

Investigation Work Plan – Final

May 2016

Former Reid Hospital Site
1401 Chester Boulevard
Richmond, Indiana

www.erm.com



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1.0 INTRODUCTION & BACKGROUND

1.1 INTRODUCTION

On behalf of Reid Health (Reid), Environmental Resources Management (ERM) is pleased to submit this Investigation Work Plan (IWP) to the Indiana Brownfields Program (IBP) to summarize a proposed scope of work at the former Reid Hospital site located at 1401 Chester Boulevard in Richmond, Indiana (hereinafter the “Site”). This IWP has been prepared based on previously prepared documents as well as recent discussions with the Indiana Department of Environmental Management (IDEM) and the IBP. The most recent references for the preparation of this IWP are as follows:

- *Phase I Site Investigation* (Phase I) dated February, 27, 2014 ,
- *Phase II Limited Subsurface Investigation* (Phase II) report dated August 29, 2014, and
- Discussions with IDEM via conference calls on January 8 and 27, 2016 and email correspondence on February 8 and March 1, 2016

The purpose of this work plan is to further evaluate the findings and address the data gaps identified in the Phase II to further develop the conceptual site model (CSM) consistent with IDEM’s Remediation Closure Guide (RCG) dated March 22, 2012 (with corrections through July 9, 2012). A Site plan with the Phase II subsurface investigation locations is provided as Figure 1.

1.2 SITE HISTORY & BACKGROUND

Based on the Phase I and II completed in 2014 at the Site by CardnoATC, the western half of the Site consists of 11 interconnected former hospital buildings surrounded by paved parking areas and access driveways. The buildings range from 1 to 7 stories and were constructed between 1904 and 1983 through various facility expansions. Exterior finishes of the hospital buildings include brick, concrete, glass, metal, stone, clay tile roofing, and vinyl siding. Interior portions of the buildings consist of offices, a morgue, emergency facilities, laboratories, patient rooms, restrooms, operating rooms, maintenance areas, storage rooms, a gift shop, and lobby areas.

As presented in the Phase I, a former power plant is located to the north of the vacant hospital buildings. The ground level floor of the power plant contained two water heater tanks and a deaerator tank. A maintenance building is located to the northeast of the power plant across a paved parking area. Wooded land surrounds the property with a steep south facing slope located along the northern boundary of the western portion of the property. Wooded land along

the western boundary of the northeast portion of the property consists of a hillside that slopes steeply to the east. A paved parking lot and access drive is located on the southern portion of the eastern half of the property. The rest of the eastern half of the property consists of wooded land as well as a dirt access road along the river.

An access road branches to the north to an open area of land centrally located on the eastern half of the property. According to prior reports, this area was previously used to dump construction and demolition debris. The access road continues east along the river and leads to a residence located to the east of the property.

1.3 REGULATORY STATUS AND INVESTIGATION SUMMARY

The Site is enrolled in the IBP, which retained CardnoATC to complete a Phase I ESA in February 2014. During the Phase I ESA Site walk, CardnoATC identified certain recognized environmental conditions (RECs) at the Site, which included:

- A former maintenance building;
- A suspected dry cleaning operation off-Site to the west/northwest;
- The fill area within the southern half of the property;
- A former print shop located on-Site; and
- A dumping area on the eastern portion of the property in a cleared portion of the wooded land.

To further evaluate the RECs, CardnoATC completed a Limited Subsurface Investigation (LSI), which is summarized in the Phase II, in which they excavated 10 test pits and installed 15 soil borings. A total of 15 surface soil samples, 15 subsurface soil samples, and 15 groundwater samples were collected from depths ranging from the surface to 30 feet below ground surface (ft bgs). Samples were screened in the field for the presence of total photoionizable vapors (TPVs), methane, and radioactivity. CardnoATC collected and analyzed soil and groundwater samples based on their proximity to on-Site RECs. Sample analytes included volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), priority pollutant list (PPL) metals, polychlorinated biphenyls (PCBs), dioxins, lithium (groundwater only), and radionuclides (groundwater only).

CardnoATC's findings from the Phase II ESA were as follows:

- VOCs were not found to be a suite of COCs for the Site.
- The presence of thallium and arsenic in the subsurface soil as well as arsenic in groundwater near the former maintenance building do not pose a risk to human health or the environment as long as the future use of the property is limited to non-residential use and doesn't include groundwater use.
- Historic dry cleaning operations upgradient of the Site do not appear to have adversely affected soil and groundwater.
- The presence of fill material may warrant further evaluation due to the presence of dioxins and radionuclides in soil and groundwater.
- The presence of chemicals in soil and groundwater near the former print shop do not represent a risk to human health and the environment as long as the future use of the property is limited to non-residential use and doesn't include groundwater use.
- The presence of contaminants in soil and groundwater near the former dumping area on the eastern portion of the Site in a cleared portion of wooded land may require further evaluation.

2.0 PROJECT APPROACH & DATA GAPS

The scope of work presented herein focuses on the Site areas and constituents that require further evaluation based on the future land use as well as the findings and the data gaps identified in the Phase II ESA. In addition, this scope has been tailored in anticipation that an institutional control (IC) in the form of an environmental restrictive covenant (ERC) would be placed on the property in the future to prevent, at a minimum, potable groundwater use and redevelopment under a residential land use scenario.

This IWP has been prepared consistent with current IDEM guidance including the Remediation Closure Guide (RCG) dated March 22, 2012 (with updates through July 9, 2012) and the Remediation Program Guide (RPG) dated February 2012. Analytical data collected through the implementation of this work plan will be compared to the 2016 IDEM RCG screening levels, which are consistent with EPA Region 5. Where constituents have no screening levels (eq. radiochemistry) ERM will evaluate the data based on EPA methods or other appropriate methods.

IDEM's Special Notice of Liability (SNL) letter dated May 14, 2015 specifically identifies arsenic, asbestos, chromium, thallium, lead, Aroclor 1232, petroleum aromatic hydrocarbons (PAHs), dioxin, lithium, and radionuclides as the hazardous substances documented at the Site. With the exception of asbestos, each of these potential constituents of potential concern (COC) will be further investigated through the implementation of this IWP with the intent of 1) eliminating them from being a COC, 2) establishing certain conditions whereby these materials are naturally occurring (e.g. background), or 3) resulting in the need for certain IC or remedial activities to achieve site closure.

Based on the radionuclide concentrations summarized in the Phase II, ERM believes these constituents could be from naturally occurring substances. However, there is currently insufficient Site analytical data to confirm this assertion. Section 2.1 provides a summary of the known radiological isotopes used at the Site and the licenses obtained by Reid during their option to better understand the historical use of radiological materials at the Site.

In addition, the dioxin concentrations detected in soil and groundwater, as summarized in the Phase II, were at very low toxicity endpoints relative to the toxicity equivalent factors (TEFs) published by EPA. This is a strong indication that additional analytical data, especially collected from on-Site groundwater, could result in the removal of this chemical from the COC list. However, additional analytical data is necessary to further evaluate dioxin in groundwater. Using dioxin data from the Phase II, ERM calculated the toxicity equivalence

(TEQ) concentrations based on soil and groundwater, which are summarized in Section 2.2.

2.1 RADIONUCLIDES AND RADIOACTIVE MATERIALS

Based on information provided to ERM, Reid obtained two Radiological Materials Licenses (RMLs) from the U.S. Nuclear Regulatory Commission (NRC) to possess and manage radiological isotopes for medical purposes. Based on ERM's review, two RMLs appear to have been issued to Reid. The first RML (No. 13-03284-02) was issued for activities at both the Site and the new hospital site located 1100 Reid Parkway in Richmond, Indiana. This RML is related to the possession and use of a number of isotopes that include Cobalt-57, Cesium-137, Iodine 125, and Barium-133. The second RML (No. 13-03284-03) is solely associated with the former hospital address (1401 Chester Boulevard). This RML appears to be related to a teletherapy unit and the associated Cobalt-60 source.

In October 1998, Reid requested that the NRC remove RML No. 13-03284-02 from the 1401 Chester Boulevard address. As part of this effort, Reid relocated the 8 sealed radiological sources to 1100 Reid Parkway. Leak tests were performed on 8 sealed sources (Co-57, Cs-137, Ba-133) and data provided to the NRC indicated that no sources were found to have been leaking. In addition, surveys were performed in areas where the sealed sources had been utilized. These consisted of the following areas:

- Nuclear Medicine Imaging Room;
- Nuclear Medicine Department Hot Lab;
- Nuclear Medicine Treadmill Testing Room;
- Nuclear Medicine Department attic storage room; and
- Radiation Oncology Hot Lab.

Documentation indicates that visual inspections and surveys were performed in September 2008. No survey areas were found to be in excess of background radiological readings, which is at or below 0.03 milliroentgens per hour (mR/hr). The results of 44 wipe samples collected from the areas indicated that there was no contamination present. Amendment 62 to RML No. 13-03284-02 was provided by the NRC on January 26, 2009, eliminating 1401 Chester Boulevard from the license.

In September 1999, Reid requested the permanent termination of RML No. 13-03284-03. Reid removed all radiological sources from the site and completed the appropriate final clearance surveys. Reid completed leak tests on source materials and provided radiological clearance survey data of the cobalt

teletherapy unit area. Documentation indicated that wipe tests were below the clearance level of 0.005 micro curries and surveys were less than background readings. In addition, Reid provided appropriate documentation that the cobalt source had been transferred off-Site to another licensee. Based on receipt of this information, Amendment 22 to RML No. 13-03284-03 was provided by the NRC on December 3, 1999, terminating the RML for the 1401 Chester Boulevard address.

Based on the removal and confirmation testing, residual radiological activity was well below naturally occurring background levels. As such, no further concerns were identified relative to the RMLs or post-operational activities.

In April 2005, a Phase I ESA was conducted and identified the following:

- Reid's B-Wing housed a radiological school. The Phase I ESA indicated that there was no visual evidence of chemical spills or staining in this area;
- The Tower and Service Wing housed the Radiological Department. The report indicated that the building appeared in good condition with no evidence of spills or staining.

The Phase II conducted by CardnoATC indicated the following:

- No soils were sampled for radiological constituents;
- Groundwater samples collected from the fill area (locations SB-5, SB-6, SB-7), outside of the fill area (locations SB-8 thru SB-12), and the former dumping area (locations SB-13, SB-14) were sampled for radionuclides;
- Radiological analysis included Gross alpha/beta, bismuth-214, lead-212, lead-214, Potassium-40, radium-226, radium-228, thallium-208, thallium-234, thorium-234, and uranium-235; and
- Gross alpha analysis indicated that the EPA drinking water standard of 15 pCi/L was exceeded for samples collected from locations SB-6 thru SB-14.

While the Phase II summarizes the detections of radiological constituents in groundwater, there is no discussion in the Phase II as to why these specific isotopes were analyzed. However, it is clear based on documentation following the removal of sources that no contamination was identified either on RML sources or within those survey areas where radiological sources were located.

It should be noted that the isotopes identified in groundwater samples are all decay products of naturally occurring radiological materials common in soil. There are three radiological natural decay series led by uranium-238, thorium-232, and uranium-235 that decay into the isotopes identified in groundwater

samples with the exception of potassium-40, also a naturally occurring isotope in soils. Additionally, only alpha-emitting radiation was detected in samples collected in the Phase II while the equipment and supplies licensed by Reid only emitted gamma radiation. As such, there does not appear to be a correlation between these isotopes and those used via Reid's radiological licenses. However, based on prior discussions, ERM understands that the IBP is concerned with radiological constituents used prior to the sources with RMLs. As such, ERM is proposing to collect additional data to further evaluate radionuclides on the property.

2.2 TOXICITY EVALUATION OF DIOXIN CONCENTRATIONS

Dioxins are a suite of compounds that are generally associated with the combustion of certain materials and have a complex chemistry. While IDEM doesn't have a screening level for the individual dioxin congeners, IDEM does have a published screening level for 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), which is the benchmark dioxin congener used in the evaluation of human health risk exposures.

Using US EPA's approved toxicity equivalence (TEQ) approach, ERM evaluated the Phase II dioxin data in accordance with methodology described in the US Environmental Protection Agency Document: *Recommended Toxicity Equivalence Factors (TEFs) for Human Health Risk Assessments of 2,3,7,8 Tetrachlorodibenzo-p-dioxin and Dioxin-Like Compounds* (EPA/100/R 10/005 | December 2010). A summary of the TEQ evaluation for both soil and groundwater is provided on Tables 1 and 2, respectively. The TEQ evaluation normalizes the concentrations of each dioxin congener based on toxicity relative to that of TCDD. The TEQs for each sample are calculated then summed to determine the TCDD equivalence concentration. That value is used for comparison to IDEM's TCDD screening level.

US EPA's published dioxin calculator uses a modified version of the Kaplan-Meier statistical technique and the arbitrary substitution method (the MDL, $\frac{1}{2}$ MDL, or zero) for non-detect values to assess the sensitivity of the dioxin data to non-detects. US EPA's dioxin calculator cannot be used for the analytical data summarized in the Phase II because not enough dioxin congeners were detected to permit the appropriate use of the Kaplan-Meier technique.

Table 3 summarizes the dioxin TEQ calculations for both soil and groundwater that were calculated within Tables 1 and 2. Based on the TEQ calculations, dioxins in soil are below the IDEM screening levels for TCDD if non-detects were evaluated at the MDL, $\frac{1}{2}$ the MDL, and zero. Based on discussions with the IBP, ERM and the IBP agreed that no further delineation of dioxins detected during CardnoATC's Phase II investigation is necessary. However, dioxin soil sampling is proposed near the former incinerator.

Based on the Phase II, only two dioxin compounds were detected in groundwater: 1,2,3,4,6,7,8-HpCDD and OCDD. As compared to TCDD, OCDD is much less soluble, it is not bioaccumulated as effectively, and it is toxicologically less important based on their TEFs. Given the elevated concentration of OCDD detected in SB-8 (2,600 pg/L), it is likely that the elevated OCDD concentrations were the result of highly turbid conditions in the groundwater samples. However, no turbidity measurements were collected as part of the Phase II, so the impact that the turbidity had on the dioxin concentrations cannot be evaluated.

For groundwater, the TEQ calculations indicate that dioxin exceeds IDEM screening level for TCDD if non-detects were evaluated inserted at ½ times or at the MDL. No additional evaluation of dioxin in groundwater would be required if non-detects were evaluated at zero. Based on the uncertainties relative to the dioxin groundwater from the Phase II, and the TEQ calculation, dioxin groundwater samples are proposed in this IWP and will be collected from permanent wells, and the turbidity will be monitored during collection.

2.3 SITE HYDROGEOLOGY AND HYDRAULIC COMMUNICATION

Based on the potentiometric map prepared by CardnoATC in the Phase II ESA, the hydrogeology beneath the Site appears to be dynamic and not fully characterized to understand groundwater flow. Figure 2 depicts the potentiometric interpretation from the Phase II. Multiple groundwater elevations are noted in the northern and western portion of the Site that may indicate multiple saturated units with the uppermost saturated units potentially being discontinuous. This variability makes it difficult to discern the flow patterns at the Site, and in turn, determine the downgradient receptors.

Based on survey data provided by CardnoATC, an important factor to the dynamic hydrogeology on-Site is the topographic elevation changes that occur from north to south, including an approximate 28 ft. elevation increase from SB-1 to SB-2 and then an approximate 40 ft. elevation decrease from SB-2 to SB-10. While groundwater samples collected during the Phase II ESA appear to have been taken from shallowest saturated unit at each investigation location, there is no evidence that these units are laterally continuous or in communication with one another. Further evaluation is necessary to determine the hydrogeologic CSM and flow conditions for the Site.

3.0 SCOPE OF WORK

The proposed soil boring, monitoring well, and groundwater sampling locations are depicted on Figures 3 and 4. Table 4 presents a soil and groundwater sampling and analysis matrix, which summarizes the sample identifications, the objective and rationale for the soil borings, the depths and anticipated sampling intervals, as well as the constituents that will be sampled at each proposed location.

In summary a total of 20 soil borings will be installed as part of this effort. In addition, 8 of the soil borings will be converted to permanent two-inch monitoring wells to evaluate groundwater flow and collect groundwater samples in accordance with the sample matrix presented in Table 4. Field sampling of investigative and monitoring data for the purpose of analytical testing and evaluation will be done in accordance with the Site QAPP provided in Appendix A.

3.1 ANALYTICAL SUITE

Based on the constituents requiring further evaluation from the Phase II, ERM is proposing a targeted analytical suite for analysis.

- PAHs – US EPA Test Method 8270
- Arsenic, thallium, lead and chromium – US EPA Test Method 6010
- Gross Alpha/Beta – US EPA Test Method 900.0
- Radium 226 – US EPA Test Method 903.1
- Gamma Spec – US EPA Test Method 901.1
- Dioxins/furans (all 17 compounds) – US EPA Test Method 8290
- Polychlorinated Biphenyls (PCBs) – US EPA Test Method 8082A

Laboratory analysis performed as part of this IWP will be conducted by Test America in North Canton, Ohio and Sacramento, California. Laboratory data deliverables will be prepared consistent with Section 3.9 and Table 3-A of IDEM's RCG. A copy of Test America's QAPP is provided as an attachment to ERM's QAPP in Appendix A.

3.2 SUBSURFACE UTILITY CLEARANCE

ERM will utilize the Indiana 811 one-call system to identify the public utilities in the vicinity of the proposed investigation locations at least 48 hours prior to conducting any subsurface work. ERM has also instituted a rigorous company-wide sub surface clearance (SSC) policy for all drilling activities, which includes the use of a private locator service and hand auguring the first five feet of every boring location prior to the use of powered drilling equipment.

3.3 SOIL INVESTIGATION & SAMPLING

ERM is proposing to conduct the following soil sampling activities. Samples will be collected and submitted in accordance with the soil and groundwater sample and analysis matrix in Table 4.

- Direct push drilling techniques (DPT) will be utilized to advance 20 soil borings (SB-16 through SB-35). The soil borings are numerated in a continuous manner from the previous Phase II report.
- The anticipated target depths and soil sampling intervals of the soil borings are provided on Table 4 and are based on the previous Phase II. However, these depths may vary based on field observations.
- Soil borings (SB-29 through SB-34) will be used for visual and field screening delineation of fill area boundaries, and will have no analytical samples collected.
- Soil samples will be collected continuously during hand auger and soil boring advancement. Soil descriptions will be logged in the field for stratigraphic description and screened using a Foxboro combination flame ionization (FID)/photoionization detector (PID). ERM will also screen soils for radiochemistry during the investigation work using a Ludlum Model 2350-1, which is sensitive for alpha, beta, gamma, and/or neutron radiation. Soil samples will also be visually inspected in the field for the potential presence of impacts.
- Based on observation of potential impacts, a representative aliquot of soil will be removed from each boring and screened using the methods described above. Samples will be stored on ice using laboratory supplied containers and subsequently submitted for analysis. Additional samples may be collected if field conditions warrant.
- Soils not selected for analysis will be containerized in 55 gallon drums and labeled as investigative derived waste (IDW) pending analysis.

- ERM will collect 1 duplicate and 1 matrix spike/matrix spike duplication (MS/MSD) soil sample for every 20 samples in accordance with the attached QAPP and submit it to Test America for analysis for quality assurance and control (QA/QC) purposes. In addition, ERM will collect two decontamination rinsate blanks and two field equipment blanks during the drilling activities.
- Upon completion of the work (including effort included in Section 3.4) at each investigation location, the core holes will be permanently abandoned in a manner consistent with Part 2 of IDEM's *Drilling Procedures and Monitoring Well Construction Guidelines*—Non-rule Policy Document W-0053.
- Containerized soil cuttings generated during the investigation activities will be placed into 55-gallon drums, which will be labeled with pending analysis. For the purposes of this investigation, it is assumed that 1 drum of soil will be generated as part of this effort. ERM will work with Reid to identify a temporary staging area for this IDW until it is properly profiled through a waste disposal facility.
- While ERM is not anticipating the observation or detection of elevated PID measurements, ERM will be prepared to collect samples for VOCs if field observations indicate the presence of organic vapors. Samples would be submitted to the project laboratory using US EPA Test Method 8260 and preparation method 5035.

3.4 GROUNDWATER SAMPLING & HYDROGEOLOGY EVALUATION

To further evaluate groundwater flow across the Site, ERM will install 8 permanent two-inch monitoring wells at locations across the Site. Monitoring wells will be installed in accordance with IDEM's *Drilling Procedures and Monitoring Well Construction Guidelines* (IDEM, 2009B). Groundwater samples will be collected and submitted in accordance with the soil and groundwater sample and analysis matrix in Table 4 as well as the QAPP in Appendix A.

- Each well will be constructed of two-inch diameter schedule 40 PVC materials and equipped with a five-foot long, #10 slot screen.
- The wells will be installed using a 20/40 grade "prepack" sand filter pack. All wells will have a 20-40 grade sand filter pack emplaced to approximately two feet above each screen.
- Bentonite chips and/or slurry will then be added to 0.5 feet below grade. The monitoring wells will be installed with the PVC riser above grade.

- Flush-mounted well covers will be used to protect the permanent wells installed in asphalt. Prograde well covers will be used for the locations in the woods.
- To develop the wells, the driller will surge block the wells and remove a minimum of five well volumes. The purge water shall be containerized in a properly labeled 55-gallon drum.
- Groundwater samples will be collected for the parameters and at the locations identified on Table 4 using a low flow pump and purged into laboratory supplied containers.
- Groundwater quality will be monitored during monitoring well sampling for dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, conductivity, and turbidity.
- Groundwater samples will be submitted in a cooler on ice under strict chain of custody procedures.
- ERM will collect 1 duplicate and 1 MS/MSD sample for QA/QC analysis in accordance with the attached QAPP.
- Purge water in 55 gallon drums will be staged at a location on-site for profiling and disposal at a later date.

3.5 WIPE SAMPLING

As of the date of this IWP, ERM representatives have not entered the on-Site buildings to inspect the interior structures or relic equipment that may still remain on-Site. Based on discussions with the IBP on January 8, 2016, ERM understands that IDEM would like to identify potential sources of contaminants in the building that may contain PCBs. The areas of the Site buildings that have been discussed as potentially requiring wipe sampling are depicted on Figures 3, 5 and 6 and include areas where potential incineration and hydraulic equipment or radiological operations previously occurred.

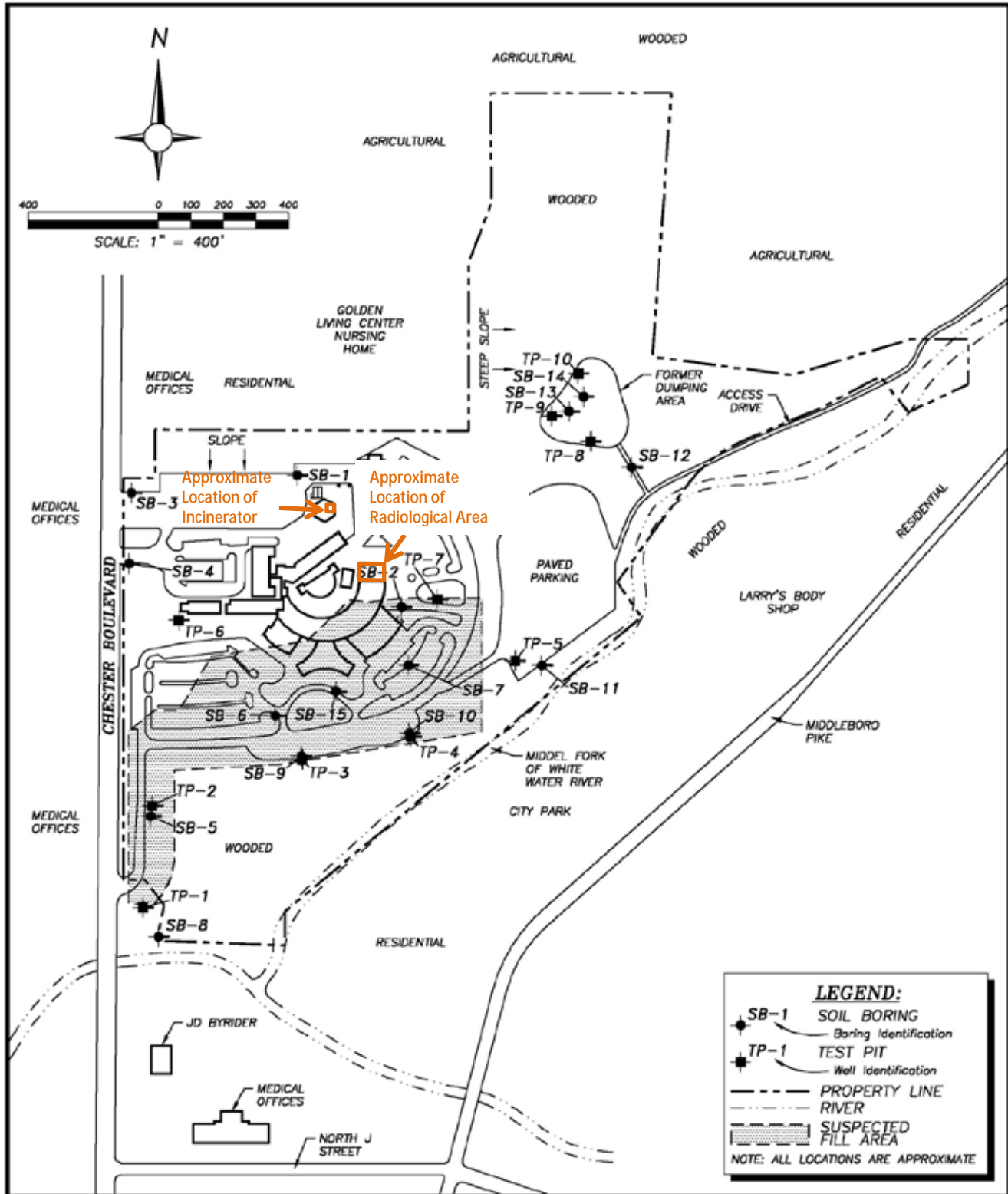
If ERM can safely and securely gain access to the buildings, ERM will inspect areas of the building and collect wipe samples for analysis of PCBs. ERM anticipates collecting up to 10 wipe samples during this effort, assuming safe access. These samples will be submitted for PCB analysis in accordance with 40 CFR 761.123. If ERM cannot safely access the buildings, the ERM project manager will notify IDEM of the safety concerns the day the access areas are inspected.

3.6 INVESTIGATION SUMMARY REPORT AND UPDATED CSM

Upon completion of the investigation effort proposed herein, ERM will prepare a summary report to summarize the work and discuss the findings of the investigation. A primary focus of the report will be to update the current CSM consistent with IDEM's RCG to make a determination on what, if any, future investigation and/or remediation is necessary. ERM anticipates this deliverable will include text, analytical data tables, groundwater elevation information, and figures including cross-sections depicting information from the Phase II ESA as well as additional information obtained during the investigation proposed herein.

Of note from recent discussions, ERM and IDEM acknowledged that there is no approved method set forth in guidance or rule by IDEM or EPA to evaluate non-detect dioxin analytical data with the TEQ calculation. Therefore both entities agreed that future dioxin evaluation at the Site would be conducted i) consistent with the TEFs published within USEPA Guidance dated December 2010 and ii) non-detects would be evaluated at ½ the MDL. The turbidity of the groundwater samples will be taken into consideration during the analysis of the TEQs.

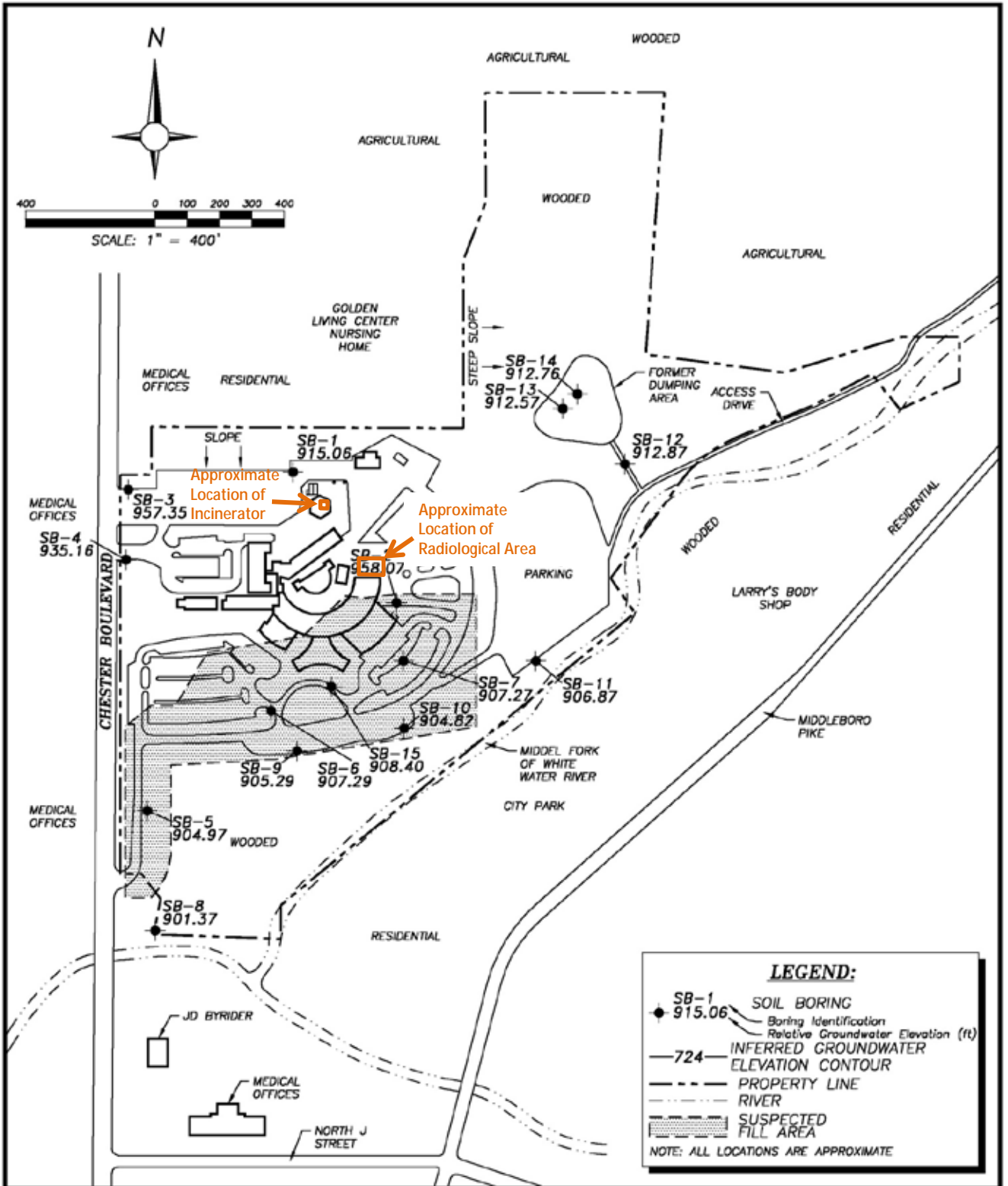
Figures



Site Plan with Phase II Investigation Locations

*Figure obtained from, "Cardno ATC Phase II Limited Subsurface Investigation" Report dated August 29, 2014.

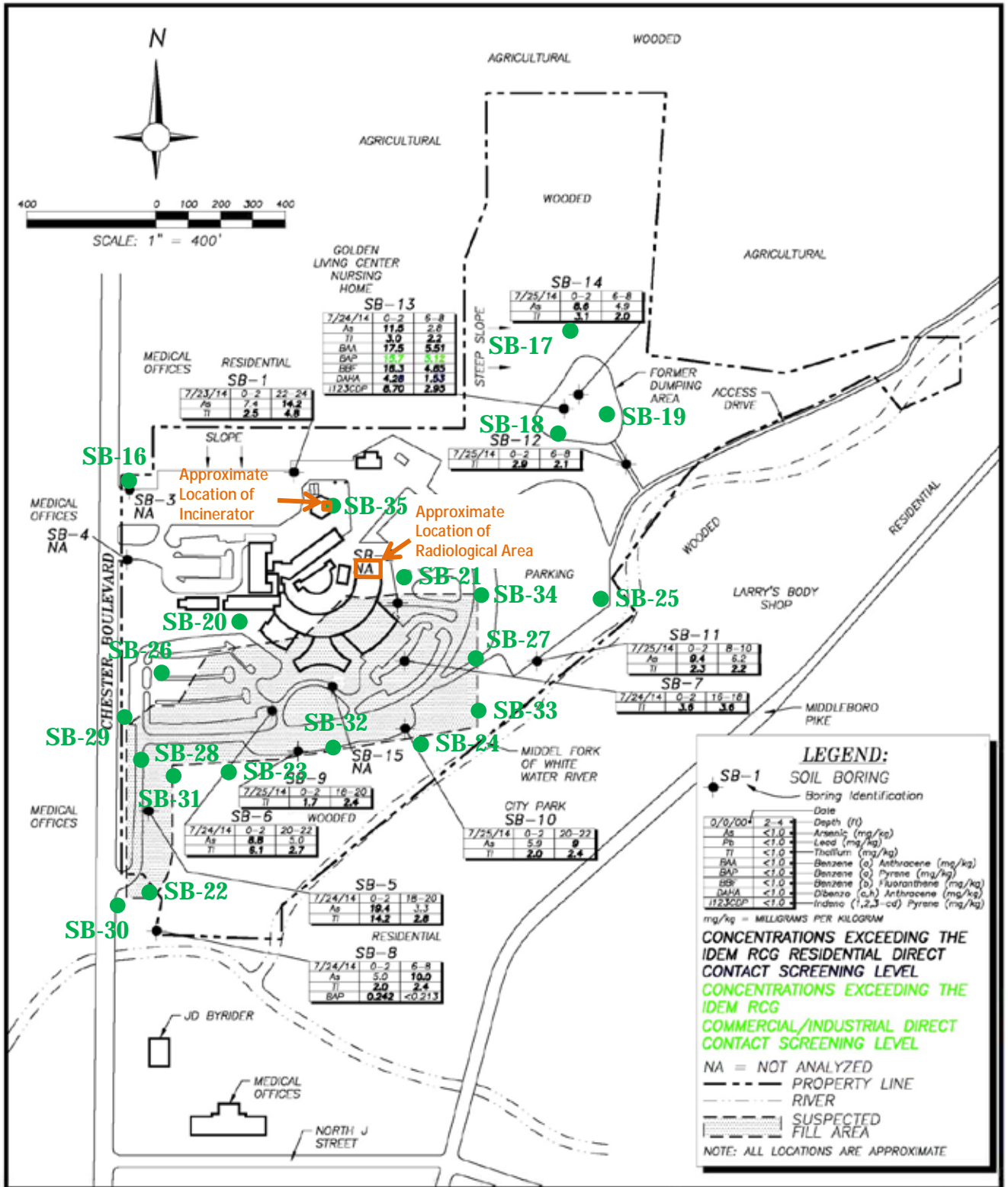
Figure 1



Potentiometric Map

*Figure obtained from, "Cardno ATC Phase II Limited Subsurface Investigation" Report dated August 29, 2014.

Figure 2

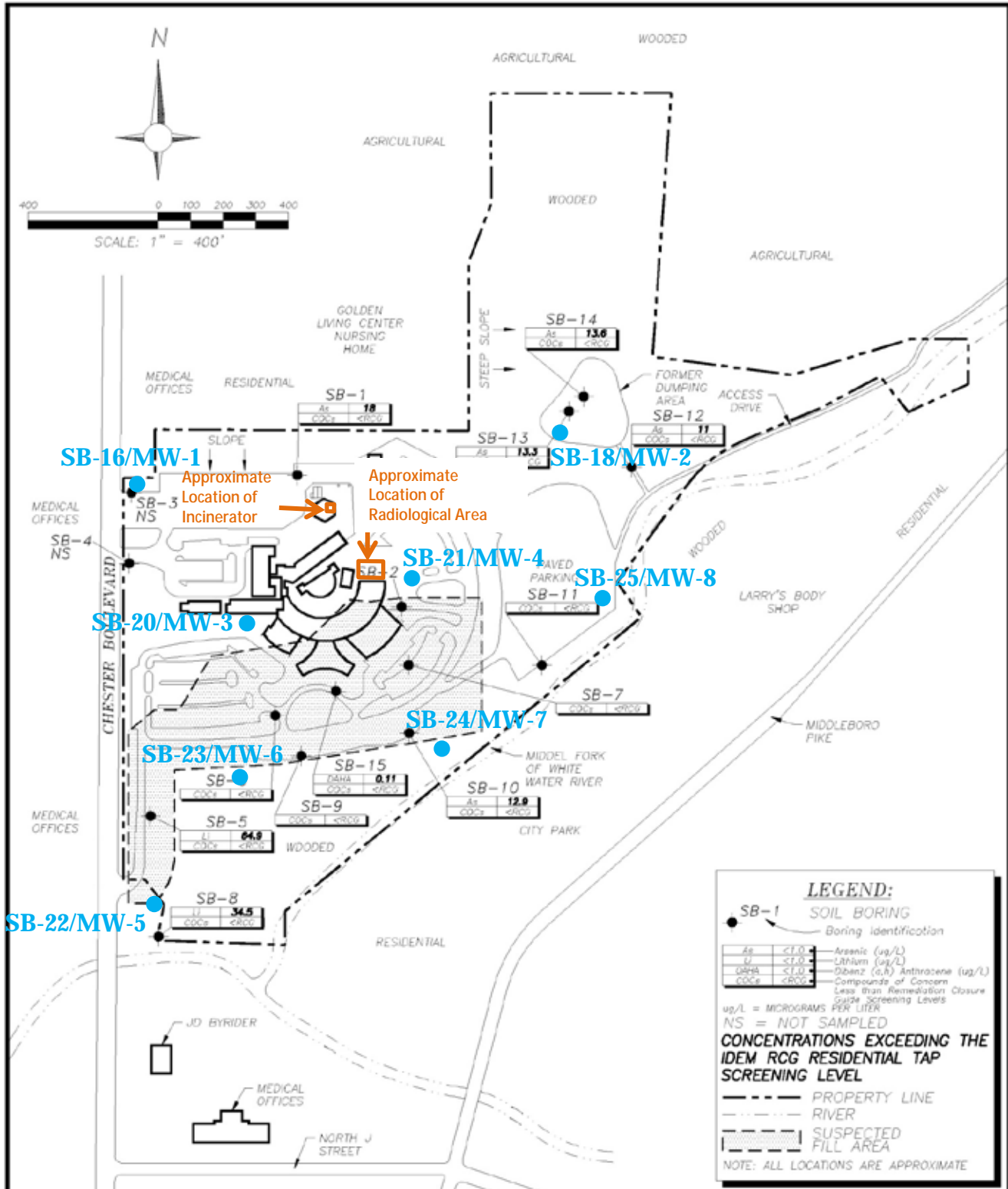


Proposed Soil Investigation Locations

- Proposed Soil Boring Locations

*Figure obtained from, "Cardno ATC Phase II Limited Subsurface Investigation" Report dated August 29, 2014.

Figure 3

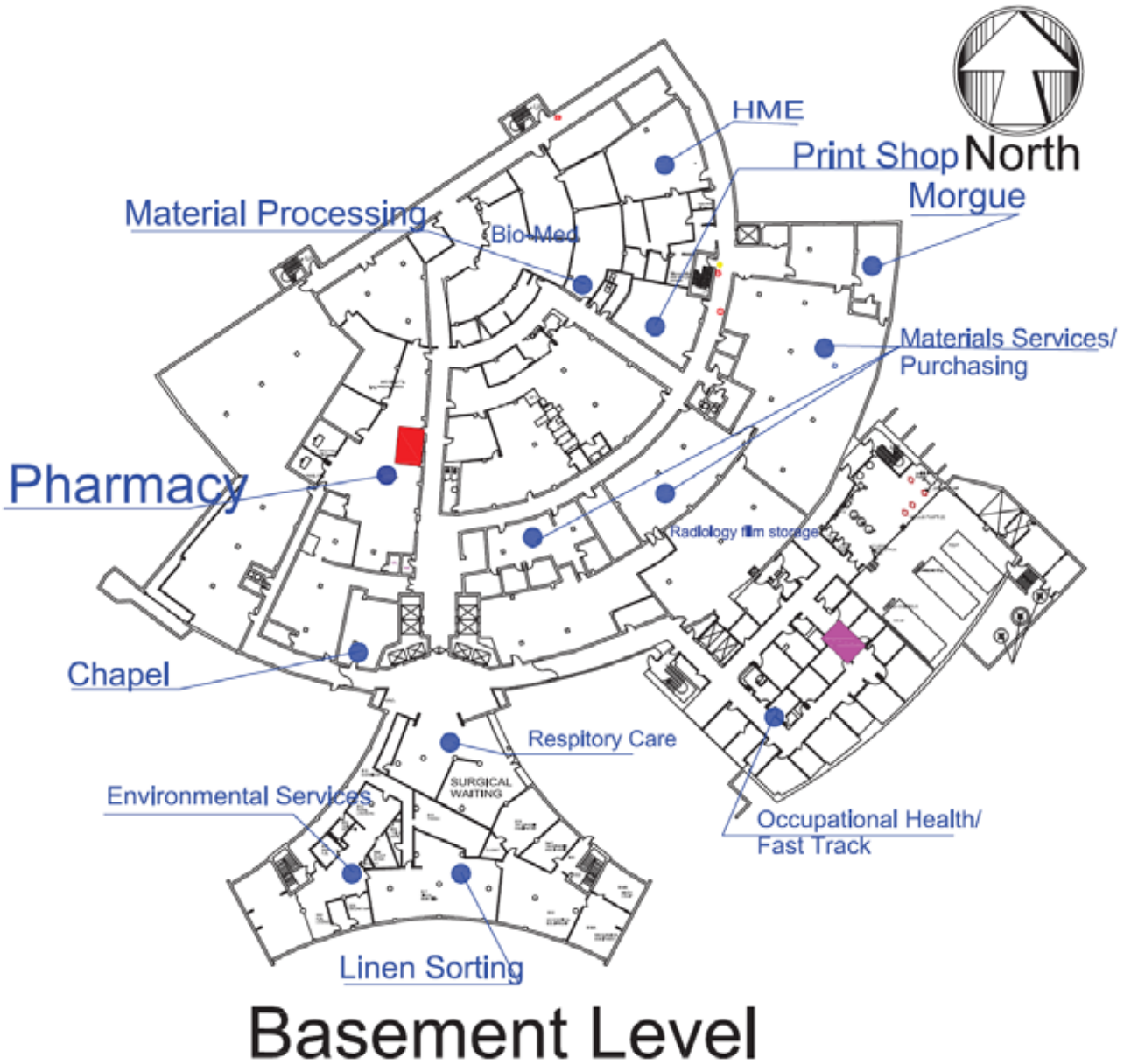


Proposed Groundwater Investigation Locations

● Proposed Groundwater Monitoring/Sample Location

*Figure extracted from, "Cardno ATC Phase II Limited Subsurface Investigation" Report dated August 29, 2014.

Figure 4



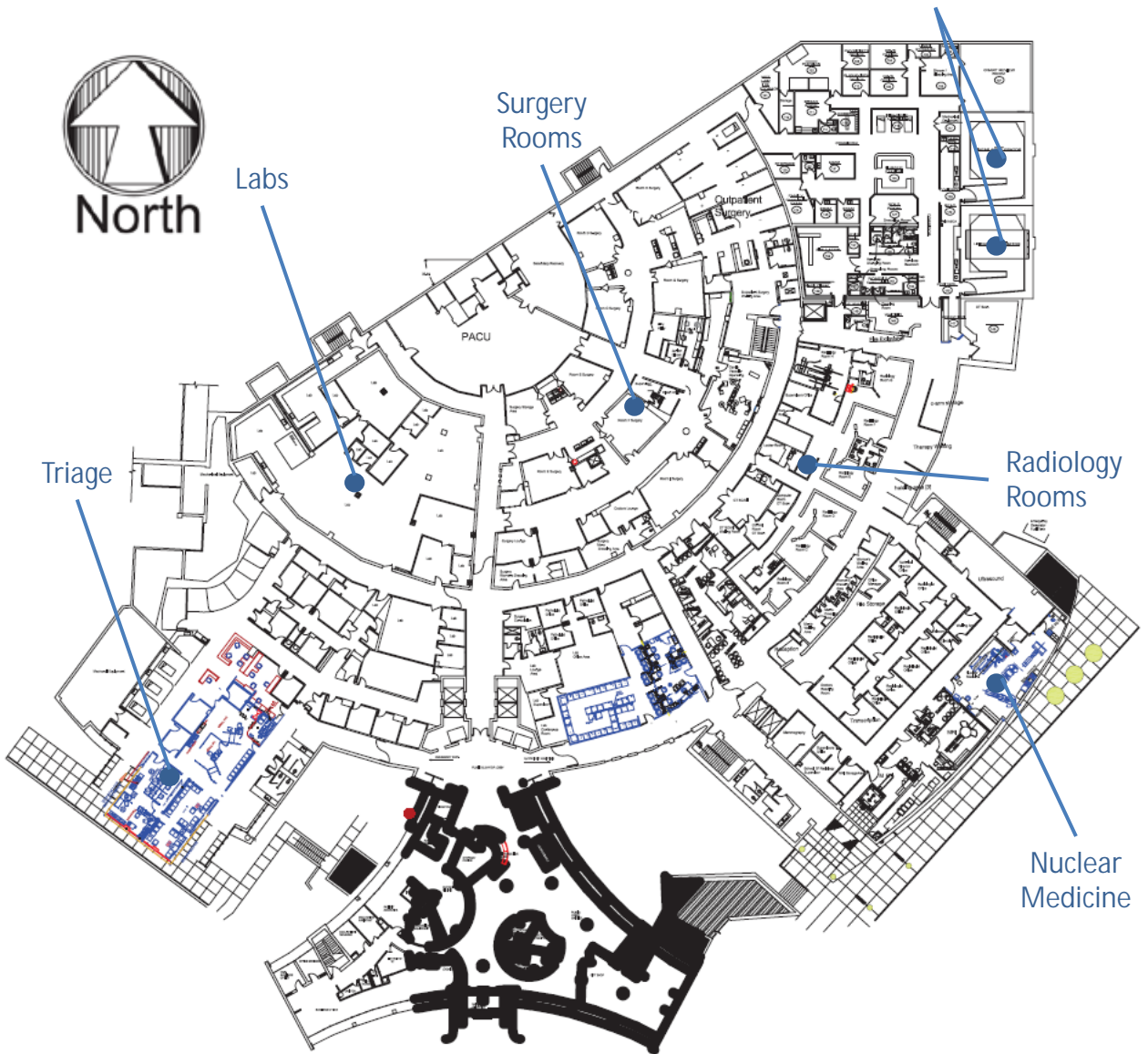
PCB Sample Locations

**Figure obtained from,
 "Cardno ATC Phase I
 Environmental Site
 Assessment " Report dated
 February 27, 2014.*

Figure 5



Linear Accelerator
(Cobalt-60 Source identified in
Nuclear Decommissioning Report)



First Level

PCB Sample Locations

**Figure obtained and modified from, "Cardno ATC Phase I Environmental Site Assessment" Report dated February 27, 2014.*

Figure 6

Tables

TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|-----------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-5 (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 1 | J | 1 | 1 | 1 | 1 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDD | 3268-87-9 | 300 | | 0.0003 | 0.09 | 0.09 | 0.09 |
| | 2,3,7,8-TCDF | 51207-31-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 7.6 | | 0.3 | 2.28 | 2.28 | 2.28 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 45 | | 0.01 | 0.45 | 0.45 | 0.45 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 42 | | 0.01 | 0.42 | 0.42 | 0.42 |
| | OCDF | 39001-02-0 | 39 | | 0.0003 | 0.0117 | 0.0117 | 0.0117 |
| SB-5 (0-2) TEQ | | | | | | 13.4517 | 8.8517 | 4.2517 |
| SB-6 (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 66 | | 0.01 | 0.66 | 0.66 | 0.66 |
| | OCDD | 3268-87-9 | 1100 | | 0.0003 | 0.33 | 0.33 | 0.33 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1.7 | | 0.1 | 0.17 | 0.17 | 0.17 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 6.3 | | 0.3 | 1.89 | 1.89 | 1.89 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 7.6 | | 0.1 | 0.76 | 0.76 | 0.76 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 6.3 | | 0.1 | 0.63 | 0.63 | 0.63 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 15 | | 0.01 | 0.15 | 0.15 | 0.15 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDF | 39001-02-0 | 14 | | 0.0003 | 0.0042 | 0.0042 | 0.0042 |
| SB-6 (0-2) TEQ | | | | | | 13.2942 | 8.9442 | 4.5942 |

TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|------------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-7 (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 8.9 | | 0.01 | 0.089 | 0.089 | 0.089 |
| | OCDD | 3268-87-9 | 80 | | 0.0003 | 0.024 | 0.024 | 0.024 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 | |
| SB-7 (0-2) TEQ | | | | | | 11.466 | 5.7895 | 0.113 |
| SB-13 (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 6.7 | U | 1 | 6.7 | 3.35 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 43 | | 0.01 | 0.43 | 0.43 | 0.43 |
| | OCDD | 3268-87-9 | 450 | | 0.0003 | 0.135 | 0.135 | 0.135 |
| | 2,3,7,8-TCDF | 51207-31-9 | 8.6 | U | 0.1 | 0.86 | 0.43 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 14 | | 0.01 | 0.14 | 0.14 | 0.14 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| OCDF | 39001-02-0 | 18 | | 0.0003 | 0.0054 | 0.0054 | 0.0054 | |
| SB-13 (0-2) TEQ | | | | | | 18.4704 | 9.5904 | 0.7104 |

TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|---------------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-5 Dup (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 2.6 | | 1 | 2.6 | 2.6 | 2.6 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 11 | | 1 | 11 | 11 | 11 |
| * EMPC | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 6.9 | | 0.1 | 0.69 | 0.69 | 0.69 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 15 | | 0.1 | 1.5 | 1.5 | 1.5 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 7.8 | | 0.1 | 0.78 | 0.78 | 0.78 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 97 | | 0.01 | 0.97 | 0.97 | 0.97 |
| | OCDD | 3268-87-9 | 540 | | 0.0003 | 0.162 | 0.162 | 0.162 |
| *EMPC | 2,3,7,8-TCDF | 51207-31-9 | 29 | | 0.1 | 2.9 | 2.9 | 2.9 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 35 | | 0.03 | 1.05 | 1.05 | 1.05 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 67 | | 0.3 | 20.1 | 20.1 | 20.1 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 62 | | 0.1 | 6.2 | 6.2 | 6.2 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 41 | | 0.1 | 4.1 | 4.1 | 4.1 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 62 | | 0.1 | 6.2 | 6.2 | 6.2 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 14 | | 0.1 | 1.4 | 1.4 | 1.4 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 250 | | 0.01 | 2.5 | 2.5 | 2.5 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 9.3 | | 0.01 | 0.093 | 0.093 | 0.093 |
| | OCDF | 39001-02-0 | 57 | | 0.0003 | 0.0171 | 0.0171 | 0.0171 |
| SB-5 Dup (0-2) TEQ | | | | | | 62.2621 | 62.2621 | 62.2621 |
| SB-14 (0-2) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 13 | | 0.01 | 0.13 | 0.13 | 0.13 |
| | OCDD | 3268-87-9 | 120 | | 0.0003 | 0.036 | 0.036 | 0.036 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 |
| SB-14 (0-2) TEQ | | | | | | 11.519 | 5.8425 | 0.166 |

TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|-------------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-5 (18-20) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDD | 3268-87-9 | 18 | | 0.0003 | 0.0054 | 0.0054 | 0.0054 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 |
| SB-5 (18-20) TEQ | | | | | | 11.4084 | 5.7069 | 0.0054 |
| SB-6 (20-22) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDD | 3268-87-9 | 51 | | 0.0003 | 0.0153 | 0.0153 | 0.0153 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 |
| SB-6 (20-22) TEQ | | | | | | 11.4183 | 5.7168 | 0.0153 |

TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|-------------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-7 (16-18) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 7.9 | | 0.01 | 0.079 | 0.079 | 0.079 |
| | OCDD | 3268-87-9 | 110 | | 0.0003 | 0.033 | 0.033 | 0.033 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 | |
| SB-7 (16-18) TEQ | | | | | | 11.465 | 5.7885 | 0.112 |
| SB-13 (6-8) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 12 | | 0.1 | 1.2 | 1.2 | 1.2 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 480 | | 0.01 | 4.8 | 4.8 | 4.8 |
| | OCDD | 3268-87-9 | 8700 | | 0.0003 | 2.61 | 2.61 | 2.61 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 49 | | 0.01 | 0.49 | 0.49 | 0.49 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| OCDF | 39001-02-0 | 340 | | 0.0003 | 0.102 | 0.102 | 0.102 | |
| SB-13 (6-8) TEQ | | | | | | 20.002 | 14.602 | 9.202 |

**TABLE 1
TOXICITY EQUIVALENCE CALCULATIONS FOR DIOXINS IN SOIL
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA**

| Sample | Congener Name | CAS Number | Result (ng/Kg) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|------------------------|---------------------|------------|----------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-14 (6-8) | 2,3,7,8-TCDD | 1746-01-6 | 1 | U | 1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 5 | U | 1 | 5 | 2.5 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDD | 3268-87-9 | 24 | | 0.0003 | 0.0072 | 0.0072 | 0.0072 |
| | 2,3,7,8-TCDF | 51207-31-9 | 1 | U | 0.1 | 0.1 | 0.05 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 5 | U | 0.03 | 0.15 | 0.075 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 5 | U | 0.3 | 1.5 | 0.75 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 5 | U | 0.1 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 5 | U | 0.01 | 0.05 | 0.025 | 0 |
| | OCDF | 39001-02-0 | 10 | U | 0.0003 | 0.003 | 0.0015 | 0 |
| SB-14 (6-8) TEQ | | | | | | 11.4102 | 5.7087 | 0.0072 |

Notes:
 TEF = Toxicity Equivalent Factors
 TEQ = Toxicity Equivalence
 ND = No detection
 MDL = Method Detection Limit
 All values in nanograms per kilogram (ng/Kg)
 EMPC = Estimated Maximum Possible Concentration
 DCSL = Direct Contact Screening Level
 MTG = Migration to Groundwater
 TEFs obtained from US EPA's *Recommended Toxicity Equivalence Factors for Human Health Assessments of 2,3,7,8-Tetrachlorodibenzo-p-dioxin and Dioxin-Like Compounds* dated December 2010

| | | | |
|-------------------------------|------------|------------|------------|
| Average TEQ (all samples) | 11.41 | 5.71 | 0.01 |
| Average TEQ (shallow samples) | 21.74 | 16.88 | 12.02 |
| Average TEQ (deep samples) | 13.14 | 7.50 | 1.87 |
| Indiana Residential DCSL | 69 | 69 | 69 |
| Indiana Commercial DCSL | 220 | 220 | 220 |
| Indiana Residential MTG | 300 | 300 | 300 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) |
|---------------------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-5 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 150 | | 0.0003 | 0.045 | 0.045 | 0.045 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| SB-5 TEQ | | | | | | 114.075 | 57.06 | 0.045 |
| SB-5 Dup | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| SB-5 Dup TEQ | | | | | | 114.06 | 57.03 | 0 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) |
|-----------------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-6 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-6 TEQ | | | | | | 114.06 | 57.03 | 0 |
| SB-7 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-7 TEQ | | | | | | 114.06 | 57.03 | 0 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) |
|-----------------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-8 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 150 | | 0.01 | 1.5 | 1.5 | 1.5 |
| | OCDD | 3268-87-9 | 2,600 | | 0.0003 | 0.78 | 0.78 | 0.78 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-8 TEQ | | | | | | 115.81 | 59.045 | 2.28 |
| SB-9 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-9 TEQ | | | | | | 114.06 | 57.03 | 0 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) |
|------------------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-10 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-10 TEQ | | | | | | 114.06 | 57.03 | 0 |
| SB-11 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| SB-11 TEQ | | | | | | 114.06 | 57.03 | 0 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) |
|------------------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|
| SB-12 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| SB-12 TEQ | | | | | | 114.06 | 57.03 | 0 |
| SB-13 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 |
| | OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 |
| SB-13 TEQ | | | | | | 114.06 | 57.03 | 0 |

TABLE 2
TOXICITY EQUIVALENCE CALCULATION FOR DIOXINS IN GROUNDWATER
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Congener Name | CAS Number | Result (pg/L) | Qualifier | TEF | TEQ (ND = MDL) | TEQ (ND = 1/2 mdl) | TEQ (ND = 0) | |
|-----------|---------------------|------------|---------------|-----------|--------|-------------------|-----------------------|-----------------|----------|
| SB-14 | 2,3,7,8-TCDD | 1746-01-6 | 10 | U | 1 | 10 | 5 | 0 | |
| | 1,2,3,7,8-PeCDD | 40321-76-4 | 50 | U | 1 | 50 | 25 | 0 | |
| | 1,2,3,4,7,8-HxCDD | 39227-28-6 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,6,7,8-HxCDD | 57653-85-7 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,7,8,9-HxCDD | 19408-74-3 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | 50 | U | 0.01 | 0.5 | 0.25 | 0 | |
| | OCDD | 3268-87-9 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| | 2,3,7,8-TCDF | 51207-31-9 | 10 | U | 0.1 | 1 | 0.5 | 0 | |
| | 1,2,3,7,8-PeCDF | 57117-41-6 | 50 | U | 0.03 | 1.5 | 0.75 | 0 | |
| | 2,3,4,7,8-PeCDF | 57117-31-4 | 50 | U | 0.3 | 15 | 7.5 | 0 | |
| | 1,2,3,4,7,8-HxCDF | 70648-26-9 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,6,7,8-HxCDF | 57117-44-9 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 2,3,4,6,7,8-HxCDF | 72918-21-9 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,7,8,9-HxCDF | 60851-34-5 | 50 | U | 0.1 | 5 | 2.5 | 0 | |
| | 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | 50 | U | 0.01 | 0.5 | 0.25 | 0 | |
| | 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | 50 | U | 0.01 | 0.5 | 0.25 | 0 | |
| | OCDF | 39001-02-0 | 100 | U | 0.0003 | 0.03 | 0.015 | 0 | |
| | SB-14 TEQ | | | | | | 114.06 | 57.03 | 0 |

Notes:

TEF = Toxicity Equivalent Factors

TEQ = Toxicity Equivalence

ND = No detection

MDL = Method Detection Limit

All values in picograms per liter (pg/L)

MCL = Maximum Contaminant Level

TEFs obtained from US EPA's *Recommended Toxicity Equivalence Factors for Human Health Assessments of 2,3,7,8-Tetrachlorodibenzo-p-dioxin and Dioxin-Like Compounds* dated December 2010

| | | | |
|---------------------|---------------|--------------|-------------|
| Average TEQ | 114.22 | 57.22 | 0.21 |
| Indiana/ US EPA MCL | 30 | 30 | 30 |

TABLE 3
DIOXIN TOXICITY EQUIVALENCE CALCULATIONS SUMMARY TABLE
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Media | Reference | TEQ (ND = MDL) | TEQ (ND = 1/2 MDL) | TEQ (ND = 0) |
|-------------|-------------------------------|-------------------|-----------------------|-----------------|
| Groundwater | Average TEQ | 114.22 | 57.22 | 0.21 |
| | Indiana/US EPA MCL | 30 | 30 | 30 |
| | | | | |
| Soil | Average TEQ (all samples) | 11.41 | 5.71 | 0.01 |
| | Average TEQ (shallow samples) | 21.74 | 16.88 | 12.02 |
| | Average TEQ (deep samples) | 13.14 | 7.50 | 1.87 |
| | Indiana Residential DCSL | 69 | 69 | 69 |
| | Indiana Commercial DCSL | 220 | 220 | 220 |
| | Indiana Residential MTG | 300 | 300 | 300 |

Notes:

ND= Non-detect

MDL= Method detection limit

TEQ= Toxicity equivalence

DCSL= Direct contact screening level

MTG= Migration to groundwater

MCL= Maximum contaminant level

Values in picograms per liter (pg/L) for groundwater and nanograms per kilogram (ng/Kg) for soil

Calculations based on US EPA's *Recommended Toxicity Equivalence Factors (TEFs) for Human Health Risk**Assessments of 2,3,7,8-Tetrachlorodibenzo-p-dioxin and Dioxin-Like Compounds* - December 2010

TABLE 4
SOIL AND GROUNDWATER SAMPLING AND ANALYSIS MATRIX
FORMER REID HOSPITAL SITE
RICHMOND, INDIANA

| Sample ID | Investigation Objective | Proposed Sample Location Details | | | | Soil | | | | | | | | | Groundwater | | | | | | | | |
|------------|--|---|------------------------|---|---|---------|----------|------|----------|---------|---------------|---------|------|------|-------------|----------|----------|---------------|---------|--------|------|------|----------------------------|
| | | Approximate Total Boring Depth (ft bgs) | Number of Soil Samples | Anticipated Soil Sampling Depth Interval (ft bgs) | Anticipated Groundwater Sample Depth (ft bgs) | Arsenic | Chromium | Lead | Thallium | Dioxins | Radionuclides | Lithium | PAHs | PCBs | Arsenic | Chromium | Thallium | Radionuclides | Lithium | Dioxin | PAHs | PCBs | Groundwater Flow Direction |
| SB-16/MW-1 | Upgradient/Offsite Baseline Data and Groundwater Flow | 25 | 2 | 0 to 2, 15 to 20 | 20 to 25 | X | X | X | X | | X | X | X | | X | X | X | X | X | X | X | | X |
| SB-17 | Upgradient/Offsite Baseline Data and Groundwater Flow | 15 | 2 | 0 to 2, 6 to 15 | NS | X | X | X | X | | X | X | X | | | | | | | | | | |
| SB-18/MW-2 | Delineation of PAHs in Dumping Area | 15 | 2 | 0 to 2, 6 to 8 | 10 to 15 | X | | X | X | | X | X | X | | X | X | X | X | X | X | X | | X |
| SB-19 | Delineation of PAHs in Dumping Area | 15 | 2 | 0 to 2, 6 to 8 | NS | X | | X | X | | X | X | X | | | | | | | | | | |
| SB-20/MW-3 | Radionuclide Investigation and Groundwater Flow | 30 | 0 | NS | 20 to 30 | | | | | | | | | | X | X | X | X | X | X | X | | X |
| SB-21/MW-4 | Radionuclide Investigation and Groundwater Flow | 30 | 0 | NS | 20 to 30 | | | | | | | | | | X | X | X | X | X | X | X | | X |
| SB-22/MW-5 | Radionuclide & PCB Evaluation - Groundwater Flow | 10 | 2 | 0 to 2 (fill), 2 to 10 (below fill) | 5 to 10 | X | | X | X | | X | X | X | X | X | X | X | X | X | X | X | X | X |
| SB-23/MW-6 | Groundwater Flow | 25 | 2 | 0 to 2 (fill), 10 to 20 (below fill) | 20 to 25 | | | | | | X | X | | | X | X | X | X | X | X | X | | X |
| SB-24/MW-7 | Groundwater Flow & Characterization | 30 | 2 | 0 to 2 (fill), 10 to 20 (below fill) | 20 to 30 | | | | | | X | X | | | X | X | X | X | X | X | X | | X |
| SB-25/MW-8 | Groundwater Flow & Characterization | 15 | 2 | 0 to 2 (fill), 10 to 15 (below fill) | 10 to 15 | | | | | | X | X | | | X | X | X | X | X | X | X | | X |
| SB-26 | Delineation of Fill Material | 25 | 2 | 0 to 2 (fill), 10 to 20 (below fill) | NS | | | | | | X | X | | | | | | | | | | | |
| SB-27 | Delineation of Fill Material | 25 | 2 | 0 to 2 (fill), 10 to 20 (below fill) | NS | | | | | | X | X | | | | | | | | | | | |
| SB-28 | Delineate/Confirm Lead and PCBs | 20 | 2 | 0 to 2 (fill), 10 to 20 (below fill) | NS | X | | X | X | | X | X | | X | | | | | | | | | |
| SB-29 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-30 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-31 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-32 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-33 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-34 | Visual and field screening delineation of fill area boundaries | 13 | NS | NS | NS | | | | | | | | | | | | | | | | | | |
| SB-35 | Investigate Former Incinerator | 20 | 2 | 0 to 2, 10 to 20 | NS | | | | X | X | X | X | | | | | | | | | | | |

Notes:
 SB = Soil Boring
 ft bgs = feet below ground surface
 NS = No sample anticipated
 All samples submitted for laboratory analysis will be analyzed by the laboratory using standard US EPA Test Methods
 Anticipated depths are estimated based on boring logs and findings from the Phase II ESA
 Table does not summarize wipe samples or surveys that will be conducted as part of the SIWP implementation
 Additional soil and/or groundwater samples may be collected if conditions or field observations warrant

Appendix A

Title and Approval Page

QUALITY ASSURANCE PROJECT PLAN

**Former Reid Hospital Site
1401 Chester Boulevard
Richmond, Indiana 47374**

May 2016

ERM Project No. 0315592

John Markey, ERM, *Partner* *Date*

Aaron Friedrich, L.P.G. (IN# 2254), ERM, *Project Manager* *Date*

Andrea Robertson, Indiana Brownfield Program, *Project Manager* *Date*

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| Appendix C | <i>Sample Field Forms</i> |

LIST OF ACRONYMS AND ABBREVIATIONS

| | |
|----------|--|
| AOC | Area of Concern |
| COC | Chemical of concern |
| DQO | Data quality objective |
| DTW | Depth to water |
| EDD | Electronic data deliverable |
| ERM | Environmental Resources Management, Inc. |
| FID | Flame ionization detector |
| H&S | Health and safety |
| IDEM | Indiana Department of Environmental Management |
| MDL | Method detection limit |
| MPC | Measurement performance criteria |
| MS | Matrix spike |
| MSD | Matrix spike duplicate |
| PAH | Polycyclic aromatic hydrocarbon |
| PCB | Polychlorinated biphenyl |
| PF | Power factor |
| PIC | Partner-in-charge |
| PID | Photoionization detector |
| PM | Project manager |
| QA | Quality assurance |
| QAM | Quality assurance manager |
| QAPP | Quality Assurance Project Plan |
| QC | Quality control |
| RCG | Remediation Closure Guide |
| RCRA | Resource Conservation and Recovery Act |
| REID | Reid Health |
| RL | Reporting limit |
| RPD | Relative percent difference |
| RWP | Remediation Work Plan |
| SIWP | Site Investigation Work Plan |
| SL | Screening level |
| SOP | Standard operating procedure |
| SVOC | Semi-volatile organic compound |
| TA | Test America, Inc. |
| TOC | Top of casing |
| UFP-QAPP | Uniform Federal Policy for Quality Assurance Project Plans |
| USEPA | United States Environmental Protection Agency |
| VOC | Volatile organic compound |
| VRA | Voluntary Remediation Agreement |
| VRP | Voluntary Remediation Program |

1.0 INTRODUCTION AND SITE HISTORY

1.1 INTRODUCTION

Environmental Resources Management, Inc. (ERM) has developed this Quality Assurance Project Plan (QAPP) on behalf of Reid Health (Reid), for subsurface investigation activities at the former Reid Hospital site located at 1401 Chester Boulevard in Richmond, Indiana (Site). The subsurface investigation work aims to further evaluate the findings from previous investigation work including the work presented in the *Phase II Limited Subsurface Investigation* (Phase II) dated August 29, 2014. The Phase II was completed with oversight from the Indiana Brownfields Program (IBP).

This QAPP presents the organization, objectives, functional activities, and quality assurance (QA) / quality control (QC) procedures associated with the Site investigation activities to be implemented. This plan also addresses specific protocols for sampling, sample handling, sample storage, chain-of-custody procedures, and field and laboratory analyses. Investigation-specific work plans will be completed as separate documents as field investigations are planned and identified. Each work plan will provide details pertaining only to that investigation.

The format of the document generally follows the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) guidance. This QAPP is being submitted as **Appendix A** of the Site Investigation Work Plan (IWP). The QAPP will be updated, if necessary, and referenced during the implementation of any future Site investigation work.

1.2 SITE HISTORY AND BACKGROUND

Based on the Phase I and II completed in 2014 at the Site by CardnoATC, the western half of the Site consists of eleven interconnected hospital buildings surrounded by paved parking areas and access drives. The buildings range from 1 to 7 stories and were constructed between 1904 and 1983 through various facility expansions. Exterior finishes of the hospital buildings include brick, concrete, glass, metal, stone, clay tile roofing, and vinyl siding. Interior portions of the buildings consist of offices, a morgue, emergency room areas, laboratories, radiology imaging rooms, patient rooms, restrooms, operating rooms, maintenance areas, storage rooms, a gift shop, and lobby areas.

As presented in the Phase I, a former power plant is located to the north of the vacant hospital buildings. A maintenance building is located to the northeast of the power plant across a paved parking area. Wooded land surrounds the property with a steep southward slope located along the northern boundary of the western portion of the property. Wooded land along the western boundary

of the northeast portion of the property consists of a hillside that slopes steeply to the east. A paved parking lot and access drive is located on the southern portion of the eastern half of the property. The rest of the eastern half of the property consists of wooded land as well as a dirt access road along the river.

An access road branches to the north to an open area of land centrally located on the eastern half of the property. According to prior reports, this area was previously used to dump construction and demolition debris. The access road continues east along the river and leads to a residence located to the east of the property.

Sample analytes during the Phase II included volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), priority pollutant list (PPL) metals, polychlorinated biphenyls (PCBs), dioxins, lithium (groundwater only), and radionuclides (groundwater only).

2.0 **PROJECT MANAGEMENT AND OBJECTIVES**

2.1 **TITLE AND APPROVAL PAGE**

The QAPP's Title and Approval page are provided on the 2nd page of this document, after the cover page.

2.2 **TABLE OF CONTENTS**

The Table of Contents follows the Title and Approval page.

2.2.1 ***Personnel Responsibilities***

Table 2-1 presents the name, organization, and title of the individuals who will have responsibility for the quality assurance of the project, and their responsibilities. The ERM Field Leader may change, depending on availability at the time of sampling.

The ERM Field Leader may delegate specific field activities to qualified junior ERM staff geologists / engineers and act as the ERM health and safety (H&S) officer during the field activities. The ERM Quality Assurance Manager (QAM) may delegate some responsibilities to ERM personnel with the training and experience required for the specific task. The TA PMs may delegate their responsibilities to their respective laboratory QAM. If TA requires client input on the selection of optional procedures in the analytical methods, the TA PM will notify the ERM PM and ERM QAM of this requirement before the project starts.

All subcontractors to ERM will be certified under the ERM contractor prequalification program (Avetta) as meeting ERM's H&S and other requirements. All subcontractors will provide a PM in charge of meeting the requirements of the activities for which his or her company was retained.

2.2.2 ***Special Training Requirements/Certification***

Samples will be collected by experienced geologists or field samplers, who will have the necessary H&S training for the activities they will perform. In addition, samples collected in the field will be conducted by appropriately trained personnel for the media and the analysis required.

All subcontractors will be experienced in the work they will be performing and will be licensed or certified, as applicable. ERM selected Test America as the project laboratory. Test America, Inc. (TA) is accredited by the National Environmental Laboratory Accreditation Program (NELAP). The QAPP provided by TA for the specific analytical procedures is provided in Appendix B.

2.3 PROJECT PLANNING AND PROBLEM DEFINITION

2.3.1 Project Planning

Reid and ERM will work with the IBP to discuss current and future proposed sample locations to ensure the adequate collection of data necessary for the purposes of investigation is obtained. Future work plans and/or sampling and Analysis Plans (SAPs) stating and summarizing the sampling work and objectives will be submitted to the IBP for approval prior to beginning work.

2.3.2 Problem Definition

2.3.2.1. Project Objectives

Based on the *Special Notice of Liability Letter – Information Request* dated May 14, 2015 issued by the Indiana Department of Environmental Management (IDEM), the hazardous substances documented at the Site are arsenic, asbestos, chromium, thallium, lead, Aroclor 1232, petroleum aromatic hydrocarbons (PAHs), dioxin, lithium, and radionuclides. As indicated in **Section 1.0**, the purpose of this investigation is to further evaluate the findings included in the *Phase II Limited Subsurface Investigation* report dated August 29, 2014 with respect to the hazardous substances identified by the IDEM.

IDEM’s Special Notice of Liability (SNL) letter dated May 14, 2015 specifically identifies arsenic, asbestos, chromium, thallium, lead, Aroclor 1232, petroleum aromatic hydrocarbons (PAHs), dioxin, lithium, and radionuclides as the hazardous substances documented at the Site. With the exception of asbestos, each of these potential constituents of potential concern (COC) will be further investigated through the implementation of this IWP with the intent of 1) eliminating them from being a COC, 2) establishing certain conditions whereby these materials are naturally occurring (e.g. background), or 3) resulting in the need for certain IC or remedial activities to achieve site closure. Upon completion of the investigation effort, ERM will prepare a summary report to summarize the work and discuss the findings of the investigation. A primary focus of the report will be to update the current CSM consistent with IDEM’s RCG to make a determination on whether future investigation and/or remediation may be necessary.

2.4 QUALITY ASSURANCE OBJECTIVES FOR DATA MEASUREMENT

2.4.1 Measurement Performance Criteria

The overall QA objectives for newly collected data are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, field measurement, and reporting that will provide data that are scientifically valid, are to a degree of quality consistent with their intended use, and are defensible in a court of law. This section defines the goals for the QC effort and the

measurement performance criteria (MPCs) for the sampling, including precision, accuracy/bias, sensitivity, completeness, representativeness, and comparability of field and laboratory analyses.

2.4.1.1. *Definitions*

Table 2-2 presents the definitions of the MPCs, based on the UFP-QAPP definitions.

2.4.1.2. *Accuracy, Precision, and Sensitivity of Analyses*

Field Instruments

The accuracy, precision, and sensitivity of analyses for the field instruments are summarized in **Table 2-3**. If any of the type of field instruments detailed above is not available and a different one has to be selected, the accuracy, precision, and sensibility of the replacement instrument will be similar to those indicated in **Table 2-3**.

Laboratory Equipment

The method detection limits and practical quantitation limits for the laboratory equipment are shown in **Table 2-4** for the media to be sampled, along with associated screening levels and other applicable benchmarks. Method detection limits (MDLs) are also included.

The acceptance criteria for the relative percent difference (RPD) of laboratory analytical data for field duplicates will be 40% for all media to be sampled.

2.4.1.3. *Representativeness, Comparability, and Completeness*

Table 2-5 summarizes the representativeness, comparability, and completeness requirements for the project.

2.5 **PROJECT OVERVIEW AND SCHEDULE**

The specific sampling that is proposed at the Site to further evaluate the potential exposure pathways include:

- Soil sampling
- Groundwater sampling

More information on this sampling can be found in the SIWP dated March 2016, or other SAPs and work plans submitted for the project, as well as the following sections of this QAPP:

- **Section 3.0:** Sampling procedures,

- **Section 4.0:** Assessment/oversight procedures, and
- **Section 5.0:** Review procedures.

The project schedule will proceed, beginning with the site investigation. Upon completion of the field investigation, applicable tables and figures will be produced from the data that was collected, and a report summarizing the investigation findings will be produced. If any further investigation and/or remediation work is needed, a work plan will be submitted to IDEM for approval, and the work will follow this same chronology of events.

3.0 MEASUREMENT/DATA ACQUISITION

3.1 SAMPLING TASKS

3.1.1 *Sampling Locations*

A summary of investigation areas, sampling locations, samples, and analytical methods are provided in the IWP dated March 2016, or in a more current work plan submitted to the IBP. Future Work Plans will be updated to reflect the proposed investigation locations.

3.1.2 *Parameters to Be Tested*

The parameters to be tested in samples collected include:

- PAHs – US EPA Test Method 8270
- Arsenic, Chromium, Lead, Lithium, and Thallium – US EPA Test Method 6010
- Gross Alpha/Beta – US EPA Test Method 900.0
- Radium 226 – US EPA Test Method 903.1
- Gamma Spec – US EPA Test Method 901.1
- Dioxins/Furans (all 17 compounds) – US EPA Test Method 8290A
- Polychlorinated Biphenyls (PCBs) – US EPA Test Method 8082A

Supporting data collected in the field include the following:

- Qualitative geologic descriptions of the soil;
- Soil screening using a combination photoionization detector (PID) / flame-ionization detector (FID);
- Radiological Meter
- Static water level in monitoring wells;
- Groundwater quality parameters including dissolved oxygen, oxidation-reduction potential, turbidity, specific conductance, pH, and temperature

3.1.3 *Intended data usage*

The data uses for the analyses conducted during the field investigations include the following:

- Field soil classification information to identify subsurface soil.
- Groundwater measurements in monitoring wells to determine the potentiometric surface.
- PID and FID data from screening of soil samples to select sampling intervals.
- Groundwater qualitative descriptions and field measurements for low-flow (micro-purge) sampling to check the stability of the groundwater prior to sampling.
- Laboratory analysis of soils and groundwater to identify the concentration and extent of any COCs and determine the need for additional investigations or remediation activities.

3.1.4 *Sampling Procedures and Requirements*

All samples will be collected by following the Standard Operating Procedures (SOPs) listed below:

- **SOP #10** for soil boring installation and sample collection and surveying;
- **SOP #20** for monitoring well installation, development and surveying;
- **SOP #30** for groundwater sampling by micro-purge methods;
- **SOP #100** for field instrument testing and inspection;
- **SOP #110** for field documentation;
- **SOP #120** for label contents, packaging, marking and shipping of samples;
- **SOP #130** for field sample custody.
- **SOP #180** for Surface Wipe (Smear) Sampling
- **SOP #190** for Decontamination Procedures

All SOPs referenced in this section are included in **Appendix A** of this QAPP.

3.1.4.1. *Sample Collection Procedures*

Soil

Soil sample collection will be performed in accordance with **SOP #10**.

Groundwater

Monitoring wells will be installed using a direct push drill rig, a hollow-stem auger rig, or a vibratory drilling rig under the oversight of qualified ERM field personnel. **SOP #20** has the procedures to be followed for monitoring well installation. **SOP #30** has the procedures to be followed for Groundwater Sampling using low flow (micro-purge) methods. In addition, SOP #20 is consistent with IDEM's *Drilling Procedures and Monitoring Well Construction Guidelines – Nonrule Policy Document #W-0053*.

Following completion of the drilling and well installation work, the following activities will be performed in the order indicated:

- Develop each well using a submersible pump and dedicated tubing or a disposable bailer, in accordance with **SOP #20**.
- Oversee the surveying of the locations and elevations of all monitoring wells by a subcontractor, in accordance with the procedures in **SOP #20**.
- Measure the depth to groundwater at each of the monitoring wells using an electronic water level meter or oil/water interface meter in accordance with **SOP #30**.
- Purge each monitoring well with a submersible bladder pump in accordance with **SOP #30**.
- Collect groundwater samples with a submersible bladder pump and/or Teflon® bailer in accordance with **SOP #30**.

Surface Wipes

Surface wipe sample collection will be performed in accordance with **SOP #180**.

3.1.4.2. *Decontamination Procedures*

Personnel, equipment, and instrument decontamination procedures are described in **SOP#190**. The laboratories will provide certified-clean sample containers for the analyses they will perform. After sample preservation, where required, the exterior of the sample containers will be wiped off before labeling.

3.1.4.3. *Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures*

Hand-held field equipment for this project will include, but is not limited to, a PID, FID, water level or oil/water interface meter, bailers, YSI 556 or similar water quality meter, Ludlum Model 2350-1 radiation survey and count meter or similar, and submersible pumps. All ERM field personnel will be familiar with the calibration, operation, and maintenance of all field instruments and will maintain their proficiency. Operating procedures outlined in the manual for each instrument will be followed. Calibration details will be recorded in the field

notebook. Calibration of field instruments will be performed according to the manufacturer's procedures at the intervals specified by the manufacturer or more frequently as conditions dictate. In the event that a field instrument cannot be calibrated, it will be returned to the manufacturer or the rental company for service.

Field sampling equipment testing and inspection procedures are described in **SOP #100**, included in **Appendix A**. The equipment type, manufacturer, supplier, inspections performed, findings, and solutions (as applicable), will be recorded in the field notebook.

3.1.4.4. *Sampling Supply Inspection and Acceptance Procedures*

Supplies and consumables for this project may include, but are not limited to calibration gases and standard solutions, detergent for equipment cleaning, distilled water, deionized water, hoses, tubing, bailers, and sample containers. The ERM Field Leader will be responsible for defining, obtaining, inspecting, and accepting the supplies and consumables related to the task he or she will perform to ensure they conform to the order placed, are available in sufficient quantity, and are in good condition. ERM has reliable suppliers of sampling equipment, consumables, and field instruments. These suppliers also provide the appropriate calibration gases and standard solutions. Sample containers will be provided by the laboratory that will perform the specific analysis. See **Section 3.2.4** for the laboratory procedures.

3.1.4.5. *Field Documentation Procedures*

Field observations and measurements taken in the field will be recorded in a field notebook and on field data collection forms, including soil boring logs, well construction diagrams, and groundwater sampling field forms. Examples of these forms are included in **Appendix C**. The procedures for documenting field activities are described in **SOP #110** included in **Appendix A**.

3.2 **ANALYTICAL TASKS**

The QA Manual for TA is presented in **Appendix B**.

3.2.1 *Analytical SOPs*

The soil and groundwater samples will be analyzed using the methods indicated in Section 3.1.1.2 above. MDLs and PQLs for each method and analyte are presented in **Table 2-4**. The laboratory will report only the analytes listed on that table for each method.

Corrective actions for laboratory analysis problems are presented in **Sections 4.1.2.2 and 4.1.2.3**.

3.2.2 *Analytical Instrument Calibration Procedures*

The laboratories will follow the calibration procedures and frequency for laboratory instrumentation specified in the analytical methods to be used for analysis of the samples. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will either maintain a factory-trained repair staff and in-house spare parts or service contracts with vendors. The calibration procedures followed by the laboratory is presented in **Appendix B**.

3.2.3 *Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures*

As part of their QA/QC programs, a routine preventive maintenance program is conducted by TA to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments are maintained in accordance with manufacturers' specifications and schedules. This maintenance is documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacturers' maintenance is provided by factory representatives or factory-trained laboratory personnel. **Appendix B** contains specific information about the laboratories' procedures.

3.2.4 *Analytical Supply Inspection and Acceptance Procedures*

Appendix B has the procedures followed by the laboratory to obtain their supplies and consumables and to document the source and cleanliness of the containers.

3.3 **SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES**

3.3.1 *Sample Collection Documentation*

3.3.1.1. *Sample Designation*

Samples will be designated as described below:

- Groundwater will be designated by monitoring well as MW-X-YYYYMMDD-01, where X will be the monitoring well identifier.
- Soil samples will be designated as SB-X-Depth-YYYYMMDD-01, where X is the sequential number of the soil boring.

QC samples will be designated as follows:

- Trip blanks will be designated as TB-X-YYYYMMDD-01, where X will be sequential sample number.

- Rinsate blanks will be designated as RB-X-YYYYMMDD-01, where X is the sequential sample number.
- Field duplicates will be designated DUP-X-YYYYMMDD-01, where X is the sequential sample number.
- Matrix spike/matrix spike duplicate (MS/MSD) samples will be designated as MS/MSD analysis in the “Special Instructions” section of the chain-of-custody forms. No specific name designation will be necessary.

3.3.1.2. *Sample Label Contents*

A label describing the contents of the sample and which analyses to perform on the sample will be placed on each sample container in accordance with the procedures specified in **SOP #120**, included in **Appendix A**.

The information recorded on the sample label will also be recorded in the field notebook, field forms (as appropriate), and chain-of-custody for each sample.

3.3.2 *Sample Handling and Tracking System*

3.3.2.1. *Sample Handling*

The ERM Field Leader for each phase of the project will be responsible for the handling, custody, storage, and shipping of samples collected in the field. The laboratory sample receiver and the analysts and technicians performing the extraction and analysis will be responsible for handling, custody, and storage of the samples as indicated in **Appendix B**. The laboratory will assign a unique identification number to each sample in accordance with **Appendix B**. The laboratory PM will be responsible for sample disposal. Unless agreed otherwise, the laboratories will retain samples before disposal for a minimum period of 30 days after submittal of the analytical data package.

3.3.2.2. *Sample Delivery*

Sample packaging, marking and labeling, and shipping procedures will be performed as indicated in **SOP #120**, included in **Appendix A**.

The samples will be shipped on the same day they are collected via an overnight carrier or delivered to the laboratory by a laboratory courier or ERM personnel either on the same day of collection or before 10 am on the next day, whenever possible. The laboratory will be notified at the time of shipment.

3.3.3 *Sample Custody*

A sample will be considered under a person's custody if it is (1) in a person's physical possession, (2) in view of the person after he has taken possession, (3)

secured by that person so that no one can tamper with the sample, or (4) secured by that person in an area that is restricted to only authorized personnel. The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact.

3.3.3.1. *Field Custody*

The field sampler(s) will be responsible for the care and custody of the samples until they are transferred or properly dispatched. As few personnel as possible will handle the samples.

To provide documentation necessary to trace sample possession from the time of collection to the time of receipt by the analytical laboratory, a chain-of-custody record will be completed and will accompany each shipment of samples to the laboratory. Copies of the chain-of-custody form for the laboratory are attached in **Appendix C**. See **SOP #130** in **Appendix A** for the chain-of custody and other field sample custody procedures.

3.3.3.2. *Laboratory Chain-of-Custody Procedures*

The chain-of-custody procedures followed by the laboratory can be found in **Appendix B**.

3.4 **QUALITY CONTROL SAMPLES**

QC samples provide measurable data quality indicators used to evaluate the different components of the measurement system, including sampling and analysis. This section describes the types of QC samples to be used for the project.

3.4.1 *Sampling Quality Control Samples*

The QC samples collected or labeled during field sampling include equipment rinsate blanks, trip blanks, blind field duplicates, and MS/MSDs. The use of each QC sample, sampling procedures, rate of sample collection, expected total number of QC samples, and QC limits are presented in **Table 3-1**.

All QC samples will be preserved, handled, and delivered to the laboratory by following the same procedures as those used for the investigative samples.

3.4.2 *Analytical Quality Control Samples*

The laboratory responsible for performing the groundwater analyses will follow the QC requirements in the corresponding analytical method. If the laboratory QC requirements are more stringent than those of the methods being used, the most stringent QC requirements will apply.

The laboratories will perform the internal QC checks specified in the analytical methods they are following. Depending on the analytical method, the QC checks may include analyzing sample spikes, surrogate spikes, reference samples, laboratory control samples, storage blanks, and/or method blanks. The frequency of QC checks, the compounds to be used for spikes, and the QC acceptance criteria are described, as appropriate, in the analytical methods to be used and in **Appendix B** if more stringent than the methods' requirements.

The laboratories' MDLs and PQLs for each compound and medium are presented in **Table 2-4**. Corrective actions for not meeting the control limits will be implemented in accordance with the analytical method being followed and with **Sections 4.1.2.2 and 4.1.2.3**. The laboratory will document internally that both initial and ongoing instrument and analytical QC criteria have been met. The data packages to be provided will contain all of the information needed to evaluate compliance with the analytical methods' required QC checks. The contents of the laboratories' data packages are described in **Section 3.5.2.2**.

3.5 DATA MANAGEMENT TASKS

3.5.1 *Project Documentation and Records*

Field information documentation records are described in **Section 3.1.2.5** and the contents of the field data package are presented in **Section 3.5.2.1**. Laboratory-generated documentation and records will be, at a minimum, as required by the analytical method each laboratory is following. Further information on laboratory documentation and records is presented in **Appendix B**. The contents of the laboratory data package are discussed in **Section 3.5.2.2**. Deliverables for other subcontractors are described in **Section 3.5.2.3**.

Information noted on the field notebook will be used to prepare soil boring logs and monitoring well construction diagrams, calculate groundwater elevations and prepare piezometric surface maps, and prepare tables of field data. Boring log and monitoring well construction diagram templates are located in **Appendix C**.

Each laboratory and contractor is responsible for reporting the data generated to ERM. As the Reid contractor, ERM is responsible for reporting all data generated for the sampling activities to Reid.

Data collected during the investigation activities will be submitted in a summary report following the conclusion of the work activities.

3.5.2 *Data Package Deliverables*

3.5.2.1. *Sample Collection and Field Measurements Data Package Deliverables*

For the field measurements, the data package deliverables include the original and copies of the field notebooks, groundwater sampling field forms, chain-of-custody forms, air bills or record of pickup by laboratory courier (if samples are not delivered by ERM personnel to the laboratory), performance assessment checklists, and any correspondence with the laboratories that define the project requirements, requests changes to the chain-of-custody forms (e.g., place samples on hold), or similar information that defines the laboratory work. **SOPs #110 and #130 in Appendix A** describe the contents of the field notebooks and the chain-of-custody forms, respectively.

3.5.2.2. *Laboratory and Subcontractor Data Package Deliverables*

TA will provide documentation of the laboratory analyses in accordance with IDEM's *RCG Section 3.9 and Table 3-A*. The contents of the laboratory data package must be sufficient to allow data validation up to Stage 4 (if needed), as described in Appendix A of the USEPA's *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*, EPA-540-R-08-05, January 13, 2009 . The laboratory will provide, at a minimum, the following data formats:

- Excel or text file of the analytical data suitable for upload into Earthsoft's EQulS Software;
- Adobe Acrobat file (.pdf) showing the results of analytical analysis, and QA/QC objectives;

The laboratory will provide electronic copies of the following documentation as a report of the survey performed at the Site:

- AutoCAD file showing the surveyed information;
- Excel or text file listing the coordinates for each sampling location; and
- Adobe Acrobat file (.pdf) showing the surveyed information (i.e., a picture of the AutoCAD file).

Other subcontractors, if used, will provide documentation of the activities performed by them in accordance with the requirements established in their subcontract with ERM.

3.5.3 *Data Reporting Formats*

Guidelines for recording of field data in the field notebook are specified in **SOP #110 in Appendix A**. Each laboratory will provide ERM with sample data

packages, in accordance with the requirements in **Section 3.5.2.2**, in portable data file format (.pdf), and an electronic data deliverable (EDD) that will present the analytical results in tabular form, with samples listed in rows and compounds analyzed listed in columns. At a minimum, the EDD will include the analytical result or reporting limit for each compound, the sample name, date of collection, and applicable data qualifiers. Description of qualifiers will be provided in a separate page of the data package. The laboratory will provide data with a maximum of three significant figures and will ensure that the concentration of each constituent in each sample is the same in both the data package and the EDD.

EarthSoft's EQUS software suite will be used as the primary management and storage tool for field and laboratory data collected during the investigations, including laboratory analysis and field data.

EQUS is a SQL Server-based Relational Database Management System with a data model specifically designed to manage environmental field and analytical data. The SQL database is located on redundant secure servers hosted by EarthSoft. Access to the system is conducted through a web portal running on a standard browser (EQUS Online) or through desktop software (EQUS Professional). This system allows consultants, clients, and regulators to securely access the data in various formats regardless of their respective locations. Since EQUS is an independent third-party software package, it is widely used in the environmental industry. It contains numerous plug-ins to additional data analysis and visualization tools, and most major laboratories, including TA, are familiar with it and can enter data directly into the system.

3.5.4 *Data Handling and Management*

3.5.4.1. *Data Recording*

Field data will be recorded as indicated in **Sections 3.1.2.5 and 3.3.3.1**. Performance assessment checks will be performed at least once during field work to ensure there are no transcription errors or discrepancies between the field notebook and chain-of-custody form information, in accordance with **Section 4.1.1.1**. Laboratory data will be recorded in accordance with TA's QA Manual (**Appendix B**). Audits of laboratory data recording will be performed as indicated in TA's QA Manual.

3.5.4.2. *Data Transformations and Data Reduction*

Analytical data tables will contain the sample name, sample location, sample date, sample reporting limits for non-detected compounds, and detected analytical results. The information presented in figures may include analytical results on a Site map, geological cross sections, and groundwater piezometric surface maps.

Field and analytical data will be summarized in reports, as applicable, as follows:

- Geological field observations and field screening (PID or FID) will be summarized in soil boring logs;
- Radiological readings
- Depth to water or product will be summarized in tables and on groundwater sampling field data forms and used to calculate groundwater elevations for groundwater piezometric surface maps;
- Groundwater quality data obtained during purging will be summarized in tables and on groundwater sampling field data forms;
- Laboratory data will be summarized in tables and figures; and
- Laboratory reports will be included in appendices (either hard copies or electronic copies on CD or DVD).

Each data summary document (i.e., boring log, table, and figure) will be checked for accuracy upon completion. If data are tabulated or calculations performed, an independent peer review will be conducted by an ERM staff member (who is a peer of the person making the calculations) to ensure that the data were entered correctly from hard copies, the comparison criteria (e.g., screening levels) were entered at the correct values for the correct constituents, and the exceedances were correctly identified. The ERM PM will also spot-check data summary documents throughout the report preparation.

TA will perform data reduction for the analyses it performs as described in **Appendix B**. Reduction of laboratory data will ensure that actual quantities reported are accurate and appropriately qualified. The number of significant figures is indicated in **Section 3.5.3**.

3.5.4.3. *Data Transfer and Transmittal*

Samples collected in the field will be analyzed at accredited analytical laboratories such as those chosen for this project, and the results received in electronic formats that have been previously established with ERM subcontract laboratories. TA's data transfer and transmittal procedures, provided in **Appendix B**, describe the laboratory report formats for this project. The sample data package will be submitted electronically via e-mail or access to a laboratory-maintained and secure web page.

3.5.4.4. *Data Analysis*

After laboratory analytical results have been received and are verified and validated, the data will be used to interpret the current site conditions. Data

tables will be produced to evaluate spatial conditions and produce figures as appropriate.

The laboratories' data equipment and computer software that will be used to process, compile, and analyze project data are described in **Appendix B**.

3.5.5 Data Tracking and Control

3.5.5.1. Data Tracking

Field and laboratory tracking procedures are outlined in **Sections 3.3.3.1 and 3.3.3.2**, respectively. Hard-copy files, if any, will be tracked by using a form where ERM staff members removing hard-copy files will add their name, the document removed, and the date of document removal and return to its original location.

3.5.5.2. Data Storage, Archiving, and Retrieval

Electronic project files, including, but not limited to .pdf, .docx, .xlsx, .txt, or .mdb files, will be stored on the ERM internal server in a client- and project-specific folder, and will be maintained by the ERM PM. Hard copies, if any, will be maintained in a file cabinet drawer or in boxes under the ERM PM's supervision in ERM's Indiana office until the end of the project. Hard copies of field information (notebooks, chain-of-custody forms, airbills, etc.) will be scanned and saved in the project's electronic folder.

TA will provide only electronic data packages. Once those data packages are received by an ERM staff member, they will be saved in the project's electronic folder on the ERM internal server. Instrument calibration and maintenance records, as well as records of container cleanliness, internal or external audits, and other internal laboratory information will be archived by TA.

Tables, figures, and reports that use the data collected will be appropriately named and filed under the client- and project-specific folder on the ERM internal server. Each staff member using the electronic data is responsible for leaving the file at the same location and for naming new files produced with the project data in a clear way to allow their identification as project files. Only the ERM PM can re-organize the electronic files, if necessary. Any hard-copy document removed from the file will be returned to its original location upon completion of its use by the person who removed it.

ERM's Database Management processes are based on EarthSoft's EQuiS Environmental Data Management System. Several automated a semi-automated quality checks are applied to the data before insertion into the permanent database, and a full QA of the submitted data against the final lab reports is conducted before releasing the data for reporting. The server housing this

database is operated at an off-site location with redundant backup and guaranteed uptime.

3.5.5.3. *Data Security*

Each ERM project has an electronic folder for each project. These are housed on the ERM company-wide server, which is password restricted. Servers in each project office are backed up each weekday and labeled. The tapes are reused weekly. In addition, 12 monthly tapes are used to back up files on the first day of the month. These are retained for one year. At the end of the project, the electronic files will be stored either on the ERM PM's office server or in an appropriate, secure location that will be password-restricted.

The storage facility used for storage of hard-copy files is responsible for ensuring the security of the files. However, given that all hard-copy documents are scanned as they are received and saved in the project's electronic file, the loss of the hard-copy files is not critical.

4.0 ASSESSMENT/OVERSIGHT

4.1 ASSESSMENTS AND RESPONSE ACTIONS

In addition to the QA/QC requirements described in **Sections 2.4 and 3.4** of this QAPP, a review of the QA/QC procedures will be performed periodically for field and laboratory activities as described in the next sections.

4.1.1 *Planned Assessment*

4.1.1.1. *Field Activities*

Planned field assessments include a review of the field activities to ensure the QAPP procedures are being followed and an internal review of the field documentation to ensure all planned locations have been sampled correctly and that the analytical methods requested for each sample are correct.

At this point, no external field audits are planned for investigative sampling and remedial activities. If conducted, external field audits may include a review of the same procedures included in the internal audits.

4.1.1.2. *Laboratories*

TA's QAMs will perform internal performance and system audits of laboratory operations in accordance with the procedures and timing in the laboratory QA manual (**Appendix B**). TA's QAM will notify the ERM QAM of any findings that require corrective actions that cannot be applied at the laboratory (e.g., re-sampling), as indicated in **Section 4.1.2.3**.

In addition, external audits of the analytical laboratories may be performed by Reid, ERM, or a subcontractor of either entity. If serious deficiencies are discovered, corrective measures will be undertaken and documented. No external audits are planned at this time.

TA will document internally that both initial and ongoing instrument and analytical QC criteria have been met. The data package provided by TA will include a summary of the QC checks, and all raw data for both the QC checks and the samples.

4.1.2 *Assessment Findings and Corrective Action Responses*

4.1.2.1. *Field Activities*

If a problem occurs in the field that is immediately correctable by direct action, the ERM Field Leader will be responsible for discussing the issue with the field sampler(s) and ensuring that the action is taken. For example, if poor sampling

techniques are observed during sample collection, the ERM Field Leader will explain the issues and how to resolve them to the field sampler, the sample will be re-collected under the supervision of the ERM Field Leader, and steps will be taken to prevent a reoccurrence of the problem (e.g., training of all samplers, additional audits). No additional work that depends on the nonconforming activity will be performed until the corrective actions are discussed.

If a sampling procedure has to be adjusted to accommodate site-specific conditions but does not impact the quality of the data (e.g., re-positioning a sampling location because of an obstruction, changing the field instrument because of malfunctioning), the ERM Field Leader will make the decision on the spot, record the change in the field notebook and in an Assessment Checklist, and notify the ERM PM within 24 hours. If the change will impact the quality of the data (e.g., insufficient sample volume), the ERM PM will be notified as soon as possible. The ERM PM will then contact Reid and, if necessary, the IDEM PM, as soon as possible to discuss the change and obtain approval.

4.1.2.2. *Laboratory Analyses*

The department supervisors at TA will evaluate any problems that occur during analysis that are immediately correctable (i.e., would not require additional field work to correct) and, if necessary, will enlist the TA QAM to solve them. Corrective action procedures of the laboratory are presented in the QA manual (**Appendix B**).

4.1.2.3. *Other Corrective Actions*

Certain problems, such as determining that insufficient sample volume is available at the laboratory for analysis, or that QA/QC RPDs are not met, are not always immediately correctable. If such a problem is encountered, the TA QAM will contact the ERM PM, who will then contact Reid. These parties will reach an agreement as to the corrective action warranted. The ERM PM will be responsible for implementing the agreed-upon action. This same procedure will be followed if audit results or unacceptable data indicate that re-sampling is necessary.

If there is a problem with laboratory performance that is not immediately correctable, the proposed corrective action will be discussed in a proposal by the laboratory QAM. This proposal will be presented by the ERM PM to Reid. The corrective action will be implemented only after full agreement on the required action has been reached. The TA PM will be responsible for implementing any corrective actions.

Before implementing significant corrective actions, such as modifying an analytical method, the ERM PM will obtain the approval of the IDEM PM.

4.2

QA MANAGEMENT REPORTS

The TA QAM will provide to the ERM PM written reports of required corrective action, if any, and issues with the QC samples as part of the data package narrative. These reports, along with the results of any field or external laboratory audits conducted, will form the basis of the project QA report that will be prepared by the ERM QAM and included as a section or appendix in the future reports submitted to the IDEM PM. Any problems serious enough to require significant actions (e.g., changing an approved SOP) will be reported to the IDEM PM within five days of the occurrence. The project QA report will include the following information:

- Whether there was any deviation of the QAPP procedures;
- A data quality assessment in terms of precision, accuracy, completeness, sensitivity, representativeness, and comparability;
- A statement as to whether the QA objectives were met;
- Problems that resulted in QA/QC issues and corrective actions taken; and
- Any limitations to the use of the data.

The laboratories' requirements for QA management reports are included in **Appendix B**.

4.3

FINAL PROJECT REPORT

Each laboratory and subcontractor is responsible for reporting the data generated to ERM, as specified in **Sections 3.5.2.1 and 3.5.2.2**, respectively. As the Reid contractor, ERM is responsible for reporting all data generated for the sampling activities to Reid.

5.0 DATA REVIEW

This section describes the steps to be taken and procedures to be followed to evaluate the data collected to ensure that project decisions are made with data that meet the DQOs and MPCs established for the project.

5.1 OVERVIEW

The data review will consist of several steps, including verification (Step I), validation (Step II), and usability assessment (Step III). Steps II and III will be streamlined, as described in **Section 5.2**. These steps include the following activities:

- Step I: Verification – Review for completeness of records.
- Step II: Validation – Assessment and documentation of compliance with methods, procedures, and contracts.
- Step III: Usability Assessment – Determination of the adequacy of data, based on the results of validation and verification, for the decisions being made.

Specific information to be reviewed and the review procedures are presented in the next section. Each step may have more than one person responsible for it.

5.2 DATA REVIEW STEPS

5.2.1 Step I: Verification

The objective of this step is to determine if the required information to evaluate if the field and laboratory data are usable has been received from the different sources generating them. The information to be verified and the procedures followed are summarized in **Table 5-1**. The laboratory procedures for data verification are included in the TA QA manual (**Appendix B**).

5.2.2 Step II: Validation

During this step, compliance with methods, procedures, and contracts will be evaluated. **Table 5-2** indicates the issues that will be validated, the items that will be checked and the organization and individual responsible for doing the validation. Analytical data will not undergo data validation beyond what's listed in **Table 5-2**, unless data discrepancies are noted. Even in this case, the validation will only consist of reviewing the raw data for the specific sample(s) affected.

5.2.3 *Step III: Usability Assessment*

5.2.3.1. *Data Limitations and Actions from Usability Assessment*

Table 5-3 lists the MPCs discussed in **Section 2.4.1**, the procedure for evaluating compliance with each MPC, and the information to be included in the usability report.

5.2.3.2. *Activities*

The usability assessment will be performed by the ERM PM and the ERM QAM, with input from the ERM Field Leader or the laboratory, if necessary. The usability assessment report will be included in the project QA report to be submitted to the IDEM PM in the formal investigation report, as indicated in **Section 4.3**.

Field-collected data (PID/FID readings, groundwater stability monitoring readings, depth-to-water measurements) will not be assessed for usability, because these data will be used in a relative, semi-quantitative manner. However, any discrepancies (e.g., a depth-to-water measurement that is inconsistent with previous results or with the results for surrounding wells completed in the same saturated zone) will be investigated for possible causes, and corrected before submitting the report of the related activities.

5.2.4 *EarthSoft's EQulS Data*

EarthSoft's EQulS software has been designed specifically for environmental data; it contains stringent QC procedures through which datasets must pass in order to be available for reporting. The automated checks require conformance of all inputs to match database field formats, use of applicable reference values, required parent-daughter relationships, and checks for missing or duplicate data. These automated data checks are complemented by additional manual reviews, as outlined in Section 6, to ensure that accurate and complete data are being maintained and transferred to all parties regardless of format. Streamlining Data Review

5.2.5 *Data Review Steps to Be Streamlined*

Laboratory data validation in accordance with the EPA Contract Laboratory Program *National Functional Guidelines for Organic Data Review* (2008) will not be performed as IDEM conducts their own validation of the provided laboratory data packages. If deemed necessary by ERM or Reid, some or all of the samples will be validated prior to submittal to the IDEM PM by following the above-indicated EPA guideline.

5.2.6 *Criteria for Streamlining Data Review*

The criteria for streamlining data review are discussed in **Section 5.2.5**.

5.2.7 *Amounts and Types of Data Appropriate for Streamlining*

All future analytical laboratory data can be streamlined, as indicated in **Section 5.2.5**.

Table 2-1
Personnel Responsibilities
Former Reid Hospital Site
1401 Chester Blvd. Richmond, Indiana

| <i>Name, Title</i> | <i>Responsibilities</i> |
|--|---|
| Mr. John Markey, ERM (PIC) | <ul style="list-style-type: none"> · Overall responsibility for ensuring that the project meets the requirements of Reid and the IDEM. · Provide input to the ERM PM on the strategies and activities required to complete the project and provide peer review of all documents. |
| Mr. Aaron Friedrich L.P.G. (Indiana #2254), ERM PM | <ul style="list-style-type: none"> · Develop the strategies and activities required to complete the project, in consultation with other specialized ERM staff members. · Supervise the preparation, quality, and submittal of all documents. · Establish and manage all budgets and schedules. · Selects and oversees subcontractors and project staff. · Ensure that the project field activities meet ERM's and Reid's H&S and QA requirements, in consultation with the ERM HSO and the ERM QAM. · Maintain and update the QAPP (with input from the QAM and other specialized ERM personnel). · Distribute updated QAPPs to the persons in the distribution list. · Maintain the final evidence file in accordance with the QAPP. |
| Chris Burrows ERM Field Leader | <ul style="list-style-type: none"> · Lead and coordinate the day-to-day activities of the field crews under his or her supervision. · Ensure that the requirements of the WP and the QAPP are followed. · Direct and supervise the activities of the subcontractors (except for the laboratories), and schedule and request containers from the laboratories. |
| Austin Taylor ERM HSO | <ul style="list-style-type: none"> · Be responsible for the safe implementation of the field activities, in accordance with ERM's and Reid's H&S requirements. |
| Mrs. Teresa Kennedy, ERM QAM | <ul style="list-style-type: none"> · Ensure the overall quality of the project field activities. · Act independently of the ERM staff generating information for the project. · Prepare and update the QAPP as the project advances from the site investigation to the remedial action implementation. · Provide QA assistance to ERM project staff members. · Direct and supervise the activities of the laboratories, except for scheduling and sample container requests. · Review or supervise the review of the field notebooks to determine whether the proper QA/QC procedures were followed during the field work. · Perform or oversee any required data validation and assessing the usability of the data. |
| Mr. Kenneth Dow, ERM Radiological Task Manager | <ul style="list-style-type: none"> · Responsible for the implementation of the radiological characterization · Ensure that radiological subcontractors, if any, are following applicable provisions of the QAPP and SOPs · Primary point of contact with radiological subcontractors and is responsible for the activities performed by the subcontractors · Responsible for QA/QC and data review of the radionuclide data |

Table 2-1
Personnel Responsibilities
 Former Reid Hospital Site
 1401 Chester Blvd. Richmond, Indiana

| <i>Name, Title</i> | <i>Responsibilities</i> |
|---|--|
| Elizabeth Hoerchler, Test America PM | <ul style="list-style-type: none"> · Ensure the proper review of the QAPP. · Approve the QAPP on behalf of Test America. · Ensure the analyses of all samples are performed and documented in accordance with the requirements of, in order of preference, this QAPP, the analytical methods, and the laboratory's QA Manual. · Ensure that the implementation of the QA program detailed in his or her laboratory's QA Manual is audited and the audit properly documented. |

Key: CAD = Computer-aided design; ERM = Environmental Resources Management; H&S = health and safety; HSO = H&S officer; IDEM = Indiana Department of Environmental Management; PIC = partner in charge PM = project manager; QA = quality assurance; QAM = quality assurance manager; QAPP = Quality Assurance Project Plan; QC = quality control; RWP = Remediation Work Plan

Table 2-2
Measurement Performance Criteria Definitions
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| <i>MPC</i> | <i>Definition</i> |
|-------------------------------------|--|
| Precision | Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms. Precision data indicate how consistent and reproducible the field sampling or analytical procedures have been. Overall project precision is measured by collecting data from co-located field duplicate (or replicate) samples. Precision specific to the laboratory is measured by analyzing laboratory duplicate (or replicate) samples. Two samples collected from the same location or two aliquots of the same sample analyzed by the laboratory are duplicates. If more than two samples or aliquots are used, they are called “replicates.” |
| Accuracy/Bias | Accuracy is the degree of agreement between an observed value (sample result) and an accepted reference value; bias describes the systematic or persistent distortion associated with a measurement process. The terms accuracy and bias are used interchangeably in the UFP-QAPP and this QAPP. Examples of QC measures for accuracy include proficiency testing (PT) samples, matrix spikes (MSs), laboratory control samples (LCSs), and equipment blanks. |
| Sensitivity and Quantitation Limits | Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. The quantitation limit or reporting limit (RL) is the minimum concentration of an analyte that can be routinely identified and quantified above the method detection limit (MDL) by a laboratory. Sensitivity can be measured by calculating the percent recovery of the analytes at the RL. Sensitivity can also be evaluated by comparing the quantitation limits achieved to the project’s quantitation limits and screening or closure levels. |
| Representativeness | Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental conditions of a site. It takes into consideration the magnitude of the site area represented by one sample and indicates the feasibility and reasonableness of that design rationale. Representativeness also reflects the ability of the sample team to collect samples and the ability of the laboratory personnel to analyze those samples so that the generated data accurately and precisely reflect site conditions. Sample homogeneity and sampling and subsampling variability should be considered when developing criteria for representativeness. The use of statistical sampling designs and standardized SOPs for sample collection and analysis help to ensure that samples are representative of site conditions. |
| Comparability | Comparability is the degree to which different methods or data agree or can be represented as similar. It describes the confidence that two data sets can contribute to a common analysis and interpolation. Consistency in sampling and analytical procedures within and between data sets addresses comparability. |
| Completeness | Completeness is a measure of the amount of valid data collected using a measurement system. It is expressed as a percentage of the number of measurements that are specified in the QAPP. |

Key: LCSs = Laboratory control samples; MDL = method detection limit; MSs = matrix spikes; PT = proficiency testing; QAPP = Quality Assurance Project Plan; QC = quality control; RL = reporting limit; SOPs = Standard Operating Procedures; UFP-QAPP = Uniform Federal Policy for Quality Assurance Project Plans

Table 2-3
Accuracy, Precision, and Sensitivity of Field Instruments
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| <i>Analyses</i> | <i>MPC Requirement</i> |
|-------------------------------|---|
| Screening | <p>The accuracy of the photoionization detector (PID) (MiniRAE 2000, or equivalent) will be evaluated by daily pre-measurement calibration with a standard reference gas. The minimum detection limit of the PID used will be 0.1 volumetric parts per million (Vppm) in the 0.5 to 500 Vppm scale. The precision of the PID will be evaluated by duplicate readings of the calibration standard reference gas. If readings vary more than 10% on the 0 to 500 Vppm scale, the PID will be replaced.</p> <p>The accuracy of the flame ionization detector (FID) (Foxboro TVA-1000, or equivalent) will be evaluated by daily pre-measurement calibration with a standard reference gas. The minimum detection limit of the FID will be 0.3 Vppm in the 1.3 to 10,000 Vppm scale. Precision and sensitivity of the FID will be evaluated by duplicate readings of the calibration standard reference gas. If readings vary more than 10% on the 0 to 500 Vppm scale, the PID will be replaced.</p> |
| Radiological Screening | The response of the Ludlum Model 2350-1 ratemeter/scaler/data logger with a 3" X 3" NAI gamma scintillator detector (or equivalent meter) will be evaluated as part of a daily pre-measurement calibration with a sealed radiological source (button source) standard. The calibration sticker will be verified prior to use to ensure that it is current. The response of the meter will also be checked at the end of day. If the meter fails to respond to the check source, it will be replaced. |
| Specific Conductance | Accuracy of field measurements will be ensured by conducting pre-measurement calibration using solutions of known specific conductance. The measured specific conductance of the standard solution must be within 5% of the actual specific conductance of the solution. The sensitivity of the specific conductivity meter is 2.5 µmhos/cm in the 0 to 500 µmhos/cm range. Accuracy and precision will be ± 3% and ± 1%, respectively. |
| pH | The accuracy of field measurements will be ensured by conducting pre-measurement calibration using at least two standard buffer solutions. (The pH meter will be calibrated using two standard buffer solutions, and then the pH of both solutions will be measured.) The instrument will have a range of pH 0 to 14, a sensitivity of pH 0.01, an accuracy of ± pH 0.1, and a precision of ± pH 0.05. The pH measurement must be within ± 0.10 pH units of the actual buffer solution values, or the meter will require recalibration. Precision will be assessed through duplicate measurements. (The electrode will be withdrawn from the sample, rinsed with deionized water, and re-immersed between each duplicate.) The duplicate measurement must be within ± 0.10 pH units of the initial measurement, or the meter will require recalibration. The instrument used will be capable of providing measurements to 0.01 pH units. |
| Temperature | Sample temperature will be measured with the temperature probe on the pH meter. The range, sensitivity, accuracy, and precision of the meter will be 0 to 55°C, 0.01°C, ± 1.0°C, and ± 0.3°C, respectively. The precision and accuracy of the field temperature probe will not be verified because of the difficulty of evaluating these parameters in the field. The instrument selected for the field investigation will include automatic calibration of the temperature sensor. |
| Dissolved Oxygen | The accuracy of field measurements will be verified through review of the pre-measurement calibration report provided by the rental company to ensure that the meter (YSI 556 MPS, or equivalent) was calibrated successfully. The instrument will have a sensitivity of 0.01 mg/L, an accuracy of ± 0.2 mg/L or ± 2% of the reading, whichever is greater, for a range of 0 – 20 mg/L and of 6% of the reading for a range of 20 – 50 mg/L. The meter will be calibrated prior to each rental period by the rental company. |
| Oxidation-Reduction Potential | The accuracy of field measurements of the meter (YSI 556 MPS, or equivalent) will be verified through calibration performed by the rental company before shipping the instrument; a calibration statement will be required from the rental company. The instrument's sensitivity and accuracy will be 0.1 mV and ± 20 mV in a range of -999 to +999 mV. The meter will be calibrated prior to each rental period by the rental company. |
| Turbidity | The accuracy of field measurements will be assessed through daily pre-measurement calibration. The instrument will have a range of 0 to 800 NTUs, a sensitivity of 0.1 NTU, an accuracy of ±5%, and a precision of ± 3%. Precision will be assessed by duplicate readings of the calibration standard after calibration is completed. |

Key: °C = Degrees Centigrade; cm = centimeters; mg/L = milligrams per liter; mV = millivolt; NTUs = Nephelometric turbidity units ; µmhos = micro mhos (the mho is a conductance unit, reciprocal of the ohm [unit of electrical resistance])

Table 2-5
Representativeness, Comparability, and Completeness Requirements
 Former Reid Hospital Site
 1401 Chester Blvd, Richmond, Indiana

| <i>MPC</i> | <i>Requirement</i> |
|--------------------|---|
| Representativeness | <ul style="list-style-type: none"> · The judgmental sampling design used at the Site will provide at least one sample for each area of concern. · All field sampling as well as laboratory testing and analysis will be performed in a standardized manner that adheres to the procedures specified in this QAPP. |
| Comparability | <p>Will be ensured by conducting all monitoring, screening, sampling, and analysis during the investigation and remedy implementation in a similar manner, as addressed in this QAPP. Specifically, data comparability will be ensured by:</p> <ul style="list-style-type: none"> · Reporting results in appropriate units; · Using the same or similar sampling procedures in all investigation areas; · Using the same or equivalent analytical procedures during all phases of the investigation; and · Following the same or equivalent QA/QC requirements in all investigative activities. |
| Completeness | <ul style="list-style-type: none"> · Field activities are expected to generate at least 90% of the planned samples. · The laboratories are expected to provide data meeting the QC acceptance criteria for 95% or better of the samples analyzed. The completeness of an analysis will be documented by the laboratory with items such as QC data and, if required, chromatograms and spectra, to allow the data user to assess the quality of the results. |

Key: QA = Quality assurance; QAPP = Quality Assurance Project Plan; QC = quality control

Table 3-1
Field Quality Control Samples
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Type of QC Sample | Use | Sampling Procedure | Rate | QC Limit |
|--------------------------|---|---|--|--|
| Equipment Rinsate Blanks | Determine the effectiveness of the decontamination procedure if non-dedicated equipment is used during sampling and whether the potential for cross contamination has been minimized. | Equipment rinsate blanks will only be collected from non-dedicated sampling equipment that is to be re-used during the project (e.g., pumps, hand augers). Equipment rinsate samples will be prepared by collecting laboratory-supplied reagent-grade distilled or deionized water that has been poured over decontaminated sampling equipment. | 1 per 20 samples or less per medium. | No QC limit will be applied to the blanks; instead, they will provide information on the potential for cross contamination and will be used to qualify the samples if necessary. |
| Trip Blanks | Assess the potential for VOC contamination of soil or groundwater water samples as a result of contaminant migration during sample shipment and storage. | Trip blanks, which will be provided by the laboratory, are vials containing laboratory-deionized or distilled water and will be taken to the field with the sample containers and will not be opened in the field. | One per sample cooler containing soil or groundwater samples for VOC analysis. One nitrogen field blank per shipment of sub-slab soil gas samples. | No QC limit will be applied to the blanks; instead, they will provide information on the potential for contaminant migration during sample shipment and storage, and will be used to qualify the samples if necessary. |
| Field Duplicates | Blind duplicates provide an estimate of the reproducibility of sampling and analytical procedures. | The duplicates will be collected after the investigative sample at the location has been collected and will be analyzed for the same parameters as the investigative samples. The duplicates will not be identified as such to the laboratory. | 1 per 20 samples or less per medium. | The relative percent difference (RPD) for all sample media will be 40% |

Table 3-1
Field Quality Control Samples
 Former Reid Hospital Site
 1401 Chester Blvd, Richmond, Indiana

| Type of QC Sample | Use | Sampling Procedure | Rate | QC Limit |
|-------------------|--|---|--------------------------------------|---|
| MS/MSDs | Provide information about the effect of the sample matrix on the analytical methodology. | MS/MSD samples are collected in the same way as field duplicates. Three times the investigative sample volume will be required for groundwater samples designated for MS/MSD analysis. No extra volume is required for soil MS/MSD samples. The samples will be identified as MS/MSD samples so that the laboratory can perform the necessary spike evaluation procedures. The sample locations will target areas that field screening indicates as possibly moderately impacted. | 1 per 20 samples or less per medium. | The RPD for all sample media MS/MSD samples will be 30% |

Key: MS = Matrix spike; MSDs = matrix spike duplicates; QAPP = Quality Assurance Project Plan; QC = Quality control; RPD = relative percent difference; VOC = volatile organic compound

Notes:

All QC samples will be preserved, handled, and delivered to the laboratory by following the same procedures as those used for the investigative samples.

Table 5-1
Verification (Step I) Process
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Verification Input | Items to Be Verified | Internal/ External | Person Responsible for Verification |
|---------------------------|--|---------------------------|---|
| Field Notes | (1) All records are present and complete for each day of field activities; (2) all planned samples, including field QC samples, were collected and sample collection locations are documented; (3) changes/exceptions are documented and were reported in accordance with requirements; and (4) any required field measurement was performed and results are documented. | Internal | ERM PM or his/her designee |
| Chain-of-Custody Forms | (1) Chain-of-custody form entries are consistent with the field logbook; (2) appropriate analytical methods and sample preservation are recorded; (3) the required volume of sample was collected and sufficient sample volume was available for QC samples (e.g., matrix spike/matrix spike duplicate); (4) all required signatures and dates are present; and (5) there are no transcription errors in the sample designations and sampling date between the chain-of-custody forms and the field notes. | Internal | ERM PM or his/her designee |
| Audit Reports | (1) All planned audits were conducted; and (2) any deficiencies noted in the Performance Assessment Checklist had a corrective action that was implemented accordance to the QAPP. | Internal | ERM PM or his/her designee |
| Laboratory Data | (1) All required contents of the laboratory package are included; and (2) the organization and complete contents of each data package are included as separate page(s) at the beginning of the data package. | External | ESC PM and Pace - Minnesota PM for their respective data packages |
| | (1) All required contents of the laboratory package are included; (2) there are no transcription errors in the sample designations and sampling date between the chain-of-custody form and the laboratory reports; (3) all samples for which analyses were requested in the chain-of-custody form were analyzed; (4) all analyses requested in the chain-of-custody forms were performed; (5) any problem with the samples upon receipt (e.g., high temperature, broken containers) were noted and reported to the ERM PM; and (6) the narrative describes all quality control exceptions. | Internal | ERM QAM or his/her designee |

Key: ERM = Environmental Resources Management, Inc.; PM = Project Manager; QAM = Quality assurance manager; QC = quality control; QAPP = Quality Assurance Project Plan

Table 5-2
Validation (Step II) Process
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Item | Items to Be Validated | Internal/ External | Person Responsible for Verification |
|--|---|-----------------------|--|
| Analytes | The required lists of analytes were reported as specified in the QAPP. | Internal | ERM PM or designee |
| Chain-of-Custody | Data was traceable from time of sample collection until reporting of data. | Internal | ERM PM or designee |
| Holding Times | (1) Holding times were met; or (2) if not, deviations were documented, appropriate notifications were made (consistent with procedural requirements), and approval to proceed was received prior to analysis. | Internal | ERM QAM or designee |
| Sample Handling | QAPP and laboratory sample handling, receipt, and storage procedures were followed, and any deviations were documented. | Internal | ERM QAM or designee |
| Sampling Procedures | Required sampling methods were used and any deviations were documented. | Internal | ERM PM or designee |
| Field Transcription | Transcription of sampling or measurement data (i.e., from field notebook to reports) is accurate. | Internal | ERM PM or designee |
| Laboratory Transcription | Transcription of analytical data from raw notebooks or instruments to the software that produces the laboratory report is accurate. | Laboratory | Laboratory QAM or designee |
| Analytical Methods and Procedures ¹ | (1) Required analytical methods (off-site laboratory) were used and any deviations were noted; and (2) the QC samples met performance criteria and any deviations have been documented. | Laboratory | Laboratory QAM or designee |
| | (1) Required analytical methods (off-site laboratory) were used and any deviations were noted; (2) the reporting limits specified in the QAPP were met; and (3) the QC samples met performance criteria and any deviations have been documented (spot check). | Internal | ERM QAM or designee |
| Data Qualifiers | Laboratory data qualifiers were defined and applied as specified in the methods, procedures, or the laboratory's QA manual. | Laboratory | Laboratory QAM or designee |
| | | Internal | ERM QAM or designee (spot check) |
| Communication | Required communication procedures were followed by field or laboratory personnel. | Internal | ERM QAM or designee |

Key:

ERM = Environmental Resources Management, Inc.; PM = project manager; QA = quality assurance; QAM = Quality assurance manager; QAPP = Quality Assurance Project Plan; QC = quality control

¹ Validation in accordance with the U.S. Environmental Protection Agency's National Functional Guidelines for Data Review will not be performed.

Table 5-3
Usability Assessment (Step III) Process
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Type of Data | MPC | Evaluation Procedure | Usability Report Information |
|--------------|---|--|---|
| Field | Precision, Accuracy/Bias, and Sensitivity and Quantitation Limits | None; the field data will be used in a qualitative way and, although MPCs were selected to ensure appropriate field evaluation, the data are used in a qualitative way and don't impact the usability of the analytical data to determine if the site requires remedial actions. | No comment required. |
| | Representativeness | <ul style="list-style-type: none"> · At least two samples were obtained per area of concern. · The extent of impacts was defined. · Field sampling was performed in a standardized manner that adhered to the procedures and requirements of this QAPP | <ul style="list-style-type: none"> · Discuss and compare issues for each matrix, analytical group, and concentration level, if any of the items listed was not met. · Describe the use limitations if representativeness was determined to be poor for a specific matrix, analytical group, or concentration level. |
| | Comparability | <ul style="list-style-type: none"> · Results were reported in appropriate units. · The same or similar sampling procedures were used in all investigation areas. · The same or equivalent analytical procedures were used during all phases of the investigation and remedy implementation. · The same or equivalent QA/QC requirements were followed in all investigative activities. | <ul style="list-style-type: none"> · Discuss and compare overall comparability for each matrix, analytical group, and concentration level. · Describe the use limitations if comparability was not met. |
| | Completeness | $\% \text{ Completeness} = \frac{\text{Valid Data Obtained} \times 100}{\text{Total Data Planned}}$ | <ul style="list-style-type: none"> · Discuss and compare overall completeness for each matrix, analytical group, and concentration level. · Describe the use limitations if completeness was not met. |
| Laboratory | Precision | $RPD = \frac{(\text{First Value} - \text{Second Value}) \times 100}{(\text{First Value} + \text{Second Value})/2}$ | <ul style="list-style-type: none"> · Discuss and compare overall field and laboratory duplicate precision data for each matrix. · Describe use limitations if overall precision is poor or when it is limited to a specific sampling or laboratory group, data package, matrix, or concentration level. |

Table 5-3
Usability Assessment (Step III) Process
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Type of Data | MPC | Evaluation Procedure | Usability Report Information |
|------------------------|-------------------------------------|--|---|
| Laboratory (cont'd) | Accuracy/Bias | $\%R = \frac{(Spiked\ Result - Unspiked\ Result) \times 100}{Spike\ Added}$ | <ul style="list-style-type: none"> · Discuss and compare overall matrix spike / matrix spike or laboratory control sample accuracy/bias data for each matrix. · Describe use limitations if overall accuracy/bias is poor or when it is limited to a specific sampling or laboratory group, data package, matrix, or concentration level. |
| | Representativeness | <ul style="list-style-type: none"> · Laboratory analyses were performed in a standardized manner that adhered to the procedures and requirements of this QAPP. | <ul style="list-style-type: none"> · Discuss and compare issues for each matrix, analytical group, and concentration level, if representativeness was not met. · Describe the use limitations if representativeness was determined to be poor for a specific matrix, analytical group, or concentration level. |
| | Comparability | <ul style="list-style-type: none"> · Results were reported in appropriate units. · The same or equivalent analytical procedures were used during all phases of the investigation and remedy implementation. · The same or equivalent QA/QC requirements were followed in all phases of the investigations and during remedy implementation. | <ul style="list-style-type: none"> · Discuss and compare overall comparability for each matrix, analytical group, and concentration level. · Describe the use limitations if comparability was not met. |
| | Sensitivity and Quantitation Limits | Compare quantitation limits to those required for the project in Tables 2-9 through 2-12 of the QAPP. | <ul style="list-style-type: none"> · Discuss and compare overall quantitation limits for each matrix, analytical group, and concentration level. · Describe the use limitations if the quantitation limits were not met for all samples. |
| | Completeness | $\% \text{ Completeness} = \frac{Valid\ Data\ Obtained \times 100}{Total\ Data\ Planned}$ | <ul style="list-style-type: none"> · Discuss and compare overall completeness for each matrix, analytical group, and concentration level. · Describe the use limitations if completeness was not met for any matrix, analytical group, or concentration level. |

Key: MPCs = Measurement performance criteria; QA/QC = quality assurance/quality control; QAPP = Quality Assurance Project Plan; %R = percent recovery.

Table 5-4
Usability Assessment Items for Consideration
Former Reid Hospital Site
1401 Chester Blvd, Richmond, Indiana

| Item for Consideration | Assessment Activity |
|---|--|
| Usability Assessment Documentation | Ensure all necessary information was provided. |
| Deviations | Determine the impact of deviations on the usability of data. |
| Sampling Locations | Determine if changes to sample locations meet the project objectives. |
| Sampling Procedures | Determine if changes to sampling procedures meet the project objectives. |
| Chain of Custody | Assess whether any problems with documentation or custody procedures prevent the use of the data. |
| Holding Times | Determine the acceptability of data from analyses performed outside holding times. |
| Damaged Samples | Assess whether data from damaged samples is usable and, if not, determine whether resampling is needed. |
| Analytical Methods | Evaluate the impact of deviations from specified analytical methods on data quality. |
| Quality Control Samples | Determine the effects of unacceptable QC sample results. |
| Matrix | Evaluate matrix effects on the usability of the data. |
| Meteorological Data and Site Conditions | Evaluate if meteorological and/or site conditions had an impact on the usability of the data. |
| Comparability | Assess whether the new data are comparable to previous results, considering concentration trends and sampling locations. |
| Completeness | Determine if missing information affects the data usability. |
| Data Restrictions | Specify the restrictions on use of data that did not meet the Measurement Performance Criteria. |
| Usability Decision | Assess if decisions can be made for the specific data that was collected to make them. |
| Measurement Performance Criteria | Discuss and compare the overall precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level and describe limitations on the use of project data. |

APPENDIX A
STANDARD OPERATING
PROCEDURES

Appendix A
Standard Operating Procedure #10
Soil Boring
Former Reid Memorial Hospital Site
1401 Chester Blvd, Richmond, Indiana

This Standard Operating Procedure (SOP) details the procedures to be used to sample soil borings and install temporary wells, and to survey the sampling locations.

Equipment and Supplies

The following is a listing of equipment and supplies to use during drilling of borings, soil sample collection, and installation of temporary wells (if applicable). Other unspecified equipment may also be used either in addition to or as a replacement (if it is functionally equivalent) for the following list.

- Site map with the locations of the soil borings marked.
- A copy of the Quality Assurance Project Plan (QAPP).
- Geoprobe® and stainless steel samplers equipped with disposable acetate liners.
- Photoionization detector (PID) or flame ionization detector (FID).
- Trowel, zip-lock bags.
- Disposable gloves.
- Indelible-ink markers.
- Field notebook and pens.

Field Documentation

See SOP #110 of the QAPP for field documentation procedures.

Decontamination

Decontamination procedures will be followed in accordance with SOP#190.

Sampling Equipment and Instrument Testing, Inspection, and Calibration

If not done before mobilization, perform sampling equipment and instrument testing and inspection as indicated in SOP #100 and perform calibration of the instruments.

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Boring Drilling

- Use the site map to locate the boring at the Site. If obstacles are present or subsurface clearance indicated the potential for a subsurface structure or utility, move the boring in the direction most appropriate for the purpose of the boring and obtain subsurface clearance for the new location.
- Check the specific boring location for the parameters for which samples are required and the depths at which they are required.
- Ask the driller to begin drilling the borehole.
- Describe and log each soil sample core recovered from each boring. Include the recovery length, composition, structure, grain size, density, sorting, color, and moisture content of the soil sample from visual observation. Use a Munsell® color chart to accurately identify the color of the soil.
- For each soil interval: (1) visually examine and describe the subsurface geology; (2) inspect the soil for visible evidence of contamination; and (3) perform field screening with a PID and/or FID for the presence of organic vapors by following the procedures below.

Field Screening for Organic Vapors

- Place a composite of each 2-foot soil core in a plastic bag that can be zipped and lock it.
- Place each plastic bag in a warm, shaded area.
- After approximately 10-15 minutes, open a small portion of the zipper and insert the probe of the PID or FID.
- Record the measurement after it stabilizes.

Soil Sampling Procedures

- For samples collected for VOCs analysis, follow the sampling procedures for 5035A sampling techniques as defined in IDEMs *Sampling Soil and Waste for Volatile Organic Compounds* guidance dated March 20, 2008 (Aug 15, 2012 Rev) including:

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Standard Operating Procedure #10
Soil Boring

Former Reid Memorial Hospital Site
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- Collect the number of soil samples from the predetermined depth intervals by placing a portion of soil from the core or the soil core into the appropriate laboratory-supplied container according to the procedures directed by the laboratory. Preserve the soil samples as necessary. See Worksheet #19 in Appendix A (TAL Quality Assurance Manual) of the QAPP for the appropriate containers, sample volume, and preservatives.
- Follow SOP #120 for the contents of the container label and to package, mark and label, and ship the sample containers. The laboratory to which samples will be submitted is listed in Table 2-2 of the QAPP.

Boring Surveying

- Retain a certified land surveyor to survey ground surface elevation to an accuracy of 0.01 foot and the eastern and northern coordinates of each boring with a horizontal accuracy of 0.1 foot.
- Use the State Plane Coordinate system for the boring coordinates.
- Locate the new borings in relation to the existing site surveys.

Appendix A
Standard Operating Procedure #20
Monitoring Well Installation,
Development, and Surveying
Former Reid Memorial Hospital Site
1401 Chester Blvd, Richmond, Indiana

This Standard Operating Procedure (SOP) details the procedures to be used to install, develop, and survey permanent monitoring wells at the Site.

Equipment

The following is a listing of equipment that will be used to install, develop, and survey monitoring wells. Other unspecified equipment may also be used either in addition to or as a replacement (if it is functionally equivalent) for the following list.

- Site map with the locations of the monitoring wells marked.
- A copy of the Quality Assurance Project Plan (QAPP).
- A variable flow, electric powered, pump capable of producing variable flow between approximately 0.25 gallons per minute (gpm) and 2 gpm.
- LDPE or Teflon-lined discharge tubing.
- Disposable polyethylene or polyvinyl chloride (PVC) bailers (if needed).
- A water level meter.
- Field notebook.

Field Documentation

See SOP #110 of the Quality Assurance Project Plan (QAPP) for field documentation procedures.

Decontamination

Decontamination procedures will be followed in accordance with SOP#190.

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Monitoring Well Installation

All monitoring wells will be installed in a continuously sampled soil boring or adjacent to a continuously sampled soil boring. Follow all applicable federal, state, and local regulations concerning groundwater monitoring well installation.

Monitoring Wells

- Advance the boring into unconsolidated deposits by using a drill rig equipped with 3.5”-inner diameter (ID) hollow-stem augers, or direct push 4.25” rods for 2-inch diameter wells. The soil will be sampled as indicated in SOP #10.
- Construct the monitoring wells with 2-inch inner-diameter polyvinyl chloride (PVC), 0.010-inch machine-slotted screen and solid 2-inch PVC pipe to grade, or 2-inch inner diameter Prepacked PVC, 0.010-inch machine-slotted screen and 2-inch PVC pipe to grade.
- Fill the remaining annulus between the well screen and the hollow-stem augers with 50-70 mesh silica sand (#5 global quartz) to 1 foot above the screened interval of the well.
- Place a 2-foot-thick bentonite seal above the sand pack. If the top of the sand pack is above the water table, bentonite pellets may be poured directly into the borehole and hydrated at the time of installation with commercial-grade distilled water.
- Fill the remaining annular space from the top of the seal to approximately six inches below ground surface (BGS) with a cement/ bentonite grout. The grout will contain 5% of bentonite by weight.
- Place a minimum 4-inch-diameter, locking, protective steel, stickup casing over the riser and set in concrete or, complete the wells at the surface with a bolt-down flush cover set in concrete.
- Use expandable, locking caps to seal the well casing at the surface.
- Cap and secure the protective casing with a keyed-alike lock that matches all the new ground water monitoring wells installed at the site.

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- Document the well installation in the field notebook and any other forms indicated in SOP #110.

Well Development

- Develop new monitoring wells following installation to help provide low-turbidity, representative groundwater samples.
- Start well development no sooner than 24 hours following the installation of the wells to allow the bentonite seal and grout to set.
- Evacuate groundwater during development with an electric submersible pump or dedicated disposable polyethylene or polyvinyl chloride (PVC) bailers. The pump's flow rate will be less than or equal to 2 gpm.
- Periodically during development, a surge block designed to displace water within the well screen will be inserted and removed from the well. This will be done to free sediment from within the wells sand pack for removal during development.
- Develop the wells until the turbidity in the water decreases based on qualitative observations or until five times the standing well volume have been removed. The development method and volume of water removed during development will be recorded in the field book and on the well construction diagram.

Well Surveying

- Retain a certified land surveyor to survey the eastern and northern coordinates of each well with a horizontal accuracy of 0.1 foot and the elevations of the top of inner casing on the north side of the well and ground surface elevations adjacent to the protective cover with a vertical accuracy of 0.01 foot and a horizontal accuracy of 0.1 foot.
- Use the State Plane Coordinate System for the boring coordinates.
- Locate the new monitoring wells in relation to the existing site surveys.

Appendix A
Standard Operating Procedure #30
Groundwater Sampling (Micro-Purge Method)
Former Reid Memorial Hospital Site
1401 Chester Blvd, Richmond, Indiana

This Standard Operating Procedure (SOP) details the procedures to be used to collect groundwater samples via the micro-purge method for laboratory analysis of site-specific compounds.

Sampling Equipment and Supplies

The following is a listing of equipment and supplies that will be used during sampling. Other unspecified equipment, where it is functionally equivalent, may also be used either in addition to or as a replacement for the following list.

- Site map with the locations of the monitoring wells.
- A copy of the Quality Assurance Project Plan (QAPP).
- A bladder pump capable of producing variable flow between approximately 100 milliliters per minute (mL/min) and 2 gallons per minute (gpm).
- MP-50 compressor Controller or similar.
- Low Density Polyethylene (LDPE) discharge tubing.
- Teflon or stainless steel bailers (if needed).
- A water level meter.
- Disposable gloves.
- Laboratory-supplied containers and shipping coolers.
- An in-line flow cell and water quality monitor capable of measuring dissolved oxygen, oxidation-reduction potential, turbidity, specific conductance, pH, and temperature.
- Containers to store the purged water.
- Preservatives.
- Ice.
- Indelible-ink markers.
- Labels.
- Chain-of-custody form.
- Field notebook and pens.

Field Documentation

See SOP #110 of the QAPP for field documentation procedures.

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Groundwater Sampling (Micro-Purge Method)
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Decontamination

Decontamination procedures will be followed in accordance with SOP#190.

Sampling Equipment and Instrument Testing and Inspection

Perform sampling equipment and instrument testing and inspection as indicated in SOP #100.

Static Water Level Measurement

- Collect static groundwater levels at the wells no sooner than 48-hours after their initial development to ensure that the final set of measurements is representative of equilibrium conditions (quasi-static water levels).
- Obtain static groundwater levels before sampling any of the monitoring wells. Obtain static groundwater levels from all the monitoring wells at the site in as short a time-span as possible; preferably within one day.
- Unlock the wells and remove the expandable pressure cap.
- Record in the field notebook whether the well was under a positive or negative pressure when the cap was removed, and if any was observed, allow the pressure to equilibrate for approximately 20 minutes before gauging the depth to water.
- Take the water level measurements by slowly lowering the meter tip and tape into the monitoring well until the buzzer and the light signal that liquid has been reached. Establish the water level measurement relative to the measuring point on the monitoring well, which will be the north side of the well or a point previously marked by a surveyor, if present.
- Raise and lower the meter tip and tape until the buzzer and light signal are repeated twice at a given point. If the water level changes (barometric compensation of a confined potentiometric surface), take measurements at intervals until a stable reading is obtained. Record all measurements and time of collection in the field notebook.
- Record the final stabilized depth-to-water measurement and the time when measured in the field notebook and in any other form indicated in SOP #110. This

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Standard Operating Procedure #30
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reading indicates the distance between the measuring point at the top of the well casing and the water.

- Measure and record the total depth of each well prior to purging and sampling the well in the field notebook and any other form indicated in SOP #110. This total depth measurement is to be used to judge if the well may have “silted up” or been damaged since the last sampling event; since this SOP details the micro-purge method for groundwater sampling, a minimum purge volume need not be calculated.
- Shut and lock the wells that will not have a water sample collected, if any.
- Record the measurements in the field notebook as indicated in SOP #110.

Well Purging

- Purge each monitoring well by: (1) using the procedures in the Indiana Department of Environmental Management’s (IDEM’s) *Micro-Purge Sampling Option* guidance dated June 3, 2005 (Revised Nov 3, 2009) (2) until the well goes dry; or (3) until three well volumes have been extracted. If procedure #1 cannot be completed to the necessary parameters, use one of the other two procedures.
- If a well goes dry, stop the purging procedure and allow it to recharge for 24 hours or less and then sample it with a clean Teflon bailer. Note that bailers will only be used if recharge rates are below functional pumping rates for the low-flow sampling methods.
- Record the type of well purging equipment, any operating settings, purge volumes, and any water quality measurements in the field notebook or on field sheets, as specified in SOP #110.

Sample Collection

- Obtain groundwater samples once purging is complete by disconnecting the flow-through cell and filling the sample containers directly from the discharge tubing or bailer.

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- Collect samples for other analytical fractions by pouring water directly into the container.
- Preserve the groundwater samples as necessary. See the QAPP for the appropriate containers, sample volume, and preservatives.
- Follow SOP #120 for the contents of the container label and to package, mark and label, and ship the sample containers. The laboratory to which samples will be submitted is listed in the QAPP.
- Secure the well caps and lock the protective casing at each location after sampling has been completed.

Appendix A
Standard Operating Procedure #100
Field Instrument Testing and Inspection
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Equipment to be used during the field sampling will be examined to certify that it is in proper operating condition. For equipment owned by ERM, this includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment problem is not overlooked, and to ensure all necessary repairs to equipment have been carried out. For rental equipment, this second maintenance step is not necessary. In addition, all field equipment will be cleaned at the beginning of each day and between samples to help ensure proper performance.

Field instruments will be checked before they are shipped or carried to the field and daily before use. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Initial and daily preventive maintenance for all instruments will include the following;

- Check battery strength and/or charge strength before use. Replace or recharge if insufficient.
- Check any tubing and connections. Ensure that tubing is in good shape, and that all connections are snug. Replace and/or tighten if necessary.
- Check all electrical connections and wiring. Make sure all connections are clean and tight, and that any wiring is dry and free of cracks and exposed insulation.
- Check all inlet and outlet filters. If dirty and/or wet, replace before use.

Critical spare parts, such as tape, pH probes, electrodes, and batteries will be kept on site to minimize instrument downtime. Should field equipment fail, the Field Leader will be contacted immediately and will either provide replacement equipment or have the malfunction repaired immediately. Backup instruments and equipment will be available on site or within one-day shipment to avoid delays in the field schedule.

The equipment type, manufacturer, supplier, inspections performed, findings, and solutions (as applicable), will be recorded in the field notebook.

Appendix A
Standard Operating Procedure #110
Field Documentation
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This Standard Operating Procedure (SOP) describes the procedures to be used to document field activities in a field notebook.

1. Use bound field notebooks assigned to the Site to document the field activities performed at the Site and store them in a secure location when not in use. Permanently label each notebook with the Site name, ERM project number, and notebook number on the front cover.
2. Describe in field notebook entries the data- and sample-collection activities performed in as much detail as possible so that field staff going to the Site could reconstruct a particular situation without reliance on memory.
3. Print all entries with waterproof indelible ink and do not make any erasures. If an incorrect entry is made, cross out the information with a single strike mark so that it remains legible, initial the error, and note the date of the change. The correction must be written adjacent to the error. Do not remove any pages, even if mutilated or illegible, from the notebook.
4. Add the following to the title page of each notebook:
 - Notebook number,
 - Project name and number,
 - Project Site address and Site contact telephone numbers,
 - Emergency telephone numbers,
 - A return address should the notebook get lost,
 - Project start date, and
 - Project end date, when available.
5. Begin each day's entry on a new page. At the beginning of each day, record the date, start time, weather, names of all sampling team members present, level of personal protection being used, planned activities for the day, and the signature of the person making the entry.
6. Enter the names of visitors to the Site and the purpose of their visit in the field notebook each day that they are present on the Site.
7. Account for times of inactivity and times when multiple tasks are being performed concurrently. Reference other notebooks with supplemental information.

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Standard Operating Procedure #110
Field Documentation
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8. Record instruments used, inspections and calibrations performed, and procedures followed (e.g., manufacturer's instruction, SOP).
9. Record measurements made (e.g., photoionization detector measurements, depths to groundwater, and distances to locate soil borings from benchmarks), including any duplicate field measurements.
10. Enter the equipment used to collect samples; the time of sampling; sample number and physical description; depth at which the sample was collected; whether the sample is a grab or a composite (and if composited, how it was composited); volume and number of sample containers; preservation; type of sample (investigative, duplicate, trip blank, etc.); the unique sample number corresponding to each QC sample; any deviation from the procedures in the Quality Assurance Project Plan (QAPP); any photographs taken and their description; and requested analyses. Complete chain-of-custody forms and note on the chain of custody if field screening or previous data indicates that a sample has potentially high concentrations to notify the lab of the possibility. If field screening indicates that a sample has potential matrix interferences such as apparent sludge or oil, provide extra sample volume as possible based on the amount of sample volume available. Sample designation procedures are presented in SOP # 120.
11. Note soil lithological description in the field notebook and/or on the boring log Also note sample information on the boring log if applicable.
12. Record any deviations from the sample collection/handling procedures provided in the previous reports or in the corresponding work plan in the field notebooks, along with appropriate explanations.
13. If photographs of the Site or the sample locations are taken, enter a photograph log with the photo number, a description of the cardinal direction of the photograph, and a description of what was photographed.

Appendix A
Standard Operating Procedure #120
Sample Labeling, Packaging, Marking and Labeling, and Shipping
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This Standard Operating Procedure describes the procedures to be used to prepare the sample container label and to package, mark and label, and ship the sample containers.

Sample Label Contents

After placing the sample into an appropriate container, the field sampler will affix a properly completed sample label or complete the laboratory supplied label. If not printed from a computer, the information should be hand-written in the label making sure the number 5 and the letter “S” are clearly different. All information will be recorded by the field sampler on the sample label in water resistant ink. All samples will be identified with labels that are securely attached to the sample containers. Each label will include the following information:

- Unique sample identification;
- Site name;
- Name and affiliation of the sampler;
- Date and time of collection;
- Requested analyses; and
- Preservatives used (if any).

The information described above will be carefully recorded on the sample label, field notebook, field forms (as appropriate), and chain-of-custody for each sample.

Sample Packaging, Marking and Labeling, and Shipping

Sample packaging, marking and labeling, and shipping procedures will be performed as follows:

- After sample preservation, where required, wipe off the exterior of the sample containers, tighten caps, complete sample paperwork as indicated in the QAPP, and attach the sample labels to the sample containers.
- Place a large plastic bag (i.e. garbage bag) inside of the cooler.

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Standard Operating Procedure #120
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- Place the sample containers inside the large plastic bag, inside the cooler and place packing material around the samples to minimize the possibility of container breakage.
- Add wet ice sealed in individual self-sealing plastic bags to maintain the temperature of 6°C or below;
- Fill the remaining space in the cooler with additional packing material and tie the large plastic bag closed;
- Enclose chain-of-custody forms and any other shipping or sample documentation accompanying the shipment in a self-sealing plastic bag and place them inside the cooler;
- Close the cooler and seal it with tape. If the cooler has a drain, tape it shut. Seal the coolers with custody seals in such a manner that the custody seal would be broken if the cooler were opened. Then, cover the custody seals with clear plastic tape. If the samples will be delivered by the sampling crew to the laboratory, sealing is not required.

The samples will be shipped on the same day they are collected via an overnight carrier or delivered to the laboratory by the laboratory's courier or the ERM Field Leader on the same day of collection or before 10 am on the next day. The laboratory will be notified at the time of shipment. If an overnight carrier, such as FedEx, is used and delivery will be on a Saturday, Saturday delivery will be marked on the delivery slip and the laboratory will be notified of the Saturday delivery at the time of shipment.

Samples will be designated as described below:

- Groundwater will be designated by monitoring well as MW-X-YYYYMMDD-01, where X will be the monitoring well identifier.
- Soil samples will be designated as SB-X-Depth-YYYYMMDD-01, where X is the sequential number of the soil boring.

QC samples will be designated as follows:

- Trip blanks will be designated as TB-X-YYYYMMDD-01, where X will be sequential sample number.
- Rinsate blanks will be designated as RB-X-YYYYMMDD-01, where X is the sequential sample number.
- Field duplicates will be designated DUP-X-YYYYMMDD-01, where X is the sequential sample number.

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- Matrix spike/matrix spike duplicate (MS/MSD) samples will be designated as MS/MSD analysis in the “Special Instructions” section of the chain-of-custody forms. No specific name designation will be necessary.

Appendix A
Standard Operating Procedure #130
Field Sample Custody
Former Reid Memorial Hospital Site
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This Standard Operating Procedure describes the procedures for field sample custody. To provide documentation necessary to trace sample possession from the time of collection to the time of receipt by the analytical laboratory, a chain-of-custody record will be completed and will accompany each shipment of samples to the laboratory. A copy of the chain-of-custody form is attached in the Quality Assurance Project Plan (QAPP). The procedures for field sample custody are as follows:

- Samples will be accompanied by a properly completed chain-of-custody form supplied by the laboratories. When transferring possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time of the exchange on the records. This record documents the transfer of custody of samples from the sampler to another person, to a laboratory, or to/from a secure storage area.
- The field portion of the chain-of-custody documentation will include the project name, the sample number, date and time of collection of each sample, whether the sample is grab or composite, number of containers, preservation used, type of quality control sample if applicable (especially which sample is to be used for the matrix spike/matrix spike duplicate analysis), analyses requested, and any information regarding lack of preservation, suspected high concentrations (based on field screening or previous analytical results), reason for additional containers, whether the sample should be held, and any other note for the laboratory.
- Samples will be properly packaged for shipment and dispatched or delivered to the appropriate laboratory for analysis, with a separate signed chain-of-custody enclosed in each sample box or cooler. Shipping containers will be secured with tape and custody seals for shipment to the laboratory. A copy of the chain-of-custody form will remain with the ERM samplers to be placed in the project files upon arrival at the office.
- If an overnight carrier or courier is used for shipment, their air bill will be used as the record of shipment. Receipts from the air bill will be retained as part of the custody documentation. Commercial carriers are not required to sign off on the chain-of-custody forms as long as the custody forms are sealed inside the sample coolers, and the custody seals remain intact.

Appendix A
Standard Operating Procedure #180
Surface Wipe (Smear) Sampling
Former Reid Memorial Hospital
1401 Chester Boulevard, Richmond, Indiana

This Standard Operating Procedure (SOP) details the procedures to be used to conduct surface wipe (smear) sampling on non-porous surfaces. Characterization of surfaces may provide indication of the degree of impact to these features.

Sampling Equipment and Supplies

The following is a listing of equipment and supplies that will be used during sampling. Other unspecified equipment, where it is functionally equivalent, may also be used either in addition to or as a replacement for the following list.

- Site map with the sample locations
- A copy of the Health and Safety Plan and a copy of the Quality Assurance Project Plan (QAPP).
- 100 cm² sampling guide
- Sample wipe
- H&S equipment
- Disposable gloves.
- Indelible-ink markers.
- Chain-of-custody form.
- Field notebook and pens.

Field Documentation

See SOP #110 for field documentation procedures.

Decontamination

Perform sampling equipment and instrument decontamination as indicated in the HASP.

Sample Point Location

The location and number of sampling points should be determined as part of the development of a work plan for the project site. The following provides recommendations on how to select sampling locations:

Survey Techniques and Data Acquisition

Surface Wipe

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Surface Wipe (Smear) Sampling
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- Prepare the surface by brushing off any loose debris or dirt.
- Lay or tape the 100 cm² sampling grid onto the non-porous surface.
- Open the sampling container and remove the pre-moistened wipe using a gloved hand.
- Wipe the surface using a firm “S” style stroke covering the entire surface (edge to edge) of the sample guide.
- Fold the exposed wipe inwards (fold in half with surface wiped on the inside).
- Repeat the firm S-stroke using the once-folded wipe at a right angle to the previous direction of travel.
- Fold the exposed wipe inwards again (fold in half with surface wiped on the inside).
- Using the twice-folded wipe, follow the firm s-stroke starting at the original point, and follow the same direction of travel.
- Place the wipe in the laboratory provided container or zip-style bag.
- Remove gloves and discard templates between sample locations.
- Complete the survey using the same techniques for all remaining points.
- If obstructions are encountered (e.g. debris) that prevent a location from being used, offset from that location in the most appropriate direction

Appendix A
Standard Operating Procedure #190
Equipment Decontamination Procedures
Former Reid Memorial Hospital Site
1401 Chester Boulevard, Richmond, Indiana

This Standard Operating Procedure (SOP) details the procedures to be used to decontaminate reusable sample equipment (i.e. drilling tooling, sample pumps, etc.)

Sampling Equipment and Supplies

The following is a listing of equipment and supplies that will be used during sampling. Other unspecified equipment, where it is functionally equivalent, may also be used either in addition to or as a replacement for the following list.

- A copy of the Quality Assurance Project Plan (QAPP).
- Alconox® or similar detergent
- Potable water
- H&S equipment
- Disposable gloves.
- Buckets or spray bottles
- Plastic Sheeting
- Pressure Washer/Steam Cleaner

Field Documentation

See SOP #110 for field documentation procedures.

Reusable Equipment Decontamination Procedures

All non-dedicated/reusable sampling equipment will be decontaminated between sample locations via the following procedure:

- Brush off excess dirt/debris with a scrub brush;
- Rinse with an Alconox® or similar solution and/or steam clean as applicable
- Rinse with potable water
- Allow to air dry
- Keep equipment wrapped in plastic sheeting when not in use

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Standard Operating Procedure #190
Equipment Decontamination Procedures
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- Nitrile gloves will be worn throughout the decontamination process. Gloves will be changed before the start of decontamination, as well as before reassembling sampling equipment after decontamination is complete.

APPENDIX B
TEST AMERICA
QUALITY ASSURANCE MANUAL

Quality Assurance Manual

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**Quality Assurance Manual
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| | |
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| CA-Q-S-009 | Root Cause Analysis |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOPs) |
| CW-Q-S-004 | Management Systems Review |
| CW-L-S-002 | Internal Investigation of Potential Data Discrepancies and Determination for Data Recall |
| CA-L-S-002 | Subcontracting Procedures |
| CW-L-P-004 | Ethics Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CW-F-P-002 | Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CA-C-S-001 | Work Sharing Process |
| CA-T-P-001 | Qualified Products List |
| CA-T-P-002 | Selection of Calibration Points |
| CA-I-P-002 | Electronic Reporting and Signature Policy |
| CW-F-S-007 | Controlled Purchases Policy |
| CA-Q-M-002 | Corporate Quality Management Plan |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |

REFERENCED LABORATORY SOPs

TestAmerica St. Louis Standard Operating Procedures are listed in [Appendix 7](#).

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica St. Louis's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with U.S. Department of Energy Quality Systems for Analytical Services (QSAS, current revision), U.S. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM, current version), The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in [Appendix 3](#). The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense/Department of Energy, *Quality Systems Manual, Version 5.0*, July 2013.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 19th, 20th and 21st, and on-line Editions.
- U.S. Department of Energy Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- U.S. Department of Energy Order 414.1C, *Quality Assurance*, June 17, 2005.
- U.S. Department of Energy, *Quality Systems for Analytical Services*, Revision 2.9, January 2012.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Federal Register 10CFR 50 Appendix B
- Toxic Substances Control Act (TSCA).
- ASME NQA-1-2000 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- ASME NQA-1-1994 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- Federal Register 10CFR21 and 10CFR50.55e

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization. Refer to [Appendix 4](#) for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in [Appendix 3](#). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Technical Directors and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed **annually** by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica St. Louis is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Executive Officer, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica St. Louis is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's St. Louis laboratory.

4.2.2 Laboratory Director (LD) or Designee

The St. Louis Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to his/her respective General Manager (GM). The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific Responsibilities include, but are not limited to:

- The Laboratory Director is responsible for maintaining positive operating margin to the company at the laboratory level and for meeting and exceeding the annual budget.
- Ensures that personnel are free from commercial, financial and other undue pressures which might adversely affect their quality of work
- Supervise all laboratory personnel and provide guidance and direction as needed.
- Ensure that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Responsible for ensuring compliance and integration of facility operation with corporate and regulatory policies and procedures.

- Ensures that appropriate corrective actions are taken to address issues identified by external and internal audits.
- The laboratory Director has signatory authority for the QAM, policies, SOPs and contracts (as defined by TestAmerica policy).

4.2.3 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation, maintenance and improvement of the quality system.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary the procedures may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.

- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Has final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of the analytical data.
- Evaluation of the thoroughness and effectiveness of training.
- **Compliance with ISO 17025** (where applicable)
- Providing Quality Systems training to all new personnel and ensuring that all personnel understand their contributions to the quality system.
- Evaluate the effectiveness of training.
- Has signatory authority over the QAM, SOPs and policies pertaining to QA/QC
- Compliance with the NELAC Standards (where applicable)
- Compliance with the QSM (where applicable)

4.2.4 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e. SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

4.2.5 Technical Director

The Technical Director(s) report(s) directly to the Laboratory Director. The scope of responsibility ranges from the new hire process and existing technology through the on going training and development programs for existing analysts and second and third generation instrumentation.

Specific responsibilities include:

- Assists in coordinating, writing and reviewing SOPs.
- May assist in the review of proposals

- Solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients.
- Investigates technical issues identified by QA, and directs evaluation of new methods.
- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

4.2.6 Manager of Project Management/Customer Service Manager

In addition to filling the requirements of Project Manager for key accounts, he/she fulfills supervisory duties and responsibilities. As Manager, he supervises the Project Management staff, sets standards for and monitors productivity, manages the assignment of accounts and the daily workload and tracks and maintains information for various revenue reports. With the QA Manager, he determines acceptable corrective actions for the nonconformance occurring within his group, develops and reviews standard operating procedures for the group.

Additional responsibilities include:

- Has signatory authority for final reports.
- Training of the Project Management staff
- Notify supervisors of incoming projects and sample delivery schedules
- Coordinate requests for sample containers and sample pick-up/deliveries

4.2.7 Project Manager

- Coordinates and manages customers' projects through all phases of laboratory operations, ensuring fulfillment of TestAmerica's commitment to client requirements, error-free work, and on-time delivery.
- Responsible to ensure that clients get timely responses to status inquiries, resolutions to problems and the agreed upon deliverables
- Discusses with clients any project related problems, resolves service issues and coordinates technical details with the lab staff
- Responsible for staff familiarization with specific quotes, sample log-in review and final report accuracy and completeness
- Maintains communications with clients and Account Executives and serves as a liaison between clients and laboratory operations to meet client's needs.
- Works closely with business unit personnel to manage quotations and change orders for existing scopes of work.
- Generates narratives outlining project observations, QC excursions, and laboratory comments.
- Has signatory authority for final reports.

4.2.8 Department Manager/Supervisor

The Department Manager/Supervisor is responsible for the overall operations of a specific laboratory area.

These responsibilities include but are not limited to:

- Meeting client satisfaction goals, managing the human resources within the department, and ensuring health and safety and quality assurance plan compliance.
- Serves as a technical resource to department employees, as well as Project Managers, sales personnel, and clients.
- Make recommendations to laboratory management in regard to process improvements.
- Ensure analysts in their department adhere to applicable SOPs and the QAM.

4.2.9 Chemist/Analyst

- Laboratory analysts are responsible for the generation of data by preparing and analyzing samples according to written SOPs and client requirements.
- They are responsible for understanding the requirements in the QAM and the SOPs associated with their specific function.
- Perform the initial technical review of sample preparation information, calculations, qualitative identifications and raw data with the authority to stop, accept, or reject data based on compliance with self-defined QC criteria.
- The laboratory analyst also provides prompt documentation and notification to the Group Leader of problems or anomalies detected.
- Monitor, calibrate, and maintain standard laboratory equipment such as refrigerators, ovens, water systems, and pipettes, and instrumentation, as necessary.

4.2.10 Environmental Health and Safety Coordinator

- The Environmental Health and Safety Coordinator is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment.
- Monitors all areas for unsafe conditions, acts, and potential hazards. Enforces environmental, health, and safety policies and procedures. Maintains regulatory compliance with local, state, and federal laws.
- Makes safety and health recommendations to laboratory management in conjunction with the facility safety committee.
- Develops and maintains the facility's health and safety and waste disposal procedures.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.

- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.11 Radiation Safety Officer (RSO)

- Under the direction of the Laboratory Director, implements the radiation protection program that, as a minimum, provides compliance with pertinent regulatory requirements, license provisions, and the Radiation Protection Program.
- Maintains direct access to the Laboratory Director on matters relating to radiological protection.
- Maintains sufficient organizational independence to review and evaluate activities involving the use of radioactive materials.
- Provides Authorized Users and radiation workers with the instruments, protective devices, dosimetry, training, and other items needed to perform their work in accordance with the radiological protection program elements.
- Maintains original copies of all St. Louis licenses/permits, including attachments and amendments, for radioactive materials.
- Directs program to monitor and control radioactive materials throughout the laboratory
- Conducts radiation safety training
- Maintains inventory of standards, tracers, and radiological samples
- Manages segregated area for storing radioactive and mixed wastes

4.3 Deputies

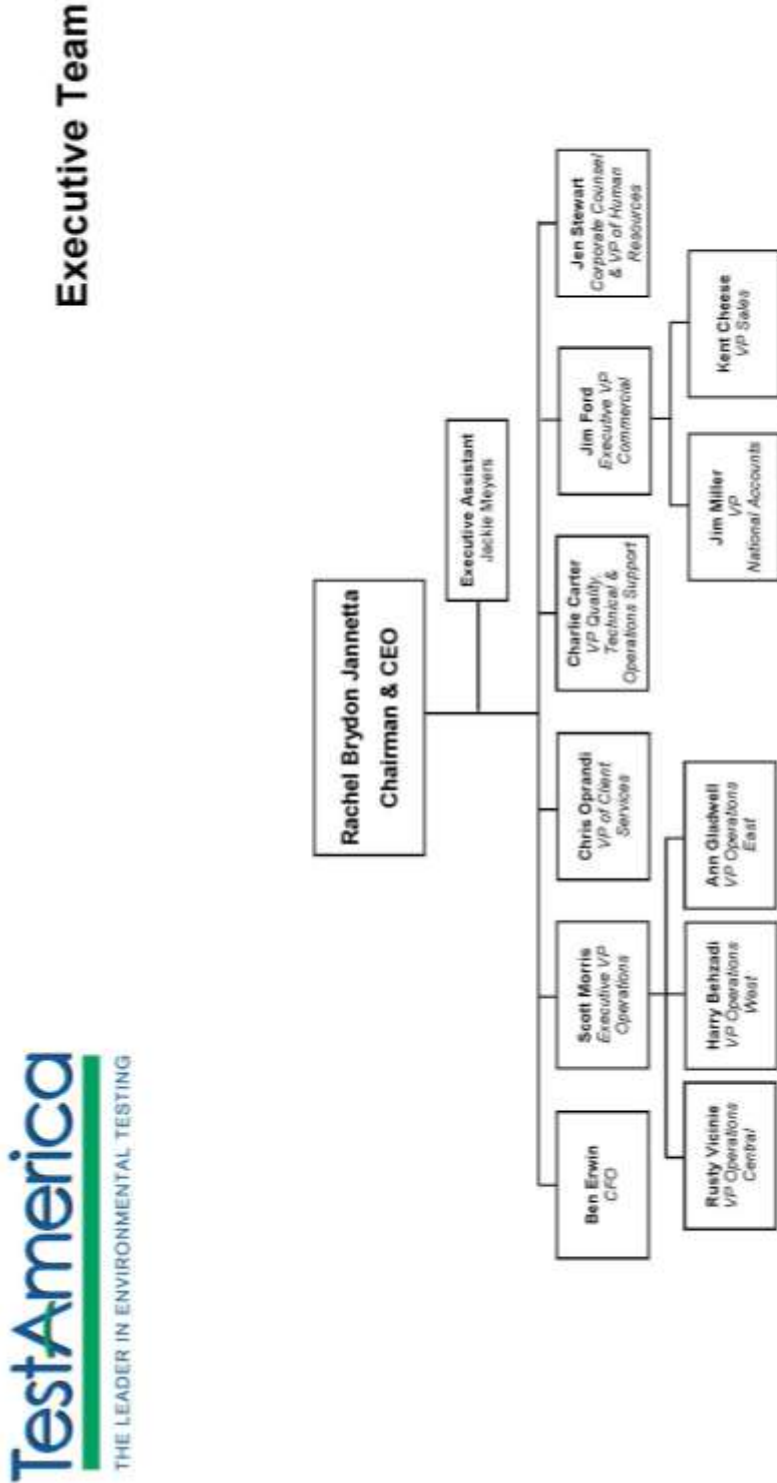
The following table defines who assumes the responsibilities of key personnel in their absence:

| Key Personnel | Deputy |
|--|---|
| Elaine Wild* Laboratory Director | Aaron Dickson Lab Operations Manager |
| Marti Ward Quality Manager | Tony Byrd Quality Assurance Specialist |
| Kristen Ely* Inorganics Technical Manager | Matt Souris [Metals Deputy] Metals Analyst Jacob Boyd [Wet Chem Deputy] Wet Chem Group Lead |
| Chris Hough* Radiochemistry Technical Manager | Rachel Muller [Count Room Deputy] Radiochemistry Analyst Supervisor Sarah Bernsen [Prep Deputy] Radiochemistry Prep Supervisor |
| Michael Ridenhower EHS Coordinator | Terry Romanko* Technical/QA Director |
| Michael Ridenhower Radiation Safety Officer | Terry Romanko* Technical/QA Director |
| Rhonda Ridenhower Manager of Project Management | Jayna Awalt Project Manager |
| Jeff Winkler* Extractable Organics Technical Supervisor | Aaron Dickson Lab Operations Manager |
| Andrew Buettner* Volatile Organics Technical Manager | Gary Bonkoski Volatile Organics Analyst |

In the event that key Technical Managers are absent for a period exceeding 15 consecutive calendar days, the deputy will temporarily perform the absentee's functions. If the absence exceeds thirty-five consecutive calendar days, the primary accreditation body shall be notified in writing.

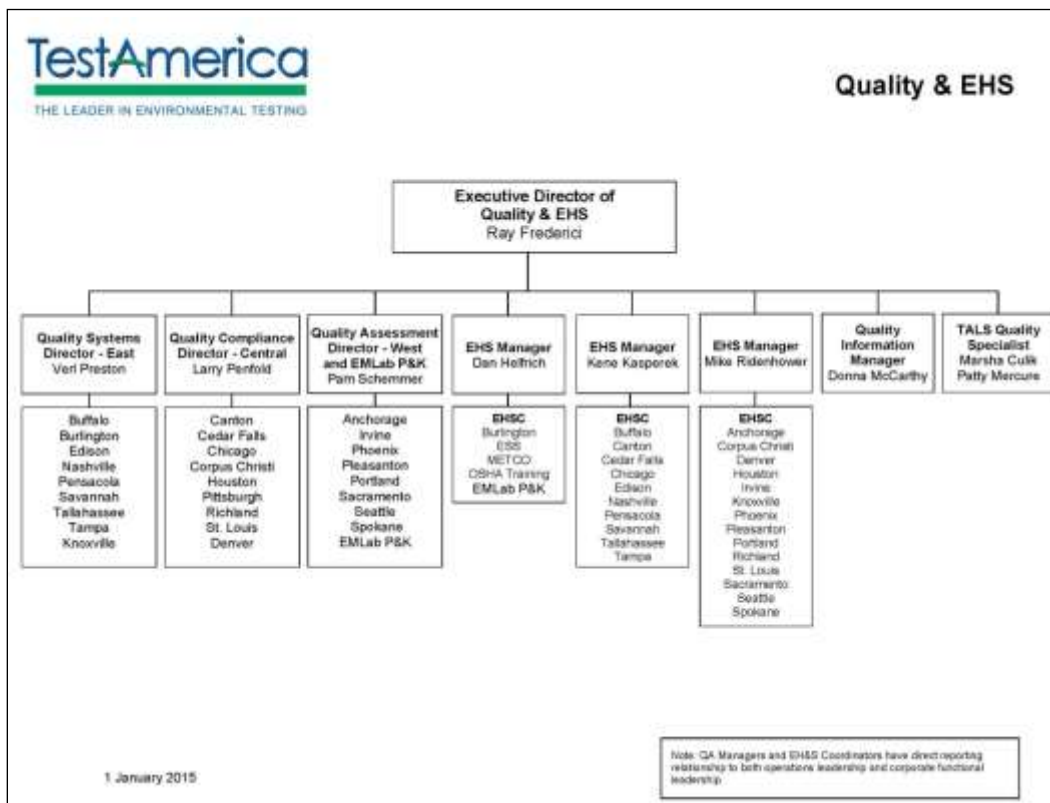
Technical Managers are designated with an asterisk (*).

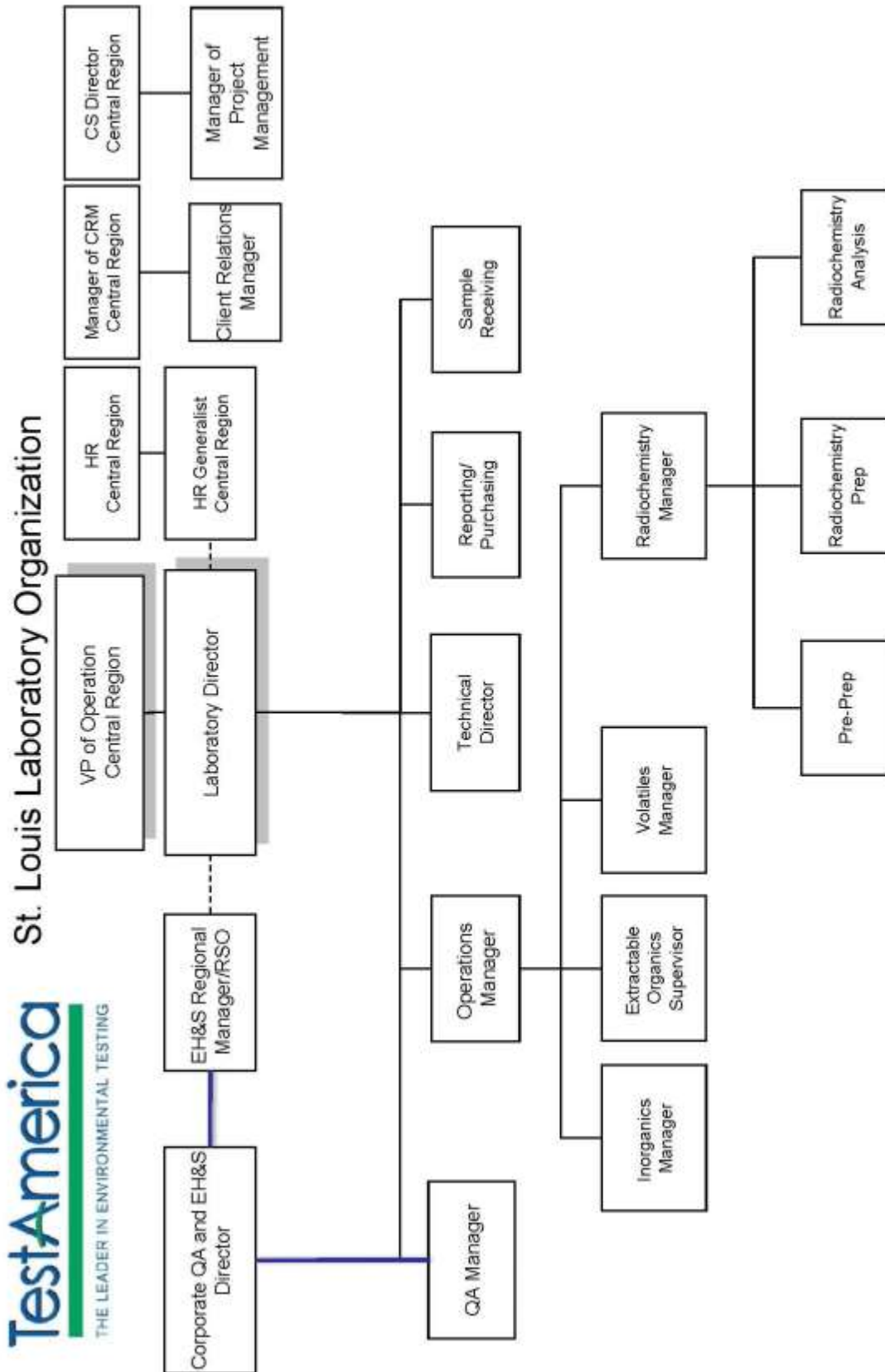
Figure 4-1. Corporate and Laboratory Organization Charts



12 August 2014







Note: QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.

SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.
- ❖ TestAmerica St. Louis' policy includes compliance with the Department of Defense QSM and the Department of Energy QSAS.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for familiarizing themselves with the quality program documentation and implementing those policies and procedures to ensure the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).

- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums
- Laboratory Waste Management Plan
- Laboratory Radiation Safety Program

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies

- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "*analytical quality control*". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit/Minimum Detectable Activity/Detection Limit) or quantified (Reporting Limit/Limit of Quantitation).

5.5 Criteria for Quality Indicators

The laboratory maintains Quality limits Reference Data through the LIMS containing the precision and accuracy acceptability limits for performed analyses. This data is managed by the laboratory's QA department. Printed and/or electronic copies of method specific QC limits are available upon request. Unless otherwise noted, limits are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP ST-QA-0014 and Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated to show warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. See SOP ST-QA-0014 "Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts".

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. ST-QA-0023, "Control of Records".

The laboratory QA Department also maintains access (controls) to various references and document sources integral to the operation of the laboratory. This includes reference methods, regulations and instrument manuals (hard or electronic copies).

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, validation requests and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a technical manager submits a draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version

information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years. When related to DoD (Department of Defense) work, the review will be done annually. Revisions are made as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder.

For changes to SOPs, refer to SOP No. CW-Q-S-002, "Writing a Standard Operating Procedure SOP" and laboratory SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

Forms, worksheets, work instructions and information are organized electronically by department in the QA folder on the network server. There is an index. Hard copies are kept in QA files. In order to develop a new form, worksheet or work instruction, the user submits a draft to the QA Department and technical manager for suggestions, approval and validation (where required) before use. Upon approval, QA personnel add the identifying control information to the document. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 14.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation. SOP ST-PM-0001, "Project Setup and Quote", outlines the process at the TestAmerica St. Louis laboratory.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Sales Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Management Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Legal Contracts Director, Account Executive or local customer Service Manager or Project Manager then submits the final proposal to the client. In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. A copy is kept in the Project Management directory on the network server.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log or e-mail chain of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the Project Manager's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

Project Manager's are the primary client contact and they ensure resources are available to meet project requirements. Although Project Manager's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources is sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, a "Client Requirement Memo" may be associated with each sample lot as a reminder of special sample receipt instructions and analytical requirements.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation may include letters, e-mails, variances and/or contract addendum.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the Client Requirement Memo and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request".

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers/Directors are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accreditation work where required.

For Department of Defense/Department of Energy projects the subcontractor and/or Work Share laboratories used must have an established and documented laboratory quality system that complies with DoD QSM/DOE QSAS requirements. The subcontractor and/or Work Share laboratories are evaluated following the procedures outlined below. The subcontractor and/or Work Share laboratory must receive project-specific approval from the DoD/DOE client before any samples are analyzed.

The DoD QSM requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be accredited by DoD or its designated representatives.
3. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
4. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

The DOE QSAS has the following requirements for subcontracting:

“The laboratory shall not use any sub-tier laboratories or subclients (including those possessing the same or similar corporate name) for performance of work under this specification without written approval from the Procurement Representative. The laboratory using the sub-tier laboratory or sub-client shall document and is responsible for ensuring that such sub-client meets all of the requirements of this specification, including being available for client inspections and audits.

Some clients may not allow any subcontracting to third party (sub-tier) laboratories. If this is the case, then this will be specifically noted in the site-specific contracts via Contracts, Task Orders, Laboratory Delivery Orders, etc.”

Project Managers (PM), Customer Service Managers (CSM), or Account Executives (AE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Account Executive (AE) or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors, A2LA accreditation or State Certification).
- Firms identified in accordance with the company’s Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC accreditation laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

With the exception of DoD and DOE programs noted above, all TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must

provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers and Sales Personnel.

8.3 Oversight and Reporting

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or EDS, AEs or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accreditation work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

With the exception of DoD and DOE programs, the Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001, laboratory SOP ST-QA-0037, "Procurement of Quality Related Items" and ST-QA0002, "Standard and Reagent Preparation".

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOPs.

The procedure for purchasing/ordering quality related items can be found in the laboratory SOP ST-QA-0037, "Procurement of Quality Related Items".

9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDS) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Radiochemical standards can be re-verified and a new expiration date applied. See SOP ST-QA-0002, "Standard and Reagent Preparation".

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- $\mu\text{mho/cm}$ (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified “clean” by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer’s certification and traceability statements are maintained in electronic files on the network server. These records include date of receipt, lot number (when applicable), and expiration date (when applicable).

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Standards and reference materials are stored separately from samples. Radiochemical standards are stored in a controlled access cabinet. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica’s Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer’s operation manual is accessible to the laboratory.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc. As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

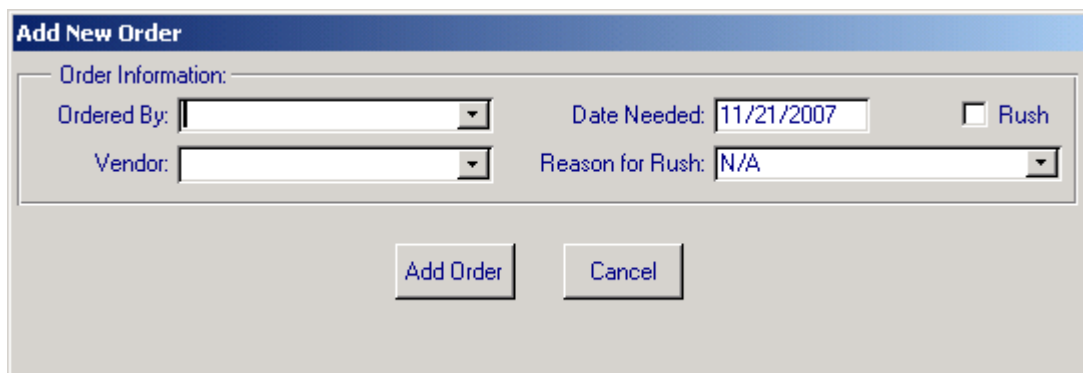
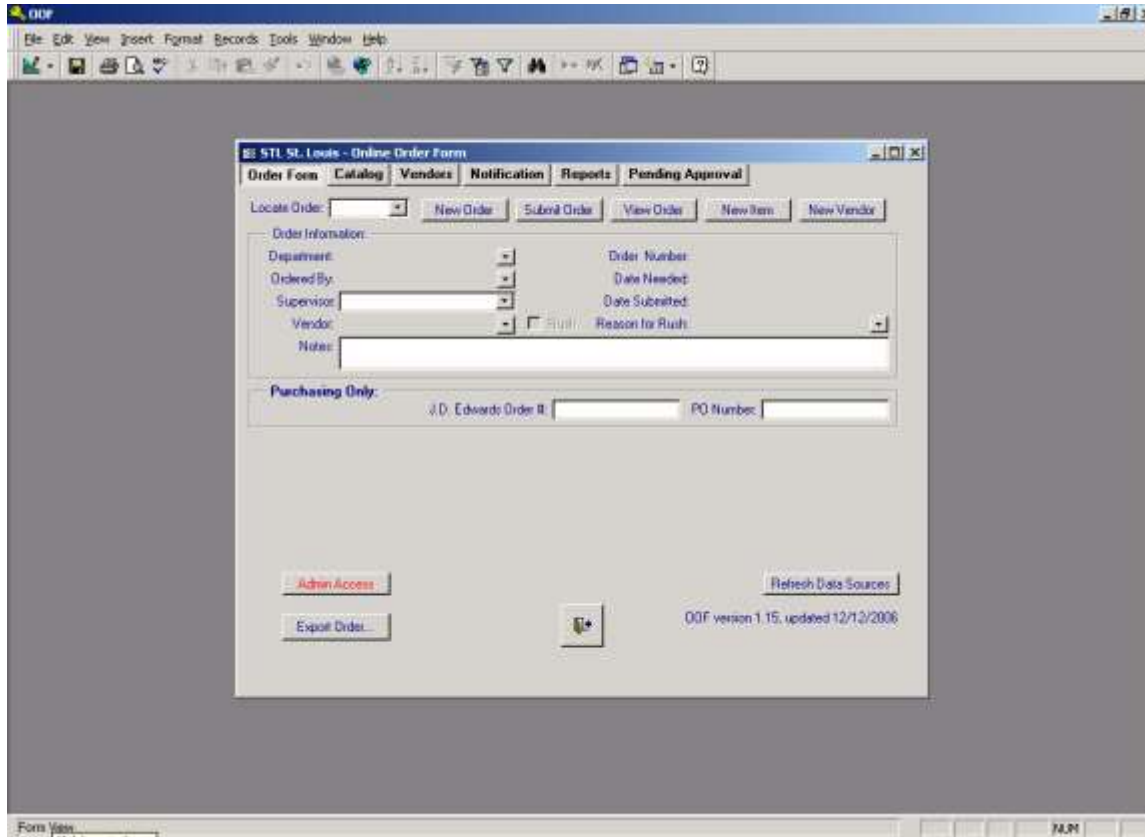
The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the J.D. Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

Figure 9-1.
Electronic Order Form



SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in the laboratory's Validation Database.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP ST-QA-0036 "Non-conformance Memorandum (NCM)/Validation Request and Corrective Action Processes".

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a

systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the QA Manager or Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the case narrative sent with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non- NELAC state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. For DOE and other programs where required, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures and will be entered into the LIMS non-conformance data base. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

When applicable (i.e. DOE and DoD projects), the laboratory notifies affected clients of potential data quality issues. Corrective actions taken to resolve the issues are submitted to the client in a timely and responsive manner.

For projects invoking Federal Regulation 10 CFR21, laboratory SOP ST-QA-0042, "Evaluating and Reporting of 10 CFR 21 Defects and Non-compliances", shall be followed.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Monthly the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may need to be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, Technical Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Validation Requests (refer to SOP ST-QA-0036).

For DOE, DoD and other programs where required, the client will be informed of proposed corrective actions.

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc...

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Validation Request - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

Health and Safety violations are documented in the EH&S Quarterly Inspection Reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or Validation Request must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or Validation Request is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and Validation Request is entered into a database for tracking purposes and a monthly summary of all corrective actions may be printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and Validation Requests for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or Validation Request.

Table 12-1 includes *examples* of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out and not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 12-1. Example – General Corrective Action Procedures

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|---|-----------------------------|---|
| Initial Instrument Blank (Analyst) | - Instrument response < RL. | - Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc... |

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|--|--|---|
| Initial Calibration Standards <i>(Analyst, Technical Manager(s))</i> | - Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP. | - Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source) <i>(Analyst, Technical Manager(s))</i> | - % Recovery within control limits. | - Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards <i>(Analyst, Data Reviewer)</i> | % Recovery within control limits documented in QC Browser database | - reanalyze standard -if still unacceptable, recalibrate and rerun affected samples |
| Matrix Spike / Matrix Spike Duplicate (MS/MSD) <i>(Analyst, Data Reviewer)</i> | - % Recovery within limits documented in the LIMS | - If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers. |

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|--|--|---|
| Laboratory Control Sample (LCS) (Analyst, Data Reviewer) | - % Recovery within limits specified in the LIMS | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates (Analyst, Data Reviewer) | - % Recovery within limits of method or within three standard deviations of the historical mean. | - Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB) (Analyst, Data Reviewer) | < Reporting Limit ¹ | - Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. |
| Proficiency Testing (PT) Samples (QA Manager, Technical Manager(s)) | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |
| Internal / External Audits (QA Manager, Technical Manager(s) Laboratory Director) | - Defined in Quality System documentation such as SOPs, QAM, etc... | - Non-conformances must be investigated through Validation system and necessary corrections must be made. |

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management) | - SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall. | - Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002. |
| Client Complaints (Project Managers, Lab Director/Manager, Sales and Marketing) | - | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). |
| QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, Technical Manager(s)) | - QAM, SOPs. | - Corrective action is determined by the type of issue. For example, NCMs and Validations for the month are reviewed and possible trends are investigated. |
| Health and Safety Violation (Safety Officer, Lab Director/Manager, Technical Manager(s)) | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected |

Note:

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc... These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

TestAmerica St. Louis uses a series of spreadsheets and/or databases to track changes to major capabilities (e.g. equipment, accreditations, etc.). An equipment list is maintained by the QA department. Accreditations are maintained via the OASIS Total Access program on the TestAmerica intranet site.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department electronically, which are backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Data Reporting Group (raw data, analytical records, lab reports) and the QA Department (logbooks, standards, certificates, Quality documents).

Table 14-1. Record Index¹

| | <u>Record Types</u> ¹ : | <u>Retention Time</u> : |
|---------------------------|---|--|
| Technical Records | <ul style="list-style-type: none"> - Raw Data - Logbooks² - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports | 5 Years from analytical report issue* |
| Official Documents | <ul style="list-style-type: none"> - Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals | 5 Years from document retirement date* |
| QA Records | <ul style="list-style-type: none"> - Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation | 5 Years from archival* <u>Data Investigation</u> : 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |
| Project Records | <ul style="list-style-type: none"> - Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports | 5 Years from analytical report issue* |

| | Record Types ¹: | Retention Time: |
|-------------------------------|--|------------------------|
| Administrative Records | Finance and Accounting | 10 years |
| | EH&S Manual, Permits | 7 years |
| | Disposal Records | Indefinitely |
| | Employee Handbook | Indefinitely |
| | Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual |
| | Administrative Policies Technical Training Records | 7 years |

¹ Record Types encompass hardcopy and electronic records.

² Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.2

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. For projects/programs that require a retention time longer than five years, the Project Manager informs the Reporting Group of the extended storage requirement. The Data Reporting Group tracks these requirements.

Table 14-2. Example: Special Record Retention Requirements

| Program | ¹Retention Requirement |
|---|---|
| Drinking Water – All States | 5 years (project records) 10 years - Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 10 years |
| NY Potable Water NYCRR Part 55-2 | 10 years |
| Ohio VAP | 10 years and State contacted prior to disposal |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |

¹Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the laboratory report. The chain of custody would indicate the name of the sampler. A log of names, initials and signatures for all individuals responsible for signing or initialing laboratory records is maintained in the Human Resources Department. If any sampling notes are provided with a work order, they are kept with the laboratory report.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in the Reagent Log in the LIMS and relevant printouts can be included in the data packages as needed.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times,

incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.

- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs or posted on the instrument.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.
- Chain of Custody protocols required by DOE and DoD

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are numbered sequentially. Within each logbook, pages are sequentially numbered. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the Reagents Log Program in LIMS. Records are considered archived when moved off-site or are so labeled. Dual storage of these records is maintained by the IT Department during its daily and weekly back-ups of the laboratory network. These back-up tapes are stored off-site.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer

agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party Records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

| Description | Performed by | Frequency |
|------------------------|---|--|
| Quality Systems Audits | QA Department, QA approved designee, or Corporate QA | All areas of the laboratory annually |
| Method Audits | Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-003) | Methods Audits Frequency: 50% of methods annually 100% of methods annually (DoD Labs) |
| Special | QA Department or Designee | Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits. |
| Performance Testing | Analysts with QA oversight | Two successful per year for each NELAC field of testing or as dictated by applicable regulatory requirements |

15.1.1 Audit Planning/Reporting

An audit plan is developed to identify the scope of the audit, the time frame, the personnel involved, the activities to be included, reference documents (i.e. Methods, SOPs, Checklists, and Client Requirement Memos) and persons to be notified of results. The audit team is selected prior to the audit. The size of the team is dependent on the scope of the audit. The lead auditor organizes and directs the audit. The audit report is issued to the appropriate departments by the lead auditor in hardcopy or electronically. The audit report is signed or otherwise endorsed by the Lead Auditor. The report describes the scope of the audit, identified auditors and persons contacted, summarizes results and describes all non-conformances found.

15.1.2 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.3 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.4 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.5 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.6 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil and Radiochemistry.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been

affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Director, Technical Managers, QA Manager, EH&S Manager and Radiation Safety Officer) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that is related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics

- Internal and External audit outcomes & corrective actions
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
 - Adequacy of staff, equipment and facility resources.
 - Adequacy of policies and procedures.
 - Future plans for resources and testing capability and capacity.
 - Changes in the volume and type of work
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- Laboratory health and safety issues
- Radioactive materials management issues

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual. Quality system changes and improvements are incorporated into the laboratory's yearly goals.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, VP of Quality, Technical and Operations Support, General Managers and Quality Directors receive a monthly report from the Corporate Quality Director summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

Management is responsible for authorizing specific personnel to perform specific tests (i.e. environmental testing, issue reports, interpret data, operate equipment).

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

The laboratory ensures that all personnel, including part time, temporary, contracted and administrative personnel, are trained in basic laboratory QA and safety programs.

Personnel dealing with sample receipt, radioactive waste management and materials shipping are trained in waste management, shipping and handling, and hazardous and/or radioactive materials control as appropriate.

17.2 Education and Experience Requirements for Technical Personnel

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|--|--|---|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC | A college degree in an applied science or 2 years of college and at least 1 year of college chemistry | Or 2 years prior analytical experience is required |
| ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS | A college degree in an applied science or 2 years of college chemistry | or 5 years of prior analytical experience |
| Spectra Interpretation | A college degree in an applied science or 2 years of college chemistry | And 2 years relevant experience Or 5 years of prior analytical experience |
| Technical Managers – <u>General</u> | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience | And 2 years experience in environmental analysis of representative analytes for which they will oversee |
| Technical Managers – <u>Wet Chem</u> only (no advanced instrumentation) | Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | And 2 years relevant experience |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewers or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels. See the laboratory SOP ST-QA-0044 Training for additional information.

Orientation to the laboratory’s policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|---|--|-------------------|
| Environmental Health & Safety | Prior to lab work | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics – Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |
| Ethics – Comprehensive Refresher | Annually | All |
| Computer Security Awareness | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to “Demonstration of Capability” in Section 19.

The following documentation must be on file at the laboratory for each employee:

- Ethics Training documentation
- Signed Ethics agreement
- Signed Confidentiality agreement
- TNI statement of qualification
- Copy of degree, if applicable
- New Employee Orientation checklist
- Safety Orientation checklist

In addition to items listed above, the following documentation is also included in the employee training record:

- Department training checklist
- Demonstration of Capability (IDOC/DOC)
- Manual Integration training, if applicable
- Annual evidence of continuing DOC (may be successful analysis of a blind sample on the specific test method, or a similar method or four successful LCS analyses.
- Specialty training as applicable

The training of technical staff is kept up to date by:

- Each employee must have documentation filed with the QA department that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintain documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice match SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and quarterly refreshers for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity. The Ethics Statement is re-signed annually.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 52,000 ft² secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, radiological sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- Separate high and low level radiochemical preparation areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in [Appendix 2](#).

18.5 Building Security

Building keys are distributed to management as necessary. The Human Resources Manager maintains a list of all employees who have been issued keys. Electronic “swipe” cards are issued to all laboratory employees.

All visitors to the laboratory enter through the main entrance and sign in and out in a visitor’s logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are given a visitor’s badge and are escorted by laboratory personnel at all times. Vendors may be issued badges which state that escorts are not required. Visitors and vendors must sign out before leaving the premises.

Entry via the warehouse dock area is permitted for client sample delivery or material supply delivery, without Visitor Log sign-in. The Sample Control Department is responsible for the proper escorting of these visitors.

Vendors issued electronic swipe cards are not required to sign in or out. Visitors from other TestAmerica facilities, while required to sign the Visitor’s log, may not require visitor badges.

At the laboratory’s discretion, visitors may be asked to show photo identification.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 and the laboratory's SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.
- A listing of TestAmerica St. Louis' SOPs is included in [appendix 7](#).

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate

analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.
- Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.
- HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.
- Method 1664, Revision A: N-Hexane Extractable Material (HEM: Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM): Non-polar Material by Extraction and Gravimetry, EPA-821-R-98-002, February 1999
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods).
- Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such,

the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly perform the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

For tasks where spiking is not possible (prep techniques including but not limited to compositing, drying and grinding, sub-sampling) the initial demonstration of capability is documented in the analysts training record by the analyst and supervisor signing off on the relevant SOP on the department training checklist. The yearly review and the analyst's acknowledgement of revisions to the SOP serve as the continuing demonstration of capability.

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).

- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted.

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, may confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial and continuing demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The validation process may include one, or a combination of the following: calibration using known reference standards, comparison of results achieved with other methods, PT samples, etc. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be

confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in a SOP, a SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots

is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. ST-QA-0016 "MDL/IDL, LOD/LOQ Determination", for details on the laboratory's MDL process.

19.8 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)

For radiochemical analyses, the MDA/MDC is determined based on normal factors and conditions which influence measurement. The MDA/MDC is used to evaluate the capability of a method relative to the required RLs. Sample size, count duration, tracer recovery, detector background and detector efficiency all contribute to determining the sample's MDA/MDC.

The Minimum Detectable Concentration (MDC) for a radionuclide by radiochemical measurement is determined from the blank/background variability associated with the appropriate detector, the detector efficiency, sample aliquot size and chemical yield. The background variability is proportional to the sample count time.

NOTE: The background variability is based on the analytical test and derived by: 1) using sample specific parameters, or 2) process blank specific parameters, or 3) by averaging the multiple MDCs derived in 1 or 2.

Matrix material is used whenever possible and is of a similar composition as the client samples.

The MDC is calculated for individual samples (depending on counting technique) using the formulas provided in [Appendix 6](#). The MDC is expected to be less than the client required detection limit. Cesium-137 is the MDC analyte of interest for gamma evaluation.

If the sample MDC is greater than the client required detection limit (CRDL) or reporting limit (RL), the Data Reviewer shall examine the sample volume/weight, counting time, tracer yield and/or other relevant factors. The Data Reviewer shall decide the corrective action which may include reanalysis, recounting or data acceptance and document per laboratory procedure.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like the MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 times the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.10 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP ST-QA-0016, "MDL/IDL, LOD/LOQ Determination", for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects where required. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analytes retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides

additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as human factors, adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor $k=2$.

19.13.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of $k = 3$. As an example, for a reported result of 1.0 mg/L with a LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ± 0.5 mg/L. This approach may be used for chemical analyses. For radiochemical uncertainty determination, see the calculations in [Appendix 6](#).

19.13.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. (Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items).

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples $\leq 5x$ the

reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in laboratory SOPs ST-IS-0001 "Software Change Management", ST-IS-0002, "Software Testing, Verification and Validation", and ST-IS-0003, "Information Systems". The laboratory is currently running QuantIMS which is a custom in-house developed laboratory information management system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.15.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, and secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls such as password protection or website access approval.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and second level reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and the laboratory SOP ST-QA-0040, "Manual Integration Procedure".

Analytical results are reduced to the appropriate concentration units as specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the reporting departments archive files. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (i.e. month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or picocuries per liter (pCi/L) or micrograms per liter ($\mu\text{g/L}$) for liquids and milligrams per kilogram (mg/kg), micrograms per kilogram ($\mu\text{g/kg}$) or picocuries per gram (pCi/g) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with

the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst reviews what has been entered to check for errors. If printed, the printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. Where possible, the data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file. For instruments without the capability of file storage the data is scanned to a pdf file and archived.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Logbooks have sequentially numbered pages.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the QA Manager or Technical Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Data review procedures are outlined in SOP ST-PM-0004, "Data Review, Verification and Reporting" to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (ST-QA-0040). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into LIMS. The Sample Control Supervisor, or designee, reviews the transcription of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.15.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add/review data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration

results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. One hundred percent of all manual integrations are reviewed. The review is documented on the chromatogram by the analyst responsible for the integration and on the Second Review Checklist by the peer reviewer. Manual integrations are also periodically electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is created for the client.

19.15.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.15.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. When complete, the report is sent out to the client.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet

quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. ST-QA-0040, entitled "Manual Integration Procedure".

- 19.15.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 19.15.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 19.15.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 19.15.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations done on samples, calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc. unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation
Analyst Demonstration of Capability

TestAmerica St. Louis

Analyst Name

M/DD/YYYY

Preparation Method(s):

Analytical Method(s):

Matrix: Solid/Water/Waste, etc...

Method Description:

Preparation SOP No: ST-XX-####

Analytical SOP No: ST-XX-####

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
 2. The test method(s) was performed by the analyst identified on this certificate.
 3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site. **These documents have been reviewed by the analyst as part of this DOC.**
 4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
 5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.
-

Analyst

Signature

Date

Dept Supervisor

Signature

Date

QA Manager

Signature

Date

SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures maybe/are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. Folder pockets are used in some logbooks to store service receipts.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses. The instrument is “tagged-out” by the analyst who observed the issue, the department manager or the QA department. A non-conformance memo, or some other “tag”, is posted on the affected instrument.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study or MDL verification sample) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

Refer to SOP ST-QA-0005, "Calibration and Verification Procedures for Thermometers, Balances, Weights and Pipettes," for detailed information.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometers are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks or filed in QA records. Monitoring of method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP ST-QA-0005.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. (Sample storage is monitored 7 days a week for units storing DOE and/or DoD samples).

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between $> 0^{\circ}\text{C}$ and $\leq 6^{\circ}\text{C}$; freezers are kept below 10°C .

Specific temperature settings/ranges for other refrigerators, ovens water baths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is non-critical. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards. This also does not apply to radiochemical methods.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification (Organic/Inorganic)

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Standard.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

- a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or
- b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in [Appendix 6](#)). Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.4.2 Radiochemical Calibrations

20.4.2.1 CALIBRATION STANDARDS

Shelf life for stock radioactive standards shall not exceed 5 half lives. Shelf life for stock solutions prepared in the laboratory from salts, metals or dilution from a parent solution shall be no greater than one year, unless stated otherwise on the calibration certificate from the manufacturer. Standards in the form of a soil, sealed sources, filter, plated sources and sealed epoxy Marinelli beakers do not always have an expiration date. After the 1 year shelf life of the stock solution has expired, it must be re-verified.

If the standard is not re-verified, the standard shall be removed or clearly designated as acceptable for qualitative purposes only.

The expiration date of the secondary standard shall not exceed the expiration date of the primary standard.

The accuracy of calibration standards is checked by comparison with a calibration verification standard from a second source. In cases where a second standard source is not available, a

source from a different vendor is acceptable. All cases where this requirement cannot be met shall be documented with a nonconformance memo.

When a traceable standard is not available to use for calibration or verification activities, a non-traceable standard may be used if written client approval is obtained (when required).

Calibration standards are prepared using the appropriate procedures.

For each analyte of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods.

Standards for instrument calibration are obtained from a variety of sources. All radioactive standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained, containing concentration/activity, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

The frequency of calibration can be found in the laboratory's radiochemical methods and [Table 20-4](#).

20.4.3 RADIOCHEMICAL CONTINUING INSTRUMENT CALIBRATION, VERIFICATION and RADIOCHEMICAL BACKGROUND MEASUREMENT

Performance checks shall be performed using appropriate check sources and monitored to ensure that the instruments are running properly and that detector response has not significantly changed. Background measurements are made according to the schedule on Table 20-4 and monitored to ensure that the laboratory maintains its capability to meet required data quality objectives.

20.4.4 RADIOCHEMICAL INSTRUMENT CONTAMINATION MONITORING

The laboratory radiochemical instrumentation SOPs specify the requirements for monitoring radiochemical instrumentation. The SOP specifies the monitoring frequencies and criteria for initiating corrective action.

20.5 Tentatively Identified Compounds (TIC) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See SOPs ST-MS-0001 and ST-MS-0002 for guidelines on making tentative identifications and reporting TICs.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|-----------------------------|------------------|--------------|---------------|-----------------------------|-------------------------------|
| GC/MS – “G” GC System | Hewlett Packard | 5890 | 2807A11075 | 1987 | NEW |
| GC/MS – “G” Concentrator | Tekmar | LSC3000 | 98175006 | 1992 | NEW |
| GC/MS – “G” Autosampler | Varian | Archon | 13540 | 2001 | NEW |
| GC/MS – “F” | Hewlett Packard | 5973 | DE00020247 | 1998 | NEW |
| GC/MS – “F” GC System | Hewlett Packard | 6890 | US80221392 | 1998 | NEW |
| GC/MS – “F” Concentrator | IO | Eclipse 4660 | D530466888P | 2002 | NEW |
| GC/MS – “F” Autosampler | Varian | Archon | 14613 | 2001 | NEW |
| GC/MS – “L” | Hewlett Packard | 5973 | CN10339019 | 2004 | NEW |
| GC/MS – “L” Concentrator | Teledyne Tekmar | Velocity XPT | US03346007 | 2004 | NEW |
| GC/MS – “L” Autosampler | Teledyne Tekmar | SOLATEk 72 | US03349002 | 2004 | NEW |
| GC/MS – “M” | Hewlett Packard | 5973 | CN10412013 | 2004 | NEW |
| GC/MS – “M” Concentrator | Teledyne Tekmar | Velocity XPT | US0412001 | 2004 | NEW |
| GC/MS – “M” Autosampler | Teledyne Tekmar | SOLATEk 72 | US04119003 | 2004 | NEW |
| GC/MS – “N” | Hewlett Packard | 5973 | CN10512032 | 2005 | NEW |
| GC/MS – “N” GC System | Hewlett Packard | 6890 | US44621325 | 2005 | NEW |
| GC/MS – “N” Concentrator | Tekmar/Dohrman | Velocity XPT | US03247002 | 2009 | Used |
| GC/MS – “N” Autosampler | Teledyne Teckmar | Solatek 72 | US03100004 | 2009 | Used |
| GC/MS – “K” | Hewlett Packard | 5973 | US81221525 | 1998 | NEW |
| GC/MS – “K” GC System | Hewlett Packard | 6890 | US00022347 | 1998 | NEW |
| GC/MS – “K” Series Injector | Hewlett Packard | 7683 | CN31530345 | 1998 | NEW |
| GC/MS – “K” Autosampler | Hewlett Packard | G2614A | US83501656 | 1998 | NEW |
| GC/MS – “J” | Hewlett Packard | 5973 | US80321385 | 1998 | NEW |
| GC/MS – “J” GC System | Hewlett Packard | 6890 | US00021127 | 1998 | NEW |
| GC/MS – “J” Series Injector | Hewlett Packard | 7683 | US81801195 | 1998 | NEW |
| GC/MS – “J” Autosampler | Hewlett Packard | G2614A | US80600251 | 1998 | NEW |
| GC/MS – “I” | Hewlett Packard | 5973 | CN10514049 | 2005 | NEW |
| GC/MS – “I” GC System | Hewlett Packard | G2579A | US44621455 | 2005 | NEW |
| GC/MS – “I” Series Injector | Hewlett Packard | 7683 | CN51224243 | 2005 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|-----------------|-----------------------------------|---------------|-----------------------------|-------------------------------|
| GC/MS – “I” Autosampler | Hewlett Packard | G2614A | CN42229061 | 2005 | NEW |
| GC/MS – “X” | Agilent | 5973 | US10461280 | 2008 | NEW |
| GC/MS – “X” GC System | Agilent | 6890N | US10144027 | 2008 | NEW |
| GC/MS – “X” Series Injector | Tekmar | 7683 | US01330017 | 2008 | NEW |
| GC/MS – “X” Autosampler | IO | G2614A | 1411 | 2008 | NEW |
| GC/MS – “Y” | Hewlett Packard | 5970 | 3449A02079 | 2009 | Used |
| GC/MS – “Y” GC System | Hewlett Packard | 5890 | 3336A57239 | 2009 | Used |
| GC/MS – “Y” Concentrator | Tekmar | Tekmar 3000 | 93300001 | 2009 | NEW |
| GC/MS – “Y” Autosampler | Varian | Archon | 12541 | 2009 | Used |
| GC/MS – “Z” | Hewlett Packard | 5973 | US80230105 | 2010 | Refurbished |
| GC/MS – “Z” GC System | Hewlett Packard | 6890 | US00009101 | 2010 | Refurbished |
| GC/MS – “Z” Concentrator | IO | Eclipse 4660 | E002466503P | 2010 | NEW |
| GC/MS – “Z” Autosampler | Varian | Archon | MS1003W019 | 2010 | NEW |
| LC/MS/MS – “R” Mass Spectrometer | Waters | Quattro Premier XE | VAB461 | 2006 | NEW |
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity PDA Detector | L05UPD807N | 2006 | NEW |
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity Sample Manager | 60UPS056M | 2006 | NEW |
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity Binary Solvent Man. | C06UPB008M | 2006 | NEW |
| LC/MS/MS – “T” Mass Spectrometer | Micromass | Ultima | VB280 | 2008 | NEW |
| LC/MS/MS – “T” HPLC – “Q” ALS Therm | Hewlett Packard | G1330A | DE13201124 | 1999 | NEW |
| LC/MS/MS – “T” HPLC – “Q” Quat Pump | Hewlett Packard | G1311A | DE14916965 | 1999 | NEW |
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Xevo | VBA453 | 2010 | NEW |
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Acquity Sample Manager | H07UPB932M | 2010 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|-----------------|--------------------------------------|---------------|-----------------------------|-------------------------------|
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Acquity Binary Solvent Manager | H07UPa802M | 2010 | NEW |
| GC – “L” | Hewlett Packard | 5890 | 2413A04451 | 1987 | NEW |
| GC – “L” Autosampler | Varian | Archon | 160098 | 2000 | NEW |
| GC – “L” Concentrator | Tekmar | LSC3000 | 93300001 | 1997 | NEW |
| GC – “K” | Agilent | 6890 | US00039258 | 2000 | NEW |
| GC – “K” Autosampler | Agilent | 7683 | US04709936 | 2000 | NEW |
| GC – “E” | Hewlett Packard | 6890 | US00011425 | 2000 | NEW |
| GC – “E” Autosampler | Hewlett Packard | 6890 | US71701354 | 2000 | NEW |
| GC – “M” | Agilent | 6890 | US10328036 | 2003 | NEW |
| GC – “M” Autosampler | Agilent | 7683 | CN32624339 | 2003 | NEW |
| GC – “O” | Agilent | 6890 | CN10422045 | 2004 | NEW |
| GC – “O” Autosampler | Agilent | 7683 | CN51132513 | 2004 | NEW |
| GC – “P” | Agilent | 6890N | CN10510018 | 2005 | NEW |
| GC – “P” Autosampler | Agilent | 7683 | CN51532846 | 2005 | NEW |
| GC – “V” | Agilent | 6890 | US00008573 | 2009 | USED |
| GC – “V” (Auto Sampler) | Agilent | G1530A | US8090377 | 2009 | USED |
| HPLC – “N” | Hewlett Packard | G1329A | DE91603153 | 1999 | NEW |
| HPLC – “N” ALS Therm | Hewlett Packard | G1330A | DE82203165 | 1999 | NEW |
| HPLC – “N” COLCOM | Hewlett Packard | G1316A | DE91609858 | 1999 | NEW |
| HPLC – “N” DAD | Hewlett Packard | G1315A | DE91605478 | 1999 | NEW |
| HPLC – “N” Degasser | Hewlett Packard | G1322A | JP73016399 | 1999 | NEW |
| HPLC – “N” Quat Pump | Hewlett Packard | G1311A | DE91605960 | 1999 | NEW |
| HPLC – “N” FLD | Hewlett Packard | G1321A | DE92001122 | 1999 | NEW |
| HPLC LCE (DAD) | Agilent | G1315D | DE64255811 | 2010 | USED |
| HPLC LCE (COL) | Agilent | G1316A | DE63065337 | 2010 | USED |
| HPLC LCE (Auto Sampler) | Agilent | G1329A | DE64764168 | 2010 | USED |
| HPLC LCE (Pump) | Agilent | G1311A | DE62962744 | 2010 | USED |
| GPC-1 | O-I Analytical | Autoprep 2000 | E427330254 | 2011 | NEW |
| ICP-MS – “6100” | Perkin Elmer | ELAN 6100 | 0859907 | 1999 | NEW |
| ICP-MS – “6100” Autosampler | Perkin Elmer | AS-91 | 4123 | 1999 | NEW |
| ICP-MS – “7500” | Agilent | 7500CX | JP82802890 | 2009 | NEW |
| ICP-MS – “7700” | Agilent | 7700 | JP10110271 | 2011 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|--|------------------------|----------------|----------------------|-----------------------------|-------------------------------|
| ICP-MS – “9000” | Perkin Elmer | ELAN 9000 | P1000302 | 2013 | USED |
| ICP – “6500 Duel View” | Thermo Fisher | 6000 Series | 20105013 | 2011 | NEW |
| CVAA | Leeman Labs | Hydra AA 2 | 0035 | 2011 | NEW |
| IC – “S” Chromatography Oven | Dionex | LC30 | 98070139 | 2008 | NEW |
| IC – “S” Conductivity Detector | Dionex | CD20 | 99070231 | 2008 | NEW |
| IC – “S” Gradient Pump | Dionex | GP50 | 99070382 | 2008 | NEW |
| IC – “S” Autosampler | Dionex | AS40 | 00090205 | 2008 | NEW |
| IC – “2500” Chromatography Oven | Dionex | LC25 | 03120540 | 2004 | NEW |
| IC – “2500” Conductivity Detector | Dionex | CD25 | 03120540 | 2004 | NEW |
| IC – “2500” Gradient Pump | Dionex | GP50 | 03120633 | 2004 | NEW |
| IC – “2500” Autosampler | Dionex | AS40 | 07020461 | 2004 | NEW |
| IC – “1500” Ion Chromatography System | Dionex | ICS-1500 | 03080236 | 2008 | NEW |
| IC – “1500” Autosampler | Dionex | ASM-3 | 920937 | 2008 | NEW |
| TOC | Shimadzu | TOC-5050A | 36501107 | 1999 | NEW |
| TOX | Mitsubishi | 100 TOX | A7M00017 | 1999 | NEW |
| TOC | Shimadzu | TOC-VCPN | H51404635090 | 2010 | NEW |
| Solid Sample Module | Shimadzu | SSM-5000A | H52504700582NK | 2010 | NEW |
| Discrete Analyzer | Systea | Easy Chem-Plus | 0901262 | 2010 | NEW |
| UV Spec 1 | Thermospectroni c | Genysis | 3SGF211001 | 2003 | NEW |
| UV Spec 2 | Thermospectroni c | Genysis | 3SGR172002 | 2013 | NEW |
| UV Spec | Shimadzu | UV-2401PC | A1083 (320053LP) | 2013 | USED |
| TRAACS – “1” | Technicon | Traacs 800 | 0103011 | 1988 | NEW |
| BOD | Man-Tech Associates | 04-227 | 270D3XB245 | 2003 | NEW |
| Ignitability Apparatus: Open Cup | Fisher | D-92 | 906N0014 | 1998 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|------------------|-----------------|--|-----------------------------|-------------------------------|
| Ignitability Apparatus: Closed Cup | Fisher | 162 | 1149 | 1992 | NEW |
| Multimeter | Thermo | 5 Star | B15814 | 2009 | NEW |
| Multimeter | Thermo | 5 Star | 015748 | 2009 | NEW |
| Alpha Spectrometer – “AV1 - AV24” “AV43 - AV122” “AV123 - AV226” “AV227 – AV247” | Ortec | Multi-Component | Multiple* | 1987-2011 | NEW |
| Gamma Spectrometer Intrinsic Germanium Detector “GE1 - GE10” “GE11 – GE19” | Tennelec / Ortec | Multi-Component | Multiple* | 1991-2011 | NEW |
| GFPC – “Protean” | Protean | MPC-9604 | 233126-BO 236534-BO 236532-BO 236533-BO | 2003 | NEW |
| GFPC – “Orange” | Protean | MPC-9604 | 08217155 08217156 08217154 08217153 10181186 10181187 | 2008-2010 | NEW |
| GFPC – “Purple” | Protean | MPC-9604 | 10181185 10181184 10029177 10029178 10029179 10029180 | 2010 | NEW |
| GFPC “Green” | Tennelec | LB5100 | 31360 | 2000 | NEW |
| LSC – “3180” Pink Teal Aquau Brown | Packard | Tricarb 3180 | DG06095123 DG01117382 DG01117385 DG01117384 DG01117383 | 2009-2011 | NEW |
| LSC – “3170” | Packard | Tricarb 3170 | 429670/429774 | 2002 | NEW |

Table 20-2. Example: Schedule of Routine Maintenance

Inductively Coupled Plasma

DAILY OR AS NEEDED - CHECK

- Gas supply
- Waste and rinse solution levels
- Droplet size (nebulizer)
- Replace orange/green tubing

WEEKLY

- Check water level in cool flow
- Nebulizer rinse
- Replace waste line
- Clean injector tip
- Check /Clean plasma torch assembly
- Replace sample tubing
- Clean spray chamber

MONTHLY

- Check /Clean air filter of power unit
- Clean fast autosampler valve and rotor

ANNUALLY

- Check vacuum system oil
- Check /Replace coolant water filter

Inductively Coupled Plasma/Mass Spectrometer

DAILY OR AS NEEDED

- Check Waste and rinse water container levels
- Check/ Replace sample, internal and waste lines
- Clean cones (7500, 7700)
- Clean cone

WEEKLY

- Check /Clean interface cones
- Check Roughing pump oil level and color
- Replace Waste Tubing

MONTHLY

- Check /Change pump oil (6100)
- Check /Clean auto lens (6100)
- Clean torch & injector tip (6100)
- Clean auto lense (6100)
- Clean torch (7500, 7700)
- Move data set files (7500, 7700)

Cold Vapor Automatic Analysis

DAILY OR AS NEEDED

- Check /Pump and drain tubing
- Check Gas pressure
- Instrument parameter check

WEEKLY

- Check /Change sample, reductant and draining tubings

MONTHLY

- Change/rinse tubing
- Check/change waste tubing

QUARTERLY

- Check /Change drying tube

TOX

DAILY OR AS NEEDED

- Cell Performance Test
- Electrodes
- Cell Fluid, Dehydrating Fluid and Electrolyte
- Adsorption module (cleaned at end of use)

Autoanalyzer Traacs- 1

DAILY

- Washout procedure (at end)

AS NEEDED

- Check /Change tubing
- Lubricate Probe shaft
- Lubricate oil rollers

TOC

DAILY OR AS NEEDED

- Air Supply and Gas Flow Rate (150mm)
- Humidifier
- A/LS Rinse Tank

MONTHLY

- Check /Inspect SO₃ scrubber – change if crystals at inlet are not white.
- Check /Inspect halogen scrubber – change if black color approaches outlet end.

ANNUALLY

- Check /Change CO₂ absorber

Ion Chromatography

DAILY OR AS NEEDED

- Plumbing for leaks
- Gases and Pump Pressure
- Conductivity meter
- Fill eluent
- Column replacement

UV Spec

DAILY OR AS NEEDED

- Rinse out Sample Cuvettes (after each use)

BOD

DAILY

- Calibration

As Needed

- Change membrane

Discrete Analyzer

DAILY

- Auto zero
- Perform rinse at completion of analysis
- Check DI water bottle/refill

Alpha Spectrometer

DAILY

- Pulsars

MONTHLY

- Backgrounds
- Clean detectors
- Continuing calibration verifications

ANNUALLY

- Calibrations

Gamma Spectrometer

DAILY

- Continuing calibration blank/continuing calibration verification

MONTHLY

- Clean/Long Backgrounds

ANNUALLY

- calibration checks

Gas Flow Proportional Counting

DAILY OR AS NEEDED

- Gas level
- Calibration verifications

MONTHLY

- Clean/Long Backgrounds

ANNUALLY

- Calibrations

Liquid Scintillation Counter

WEEKLY OR AS NEEDED

- Clean Fan

YEARLY

- Serviced by vendor

Semi-volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED

- Gas supply, column flow and inlet pressure
- Fill solvent rinse vials
- Check /Injection Port Cleaning
- Check /Change Septum, injection port liner, and seals
- Check /Trim Column
- Check/replace injection syringe

ANNUALLY

- Check /Replace pump oil

AS NEEDED

- Replace column
- Clean ion source
- Replace multiplier
- Replace electronic circuit board
- Replace detector
- Replace transfer lines

Volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED

- Gas supply, column flow and inlet pressure

QUARTERLY

- Check Trim Column
- Check/Change Trap

SEMI-ANNUALLY

- Check/Replace Column
- Check/Clean Source
- Check/Injection port maintenance

ANNUALLY

- Check/ Replace pump oil

High Pressure Liquid Chromatograph (HPLC)

DAILY OR AS NEEDED

- Ensure column flow and pressure are correct
- Ensure HPLC solvents are sufficient to run
- Ensure proper DAD signals are on
- Visibly check for leaks

MONTHLY

- Check/Change Purge Valve Frit

SEMIANNUALLY

- Check/Change Guard Cartridge and Frit Cap

BIANNUALLY

- Check/Replace Column
- Check/Replace UV Source
- Check/Replace Visible Source
- Check/Replace pump seals

Semi-Volatile Gas Chromatograph (Dual ECD)

DAILY OR AS NEEDED

- Ensure column flow and inlet pressure are correct
- Ensure temperature for oven, inlet(s), and detector(s) are correct
- Ensure solvent rinse vials are full
- Ensure injection syringe is secure in tower and plunger is engaged

MONTHLY

- Check/Replace injection port septum
- Visibly inspect injection port liner; replace if contaminated
- Check /Remove injection syringe and ensure plunger is free moving
- Check system for leaks (injection port, detector(s) and any column connectors)

SEMIANNUALLY

- Perform Radioactive leak test

Semi-Volatile Gas Chromatograph (FID)

DAILY OR AS NEEDED

- Check gas supply, column flow, and inlet pressure
- Fill solvent rinse vials

MONTHLY

- Check/Replace septum, injection port liner and seals
- Check/ Trim Guard Column

SEMIANNUALLY

- Check/ Replace Column

Volatile Gas Chromatograph

DAILY OR AS NEEDED

- Check gas supply, column flow and inlet pressure
- Change trap
- Trim column

SEMIANNUALLY

- Check/Replace Column
- Check/Injection port maintenance

ANNUALLY

- Check /Clean PID/FID

Liquid Chromatograph Mass Spectrometer Mass Spectrometer (LCMSMS)

DAILY OR AS NEEDED

- Check level of solution in reservoirs
- Check gas supply, column flow and system pressure
- Sonicate inlet check valves
- Clean ionization probes/corona pin
- Ballast Rough Pump

SEMIANNUALLY

- Check/Replace Column
- Check/Clean source
- Check/Injector maintenance

ANNUALLY

- Check/Replace pump oil

Table 20-3 Example: Periodic Calibration

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|----------------------------------|--|--|--|---|
| Analytical Balance | Accuracy determined using working weights that are annually checked against weights traceable to the International System of Units (SI) through a NMI. Minimum of 2 standards bracketing the weight of interest. Inspected and checked by ISO17025 accredited vendor annually. | Each day of use | ± 0.1% (QSM requires ± 0.1% or ±0.5 mg, whichever is greater) | Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service. |
| Top Loading Balance | Accuracy determined using ISO17025-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and checked by ISO17025 accredited vendor annually | Each day of use | ± 2.0% (QSM requires ± 2% or ±0.02 g, whichever is greater) | Clean. Replace. |
| ISO17025-accredited NIST Weights | Verification of standard mass using weights traceable to the International System of Units (SI) through a NMI | 5 years | Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory. | Replace. |
| NIST-Traceable Thermometer | Accuracy determined by ISO17025-accredited measurement laboratory. | 5 years | As per certificate. | Replace. |
| Thermometer | Against NIST-traceable thermometer | Yearly at appropriate temperature range for intended use | ± 1.0 °C | Replace |
| Digital thermometer | Against NIST-traceable thermometer | Quarterly | ± 1.0 °C | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|--|---|--|--|
| Refrigerator | Temperature checked using NIST-traceable thermometer. | Daily. If out of range, check again after several hours | 0 – 6 °C | Adjust. Repair. While waiting for repair, seal door, attach “Out of Service” sign, move items to functional unit. Notify supervisor. |
| Freezer | Temperature checked using NIST-traceable thermometer | Daily. If out of range, check again after several hours | <-10 °C | Adjust. Repair. While waiting for repair, seal door, attach “Out of Service” sign, move items to functional unit. Notify supervisor. |
| Oven | Temperature checked using NIST-traceable thermometer. | When in use. | 103 ± 2 °C (moisture determination) 180 ± 2°C (TDS) (DoD: ±5% of set temp) | Adjust. Replace. |
| Incubator | Temperature checked using NIST-traceable thermometer. | When in use. For microbiology, twice daily when in use. | BOD: 20 ± 1.0 °C | Adjust. Replace. |
| Water Bath | Temperature checked using NIST-traceable thermometer. | When in use. | ± 5 °C | Adjust. Replace. |
| Volumetric Dispensing Devices - pipettes | On delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number. Before first use: 10 replicate measurements with %RSD ≤ 1%. | Day of use 3 reps | ± 2% bias Precision RSD ≤ 1% | Adjust. Replace. |
| Non-volumetric labware (applicable only when measuring initial sample vol. or final extract/digest ate volume | Gravimetric – 10 reps before use | By lot before first use or upon evidence of deterioration | Bias: Mean within ± 3%of nominal volume Precision RSD ≤ 3% of stated value (based on 10 replicate measures) | replace |
| Volumetric glassware | The laboratory uses only Class A volumetric glassware. Calibration not required | N/A | Check for deterioration | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---------------------------------|--|---|----------------------------------|---|
| Glass Microliter Syringes | None | Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy. | $\pm 1\%$ | Not applicable. |
| Conductivity Meter | Cell impedance calibrated with three KCl standards. | Each use. | $r \geq 0.99$ | Recalibrate. |
| Deionized Water | Check in-line conductivity meter on system with conductivity meter in Inorganic Department. | Daily | $<10 \mu\text{mhos}/\text{cm}^2$ | Record on log. Report discrepancies to QA Department |

Table 20-4 Radiochemistry Calibration, Verification & Background Criteria

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|--------------------|-----------------------|--|--|
| Gamma Spectroscopy | Initial Calibration | Energy, FWHM and energy calibrations shall be established for the germanium spectroscopy systems annually , or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters. | The curve should have eight calibration points used to determine the energy relationship of the calibration. The calibration source must have radionuclides that “blanket” the intended range of calibration. The energy difference should be less than 0.05% for all points or with 2 keV for calibration points. Computed efficiency test for all points should have a percent difference less than 8%. The FWHM must be less than 3.0 keV at 1332 keV. FWHM difference should be less than 8% for all points. |
| Gamma Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the germanium spectroscopy systems monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements. | Background count time is 12 hours. |
| Gamma Spectroscopy | Continuing | Daily Checks The energy, resolution and efficiency calibrations for a detector shall be checked with its respective source each day that the germanium spectroscopy system is used. The detector background shall be checked each day that the germanium spectroscopy system is used. | Calibration (efficiency, resolution, energy alignment, and background) quality control parameters will be found not acceptable if the result is outside the established limits (2σ to 3σ range) and marked as “action”. The Daily QC check may only be recounted once without corrective action. |
| Alpha Spectroscopy | Initial Calibration | Energy calibrations shall be established for the alpha spectroscopy systems yearly , or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters. Efficiency calibrations shall be established for the alpha spectroscopy systems yearly , or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 meV. Final peak energy positions of all observed isotopes shall be within ± 40 keV of expected energy. Efficiency should fall between 20 and 32%. |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|-------------------------------|-----------------------|--|---|
| Alpha Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the alpha spectroscopy systems monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters. | Background count time is 960 minutes. |
| Alpha Spectroscopy | Continuing | Daily Checks Routine pulser quality control verifications are to be performed each day of use. The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used. | Routine calibration, background and pulser quality control parameters using the "Boundary" out-of-range test will be found unacceptable if the value is outside reasonable parameter tolerance. The routine quality control check should be rerun to determine the statistical significance of the errant parameter. |
| Gas Flow Proportional Counter | Initial Calibration | Mass attenuation alpha/beta curves should be performed on an annual basis, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | The efficiency calibration shall consist of at least seven single or dual sets of mass attenuated calibration standards. The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second. The coefficient of determination (r^2) shall be greater than or equal to 0.9. |
| Gas Flow Proportional Counter | Initial Background | Background established for the GFPC monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters. | Backgrounds are counted for 1,000 minutes Alpha < 0.2 counts per minute Beta < 2.0 counts per minute |
| Gas Flow Proportional Counter | Continuing | Daily Checks Efficiency check and background check | |

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices that are used to deliver volume critical measurements. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation) or another accreditation organization that is a signatory to a MRA (Mutual recognition Arrangement) of one or more of the following cooperation's – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation).. A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to TestAmerica St. Louis contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All liquid thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, and NIST with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Reagents Log Identification Number generated by LIMS and an expiration date. All documentation received with the reference standard is retained as a QC record and references the Standards Log Standard Identification Number. Reference standards that are used in the radiochemical laboratory shall be obtained from NIST, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. When traceable standards are not available, written approval for use must be obtained from DOE clients.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. Radiochemical standards must be verified prior to initial use. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual and the analytical method SOPs "Standards and Reagents" section for additional details. Radiochemical standards and reference material are stored separately from samples and are protected in a controlled cabinet or refrigerator. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.] Purchased stock mixtures and reagents are labeled to indicate the date they are opened.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in a directory on the laboratory network drive. Records must be kept of the date of receipt and date

of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and ST-QA-0002, "Standard and Reagent Preparation".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database:

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds; these records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (assigned by the LIMS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the MSDS documents available on the TestAmerica intranet site).

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

1. with the manufacturer's recommendations;
2. with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) is measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time. The

laboratory SOP ST-PM-0002 contains a table listing preservation, container and holding time information.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & sub-sampling are located in SOP ST-QA-0038, "Procedure for Compositing and Sub-sampling".

NOTE: *Unless otherwise noted by individual preparation SOPs, the following statements apply to sample aliquots of volume (liquid) for testing analysis.*

- Density Requirement – If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).
- Volume Determination – Aliquot volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous, or suspected of having a density greater than 1.2, will have aliquots taken for density analysis to correct volume for density

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her

view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored with the other login paperwork.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal, retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are described in SOP ST-PM-0002, "Sample Receipt and Chain of Custody".

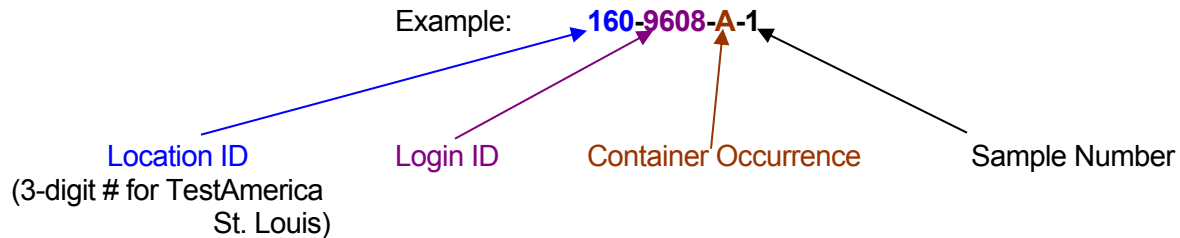
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. Coolers received from a known or potential radiologically contaminated site are frisked prior to opening. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a "Condition Upon Receipt" form (CUR) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following four pieces of information:



The above example indicates TestAmerica St. Louis (location 160), Login ID 9608 (unique to a particular job/client), container "A" of sample number 1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. For example, when a 1-liter amber bottle is sent through a Liquid/Liquid Extraction and extraction vial is created from the prep step. The vial would be a secondary container and would be labeled as follows:

160-9608-A-1-A

Secondary Container Occurrence - the Secondary ID has five components

The IDs are 'bar-coded' on the LIMS generated laboratory sample label attached to each container.

These steps allow the samples to be tracked through the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined and noted in the Case Narrative.

- 23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.
- 23.3.2** For samples received from a potentially radioactive site, an aliquot is removed from the container to perform a "rad screen."
- 23.3.3** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:
- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
 - Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according to SOP ST-PM-0002.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples having high levels of radiochemical contamination are labeled as such. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and are analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to a dry room temperature sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only.

Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. The sample itself is clearly "HAZARDOUS" or "FOREIGN SOIL". Any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, the sample is labeled as such. Potentially radioactive samples are "screened" prior to release to the laboratory. The RAD category is entered into the LIMS and alerts the analyst to the radiation level associated with the sample. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility (see SOPs *ST-HS-0006, "Quarantine Soils Procedure"*, and the *Radiation Protection SOPs* for more details).

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: ST-HS-0004, "Hazardous Waste Management Plan"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally

maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-2. Example: Sample Acceptance Policy

TestAmerica St. Louis Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. STL St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

DoD QSM SAMPLE ACCEPTANCE POLICY:

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.

The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation. Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented on a Condition Upon Receipt Form (CUR) for the project records and the client must be contacted for instructions. If the client decides to proceed with analysis, the project report shall clearly indicate any of the above conditions and the resolution.

If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction from the client to the contrary, the sample will be rejected.

For DoD QSM project work, sample containers must be certified to meet the "less than" ½ the RL criteria for the analytes of concern. Analytes for which this certification can not be obtained will be noted in the Case Narrative. Upon DoD project approval, the laboratory will analyze method blanks prepared in the containers of concern, qualify and narrate the sample analytes which do not meet the criteria, or take other appropriate action as determined by the DoD project site.

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS), tracers and carriers). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the **exact** same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance. PT samples must be evaluated the same as regular environmental samples. The laboratory shall employ the same quality control, sequence of analytical steps, and replicates as used when analyzing routine samples.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

| Control Type | Details |
|--------------------|---|
| Method Blank (MB) | <p>are used to assess preparation and analysis for possible contamination during the preparation and processing steps.</p> <p>The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.</p> <p>The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.</p> <p>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).</p> <p>Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than $\frac{1}{10}$ of the amount measured in the sample.</p> |
| Calibration Blanks | are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. |
| Instrument Blanks | are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content. |

Table 24-1. Example – Negative Controls

| Control Type | Details |
|-------------------------------|--|
| Trip Blank ¹ | are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. |
| Field Blanks ¹ | are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks ¹ | are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC) |
| Holding Blanks | also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory |

¹ When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-2. Sample Matrix Control

| Control Type | Details |
|--------------|---------|
|--------------|---------|

Table 24-2. Sample Matrix Control

| Control Type | Details | |
|-------------------------|--------------------------------|---|
| Matrix Spikes (MS) | Use | Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used; |
| | Typical Frequency ¹ | At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details |
| | Description | Essentially a sample fortified with a known amount of the test analyte(s). |
| Surrogate | Use | Measures method performance to sample matrix (organics only). |
| | Typical Frequency ¹ | Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery. |
| | Description | Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples. |
| Duplicates ² | Use | For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. |
| | Typical Frequency ¹ | Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. |
| | Description | Performed by analyzing two aliquots of the same field sample independently or an additional LCS. |
| Internal Standards | Use | Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements. |
| | Typical Frequency ¹ | All organic and ICP methods as required by the analytical method. |
| | Description | Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance. |
| Tracers and Carriers | Use | Chemically mimic and do not interfere with the target analytes through radiochemical separations. Isotopic tracers are typically radioactive materials while carriers are typically non-radioactive |
| | Typical Frequency ¹ | Added to each client sample, method blank, LCS and matrix QC sample, as required by the specific method. |
| | Description | Added to samples to determine the overall chemical yield of the analytical preparation steps. Each sample is spiked separately with the same material and individual sample yields are determined. The tracer/carrier is added to the sample at the very beginning of the preparation steps. For solid samples the tracer/carrier is added after grinding, but before muffling or dissolution. |

¹ See the specific analytical SOP for type and frequency of sample matrix control samples.

² LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits

with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on a semi-annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by $\leq 5\%$ from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. The QA department can generate a Quality Control Limit summary that contains tables that summarize the precision and accuracy acceptability limits for the analyses performed at TestAmerica St. Louis. The information is stored in the LIMS and includes an effective date and is updated each time new limits are generated. Unless otherwise noted, these limits are laboratory generated. The limits are approved in the LIMS system after review by the QA department. The LIMS maintains an archive of all limits used in the laboratory. Historical limits can be found in the LIMS program. See laboratory SOP ST-QA-0014, "Evaluation of Analytical Accuracy and Precision through the Use of Control Charts".

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for NELAC and Department of Defense (DoD) work, there are an allowable number of Marginal Exceedances (ME):

| | |
|------------------|-------------------------------------|
| <11 analytes | 0 marginal exceedances are allowed. |
| 11 – 30 Analytes | 1 marginal exceedance is allowed |
| 31-50 Analytes | 2 marginal exceedances are allowed |
| 51-70 Analytes | 3 marginal exceedances are allowed |
| 71-90 Analytes | 4 marginal exceedances are allowed |
| > 90 Analytes | 5 marginal exceedances are allowed |

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of

the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.6.5 If radiochemical tracer or carrier recovery is outside limits the sample is re-analyzed to confirm matrix interference. If recoveries confirm, or there was obvious interference, results are reported from the original run and a note is included with the case narrative. If the re-analysis meets the recovery criteria, the second run is reported (or both are reported if requested by the client). When samples are non-detect for the target analytes and the carrier/tracer recovery indicates a high bias in the analysis, the samples are not re-run unless required by the client.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method; including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation

requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report for Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number or SDG number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

- 25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.
- 25.2.9** Date reported or date of revision, if applicable.
- 25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).
- 25.2.11** Practical quantitation limits or reporting limit.
- 25.2.12** Method detection limits (if requested)
- 25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).
- 25.2.14** Sample results.
- 25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
- 25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 regarding additional addenda).
- 25.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- 25.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.
- 25.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.
- 25.2.20** When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.
- 25.2.21** A narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
- 25.2.22** When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.
- 25.2.23** Appropriate laboratory certification number for the state of origin of the sample, if applicable.
- 25.2.24** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary data). A complete report must be sent once all of the work has been completed.

25.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.26 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form and as an electronic (pdf) file. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica St. Louis offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without

errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory

outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the job number/SDG number followed by "rev".

When the report is re-issued, a notation of “Revised “is placed on the cover/signature page of the report *and at the top of the narrative page* with a brief explanation of reason for the re-issue.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

SECTION 26. REVISION HISTORY

26.1 CHANGES TO REVISION 0

- 26.1.1 Updated to conform to new corporate Template. Information that was specific to the company at large and less specific to the individual laboratory was removed from the template and is now found in the Corporate Quality Management Plan (CQMP).
- 26.1.2 The Quality Policy Statement was updated to include compliance with NELAC standards.
- 26.1.3 Section 10 (Services to Client) was merged with Section 7 (renamed)
- 26.1.4 Section 10 was left intentionally blank.
- 26.1.5 Section 16 (Audits) was given new text.
- 26.1.6 Section 17 (Management Reviews) revised QA report section, some tables were removed
- 26.1.7 Section 21 (Calibrations) removed information that can be found in method SOPs
- 26.1.8 Radiochemistry calculations in Appendix 6 were updated
- 26.1.9 Tables, figures and appendices were updated and re-numbered

26.2 CHANGES TO REVISION 1(06/02/09)

- 26.2.1 Added reference to ASME NQA-1-2000 to Section 3.1
- 26.2.2 Updated Ethics Agreement in Appendix 1
- 26.2.3 Updated radiochemistry calculations in Appendix 6.

26.3 CHANGES TO REVISION 2 (08/31/09)

- 26.3.1 Added reference to DoD QSM 4.1 to Section 3.1
- 26.3.2 Updated QA Manager job description in Section 4.2.3
- 26.3.3 Updated laboratory organizational chart
- 26.3.4 Added Quality Program objectives to Section 5.1; clarified staff responsibilities regarding QA documents
- 26.3.5 Added QAM review cycle to Table 16-1
- 26.3.6 Added freezer temperature criteria to Section 21.3.4
- 26.3.7 Updated Calibration information in Table 21-3
- 26.3.8 Added current Florida NELAC cert to Appendix 3
- 26.3.9 Signatures moved from Title Page to Cover per DoD Requirements

26.4 CHANGES TO REVISION 3 (08/31/10)

- 26.4.1 Section 2: list of Cross-walk references to the ISO 17025 requirements added
- 26.4.2 Section 4.2: QA Manager responsibilities updated
- 26.4.3 Section 4: Organizational Charts updated in figure 4-1
- 26.4.4 Section 5.1: Addition to quality Policy Statement regarding continuous improvement
- 26.4.5 Section 7: Figure 7-1 removed
- 26.4.6 Section 13: Table 13-3 "General Corrective Actions" added
- 26.4.7 Section 13.3.3: Root cause analysis added
- 26.4.8 Sections 3.1 & 20.4: Source methods references updated
- 26.4.9 Section 18.3: Evidence of successful training added
- 26.4.10 Section 20.15.5: text on manual integrations and Mint Miner[®] expanded
- 26.4.11 Section 21: Table 21-1 "instrument List", updated
- 26.4.12 Section 21.3.5: requirement for non-volumetric labware added
- 26.4.13 Section 21.4: calibration standards section expanded
- 26.4.14 Section 24.2.2: Unique sample ID section added

- 26.4.15 Section 24.3: Sample Acceptance Policy moved to appear in Table of Contents
- 26.4.16 Section 24.6: added note on Trip blanks
- 26.4.17 Section 26.2.18: added narrative requirement reproduction of laboratory reports
- 26.4.18 Information in Appendices 1,2,3,5 & 7 updated
- 26.4.19 Added "End of Document" statement
- 26.4.20 General grammatical edits and corrections

- 26.5 **CHANGES TO REVISION 4**
- 26.5.1 10/08/10: Added Section 20.4.2.4 to address DOCs for tests without analyte spikes
- 26.5.2 8/31/11: Removed the 'effective date' by section and applied it to the entire document. Continuous document pagination implemented.
- 26.5.3 2009 TNI Standard references added to the Table of Contents only – citations removed from the section titles within the document. Updated all references from the 2003 NELAC Standards to the 2009 TNI standard
- 26.5.4 Use of the title 'Technical Manager' from the TNI Standard is defined and implemented.
- 26.5.5 Section 10 (previously left empty) removed. Other section numbers adjusted accordingly.
- 26.5.6 Section 4: Additional Quality Assurance and Technical Manager (a.k.a., Supervisors) responsibilities assigned based on the TNI Standard
- 26.5.7 Section 8: Clarification of subcontracting procedures
- 26.5.8 Table 12-1: Updated for additional corrective action procedures
- 26.5.9 Section 15: Updates reflect current internal audit process as defined in CA-Q-S-004. Table 15-1 updated.
- 26.5.10 Section 19: Verification of MDLs/RLs updated to TNI Standard
- 26.5.11 Section 25: added statement regarding the listing of non-accredited methods in the lab report
- 26.5.12 Appendix 2: updated laboratory floor plan
- 26.5.13 Appendix 4: added/removed glossary terms/acronyms
- 26.5.14 Appendix 5: Certification table updated
- 26.5.15 Appendix 6: updated and clarified calculations
- 26.5.16 Appendix 7: updated SOP list

- 26.6 **CHANGES TO REVISION 5**
- 26.6.1 Grammatical and format corrections made throughout entire document
- 26.6.2 Updated signature page
- 26.6.3 REFERENCED CORPORATE SOPs AND POLICIES updated
- 26.6.4 Section 4.3: Deputies updated
- 26.6.5 Figure 4-1 Corporate and Laboratory Organization Charts updated
- 26.6.6 Section 5.5: Criteria for Quality Indicators updated
- 26.6.7 Changed TNI to NELAC where applicable
- 26.6.8 Section 9.3.3: Specifications: updated compressed gasses paragraph
- 26.6.9 Replaced Clouseau with LIMS where applicable
- 26.6.10 Section 11.2: Responsibilities and Authorities removed COO
- 26.6.11 Section 12: Removed Clouseau screen shots
- 26.6.12 Section 14: Replaced reference to standards log program with LIMS
- 26.6.13 Section 15: updated reference to Internal Auditing SOP to CA-Q-S-003
- 26.6.14 Section 15: Added Audit Planning/Reporting section
- 26.6.15 Sections 19.15.2 & 19.15.3: updated

- 26.6.16 Section 20.2: Added "tagged-out" requirements
- 26.6.17 Table 20-1, 20-2, 20-4 updated
- 26.6.18 Section 22.5: Addition of aqueous sample aliquot density requirement and volume determination
- 26.6.19 Section 23.2.1.1: Replaced QuantIMS with TALS unique sample identification.
- 26.6.20 Section 23.3: Updated to indicate that variation from policy to be noted in case narrative
- 26.6.21 Section 24.6.1: updated to reference LIMS instead of QC Browser
- 26.6.22 Appendix 3: updated NELAC certification
- 26.6.23 Appendix 4: added new glossary terms and acronyms
- 26.6.24 Appendix 5: updated St. Louis certifications
- 26.6.25 Appendix 6: added organic calculation "On column concentrations"
- 26.6.26 Appendix 7: updated laboratory SOP listing

26.7 CHANGES TO REVISION 6

- 26.7.1 Section 3.1, updated references
- 26.7.2 Section 4.1, changed Chief Operating Officer to Chief Executive Officer
- 26.7.3 Section 4.2, updated QA Manager, Technical Manager and Technical Director Responsibilities
- 26.7.4 Section 4.3, updated responsibilities table of key personnel
- 26.7.5 Figure 4-1, updated Corporate and Lab Org Chart
- 26.7.6 Table 14-1, removed 7 year requirement and replaced it with reference to HR Manual
- 26.7.7 Section 19.13.4, revised explanation of the meaning of the lab's uncertainty statement to more closely conform to A2LA and NIST language
- 26.7.8 Table 20-4, updated to reflect practice
- 26.7.9 Section 24.1, statement added to clarify and emphasize treatment of QC samples and PT samples
- 26.7.10 Appendix 3: updated NELAC certification
- 26.7.11 Appendix 5: updated St. Louis certifications
- 26.7.12 Appendix 6: updated calculations
- 26.7.13 Appendix 7: updated SOP listing

26.8 CHANGES TO REVISION 7 (02/02/2015)

- 26.8.1 Section 4.3, updated Key Personnel Deputy table
- 26.8.2 Figure 4-1, updated organizational charts
- 26.8.3 Section 17.3, added reference to see SOP ST-QA-0044 Training
- 26.8.4 Table 20-3, updated Example: Periodic Calibration
- 26.8.5 Appendix 5, update lab certifications, accreditations, validations

Appendix 1. *Example:* Ethics & Confidentiality Agreements



EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), are committed to ensuring the highest standard of ethical and professional conduct in all business activities. The Company and its employees will comply with all applicable laws, regulations and policies. We will ensure the highest standards of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform, the data I report in connection with my employment at the Company, and all business activities, I agree that:

- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers in any aspect of my job, including timekeeping, accounting, and compliance with all safety, environmental and employment regulations.*
- I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions; nor will I intentionally alter or omit dates, dollar values or other business related information in order to achieve desired financial results.*
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- I shall not accept gifts of a value that would adversely influence judgment.*
- I shall avoid conflicts of interest and report any potential conflicts to the management (e.g., employment or consulting with competitors, clients, or vendors);*
- I shall not participate in unfair competition practices (e.g., slandering competitors, collusion with other labs to restrict others from bidding on projects);*
- I shall not take any action, personally, or on behalf of the Company, which violates any applicable law, regulation, or internal policy, or which causes the Company to incur financial risk or loss or causes the Company to report incorrect financial information.*
- I will not intentionally report values that are inconsistent with the actual values observed or measured;*
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;*
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's;*
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the facility's Standard Operating Procedures, EPA Manual, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- I shall not compare or disclose results for any Proficiency Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica facility, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- I shall not misrepresent certifications and status of certifications to clients or regulators;*
- I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Facility Director and Quality Assurance Officer/Manager (where applicable). The Facility Director or Quality Assurance Officer/Manager (where applicable) will initial and date the information and return a copy to me; I shall*

Company Confidential & Proprietary

[THIS IS A CONTROLLED DOCUMENT. WHEN PRINTED IT BECOMES UNCONTROLLED]



not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.

- I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices or illegal or unethical business activities, or if I am in doubt or uncertain as to whether or not such laboratory practices or business activities are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Facility Director, all supervisors and managers with direct line reporting relationship between me and the Facility Director, and the local Quality Assurance representative (where applicable), excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that all of my dealings as an employee must be in compliance with applicable Federal and State laws, including safety regulations, environmental regulations, accounting rules, and employment laws, such as the Drug Free Workplace Act and anti-discrimination and harassment legislation.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

Employee Printed Name _____

EMPLOYEE SIGNATURE _____ Date _____



CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____ (printed name), understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge through no act of my own. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). In the event that any provision of this Agreement is held to be unenforceable because of the scope, duration or area of its applicability, the court making such determination shall have the power to modify any or all such terms, and those terms shall then be applicable in such modified form and the other provisions of this Agreement shall remain in force.
6. I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

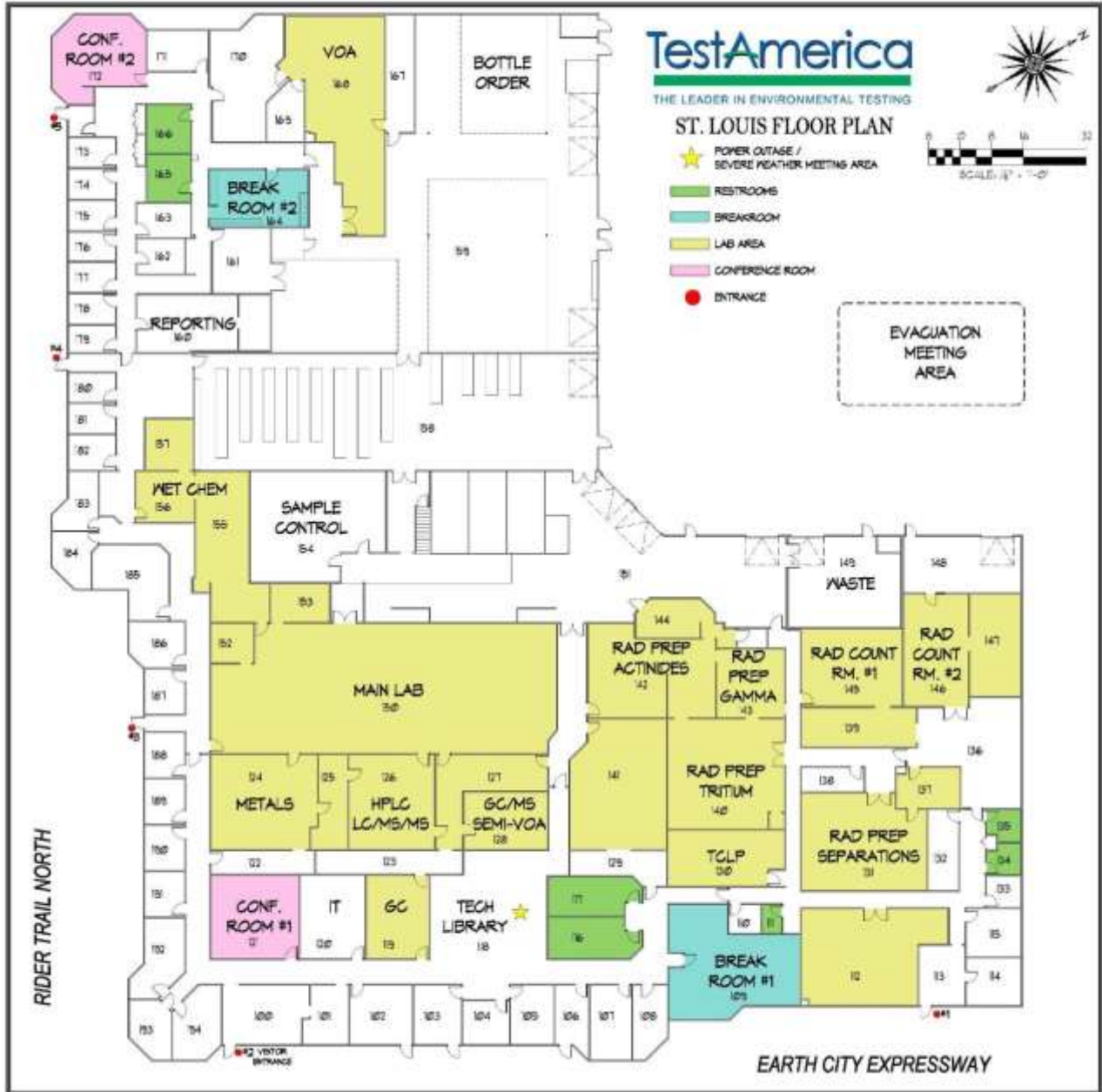
I have executed this Agreement, intending to be legally bound.

Printed Name

Signature

Date

Appendix 2. Laboratory Floor Plan



Appendix 3: Example: NELAC/TNI Certified Tests

| | | |
|---|--|---|
|  | <p>STATE OF LOUISIANA DEPARTMENT OF ENVIRONMENTAL QUALITY Is hereby granting a Louisiana Environmental Laboratory Accreditation to</p> |  |
| <p>TestAmerica Laboratories Inc 13715 Rider Trail N Earth City, Missouri 63045-1205</p> | | |
| <p>Agency Interest No. 106151</p> | | |
| <p>According to the Louisiana Administrative Code, Title 33, Part I, Subpart 3, LABORATORY ACCREDITATION, the State of Louisiana formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed in the attachment.</p> | | |
| <p>The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part I, Subpart 3 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part I. Please contact the Department of Environmental Quality, Louisiana Environmental Laboratory Accreditation Program (LELAP) to verify the laboratory's scope of accreditation and accreditation status.</p> | | |
| <p>Accreditation by the State of Louisiana is not an endorsement or a guarantee of validity of the data generated by the laboratory. To be accredited initially and maintain accreditation, the laboratory agrees to participate in two single-blind, single-concentration PT studies, where available, per year for each field of testing for which it seeks accreditation or maintains accreditation as required in LAC 33:14711.</p> | | |
|  _____ | Certificate Number: 04080 | |
| <p>Lourdes Iturralde, Administrator Notifications and Accreditations Section Public Participation & Permit Support Services Division</p> | Expiration Date: June 30, 2015 Issued On: July 1, 2014 | |



STATE OF LOUISIANA
DEPARTMENT OF ENVIRONMENTAL QUALITY
Issue Date: July 1, 2014

TestAmerica Laboratories Inc
AI Number: 106151
Expiration Date: June 30, 2015

13715 Rider Trail N, Earth City, Missouri 63045-1205

Certificate Number: 04080

Air Emissions

| Analyte | Method Name | Method Code | Type | AB |
|---------|-------------|-------------|------|------|
| NONE | NONE | NONE | NONE | NONE |

Non Potable Water

| Analyte | Method Name | Method Code | Type | AB |
|---|--------------------|-------------|-------|----|
| 2755 - Americium-241 | Eichrom ACW03 | 2259 | NELAP | LA |
| 2940 - Plutonium | Eichrom ACW03 | 2259 | NELAP | LA |
| 3035 - Uranium | Eichrom ACW03 | 2259 | NELAP | LA |
| 100499 - Neptunium | Eichrom ACW08 | 2260 | NELAP | LA |
| 1170 - Thorium | Eichrom ACW08 | 2260 | NELAP | LA |
| 2900 - Lead-210 | Eichrom OTW01 | 2264 | NELAP | LA |
| 1170 - Thorium | Eichrom ACW10 | 2269 | NELAP | LA |
| 4735 - 1,4-Dioxane (1,4- Diethyleneoxide) | EPA 8260 SIM | 2995 | NELAP | LA |
| 1923 - Reactive Cyanide | EPA 7.3.3.2, Rev.3 | 10001204 | NELAP | LA |
| 1925 - Reactive sulfide | EPA 7.3.4.2, Rev.3 | 10001408 | NELAP | LA |
| 1610 - Conductivity | EPA 120.1 | 10006209 | NELAP | LA |
| 1900 - pH | EPA 150.1 | 10008205 | NELAP | LA |
| 1955 - Residue-filterable (TDS) | EPA 160.1 | 10009004 | NELAP | LA |
| 1960 - Residue-nonfilterable (TSS) | EPA 160.2 | 10009402 | NELAP | LA |
| 1950 - Residue-total | EPA 160.3 | 10009800 | NELAP | LA |
| 1000 - Aluminum | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1005 - Antimony | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1010 - Arsenic | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1015 - Barium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1020 - Beryllium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1025 - Boron | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1030 - Cadmium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1035 - Calcium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1040 - Chromium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1050 - Cobalt | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1055 - Copper | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1070 - Iron | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1075 - Lead | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1085 - Magnesium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1090 - Manganese | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1100 - Molybdenum | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1105 - Nickel | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1125 - Potassium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1140 - Selenium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1990 - Silica as SiO2 | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1150 - Silver | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1155 - Sodium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1160 - Strontium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1165 - Thallium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1175 - Tin | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1180 - Titanium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1185 - Vanadium | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |
| 1190 - Zinc | EPA 200.7, Rev.4.4 | 10013806 | NELAP | LA |

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.

Non Potable Water

| Analyte | Method Name | Method Code | Type | AB |
|----------------------------------|----------------|-------------|-------|----|
| 1000 - Aluminum | EPA 200.8 | 10014401 | NELAP | LA |
| 1005 - Antimony | EPA 200.8 | 10014401 | NELAP | LA |
| 1010 - Arsenic | EPA 200.8 | 10014401 | NELAP | LA |
| 1015 - Barium | EPA 200.8 | 10014401 | NELAP | LA |
| 1020 - Beryllium | EPA 200.8 | 10014401 | NELAP | LA |
| 1030 - Cadmium | EPA 200.8 | 10014401 | NELAP | LA |
| 1040 - Chromium | EPA 200.8 | 10014401 | NELAP | LA |
| 1050 - Cobalt | EPA 200.8 | 10014401 | NELAP | LA |
| 1055 - Copper | EPA 200.8 | 10014401 | NELAP | LA |
| 1075 - Lead | EPA 200.8 | 10014401 | NELAP | LA |
| 1085 - Magnesium | EPA 200.8 | 10014401 | NELAP | LA |
| 1090 - Manganese | EPA 200.8 | 10014401 | NELAP | LA |
| 1100 - Molybdenum | EPA 200.8 | 10014401 | NELAP | LA |
| 1105 - Nickel | EPA 200.8 | 10014401 | NELAP | LA |
| 1140 - Selenium | EPA 200.8 | 10014401 | NELAP | LA |
| 1150 - Silver | EPA 200.8 | 10014401 | NELAP | LA |
| 1165 - Thallium | EPA 200.8 | 10014401 | NELAP | LA |
| 1170 - Thorium | EPA 200.8 | 10014401 | NELAP | LA |
| 3035 - Uranium | EPA 200.8 | 10014401 | NELAP | LA |
| 1185 - Vanadium | EPA 200.8 | 10014401 | NELAP | LA |
| 1190 - Zinc | EPA 200.8 | 10014401 | NELAP | LA |
| 1095 - Mercury | EPA 245.1 | 10036201 | NELAP | LA |
| 1535 - Bromate | EPA 300.0 | 10053006 | NELAP | LA |
| 1540 - Bromide | EPA 300.0 | 10053006 | NELAP | LA |
| 1575 - Chloride | EPA 300.0 | 10053006 | NELAP | LA |
| 1730 - Fluoride | EPA 300.0 | 10053006 | NELAP | LA |
| 1810 - Nitrate as N | EPA 300.0 | 10053006 | NELAP | LA |
| 1840 - Nitrite as N | EPA 300.0 | 10053006 | NELAP | LA |
| 1870 - Orthophosphate as P | EPA 300.0 | 10053006 | NELAP | LA |
| 2000 - Sulfate | EPA 300.0 | 10053006 | NELAP | LA |
| 1505 - Alkalinity as CaCO3 | EPA 310.1 | 10054601 | NELAP | LA |
| 1895 - Perchlorate | EPA 314, Rev.1 | 10055604 | NELAP | LA |
| 1940 - Total residual chlorine | EPA 330.1 | 10057804 | NELAP | LA |
| 1635 - Cyanide | EPA 335.4 | 10061402 | NELAP | LA |
| 1730 - Fluoride | EPA 340.2 | 10052201 | NELAP | LA |
| 3751 - Ammonia | EPA 350.1 | 10063408 | NELAP | LA |
| 1810 - Nitrate as N | EPA 353.1 | 10066805 | NELAP | LA |
| 1820 - Nitrate-Nitrite | EPA 353.1 | 10066805 | NELAP | LA |
| 1910 - Total Phosphorus | EPA 365.2 | 10070403 | NELAP | LA |
| 2005 - Sulfide | EPA 376.1 | 10074007 | NELAP | LA |
| 1530 - Biochemical oxygen demand | EPA 405.1 | 10075408 | NELAP | LA |
| 1565 - Chemical oxygen demand | EPA 410.4 | 10077006 | NELAP | LA |
| 2040 - Total Organic Carbon | EPA 415.1 | 10078203 | NELAP | LA |
| 7355 - 4,4'-DDD | EPA 608 | 10103603 | NELAP | LA |
| 7360 - 4,4'-DDE | EPA 608 | 10103603 | NELAP | LA |
| 7365 - 4,4'-DDT | EPA 608 | 10103603 | NELAP | LA |
| 7025 - Aldrin | EPA 608 | 10103603 | NELAP | LA |
| 8880 - Aroclor-1016 (PCB-1016) | EPA 608 | 10103603 | NELAP | LA |
| 8885 - Aroclor-1221 (PCB-1221) | EPA 608 | 10103603 | NELAP | LA |
| 8890 - Aroclor-1232 (PCB-1232) | EPA 608 | 10103603 | NELAP | LA |
| 8895 - Aroclor-1242 (PCB-1242) | EPA 608 | 10103603 | NELAP | LA |
| 8900 - Aroclor-1248 (PCB-1248) | EPA 608 | 10103603 | NELAP | LA |
| 8905 - Aroclor-1254 (PCB-1254) | EPA 608 | 10103603 | NELAP | LA |
| 8910 - Aroclor-1260 (PCB-1260) | EPA 608 | 10103603 | NELAP | LA |
| 7250 - Chlordane (tech.) | EPA 608 | 10103603 | NELAP | LA |
| 7470 - Dieldrin | EPA 608 | 10103603 | NELAP | LA |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.

| Non Potable Water | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 7510 - Endosulfan I | EPA 608 | 10103603 | NELAP | LA | |
| 7515 - Endosulfan II | EPA 608 | 10103603 | NELAP | LA | |
| 7520 - Endosulfan sulfate | EPA 608 | 10103603 | NELAP | LA | |
| 7540 - Endrin | EPA 608 | 10103603 | NELAP | LA | |
| 7530 - Endrin aldehyde | EPA 608 | 10103603 | NELAP | LA | |
| 7685 - Heptachlor | EPA 608 | 10103603 | NELAP | LA | |
| 7690 - Heptachlor epoxide | EPA 608 | 10103603 | NELAP | LA | |
| 8250 - Toxaphene (Chlorinated camphene) | EPA 608 | 10103603 | NELAP | LA | |
| 7110 - alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 608 | 10103603 | NELAP | LA | |
| 7115 - beta-BHC (beta-Hexachlorocyclohexane) | EPA 608 | 10103603 | NELAP | LA | |
| 7105 - delta-BHC | EPA 608 | 10103603 | NELAP | LA | |
| 7120 - gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 608 | 10103603 | NELAP | LA | |
| 5160 - 1,1,1-Trichloroethane | EPA 624 | 10107207 | NELAP | LA | |
| 5110 - 1,1,2,2-Tetrachloroethane | EPA 624 | 10107207 | NELAP | LA | |
| 5165 - 1,1,2-Trichloroethane | EPA 624 | 10107207 | NELAP | LA | |
| 4630 - 1,1-Dichloroethane | EPA 624 | 10107207 | NELAP | LA | |
| 4640 - 1,1-Dichloroethylene | EPA 624 | 10107207 | NELAP | LA | |
| 4610 - 1,2-Dichlorobenzene | EPA 624 | 10107207 | NELAP | LA | |
| 4635 - 1,2-Dichloroethane (Ethylene dichloride) | EPA 624 | 10107207 | NELAP | LA | |
| 4655 - 1,2-Dichloropropane | EPA 624 | 10107207 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 624 | 10107207 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 624 | 10107207 | NELAP | LA | |
| 4500 - 2-Chloroethyl vinyl ether | EPA 624 | 10107207 | NELAP | LA | |
| 4325 - Acrolein (Propenal) | EPA 624 | 10107207 | NELAP | LA | |
| 4340 - Acrylonitrile | EPA 624 | 10107207 | NELAP | LA | |
| 4375 - Benzene | EPA 624 | 10107207 | NELAP | LA | |
| 4395 - Bromodichloromethane | EPA 624 | 10107207 | NELAP | LA | |
| 4400 - Bromoform | EPA 624 | 10107207 | NELAP | LA | |
| 4455 - Carbon tetrachloride | EPA 624 | 10107207 | NELAP | LA | |
| 4475 - Chlorobenzene | EPA 624 | 10107207 | NELAP | LA | |
| 4575 - Chlorodibromomethane | EPA 624 | 10107207 | NELAP | LA | |
| 4485 - Chloroethane (Ethyl chloride) | EPA 624 | 10107207 | NELAP | LA | |
| 4505 - Chloroform | EPA 624 | 10107207 | NELAP | LA | |
| 4765 - Ethylbenzene | EPA 624 | 10107207 | NELAP | LA | |
| 4950 - Methyl bromide (Bromomethane) | EPA 624 | 10107207 | NELAP | LA | |
| 4960 - Methyl chloride (Chloromethane) | EPA 624 | 10107207 | NELAP | LA | |
| 4975 - Methylene chloride (Dichloromethane) | EPA 624 | 10107207 | NELAP | LA | |
| 5115 - Tetrachloroethylene (Perchloroethylene) | EPA 624 | 10107207 | NELAP | LA | |
| 5140 - Toluene | EPA 624 | 10107207 | NELAP | LA | |
| 5170 - Trichloroethene (Trichloroethylene) | EPA 624 | 10107207 | NELAP | LA | |
| 5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | EPA 624 | 10107207 | NELAP | LA | |
| 5235 - Vinyl chloride | EPA 624 | 10107207 | NELAP | LA | |
| 5260 - Xylene (total) | EPA 624 | 10107207 | NELAP | LA | |
| 4680 - cis-1,3-Dichloropropene | EPA 624 | 10107207 | NELAP | LA | |
| 4700 - trans-1,2-Dichloroethylene | EPA 624 | 10107207 | NELAP | LA | |
| 4685 - trans-1,3-Dichloropropylene | EPA 624 | 10107207 | NELAP | LA | |
| 5155 - 1,2,4-Trichlorobenzene | EPA 625 | 10107401 | NELAP | LA | |
| 4610 - 1,2-Dichlorobenzene | EPA 625 | 10107401 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 625 | 10107401 | NELAP | LA | |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.

| Non Potable Water | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4620 - 1,4-Dichlorobenzene | EPA 625 | 10107401 | NELAP | LA | |
| 6840 - 2,4,6-Trichlorophenol | EPA 625 | 10107401 | NELAP | LA | |
| 6000 - 2,4-Dichlorophenol | EPA 625 | 10107401 | NELAP | LA | |
| 6130 - 2,4-Dimethylphenol | EPA 625 | 10107401 | NELAP | LA | |
| 6175 - 2,4-Dinitrophenol | EPA 625 | 10107401 | NELAP | LA | |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 625 | 10107401 | NELAP | LA | |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 625 | 10107401 | NELAP | LA | |
| 5795 - 2-Chloronaphthalene | EPA 625 | 10107401 | NELAP | LA | |
| 5800 - 2-Chlorophenol | EPA 625 | 10107401 | NELAP | LA | |
| 6360 - 2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol) | EPA 625 | 10107401 | NELAP | LA | |
| 6490 - 2-Nitrophenol | EPA 625 | 10107401 | NELAP | LA | |
| 5945 - 3,3'-Dichlorobenzidine | EPA 625 | 10107401 | NELAP | LA | |
| 5660 - 4-Bromophenyl phenyl ether | EPA 625 | 10107401 | NELAP | LA | |
| 5700 - 4-Chloro-3-methylphenol | EPA 625 | 10107401 | NELAP | LA | |
| 5825 - 4-Chlorophenyl phenylether | EPA 625 | 10107401 | NELAP | LA | |
| 6500 - 4-Nitrophenol | EPA 625 | 10107401 | NELAP | LA | |
| 5500 - Acenaphthene | EPA 625 | 10107401 | NELAP | LA | |
| 5505 - Acenaphthylene | EPA 625 | 10107401 | NELAP | LA | |
| 5555 - Anthracene | EPA 625 | 10107401 | NELAP | LA | |
| 5575 - Benzo(a)anthracene | EPA 625 | 10107401 | NELAP | LA | |
| 5580 - Benzo(a)pyrene | EPA 625 | 10107401 | NELAP | LA | |
| 5585 - Benzo(b)fluoranthene | EPA 625 | 10107401 | NELAP | LA | |
| 5590 - Benzo(g,h,i)perylene | EPA 625 | 10107401 | NELAP | LA | |
| 5600 - Benzo(k)fluoranthene | EPA 625 | 10107401 | NELAP | LA | |
| 5670 - Butyl benzyl phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 5855 - Chrysene | EPA 625 | 10107401 | NELAP | LA | |
| 6065 - Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP) | EPA 625 | 10107401 | NELAP | LA | |
| 5925 - Di-n-butyl phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 6200 - Di-n-octyl phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 5895 - Dibenz(a,h) anthracene | EPA 625 | 10107401 | NELAP | LA | |
| 6070 - Diethyl phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 6135 - Dimethyl phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 6265 - Fluoranthene | EPA 625 | 10107401 | NELAP | LA | |
| 6270 - Fluorene | EPA 625 | 10107401 | NELAP | LA | |
| 6275 - Hexachlorobenzene | EPA 625 | 10107401 | NELAP | LA | |
| 4835 - Hexachlorobutadiene | EPA 625 | 10107401 | NELAP | LA | |
| 6285 - Hexachlorocyclopentadiene | EPA 625 | 10107401 | NELAP | LA | |
| 4840 - Hexachloroethane | EPA 625 | 10107401 | NELAP | LA | |
| 6315 - Indeno(1,2,3-cd) pyrene | EPA 625 | 10107401 | NELAP | LA | |
| 6320 - Isophorone | EPA 625 | 10107401 | NELAP | LA | |
| 5005 - Naphthalene | EPA 625 | 10107401 | NELAP | LA | |
| 5015 - Nitrobenzene | EPA 625 | 10107401 | NELAP | LA | |
| 6605 - Pentachlorophenol | EPA 625 | 10107401 | NELAP | LA | |
| 6615 - Phenanthrene | EPA 625 | 10107401 | NELAP | LA | |
| 6625 - Phenol | EPA 625 | 10107401 | NELAP | LA | |
| 6665 - Pyrene | EPA 625 | 10107401 | NELAP | LA | |
| 5760 - bis(2-Chloroethoxy)methane | EPA 625 | 10107401 | NELAP | LA | |
| 5765 - bis(2-Chloroethyl) ether | EPA 625 | 10107401 | NELAP | LA | |
| 5780 - bis(2-Chloroisopropyl) ether | EPA 625 | 10107401 | NELAP | LA | |
| 6245 - bis(2-Ethoxyethyl) phthalate | EPA 625 | 10107401 | NELAP | LA | |
| 6545 - n-Nitrosodi-n-propylamine | EPA 625 | 10107401 | NELAP | LA | |
| 6530 - n-Nitrosodimethylamine | EPA 625 | 10107401 | NELAP | LA | |
| 6535 - n-Nitrosodiphenylamine | EPA 625 | 10107401 | NELAP | LA | |
| 2835 - Gross alpha-beta | EPA 900 | 10112400 | NELAP | LA | |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

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| Non Potable Water | | | | | |
|--|-----------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 2830 - Gross-alpha | EPA 900 | 10112400 | NELAP | LA | |
| 2840 - Gross-beta | EPA 900 | 10112400 | NELAP | LA | |
| 2800 - Cesium-134 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2805 - Cesium-137 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2855 - Gross gamma | EPA 901.1 | 10112808 | NELAP | LA | |
| 100586 - Photon Emitters | EPA 901.1 | 10112808 | NELAP | LA | |
| 2955 - Radioactive cesium | EPA 901.1 | 10112808 | NELAP | LA | |
| 3070 - Zinc-65 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2965 - Radium-226 | EPA 903 | 10113209 | NELAP | LA | |
| 2750 - Total alpha radium | EPA 903 | 10113209 | NELAP | LA | |
| 3005 - Strontium-90 | EPA 905 | 10113801 | NELAP | LA | |
| 3030 - Tritium | EPA 906 | 10114008 | NELAP | LA | |
| 1860 - Oil & Grease | EPA 1664A (HEM) | 10127807 | NELAP | LA | |
| 100004 - Acid Digestion of Aqueous samples and Extracts for Total Metals | EPA 3010A | 10133605 | NELAP | LA | |
| 1444 - Separatory Funnel Liquid-liquid extraction | EPA 3510C | 10138202 | NELAP | LA | |
| 1410 - Continuous Liquid-liquid extraction | EPA 3520C | 10139001 | NELAP | LA | |
| 1000 - Aluminum | EPA 6010C | 10155803 | NELAP | LA | |
| 1005 - Antimony | EPA 6010C | 10155803 | NELAP | LA | |
| 1010 - Arsenic | EPA 6010C | 10155803 | NELAP | LA | |
| 1015 - Barium | EPA 6010C | 10155803 | NELAP | LA | |
| 1020 - Beryllium | EPA 6010C | 10155803 | NELAP | LA | |
| 1025 - Boron | EPA 6010C | 10155803 | NELAP | LA | |
| 1030 - Cadmium | EPA 6010C | 10155803 | NELAP | LA | |
| 1035 - Calcium | EPA 6010C | 10155803 | NELAP | LA | |
| 1040 - Chromium | EPA 6010C | 10155803 | NELAP | LA | |
| 1050 - Cobalt | EPA 6010C | 10155803 | NELAP | LA | |
| 1055 - Copper | EPA 6010C | 10155803 | NELAP | LA | |
| 1070 - Iron | EPA 6010C | 10155803 | NELAP | LA | |
| 1075 - Lead | EPA 6010C | 10155803 | NELAP | LA | |
| 1080 - Lithium | EPA 6010C | 10155803 | NELAP | LA | |
| 1085 - Magnesium | EPA 6010C | 10155803 | NELAP | LA | |
| 1090 - Manganese | EPA 6010C | 10155803 | NELAP | LA | |
| 1100 - Molybdenum | EPA 6010C | 10155803 | NELAP | LA | |
| 1105 - Nickel | EPA 6010C | 10155803 | NELAP | LA | |
| 1909 - Phosphorus | EPA 6010C | 10155803 | NELAP | LA | |
| 1125 - Potassium | EPA 6010C | 10155803 | NELAP | LA | |
| 1140 - Selenium | EPA 6010C | 10155803 | NELAP | LA | |
| 1150 - Silver | EPA 6010C | 10155803 | NELAP | LA | |
| 1155 - Sodium | EPA 6010C | 10155803 | NELAP | LA | |
| 1160 - Strontium | EPA 6010C | 10155803 | NELAP | LA | |
| 1165 - Thallium | EPA 6010C | 10155803 | NELAP | LA | |
| 1175 - Tin | EPA 6010C | 10155803 | NELAP | LA | |
| 1180 - Titanium | EPA 6010C | 10155803 | NELAP | LA | |
| 1185 - Vanadium | EPA 6010C | 10155803 | NELAP | LA | |
| 1190 - Zinc | EPA 6010C | 10155803 | NELAP | LA | |
| 1000 - Aluminum | EPA 6020A | 10156408 | NELAP | LA | |
| 1005 - Antimony | EPA 6020A | 10156408 | NELAP | LA | |
| 1010 - Arsenic | EPA 6020A | 10156408 | NELAP | LA | |
| 1015 - Barium | EPA 6020A | 10156408 | NELAP | LA | |
| 1020 - Beryllium | EPA 6020A | 10156408 | NELAP | LA | |
| 1025 - Boron | EPA 6020A | 10156408 | NELAP | LA | |
| 1030 - Cadmium | EPA 6020A | 10156408 | NELAP | LA | |
| 1035 - Calcium | EPA 6020A | 10156408 | NELAP | LA | |
| 1034 - Cerium | EPA 6020A | 10156408 | NELAP | LA | |

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| Non Potable Water | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 1037 - Cesium | EPA 6020A | 10156408 | NELAP | LA | |
| 1040 - Chromium | EPA 6020A | 10156408 | NELAP | LA | |
| 1050 - Cobalt | EPA 6020A | 10156408 | NELAP | LA | |
| 1055 - Copper | EPA 6020A | 10156408 | NELAP | LA | |
| 1070 - Iron | EPA 6020A | 10156408 | NELAP | LA | |
| 1072 - Lanthanum | EPA 6020A | 10156408 | NELAP | LA | |
| 1075 - Lead | EPA 6020A | 10156408 | NELAP | LA | |
| 1080 - Lithium | EPA 6020A | 10156408 | NELAP | LA | |
| 1085 - Magnesium | EPA 6020A | 10156408 | NELAP | LA | |
| 1090 - Manganese | EPA 6020A | 10156408 | NELAP | LA | |
| 1100 - Molybdenum | EPA 6020A | 10156408 | NELAP | LA | |
| 1103 - Neodymium | EPA 6020A | 10156408 | NELAP | LA | |
| 1105 - Nickel | EPA 6020A | 10156408 | NELAP | LA | |
| 1909 - Phosphorus | EPA 6020A | 10156408 | NELAP | LA | |
| 1125 - Potassium | EPA 6020A | 10156408 | NELAP | LA | |
| 1127 - Praseodymium | EPA 6020A | 10156408 | NELAP | LA | |
| 1140 - Selenium | EPA 6020A | 10156408 | NELAP | LA | |
| 1145 - Silicon | EPA 6020A | 10156408 | NELAP | LA | |
| 1150 - Silver | EPA 6020A | 10156408 | NELAP | LA | |
| 1155 - Sodium | EPA 6020A | 10156408 | NELAP | LA | |
| 1160 - Strontium | EPA 6020A | 10156408 | NELAP | LA | |
| 1165 - Thallium | EPA 6020A | 10156408 | NELAP | LA | |
| 1170 - Thorium | EPA 6020A | 10156408 | NELAP | LA | |
| 1175 - Tin | EPA 6020A | 10156408 | NELAP | LA | |
| 1180 - Titanium | EPA 6020A | 10156408 | NELAP | LA | |
| 1183 - Tungsten | EPA 6020A | 10156408 | NELAP | LA | |
| 3035 - Uranium | EPA 6020A | 10156408 | NELAP | LA | |
| 1185 - Vanadium | EPA 6020A | 10156408 | NELAP | LA | |
| 1190 - Zinc | EPA 6020A | 10156408 | NELAP | LA | |
| 1192 - Zirconium | EPA 6020A | 10156408 | NELAP | LA | |
| 1045 - Chromium VI | EPA 7196A | 10162400 | NELAP | LA | |
| 1095 - Mercury | EPA 7470A | 10165807 | NELAP | LA | |
| 9369 - Diesel range organics (DRO) | EPA 8015B | 10173601 | NELAP | LA | |
| 4785 - Ethylene glycol | EPA 8015B | 10173601 | NELAP | LA | |
| 9408 - Gasoline range organics (GRO) | EPA 8015B | 10173601 | NELAP | LA | |
| 6657 - Propylene Glycol | EPA 8015B | 10173601 | NELAP | LA | |
| 6835 - 2,4,5-Trichlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6840 - 2,4,6-Trichlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6000 - 2,4-Dichlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6130 - 2,4-Dimethylphenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6175 - 2,4-Dinitrophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6005 - 2,6-Dichlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 5800 - 2-Chlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6360 - 2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol) | EPA 8041 | 10176600 | NELAP | LA | |
| 6400 - 2-Methylphenol (o-Cresol) | EPA 8041 | 10176600 | NELAP | LA | |
| 6490 - 2-Nitrophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6412 - 3+4 Methylphenol | EPA 8041 | 10176600 | NELAP | LA | |
| 5700 - 4-Chloro-3-methylphenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6500 - 4-Nitrophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 8620 - Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8041 | 10176600 | NELAP | LA | |
| 6605 - Pentachlorophenol | EPA 8041 | 10176600 | NELAP | LA | |
| 6625 - Phenol | EPA 8041 | 10176600 | NELAP | LA | |
| 7355 - 4,4'-DDD | EPA 8081B | 10178800 | NELAP | LA | |
| 7360 - 4,4'-DDE | EPA 8081B | 10178800 | NELAP | LA | |

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|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 7365 - 4,4'-DDT | EPA 8081B | 10178800 | NELAP | LA | |
| 7025 - Aldrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7250 - Chlordane (tech.) | EPA 8081B | 10178800 | NELAP | LA | |
| 7470 - Dieldrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7510 - Endosulfan I | EPA 8081B | 10178800 | NELAP | LA | |
| 7515 - Endosulfan II | EPA 8081B | 10178800 | NELAP | LA | |
| 7520 - Endosulfan sulfate | EPA 8081B | 10178800 | NELAP | LA | |
| 7540 - Endrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7530 - Endrin aldehyde | EPA 8081B | 10178800 | NELAP | LA | |
| 7535 - Endrin ketone | EPA 8081B | 10178800 | NELAP | LA | |
| 7685 - Heptachlor | EPA 8081B | 10178800 | NELAP | LA | |
| 7690 - Heptachlor epoxide | EPA 8081B | 10178800 | NELAP | LA | |
| 7810 - Methoxychlor | EPA 8081B | 10178800 | NELAP | LA | |
| 8250 - Toxaphene (Chlorinated camphene) | EPA 8081B | 10178800 | NELAP | LA | |
| 7110 - alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |
| 7240 - alpha-Chlordane | EPA 8081B | 10178800 | NELAP | LA | |
| 7115 - beta-BHC (beta-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |
| 7105 - delta-BHC | EPA 8081B | 10178800 | NELAP | LA | |
| 7120 - gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |
| 7245 - gamma-Chlordane | EPA 8081B | 10178800 | NELAP | LA | |
| 8880 - Aroclor-1016 (PCB-1016) | EPA 8082A | 10179201 | NELAP | LA | |
| 8885 - Aroclor-1221 (PCB-1221) | EPA 8082A | 10179201 | NELAP | LA | |
| 8890 - Aroclor-1232 (PCB-1232) | EPA 8082A | 10179201 | NELAP | LA | |
| 8895 - Aroclor-1242 (PCB-1242) | EPA 8082A | 10179201 | NELAP | LA | |
| 8900 - Aroclor-1248 (PCB-1248) | EPA 8082A | 10179201 | NELAP | LA | |
| 8905 - Aroclor-1254 (PCB-1254) | EPA 8082A | 10179201 | NELAP | LA | |
| 8910 - Aroclor-1260 (PCB-1260) | EPA 8082A | 10179201 | NELAP | LA | |
| 8655 - 2,4,5-T | EPA 8151A | 10183207 | NELAP | LA | |
| 8545 - 2,4-D | EPA 8151A | 10183207 | NELAP | LA | |
| 8560 - 2,4-DB | EPA 8151A | 10183207 | NELAP | LA | |
| 8555 - Dalapon | EPA 8151A | 10183207 | NELAP | LA | |
| 8595 - Dicamba | EPA 8151A | 10183207 | NELAP | LA | |
| 8605 - Dichloroprop (Dichloroprop) | EPA 8151A | 10183207 | NELAP | LA | |
| 8620 - Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8151A | 10183207 | NELAP | LA | |
| 8650 - Silvex (2,4,5-TP) | EPA 8151A | 10183207 | NELAP | LA | |
| 5105 - 1,1,1,2-Tetrachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5160 - 1,1,1-Trichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5110 - 1,1,2,2-Tetrachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260B | 10184802 | NELAP | LA | |
| 5165 - 1,1,2-Trichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4630 - 1,1-Dichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4640 - 1,1-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4670 - 1,1-Dichloropropene | EPA 8260B | 10184802 | NELAP | LA | |
| 5150 - 1,2,3-Trichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5180 - 1,2,3-Trichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5210 - 1,2,4-Trimethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4570 - 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260B | 10184802 | NELAP | LA | |
| 4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260B | 10184802 | NELAP | LA | |

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| Non Potable Water | | | | | |
|---|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4610 - 1,2-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4635 - 1,2-Dichloroethane (Ethylene dichloride) | EPA 8260B | 10184802 | NELAP | LA | |
| 4655 - 1,2-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 5215 - 1,3,5-Trimethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4660 - 1,3-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4735 - 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4665 - 2,2-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 4410 - 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260B | 10184802 | NELAP | LA | |
| 4500 - 2-Chloroethyl vinyl ether | EPA 8260B | 10184802 | NELAP | LA | |
| 4535 - 2-Chlorotoluene | EPA 8260B | 10184802 | NELAP | LA | |
| 4860 - 2-Hexanone | EPA 8260B | 10184802 | NELAP | LA | |
| 4995 - 4-Methyl-2-pentanone (MIBK) | EPA 8260B | 10184802 | NELAP | LA | |
| 4315 - Acetone | EPA 8260B | 10184802 | NELAP | LA | |
| 4320 - Acetonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4325 - Acrolein (Propenal) | EPA 8260B | 10184802 | NELAP | LA | |
| 4340 - Acrylonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4355 - Allyl chloride (3-Chloropropene) | EPA 8260B | 10184802 | NELAP | LA | |
| 4375 - Benzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4385 - Bromobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4390 - Bromochloromethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4395 - Bromodichloromethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4400 - Bromoform | EPA 8260B | 10184802 | NELAP | LA | |
| 4450 - Carbon disulfide | EPA 8260B | 10184802 | NELAP | LA | |
| 4455 - Carbon tetrachloride | EPA 8260B | 10184802 | NELAP | LA | |
| 4475 - Chlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4575 - Chlorodibromomethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4485 - Chloroethane (Ethyl chloride) | EPA 8260B | 10184802 | NELAP | LA | |
| 4505 - Chloroform | EPA 8260B | 10184802 | NELAP | LA | |
| 4525 - Chloroprene (2-Chloro-1,3-butadiene) | EPA 8260B | 10184802 | NELAP | LA | |
| 4595 - Dibromomethane (Methylene bromide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4625 - Dichlorodifluoromethane (Freon-12) | EPA 8260B | 10184802 | NELAP | LA | |
| 4725 - Diethyl ether | EPA 8260B | 10184802 | NELAP | LA | |
| 4755 - Ethyl acetate | EPA 8260B | 10184802 | NELAP | LA | |
| 4810 - Ethyl methacrylate | EPA 8260B | 10184802 | NELAP | LA | |
| 4765 - Ethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4835 - Hexachlorobutadiene | EPA 8260B | 10184802 | NELAP | LA | |
| 4870 - Iodomethane (Methyl iodide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4875 - Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260B | 10184802 | NELAP | LA | |
| 4900 - Isopropylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4925 - Methacrylonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4950 - Methyl bromide (Bromomethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 4960 - Methyl chloride (Chloromethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 4990 - Methyl methacrylate | EPA 8260B | 10184802 | NELAP | LA | |
| 5000 - Methyl tert-butyl ether (MTBE) | EPA 8260B | 10184802 | NELAP | LA | |
| 4975 - Methylene chloride (Dichloromethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8260B | 10184802 | NELAP | LA | |
| 5035 - Pentachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5080 - Propionitrile (Ethyl cyanide) | EPA 8260B | 10184802 | NELAP | LA | |

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|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 5100 - Styrene | EPA 8260B | 10184802 | NELAP | LA | |
| 5115 - Tetrachloroethylene (Perchloroethylene) | EPA 8260B | 10184802 | NELAP | LA | |
| 5140 - Toluene | EPA 8260B | 10184802 | NELAP | LA | |
| 5170 - Trichloroethene (Trichloroethylene) | EPA 8260B | 10184802 | NELAP | LA | |
| 5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | EPA 8260B | 10184802 | NELAP | LA | |
| 5225 - Vinyl acetate | EPA 8260B | 10184802 | NELAP | LA | |
| 5235 - Vinyl chloride | EPA 8260B | 10184802 | NELAP | LA | |
| 5260 - Xylene (total) | EPA 8260B | 10184802 | NELAP | LA | |
| 4645 - cis-1,2-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4680 - cis-1,3-Dichloropropene | EPA 8260B | 10184802 | NELAP | LA | |
| 5240 - m+p-xylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4435 - n-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5250 - o-Xylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4440 - sec-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4445 - tert-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4700 - trans-1,2-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4685 - trans-1,3-Dichloropropylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4605 - trans-1,4-Dichloro-2-butene | EPA 8260B | 10184802 | NELAP | LA | |
| 6703 - 1,1'-Biphenyl (BZ-0) | EPA 8270D | 10186002 | NELAP | LA | |
| 6715 - 1,2,4,5-Tetrachlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 4610 - 1,2-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 4735 - 1,4-Dioxane (1,4- Diethyleneoxide) | EPA 8270D | 10186002 | NELAP | LA | |
| 6420 - 1,4-Naphthoquinone | EPA 8270D | 10186002 | NELAP | LA | |
| 6380 - 1-Methylnaphthalene | EPA 8270D | 10186002 | NELAP | LA | |
| 6425 - 1-Naphthylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6735 - 2,3,4,6-Tetrachlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6835 - 2,4,5-Trichlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6840 - 2,4,6-Trichlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6000 - 2,4-Dichlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6130 - 2,4-Dimethylphenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6175 - 2,4-Dinitrophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8270D | 10186002 | NELAP | LA | |
| 6005 - 2,6-Dichlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8270D | 10186002 | NELAP | LA | |
| 5515 - 2-Acetylaminofluorene | EPA 8270D | 10186002 | NELAP | LA | |
| 5795 - 2-Chloronaphthalene | EPA 8270D | 10186002 | NELAP | LA | |
| 5800 - 2-Chlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6360 - 2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol) | EPA 8270D | 10186002 | NELAP | LA | |
| 5145 - 2-Methylaniline (o-Toluidine) | EPA 8270D | 10186002 | NELAP | LA | |
| 6385 - 2-Methylnaphthalene | EPA 8270D | 10186002 | NELAP | LA | |
| 6400 - 2-Methylphenol (o-Cresol) | EPA 8270D | 10186002 | NELAP | LA | |
| 6430 - 2-Naphthylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6460 - 2-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA | |
| 6490 - 2-Nitrophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6412 - 3+4 Methylphenol | EPA 8270D | 10186002 | NELAP | LA | |
| 5945 - 3,3'-Dichlorobenzidine | EPA 8270D | 10186002 | NELAP | LA | |
| 6120 - 3,3'-Dimethylbenzidine | EPA 8270D | 10186002 | NELAP | LA | |
| 6355 - 3-Methylcholanthrene | EPA 8270D | 10186002 | NELAP | LA | |
| 6465 - 3-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA | |
| 5540 - 4-Aminobiphenyl | EPA 8270D | 10186002 | NELAP | LA | |

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| Analyte | Method Name | Method Code | Type | AB |
|--|-------------|-------------|-------|----|
| 5660 - 4-Bromophenyl phenyl ether | EPA 8270D | 10186002 | NELAP | LA |
| 5700 - 4-Chloro-3-methylphenol | EPA 8270D | 10186002 | NELAP | LA |
| 5745 - 4-Chloroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 5825 - 4-Chlorophenyl phenylether | EPA 8270D | 10186002 | NELAP | LA |
| 4540 - 4-Chlorotoluene | EPA 8270D | 10186002 | NELAP | LA |
| 6470 - 4-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 6500 - 4-Nitrophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6510 - 4-Nitroquinoline 1-oxide | EPA 8270D | 10186002 | NELAP | LA |
| 6570 - 5-Nitro-o-toluidine | EPA 8270D | 10186002 | NELAP | LA |
| 6115 - 7,12-Dimethylbenz(a) anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5500 - Acenaphthene | EPA 8270D | 10186002 | NELAP | LA |
| 5505 - Acenaphthylene | EPA 8270D | 10186002 | NELAP | LA |
| 5510 - Acetophenone | EPA 8270D | 10186002 | NELAP | LA |
| 5545 - Aniline | EPA 8270D | 10186002 | NELAP | LA |
| 5555 - Anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5560 - Aramite | EPA 8270D | 10186002 | NELAP | LA |
| 7065 - Atrazine | EPA 8270D | 10186002 | NELAP | LA |
| 5562 - Azobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 5570 - Benzaldehyde | EPA 8270D | 10186002 | NELAP | LA |
| 5575 - Benzo(a)anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5580 - Benzo(a)pyrene | EPA 8270D | 10186002 | NELAP | LA |
| 5585 - Benzo(b)fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 5590 - Benzo(g,h,i)perylene | EPA 8270D | 10186002 | NELAP | LA |
| 5600 - Benzo(k)fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 5610 - Benzoic acid | EPA 8270D | 10186002 | NELAP | LA |
| 5630 - Benzyl alcohol | EPA 8270D | 10186002 | NELAP | LA |
| 5670 - Butyl benzyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7180 - Caprolactam | EPA 8270D | 10186002 | NELAP | LA |
| 5680 - Carbazole | EPA 8270D | 10186002 | NELAP | LA |
| 7260 - Chlorobenzilate | EPA 8270D | 10186002 | NELAP | LA |
| 5855 - Chrysene | EPA 8270D | 10186002 | NELAP | LA |
| 4557 - Cyclohexanol | EPA 8270D | 10186002 | NELAP | LA |
| 6065 - Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP) | EPA 8270D | 10186002 | NELAP | LA |
| 5925 - Di-n-butyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 6200 - Di-n-octyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7405 - Diallate | EPA 8270D | 10186002 | NELAP | LA |
| 5895 - Dibenz(a,h) anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5905 - Dibenzofuran | EPA 8270D | 10186002 | NELAP | LA |
| 6070 - Diethyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7475 - Dimethoate | EPA 8270D | 10186002 | NELAP | LA |
| 6135 - Dimethyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 8625 - Disulfoton | EPA 8270D | 10186002 | NELAP | LA |
| 4810 - Ethyl methacrylate | EPA 8270D | 10186002 | NELAP | LA |
| 6260 - Ethyl methanesulfonate | EPA 8270D | 10186002 | NELAP | LA |
| 7580 - Famphur | EPA 8270D | 10186002 | NELAP | LA |
| 6265 - Fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 6270 - Fluorene | EPA 8270D | 10186002 | NELAP | LA |
| 6275 - Hexachlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4835 - Hexachlorobutadiene | EPA 8270D | 10186002 | NELAP | LA |
| 6285 - Hexachlorocyclopentadiene | EPA 8270D | 10186002 | NELAP | LA |
| 4840 - Hexachloroethane | EPA 8270D | 10186002 | NELAP | LA |
| 6295 - Hexachloropropene | EPA 8270D | 10186002 | NELAP | LA |
| 6315 - Indeno(1,2,3-cd) pyrene | EPA 8270D | 10186002 | NELAP | LA |
| 7725 - Isodrin | EPA 8270D | 10186002 | NELAP | LA |
| 6320 - Isophorone | EPA 8270D | 10186002 | NELAP | LA |

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|---|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 6325 - Isosafrole | EPA 8270D | 10186002 | NELAP | LA | |
| 6345 - Methapyrilene | EPA 8270D | 10186002 | NELAP | LA | |
| 4990 - Methyl methacrylate | EPA 8270D | 10186002 | NELAP | LA | |
| 6375 - Methyl methanesulfonate | EPA 8270D | 10186002 | NELAP | LA | |
| 7825 - Methyl parathion (Parathion, methyl) | EPA 8270D | 10186002 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8270D | 10186002 | NELAP | LA | |
| 5015 - Nitrobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 6590 - Pentachlorobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 5035 - Pentachloroethane | EPA 8270D | 10186002 | NELAP | LA | |
| 6600 - Pentachloronitrobenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 6605 - Pentachlorophenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6610 - Phenacetin | EPA 8270D | 10186002 | NELAP | LA | |
| 6615 - Phenanthrene | EPA 8270D | 10186002 | NELAP | LA | |
| 6625 - Phenol | EPA 8270D | 10186002 | NELAP | LA | |
| 6665 - Pyrene | EPA 8270D | 10186002 | NELAP | LA | |
| 5095 - Pyridine | EPA 8270D | 10186002 | NELAP | LA | |
| 6125 - a-a-Dimethylphenethylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 5760 - bis(2-Chloroethoxy)methane | EPA 8270D | 10186002 | NELAP | LA | |
| 5765 - bis(2-Chloroethyl) ether | EPA 8270D | 10186002 | NELAP | LA | |
| 5780 - bis(2-Chloroisopropyl) ether | EPA 8270D | 10186002 | NELAP | LA | |
| 5025 - n-Nitroso-di-n-butylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6545 - n-Nitrosodi-n-propylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6525 - n-Nitrosodiethylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6530 - n-Nitrosodimethylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6535 - n-Nitrosodiphenylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6550 - n-Nitrosomethylethylamine | EPA 8270D | 10186002 | NELAP | LA | |
| 6555 - n-Nitrosomorpholine | EPA 8270D | 10186002 | NELAP | LA | |
| 6560 - n-Nitrosopiperidine | EPA 8270D | 10186002 | NELAP | LA | |
| 6565 - n-Nitrosopyrrolidine | EPA 8270D | 10186002 | NELAP | LA | |
| 5090 - n-Propylbenzene | EPA 8270D | 10186002 | NELAP | LA | |
| 8290 - o,o,o-Triethyl phosphorothioate | EPA 8270D | 10186002 | NELAP | LA | |
| 5500 - Acenaphthene | EPA 8310 | 10187607 | NELAP | LA | |
| 5505 - Acenaphthylene | EPA 8310 | 10187607 | NELAP | LA | |
| 5555 - Anthracene | EPA 8310 | 10187607 | NELAP | LA | |
| 5575 - Benzo(a)anthracene | EPA 8310 | 10187607 | NELAP | LA | |
| 5580 - Benzo(a)pyrene | EPA 8310 | 10187607 | NELAP | LA | |
| 5585 - Benzo(b)fluoranthene | EPA 8310 | 10187607 | NELAP | LA | |
| 5590 - Benzo(g,h,i)perylene | EPA 8310 | 10187607 | NELAP | LA | |
| 5600 - Benzo(k)fluoranthene | EPA 8310 | 10187607 | NELAP | LA | |
| 5855 - Chrysene | EPA 8310 | 10187607 | NELAP | LA | |
| 5895 - Dibenz(a,h) anthracene | EPA 8310 | 10187607 | NELAP | LA | |
| 6265 - Fluoranthene | EPA 8310 | 10187607 | NELAP | LA | |
| 6270 - Fluorene | EPA 8310 | 10187607 | NELAP | LA | |
| 6315 - Indeno(1,2,3-cd) pyrene | EPA 8310 | 10187607 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8310 | 10187607 | NELAP | LA | |
| 6615 - Phenanthrene | EPA 8310 | 10187607 | NELAP | LA | |
| 6665 - Pyrene | EPA 8310 | 10187607 | NELAP | LA | |
| 6885 - 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8321A | 10189001 | NELAP | LA | |
| 6160 - 1,3-Dinitrobenzene (1,3-DNB) | EPA 8321A | 10189001 | NELAP | LA | |
| 8655 - 2,4,5-T | EPA 8321A | 10189001 | NELAP | LA | |
| 9651 - 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8321A | 10189001 | NELAP | LA | |
| 8545 - 2,4-D | EPA 8321A | 10189001 | NELAP | LA | |
| 8560 - 2,4-DB | EPA 8321A | 10189001 | NELAP | LA | |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8321A | 10189001 | NELAP | LA | |
| 6181 - 2,6-Diamino-4-nitrotoluene | EPA 8321A | 10189001 | NELAP | LA | |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8321A | 10189001 | NELAP | LA | |

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|---|----------------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 9303 - 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8321A | 10189001 | NELAP | LA | |
| 9507 - 2-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA | |
| 6150 - 3,5-Dinitroaniline | EPA 8321A | 10189001 | NELAP | LA | |
| 9510 - 3-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA | |
| 9306 - 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8321A | 10189001 | NELAP | LA | |
| 9513 - 4-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA | |
| 8555 - Dalapon | EPA 8321A | 10189001 | NELAP | LA | |
| 8595 - Dicamba | EPA 8321A | 10189001 | NELAP | LA | |
| 8605 - Dichloroprop (Dichloroprop) | EPA 8321A | 10189001 | NELAP | LA | |
| 8620 - Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8321A | 10189001 | NELAP | LA | |
| 7775 - MCPA | EPA 8321A | 10189001 | NELAP | LA | |
| 7780 - MCPP | EPA 8321A | 10189001 | NELAP | LA | |
| 6415 - Methyl-2,4,6-trinitrophenylitramine (tetryl) | EPA 8321A | 10189001 | NELAP | LA | |
| 6485 - Nitroglycerin | EPA 8321A | 10189001 | NELAP | LA | |
| 9522 - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8321A | 10189001 | NELAP | LA | |
| 9558 - Pentaerythritoltetranitrate | EPA 8321A | 10189001 | NELAP | LA | |
| 9432 - RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8321A | 10189001 | NELAP | LA | |
| 8650 - Silvex (2,4,5-TP) | EPA 8321A | 10189001 | NELAP | LA | |
| 2045 - Total Organic Halides (TOX) | EPA 9020B | 10194408 | NELAP | LA | |
| 2005 - Sulfide | EPA 9030 | 10195207 | NELAP | LA | |
| 1610 - Conductivity | EPA 9050A | 10198808 | NELAP | LA | |
| 1540 - Bromide | EPA 9056A | 10199607 | NELAP | LA | |
| 1575 - Chloride | EPA 9056A | 10199607 | NELAP | LA | |
| 1730 - Fluoride | EPA 9056A | 10199607 | NELAP | LA | |
| 1810 - Nitrate as N | EPA 9056A | 10199607 | NELAP | LA | |
| 1840 - Nitrite as N | EPA 9056A | 10199607 | NELAP | LA | |
| 1870 - Orthophosphate as P | EPA 9056A | 10199607 | NELAP | LA | |
| 2000 - Sulfate | EPA 9056A | 10199607 | NELAP | LA | |
| 2835 - Gross alpha-beta | EPA 9310 | 10208205 | NELAP | LA | |
| 2830 - Gross-alpha | EPA 9310 | 10208205 | NELAP | LA | |
| 2840 - Gross-beta | EPA 9310 | 10208205 | NELAP | LA | |
| 100210 - Alpha Emitting Radium Isotopes | EPA 9315 | 10208409 | NELAP | LA | |
| 2965 - Radium-226 | EPA 9315 | 10208409 | NELAP | LA | |
| 2975 - Total radium | EPA 9315 | 10208409 | NELAP | LA | |
| 2970 - Radium-228 | EPA 9320 | 10208603 | NELAP | LA | |
| 4323 - Acetylene | EPA RSK-175 (GC/FID) | 10212905 | NELAP | LA | |
| 4747 - Ethane | EPA RSK-175 (GC/FID) | 10212905 | NELAP | LA | |
| 4752 - Ethylene | EPA RSK-175 (GC/FID) | 10212905 | NELAP | LA | |
| 4926 - Methane | EPA RSK-175 (GC/FID) | 10212905 | NELAP | LA | |
| 1780 - Ignitability | EPA 1010A | 10234807 | NELAP | LA | |
| 2830 - Gross-alpha | EPA 900.0 (GPC) | 10242601 | NELAP | LA | |
| 2840 - Gross-beta | EPA 900.0 (GPC) | 10242601 | NELAP | LA | |
| 1645 - Total Cyanide | EPA 9010C | 10243002 | NELAP | LA | |
| 1645 - Total Cyanide | EPA 9012B | 10243206 | NELAP | LA | |
| 1900 - pH | EPA 9040C | 10244403 | NELAP | LA | |
| 1900 - pH | EPA 9045D | 10244607 | NELAP | LA | |
| 2040 - Total Organic Carbon | EPA 9060A | 10244801 | NELAP | LA | |
| 1406 - Purge and trap for aqueous phase samples | EPA 5030C | 10284603 | NELAP | LA | |
| 1895 - Perchlorate | EPA 6850 | 10304606 | NELAP | LA | |

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| Non Potable Water | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 5105 - 1,1,1,2-Tetrachloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 5160 - 1,1,1-Trichloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 5110 - 1,1,2,2-Tetrachloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260C | 10307003 | NELAP | LA | |
| 5165 - 1,1,2-Trichloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4630 - 1,1-Dichloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4640 - 1,1-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4670 - 1,1-Dichloropropene | EPA 8260C | 10307003 | NELAP | LA | |
| 5150 - 1,2,3-Trichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5180 - 1,2,3-Trichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 5182 - 1,2,3-Trimethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5210 - 1,2,4-Trimethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4570 - 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260C | 10307003 | NELAP | LA | |
| 4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4697 - 1,2-Dichloro-1,1,2-trifluoroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4610 - 1,2-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4635 - 1,2-Dichloroethane (Ethylene dichloride) | EPA 8260C | 10307003 | NELAP | LA | |
| 4655 - 1,2-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 5215 - 1,3,5-Trimethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4660 - 1,3-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 4835 - 1,3-Hexachlorobutadiene | EPA 8260C | 10307003 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4735 - 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4510 - 1-Chlorohexane | EPA 8260C | 10307003 | NELAP | LA | |
| 4665 - 2,2-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 4410 - 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260C | 10307003 | NELAP | LA | |
| 4500 - 2-Chloroethyl vinyl ether | EPA 8260C | 10307003 | NELAP | LA | |
| 4535 - 2-Chlorotoluene | EPA 8260C | 10307003 | NELAP | LA | |
| 4860 - 2-Hexanone | EPA 8260C | 10307003 | NELAP | LA | |
| 5020 - 2-Nitropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 4540 - 4-Chlorotoluene | EPA 8260C | 10307003 | NELAP | LA | |
| 4910 - 4-Isopropyltoluene (p-Cymene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4995 - 4-Methyl-2-pentanone (MIBK) | EPA 8260C | 10307003 | NELAP | LA | |
| 4315 - Acetone | EPA 8260C | 10307003 | NELAP | LA | |
| 4320 - Acetonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4325 - Acrolein (Propenal) | EPA 8260C | 10307003 | NELAP | LA | |
| 4340 - Acrylonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4355 - Allyl chloride (3-Chloropropene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4375 - Benzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4385 - Bromobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4390 - Bromochloromethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4395 - Bromodichloromethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4400 - Bromoform | EPA 8260C | 10307003 | NELAP | LA | |
| 4450 - Carbon disulfide | EPA 8260C | 10307003 | NELAP | LA | |
| 4455 - Carbon tetrachloride | EPA 8260C | 10307003 | NELAP | LA | |
| 4475 - Chlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4575 - Chlorodibromomethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4485 - Chloroethane (Ethyl chloride) | EPA 8260C | 10307003 | NELAP | LA | |
| 4505 - Chloroform | EPA 8260C | 10307003 | NELAP | LA | |

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|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4525 - Chloroprene (2-Chloro-1,3-butadiene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4555 - Cyclohexane | EPA 8260C | 10307003 | NELAP | LA | |
| 4560 - Cyclohexanone | EPA 8260C | 10307003 | NELAP | LA | |
| 9375 - Di-isopropylether (DIPE) (Isopropyl ether) | EPA 8260C | 10307003 | NELAP | LA | |
| 4595 - Dibromomethane (Methylene bromide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4625 - Dichlorodifluoromethane (Freon-12) | EPA 8260C | 10307003 | NELAP | LA | |
| 4725 - Diethyl ether | EPA 8260C | 10307003 | NELAP | LA | |
| 4750 - Ethanol | EPA 8260C | 10307003 | NELAP | LA | |
| 4755 - Ethyl acetate | EPA 8260C | 10307003 | NELAP | LA | |
| 4810 - Ethyl methacrylate | EPA 8260C | 10307003 | NELAP | LA | |
| 4770 - Ethyl-t-butyl ether (ETBE) (2-Ethoxy-2-methylpropane) | EPA 8260C | 10307003 | NELAP | LA | |
| 4765 - Ethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4835 - Hexachlorobutadiene | EPA 8260C | 10307003 | NELAP | LA | |
| 4870 - Iodomethane (Methyl iodide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4875 - Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260C | 10307003 | NELAP | LA | |
| 4895 - Isopropyl alcohol (2-Propanol, Isopropanol) | EPA 8260C | 10307003 | NELAP | LA | |
| 4900 - Isopropylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4925 - Methacrylonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4940 - Methyl acetate | EPA 8260C | 10307003 | NELAP | LA | |
| 4950 - Methyl bromide (Bromomethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 4960 - Methyl chloride (Chloromethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 4990 - Methyl methacrylate | EPA 8260C | 10307003 | NELAP | LA | |
| 5000 - Methyl tert-butyl ether (MTBE) | EPA 8260C | 10307003 | NELAP | LA | |
| 4975 - Methylene chloride (Dichloromethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8260C | 10307003 | NELAP | LA | |
| 5035 - Pentachloroethane | EPA 8260C | 10307003 | NELAP | LA | |
| 5080 - Propionitrile (Ethyl cyanide) | EPA 8260C | 10307003 | NELAP | LA | |
| 5100 - Styrene | EPA 8260C | 10307003 | NELAP | LA | |
| 4370 - T-amylmethylether (TAME) | EPA 8260C | 10307003 | NELAP | LA | |
| 5115 - Tetrachloroethylene (Perchloroethylene) | EPA 8260C | 10307003 | NELAP | LA | |
| 5120 - Tetrahydrofuran (THF) | EPA 8260C | 10307003 | NELAP | LA | |
| 5140 - Toluene | EPA 8260C | 10307003 | NELAP | LA | |
| 5170 - Trichloroethene (Trichloroethylene) | EPA 8260C | 10307003 | NELAP | LA | |
| 5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | EPA 8260C | 10307003 | NELAP | LA | |
| 5225 - Vinyl acetate | EPA 8260C | 10307003 | NELAP | LA | |
| 5235 - Vinyl chloride | EPA 8260C | 10307003 | NELAP | LA | |
| 5260 - Xylene (total) | EPA 8260C | 10307003 | NELAP | LA | |
| 4645 - cis-1,2-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4680 - cis-1,3-Dichloropropene | EPA 8260C | 10307003 | NELAP | LA | |
| 4600 - cis-1,4-Dichloro-2-butene | EPA 8260C | 10307003 | NELAP | LA | |
| 5240 - m+p-xylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4425 - n-Butyl alcohol (1-Butanol, n-Butanol) | EPA 8260C | 10307003 | NELAP | LA | |
| 4435 - n-Butylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5090 - n-Propylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5250 - o-Xylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4440 - sec-Butylbenzene | EPA 8260C | 10307003 | NELAP | LA | |

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| Non Potable Water | | | | | |
|---|------------------------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4420 - tert-Butyl alcohol | EPA 8260C | 10307003 | NELAP | LA | |
| 4445 - tert-Butylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4700 - trans-1,2-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4685 - trans-1,3-Dichloropropylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4605 - trans-1,4-Dichloro-2-butene | EPA 8260C | 10307003 | NELAP | LA | |
| 6885 - 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8330B | 10308006 | NELAP | LA | |
| 6160 - 1,3-Dinitrobenzene (1,3-DNB) | EPA 8330B | 10308006 | NELAP | LA | |
| 9651 - 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 9303 - 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8330B | 10308006 | NELAP | LA | |
| 9507 - 2-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 9510 - 3-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 9306 - 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8330B | 10308006 | NELAP | LA | |
| 9513 - 4-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 6415 - Methyl-2,4,6-trinitrophenylnitramine (tetryl) | EPA 8330B | 10308006 | NELAP | LA | |
| 5015 - Nitrobenzene | EPA 8330B | 10308006 | NELAP | LA | |
| 6485 - Nitroglycerin | EPA 8330B | 10308006 | NELAP | LA | |
| 9522 - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8330B | 10308006 | NELAP | LA | |
| 9432 - RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8330B | 10308006 | NELAP | LA | |
| 2800 - Cesium-134 | EPA 901.1 | 10308608 | NELAP | LA | |
| 2805 - Cesium-137 | EPA 901.1 | 10308608 | NELAP | LA | |
| 2815 - Cobalt-60 | EPA 901.1 | 10308608 | NELAP | LA | |
| 2826 - Gamma Emitters | EPA 901.1 | 10308608 | NELAP | LA | |
| 3070 - Zinc-65 | EPA 901.1 | 10308608 | NELAP | LA | |
| 2970 - Radium-228 | EPA 904.0 | 10309805 | NELAP | LA | |
| 100543 - Strontium, total | EPA 905.0 | 10310006 | NELAP | LA | |
| 3005 - Strontium-90 | EPA 905.0 | 10310006 | NELAP | LA | |
| 3030 - Tritium | EPA 906.0 | 10310200 | NELAP | LA | |
| 2965 - Radium-226 | EPA 9315 | 10311009 | NELAP | LA | |
| 1505 - Alkalinity as CaCO3 | SM 2320 B-97, Online Edition | 20045607 | NELAP | LA | |
| 1950 - Residue-total | SM 2540 B-97, Online Edition | 20049405 | NELAP | LA | |
| 1955 - Residue-filterable (TDS) | SM 2540 C-97, Online Edition | 20050402 | NELAP | LA | |
| 1960 - Residue-nonfilterable (TSS) | SM 2540 D-97, Online Edition | 20051201 | NELAP | LA | |
| 1900 - pH | SM 4500-H+ B-2000 | 20105219 | NELAP | LA | |
| 1530 - Biochemical oxygen demand | SM 5210 B-2001 | 20135255 | NELAP | LA | |
| 2758 - Antimony 124 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1006 - Antimony 125 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2765 - Barium-133 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1021 - Beryllium-7 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2772 - Bismuth-212 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2773 - Bismuth-214 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2794 - Cerium-141 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2800 - Cesium-134 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2805 - Cesium-137 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2812 - Cobalt-57 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2815 - Cobalt-60 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1068 - Europium-152 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1069 - Europium-154 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1078 - Europium-155 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2826 - Gamma Emitters | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |

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Non Potable Water

| Analyte | Method Name | Method Code | Type | AB |
|--------------------------------------|----------------------------------|-------------|-------|----|
| 2875 - Iodine-131 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2880 - Iridium-192 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2902 - Lead-212 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2903 - Lead-214 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2905 - Manganese-54 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2908 - Mercury-203 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2918 - Niobium-94 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1107 - Niobium-95 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2946 - Potassium-40 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2952 - Protactinium-234 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2960 - Radium-224 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2965 - Radium-226 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2988 - Ruthenium-103 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1136 - Ruthenium-106 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1156 - Sodium-22 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1164 - Strontium-85 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1166 - Thallium-208 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3031 - Thorium-227 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1171 - Thorium-228 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3032 - Thorium-231 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3028 - Thorium-234 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2942 - Tin-113 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3037 - Uranium-235 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3038 - Uranium-238 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3067 - Yttrium-88 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3070 - Zinc-65 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3072 - Zirconium-95 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2930 - Plutonium-238 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 2932 - Plutonium-239 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3036 - Uranium-234 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3038 - Uranium-238 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3005 - Strontium-90 | HASL 300 Sr-02-RC (GPC), 28th ED | 90009204 | NELAP | LA |
| 3005 - Strontium-90 | HASL 300 Sr-03-RC, 28th ED | 90009806 | NELAP | LA |
| 9408 - Gasoline range organics (GRO) | IDNR OA-1 | 90016403 | NELAP | LA |
| 9369 - Diesel range organics (DRO) | IDNR OA-2 | 90016607 | NELAP | LA |

Solid Chemical Materials

| Analyte | Method Name | Method Code | Type | AB |
|---|--------------------|-------------|-------|----|
| 2755 - Americium-241 | Eichrom RAW03 | 2257 | NELAP | LA |
| 2755 - Americium-241 | Eichrom ACW03 | 2259 | NELAP | LA |
| 2940 - Plutonium | Eichrom ACW03 | 2259 | NELAP | LA |
| 3035 - Uranium | Eichrom ACW03 | 2259 | NELAP | LA |
| 100499 - Neptunium | Eichrom ACW08 | 2260 | NELAP | LA |
| 1170 - Thorium | Eichrom ACW08 | 2260 | NELAP | LA |
| 2900 - Lead-210 | Eichrom OTW01 | 2264 | NELAP | LA |
| 2912 - Nickel-63 | Eichrom NiW01 | 2267 | NELAP | LA |
| 1170 - Thorium | Eichrom ACW10 | 2269 | NELAP | LA |
| 3000 - Strontium-89 (calc.) | EPA 905 (Modified) | 2441 | NELAP | LA |
| 3005 - Strontium-90 | EPA 905 (Modified) | 2441 | NELAP | LA |
| 3030 - Tritium | EPA 906 (Modified) | 2442 | NELAP | LA |
| 4735 - 1,4-Dioxane (1,4- Diethyleneoxide) | EPA 8260 SIM | 2995 | NELAP | LA |
| 1923 - Reactive Cyanide | EPA 7.3.3.2, Rev.3 | 10001204 | State | LA |
| 1925 - Reactive sulfide | EPA 7.3.4.2, Rev.3 | 10001408 | NELAP | LA |
| 1730 - Fluoride | EPA 340.2 | 10062007 | NELAP | LA |

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| Solid Chemical Materials | | | | | |
|--|------------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 1795 - Kjeldahl nitrogen - total | EPA 351.2, Rev.2 | 10065404 | NELAP | LA | |
| 1565 - Chemical oxygen demand | EPA 410.4 | 10077006 | NELAP | LA | |
| 2800 - Cesium-134 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2805 - Cesium-137 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2815 - Cobalt-60 | EPA 901.1 | 10112808 | NELAP | LA | |
| 2826 - Gamma Emitters | EPA 901.1 | 10112808 | NELAP | LA | |
| 100586 - Photon Emitters | EPA 901.1 | 10112808 | NELAP | LA | |
| 1466 - Toxicity Characteristic Leaching Procedure (TCLP) | EPA 1311 | 10118806 | NELAP | LA | |
| 1460 - Synthetic Precipitation Leaching Procedure | EPA 1312 | 10119003 | NELAP | LA | |
| 100007 - Acid Digestion of Sediments, Sludges, and soils | EPA 3050B | 10135601 | NELAP | LA | |
| 1402 - Alkaline Digestion for Hexavalent Chromium | EPA 3060A | 10136604 | NELAP | LA | |
| 1444 - Separatory Funnel Liquid-liquid extraction | EPA 3510C | 10138202 | NELAP | LA | |
| 1468 - Ultrasonic Extraction | EPA 3550C | 10142004 | NELAP | LA | |
| 1000 - Aluminum | EPA 6010C | 10155803 | NELAP | LA | |
| 1005 - Antimony | EPA 6010C | 10155803 | NELAP | LA | |
| 1010 - Arsenic | EPA 6010C | 10155803 | NELAP | LA | |
| 1015 - Barium | EPA 6010C | 10155803 | NELAP | LA | |
| 1020 - Beryllium | EPA 6010C | 10155803 | NELAP | LA | |
| 1025 - Boron | EPA 6010C | 10155803 | NELAP | LA | |
| 1030 - Cadmium | EPA 6010C | 10155803 | NELAP | LA | |
| 1035 - Calcium | EPA 6010C | 10155803 | NELAP | LA | |
| 1040 - Chromium | EPA 6010C | 10155803 | NELAP | LA | |
| 1050 - Cobalt | EPA 6010C | 10155803 | NELAP | LA | |
| 1055 - Copper | EPA 6010C | 10155803 | NELAP | LA | |
| 1070 - Iron | EPA 6010C | 10155803 | NELAP | LA | |
| 1075 - Lead | EPA 6010C | 10155803 | NELAP | LA | |
| 1080 - Lithium | EPA 6010C | 10155803 | NELAP | LA | |
| 1085 - Magnesium | EPA 6010C | 10155803 | NELAP | LA | |
| 1090 - Manganese | EPA 6010C | 10155803 | NELAP | LA | |
| 1100 - Molybdenum | EPA 6010C | 10155803 | NELAP | LA | |
| 1105 - Nickel | EPA 6010C | 10155803 | NELAP | LA | |
| 1125 - Potassium | EPA 6010C | 10155803 | NELAP | LA | |
| 1140 - Selenium | EPA 6010C | 10155803 | NELAP | LA | |
| 1145 - Silicon | EPA 6010C | 10155803 | NELAP | LA | |
| 1150 - Silver | EPA 6010C | 10155803 | NELAP | LA | |
| 1155 - Sodium | EPA 6010C | 10155803 | NELAP | LA | |
| 1160 - Strontium | EPA 6010C | 10155803 | NELAP | LA | |
| 1165 - Thallium | EPA 6010C | 10155803 | NELAP | LA | |
| 1175 - Tin | EPA 6010C | 10155803 | NELAP | LA | |
| 1180 - Titanium | EPA 6010C | 10155803 | NELAP | LA | |
| 1185 - Vanadium | EPA 6010C | 10155803 | NELAP | LA | |
| 1190 - Zinc | EPA 6010C | 10155803 | NELAP | LA | |
| 1000 - Aluminum | EPA 6020A | 10156408 | NELAP | LA | |
| 1005 - Antimony | EPA 6020A | 10156408 | NELAP | LA | |
| 1010 - Arsenic | EPA 6020A | 10156408 | NELAP | LA | |
| 1015 - Barium | EPA 6020A | 10156408 | NELAP | LA | |
| 1020 - Beryllium | EPA 6020A | 10156408 | NELAP | LA | |
| 1025 - Boron | EPA 6020A | 10156408 | NELAP | LA | |
| 1030 - Cadmium | EPA 6020A | 10156408 | NELAP | LA | |
| 1035 - Calcium | EPA 6020A | 10156408 | NELAP | LA | |
| 1034 - Cerium | EPA 6020A | 10156408 | NELAP | LA | |

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| Solid Chemical Materials | | | | | |
|---|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 1040 - Chromium | EPA 6020A | 10156408 | NELAP | LA | |
| 1050 - Cobalt | EPA 6020A | 10156408 | NELAP | LA | |
| 1055 - Copper | EPA 6020A | 10156408 | NELAP | LA | |
| 1070 - Iron | EPA 6020A | 10156408 | NELAP | LA | |
| 1072 - Lanthanum | EPA 6020A | 10156408 | NELAP | LA | |
| 1075 - Lead | EPA 6020A | 10156408 | NELAP | LA | |
| 1080 - Lithium | EPA 6020A | 10156408 | NELAP | LA | |
| 1085 - Magnesium | EPA 6020A | 10156408 | NELAP | LA | |
| 1090 - Manganese | EPA 6020A | 10156408 | NELAP | LA | |
| 1100 - Molybdenum | EPA 6020A | 10156408 | NELAP | LA | |
| 1103 - Neodymium | EPA 6020A | 10156408 | NELAP | LA | |
| 1105 - Nickel | EPA 6020A | 10156408 | NELAP | LA | |
| 1125 - Potassium | EPA 6020A | 10156408 | NELAP | LA | |
| 1127 - Praseodymium | EPA 6020A | 10156408 | NELAP | LA | |
| 1140 - Selenium | EPA 6020A | 10156408 | NELAP | LA | |
| 1150 - Silver | EPA 6020A | 10156408 | NELAP | LA | |
| 1155 - Sodium | EPA 6020A | 10156408 | NELAP | LA | |
| 1160 - Strontium | EPA 6020A | 10156408 | NELAP | LA | |
| 1165 - Thallium | EPA 6020A | 10156408 | NELAP | LA | |
| 1170 - Thorium | EPA 6020A | 10156408 | NELAP | LA | |
| 1175 - Tin | EPA 6020A | 10156408 | NELAP | LA | |
| 1180 - Titanium | EPA 6020A | 10156408 | NELAP | LA | |
| 1184 - Uranium | EPA 6020A | 10156408 | NELAP | LA | |
| 1185 - Vanadium | EPA 6020A | 10156408 | NELAP | LA | |
| 1190 - Zinc | EPA 6020A | 10156408 | NELAP | LA | |
| 1192 - Zirconium | EPA 6020A | 10156408 | NELAP | LA | |
| 1045 - Chromium VI | EPA 7196A | 10162400 | NELAP | LA | |
| 1095 - Mercury | EPA 7471B | 10166402 | NELAP | LA | |
| 9369 - Diesel range organics (DRO) | EPA 8015B | 10173601 | NELAP | LA | |
| 4785 - Ethylene glycol | EPA 8015B | 10173601 | NELAP | LA | |
| 9408 - Gasoline range organics (GRO) | EPA 8015B | 10173601 | NELAP | LA | |
| 6657 - Propylene Glycol | EPA 8015B | 10173601 | NELAP | LA | |
| 7355 - 4,4'-DDD | EPA 8081B | 10178800 | NELAP | LA | |
| 7360 - 4,4'-DDE | EPA 8081B | 10178800 | NELAP | LA | |
| 7365 - 4,4'-DDT | EPA 8081B | 10178800 | NELAP | LA | |
| 7025 - Aldrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7250 - Chlordane (tech.) | EPA 8081B | 10178800 | NELAP | LA | |
| 7470 - Dieldrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7510 - Endosulfan I | EPA 8081B | 10178800 | NELAP | LA | |
| 7515 - Endosulfan II | EPA 8081B | 10178800 | NELAP | LA | |
| 7520 - Endosulfan sulfate | EPA 8081B | 10178800 | NELAP | LA | |
| 7540 - Endrin | EPA 8081B | 10178800 | NELAP | LA | |
| 7530 - Endrin aldehyde | EPA 8081B | 10178800 | NELAP | LA | |
| 7535 - Endrin ketone | EPA 8081B | 10178800 | NELAP | LA | |
| 7685 - Heptachlor | EPA 8081B | 10178800 | NELAP | LA | |
| 7690 - Heptachlor epoxide | EPA 8081B | 10178800 | NELAP | LA | |
| 7810 - Methoxychlor | EPA 8081B | 10178800 | NELAP | LA | |
| 8250 - Toxaphene (Chlorinated camphene) | EPA 8081B | 10178800 | NELAP | LA | |
| 7110 - alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |
| 7240 - alpha-Chlordane | EPA 8081B | 10178800 | NELAP | LA | |
| 7115 - beta-BHC (beta-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |
| 7105 - delta-BHC | EPA 8081B | 10178800 | NELAP | LA | |
| 7120 - gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 8081B | 10178800 | NELAP | LA | |

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| Solid Chemical Materials | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 7245 - gamma-Chlordane | EPA 8081B | 10178800 | NELAP | LA | |
| 8880 - Aroclor-1016 (PCB-1016) | EPA 8082A | 10179201 | NELAP | LA | |
| 8885 - Aroclor-1221 (PCB-1221) | EPA 8082A | 10179201 | NELAP | LA | |
| 8890 - Aroclor-1232 (PCB-1232) | EPA 8082A | 10179201 | NELAP | LA | |
| 8895 - Aroclor-1242 (PCB-1242) | EPA 8082A | 10179201 | NELAP | LA | |
| 8900 - Aroclor-1248 (PCB-1248) | EPA 8082A | 10179201 | NELAP | LA | |
| 8905 - Aroclor-1254 (PCB-1254) | EPA 8082A | 10179201 | NELAP | LA | |
| 8910 - Aroclor-1260 (PCB-1260) | EPA 8082A | 10179201 | NELAP | LA | |
| 8655 - 2,4,5-T | EPA 8151A | 10183207 | NELAP | LA | |
| 8545 - 2,4-D | EPA 8151A | 10183207 | NELAP | LA | |
| 8560 - 2,4-DB | EPA 8151A | 10183207 | NELAP | LA | |
| 8555 - Dalapon | EPA 8151A | 10183207 | NELAP | LA | |
| 8595 - Dicamba | EPA 8151A | 10183207 | NELAP | LA | |
| 8605 - Dichloroprop (Dichloroprop) | EPA 8151A | 10183207 | NELAP | LA | |
| 8620 - Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8151A | 10183207 | NELAP | LA | |
| 7775 - MCPA | EPA 8151A | 10183207 | NELAP | LA | |
| 7780 - MCPP | EPA 8151A | 10183207 | NELAP | LA | |
| 8650 - Silvex (2,4,5-TP) | EPA 8151A | 10183207 | NELAP | LA | |
| 5105 - 1,1,1,2-Tetrachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5160 - 1,1,1-Trichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5110 - 1,1,2,2-Tetrachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260B | 10184802 | NELAP | LA | |
| 5165 - 1,1,2-Trichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4630 - 1,1-Dichloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4640 - 1,1-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4670 - 1,1-Dichloropropene | EPA 8260B | 10184802 | NELAP | LA | |
| 5150 - 1,2,3-Trichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5180 - 1,2,3-Trichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5210 - 1,2,4-Trimethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4570 - 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260B | 10184802 | NELAP | LA | |
| 4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4697 - 1,2-Dichloro-1,1,2-trifluoroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4610 - 1,2-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4635 - 1,2-Dichloroethane (Ethylene dichloride) | EPA 8260B | 10184802 | NELAP | LA | |
| 4655 - 1,2-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 5215 - 1,3,5-Trimethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4660 - 1,3-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4735 - 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4665 - 2,2-Dichloropropane | EPA 8260B | 10184802 | NELAP | LA | |
| 4410 - 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260B | 10184802 | NELAP | LA | |
| 4500 - 2-Chloroethyl vinyl ether | EPA 8260B | 10184802 | NELAP | LA | |
| 4535 - 2-Chlorotoluene | EPA 8260B | 10184802 | NELAP | LA | |
| 4860 - 2-Hexanone | EPA 8260B | 10184802 | NELAP | LA | |
| 4995 - 4-Methyl-2-pentanone (MIBK) | EPA 8260B | 10184802 | NELAP | LA | |
| 4315 - Acetone | EPA 8260B | 10184802 | NELAP | LA | |
| 4320 - Acetonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4325 - Acrolein (Propenal) | EPA 8260B | 10184802 | NELAP | LA | |

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| Solid Chemical Materials | | | | | |
|--|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4340 - Acrylonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4355 - Allyl chloride (3-Chloropropene) | EPA 8260B | 10184802 | NELAP | LA | |
| 4375 - Benzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4385 - Bromobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4390 - Bromochloromethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4395 - Bromodichloromethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4400 - Bromoform | EPA 8260B | 10184802 | NELAP | LA | |
| 4450 - Carbon disulfide | EPA 8260B | 10184802 | NELAP | LA | |
| 4455 - Carbon tetrachloride | EPA 8260B | 10184802 | NELAP | LA | |
| 4475 - Chlorobenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4575 - Chlorodibromomethane | EPA 8260B | 10184802 | NELAP | LA | |
| 4485 - Chloroethane (Ethyl chloride) | EPA 8260B | 10184802 | NELAP | LA | |
| 4505 - Chloroform | EPA 8260B | 10184802 | NELAP | LA | |
| 4525 - Chloroprene (2-Chloro-1,3-butadiene) | EPA 8260B | 10184802 | NELAP | LA | |
| 4595 - Dibromomethane (Methylene bromide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4625 - Dichlorodifluoromethane (Freon-12) | EPA 8260B | 10184802 | NELAP | LA | |
| 4725 - Diethyl ether | EPA 8260B | 10184802 | NELAP | LA | |
| 4755 - Ethyl acetate | EPA 8260B | 10184802 | NELAP | LA | |
| 4810 - Ethyl methacrylate | EPA 8260B | 10184802 | NELAP | LA | |
| 4765 - Ethylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4835 - Hexachlorobutadiene | EPA 8260B | 10184802 | NELAP | LA | |
| 4870 - Iodomethane (Methyl iodide) | EPA 8260B | 10184802 | NELAP | LA | |
| 4875 - Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260B | 10184802 | NELAP | LA | |
| 4900 - Isopropylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4925 - Methacrylonitrile | EPA 8260B | 10184802 | NELAP | LA | |
| 4950 - Methyl bromide (Bromomethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 4960 - Methyl chloride (Chloromethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 4990 - Methyl methacrylate | EPA 8260B | 10184802 | NELAP | LA | |
| 5000 - Methyl tert-butyl ether (MTBE) | EPA 8260B | 10184802 | NELAP | LA | |
| 4975 - Methylene chloride (Dichloromethane) | EPA 8260B | 10184802 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8260B | 10184802 | NELAP | LA | |
| 5035 - Pentachloroethane | EPA 8260B | 10184802 | NELAP | LA | |
| 5080 - Propionitrile (Ethyl cyanide) | EPA 8260B | 10184802 | NELAP | LA | |
| 5100 - Styrene | EPA 8260B | 10184802 | NELAP | LA | |
| 5115 - Tetrachloroethylene (Perchloroethylene) | EPA 8260B | 10184802 | NELAP | LA | |
| 5140 - Toluene | EPA 8260B | 10184802 | NELAP | LA | |
| 5170 - Trichloroethene (Trichloroethylene) | EPA 8260B | 10184802 | NELAP | LA | |
| 5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | EPA 8260B | 10184802 | NELAP | LA | |
| 5225 - Vinyl acetate | EPA 8260B | 10184802 | NELAP | LA | |
| 5235 - Vinyl chloride | EPA 8260B | 10184802 | NELAP | LA | |
| 5260 - Xylene (total) | EPA 8260B | 10184802 | NELAP | LA | |
| 4645 - cis-1,2-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4680 - cis-1,3-Dichloropropene | EPA 8260B | 10184802 | NELAP | LA | |
| 5240 - m+p-xylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4435 - n-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 5250 - o-Xylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4440 - sec-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4445 - tert-Butylbenzene | EPA 8260B | 10184802 | NELAP | LA | |
| 4700 - trans-1,2-Dichloroethylene | EPA 8260B | 10184802 | NELAP | LA | |
| 4685 - trans-1,3-Dichloropropylene | EPA 8260B | 10184802 | NELAP | LA | |

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Solid Chemical Materials

| Analyte | Method Name | Method Code | Type | AB |
|--|-------------|-------------|-------|----|
| 4605 - trans-1,4-Dichloro-2-butene | EPA 8260B | 10184802 | NELAP | LA |
| 6715 - 1,2,4,5-Tetrachlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4610 - 1,2-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4615 - 1,3-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4620 - 1,4-Dichlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 6165 - 1,4-Dinitrobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4735 - 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8270D | 10186002 | NELAP | LA |
| 6420 - 1,4-Naphthoquinone | EPA 8270D | 10186002 | NELAP | LA |
| 6380 - 1-Methylnaphthalene | EPA 8270D | 10186002 | NELAP | LA |
| 6425 - 1-Naphthylamine | EPA 8270D | 10186002 | NELAP | LA |
| 4659 - 2,2'-Oxybis(1-chloropropane) | EPA 8270D | 10186002 | NELAP | LA |
| 6735 - 2,3,4,6-Tetrachlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6835 - 2,4,5-Trichlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6840 - 2,4,6-Trichlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6000 - 2,4-Dichlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6130 - 2,4-Dimethylphenol | EPA 8270D | 10186002 | NELAP | LA |
| 6175 - 2,4-Dinitrophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8270D | 10186002 | NELAP | LA |
| 6005 - 2,6-Dichlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8270D | 10186002 | NELAP | LA |
| 5515 - 2-Acetylaminofluorene | EPA 8270D | 10186002 | NELAP | LA |
| 5795 - 2-Chloronaphthalene | EPA 8270D | 10186002 | NELAP | LA |
| 5800 - 2-Chlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6360 - 2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol) | EPA 8270D | 10186002 | NELAP | LA |
| 5145 - 2-Methylaniline (o-Toluidine) | EPA 8270D | 10186002 | NELAP | LA |
| 6385 - 2-Methylnaphthalene | EPA 8270D | 10186002 | NELAP | LA |
| 6400 - 2-Methylphenol (o-Cresol) | EPA 8270D | 10186002 | NELAP | LA |
| 6430 - 2-Naphthylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6460 - 2-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 6490 - 2-Nitrophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6412 - 3+4 Methylphenol | EPA 8270D | 10186002 | NELAP | LA |
| 5945 - 3,3'-Dichlorobenzidine | EPA 8270D | 10186002 | NELAP | LA |
| 6465 - 3-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 5540 - 4-Aminobiphenyl | EPA 8270D | 10186002 | NELAP | LA |
| 5660 - 4-Bromophenyl phenyl ether | EPA 8270D | 10186002 | NELAP | LA |
| 5700 - 4-Chloro-3-methylphenol | EPA 8270D | 10186002 | NELAP | LA |
| 5745 - 4-Chloroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 5825 - 4-Chlorophenyl phenylether | EPA 8270D | 10186002 | NELAP | LA |
| 6470 - 4-Nitroaniline | EPA 8270D | 10186002 | NELAP | LA |
| 6500 - 4-Nitrophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6510 - 4-Nitroquinoline 1-oxide | EPA 8270D | 10186002 | NELAP | LA |
| 6570 - 5-Nitro-o-toluidine | EPA 8270D | 10186002 | NELAP | LA |
| 6115 - 7,12-Dimethylbenz(a)anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5500 - Acenaphthene | EPA 8270D | 10186002 | NELAP | LA |
| 5505 - Acenaphthylene | EPA 8270D | 10186002 | NELAP | LA |
| 5510 - Acetophenone | EPA 8270D | 10186002 | NELAP | LA |
| 5545 - Aniline | EPA 8270D | 10186002 | NELAP | LA |
| 5555 - Anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5560 - Aramite | EPA 8270D | 10186002 | NELAP | LA |
| 7065 - Atrazine | EPA 8270D | 10186002 | NELAP | LA |
| 5562 - Azobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 5570 - Benzaldehyde | EPA 8270D | 10186002 | NELAP | LA |
| 5575 - Benzo(a)anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5580 - Benzo(a)pyrene | EPA 8270D | 10186002 | NELAP | LA |

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Solid Chemical Materials

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|--|-------------|-------------|-------|----|
| 5585 - Benzo(b)fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 5590 - Benzo(g,h,i)perylene | EPA 8270D | 10186002 | NELAP | LA |
| 5600 - Benzo(k)fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 5610 - Benzoic acid | EPA 8270D | 10186002 | NELAP | LA |
| 5630 - Benzyl alcohol | EPA 8270D | 10186002 | NELAP | LA |
| 5670 - Butyl benzyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7180 - Caprolactam | EPA 8270D | 10186002 | NELAP | LA |
| 5680 - Carbazole | EPA 8270D | 10186002 | NELAP | LA |
| 7260 - Chlorobenzilate | EPA 8270D | 10186002 | NELAP | LA |
| 5855 - Chrysene | EPA 8270D | 10186002 | NELAP | LA |
| 4557 - Cyclohexanol | EPA 8270D | 10186002 | NELAP | LA |
| 6065 - Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP) | EPA 8270D | 10186002 | NELAP | LA |
| 5925 - Di-n-butyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 6200 - Di-n-octyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7405 - Diallate | EPA 8270D | 10186002 | NELAP | LA |
| 5895 - Dibenz(a,h) anthracene | EPA 8270D | 10186002 | NELAP | LA |
| 5905 - Dibenzofuran | EPA 8270D | 10186002 | NELAP | LA |
| 6070 - Diethyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 7475 - Dimethoate | EPA 8270D | 10186002 | NELAP | LA |
| 6135 - Dimethyl phthalate | EPA 8270D | 10186002 | NELAP | LA |
| 8625 - Disulfoton | EPA 8270D | 10186002 | NELAP | LA |
| 6260 - Ethyl methanesulfonate | EPA 8270D | 10186002 | NELAP | LA |
| 7580 - Famphur | EPA 8270D | 10186002 | NELAP | LA |
| 6265 - Fluoranthene | EPA 8270D | 10186002 | NELAP | LA |
| 6270 - Fluorene | EPA 8270D | 10186002 | NELAP | LA |
| 6275 - Hexachlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 4835 - Hexachlorobutadiene | EPA 8270D | 10186002 | NELAP | LA |
| 6285 - Hexachlorocyclopentadiene | EPA 8270D | 10186002 | NELAP | LA |
| 4840 - Hexachloroethane | EPA 8270D | 10186002 | NELAP | LA |
| 6295 - Hexachloropropene | EPA 8270D | 10186002 | NELAP | LA |
| 6315 - Indeno(1,2,3-cd) pyrene | EPA 8270D | 10186002 | NELAP | LA |
| 7725 - Isodrin | EPA 8270D | 10186002 | NELAP | LA |
| 6320 - Isophorone | EPA 8270D | 10186002 | NELAP | LA |
| 6325 - Isosafrole | EPA 8270D | 10186002 | NELAP | LA |
| 6345 - Methapyrilene | EPA 8270D | 10186002 | NELAP | LA |
| 4990 - Methyl methacrylate | EPA 8270D | 10186002 | NELAP | LA |
| 6375 - Methyl methanesulfonate | EPA 8270D | 10186002 | NELAP | LA |
| 7825 - Methyl parathion (Parathion, methyl) | EPA 8270D | 10186002 | NELAP | LA |
| 5005 - Naphthalene | EPA 8270D | 10186002 | NELAP | LA |
| 5015 - Nitrobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 6590 - Pentachlorobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 5035 - Pentachloroethane | EPA 8270D | 10186002 | NELAP | LA |
| 6600 - Pentachloronitrobenzene | EPA 8270D | 10186002 | NELAP | LA |
| 6605 - Pentachlorophenol | EPA 8270D | 10186002 | NELAP | LA |
| 6610 - Phenacetin | EPA 8270D | 10186002 | NELAP | LA |
| 6615 - Phenanthrene | EPA 8270D | 10186002 | NELAP | LA |
| 6625 - Phenol | EPA 8270D | 10186002 | NELAP | LA |
| 6665 - Pyrene | EPA 8270D | 10186002 | NELAP | LA |
| 5095 - Pyridine | EPA 8270D | 10186002 | NELAP | LA |
| 6125 - a-a-Dimethylphenethylamine | EPA 8270D | 10186002 | NELAP | LA |
| 5760 - bis(2-Chloroethoxy)methane | EPA 8270D | 10186002 | NELAP | LA |
| 5765 - bis(2-Chloroethyl) ether | EPA 8270D | 10186002 | NELAP | LA |
| 5780 - bis(2-Chloroisopropyl) ether | EPA 8270D | 10186002 | NELAP | LA |
| 5025 - n-Nitroso-di-n-butylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6545 - n-Nitrosodi-n-propylamine | EPA 8270D | 10186002 | NELAP | LA |

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Solid Chemical Materials

| Analyte | Method Name | Method Code | Type | AB |
|---|-------------|-------------|-------|----|
| 6525 - n-Nitrosodiethylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6530 - n-Nitrosodimethylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6535 - n-Nitrosodiphenylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6550 - n-Nitrosomethylethylamine | EPA 8270D | 10186002 | NELAP | LA |
| 6555 - n-Nitrosomorpholine | EPA 8270D | 10186002 | NELAP | LA |
| 6560 - n-Nitrosopiperidine | EPA 8270D | 10186002 | NELAP | LA |
| 6565 - n-Nitrosopyrrolidine | EPA 8270D | 10186002 | NELAP | LA |
| 5090 - n-Propylbenzene | EPA 8270D | 10186002 | NELAP | LA |
| 5500 - Acenaphthene | EPA 8310 | 10187607 | NELAP | LA |
| 5505 - Acenaphthylene | EPA 8310 | 10187607 | NELAP | LA |
| 5555 - Anthracene | EPA 8310 | 10187607 | NELAP | LA |
| 5575 - Benzo(a)anthracene | EPA 8310 | 10187607 | NELAP | LA |
| 5580 - Benzo(a)pyrene | EPA 8310 | 10187607 | NELAP | LA |
| 5585 - Benzo(b)fluoranthene | EPA 8310 | 10187607 | NELAP | LA |
| 5590 - Benzo(g,h,i)perylene | EPA 8310 | 10187607 | NELAP | LA |
| 5600 - Benzo(k)fluoranthene | EPA 8310 | 10187607 | NELAP | LA |
| 5680 - Carbazole | EPA 8310 | 10187607 | NELAP | LA |
| 5855 - Chrysene | EPA 8310 | 10187607 | NELAP | LA |
| 5895 - Dibenz(a,h) anthracene | EPA 8310 | 10187607 | NELAP | LA |
| 6265 - Fluoranthene | EPA 8310 | 10187607 | NELAP | LA |
| 6270 - Fluorene | EPA 8310 | 10187607 | NELAP | LA |
| 6315 - Indeno(1,2,3-cd) pyrene | EPA 8310 | 10187607 | NELAP | LA |
| 5005 - Naphthalene | EPA 8310 | 10187607 | NELAP | LA |
| 6615 - Phenanthrene | EPA 8310 | 10187607 | NELAP | LA |
| 6665 - Pyrene | EPA 8310 | 10187607 | NELAP | LA |
| 6885 - 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8321A | 10189001 | NELAP | LA |
| 6160 - 1,3-Dinitrobenzene (1,3-DNB) | EPA 8321A | 10189001 | NELAP | LA |
| 8655 - 2,4,5-T | EPA 8321A | 10189001 | NELAP | LA |
| 9651 - 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8321A | 10189001 | NELAP | LA |
| 8545 - 2,4-D | EPA 8321A | 10189001 | NELAP | LA |
| 8560 - 2,4-DB | EPA 8321A | 10189001 | NELAP | LA |
| 5882 - 2,4-Diamino-6-nitrotoluene | EPA 8321A | 10189001 | NELAP | LA |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8321A | 10189001 | NELAP | LA |
| 6181 - 2,6-Diamino-4-nitrotoluene | EPA 8321A | 10189001 | NELAP | LA |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8321A | 10189001 | NELAP | LA |
| 9303 - 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8321A | 10189001 | NELAP | LA |
| 9507 - 2-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA |
| 6150 - 3,5-Dinitroaniline | EPA 8321A | 10189001 | NELAP | LA |
| 9510 - 3-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA |
| 9306 - 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8321A | 10189001 | NELAP | LA |
| 9513 - 4-Nitrotoluene | EPA 8321A | 10189001 | NELAP | LA |
| 8555 - Dalapon | EPA 8321A | 10189001 | NELAP | LA |
| 8595 - Dicamba | EPA 8321A | 10189001 | NELAP | LA |
| 8605 - Dichloroprop (Dichloroprop) | EPA 8321A | 10189001 | NELAP | LA |
| 8620 - Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8321A | 10189001 | NELAP | LA |
| 7775 - MCPA | EPA 8321A | 10189001 | NELAP | LA |
| 7780 - MCPP | EPA 8321A | 10189001 | NELAP | LA |
| 6415 - Methyl-2,4,6-trinitrophenylnitramine (tetryl) | EPA 8321A | 10189001 | NELAP | LA |
| 5015 - Nitrobenzene | EPA 8321A | 10189001 | NELAP | LA |
| 6485 - Nitroglycerin | EPA 8321A | 10189001 | NELAP | LA |
| 9522 - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8321A | 10189001 | NELAP | LA |

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Solid Chemical Materials

| Analyte | Method Name | Method Code | Type | AB |
|--|-----------------|-------------|-------|----|
| 9558 - Pentaerythritoltetranitrate | EPA 8321A | 10189001 | NELAP | LA |
| 9432 - RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8321A | 10189001 | NELAP | LA |
| 8650 - Silvex (2,4,5-TP) | EPA 8321A | 10189001 | NELAP | LA |
| 1645 - Total Cyanide | EPA 9012A | 10193405 | NELAP | LA |
| 1900 - pH | EPA 9040B | 10197203 | NELAP | LA |
| 1900 - pH | EPA 9045C | 10198400 | NELAP | LA |
| 1610 - Conductivity | EPA 9050A | 10198808 | NELAP | LA |
| 1540 - Bromide | EPA 9056A | 10199607 | NELAP | LA |
| 1575 - Chloride | EPA 9056A | 10199607 | NELAP | LA |
| 1730 - Fluoride | EPA 9056A | 10199607 | NELAP | LA |
| 1810 - Nitrate as N | EPA 9056A | 10199607 | NELAP | LA |
| 1840 - Nitrite as N | EPA 9056A | 10199607 | NELAP | LA |
| 100511 - Orthophosphate | EPA 9056A | 10199607 | NELAP | LA |
| 1870 - Orthophosphate as P | EPA 9056A | 10199607 | NELAP | LA |
| 2000 - Sulfate | EPA 9056A | 10199607 | NELAP | LA |
| 1560 - Cation exchange capacity | EPA 9081 | 10203404 | NELAP | LA |
| 2830 - Gross-alpha | EPA 9310 | 10208205 | NELAP | LA |
| 2840 - Gross-beta | EPA 9310 | 10208205 | NELAP | LA |
| 100210 - Alpha Emitting Radium Isotopes | EPA 9315 | 10208409 | NELAP | LA |
| 2975 - Total radium | EPA 9315 | 10208409 | NELAP | LA |
| 2970 - Radium-228 | EPA 9320 | 10208603 | NELAP | LA |
| 1780 - Ignitability | EPA 1010A | 10234807 | NELAP | LA |
| 2830 - Gross-alpha | EPA 900.0 (GPC) | 10242601 | NELAP | LA |
| 2840 - Gross-beta | EPA 900.0 (GPC) | 10242601 | NELAP | LA |
| 1635 - Cyanide | EPA 9010C | 10243002 | NELAP | LA |
| 1635 - Cyanide | EPA 9012B | 10243206 | NELAP | LA |
| 1645 - Total Cyanide | EPA 9012B | 10243206 | NELAP | LA |
| 2975 - Total radium | EPA 903.0 (GPC) | 10244005 | NELAP | LA |
| 1900 - pH | EPA 9040C | 10244403 | NELAP | LA |
| 1900 - pH | EPA 9045D | 10244607 | NELAP | LA |
| 2040 - Total Organic Carbon | EPA 9060A | 10244801 | NELAP | LA |
| 1745 - Free liquid | EPA 9095B | 10245600 | NELAP | LA |
| 1406 - Purge and trap for aqueous phase samples | EPA 5030C | 10284603 | NELAP | LA |
| 100017 - Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples | EPA 5035A | 10284807 | NELAP | LA |
| 5105 - 1,1,1,2-Tetrachloroethane | EPA 8260C | 10307003 | NELAP | LA |
| 5160 - 1,1,1-Trichloroethane | EPA 8260C | 10307003 | NELAP | LA |
| 5110 - 1,1,2,2-Tetrachloroethane | EPA 8260C | 10307003 | NELAP | LA |
| 5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260C | 10307003 | NELAP | LA |
| 5165 - 1,1,2-Trichloroethane | EPA 8260C | 10307003 | NELAP | LA |
| 4630 - 1,1-Dichloroethane | EPA 8260C | 10307003 | NELAP | LA |
| 4640 - 1,1-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA |
| 4670 - 1,1-Dichloropropene | EPA 8260C | 10307003 | NELAP | LA |
| 5150 - 1,2,3-Trichlorobenzene | EPA 8260C | 10307003 | NELAP | LA |
| 5180 - 1,2,3-Trichloropropane | EPA 8260C | 10307003 | NELAP | LA |
| 5155 - 1,2,4-Trichlorobenzene | EPA 8260C | 10307003 | NELAP | LA |
| 5210 - 1,2,4-Trimethylbenzene | EPA 8260C | 10307003 | NELAP | LA |
| 4570 - 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260C | 10307003 | NELAP | LA |
| 4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260C | 10307003 | NELAP | LA |
| 4610 - 1,2-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.

| Solid Chemical Materials | | | | | |
|---|-------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 4635 - 1,2-Dichloroethane (Ethylene dichloride) | EPA 8260C | 10307003 | NELAP | LA | |
| 4655 - 1,2-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 5215 - 1,3,5-Trimethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4615 - 1,3-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4660 - 1,3-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 4620 - 1,4-Dichlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4735 - 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4665 - 2,2-Dichloropropane | EPA 8260C | 10307003 | NELAP | LA | |
| 4410 - 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260C | 10307003 | NELAP | LA | |
| 4500 - 2-Chloroethyl vinyl ether | EPA 8260C | 10307003 | NELAP | LA | |
| 4535 - 2-Chlorotoluene | EPA 8260C | 10307003 | NELAP | LA | |
| 4860 - 2-Hexanone | EPA 8260C | 10307003 | NELAP | LA | |
| 4540 - 4-Chlorotoluene | EPA 8260C | 10307003 | NELAP | LA | |
| 4910 - 4-Isopropyltoluene (p-Cymene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4995 - 4-Methyl-2-pentanone (MIBK) | EPA 8260C | 10307003 | NELAP | LA | |
| 4315 - Acetone | EPA 8260C | 10307003 | NELAP | LA | |
| 4320 - Acetonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4325 - Acrolein (Propenal) | EPA 8260C | 10307003 | NELAP | LA | |
| 4340 - Acrylonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4355 - Allyl chloride (3-Chloropropene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4375 - Benzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4385 - Bromobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4390 - Bromochloromethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4395 - Bromodichloromethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4400 - Bromoform | EPA 8260C | 10307003 | NELAP | LA | |
| 4450 - Carbon disulfide | EPA 8260C | 10307003 | NELAP | LA | |
| 4455 - Carbon tetrachloride | EPA 8260C | 10307003 | NELAP | LA | |
| 4475 - Chlorobenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4575 - Chlorodibromomethane | EPA 8260C | 10307003 | NELAP | LA | |
| 4485 - Chloroethane (Ethyl chloride) | EPA 8260C | 10307003 | NELAP | LA | |
| 4505 - Chloroform | EPA 8260C | 10307003 | NELAP | LA | |
| 4525 - Chloroprene (2-Chloro-1,3-butadiene) | EPA 8260C | 10307003 | NELAP | LA | |
| 4595 - Dibromomethane (Methylene bromide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4625 - Dichlorodifluoromethane (Freon-12) | EPA 8260C | 10307003 | NELAP | LA | |
| 4725 - Diethyl ether | EPA 8260C | 10307003 | NELAP | LA | |
| 4755 - Ethyl acetate | EPA 8260C | 10307003 | NELAP | LA | |
| 4810 - Ethyl methacrylate | EPA 8260C | 10307003 | NELAP | LA | |
| 4765 - Ethylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4835 - Hexachlorobutadiene | EPA 8260C | 10307003 | NELAP | LA | |
| 4870 - Iodomethane (Methyl iodide) | EPA 8260C | 10307003 | NELAP | LA | |
| 4875 - Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260C | 10307003 | NELAP | LA | |
| 4900 - Isopropylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4925 - Methacrylonitrile | EPA 8260C | 10307003 | NELAP | LA | |
| 4950 - Methyl bromide (Bromomethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 4960 - Methyl chloride (Chloromethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 4990 - Methyl methacrylate | EPA 8260C | 10307003 | NELAP | LA | |
| 5000 - Methyl tert-butyl ether (MTBE) | EPA 8260C | 10307003 | NELAP | LA | |
| 4975 - Methylene chloride (Dichloromethane) | EPA 8260C | 10307003 | NELAP | LA | |
| 5005 - Naphthalene | EPA 8260C | 10307003 | NELAP | LA | |
| 5035 - Pentachloroethane | EPA 8260C | 10307003 | NELAP | LA | |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

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| Solid Chemical Materials | | | | | |
|---|---------------------------|-------------|-------|----|--|
| Analyte | Method Name | Method Code | Type | AB | |
| 5080 - Propionitrile (Ethyl cyanide) | EPA 8260C | 10307003 | NELAP | LA | |
| 5100 - Styrene | EPA 8260C | 10307003 | NELAP | LA | |
| 5115 - Tetrachloroethylene (Perchloroethylene) | EPA 8260C | 10307003 | NELAP | LA | |
| 5140 - Toluene | EPA 8260C | 10307003 | NELAP | LA | |
| 5170 - Trichloroethene (Trichloroethylene) | EPA 8260C | 10307003 | NELAP | LA | |
| 5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | EPA 8260C | 10307003 | NELAP | LA | |
| 5225 - Vinyl acetate | EPA 8260C | 10307003 | NELAP | LA | |
| 5235 - Vinyl chloride | EPA 8260C | 10307003 | NELAP | LA | |
| 5260 - Xylene (total) | EPA 8260C | 10307003 | NELAP | LA | |
| 4645 - cis-1,2-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4680 - cis-1,3-Dichloropropene | EPA 8260C | 10307003 | NELAP | LA | |
| 5240 - m+p-xylene | EPA 8260C | 10307003 | NELAP | LA | |
| 5090 - n-Propylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 5250 - o-Xylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4440 - sec-Butylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4445 - tert-Butylbenzene | EPA 8260C | 10307003 | NELAP | LA | |
| 4700 - trans-1,2-Dichloroethylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4685 - trans-1,3-Dichloropropylene | EPA 8260C | 10307003 | NELAP | LA | |
| 4605 - trans-1,4-Dichloro-2-butene | EPA 8260C | 10307003 | NELAP | LA | |
| 6885 - 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8330B | 10308006 | NELAP | LA | |
| 6160 - 1,3-Dinitrobenzene (1,3-DNB) | EPA 8330B | 10308006 | NELAP | LA | |
| 9651 - 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 6185 - 2,4-Dinitrotoluene (2,4-DNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 6190 - 2,6-Dinitrotoluene (2,6-DNT) | EPA 8330B | 10308006 | NELAP | LA | |
| 9303 - 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8330B | 10308006 | NELAP | LA | |
| 9507 - 2-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 9510 - 3-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 9306 - 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8330B | 10308006 | NELAP | LA | |
| 9513 - 4-Nitrotoluene | EPA 8330B | 10308006 | NELAP | LA | |
| 6415 - Methyl-2,4,6-trinitrophenylamine (tetryl) | EPA 8330B | 10308006 | NELAP | LA | |
| 5015 - Nitrobenzene | EPA 8330B | 10308006 | NELAP | LA | |
| 6485 - Nitroglycerin | EPA 8330B | 10308006 | NELAP | LA | |
| 9522 - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8330B | 10308006 | NELAP | LA | |
| 9432 - RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8330B | 10308006 | NELAP | LA | |
| 2826 - Gamma Emitters | EPA 901.1 | 10308608 | NELAP | LA | |
| 2970 - Radium-228 | EPA 904.0 | 10309805 | NELAP | LA | |
| 3005 - Strontium-90 | EPA 905.0 | 10310006 | NELAP | LA | |
| 3030 - Tritium | EPA 906.0 | 10310200 | NELAP | LA | |
| 2755 - Americium-241 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2758 - Antimony 124 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1006 - Antimony 125 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2765 - Barium-133 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 1021 - Beryllium-7 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2772 - Bismuth-212 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2773 - Bismuth-214 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2793 - Cerium-139 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2794 - Cerium-141 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2795 - Cerium-144 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |
| 2800 - Cesium-134 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA | |

TestAmerica Laboratories Inc
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Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

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Solid Chemical Materials

| Analyte | Method Name | Method Code | Type | AB |
|--------------------------------------|----------------------------------|-------------|-------|----|
| 2805 - Cesium-137 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2812 - Cobalt-57 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2813 - Cobalt-58 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2815 - Cobalt-60 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1068 - Europium-152 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1069 - Europium-154 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2826 - Gamma Emitters | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2900 - Lead-210 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2902 - Lead-212 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2903 - Lead-214 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2905 - Manganese-54 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2908 - Mercury-203 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2918 - Niobium-94 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1107 - Niobium-95 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 100586 - Photon Emitters | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2952 - Protactinium-234 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2960 - Radium-224 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2965 - Radium-226 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2970 - Radium-228 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1136 - Ruthenium-106 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2989 - Scandium-46 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1156 - Sodium-22 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1164 - Strontium-85 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1166 - Thallium-208 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3031 - Thorium-227 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 1171 - Thorium-228 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3032 - Thorium-231 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3028 - Thorium-234 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2942 - Tin-113 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3037 - Uranium-235 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3038 - Uranium-238 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3067 - Yttrium-88 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3070 - Zinc-65 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 3072 - Zirconium-95 | HASL 300 Ga-01-R, 28th ED | 90000401 | NELAP | LA |
| 2930 - Plutonium-238 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 2932 - Plutonium-239 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3036 - Uranium-234 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3038 - Uranium-238 | HASL 300 A-01-R, 28th ED | 90000605 | NELAP | LA |
| 3027 - Thorium-230 | HASL 300 G-01, 28th ED | 90002407 | NELAP | LA |
| 2995 - Strontium-89 | HASL 300 Sr-01-RC (GPC), 28th ED | 90008405 | NELAP | LA |
| 3005 - Strontium-90 | HASL 300 Sr-02-RC (GPC), 28th ED | 90009204 | NELAP | LA |
| 3005 - Strontium-90 | HASL 300 Sr-03-RC, 28th ED | 90009806 | NELAP | LA |
| 9408 - Gasoline range organics (GRO) | IDNR OA-1 | 90016403 | NELAP | LA |
| 9369 - Diesel range organics (DRO) | IDNR OA-2 | 90016607 | NELAP | LA |

Biological Tissue

| Analyte | Method Name | Method Code | Type | AB |
|---------|-------------|-------------|------|------|
| NONE | NONE | NONE | NONE | NONE |

TestAmerica Laboratories Inc
Issue Date: July 1, 2014

Certificate Number: 04080

AI Number: 106151
Expiration Date: June 30, 2015

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.



Department of Health, Bureau of Public Health Laboratories
This is to certify that

E87689

TESTAMERICA ST, LOUIS
13715 RIDER TRAIL NORTH
EARTH CITY, MO 63045


has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER -
RADIOCHEMISTRY, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER -
METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - RADIOCHEMISTRY, NON-POTABLE WATER -
VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS -
PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS,
SOLID AND CHEMICAL MATERIALS - RADIOCHEMISTRY, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: October 10, 2014 Expiration Date: June 30, 2015




Carina Blackmore, DVM, PhD, Dipl. ACVPM, CPM
Chief, Bureau of Public Health Laboratories
DH Form 1607, 7/04
NON-TRANSFERABLE E87689-39-10/10/2014
Supersedes all previously issued certificates



Rick Scott
Governor



John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 1 of 29

Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045
Matrix: Drinking Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------------|--------------------|----------------|
| 1,1,1,2-Tetrachloroethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,1,1-Trichloroethane | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,1,2,2-Tetrachloroethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,1,2-Trichloroethane | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,1-Dichloroethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,1-Dichloroethylene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,1-Dichloropropene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2,3-Trichlorobenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2,3-Trichloropropane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2,4-Trichlorobenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2,4-Trimethylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2-Dibromo-3-chloropropane (DBCP) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,2-Dichlorobenzene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,2-Dichloroethane | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,2-Dichloropropane | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 1,3,5-Trimethylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,3-Dichlorobenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,3-Dichloropropane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 1,4-Dichlorobenzene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| 2,2-Dichloropropane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 2-Butanone (Methyl ethyl ketone, MEK) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 12/10/2008 |
| 2-Chlorotoluene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 2-Hexanone | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 12/10/2008 |
| 4-Chlorotoluene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 4-Isopropyltoluene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| 4-Methyl-2-pentanone (MIBK) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 12/10/2008 |
| Acetone | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 12/10/2008 |
| Benzene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Bromobenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Bromochloromethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Bromodichloromethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Bromoform | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Carbon disulfide | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 12/10/2008 |
| Carbon tetrachloride | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Chlorobenzene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 10/10/2014

Expiration Date: 6/30/2015



Rick Scott
 Governor



John H. Armstrong, MD, FACS
 State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
 TestAmerica St. Louis
 13715 Rider Trail North
 Earth City, MO 63045
 Matrix: Drinking Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|--------------|-----------------------------------|--------------------|----------------|
| Chloroethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Chloroform | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| cis-1,2-Dichloroethylene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| cis-1,3-Dichloropropene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Dibromochloromethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Dibromomethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Dichlorodifluoromethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Dichloromethane (DCM, Methylene chloride) | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Ethylbenzene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Gamma emitters | EPA 901.1 | Radiochemistry | NELAP | 1/9/2014 |
| Gross-alpha | EPA 900.0 | Radiochemistry | NELAP | 12/10/2008 |
| Hexachlorobutadiene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Isopropylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Methyl bromide (Bromomethane) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Methyl chloride (Chloromethane) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Methyl tert-butyl ether (MTBE) | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Naphthalene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| n-Butylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| n-Propylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Radioactive cesium | EPA 901.1 | Radiochemistry | NELAP | 12/10/2008 |
| Radium-226 | EPA 903.0 | Radiochemistry | NELAP | 12/10/2008 |
| Radium-228 | EPA 904.0 | Radiochemistry | NELAP | 12/10/2008 |
| sec-Butylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Strontium-90 | DOE Sr-02 | Radiochemistry | NELAP | 12/10/2008 |
| Strontium-90 | DOE Sr-03-RC | Radiochemistry | NELAP | 12/10/2008 |
| Strontium-90 | EPA 905.0 | Radiochemistry | NELAP | 12/10/2008 |
| Styrene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| tert-Butylbenzene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Tetrachloroethylene (Perchloroethylene) | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Toluene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| trans-1,2-Dichloroethylene | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| trans-1,3-Dichloropropene | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Trichloroethene (Trichloroethylene) | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |
| Trichlorofluoromethane | EPA 524.2 | Group II Unregulated Contaminants | NELAP | 7/17/2003 |
| Tritium | EPA 906.0 | Radiochemistry | NELAP | 12/10/2008 |
| Vinyl chloride | EPA 524.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |

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Issue Date: 10/10/2014

Expiration Date: 6/30/2015

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Drinking Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|----------------|-------------|------------------------------|--------------------|----------------|
| Xylene (total) | EPA 824.2 | Other Regulated Contaminants | NELAP | 7/17/2003 |

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Issue Date: 10/10/2014

Expiration Date: 6/30/2015



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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|----------------------|--------------------|----------------|
| 1,1,1,2-Tetrachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,1-Trichloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,1-Trichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2,2-Tetrachloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2,2-Tetrachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2-Trichloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2-Trichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethylene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,3-Trichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,3-Trichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,4,5-Tetrachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2,4-Trichlorobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2,4-Trichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,4-Trichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2,4-Trimethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2-Dichloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichloropropane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3,5-Trimethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dichlorobenzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3-Dichlorobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|-----------------------------------|-------------|-----------------------------|--------------------|----------------|
| 1,3-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3-Dinitrobenzene (1,3-DNB) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dinitrobenzene (1,3-DNB) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,4-Naphthoquinone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1-Naphthylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,2-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2,3,4,6-Tetrachlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,5-T | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 2,4,5-Trichlorophenol | EPA 8041 | Extractable Organics | NELAP | 12/10/2008 |
| 2,4,5-Trichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trichlorophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trichlorophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-D | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 2,4-DB | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 2,4-Dichlorophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dichlorophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dimethylphenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dimethylphenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dimethylphenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Diamino-4-nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |

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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---------------------------------------|-------------|----------------------|--------------------|----------------|
| 2,6-Dichlorophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Chloroethyl vinyl ether | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Chloroethyl vinyl ether | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Chloronaphthalene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chloronaphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorotoluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Hexanone | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Methyl-4,6-dinitrophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methyl-4,6-dinitrophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methyl-4,6-dinitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methylnaphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methylphenol (o-Cresol) | EPA 8941 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methylphenol (o-Cresol) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Naphthylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 3,3'-Dichlorobenzidine | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 3,3'-Dichlorobenzidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3,3'-Dimethylbenzidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3,5-Dinitroaniline | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 3/4-Methylphenols (m/p-Cresols) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---------------------------------------|-------------|-----------------------------|--------------------|----------------|
| 3-Methylcholanthrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 4,4'-DDD | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDD | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDE | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDE | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDT | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDT | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Aminobiphenyl | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Bromophenyl phenyl ether | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Bromophenyl phenyl ether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloro-3-methylphenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloro-3-methylphenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloro-3-methylphenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chlorophenyl phenylether | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chlorophenyl phenylether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chlorotoluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2003 |
| 4-Methyl-2-pentanone (MIBK) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 4-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 7,12-Dimethyl(benz(a) anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n,n-Dimethylphenethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthylene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthylene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| Accenaphthylene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Acetone | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acetonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acetophenone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acetylene | RSK-175 | Volatile Organics | NELAP | 7/1/2013 |
| Acrolein (Propenal) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Acrolein (Propenal) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acrylonitrile | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Acrylonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Aldrin | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aldrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Alkalinity as CaCO3 | EPA 310.1 | General Chemistry | NELAP | 7/1/2013 |
| Alkalinity as CaCO3 | SM 2320 B | General Chemistry | NELAP | 7/1/2013 |
| Allyl chloride (3-Chloropropene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| alpha-Chlordane | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aluminum | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Aluminum | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Aluminum | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Aluminum | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Ammonia as N | EPA 350.1 | General Chemistry | NELAP | 7/1/2013 |
| Aniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Anthracene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Antimony | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Antimony | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Antimony | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Antimony | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Aramite | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Aroclor-1016 (PCB-1016) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1016 (PCB-1016) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1221 (PCB-1221) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1221 (PCB-1221) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1232 (PCB-1232) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 10/10/2014

Expiration Date: 6/30/2015



Rick Scott
 Governor



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Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
 TestAmerica St. Louis
 13715 Rider Trail North
 Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|-------------------------|-------------|-----------------------------|--------------------|----------------|
| Aroclor-1232 (PCB-1232) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1242 (PCB-1242) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1242 (PCB-1242) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1248 (PCB-1248) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1248 (PCB-1248) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1254 (PCB-1254) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1254 (PCB-1254) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1260 (PCB-1260) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1260 (PCB-1260) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Arsenic | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Arsenic | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Arsenic | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Arsenic | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Benzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Benzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Benzo(a)anthracene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)pyrene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(b)fluoranthene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(b)fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(b)fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(g,h,i)perylene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(g,h,i)perylene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(g,h,i)perylene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(k)fluoranthene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(k)fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(k)fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzoic acid | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzyl alcohol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| Beryllium | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Beryllium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Beryllium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Beryllium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| beta-BHC (beta-Hexachlorocyclohexane) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| beta-BHC (beta-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Biochemical oxygen demand | EPA 405.1 | General Chemistry | NELAP | 7/1/2013 |
| Biochemical oxygen demand | SM 5210 B | General Chemistry | NELAP | 7/1/2013 |
| bis(2-Chloroethoxy)methane | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroethoxy)methane | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroethyl) ether | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroethyl) ether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane)) | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Ethylhexyl) phthalate (DEHP) | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Ethylhexyl) phthalate (DEHP) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Boron | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Boron | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Boron | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Bromide | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Bromide | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Bromobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromochloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromodichloromethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Bromodichloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromoform | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Bromoform | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Butyl benzyl phthalate | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Butyl benzyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Cadmium | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Cadmium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Cadmium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Cadmium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Calcium | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Calcium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Calcium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Carbazole | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---------------------------|-------------|-----------------------------|--------------------|----------------|
| Carbon disulfide | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Carbon tetrachloride | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Carbon tetrachloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chemical oxygen demand | EPA 410.4 | General Chemistry | NELAP | 7/1/2013 |
| Chlordane (tech.) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Chlordane (tech.) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Chloride | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Chloride | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Chlorobenzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Chlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroform | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroform | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroprene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chromium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Chromium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Chromium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Chromium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Chromium VI | EPA 7196 | Metals | NELAP | 7/1/2013 |
| Chrysene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Chrysene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Chrysene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| cis-1,2-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| cis-1,3-Dichloropropene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| cis-1,3-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| cis-1,4-Dichloro-2-butene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Cobalt | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Cobalt | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Cobalt | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Cobalt | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Conductivity | EPA 120.1 | General Chemistry | NELAP | 7/1/2013 |
| Conductivity | EPA 9050 | General Chemistry | NELAP | 7/1/2003 |
| Copper | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Copper | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Copper | EPA 6010 | Metals | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| Copper | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Dalapon | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| delta-BHC | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| delta-BHC | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dibenz(a,h)anthracene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Dibenz(a,h)anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dibenz(a,h)anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Dibenzofuran | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dibromochloromethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Dibromochloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Dibromomethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Disamba | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dichlorodifluoromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Dichloroprop (Dichloroprop) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dieldrin | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dieldrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Diesel range organics (DRO) | EPA 8015 | Extractable Organics | NELAP | 7/1/2013 |
| Diethyl ether | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Diethyl phthalate | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Diethyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dimethyl phthalate | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Dimethyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-butyl phthalate | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-butyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-octyl phthalate | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-octyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan I | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan I | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan II | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan II | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan sulfate | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan sulfate | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |

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Issue Date: 10/10/2014

Expiration Date: 6/30/2015



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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| Endrin aldehyde | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin aldehyde | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin ketone | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Ethane | RSK-175 | Volatile Organics | NELAP | 7/1/2013 |
| Ethyl acetate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Ethyl methacrylate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Ethylbenzene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Ethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Ethylene | RSK-175 | Volatile Organics | NELAP | 7/1/2013 |
| Fluoranthene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Fluorene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Fluorene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Fluorene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Fluoride | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Fluoride | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Gamma emitters | EPA 901.1 | Radiochemistry | NELAP | 7/1/2013 |
| gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| gamma-Chlordane | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Gasoline range organics (GRO) | EPA 8015 | Volatile Organics | NELAP | 7/1/2013 |
| Gross-alpha | EPA 900.0 | Radiochemistry | NELAP | 7/1/2013 |
| Gross-alpha | EPA 9310 | Radiochemistry | NELAP | 7/1/2013 |
| Gross-beta | EPA 900.0 | Radiochemistry | NELAP | 7/1/2013 |
| Gross-beta | EPA 9310 | Radiochemistry | NELAP | 7/1/2013 |
| Heptachlor | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Heptachlor | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Heptachlor epoxide | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Heptachlor epoxide | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Hexachlorobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorobutadiene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorobutadiene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Hexachlorobutadiene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|---------------------------|--------------------|----------------|
| Hexachlorocyclopentadiene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorocyclopentadiene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachloroethane | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachloroethane | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachloropropene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Ignitability | EPA 1010 | General Chemistry | NELAP | 7/1/2013 |
| Indeno(1,2,3-cd)pyrene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Indeno(1,2,3-cd)pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Indeno(1,2,3-cd)pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Iodomethane (Methyl iodide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Iron | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Iron | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Iron | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Isodrin | EPA 8270 | Extractable Organics | NELAP | 7/1/2003 |
| Isophorone | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Isophorone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Isopropylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Isosafrole | EPA 8270 | Extractable Organics | NELAP | 6/25/2013 |
| Lead | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Lead | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Lead | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Lead | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Lithium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| m+p-Xylenes | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Magnesium | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Magnesium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Magnesium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Magnesium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Manganese | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Manganese | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Manganese | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Manganese | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Mercury | EPA 245.1 | Metals | NELAP | 7/1/2013 |
| Mercury | EPA 7470 | Metals | NELAP | 7/1/2013 |
| Methacrylonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |

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E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--------------------------------------|-------------|-----------------------------|--------------------|----------------|
| Methane | RSK-175 | Volatile Organics | NELAP | 7/1/2013 |
| Methapyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Methoxychlor | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Methyl bromide (Bromomethane) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl bromide (Bromomethane) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl chloride (Chloromethane) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl chloride (Chloromethane) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl methacrylate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl parathion (Parathion, methyl) | EPA 8270 | Extractable Organics | NELAP | 7/1/2003 |
| Methyl tert-butyl ether (MTBE) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methylene chloride | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Methylene chloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Molybdenum | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Molybdenum | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Molybdenum | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Molybdenum | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Naphthalene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Naphthalene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Naphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Naphthalene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| n-Butyl alcohol | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| n-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Nickel | EPA 200.7 | General Chemistry, Metals | NELAP | 7/1/2013 |
| Nickel | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Nickel | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Nickel | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Nitrate | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Nitrate as N | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Nitrate-nitrite | EPA 353.1 | General Chemistry | NELAP | 7/1/2013 |
| Nitrite | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Nitrite as N | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Nitroglycerin | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Nitroglycerin | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 10/10/2014

Expiration Date: 6/30/2015

Rick Scott
Governor



John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|---------------------------|----------------------|--------------------|----------------|
| n-Nitrosodiethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodimethylamine | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodimethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitroso-di-n-butylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodi-n-propylamine | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodi-n-propylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodiphenylamine | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodiphenylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosomethylethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosomorpholine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosopiperidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosopyrrolidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Propylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2003 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Oil & Grease | EPA 1664A | General Chemistry | NELAP | 7/1/2013 |
| Orthophosphate as P | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Orthophosphate as P | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| o-Toluidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2003 |
| o-Xylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Pentachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2003 |
| Pentachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Pentachloronitrobenzene (Quinzozene) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pentachlorophenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Pentachlorophenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| Pentachlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pentaerythritoltetrasulfate (PETN) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Perchlorate | EPA 314.0 | General Chemistry | NELAP | 7/1/2013 |
| Perchlorate | EPA 6850 | General Chemistry | NELAP | 7/1/2013 |
| pH | EPA 150.1 | General Chemistry | NELAP | 7/1/2013 |
| pH | EPA 9040 | General Chemistry | NELAP | 7/1/2003 |
| pH | SM 4500-H ⁺ -B | General Chemistry | NELAP | 7/1/2013 |
| Phenacetin | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Phenanthrene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Phenanthrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-----------------|-----------------------------|--------------------|----------------|
| Phenanthrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Phenol | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Phenol | EPA 8041 | Extractable Organics | NELAP | 7/1/2013 |
| Phenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| p-Isopropyltoluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2003 |
| Potassium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Potassium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Potassium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Propionitrile (Ethyl cyanide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Propylene glycol | EPA 8015 | Volatile Organics | NELAP | 2/26/2013 |
| Pyrene | EPA 625 | Extractable Organics | NELAP | 7/1/2013 |
| Pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Pyridine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Radium-226 | EPA 903.0 | Radiochemistry | NELAP | 7/1/2013 |
| Radium-228 | EPA 904.0 | Radiochemistry | NELAP | 7/1/2013 |
| Radium-228 | EPA 9320 | Radiochemistry | NELAP | 7/1/2013 |
| RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Reactive cyanide | Sec. 7.3 SW-846 | General Chemistry | NELAP | 7/24/2006 |
| Reactive sulfide | Sec. 7.3 SW-846 | General Chemistry | NELAP | 7/1/2013 |
| Residue-filterable (TDS) | EPA 160.1 | General Chemistry | NELAP | 7/1/2013 |
| Residue-filterable (TDS) | SM 2540 C | General Chemistry | NELAP | 7/1/2013 |
| Residue-nonfilterable (TSS) | EPA 160.2 | General Chemistry | NELAP | 7/1/2013 |
| Residue-nonfilterable (TSS) | SM 2540 D | General Chemistry | NELAP | 7/1/2013 |
| Residue-total | EPA 160.3 | General Chemistry | NELAP | 7/1/2013 |
| sec-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Selenium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Selenium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Selenium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Selenium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Silvex (2,4,5-TP) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |

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Issue Date: 10/10/2014

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State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|---------------|----------------------|--------------------|----------------|
| Sodium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Sodium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Sodium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Strontium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Strontium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Strontium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Strontium-90 | DOE Sr-02 | Radiochemistry | NELAP | 7/1/2013 |
| Strontium-90 | DOE Sr-03-RC | Radiochemistry | NELAP | 7/1/2013 |
| Strontium-90 | EPA 905.0 | Radiochemistry | NELAP | 7/1/2013 |
| Styrene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Sulfate | EPA 300.0 | General Chemistry | NELAP | 7/1/2013 |
| Sulfate | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Sulfide | EPA 376.1 | General Chemistry | NELAP | 7/1/2013 |
| Sulfide | EPA 9030/9034 | General Chemistry | NELAP | 6/25/2013 |
| Synthetic Precipitation Leaching Procedure | EPA 1312 | General Chemistry | NELAP | 7/24/2006 |
| tert-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Tetrachloroethylene (Perchloroethylene) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Tetrachloroethylene (Perchloroethylene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Tetryl (methyl-2,4,6-trinitrophenyltriamine) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Tetryl (methyl-2,4,6-trinitrophenyltriamine) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Thallium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Thallium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Thallium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Thallium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Thorium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Thorium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Tin | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Tin | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Tin | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Titanium | EPA 200.7 | Metals | NELAP | 7/1/2013 |
| Titanium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Titanium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Toluene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Toluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Total cyanide | EPA 335.4 | General Chemistry | NELAP | 2/26/2013 |
| Total cyanide | EPA 9010 | General Chemistry | NELAP | 7/1/2013 |

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Rick Scott
Governor



John H. Armstrong, MD, FACB
State Surgeon General & Secretary

Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Non-Potable Water

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|-----------------------------|--------------------|----------------|
| Total cyanide | EPA 9012 | General Chemistry | NELAP | 7/1/2013 |
| Total organic carbon | EPA 415.1 | General Chemistry | NELAP | 7/1/2013 |
| Total organic carbon | EPA 9060 | General Chemistry | NELAP | 7/1/2013 |
| Total organic halides (TOX) | EPA 9020 | General Chemistry | NELAP | 7/1/2013 |
| Total radium | EPA 9315 | Radiochemistry | NELAP | 7/1/2013 |
| Toxaphene (Chlorinated camphene) | EPA 608 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Toxaphene (Chlorinated camphene) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Toxicity Characteristic Leaching Procedure | EPA 1311 | General Chemistry | NELAP | 7/24/2006 |
| trans-1,2-Dichloroethylene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,2-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,3-Dichloropropene | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,3-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,4-Dichloro-2-butene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Trichloroethene (Trichloroethylene) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Trichloroethene (Trichloroethylene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Trichlorofluoromethane | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Trichlorofluoromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Tritium | EPA 906.0 | Radiochemistry | NELAP | 7/1/2013 |
| Uranium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Uranium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 200.7 | General Chemistry,Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Vinyl acetate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Vinyl chloride | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Vinyl chloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Xylene (total) | EPA 624 | Volatile Organics | NELAP | 7/1/2013 |
| Xylene (total) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Zinc | EPA 200.7 | General Chemistry,Metals | NELAP | 7/1/2013 |
| Zinc | EPA 200.8 | Metals | NELAP | 7/1/2013 |
| Zinc | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Zinc | EPA 6020 | Metals | NELAP | 7/1/2013 |

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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| 1,1,1,2-Tetrachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,1-Trichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2,2-Tetrachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1,2-Trichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,1-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,3-Trichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,3-Trichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,4,5-Tetrachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2,4-Trichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2,4-Trichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2,4-Trimethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dibromo-3-chloropropane (DBCP) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dibromoethane (EDB, Ethylene dibromide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,2-Dichloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,2-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3,5-Trimethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,3-Dinitrobenzene (1,3-DNB) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 1,3-Dinitrobenzene (1,3-DNB) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,4-Dichlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1,4-Dioxane (1,4-Diethyleneoxide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 1,4-Naphthoquinone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 1-Naphthylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,2-Dichloropropane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2,3,4,6-Tetrachlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,5-T | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |

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State Surgeon General & Secretary

Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---------------------------------------|-------------|-----------------------------|--------------------|----------------|
| 2,4,5-Trichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-D | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 2,4-DB | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 2,4-Diamino-6-nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dimethylphenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,4-Dinitrotoluene (2,4-DNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Diamino-4-nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dichlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2,6-Dinitrotoluene (2,6-DNT) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Amino-4,6-dinitrotoluene (2-am-dnt) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Butanone (Methyl ethyl ketone, MEK) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Chloroethyl vinyl ether | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Chloronaphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Chlorotoluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Hexanone | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 2-Methyl-4,6-dinitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methylnaphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Methylphenol (o-Cresol) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Naphthylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 2-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 3,3'-Dichlorobenzidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3,5-Dinitroaniline | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |

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Issue Date: 10/10/2014

Expiration Date: 6/30/2015



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Governor



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Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| 3,4-Methylphenols (m/p-Cresols) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 3-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 4,4'-DDD | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDE | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4,4'-DDT | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Amino-2,6-dinitrotoluene (4-am-dnt) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Aminobiphenyl | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Bromophenyl phenyl ether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloro-3-methylphenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chloroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Chlorophenyl phenylether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Methyl-2-pentanone (MIBK) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| 4-Nitroaniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrotoluene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| 4-Nitrotoluene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| 7,12-Dimethylbenz(a) anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n,n-Dimethylphenethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthylene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acenaphthylene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Acetone | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acetonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acetophenone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Acrolein (Propenal) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Acrylonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Aldrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Allyl chloride (3-Chloropropene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| alpha-BHC (alpha-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| alpha-Chlordane | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aluminum | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Aluminum | EPA 6020 | Metals | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|-----------------------------|--------------------|----------------|
| Aniline | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Antimony | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Antimony | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Aramite | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Aroclor-1016 (PCB-1016) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1221 (PCB-1221) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1232 (PCB-1232) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1242 (PCB-1242) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1248 (PCB-1248) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1254 (PCB-1254) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Aroclor-1260 (PCB-1260) | EPA 8082 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Arsenic | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Arsenic | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Barium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Benzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Benzo(a)anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(a)pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(b)fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(b)fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(g,h,i)perylene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(g,h,i)perylene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(k)fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzo(k)fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Benzoic acid | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Benzyl alcohol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Beryllium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Beryllium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| beta-BHC (beta-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| bis(2-Chloroethoxy)methane | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroethyl) ether | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane)) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|------------------------------------|-------------|----------------------------|--------------------|----------------|
| bis(2-Ethylhexyl) phthalate (DEHP) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Boron | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Boron | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Bromide | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Bromobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromochloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromodichloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Bromoform | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Butyl benzyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Cadmium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Cadmium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Calcium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Calcium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Carbazole | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Carbon disulfide | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Carbon tetrachloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Cation exchange capacity | EPA 9081 | General Chemistry | NELAP | 7/1/2013 |
| Chlordane (tech.) | EPA 8081 | Pesticides-Herbicides-PCBs | NELAP | 7/1/2013 |
| Chloride | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Chlorobenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroform | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chloroprene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Chromium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Chromium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Chromium VI | EPA 7196 | General Chemistry | NELAP | 7/1/2013 |
| Chrysene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Chrysene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| cis-1,2-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| cis-1,3-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Cobalt | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Cobalt | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Conductivity | EPA 9050 | General Chemistry | NELAP | 7/1/2013 |
| Copper | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Copper | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Dalapon | EPA 8151 | Pesticides-Herbicides-PCBs | NELAP | 7/1/2013 |

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E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| delta-BHC | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dibenz(a,h)anthracene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dibenz(a,h)anthracene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Dibenzofuran | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dibromochloromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Dibromomethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Dicamba | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dichlorodifluoromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Dichloroprop (Dichlorprop) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Dieldrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Diesel range organics (DRO) | EPA 8015 | Extractable Organics | NELAP | 7/1/2013 |
| Diethyl ether | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Diethyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dimethyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-butyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Di-n-octyl phthalate | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan I | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan II | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endosulfan sulfate | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin aldehyde | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Endrin ketone | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Ethyl acetate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Ethyl methacrylate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Ethylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Fluoranthene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Fluoranthene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Fluorene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Fluorene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Fluoride | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| gamma-BHC (Lindane, gamma-Hexachlorocyclohexane) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| gamma-Chlordane | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Gasoline range organics (GRO) | EPA 8015 | Volatile Organics | NELAP | 7/1/2013 |
| Gross-alpha | EPA 9310 | Radiochemistry | NELAP | 7/1/2013 |
| Gross-beta | EPA 9310 | Radiochemistry | NELAP | 7/1/2013 |

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Laboratory Scope of Accreditation

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|--|--------------------|----------------|
| Heptachlor | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Heptachlor epoxide | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Hexachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorobutadiene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Hexachlorobutadiene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachlorocyclopentadiene | EPA 8270 | Extractable Organics,Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Hexachloroethane | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Hexachloropropene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Ignitability | EPA 1010 | General Chemistry | NELAP | 7/1/2013 |
| Indeno(1,2,3-cd)pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Indeno(1,2,3-cd)pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Iodomethane (Methyl iodide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Iron | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Iron | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Isobutyl alcohol (2-Methyl-1-propanol) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Isophorone | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Isopropylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Isosafrole | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Kjeldahl nitrogen - total | EPA 351.2 | General Chemistry | NELAP | 7/1/2013 |
| Lead | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Lead | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Lithium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| m+p-Xylenes | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Magnesium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Magnesium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Manganese | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Manganese | EPA 6020 | Metals | NELAP | 7/1/2013 |
| MCPA | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| MCPP | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Mercury | EPA 7471 | Metals | NELAP | 7/1/2013 |
| Methacrylonitrile | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methapyrilene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Methoxychlor | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Methyl bromide (Bromomethane) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl chloride (Chloromethane) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|----------------------|--------------------|----------------|
| Methyl methacrylate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methyl tert-butyl ether (MTBE) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Methylene chloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Molybdenum | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Molybdenum | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Naphthalene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Naphthalene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Naphthalene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| n-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Nickel | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Nickel | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Nitrate | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Nitrite | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Nitrobenzene | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Nitroglycerin | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodiethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodimethylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitroso-di-n-butylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodi-n-propylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosodiphenylamine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosomorpholine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosopiperidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| n-Nitrosopyrrolidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Orthophosphate as P | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| o-Toluidine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| o-Xylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Paint Filter Liquids Test | EPA 9095 | General Chemistry | NELAP | 7/1/2013 |
| Pentachlorobenzene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pentachloroethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Pentachloronitrobenzene (Quinone) | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pentachlorophenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 10/10/2014

Expiration Date: 6/30/2015



Rick Scott
Governor



John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87689-39, expiration date June 30, 2015. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|---|-------------|-----------------------------|--------------------|----------------|
| Pentaerythritoltrinitrate (PETN) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| pH | EPA 9040 | General Chemistry | NELAP | 7/1/2013 |
| pH | EPA 9045 | General Chemistry | NELAP | 7/1/2013 |
| Phenacetin | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Phenanthrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Phenanthrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Phenol | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Potassium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Potassium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Propionitrile (Ethyl cyanide) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Pyrene | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Pyrene | EPA 8310 | Extractable Organics | NELAP | 7/1/2013 |
| Pyridine | EPA 8270 | Extractable Organics | NELAP | 7/1/2013 |
| Radium-228 | EPA 9320 | Radiochemistry | NELAP | 7/1/2013 |
| RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Reactive cyanide | EPA 7.3.3.2 | General Chemistry | NELAP | 7/1/2013 |
| sec-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Selenium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Selenium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Silicon | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Silver | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Silvex (2,4,5-TP) | EPA 8151 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Sodium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Sodium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Strontium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Strontium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Styrene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Sulfate | EPA 9056 | General Chemistry | NELAP | 7/1/2013 |
| Synthetic Precipitation Leaching Procedure | EPA 1312 | General Chemistry | NELAP | 7/1/2013 |
| tert-Butylbenzene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Tetrachloroethylene (Perchloroethylene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Tetryl (methyl-2,4,6-trinitrophenyltriamine) | EPA 8321 | Extractable Organics | NELAP | 7/1/2013 |
| Tetryl (methyl-2,4,6-trinitrophenyltriamine) | EPA 8330 | Extractable Organics | NELAP | 7/1/2013 |
| Thallium | EPA 6010 | Metals | NELAP | 7/1/2013 |

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State Laboratory ID: E87689 EPA Lab Code: MO00054 (314) 298-8566

E87689
TestAmerica St. Louis
13715 Rider Trail North
Earth City, MO 63045

Matrix: Solid and Chemical Materials

| Analyte | Method/Tech | Category | Certification Type | Effective Date |
|--|-------------|-----------------------------|--------------------|----------------|
| Thallium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Thorium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Tin | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Tin | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Titanium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Titanium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Toluene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Total cyanide | EPA 9010 | General Chemistry | NELAP | 7/1/2013 |
| Total cyanide | EPA 9012 | General Chemistry | NELAP | 7/1/2013 |
| Total organic carbon | EPA 9060 | General Chemistry | NELAP | 7/1/2013 |
| Total radium | EPA 9315 | Radiochemistry | NELAP | 7/1/2013 |
| Toxaphene (Chlorinated camphene) | EPA 8081 | Pesticides-Herbicides-PCB's | NELAP | 7/1/2013 |
| Toxicity Characteristic Leaching Procedure | EPA 1311 | General Chemistry | NELAP | 7/1/2013 |
| trans-1,2-Dichloroethylene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,3-Dichloropropene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| trans-1,4-Dichloro-2-butene | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Trichloroethene (Trichloroethylene) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Trichlorofluoromethane | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Uranium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Vanadium | EPA 6020 | Metals | NELAP | 7/1/2013 |
| Vinyl acetate | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Vinyl chloride | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Xylene (total) | EPA 8260 | Volatile Organics | NELAP | 7/1/2013 |
| Zinc | EPA 6010 | Metals | NELAP | 7/1/2013 |
| Zinc | EPA 6020 | Metals | NELAP | 7/1/2013 |

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Appendix 4. Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Activity, of radionuclides: The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nuclear level) for a given amount of a radionuclide. Its standard unit (SI) is the Becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7×10^{10} Bq equals 1 Ci, exactly. (ANSI)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (QSM)

Analysis: A combination of sample preparation and instrument determination. (QSM)

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together. (QSM)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (NELAC)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (NELAC)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (NELAC)

Background: Ambient signal response recorded by measurement instruments that are independent of radioactivity contributed by the radionuclides being measured in the sample. (ANSI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together

as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (NELAC)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (NELAC)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (NELAC)

- 1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
- 2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Standard (Source): A substance or reference material used to calibrate an instrument (QAMS)

Carrier: Carriers are stable counterparts of the radioactive isotope(s) to be measured. When used, carriers are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). The carrier yield is used in the data calculation to correct for all sources of analytical losses. The term carrier can also be used for a non-radioactive compound added to assist in the isolation of the target analyte(s).

Certified Reference Material (CRM): A reference material

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (NELAC)

Check source: a radioactive source, not necessarily traceable to a national standards body such as NIST in the USA that is used to confirm the continuing satisfactory operation of an instrument. (ASTM)

Clouseau: TestAmerica custom software developed to document, track and trend non-conformances throughout the laboratory. The software interfaces with the laboratory information management system, QuantIMS and the report narrative generating software, KATO, to provide the laboratory with a corrective action system.

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safe-guarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (NELAC)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Control Chart: A graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g., mean, standard deviation, range, etc.) of the data.

“A control chart has two basic uses: (1) as a tool to judge if a process was in control, and (2) as an aid in achieving and maintaining statistical control. For applications related to radiation detection instrumentation or radiochemical processes, the mean (center line) value of a historical characteristic (e.g., mean detector response), subsequent data values and control limits placed symmetrically above and below the center line are displayed on a control chart.” (MARLAP)

Count rate: The rate at which detector pulses are being registered in a selected voltage interval. The unit is reciprocal seconds (i.e., s^{-1}). Generally the count rate is uncorrected for detector efficiency. The count rate divided by the detector efficiency for a specific particle and energy will yield the source activity.

Count time: The time interval for the counting of a sample or source by a radiation detector. Depending upon the context used, this can be either the “clock” time (the entire period required to count the sample), or “live” time (the period during which the detector is actually counting). Live time is always less than or equal to clock time. (MARLAP)

Continuing Calibration Verification: The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and nonlinear calibration models. (QSM)

Correction: Actions necessary to correct or repair analysis specific non-conformances (e.g. the acceptance criteria for method specific QC and protocols as well as the associated corrective actions). The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402) A root cause analysis may not be necessary in all cases. (QSM)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (NELAC)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (NELAC)

Detection Limit (DL): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence. (QSM)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Energy Calibration: The correlation of the multi-channel analyzer (MCA) channel number to decay photon energy, obtained from the location of peaks from known radioactive standards.

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

False Negative: A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest. (QSM)

False Positive: A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest. (QSM)

Field Blank: Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Initial Calibration Verification (ICV): Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration. (QSM)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is $\pm 100\%$. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Laboratory Information Management Systems (LIMS): The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents. (QSM)

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (NELAC)

QSM Clarification: The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (NELAC)

QSM Clarification: The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement Uncertainty: An estimate of the error in a measurement often stated as a range of values that contain the true value, within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty. (QSM)

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Minimum Detectable Activity or Concentration (MDA/MDC): For radiological analyses it is the smallest amount of activity/concentration that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity/concentration and a 5% chance that the true activity/concentration went undetected.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (NELAC)

Operator Aid: A technical posting, other than formal procedures, rules, instructions (such as poster, operating manual, or notepad) that assists workers in routine tasks and are not required to be posted or displayed by any organization or procedure. All operator aids must be controlled by the facility.

Qualitative Analysis: Analysis designed to identify the components of a substance or mixture. (QSM)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (NELAC)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (NELAC)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (NELAC)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (NELAC)

Quantitative Analysis: analysis designed to determine the amounts or proportions of the components of a substance. (QSM)

RadCapture: Software used to process and report radiochemical data.

Radioactive: exhibiting radioactivity or containing radionuclides. (MARLAP)

Radioactive decay: Process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

Radioactivity: spontaneous emission of radiation, either directly from unstable atomic nuclei or as a consequence of a nuclear reaction.

Radionuclide: a nuclide that is radioactive (capable of undergoing radioactive decay). (MARLAP)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (NELAC)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (NELAC)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (NELAC)

Reporting Limit: A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix. (QSM)

Sample Transfer Utility (STU): TestAmerica custom software developed to document and track samples through the laboratory. The software interfaces with the laboratory information management system, QuantIMS. STU employs barcode technology for rapid processing of sample transfer events including removal from storage, transfer between personnel and sample disposal.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (NELAC)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (NELAC)

Standard Deviation: the square root of a variance of a random variable. The variance is a measure of the variation of the observations within a measurement set. The standard deviation is often estimated using a set of measurements of the random variable. The standard deviation has the same units as the measured quantity and therefore, is particularly convenient when describing the variability of the measured quantity. (ANSI)

Standard Operating Procedure (SOP): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (NELAC)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systematic error: An error component that produces a fixed bias in the underlying expected value of a determination, from measurement to measurement. (ANSI)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (NELAC)

Tracer: Tracers are radioactive and/or massless. Where used, they are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). Tracers are counted and the yield is used in data calculations to correct for and all sources of analytical loss.

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Unethical actions: Deliberate falsification of analytical or quality control results, where failed method or contractual requirements are made to appear acceptable. (QSM)

Acronyms:

| | |
|------|--|
| %R | Percent Recovery |
| ANSI | American National Standards Institute |
| ASTM | American Society for Testing and Materials |
| Bq | becquerel |
| CAR | Corrective Action Report |
| CCV | Continuing Calibration Verification |
| CF | Calibration Factor |
| CFR | Code of Federal Regulations |
| Ci | Curie |
| CLP | Contract Laboratory Program |
| COC | Chain of Custody |
| cpm | Counts per minute |
| cps | Counts per second |
| CRM | Certified reference material |
| CSU | Combined standard uncertainty |
| CWA | Clean Water Act |
| DER | Duplicate Error Ratio |
| DOC | Demonstration of Capability |
| DOD | Department of Defense |

| | |
|---------|--|
| DOE | Department of Energy |
| DOECAP | DOE Consolidated Audit Program |
| DOT | Department of Transportation |
| dpm | Disintegrations per minute |
| DQO | Data Quality Objectives |
| DUP | Duplicate |
| EDD | Electronic data deliverable |
| EHS | Environment, Health and Safety |
| EPA | Environmental Protection Agency |
| FWHM | Full width half maximum |
| GC | Gas Chromatography |
| GC/MS | Gas Chromatography/Mass Spectrometry |
| GFPC | Gas-flow Proportional Counter |
| HPGe | High-purity germanium |
| HPLC | High Performance Liquid Chromatography |
| ICP | Inductively Coupled Plasma Atomic Emission Spectroscopy |
| ICP-MS | ICP/Mass Spectrometry |
| ICV | Initial Calibration Verification |
| IDL | Instrument Detection Limit |
| IH | Industrial Hygiene |
| IS | Internal Standard |
| ISO | International Organization of Standardization |
| keV | Kilo electron volts |
| LAN | Local area network |
| LCL | Lower control limits |
| LCS | Laboratory Control Sample |
| LCSD | Laboratory Control Sample Duplicate |
| LIMS | Laboratory Information Management System |
| LLD | Lower Level of Detection |
| LOD | Limit of Detection |
| LLQ | Lower Level of Quantitation |
| LOQ | Limit of Quantitation (PQL) |
| LSC | Liquid scintillation counter |
| MAPEP | Mixed Analyte Performance Evaluation Program |
| MARLAP | Multi-Agency Radiological Laboratory Analytical Protocol |
| MCL | Maximum contaminant limit |
| MDA/MDC | Minimum Detectable Activity/Concentration |
| MDL | Method Detection Limit |
| MDLCK | MDL Check Standard |
| MDLV | MDL Verification Check Standard |
| ME | Marginal exceedance |
| MeV | Mega electron volts |
| MQC | Minimum quantifiable concentration |
| MQO | Measurement quality objective |
| MRL | Method Reporting Limit Check Standard |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| MSDS | Material Safety Data Sheet |
| NCM | Non-conformance memo |
| NELAC | National Environmental Laboratory Accreditation Conference |
| NELAP | National Environmental Laboratory Accreditation Program |
| NIST | National Institute of Standards and Technology |
| NVLAP | National Voluntary Laboratory Accreditation Program |
| pCi | picocurie |

| | |
|------------------|--|
| PE | Performance Evaluation |
| PT | Performance Testing |
| TNI | The NELAC Institute |
| QAM | Quality Assurance Manual |
| QA/QC | Quality Assurance / Quality Control |
| QAMS | Quality Assurance Management Systems |
| QAPP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RDL | Required detection limit |
| RF | Response Factor |
| ROI | Region of interest |
| RPD | Relative Percent Difference |
| RPP | Radiation Protection Plan |
| RSD | Relative Standard Deviation |
| RSO | Radiation Safety Officer |
| SAP | Sample and analysis plan |
| SD | Standard Deviation |
| SMO | Sample Management Office |
| SOP | Standard Operating Procedure |
| SOW | Statement of work |
| SQC | Statistical quality control |
| SRM | Standard reference material |
| TAT | Turn-Around-Time |
| TCLP | Toxicity characteristic leaching procedure |
| TLD | Thermoluminescent dosimeter |
| TPU | Total propagated uncertainty |
| TSS | Total suspended solids |
| μohms | Resistivity unit of measure |
| WET | Whole effluent toxicity |
| WMP | Waste Management Plan |
| WP | Water pollution |
| VOA | Volatiles |
| VOC | Volatile Organic Compound |

Appendix 5: Laboratory Certifications, Accreditations, Validations

TestAmerica St. Louis maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

|  | | <h2 style="text-align: right;">TestAmerica Certifications</h2> | | |
|---|---------------|--|-----------------|-----------------|
| Laboratory | Program | Authority | Identification | Expiration Date |
| TestAmerica St. Louis | DoD ELAP | L-A-B | L2305 | 01/10/2016 |
| TestAmerica St. Louis | Federal | USDA | P330-07-00122 | 01/09/2017 |
| TestAmerica St. Louis | NELAP | California | 2886 | 03/31/2015 |
| TestAmerica St. Louis | NELAP | Florida | EB7689 | 06/30/2015 |
| TestAmerica St. Louis | NELAP | Illinois | 200023 | 11/30/2015 |
| TestAmerica St. Louis | NELAP | Kansas | E-10236 | 03/31/2015 |
| TestAmerica St. Louis | NELAP | Louisiana | 04080 | 06/30/2015 |
| TestAmerica St. Louis | NELAP | Louisiana | LA150017 | 12/31/2016 |
| TestAmerica St. Louis | NELAP | New Jersey | MC002 | 06/30/2015 |
| TestAmerica St. Louis | NELAP | New York | 11616 | 03/31/2015 |
| TestAmerica St. Louis | NELAP | Pennsylvania | 68-00540 | 02/28/2015 |
| TestAmerica St. Louis | NELAP | Texas | T104704193-13-6 | 07/31/2015 |
| TestAmerica St. Louis | NELAP | Utah | MC000542013-5 | 07/31/2015 |
| TestAmerica St. Louis | NELAP | Virginia | 460230 | 06/14/2015 |
| TestAmerica St. Louis | NRC | NRC | 24-24817-01 | 12/31/2022 |
| TestAmerica St. Louis | State Program | Alaska | MC00054 | 06/30/2015 |
| TestAmerica St. Louis | State Program | Connecticut | PH-0241 | 03/31/2015 |
| TestAmerica St. Louis | State Program | Iowa | 373 | 12/01/2016 |
| TestAmerica St. Louis | State Program | Kentucky (DW) | 90125 | 12/31/2015 |
| TestAmerica St. Louis | State Program | Maryland | 310 | 09/30/2015 |
| TestAmerica St. Louis | State Program | Missouri | 780 | 06/30/2015 |
| TestAmerica St. Louis | State Program | Nevada | MC000542013-1 | 07/31/2015 |
| TestAmerica St. Louis | State Program | North Dakota | R207 | 06/30/2015 |
| TestAmerica St. Louis | State Program | Oklahoma | 9997 | 08/31/2015 |
| TestAmerica St. Louis | State Program | South Carolina | 85002001 | 06/30/2015 |
| TestAmerica St. Louis | State Program | Washington | C592 | 08/30/2015 |
| TestAmerica St. Louis | State Program | West Virginia DEP | 381 | 08/31/2015 |

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* Certification Valid - Laboratory is Pending Renewal with the Program Authority
For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com
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The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. For each organization or may be found on the corporate web site, the laboratory’s public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix 6: Calculations

Common Calculations

- Percent Recoveries (*ICV, CCV, LCS, Surrogates*) are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found}}{\text{True}} \right)$$

- Tracers and Carriers

$$\text{Recovery (\%)} = \frac{\text{measured}}{\text{added} - \text{native}} \times 100$$

Where:

Measured is the amount of tracer/carrier measured

Added is the amount of tracer/carrier added (spiked) into the sample

Native is the amount of tracer/carrier analyte native to the sample

- Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \left(\frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

- The relative percent difference (RPD) of matrix spike/matrix spike duplicates is calculated according to the following equation:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

- The relative percent difference (RPD) of sample/sample duplicates is calculated according to the following equation:

$$RPD = 100 \left[\frac{|SR - SD|}{\left(\frac{SR + SD}{2} \right)} \right]$$

Where:

SR = sample result

SD = sample duplicate result

- The percent difference (%D) is calculated as follows:

$$\%Difference = \frac{|R_1 - R_2|}{R_1} \times 100$$

Where:

R₁ = First result

R₂ = Second result

- Standard Deviation (SD) is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^N (X_i - X)^2}{N - 1}}$$

Where:

X_i = Value of X as i through N

N = Number of points

X = Average value of X_i

ADDITIONAL Calculations for Metals

- The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

- The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

Additional Calculations for Organics

- The calibration factor for an external calibration standard is calculated as follows:

$$\text{Calibration Factor (CF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

- Relative Standard Deviation (%RSD), applicable to initial calibration, is calculated as follows:

$$\%RSD = \frac{SD}{CF_{avg}} \times 100$$

Where:

CF_{avg} = The average of the initial CFs for a compound

SD = The standard deviation (using n-1) of the initial calibration CFs for a compound

- Aqueous sample concentration using external standard calibration is calculated as follows:

$$\text{Concentration (mg / L)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μ L

D_f = Dilution factor

V_t = Volume of total extract, μ L

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area or height/ng

- Non-aqueous sample concentration using external standard calibration is calculated as follows:

$$\text{Concentration (mg / kg)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μ L

D_f = Dilution factor

V_t = Volume of total extract, μ L

CF = Calibration factor, area or height/ng

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \% \text{Moisture}}{100} \quad (D = 1 \text{ if wet weight is required})$$

- *On column concentration*

On Column Concentration ($\mu\text{g/mL}$):

$$[OC] = \frac{A_x}{CF}$$

Where:

$$[OC] = \text{On Column Concentration [typically expressed in } \mu\text{g/mL (ppm)]}$$

Then substitute/derive

$$[C] = [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit ($\mu\text{g/L}$)* and we solve the equation for *concentration ($\mu\text{g/L}$)*

Then

$$[C] \equiv RL \equiv [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

Where:

RL = Reporting Limit

Additional Calculations for GC/MS SVOA

- Concentration calculation using average response factor:

$$C_{ex} = \frac{R_x C_{is}}{R_{is} RF}$$

- Concentration calculation using linear fit:

$$C_{ex} = A + B \frac{(R_x C_{is})}{R_{is}}$$

Where:

C_{ex} = Concentration in extract, $\mu\text{g/ml}$
 R_x = Response for analyte
 R_{is} = Response for internal standard
 C_{is} = Concentration of internal standard
 A = Intercept
 B = Slope

- Concentration calculation using quadratic fit:

$$C_{ex} = A + B \left(\frac{R_x C_{is}}{R_{is}} \right) + C \left(\frac{R_x C_{is}}{R_{is}} \right)^2$$

Where:

C = Curvature

- Aqueous sample concentration is calculated as follows:

$$\text{Concentration, } \mu\text{g} / \text{L} = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, μL , taking into account dilutions
 V_o = Volume of water extracted (ml)

- Sediment/soil, sludge and waste concentration is calculated as follows:

$$\text{Concentration, } \mu\text{g} / \text{kg} = \frac{C_{ex} V_t}{W_s D}$$

Where:

W_s = Weight of sample extracted or diluted in grams
 D = (100 - % moisture in sample)/100, for a dry weight basis
or 1 for a wet weight basis

Additional Calculations for GC/MS VOA

- Calculation (x) for water and water-miscible waste:

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured
 A_{is} = Area of the characteristic ion for the internal standard
 I_s = Amount of internal standard added in ng

V_o = Volume of water purged, mL

$$D_f = \text{Dilution Factor} = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

- Calculation (x) for medium level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is} are the same as for water
 V_t = Volume of total extract, mL (typically 25 mL)
 V_a = Volume of extract added for purging, μL
 W_s = Weight of sample extracted, g

$$D = \frac{100 - \% \text{moisture}}{100}$$

- Calculation (x) for low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is} are the same as for water
 D is the same as for medium level soils
 W_s = Weight of sample added to the purge vessel, g

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where:

CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

Gamma Activity Concentration

The activity concentration of a sample will be calculated using the following equation.

$$ACT_S = \frac{\text{Net_Counts}}{2.22 * E * t_S * Ab * V_A * D_C * D_S}$$

where:

| | | |
|------------------|---|--|
| ACT _S | = | the activity in pCi/(units of the volume) |
| Net_Counts | = | the net area of a peak |
| 2.22 | = | the correction factor to pCi |
| E | = | the efficiency – corrected for transmission |
| t _S | = | the count time in minutes |
| Ab | = | the gamma abundance factor |
| V _A | = | the sample aliquot volume |
| D _C | = | the decay correction during the analysis |
| D _S | = | the decay correction from collection date to start of analysis |

Gamma Uncertainty of Concentration (at 2σ confidence level)

The Total Propagated Uncertainty (TPU) will be calculated using the following equation.

The software calculates the 2σ TPU term by incorporating the stochastic counting uncertainty and by examining the nuclide library for the error in the nuclide half-life and abundance for their respective contributions. The software routine also includes the standard certificate file and the calibration standard uncertainties. Finally, a 1% factor is added in quadrature due to the uncertainty in the preparation of the sample. This is attributed to the maximum allowable variability of the balances.

$$TPU_S = 1.96 * ACT_S * \sqrt{\left(\frac{\Delta P}{P}\right)^2 + \left(\frac{\Delta Ab}{Ab}\right)^2 + \left(\frac{\Delta \epsilon}{\epsilon}\right)^2 + \left(\frac{\Delta V}{V}\right)^2 + \left(\frac{sys}{100}\right)^2 + (\Delta Decay)^2}$$

Where:

$$\Delta Decay = \left[\frac{\Delta T_{1/2}}{T_{1/2}} \right] * \left[\frac{\lambda E_r}{1 - e^{-\lambda E_r}} - \lambda(T_S + E_r) - 1 \right]$$

Where:

| | | |
|------------------|---|--|
| TPU _S | = | the 2σ uncertainty of the activity of the sample |
| ACT _S | = | the activity in pCi/(units of volume) |
| 1.96 | = | the statistical multiplication factor for 95% confidence level |
| ΔP | = | the uncertainty in the peak area |

| | | |
|----------------------|---|---|
| ΔAb | = | the uncertainty in gamma abundance |
| $\Delta \varepsilon$ | = | the uncertainty in the efficiency ε |
| ΔV | = | the uncertainty in the volume |
| sys | = | the systematic error estimate (in %)* |
| $\Delta T_{1/2}$ | = | the uncertainty in the half-life |
| $T_{1/2}$ | = | the half-life of the nuclide of interest |
| λ | = | the decay constant |
| E_r | = | the elapsed real time during count |
| T_s | = | the sample collection time |

Gamma MDC

The minimum detectable concentration will be calculated using the following equation.

$$MDC = \frac{4.65 * \sqrt{R_B * t_S} + 2.71}{2.22 * E * t_S * Ab * V_A * D_C * D_S}$$

Where:

| | | |
|-------|---|--|
| MDC | = | Minimum Detectable Activity of the sample |
| R_B | = | Count rate of detector background (in cpm) |
| t_S | = | Count time for analysis |
| E | = | Detector efficiency |
| Ab | = | Abundance of the gamma emission |
| V_A | = | sample aliquot volume |
| D_C | = | Decay during sample analysis |
| D_S | = | Decay from collection to start of analysis |

Alpha Tracer Yield Recovery

Tracer Yield Recovery

$$Y = \frac{(C_T - C_B)}{E * A_T * t_S}$$

Where:

| | | |
|-------|---|------------------------------|
| Y | = | Chemical Yield |
| C_T | = | Tracer Counts |
| C_B | = | Tracer ROI background counts |
| A_T | = | Tracer dpm |
| t_S | = | Count time for analysis |
| E | = | Detector efficiency |

Additional Information for Radiochemistry Calculations:

Zero Count Uncertainty

Certain analyses with intrinsic low background may lead to instances where both the background and the sample count results may be zero (e.g. alpha spec, Ni-59). In such circumstances, the counting uncertainty (CU) and total propagated uncertainty (TPU) will evaluate to zero. To provide a non-zero estimate of the counting uncertainty (and thus a non-zero TPU) in such an occasion, a value of one (1) will be substituted for the sample counts in the counting uncertainty and critical level equations.

Crosstalk Calculation

Alpha into Beta Crosstalk

$$\alpha \gg \beta \text{ crosstalk} = \frac{CPM_{XT}}{CPM_{\alpha} + CPM_{XT}} = y$$

$$yCPM_{\alpha} + yCPM_{XT} = CPM_{XT}$$

$$CPM_{XT} = \frac{y}{(1-y)} CPM_{\alpha} \text{ where } CPM_{\alpha} \text{ is net alpha CPM}$$

Where:

- CPM = counts per minute (S=Sample, B=Background, XT=crosstalk, α=alpha)
- T = count duration in minutes (S=Sample, B=Background)
- E = Efficiency
- V = aliquot volume
- UF = uncertainty factor (e.g. 0.05)
- Act = activity

RadCapture Version 5.1.63

Calculation equations for all methods were updated to create consistency. All methods now use the form:

$$Activity = \frac{\left(\frac{Cs}{Ts} - \frac{Cxt}{Ts} - \frac{Cb}{Tb} \right)}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncCnt } (1\sigma) = \frac{\sqrt{\frac{C_s}{T_s^2} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncTot } (1\sigma) = \sqrt{\text{UncCnt}^2 + (\text{TPUFact} * \text{Activity})^2}$$

$$\text{MDC} = \left(\frac{3.29 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} + \frac{3}{D * E * I * V * T_s * R * A} \right) * DF * UCF$$

$$\text{DLC} = \left(\frac{1.645 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} \right) * DF * UCF$$

Where:

| | | |
|-----|---|--|
| Cs | = | Sample Counts |
| Cb | = | Background Counts |
| Cxt | = | Crosstalk Counts (currently only gross beta) |
| Ts | = | Sample Count Duration |
| Tb | = | Background Count Duration |
| D | = | Decay |
| E | = | Efficiency |
| I | = | Ingrowth |
| V | = | Aliquot Volume |
| R | = | Recovery |
| A | = | Abundance (Branching Ratio) |
| DF | = | Dilution Factor |
| UCF | = | Units Conversion Factor |
| Chi | = | non-Poisson variance |

For the count uncertainty, if both Cs and Cb = 0, then 1 is forced into Cs.
For the DLC, if Cb = 0, then 1 is forced into Cb.

Gross Alpha/Beta is the only method which currently employs a crosstalk factor (and only for alpha into beta crosstalk). However, a crosstalk factor is included for all methods to create consistency. For all methods except Gross Alpha/Beta, Cxt is set to zero in the code.

Similarly, the non-Poisson variance (Chi) has only been employed for a specific client, and only for LSC methods. It is included for all methods to create consistency in the calculation equations. A table is set up in the database to list the Chi factor for each analyte. This factor may be updated on a periodic basis to reflect current operating conditions. This is controlled by an "active" date assigned in the table. The Chi factor is currently set to only be applied for

specific projects (client-based). When not directed to the Chi Table, the calculation uses zero (currently the default for all).

When both the crosstalk and Chi factors are zero, all equations are essentially equivalent to previous versions. The new DLC equation has a marked distinction modification in that it essentially represents a “non-paired” situation to take into account variation in count durations of the background and sample. When the sample and background count durations are the same, the DLC result of the new “non-paired” equation equals the result of the previous equation. Thus, for this verification only the DLC is calculated manually when the sample and background count durations are different. In addition, the factor in the second portion of the MDC equation has been changed to “3” (updated from “2.71” to reflect current generally accepted industry practice).

Equations for Isotopes by Mass and Activity ICP-MS

Activity Calculation:

$$A_c = M_c \times S$$

Where:

A_c = Activity concentration of Nuclide (e.g. pCi/g or pCi/L)

M_c = Mass concentration of nuclide (e.g. ug/g or ug/L)

S = Specific Activity of the Nuclide

The specific activity of a nuclide is a constant based upon the half-life.

Total Uranium, by Mass:

$$M_{Total} = M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-238}$$

Where:

M = Mass for each isotope from ICP - MS results

Total Uranium, by Activity:

$$A_{Total} = A_{U-233} + A_{U-234} + A_{U-235} + A_{U-236} + A_{U-238}$$

Where:

A = Activity for each isotope using conversion above from ICP - MS results

Percent U-235 (by mass):

$$\text{Percent U - 235} = \left(\frac{M_{U-235}}{(M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-238})} \right) \times 100$$

Where:

M = Mass for each isotope

Specific Activity values utilized in the calculations above were obtained from NuclideNavigator Version 3.4 and are based upon the PCNUDAT data file from the National Nuclear Data Center (NNDC) at Brookhaven National Laboratory (BNL).

| <u>Nuclide</u> | <u>Specific Activity (pCi/ug)</u> |
|----------------|-----------------------------------|
| Technetium | 17120 |
| Uranium 233 | 9636 |
| Uranium 234 | 6222 |
| Uranium 235 | 2.161 |
| Uranium 236 | 64.67 |
| Uranium 238 | 0.3361 |

Uranium, by Mass:

$$M = \frac{(A \times C) \times (G / L)}{N}$$

Where:

A = Activity in pCi/L for liquid, pCi/g for soil

C = conversion factor from pCi to Bq = 0.037

G = gram formula weight

L = Lamda = 0.693 / halflife in seconds

N = Avegado' s Number = 6.02252E + 23

Total Uranium, by Mass:

$$M_{Total} = M_{U-234} + M_{U-235} + M_{U-238}$$

Where:

M = Mass for each isotope from above equation

Percent U-235:

$$\text{Percent U - 235} = \left(\frac{M_{U-235}}{(M_{U-234} + M_{U-235} + M_{U-238})} \right) \times 100$$

Where:

M = Mass for each isotope

Appendix 7 Laboratory SOP Listing

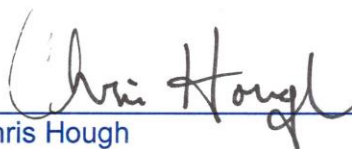
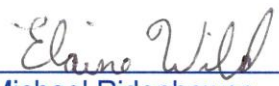
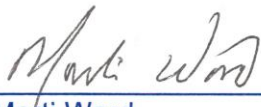
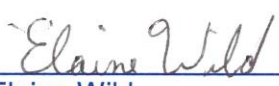
| SOP Number | SOP Title |
|------------|--|
| ST-GC-0005 | Extractable Total Petroleum Hydrocarbons |
| ST-GC-0013 | Extraction and analysis of Phenols |
| ST-GC-0014 | Aromatic Volatiles and Volatile Petroleum Hydrocarbon |
| ST-GC-0015 | PCB GC Analysis |
| ST-GC-0016 | Pesticide GC Analysis |
| ST-GC-0017 | Herbicide GC Analysis |
| ST-GC-0018 | Analysis of Water Miscible Non-Halogenated Organic |
| ST-GC-0019 | RSK-175 |
| ST-HS-0001 | Waste Minimization Plan |
| ST-HS-0002 | Facility Addendum to Corporate Safety Manual |
| ST-HS-0003 | St. Louis Facility Contingency Plan |
| ST-HS-0004 | Hazardous Waste Management Plan |
| ST-HS-0005 | Laboratory Security Systems |
| ST-HS-0006 | Quarantine Soils Procedure |
| ST-HS-0007 | Fume Hood Calibration |
| ST-IP-0001 | Reactive Cyanide & Sulfide |
| ST-IP-0002 | Acid Digestion of soil |
| ST-IP-0004 | Labware Prep for Inorganic & Trace Metal Analysis |
| ST-IP-0013 | Acid Digestion of Aqueous Samples & Extracts |
| ST-IP-0014 | Alkaline digestion of Cr+6 |
| ST-IP-0015 | Filtration Procedure for Dissolved Metals Analysis |
| ST-IP-0019 | Sulfide Distillation |
| ST-IP-0020 | Distribution Coefficients of Inorganic Species by the Batch Method |
| ST-IS-0001 | Software Change Management |
| ST-IS-0002 | Software Testing, Validation & Verification |
| ST-IS-0003 | Information Systems |
| ST-LC-0001 | HPLC Analysis of PAH/PNA |
| ST-LC-0002 | Analysis of Nitroaromatic & Nitroamine Explosives |
| ST-LC-0004 | Analysis of Perchlorates by LC/MS/MS |
| ST-LC-0005 | Analysis of Nitroaromatics by LC/MS/MS |
| ST-LC-0006 | Analysis of Herbicides by Method 8321 |
| ST-MS-0001 | GC/MS Analysis based on 8270C and 625 |
| ST-MS-0002 | Volatile Organics by GCMS |
| ST-MT-0001 | Metals by ICP/MS |
| ST-MT-0003 | Metals by ICP-AES |
| ST-MT-0005 | Mercury in Aqueous Samples by CVAA |
| ST-MT-0007 | Mercury in Solid Samples by CVAA |
| ST-MT-0008 | Total Uranium by Laser Induced Phosphorimetry (KPA) |
| ST-OP-0001 | Labware Preparation for Organic Analysis |
| ST-OP-0002 | Extraction & Cleanup of Organic Compounds from Water |
| ST-OP-0007 | Extraction of Herbicides - Water & Soil |
| ST-OP-0008 | Extraction of Nitroaromatics |
| ST-OP-0009 | TCLP/SPLP and CWET Procedures |
| ST-PM-0001 | Project Setup and Quote |

| SOP Number | SOP Title |
|------------|---|
| ST-PM-0002 | Sample Receipt & Chain of Custody |
| ST-PM-0003 | Bottle Kit Preparation |
| ST-PM-0004 | Data Review, Verification & Reporting |
| ST-QA-0002 | Standard and Reagent Preparation |
| ST-QA-0005 | Calibration & Verification Procedure for Thermometer |
| ST-QA-0014 | Evaluation of Accuracy and Precision via Control C |
| ST-QA-0016 | IDL/MDL Determination |
| ST-QA-0021 | Internal Surveillance |
| ST-QA-0023 | Document Control |
| ST-QA-0024 | Preventative Maintenance |
| ST-QA-0028 | Water System Maintenance & Monitoring |
| ST-QA-0031 | VOA Holding Blank Analysis |
| ST-QA-0035 | Preparation and Management of SOPs |
| ST-QA-0036 | Non-Conformance Memo Process |
| ST-QA-0037 | Procurement of Quality Related Items |
| ST-QA-0038 | Procedure for Compositing and Subsampling |
| ST-QA-0039 | Sample Transfer Utility |
| ST-QA-0040 | Manual Integration Procedure |
| ST-QA-0041 | Lead Auditor |
| ST-QA-0042 | 10CFR 21 Defects and Non-Compliances |
| ST-QA-0043 | DoD QSM 4.X |
| ST-QA-0044 | Training |
| ST-QAM | Quality Assurance Manual |
| ST-RC-0002 | Planchet Prep for Radiochemistry & Radiological Sc |
| ST-RC-0003 | Drying & Grinding of Soil & Solid Samples |
| ST-RC-0004 | Prep of Soil, Sludge, Filter, Biota &)/G Samples |
| ST-RC-0010 | Screening Samples for Presence of Radioactive Mate |
| ST-RC-0014 | Bulk Drying and Grinding of Soil and Solid Samples |
| ST-RC-0015 | Total Activity Screening Procedure by LSC |
| ST-RC-0020 | Determination of Gross Alpha/Beta Activity |
| ST-RC-0021 | Gross Alpha Radiation in Water - Coprecipitation |
| ST-RC-0025 | Preparation of Samples for Gamma Spectroscopy |
| ST-RC-0030 | Determination of Tritium in Water, Fluids, Soil & |
| ST-RC-0031 | Tritium Determination by Cryogenic Distillation |
| ST-RC-0036 | Chlorine-36 |
| ST-RC-0039 | Radium 226 by Alpha Spec |
| ST-RC-0040 | Total Alpha Emitting Isotopes of Radium |
| ST-RC-0041 | Radium-226 & Radium-228 by Chemical Separation |
| ST-RC-0042 | Iodine-129 in Water |
| ST-RC-0050 | Preparation of Strontium 89 & 90 |
| ST-RC-0055 | Determination of Fe55, Ni59 & Ni63 by LSC |
| ST-RC-0056 | Carbon-14 by LSC |
| ST-RC-0057 | Carbon -14/Inert Gas |
| ST-RC-0058 | Soil Prep for Sr-89, Sr-90 & Total Sr using Extraction Chromatography |
| ST-RC-0100 | Actinide Co-precipitation |
| ST-RC-0125 | Determination of TC99 using Eichrom TEVA Resin |

| SOP Number | SOP Title |
|------------|--|
| ST-RC-0210 | Determination of Po210 by Alpha Spectrometry |
| ST-RC-0211 | Determination of Pb210 by LSC |
| ST-RC-0232 | Isotopic Th/Np in Various Matrices by Eichrom TEVA |
| ST-RC-0238 | Isotopic U by Eichrom UTEVA Resin for Various Matrices |
| ST-RC-0240 | Isotopic Am/Cu/Pu/Th/U in Various Matrices Eichrom |
| ST-RC-0241 | Am/Pu/Cu/U in Various Matrices by Eichrom UTEVA & |
| ST-RC-0242 | Isotopic Th/Pu/U in Various Matrices by Eichrom Se |
| ST-RC-0245 | Determination of Pu241 by LSC |
| ST-RC-0246 | Isotopic Am/Cu/U in Various Matrices by Eichrom S |
| ST-RC-0247 | Promethium247 & Samarium151 Lanthide Resin Separation |
| ST-RC-0300 | NJ 48 Hour Gross Alpha Testing PWTA |
| ST-RC-5006 | Decontamination of Lab Glassware, Labware & Equip. |
| ST-RD-0102 | Gamma Vision Analysis |
| ST-RD-0210 | Alpha spectroscopy |
| ST-RD-0302 | Liquid Scintillation Counter Analysis |
| ST-RD-0403 | Low Background Gas Flow Proportional Counting System |
| ST-RP-0001 | Radiation Protection Program |
| ST-RP-0005 | ALARA Program |
| ST-RP-0010 | Internal Exposure Control |
| ST-RP-0020 | External Exposure Control |
| ST-RP-0030 | Radiological Contamination |
| ST-RP-0031 | Radiation Work Permits |
| ST-RP-0032 | Instrumentation and surveillance |
| ST-RP-0033 | Radiological Areas and Posting |
| ST-RP-0034 | Engineered Controls |
| ST-RP-0042 | Handling of Sealed Sources |
| ST-RP-0050 | Purchase, Receipt, Handling and ID of Radioactive |
| ST-RP-0051 | Packaging/Transportation of Radioactive Material |
| ST-RP-0100 | Radiation Protection Records |
| ST-RP-0110 | Radiation Protection Training |
| ST-RP-0120 | Emergency Response & notification |
| ST-RP-0140 | Quality Assurance in Radiological Protection |
| ST-WC-0001 | Turbidity |
| ST-WC-0002 | Cyanide Analysis by Technicon TRAACS 800 Autoanaly. |
| ST-WC-0003 | Hardness |
| ST-WC-0004 | Chemical Oxygen Demand |
| ST-WC-0005 | Percent Solids Determination |
| ST-WC-0006 | Total Organic Halides in Water (TOX) and Soil(EOX) |
| ST-WC-0011 | Analysis of pH in Water & Soil |
| ST-WC-0012 | Analysis of Sulfide in Water |
| ST-WC-0013 | Phosphorus, all Forms |
| ST-WC-0014 | Analysis of Ammonia as N in Water & Soil |
| ST-WC-0015 | Biochemical Oxygen Demand |
| ST-WC-0016 | Total Organic Carbon |
| ST-WC-0017 | Phenolics, Total Recoverable |
| ST-WC-0018 | Acidity of Water & Wastewater |

| SOP Number | SOP Title |
|------------|---|
| ST-WC-0019 | Alkalinity in Water & Soil |
| ST-WC-0020 | Prep and determination of TKN |
| ST-WC-0023 | Nitrate/Nitrite analysis by TRAACS |
| ST-WC-0025 | Conductivity in Water & Soil |
| ST-WC-0026 | Flashpoint by Pensky-Martens Closed Cup |
| ST-WC-0028 | Anions by Ion Chromatography |
| ST-WC-0029 | Residual Chlorine |
| ST-WC-0031 | Paint Filter |
| ST-WC-0033 | Hexavalent Chromium |
| ST-WC-0034 | Heat of Combustion |
| ST-WC-0036 | Determination of Solids in Water and Wastewater |
| ST-WC-0037 | Perchlorate by IC |
| ST-WC-0039 | Method 1664, N-Hexane Extractable Material |
| ST-WC-0042 | Chlorophyll-a |
| ST-WC-0044 | POTENTIOMETRIC DETERMINATION OF FLUORIDE ISE |
| ST-WC-0045 | Cation Exchange |
| ST-WC-0046 | Reactivity to Air, Water, Physical Properties |
| ST-WC-0047 | TOC in soil |
| ST-WC-0050 | Std Method for Moisture, Ash & Organic Matter |

Title: GAMMAVISION® ANALYSIS

| Approvals (Signature/Date): | | | |
|---|---------|--|---------|
|  | 6/22/15 |  | 6/22/15 |
| Chris Hough Radiochemistry Manager | Date | Michael Ridenhower Health & Safety Manager / Coordinator | Date |
|  | 6.22.15 |  | 6/22/15 |
| Marti Ward Quality Assurance Manager | Date | Elaine Wild Laboratory Director | Date |

This SOP was previously identified as SOP No. ST-RD-0102 Rev. 12

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure applies to all germanium detectors and the computer assisted germanium spectroscopy analysis system.
- 1.2 Due to the nature of gamma spectroscopy, once the system is calibrated to a particular geometry a similar matrix may be run as long as it is prepared to match a calibrated geometry.
- 1.3 This SOP is based on EPA Method 901.1, DOE EML HASL 300 Method GA-01-R and ANSI N42.14-1999.
- 1.4 The requested limits (**RL**), minimum detectable amount (**MDA**) and QC limits are maintained in the Laboratory Information Management System (**LIMS**).

2.0 SUMMARY OF METHOD

- 2.1 This procedure provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the germanium spectroscopy system.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Germanium spectrometry has potential interference. Interferences are usually in the form of radionuclides with unresolved photon emissions. These interferences are limited by the careful design/construction of the gamma spectral identification and interference libraries.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Germanium spectroscopy system utilizing a computer based data acquisition system (GammaVision[®]-32).
- 6.2 GammaVision[®]-32 (know as GammaVision) is a comprehensive, all-in-one package, for the analysis of gamma-ray spectra acquired with HPGe detectors.
- 6.3 Global Value software is an optimization tool for automation, custom reporting, quality assurance and data management (GammaVision productivity add-on software).

7.0 REAGENTS AND STANDARDS

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- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.
- 7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Samples may be collected in glass or plastic containers.
- 8.2 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

- 9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation.
- 9.2 **Batch**
 - 9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
 - 9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.
 - 9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample , and Sample Duplicate.
- 9.3 **Method Blank (MB)**
 - 9.3.1 A method blank must be counted with every sample batch.
 - 9.3.1.1 For soils, a method blank is sodium sulfate filled in the specified geometry.
 - 9.3.1.2 For waters, a method blank is DI water filled in the specified geometry.
 - 9.3.1.3 For filters, a method blank is a blank petri dish.
- 9.4 **Laboratory Control Sample (LCS)**
 - 9.4.1 An LCS must be counted with every sample batch.
 - 9.4.1.1 For water, a purchased mixed nuclide source in the specified geometry.
 - 9.4.1.2 For soil, a purchased mixed nuclide source in the specified geometry.
 - 9.4.1.3 For filters, a purchased mixed nuclide source in a petri dish.
- 9.5 **Sample Duplicate**
 - 9.5.1 A Sample Duplicate is a recounted field sample to demonstrate instrument precision, since there is no sample preparation (required to count on a different detector than the sample).
 - 9.5.1.1 If requested, the laboratory may perform a Sample Duplicate which is an additional aliquot of a field sample.
- 9.6 **Procedural Variations/ Nonconformance and Corrective Action**
 - 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager.

See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 There are two types of Calibrations performed for Gamma: Energy and Efficiency
 - 10.1.1 Energy Calibrations
 - 10.1.1.1 Frequency: the energy calibration is performed once per detector. The source is not geometry specific.
 - 10.1.1.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)
 - 10.1.1.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
 - 10.1.1.4 Range: the energy range, is 46.54 to 1836.1 keV for air filter and solid.
 - 10.1.1.5 Criteria:
 - 10.1.1.5.1 The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration.
 - 10.1.1.5.2 The energy difference (delta Δ) should be within 0.05% for all calibration points or within 0.2 keV for the calibration points.
 - 10.1.1.5.3 The FWHM must be less than 3.0 keV at 1332 keV.
 - 10.1.1.5.4 FWHM difference (delta Δ) should be within 8% for all calibration points.
 - 10.1.2 Efficiency Calibrations
 - 10.1.2.1 Frequency: the efficiency calibration is performed per geometry.
 - 10.1.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)
 - 10.1.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
 - 10.1.2.4 Range: the energy range of the calibration is dependent on the matrix that is calibrated. i.e. 46.54 to 1836.1 keV for air filter and solid, 59.5 keV to 1836.1 keV for water.
 - 10.1.2.5 Criteria:
 - 10.1.2.5.1 The curve should have at least eight points to determine the efficiency
 - 10.1.2.5.2 The calibration source must have radionuclides that “bracket” the intended range of calibration
 - 10.1.2.5.3 A minimum of 10,000 counts will be accumulated for each data point
 - 10.1.2.5.4 The efficiency difference (delta Δ) should be within 8% of the true value for each point
- 10.2 Initial Calibration Verification (ICV) [Frequency: Once]
 - 10.2.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.
 - 10.2.1.1 The ICV check does not include short half-life nuclides which may exist in the purchased standard. At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).
 - 10.2.2 An ICV must be performed with every initial calibration.
 - 10.2.3 The ICV percent recovery must be within $\pm 10\%$ of the true value for each nuclide.

- 10.2.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
- 10.3 Annual Calibration Verification (ACV) [Frequency: Annually] not geometry specific
- 10.3.1 An annual calibration verification check will be performed on each detector.
- 10.3.2 Two verification standards (second source independent from the initial calibration source) will be used for the verification checks.
- 10.3.2.1 One from a water source that surrounds the detector
- 10.3.2.2 One from a solid source that rests on top of the detector
- 10.3.3 The checks will include isotopes from the low (Am-241), medium (Cs-137) and high (Co-60) energy range.
- 10.3.4 The verification can be accomplished by using LCS samples counted on each detector.
- 10.3.5 The ACV percent recovery must be within $\pm 10\%$ of the true value for each nuclide.
- 10.3.6 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ACV, or analysis of a different ACV). Any decision to proceed with analysis of samples when the ACV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
- 10.4 Daily Checks
- 10.4.1 The detector **background** shall be checked each day that the germanium spectroscopy system is used. Limits are set at 2 sigma and 3 sigma.
- 10.4.1.1 Bkgd Countrate (background count rate for entire spectrum)
- | | | |
|---------------------|---|----------------|
| Tolerance (warning) | = | $\pm 2 \sigma$ |
| Control (out) | = | $\pm 3 \sigma$ |
- 10.4.2 The instrument **Channel, Energy, FWHM** (resolution) and **Activity Difference** (efficiency) for a detector shall be checked each day the germanium spectroscopy system is used (using a check source that is non-geometry specific).
- 10.4.2.1 **Channel** – (low and high energy) is monitored for channel alignment. Limits are set around the target Channel
- 10.4.2.1.1 QA-60 Low Energy
- | | | |
|---------------------|---|------------------|
| Tolerance (warning) | = | ± 1 channel |
| Control (out) | = | ± 2 channels |
- 10.4.2.1.2 QA-1332 High Energy
- | | | |
|---------------------|---|------------------|
| Tolerance (warning) | = | ± 2 channels |
| Control (out) | = | ± 3 channels |
- 10.4.2.2 **Energy** – (low and high energy) is monitored for energy alignment. Limits are set around a target energy
- 10.4.2.2.1 QA-60 Low Energy
- | | | |
|---------------------|---|----------------|
| Tolerance (warning) | = | ± 0.25 keV |
| Control (out) | = | ± 0.50 keV |
- 10.4.2.2.2 QA-1332 High Energy
- | | | |
|---------------------|---|----------------|
| Tolerance (warning) | = | ± 0.5 keV |
| Control (out) | = | ± 0.75 keV |
- 10.4.2.3 Full-Width at the Half Maximum (**FWHM**) - (low, mid, and high energy) is monitored for peak shape. There are no limits compared to a target FWHM. There are no lower limits (–) set for FWHM.
- 10.4.2.3.1 QA-60 Low Energy
- | | | |
|---------------------|---|-------|
| Tolerance (warning) | = | + 1.1 |
| Control (out) | = | + 1.2 |
- 10.4.2.3.2 QA-662 Mid Energy

- | | | | | |
|------------|---------|---------------------|---|-------|
| | | Tolerance (warning) | = | + 1.7 |
| | | Control (out) | = | + 1.8 |
| 10.4.2.3.3 | QA-1332 | <u>High Energy</u> | | |
| | | Tolerance (warning) | = | + 2.2 |
| | | Control (out) | = | + 2.3 |
- 10.4.2.4 **Activity Difference** (low, mid, and high energy) – is monitored to check the percent difference between the source activity and the reported activity. Limits are set around the target activity.
- | | | | | |
|------------|----------------|----------------------------|---|-----|
| 10.4.2.4.1 | QA-60/662/1332 | <u>Low/Mid/High Energy</u> | | |
| | | Tolerance (warning) | = | ± 4 |
| | | Control (out) | = | ± 5 |
- 10.4.3 If the daily check is outside of the control limits, it may be recounted or tagged out with red tag (or with an NCM). The Daily QC check may only be recounted once without corrective action.
- 10.4.3.1 If the out of control parameter is found acceptable for the rerun, the instrument can be used for the analysis of samples. *Note: No corrective action is necessary for this situation since the uncertainty can be attributed to the stochastic uncertainty of decay process (statistics), uncertainty of the sources, or a known uncorrected trend.*
- 10.4.3.2 If the instrument fails to meet the acceptance criteria for the rerun, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out". (See ST-QA-0036 for NCM details regarding tagging out of service).
- 10.4.3.3 If the QC check fails the following day for the same detector for the same specific parameter as the day before, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out" until the detector can be evaluated and/or maintenance can be performed.
- 10.4.3.4 The analyst may want to:
- 10.4.3.4.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.
- 10.4.3.4.2 Check source positioning and all instrument settings.
- 10.4.3.4.3 Check all cables for any apparent damage and confirm that all cables are routed to proper connectors and are in good working order.
- 10.4.3.4.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Corrective actions must be noted in the instrument maintenance log.
- 10.4.3.4.5 If a parameter has two successive values in the warning limits, the system will be examined for a trend and noted in the maintenance log. Decisions will be based upon the Data Quality Objectives (DQO) and the degree of the bias in relation to the parameter.
- 10.5 Background
- 10.5.1 Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements.
- 10.5.1.1 Backgrounds count for a minimum of 12 hours.
- 10.5.1.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.
- 10.5.1.1.2 After review of the monthly background, the analyst will mark each detector complete on the "Monthly Background Complete" sheet located on each gamma cave (see [attachment 2](#))
- 10.5.1.2 Monthly Background limits are set at 2 sigma and 3 sigma.

| | | | |
|------------|---|---|----------------|
| 10.5.1.2.1 | <u>Bkgd Countrate (background count rate for entire spectrum)</u> | | |
| | Tolerance (warning) | = | $\pm 2 \sigma$ |
| | Control (out) | = | $\pm 3 \sigma$ |

10.6 Calibration Software Handling

10.6.1 Gamma Detector System Energy and Shape Calibration

10.6.1.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path

“C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc” where:

10.6.1.1.1 X = Detector Number

10.6.1.1.2 Analysis method

10.6.1.1.3 Select library

10.6.1.1.4 Enter correct sample data.

10.6.1.1.5 Enter correct conversion time.

10.6.1.2 Close all detectors windows in the current instance of gamma vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.1.3 Select the menu “Analyze\Setting\Sample type....”

10.6.1.4 Select the browse button next to the “File” field and open the file. Click the “OK” button of the window to close it.

10.6.1.5 Recall the application Calibration File from the menu “Calibration \Recall Calibration...”

10.6.1.6 Select the menu “Calibrate\Calibration wizard...”

10.6.1.7 Select the option to create new energy calibrations. Select the next button.

10.6.1.8 On the energy calibration wizard page, select the file “DET_EnergyStandardMix Lib” or appropriate library for mixed gamma used the browser button if desired. Select the next button.

10.6.1.9 Select the next button to perform the energy, FWHM.

10.6.1.10 Select the edit energy button to review the energy.

10.6.1.10.1 Close the energy calibration sidebar window.

10.6.1.11 Select the save calibration button and save the calibration to “Cal\Energy\X_Energy.clb” where X is the detector.

10.6.1.12 Enter the calibration description in the format “X_ENERGY_GEOMETRY” where X is the detector number and Geometry is an appropriate geometry description when prompted. Select the Finish button to close the calibration wizard.

10.6.1.13 Print the calibration report from the menu “Calibrate \print calibration.

10.6.2 Gamma Detector System Efficiency Calibration

10.6.2.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path

“C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc” where:

10.6.2.1.1 X = Detector Number

10.6.2.1.2 Analysis method

10.6.2.1.3 Select library

10.6.2.1.4 Enter correct sample data.

10.6.2.1.5 Enter correct conversion time.

10.6.2.2 Close all detector windows in the current instance of Gamma Vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.2.3 Select the menu “Analyze\Setting\Sample Type”

10.6.2.4 Select the browse button next to the “File”, field and open the file. Click the “OK” button at the bottom of the window to close it.

10.6.2.5 Recall the applicable calibration file from the menu “Calibration\Recall Calibration” (if the geometry file currently exists)

10.6.2.6 Select the menu “Calibrate\Calibration Wizard”

- 10.6.2.7 Select the option to create new energy and efficiency calibration. Select next button.
 - 10.6.2.8 On the Energy Calibration Wizard page select the file “EnergyStandardMix Lib” or appropriate library for mixed gamma used the browser button if desired. Select the Next button.
 - 10.6.2.9 On the Efficiency Calibration Wizard page, select library file, “DET_EfficiencyCalibration.Lib” for mixed gamma sources.
 - 10.6.2.10 On the Efficiency Calibration Wizard page, select the appropriate Certification file from the directory.
 - 10.6.2.11 Select the next button to perform the energy FWHM and efficiency calibration.
 - 10.6.2.12 Select the Edit Energy button to review the energy and FWHM Calibration.
 - 10.6.2.12.1 Close the Efficiency Calibration side window.
 - 10.6.2.13 Select the save calibration button and save the calibration to Cal\X_Geometry.clb” where X is the detector and geometry is an appropriate geometry name.
 - 10.6.2.14 Enter the calibration description in the format “x_Geometry_Source number_date counted” where X is the detector number and geometry is an appropriate geometry description when prompted. Select the finish button to close the calibration wizard.
 - 10.6.2.15 Print calibration report from the menu “Calibrate\Print Calibration”
 - 10.6.2.16 Select “Analyze”, select “Entire spectrum in memory” and file print.
 - 10.6.2.17 Close the spectrum Buffer window and save the spectrum when prompted.
- 10.6.3 Detector Long Background Counting
- 10.6.3.1 Remove any samples from the detector, clean the detector, close the shield lid and start acquisition.
 - 10.6.3.2 Select detector 1 in global value quick Start
 - 10.6.3.3 Select Monthly Background PBC under Automation Groups
 - 10.6.3.4 Select Background PBC Long Count under Automation Jobs.
 - 10.6.3.5 Login using name and password.
 - 10.6.3.6 Select “OK”, ensure detector cave is empty.
 - 10.6.3.7 Repeat for each detector which background you would like to start.
 - 10.6.3.8 After the background is complete it will save as a PBC file.

11.0 PROCEDURE

- 11.1 Calibration Quality Control (**Daily Check**)
 - 11.1.1 Place the calibration quality control sample on the detector, and start acquisition.
 - 11.1.2 Select detector from global value quick sstart.
 - 11.1.3 Select Quality Control under Automation Groups.
 - 11.1.4 Select Daily Quality Control Check under Automation Jobs.
 - 11.1.5 Login with user name and password.
 - 11.1.6 Select “OK”, ensure source is on detector.
 - 11.1.7 Repeat for each detector.
 - 11.1.8 Record in the instrument run log.
- 11.2 Background Quality Control (**Daily Background**)
 - 11.2.1 Remove any samples from the detector, and start acquisition.
 - 11.2.2 Select detector global value quick start.
 - 11.2.3 Select quality control under automation groups.
 - 11.2.4 Select daily background check under automation jobs.
 - 11.2.5 Login with username and password.
 - 11.2.6 Select “OK”, ensure detector cave is empty.
 - 11.2.7 Repeat for each detector.

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11.2.8 Record in the instrument run log.

11.3 Sample Counting

11.3.1 Place the sample on the detector.

11.3.2 Select detector from global valuevalue quick start.

11.3.3 Select analyze samples under automation groups.

11.3.4 Select count sample under automation jobs.

11.3.5 Login with username and password.

11.3.6 Scan sample description from barcode report.

11.3.7 Select analysis method, sample type, geometry, library, correct date, count time, and continue.

11.3.8 Select "OK", ensure sample is on detector.

11.3.9 Record in the instrument run log.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 All calculations are performed in GammaVision-32 software; conversions are performed in RadCapture. Calculations are found in ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (MB)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the requested limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – The blank may be re-counted once to confirm the activity (in the same detector). If the re-counted MB activity exceeds the MDA and/or the requested limit, samples with less than 10 times the activity found in the blank are recounted. An NCM is written to document the excursion. Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in LIMS.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – The LCS may be re-counted once to confirm the result. If the re-counted LCS exceeds the control limit, samples that are non-detect may be reported with an NCM.

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- 13.3.2.2 LCS Spike Recovery excursion (low) – The LCS may be re-counted once to confirm the result. If the low recovery is confirmed, the batch is recounted.
- 13.3.2.3 RPD/RER Duplicate excursion – The LCS is recounted if both RPD and RER exceed criteria.

- 13.4 Duplicate
- 13.4.1 Acceptance Criteria:
- 13.4.1.1 All control analytes must be within the specified control limits for precision (RPD), max. 40% RPD, RER < 1.
- 13.4.2 Corrective Action for Duplicate not meeting acceptance criteria:
- 13.4.2.1 RPD/RER Duplicate excursion – The sample is recounted if both RPD and RER exceed criteria.
- 13.5 Insufficient Sample
- 13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the LIMS NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.
- 14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration of capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

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17.0 REFERENCES

- 17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 28th Edition, method GA-01-R, Gamma Radioassay
- 17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1
- 17.3 American National Standards Institute (ANSI) Accredited Standards Committee on Radiation Instrumentation, N42; ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
- 17.4 Ortec MCB Connections-32, Hardware Property Dialogs Manual, current version
- 17.5 MAESTRO-32, MCA Emulator, current version
- 17.6 GammaVision-32, Gamma-Ray Spectrum Analysis and MCA Emulator, current version
- 17.7 Master library Source: Gerhard Erdtmann, Werner Soyka
- 17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 17.11 Associated SOPs, Current Revision:
 - 17.11.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
 - 17.11.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
 - 17.11.3 ST-RC-0025, Preparation of Samples for Gamma Spectroscopy
 - 17.11.4 ST-QA-0002, Standards and Reagent Preparation
 - 17.11.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.11.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

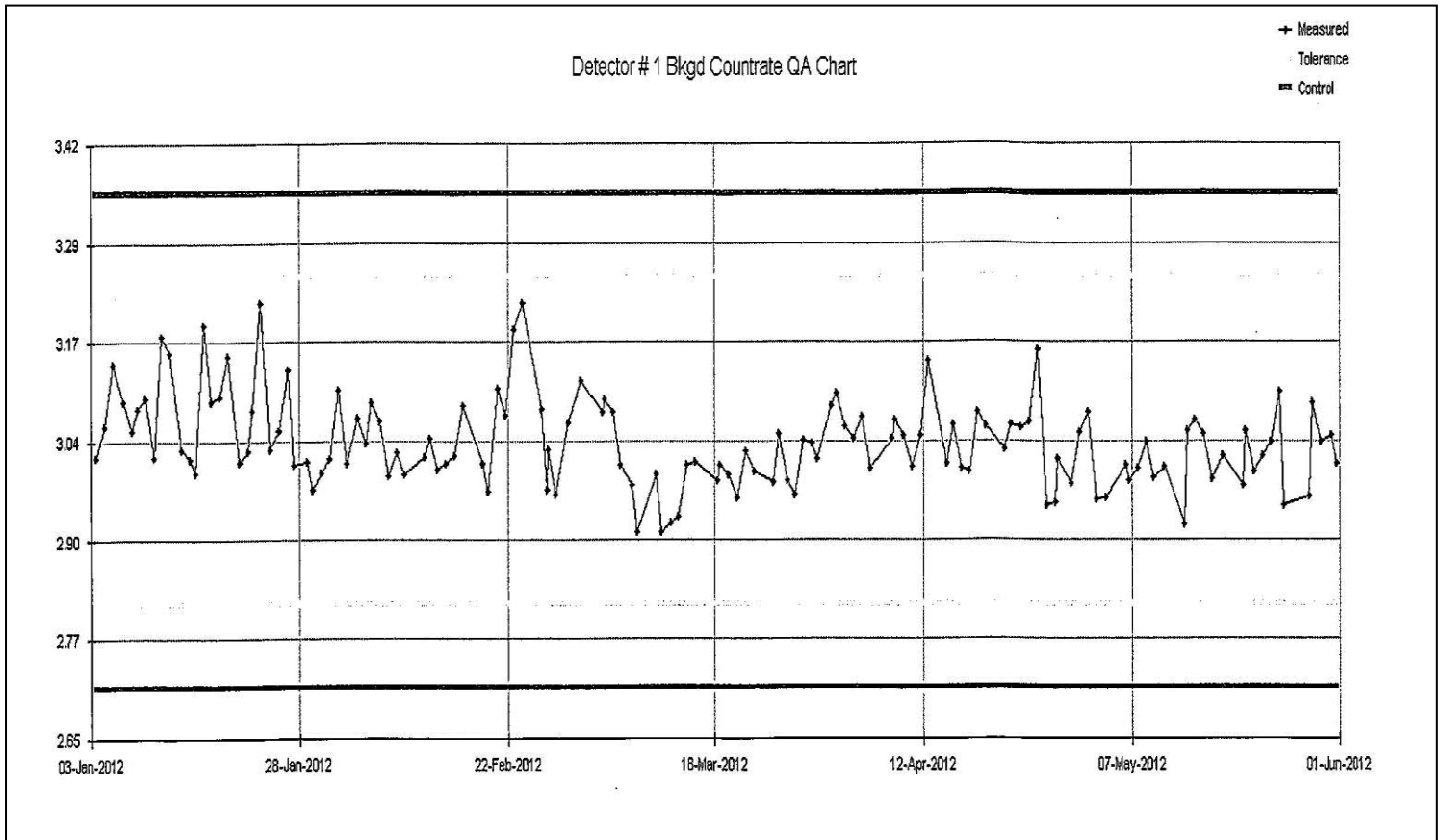
- 19.1 Annual Review, No Changes.
- 19.2 Revision 8:
 - 19.2.1 Increased background count times from 12 to 36 hours in section 10.3.1.1.
 - 19.2.2 Updated the procedure for detector long background counting in section 10.5 to reflect new software.
 - 19.2.3 Updated daily calibration checks, daily background and sample counting procedures in section 11.0 to reflect new software.
- 19.3 Revision 9:
 - 19.3.1 Replaced quartz sand with sodium sulfate to be used for soil method blanks in section 9.2.
 - 19.3.2 Updated section 10.4 regarding instrument daily checks.
 - 19.3.3 Updated data assessment and acceptance criteria in section 13.0
 - 19.3.4 Updated section 9.0 regarding batch, method blank and laboratory control samples.
 - 19.3.5 Updated the calibration points for an internal calibration in section 10.1.
 - 19.3.6 Updated the percent recovery regarding the ICV in section 10.2.
 - 19.3.7 Updated software storage file name throughout section 10.5.
- 19.4 Revision 10:
 - 19.4.1 Updated references to QuantIMS through out
 - 19.4.2 Update §10.1
 - 19.4.3 Added §10.3 Annual Calibration Verification

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- 19.4.4 Updated §10.4: 36 hour background changed to 12 hour and requirement to complete [Attachment 2](#)
- 19.4.5 Added [Attachment 2](#), “Monthly Background Complete” example
- 19.4.6 Updated §13 references to Clouseau changed to LIMS
- 19.4.7 Added §17 reference to ANSI 42.14-1999
- 19.5 Revision 11:
 - 19.5.1 Updated §1.4 with corrected terminology
 - 19.5.2 Updated §6.0 software details
 - 19.5.3 Addition/Update §10.0 major change in calibration
 - 19.5.4 Updated §13.0 additional corrective action steps
 - 19.5.5 Updated §15.0 with new verbiage
- 19.6 Revision 12: (04/16/2014)
 - 19.6.1 Spelling and grammar corrections made throughout SOP.
 - 19.6.2 Sections 10.2.3 and 10.2.5 had wording changed to common text.
 - 19.6.3 Section 10.4.3.3 was updated to add ‘for the same specific parameter as the day before’ and ‘until the detector can be evaluated and/or maintenance can be performed.’.
 - 19.6.4 Section 10.5.1.2.1 was added to provide limits for monthly backgrounds, which were not previously provided.
 - 19.6.5 Section 13.4.2 had ‘LCS’ changed to ‘duplicate’ since it is the duplicate section and LCS was incorrectly referenced.
- 19.7 Revision 13: (06/22/2015)
 - 19.7.1 Section 10.5.1.1 was updated to say “Backgrounds count for a minimum of 12 hours” new wording.
 - 19.7.2 Section 10.6.3.2 updated capitalization “global value quick”.
 - 19.7.3 Section 11.1.2 updated capitalization “global value quick start”.
 - 19.7.4 Section 11.3.2 updated capitalization “global value”.
 - 19.7.5 Section 11.3.7 updated to say “and continue.” new wording.

Attachment 1



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Attachment 2

Example of the form, actual form in use may have slight variations.

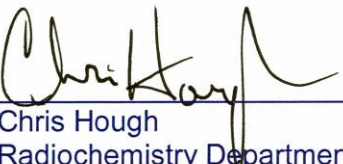



2013 Monthly Background Complete

| | |
|-------|--------------------------------------|
| Jan | Reviewed by _____ Initials & Date |
| Feb | Reviewed by _____ Initials & Date |
| Mar | Reviewed by _____ Initials & Date |
| April | Reviewed by _____ Initials & Date |
| May | Reviewed by _____ Initials & Date |
| June | Reviewed by _____ Initials & Date |
| July | Reviewed by _____ Initials & Date |
| Aug | Reviewed by _____ Initials & Date |
| Sept | Reviewed by _____ Initials & Date |
| Oct | Reviewed by _____ Initials & Date |
| Nov | Reviewed by _____ Initials & Date |
| Dec | Reviewed by _____ Initials & Date |

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**Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING
 (GFPC) SYSTEM ANALYSIS**

| Approvals (Signature/Date): | | | |
|---|---------|--|---------|
|  | 4/24/15 |  | 4/27/15 |
| Chris Hough Radiochemistry Department Manager | Date | For Michael Ridenhower Health & Safety Manager / Coordinator | Date |
|  | 4-27-15 |  | 4/24/15 |
| Marti Ward Quality Assurance Manager | Date | Elaine Wild Laboratory Director | Date |

This SOP was previously identified as SOP No. ST-RD-0403 Rev. 15

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs radium-226/228, strontium-89/90, gross alpha/beta, neptunium-236 and chlorine 36.
- 1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; and DOE EML HASL 300 method, Ba-01-R, Sr-02 and Sr-03-RC.
- 1.3 The SOP applies to GFPC analysis of liquid and solid matrices.
- 1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.
- 3.2 IQC - a computerized Quality Control Program where the counting results of Daily Radioactive check sources and Daily Background checks are entered and compared to statistical average data. A measurement within ± 3 standard deviations indicates the detector is operating within acceptable parameters.
- 3.3 α LL - discriminator setting indicating the alpha lower voltage limit.
- 3.4 Alpha Voltage Only - detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.
- 3.5 α UL - discriminator setting indicating the instruments alpha upper voltage limit.
- 3.6 β LL - discriminator setting indicating the beta lower voltage limit.
- 3.7 β UL - discriminator setting indicating the beta upper voltage limit.
- 3.8 Crosstalk - a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.
- 3.9 Plateau - a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.
- 3.10 LB4100 – LBPC (Low background Gas Flow Proportional Counting instrument).

4.0 INTERFERENCES

- 4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.
 - 4.1.1 If a sample “times out” reaching 10,000 counts before the allotted time, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits. If the chamber requires action to remove contamination and a new background check is acceptable, then a 30 minute empty chamber count should be performed to determine if a new long background needs to be performed on that detector.

- 4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm². Therefore, the maximum acceptable mass density is typically 5 mg/cm² or less that 100 mg for a 2" planchet.
- 4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm² or greater.
- 4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|--|---------------------------------|---|---|
| Silver Nitrate | Poison Corrosive Oxidizer | 0.01 ^g /m ³ (TWA) for silver, metal dust, and fume as Ag | Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact may cause redness, pain, and severe burning. Eye contact can cause blurred vision, redness, and pain. |
| Ammonium Hydroxide | Poison Corrosive | 50 ppm (NH ₃) | Inhalation symptoms may include irritation to the respiratory tract. Ingestion symptoms may include pain in the mouth, chest, and abdomen with coughing, vomiting, and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |
| TWA – Time Weighted Average | | | |

6.0 EQUIPMENT AND SUPPLIES

6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.

- 6.2 Gas mixture, 90% argon, 10% Methane
- 6.3 Blank planchets
- 6.4 PC based data acquisition system, IQC software
- 6.5 Centrifuge tubes
- 6.6 Centrifuge
- 6.7 Vortex
- 6.8 Pipettes, Eppendorf or equivalent
- 6.9 Pipette, disposable

7.0 STANDARDS AND REAGENTS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision
- 7.2 Radioactive sources to measure beta radiation,: Sr-90 and Ra-228 sources.
- 7.3 Radioactive sources to measure alpha radiation: Am-241, Th-230 and Ra-226
- 7.4 Deionized Water (DI), obtained from the Milli-Q unit.
- 7.5 Silver nitrate (AgNO_3), 0.5 N
- 7.6 Sodium chloride (NaCl), crystals
- 7.7 Sodium chloride (NaCl), 0.5 N
 - 7.7.1 Add 50 mL of DI water to a 100 mL volumetric, add 5.84 g of NaCl , dilute to 100 mL, cap and shake to dissolve. Adjust volume to 100 mL with DI water.
- 7.8 Ammonium hydroxide (NH_4OH), concentrated, 28 N
- 7.9 Ammonium hydroxide (NH_4OH), 5 %
 - 7.9.1 Add 25 mL of concentrated Ammonium Hydroxide to 475 mL of DI water. **CAUTION – Ammonium hydroxide is corrosive. Mist and vapor cause burns to every area of contact.**

7.10 **CI-36:** At least four sodium chloride standards are prepared for calibration.

- 7.10.1 Add 10 mL of DI water to 4 centrifuge tubes.
- 7.10.2 Add 0.500 mL of 0.5 N sodium chloride carrier solutions to each centrifuge tube. Swirl to mix.
- 7.10.3 Add 2 drops of 5 % ammonium hydroxide solution, swirl to mix.
- 7.10.4 Add 12 mL of 0.5 N silver nitrate solution to each centrifuge tube.
- 7.10.5 Vortex for 30 seconds.
- 7.10.6 Centrifuge and decant supernate to waste.
- 7.10.7 Proceed to section 11.4, Planchet Preparation of Silver Chloride Precipitation of SOP ST-RC-0036.
- 7.10.8 Average the four weights for the sodium chloride carrier solution, record the standardized weight in the log book and on the bottle.
- 7.10.9 NOTE: It may be necessary to use more than 0.500 mL of carrier in some large water samples or calibrate a 4 N sodium chloride carrier solution. The efficiency of the detectors will have to be calculated using the heavier sodium chloride carrier solution.

- 7.10.10 Prepare four sodium chloride calibration samples as in Section 7.10 but add a known amount of Cl-36 to each tube before the sodium chloride carrier is added. Analyze samples by GFPC and determine detector efficiency as per Section 12, Data Analysis and Calculations.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041 and ST-RC -0050, for more detailed information.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 Batch

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 Method Blank (MB)

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 Laboratory Control Sample (LCS)

9.4.1 An LCS is a blank matrix spiked with a known amount of target analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every sample batch.

9.5 Matrix Spike

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 Sample Duplicate

9.6.1 A Sample Duplicate is an additional aliquot of a field sample, processed simultaneously with, and under the same conditions as, samples through all steps of the analytical process to demonstrate precision.

- 9.7 **Procedural Variations/ Nonconformance and Corrective Action**
- 9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Additional preventative maintenance can be found in ST-QA-0024.
- 10.2 Voltage Plateau Determination
- 10.2.1 **Frequency:**
- 10.2.1.1 Performed as a part of the Initial Calibration.
- 10.2.2 **Voltage Plateau Determination on Protean MPC 9604**
- 10.2.2.1 Place the Sr 90 source or sources in the detector drawer.
- 10.2.2.2 Select detector of interest on the computer screen.
- 10.2.2.3 Click Plateau under Count Method.
- 10.2.2.4 Set time to 5min for distributed Sr90 source.
- 10.2.2.5 Select A, B, C, D.
- 10.2.2.6 Click OK.
- 10.2.2.7 When count is complete select Plateau under instrument Specific.
- 10.2.2.8 Set Beta appropriate voltage with arrows </>. Evaluate and Print report.
- 10.2.3 **Criteria for Plateaus for Protean MPC 9604**
- 10.2.3.1 Acquire 40 data points in 30V increments beginning at 705V and ending at 1875V. Slope should be no more than 5%.
- 10.3 **Discriminator Settings**
- 10.3.1 **Frequency:**
- 10.3.1.1 Performed as a part of the Initial Calibration.
- 10.3.2 **Discriminator Settings on Protean MPC 9604**
- 10.3.2.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and/or Po-210 sources
- 10.3.2.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.
- 10.4 **Initial Calibration (IC):**
- 10.4.1 **Frequency:**
- 10.4.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated initially and verified each year thereafter. Recalibration may be required if indicated during the operation of the instrument.
- 10.4.2 The specific calibration source preparations can be found in the file containing the previous calibration.
- 10.4.3 All nuclide sources shall be NIST traceable.
- 10.4.4 The efficiency calibration shall consist of at least seven mass attenuated calibration standards, unless a single point source efficiency is to be determined.
- 10.4.5 Alpha, Beta Ra226 - at least seven mass attenuated calibration standards
- 10.4.6 Air Filter – single point calibration

- 10.4.7 CI-36 – Averaged 4 point calibration.
- 10.4.8 Np – Averaged 4 point calibration.
- 10.4.9 The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second.
 - 10.4.9.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.
- 10.4.10 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.
 - 10.4.10.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.
- 10.4.11 Each standard shall be counted in every detector to be calibrated.
- 10.4.12 **IC Criteria:**
 - 10.4.12.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.
 - 10.4.12.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.
 - 10.4.12.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.
 - 10.4.12.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.
 - 10.4.12.5 Points that are visual outliers or demonstrate less than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.
 - 10.4.12.6 The coefficient of determination (r^2) shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.5 **Independent Calibration Verification (ICV)**

- 10.5.1 **Frequency:**
 - 10.5.1.1 Performed with every initial calibration
- 10.5.2 GFPC initial calibrations must be verified by a second source standard.
- 10.5.3 The ICV standard is NIST traceable.
- 10.5.4 The ICV is counted to accumulate at least 5,000 counts.

Annual Calibration Verification (ACV)

10.5.5 **Frequency:**

- 10.5.5.1 Performed annually after every initial calibration
- 10.5.6 GFPC annual calibrations must be verified by a second source standard.
- 10.5.7 The ACV standard is NIST traceable.
- 10.5.8 The ACV is counted to accumulate at least 5,000 counts.

10.5.9 **ICV / ACV Criteria:**

- 10.5.9.1 Prepare 3 verification sources varying in expected mass (low, medium and high) within the calibration range of the curve, unless a single point source is to be determined.
- 10.5.9.2 Alpha and Beta 3 sources
- 10.5.9.3 Ra226 single source
- 10.5.9.4 Air Filter single source
- 10.5.9.5 Cl-36 – single source
- 10.5.9.6 Np- single source
- 10.5.9.7 The source/standard used for the ICV shall be from an independent second source as defined within the laboratory Quality Assurance Manual.”
 - 10.5.9.7.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.
- 10.5.9.8 Count the secondary source in all detectors that were calibrated.
- 10.5.9.9 Calculate the results in terms of percentage recovery.
- 10.5.9.10 Calculate the mean results of all masses across each detector.
- 10.5.9.11 **Criteria:**
 - 10.5.9.11.1 Individual points are within 30 percent of the true value
 - 10.5.9.11.2 The mean result of all masses across all detectors is less than 10 percent.
 - 10.5.9.11.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.6 **Setting Performance Check Criteria After Calibration**

- 10.6.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.
- 10.6.2 The limits for the background check sample will be established with five points from four months. Every month the oldest months points will be removed and points from the current month will be added.
- 10.6.3 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.
- 10.6.4 The limits for alpha/beta check sources will be a running average of the four months post calibration.
 - 10.6.4.1 The limits are to be documented.
 - 10.6.4.2 The limits will be re-established monthly at the following frequency
 - 10.6.4.2.1.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.
 - 10.6.4.2.1.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.

10.6.4.2.1.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.

10.6.4.2.1.4 4th month – take first five data points from new month and five points each from the previous three months.

10.6.4.3 Limits are set.

10.7 **Alpha to Beta Crosstalk Determination**

10.7.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.

10.7.1.1 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.

10.7.1.2 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.

10.7.1.3 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.

10.7.1.3.1.1 In this manner the crosstalk factor can be determined for any given mass.

10.7.1.4 The equation of the curve shall be determined using polynomial functions.

10.7.1.5 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.8 **Beta to Alpha Crosstalk Determination**

10.8.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.

10.8.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.

10.8.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

10.9 **Long Background**

10.9.1 **Frequency:**

10.9.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)

10.9.1.2 Minimum count time: 1000 minutes.

10.9.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.

10.9.2.1 Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.

10.9.3 Check that instrument settings are as specified in 11.1.

10.9.4 **Protean Long Background Count Set Up**

10.9.4.1 Create Manual batch in RadCapture

10.9.4.2 Export Manual batch from RadCapture

10.9.4.3 At Protean instrument:

- 10.9.4.4 Select 'Detector'
- 10.9.4.5 Select 'Sample Log'
- 10.9.4.6 Select appropriate Long Background (ex: ICB;00) you want to start under sample ID
- 10.9.4.7 Change count time to 1000min
- 10.9.4.8 Select 'Start'
- 10.9.4.9 Continue these steps with detectors 1-15. .
- 10.9.4.10 Review the data for acceptance when the backgrounds are complete.

10.9.5 **Printing Protean Long Backgrounds**

- 10.9.5.1 Select 'Print Protean data' icon on the desk top
- 10.9.5.2 Select OK
- 10.9.5.3 Enter Batch #
- 10.9.5.4 Print

10.9.6 **Protean Long Background Entry into Protean**

- 10.9.6.1 Select Input data
- 10.9.6.2 Select Definitions
- 10.9.6.3 Select Calibrations
- 10.9.6.4 Select Properties
- 10.9.6.5 Select References 0-7 for Detectors 0 thru 7 and 8-15 for Detectors 8 thru 15
- 10.9.6.6 Enter Background CPM's for Alpha and Beta from printed data sheet

10.9.7 **Orange and Purple Long Background Count Set-Up**

- 10.9.7.1 Select detector 0
- 10.9.7.2 Select 'source log'
- 10.9.7.3 Select 'ICB' by clicking on the file list arrow.
- 10.9.7.4 Ensure count time is set to 1000 minutes.
- 10.9.7.5 Select 'start'
- 10.9.7.6 Continue these steps with detectors 1-23.
- 10.9.7.7 Review the data for acceptance when the backgrounds are complete.

10.9.8 **Printing Orange and Purple Long Backgrounds**

- 10.9.8.1 Select 'Data
- 10.9.8.2 Select 'Source Count Data'
- 10.9.8.3 Select 'Source Name' ICB
- 10.9.8.4 Select 'This Range' enter your date range that Long Backgrounds were performed.
- 10.9.8.5 Select 'Refresh'
- 10.9.8.6 Select 'Source Count Summary' under Reports
- 10.9.8.7 Select 'Print'
- 10.9.8.8 Select 'Landscape' under Orientation
- 10.9.8.9 Select 'OK'

10.9.9 **Long Background Criteria:**

- 10.9.9.1 Long backgrounds are evaluated at ± 3 sigma.
- 10.9.9.2 Protean is evaluated at < 0.2 CPM for alpha and < 2.0 CPM for beta, due to the lack of capability to control chart for sigma evaluation.
- 10.9.9.3 The data report is evaluated per detector.
- 10.9.9.4 If a detector is above this limit, discard planchet.

10.9.9.5 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.

10.9.9.6 Place a clean planchet in the holder and repeat steps for that detector (s) only.

10.9.9.7 Perform a new background.

10.9.9.8 Note: The detector is tagged with an out of service tag noted with LMB and date. Detector is out of service until a successful background has been achieved.

11.0 PROCEDURES

11.1 Initial Setup

11.1.1 Check the normal instrument settings for all controls as described below:

11.1.1.1 Tank Flow 8 psi

11.1.1.2 Flow Cells ≥ 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.

11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Protean remains as set by the manufacturer and does not require adjustment.

11.1.3 If counting gas has just been changed or turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes on document on separate sheet.

11.2 Record date of Daily Background and Check Source Data in runlog logbook.

11.3 Maintenance

11.3.1 Change out the counting gas when the gauge reads under 500 psi. This usually occurs every 1 to 2 weeks. Record in the instrument maintenance logbook.

11.3.2 Allow gas to purge a minimum of 30 minutes prior to operation.

11.3.3 Background and checksource checks are required following a gas bottle change.

11.4 Data Acquisition: Daily Background Check and Source Check

11.4.1 Daily Background Check:

11.4.2 Protean Instrument:

11.4.2.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.

11.4.2.2 Double click detector 0 on the Protean computer screen.

11.4.2.3 Select 'source log'

11.4.2.4 Set the time for 200 minutes.

11.4.2.5 Type or scan 'CCB;00' in the sample id box. (B is for background and 0 is for detector.)

11.4.2.6 Select 'start'

11.4.2.7 Double click detector 1 on the computer screen. Repeat steps 11.4.2.3 through 11.4.2.5 for each detector, making sure to change the number to coincide with the detector the background is counting for.

11.4.2.8 Remove planchets from detector drawers when counting is complete.

11.4.2.9 On any work station, i.e. "PC computer in the count room", double click on the IQC icon.

11.4.2.10 Select 'import data'

11.4.2.11 Select 'Protean'. Enter the current date. Click on the file list arrow.

11.4.2.12 Select 'close'

11.4.2.13 Select 'reporting'. Verify the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report.

11.4.3 Orange and Purple Instrument:

11.4.3.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.

11.4.3.2 Select detector 0.

11.4.3.3 Select 'source log'.

11.4.3.4 Select or scan 'CCB' by clicking on the file list arrows for orange.

11.4.3.5 Select 'CCB' by clicking on the file list arrows for purple.

11.4.3.6 Select 'start'

11.4.3.7 Repeat these steps with detectors 1-23.

11.4.4 Daily Background Criteria:

11.4.4.1 Review the IQC report for each detector.

11.4.4.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-count.

11.4.4.1.2 Tag detector out of service with a Tag noted with "Bkg RC".

11.4.4.1.3 If detector fails Background re-count tag detector with an out of service tag noted with date to indicate that the detector is out of service for the day.

11.5 Daily Source Check

11.5.1 Protean Instrument:

11.5.1.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15 and slowly insert each drawer into the instrument.

11.5.1.2 Double click detector 0 on the Protean computer screen.

11.5.1.3 Select 'source log'.

11.5.1.4 Set the time for 2 minutes.

11.5.1.5 Type or scan "CCVA-"#";SA00" in the sample id box. ("#" is the source container, S is for source, A is for Alpha and 0 is the detector.)

11.5.1.6 Select 'start'

11.5.1.7 Double click detector 1 on the computer screen. Repeat steps 11.5.2.3 to 11.5.2.6 for each detector.

11.5.1.8 When the counting is complete, slowly open each detector drawer. Place beta sources in detectors 0-7. Place alpha sources in detectors 8-15.

11.5.1.9 Double click detector 0 on the Protean computer screen.

11.5.1.10 Type or scan "CCVB-"#";SB00" in the sample ID box. ("#" is the source container, S is for source, B is for Beta and 0 is the detector.)

11.5.2 Double click detector 1 on the computer screen. Repeat steps 11.5.2.10 for each detector.

11.5.2.1 Remove sources from detector drawers when counting is complete

11.5.2.2 Review the IQC report for each detector.

11.5.2.3 Limits are +/- 3% (fail).

11.5.2.4 If detector fails source check a Red tag is placed on the outside of the detector to indicate detector is out of service for the day.

11.5.3 Orange and Purple Instrument:

- 11.5.3.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.
- 11.5.3.2 Select detector 0.
- 11.5.3.3 Select 'source log'.
- 11.5.3.4 Select 'CCVA-“#”;SA00.
- 11.5.3.5 Select 'start'
- 11.5.3.6 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15 select 'CCVA-“#”;SA08', 'CCVA-“#”;SA09', and so on for each correlating detector number.
- 11.5.3.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 0-7 and place alpha sources in detectors 8-15.
- 11.5.3.8 Select detector 0.
- 11.5.3.9 Select 'CCVB-“#”;SB00'.
- 11.5.3.10 Select 'start'.
- 11.5.3.11 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15, select 'CCVB-“#”;SB08', 'CCVB-“#”;SB09' and so on for each correlating detector number.
- 11.5.3.12 Repeat steps 11.5.4.1 to 11.5.4.11 for detectors 16-23.
- 11.5.3.13 Remove sources from detector drawers when counting is complete.
- 11.5.3.14 Review the IQC report for each detector.
- 11.5.3.15 Limits $\pm 3\%$ (fail)
 - 11.5.3.15.1 The individual loading samples will verify that detectors are in service.

11.5.4 Daily Source Criteria:

- 11.5.4.1 Review and save with your name and date on the IQC report for each detector.
 - 11.5.4.1.1 If a detector fails criteria, re-count source.
 - 11.5.4.1.2 If detector fails source re-count tag detector with a Red out of service tag noted with date to indicate that the detector is out of service for the day

11.5.5 Daily check Criteria:

- 11.5.5.1 Review and save with your name and date on the IQC report.
 - 11.5.5.1.1 The individuals loading samples will verify that detectors are in-service prior to loading on them.
 - 11.5.5.1.2 In addition Daily checks will be verified at 1st level review of Data.
 - 11.5.5.1.3

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis ST-QAM.
- 12.3 To calculate the efficiency of the detectors for Cl-36, divide the net counts determined of the spiked Sodium Chloride, by the known dpm of the Standard used.

$$\frac{\text{Net Counts of Spiked Silver Chloride}}{\text{Known dpm of Cl} - 36(\text{decay corrected to day counted})} = \text{Efficiency}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank
- 13.2.1 Acceptance Criteria:
- 13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
- 13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.2.1 Method Blank Contamination – (e.g. reprep/reanalysis, narration). If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.
- 13.3 Laboratory Control Sample (LCS)
- 13.3.1 Acceptance Criteria:
- 13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
- 13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed..
- 13.3.2.2 LCS Spike Recovery excursion (low) the batch is re-prepped and re-analyzed for the affected isotope.
- 13.4 RPD/RER Duplicate excursion – For the RPD/RER One or both must be within acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narrated in an NCM.
- 13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- 13.5.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).
- 13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:
- 13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.
- 13.6 Sample Result Evaluation
- 13.6.1 Tracer/Carrier recovery must be within specified limits.
- 13.6.2 Tracer/Carrier recovery low– Samples must be reextracted. Exceptions can be made and results reported with approval from the technical director, manager, or client and appropriate NCM included.

- 13.6.3 Tracer/Carrier recovery high
 - 13.6.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:
 - 13.6.3.1.1 minimum number of tracer counts
 - 13.6.3.1.2 level of uncertainty
 - 13.6.3.1.3 client project requirements/approval
- 13.6.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

| Radium | Strontium | Chloride |
|--------|-----------|----------|
| 110% | 109% | 109% |

- 13.6.5 These expectations will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.
- 13.7 Insufficient Sample
 - 13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
- 16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 ANSI N42.25-1997 – American National Standard Calibration and Usage of Alpha/Beta Proportional Counters
- 17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay, Sr-02 Strontium 90, Sr-03-RC Strontium-90 in Environmental Samples.
- 17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry
- 17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes
- 17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium-228
- 17.6 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water
- 17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta
- 17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes
- 17.9 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium-228
- 17.10 TestAmerica St. Louis Quality Assurance Manual, current revision
- 17.11 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions
- 17.12 Associated SOPs, current revisions:
- 17.12.1 ST-PM-0002 "Sample Receipt and Chain of Custody"
- 17.12.2 ST-QA-0002, "Standards and Reagent Preparation."
- 17.12.3 ST-QA-0024, "Preventative Maintenance"
- 17.12.4 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"
- 17.12.5 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".
- 17.12.6 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
- 17.12.7 ST-RC-0021, "Gross Alpha Radition in Water using Coprecipitation"
- 17.12.8 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"
- 17.12.9 ST-RC-0040, "Total Alpha Emitting Isotopes of Radium"
- 17.12.10 ST-RC-0041, "Radium 228 in Water"
- 17.12.11 ST-RC-0050, "Preparation of Strontium-89 and 90"
- 17.12.12 ST-RC-0300, "New Jersey 48-hour Gross Alpha Testing for Private Well Testing ACT (PWTA)"

18.0 MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 TestAmerica St. Louis uses thorium-230 to calibrate the GFPC system for Ra-226. Th-230 has similar alpha energies and a sufficiently long half life to eliminate the need for purification. The laboratory has historically performed well on PE programs for Ra-226, demonstrating the laboratory's ability to accurately calibrate for this isotope. Calibrating with a Ra-226 source presents a severe bias in the quantitated result. Ra-226 can be purified and separated from all other alpha emitting isotopes, but the moment after separation, alpha emitting daughters begin to grow (i.e. radon-222, polonium-28 and polonium-214). As the daughter's in-growth alpha activity changes and due to the higher alpha energies of these daughters, the measured efficiency of the GFPC changes as well. After three weeks the alpha activity from purified Ra-226 increases by a factor of four. Due to their short half lives, these daughters can not be isolated long enough to mathematically correct for the bias brought on by them. Calibrating the GFPC with Ra-226 is actually calibrating with a mix of the four isotopes and not a legitimate calibration under the cited regulation.
- 18.2 Strontium-89 short half life makes it impractical to use as a calibration standard for both radium-228 analysis, as stated in EPA method 904 and SW method 9310, and strontium-89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed strontium-90/yttrium-90 standard for its' GFPC beta calibration used in Gross Beta, strontium-90, strontium-89, and radium-228 analyses. TestAmerica St. Louis has selected the strontium-90/yttrium-90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.
- 18.3 For Ra-228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.
- 18.4 TestAmerica St. Louis does not perform a direct strontium-89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total strontium and strontium-90.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated Section 10 to address voltage increase per step, plateau slope and QC check count requirements (5000 counts)
- 19.2 Rev. 11;
19.2.1 Added instument Purple throughout section 10 and 11.
19.2.2 Adjusted procedure steps throughout section 11.
- 19.3 Rev. 12,
19.3.1 Added Sr-02-RC and Sr-03-RC to sections 1.0 and 17.0.
- 19.4 Rev. 13:
19.4.1 Added Neptunium to scope in section 1.0.
19.4.2 Updated the Quality Control Program for counting daily rad checks and daily background checks in section 3.0.
19.4.3 Updated background count set-up, printing and entering protean data in section 10.8.
- 19.5 Rev. 14: (9/12/2013)
19.5.1 Removed references to Clouseau, SAC and QuantIMS
19.5.2 Section 5.0 added silver nitrate and ammonium hydroxide
19.5.3 Section 6.0 updated to include additional equipment
19.5.4 Section 7.0 updated to include addition reagents
19.5.5 Section 9.0 added reference to prep SOPs for additional information
19.5.6 Section 10.0 added sodium chloride standard preparation & reference to ST-QA-0024

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- 19.5.7 Section 12.0 added Cl-36 detector efficiency calculation
- 19.5.8 Section 13.0 updated to include actual corrective actions and native concentration carrier requirements
- 19.5.9 Section 13.0 updated to include corrective actions
- 19.5.10 Section 17.0 added reference to ST-QA-0024
- 19.6 Rev. 15: (1/16/2015)
 - 19.6.1 Added Section 7.10
 - 19.6.2 Updated Section 9.6.1
 - 19.6.3 Updated Section 10
 - 19.6.4 Updated Section 11
 - 19.6.5 Added ANSI N42.25-1997 reference to section 17
- 19.7 Rev. 16: (05/05/2015)
 - 19.7.1 Section 11.3.3 added Background and checks are needed following a tank change.

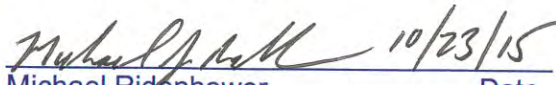
TestAmerica Preparation Procedure for PCBs for Wipe Samples

May 19, 2016

The sample preparation procedure for the extraction of PCBs of wipe samples is defined below.

1. Sample preparation is performed in the containers received.
2. Clean wipes for used for the Method Blank and Laboratory Control Sample. The LCS spike standard is added into the containers directly onto the wipe.
3. Surrogate standards are then added to all samples directly onto the wipe.
4. The appropriate amount of hexane is then added to all samples after the surrogate has been introduced. The volume of hexane added varies depending on the client container. A minimum of 30mL-100mLs is used- the wipe must be submerged in the solvent.
5. The caps are then tightened and the samples are placed on the autoshaker for 2 hours min. (4hrs max).
6. Samples are then removed from the autoshaker and transferred into a filtered KD setup, which includes rinsing the filter and associated sodium sulfate (approx 10g) with hexane. The sample aliquot is poured through and collected in the KD. The original container is rinsed with hexane. The wipe is rinsed one final time with hexane and allowed to dry. It is then returned to the original container and held for disposal for 30 days after completion of the analysis.
7. The KD apparatus is put on a bath and boiled to approx. 6-10mLs. The sample aliquot is then removed and allowed to cool. Hexane is added if needed for a final volume of 10mL or concentrated on the n-evap if it is slightly above 10mLs.
8. The final sample aliquot is collected and provided to the instrument analyst. The same analytical procedure for PCB wipes is followed as stated in SOP ST-GC-00015.

Title: PCB GC ANALYSIS
[SW-846 8000B/8000C/8082A; EPA 608]

| Approvals (Signature/Date): | | | |
|---|----------|--|----------|
|  | 10-23-15 |  | 10/23/15 |
| Jeff Winkler Extractions Supervisor | Date | Michael Ridenhower Health & Safety Manager / Coordinator | Date |
|  | 10-23-15 |  | 10/23/15 |
| Marti Ward Quality Assurance Specialist | Date | Elaine Wild Laboratory Director | Date |

This SOP was previously identified as SOP No. ST-GC-0015 Rev. 17

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes procedures to be used for the analysis of polychlorinated biphenyls (PCB) by GC/ECD. The PCBs are determined and quantitated as multi-component Aroclor mixes.
- 1.2 Sample preparation techniques are described in SOP ST-OP-0002.
- 1.3 This SOP is based on EPA SW-846 Methods 8000B, 8000C and 8082A, and EPA Method 608.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are prepared for analysis using the separatory funnel liquid / liquid extraction technique. Solid samples are prepared using sonication. Wipes are extracted by autoshaker.
- 2.2 After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The external standardization procedure is used.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Interferences in GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.
- 4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). For PCBs the most common cleanup procedure is the Sulfuric Acid cleanup (Method 3665A).
- 4.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, DDD) may interfere. When suspected, a standard of DDT analogs should be injected to determine which of the aroclor peaks may be subject to said interference, [DDT may interfere with the last major Aroclor 1254 peak in soil/sediment samples.]
- 4.4 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or high biased results.

- 4.5 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead to minimization of these interferences.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.3 PRIMARY MATERIALS USED
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (1) | Hazards | OSHA Exposure Limit (2) | Signs and symptoms of exposure/Unusual Hazards |
|--|---------------------------------|-------------------------|--|
| Hexane | Flammable Irritant | 500 ppm- (TWA) | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| Methanol | Flammable Poison Irritant | 200 ppm (TWA) | A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limits refer to the OSHA regulatory exposure limit. | | | |
| TWA – Time Weighted Average | | | |

6.0 EQUIPMENT AND SUPPLIES

- 6.1 GC/ECD system: The lab utilizes a Hewlett Packard GC 5890 dual ECD system and an Agilent GC 6890 dual micro ECD system with autosampler.

- 6.1.1 Columns used Restek Rtx-CLPesticides 30 meter, 0.53 mmID, 0.5 μ m df; Restek Rtx-CLPesticides2 30 meter, 0.52 mmID, 0.42 μ m df
- 6.1.2 GC column types, and instrument run conditions are posted in the maintenance calendar and reside in the Chemstation method.
- 6.2 Data System – Chemstation for acquisition and Chrom™ for data processing.
- 6.3 Amber and/or clear glass vials. Crimp top seals.
- 6.4 Disposal pipettes.
- 6.5 Micro syringes – 10- μ L, 250- μ L, 500- μ L, 1000- μ L. Hamilton 1700 series.
- 6.6 Volumetric flasks, Class A

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 See recipes for standards in the TALS Reagent module. Information listed in appendix to this SOP.
- 7.3 PCB primary standard solutions:
 - 7.2.1 Primary standards are prepared by dilution of neat liquid Aroclor mix 1016/1260, and from single aroclor mixes in hexane. Primary standards must be replaced after 6 months or the manufacturer's expiration date whichever is shorter. Standards must be stored in refrigerator or freezer at $\leq 6^{\circ}\text{C}$.
- 7.4 Working standards:
 - 7.7.1 The working standards are prepared in hexane from the primary standard solution for a minimum of five concentration levels of the Aroclor mix 1016/1260 and one level of the single aroclors. Working standards must be replaced after 6 months or manufacturer's expiration date whichever is shorter. All working standards expire after six months or at the expiration date of their stock standards, whichever comes sooner.
- 7.5 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up.
- 7.6 Decachlorobiphenyl (surrogate)
- 7.7 An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: 1-Bromo-2-Nitrobenzene
- 7.8 Internal Standards are added to all standards and extracts to result in 20 ng injected onto the column.
- 7.9 Copper powder
 - 7.9.1 Remove oxides (if powder is dark) by treating with dilute nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

- 7.10 DDT analog standard – used when possible interference from DDT analogs is present. This is a single standard containing DDT, DDE and DDD at a concentration judged sufficient by the analyst.
- 7.11 Initial Calibration Verification (ICV) spiking standard is similar to calibration standards, but are from a different source or vendor and are prepared and stored in the same way as calibration standards.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored at $4 \pm 2^{\circ} \text{C}$.
- 8.3 Soil samples are refrigerated at $4 \pm 2^{\circ} \text{C}$.
- 8.4 Extracts must be refrigerated at $\leq 6^{\circ} \text{C}$.
- 8.5 Sample extracts need to be isolated from all potential contaminants and all standards.

9.0 QUALITY CONTROL

- 9.1 **Batch**
 - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
 - 9.1.2 A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples, and/or 24 hours (12 hours for GC/MS).
 - 9.1.3 An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
 - 9.1.4 Instrument conditions must be the same for all standards, samples and QC samples.
 - 9.1.5 Each analytical batch may contain up to 20 environmental samples, a Method Blank (MB), a single Laboratory Control Sample (LCS) and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair. In the event that there is insufficient sample to analyze an MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.6 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual “sample batch” does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.
- 9.2 **Method Blank (MB)**
 - 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
 - 9.2.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
 - 9.2.3 DI water is used as the blank matrix for water batches.
 - 9.2.4 Sodium sulfate is used as the blank matrix for solid batches.

- 9.3 **Laboratory Control Sample (LCS)**
- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 An LCS must be prepared with every batch.
 - 9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.
 - 9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches
- 9.4 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**
- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.4.2 Additional MS/MSDs do not count towards the 20 samples in an analytical batch.
 - 9.4.3 An MS/MSD can be prepared with every batch, although it is not a method requirement. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.
- 9.5 **Procedural Variations/Nonconformance and Corrective Action**
- 9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Internal standard calibration is used.
- 10.2 Initial Calibration
- 10.2.1 Prepare an Aroclor 1016/1260 and Decachlorobiphenyl (surrogate) standard at a minimum of five concentration levels. (Six points are required if a quadratic (second order) curve is used.) The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 1. [NOTE: Quadratic regression is not allowed for South Carolina compliance work.]
 - 10.2.1.1 If a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix. See Client Requirements Sheet or Memo.
 - 10.2.1.2 A single point calibration for Aroclor 1221, 1232, 1242, 1248, 1254, 1262, and 1268 is performed with every initial calibration for pattern recognition.
 - 10.2.1.2.1 If any of the above single point Aroclors are detected, samples may be required to be re-analyzed under a five point calibration for the aroclor found. See Client Requirement Sheet to determine if this is needed.
 - 10.2.1.3 Select 3-5 major peaks in the analyte pattern. Calculate the response using the area of these individual peaks.
 - 10.2.1.3.1 Aroclor 1221 uses only 3 peaks due to the limited amount of peaks available to choose from.
 - 10.2.2 Add the internal standard mixture to result in 20 ng on column.
 - 10.2.3 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).
 - 10.2.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Corporate policy, "Calibration Point Selection", CA-Q-T-002.

- 10.3 SW 8082 criteria
 - 10.3.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$.
 - 10.3.2 If the %RSDs in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
 - 10.3.2.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.
 - 10.3.2.2 If a linear regression curve is used, r must be ≥ 0.99
 - 10.3.2.3 For South Carolina compliance work, forcing through zero is NOT allowed.
 - 10.3.2.4 Weighting of data points
 - 10.3.2.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it may be preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.
- 10.4 608 Criteria
 - 10.4.1 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit. The other standards define the working range of the detector.
 - 10.4.1.1 Refer to the TestAmerica Corporate policy, “Calibration Point Selection”, CA-Q-T-002.
 - 10.4.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 10\%$.
 - 10.4.3 If the %RSDs in the initial calibration is $> 10\%$, then calibration using a linear regression may be employed.
 - 10.4.3.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.
 - 10.4.3.2 If a linear regression curve is used, r must be ≥ 0.995
 - 10.4.3.3 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.
 - 10.4.3.3.1 Weighting of data points
 - 10.4.3.3.2 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.
- 10.5 Initial Calibration Verification (ICV)
 - 10.5.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.
 - 10.5.1.1 The ICV is not performed for the single point analytes.
 - 10.5.2 An ICV must be performed with every initial calibration.
 - 10.5.2.1 A passing ICV may be used as the opening CCV for a set of samples run following the ICV.

- 10.5.3 The ICV performance must be within +/- 20% D criteria.
 - 10.5.3.1 Only the analytes present in the ICAL are evaluated for the 20% criteria.
 - 10.5.3.2 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).
 - 10.5.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

- 10.6 Continuing Calibration Verification (CCV)
 - 10.6.1 A CCV must be run at the start of each 24 hour period.
 - 10.6.2 A CCV may be a second source or the same source as the initial calibration standards and should be made to represent the midpoint of the curve.
 - 10.6.3 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV).
 - 10.6.3.1 Analyte response factors must be verified after every 20 samples (10 samples for DOD) and at the end of the analysis run through the analysis of a CCV..
 - 10.6.4 It is adequate to verify calibration with a single mixture of Aroclors 1016 and 1260.
 - 10.6.4.1 For projects with specific Aroclor requirements, a specific Aroclor may be included in the daily calibration check.
 - 10.6.5 The calibration verification is acceptable if the %D for both 1016/1260 and the surrogate (DCB) is $\leq 20\%$.
 - 10.6.5.1 The same criterion is used if other Aroclor verifications are performed.
 - 10.6.5.2 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.
 - 10.6.5.3 If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed.
 - 10.6.5.4 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).

- 10.7 Retention Time (RT) Windows
 - 10.7.1 Retention Time (RT) windows must be determined for all analytes.
 - 10.7.2 Establishing RT windows:
 - 10.7.2.1 Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used).
 - 10.7.2.2 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.
 - 10.7.2.3 The center of the retention time window is the retention time from the average of three standards used to calculate the RT window.
 - 10.7.2.4 The center of the window is updated with the midpoint standard of the initial calibration.
 - 10.7.2.5 Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. Method 8000C also requires a new study when a column is clipped during maintenance.
 - 10.7.2.5.1 The new windows must be generated within one week of the installation of the new column.

10.7.2.5.2 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

- 10.7.3 Retention Time Criteria
 - 10.7.3.1 The retention times of AR 1016/1260 (and other aroclors if applicable) in each continuing calibration must be within the retention time windows established.
 - 10.7.3.2 The retention times of AR 1016/1260 (and other aroclors if applicable) are updated using the 1st CCV or RT marker of the day.
- 10.8 Method Detection Limit Studies
 - 10.8.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

- 11.1 Samples are prepared following ST-OP-0002
- 11.2 Allow standards, samples and sample extracts to reach ambient temperature before analysis.
- 11.3 Sulfur Removal
 - 11.3.1 Sulfur Removal with Copper Powder
 - 11.3.1.1 Transfer 1.0 mL of sample extract, and associated QC, into labeled vials.
 - 11.3.1.2 Add approximately 2g cleaned copper powder to the vial.
 - 11.3.1.3 Mix for one minute on a mechanical shaker.
 - 11.3.1.4 Allow phases to separate.
 - 11.3.1.5 Separate extract from copper by drawing the extract off with a disposable pipette.
 - 11.3.1.6 Transfer the supernate to a clean, labeled vial.
- 11.4 All analysis conditions and injection volumes for samples must be the same as for the calibration standards
- 11.5 Add internal standard to the extract to result in 20ng injected on column.
- 11.6 Sample Introduction
 - 11.6.1 Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.
- 11.7 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at $\leq 6^{\circ}\text{C}$, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Internal Standard Calculations
 - 12.2.1 *Analyte Concentration ($\mu\text{g/L}$) in sample*

Concentration ($\mu\text{g/L}$):

$$[C] = \frac{A_x * I_s * V_t * D}{A_{is} * RRF_{avg} * V_o * V_i}$$

Where:

| | | |
|-------------|---|---|
| $[C]$ | = | Analyte Concentration in sample ($\mu\text{g/L}$) |
| A_x | = | peak area or peak height |
| I_s | = | Amount of each internal standard injected (ng) |
| V_t | = | volume of concentrated extract (mL) |
| D | = | Dilution factor |
| A_{is} | = | Peak area or peak height of each internal standard |
| RRF_{avg} | = | Average relative response factor |
| V_o | = | Volume of water extracted (L) |
| V_i | = | Injection volume |

12.2.2 See Chrom software for additional calculations.

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica policy: CA-Q-S-002, "Manual Integrations". Manual integrations are denoted with an "M" flag on the Chrom quantitation report.

12.4 Identification of Aroclors

12.4.1 Tentative identification of an Aroclor occurs when multi-component peaks are found within their respective retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required.

12.4.2 Definitive Aroclor identification is based primarily on pattern recognition. Retention times and retention time windows are used to tentatively identify Aroclors, but the fingerprint produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.4.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. Source-specific information, such as historical data, indicating the anticipation of Aroclors must be documented. The pattern of peaks can serve as confirmation depending of the client specific project requirements.

12.5 Quantitation of Aroclors

12.5.1 Use three to five major peaks when calibrating Aroclors. Choose peaks distinctive of the individual Aroclor. Any manual integration made in the ICAL levels must be noted and be made in any subsequent samples to maintain consistency with the initial calibration. These same three to five peaks are then used to calculate the response/concentration of the Aroclor(s) when present in a sample.

12.5.1.1 For Aroclor 1221 only three peaks are used due to the limited number of peaks available.

12.5.1.2 In instances where less than five peaks are used those peaks that are not used are said to be "dropped" and an NCM must be written. When quantitating Aroclors 1016/1260 in an LCS/D and/or MS/MSD, all five peaks must be used. In

samples, less than the standard five peaks may be used to quantitate target analytes if there are demonstrated matrix interferences and/or if multiple, overlapping Aroclors are present. If there is a predominance of one Aroclor that elutes next to and shares peaks with another Aroclor and it is apparent to the analyst that the lesser Aroclor's concentration is elevated significantly by the more dominant Aroclor, then three peaks may be dropped and an NCM written. It is never allowable to quantitate an Aroclor using only one peak.

- 12.5.2 If well distinguishable Aroclor patterns are present, then multiple Aroclors are quantitated and reported.
 - 12.5.2.1 Aroclor elution times may overlap and one or more Aroclor peaks may be "shared" with another Aroclor. When this occurs, only the predominant Aroclor is quantitated and reported. Aroclors sharing elution time and peaks include: 1016, 1232, 1242, 1248 – these cannot be identified together (unless quantitating an MS/D where Aroclor 1016 is known to be present). Aroclors 1260 and 1262 also share peaks; only one of these can be reported (unless quantitating and MS/D where 1260 is known to be present).
- 12.5.3 Dual Column Quantitation
 - 12.5.3.1 Dual column confirmation is required for positive Aroclor identification. A secondary column using an alternate phase is employed and the sample is injected simultaneously into both a primary and secondary column. Elution times often differ, as does overall pattern/fingerprint of Aroclors between the two columns. Determination of target analytes on the secondary column is made in the same way as on the primary column. Target analytes may be reported from either column.
 - 12.5.3.2 Method 8000C states to report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. Method 8000B requires the reporting of the higher of the two columns. See Client Requirement Sheet for determination.
 - 12.5.3.2.1 For non-detect (ND) results, report from the primary channel if all QC and CCVs are acceptable. If the QC and CCVs are only acceptable on the secondary column, report the non-detects from this column.
 - 12.5.3.2.2 If the %D between the two columns is greater than 40%, report the higher result if there are obvious chromatographic interferences on the column with the lower result.
 - 12.5.3.2.3 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.
 - 12.5.3.2.3.1 If there is no evidence of chromatographic problems, report the lower result for method 8000C and the higher result for method 8000B. The data user should be advised of the disparity between the results on the two columns.
 - 12.5.3.2.3.2 Use the higher result if there is obvious chromatographic interference on the column with the lower result.
 - 12.5.3.3 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.
 - 12.5.3.4 The surrogate should be reported from the column used for the reporting of the sample results.

12.6 Dilutions

- 12.6.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample data is "E" flagged and the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over

50% of the high level standard) of the calibration range. Target analytes with resulting concentrations lower than the dilution adjusted RL should be flagged with a "J" qualifier.

12.6.2 It may be necessary to dilute samples due to matrix.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (MB)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet. South Carolina requires blank be below the RL. If blank > RL, take corrective action and re-analyze associated samples.

13.2.1.3 The Method Blank must have acceptable surrogate recoveries.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.2.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). . South Carolina requires the LCS to recover within limit of 70 -130%. If recovery is outside limits take corrective action and re-analyze associated samples.

13.3.1.2 The LCS must have acceptable surrogate recoveries.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS,

data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.3.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.5 Surrogate

13.5.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.5.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.5.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Dilutions

13.6.2 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution

13.1.1.2 Dilution: Surrogate(s) and/or spikes diluted out– Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution.

13.6.3 Carryover

13.6.3.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.6.3.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.6.3.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.6.4 Internal Standards

13.6.4.1 Acceptance Criteria

13.6.4.1.1 If the area for the internal standard in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, corrective action must be taken.

13.6.4.1.2 If the area for the internal standard in the samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some

programs require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)

- 13.6.4.2 Corrective Action for Internal Standards not meeting acceptance criteria:
- 13.6.4.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to the analytical result. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
 - 13.6.4.2.2 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples with positive hits above the RL for the analytes associated to the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.7 Insufficient Sample

- 13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report case narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
- 14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”
- 16.2 Waste Streams Produced by the Method

- 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
 - 16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
 - 16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.
 - 16.2.1.4 Expired primary and working PCB standards shall be segregated and placed into the proper satellite accumulation container specifically for PCB waste which is located within the GC lab.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B and 8000C.
- 17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8082A, 8081B Update IV, February 2007 and EPA 608 Method.
- 17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision
- 17.4 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision
- 17.5 TestAmerica Policy CA-Q-S-002, Manual Integrations
- 17.6 TestAmerica Policy CA-Q-T-002, Calibration Point selection
- 17.7 Associated SOPs
 - 17.7.1 ST-OP-0001, Labware Preparation for Organic Analysis
 - 17.7.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series
 - 17.7.3 ST-OP-0003, Extraction of PCB in Oil
 - 17.7.4 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
 - 17.7.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.7 ST-QA-0016, IDL/MDL Determination
 - 17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.7.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 MODIFICATIONS FROM REFERENCE METHOD

- 18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.
 - 18.1.1 Method Blanks for South Carolina compliance work MUST be below the RL
- 18.2 The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

- 18.3 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit.
- 18.4 SW846 Method 8000C requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated the table one regarding the levels of calibration of Aroclor 1016/1260 and the amount of the surrogate used, Decachlorobipenyl.
- 19.2 Rev 10:
19.2.1 Removing holding times for PCB's in section 8.0 per method
- 19.3 Revision 11:
19.3.1 Removed references to QuantIMS and Clouseau – replaced with LIMS.
19.3.2 Added software information to Section 6.
19.3.3 Added composition of Method Blank and LCS to Section 9.
19.3.4 Added requirement for 6 points for non-linear curves to Section 10.
19.3.5 Added Reporting limit calculations to Section 12
19.3.6 Added specific corrective actions to Section 13
19.3.7 Updated text in Section 15 to include methods beyond those approved by EPA.
- 19.4 Rev 12 (4/21/14):
19.4.1 Removed continuous extractions in section 2.
19.4.2 Replaced Target with Chrom data systems in section 6.
19.4.3 Updated location of instrument run setting in section 6.
19.4.4 Added location of standard recipes to section 7.
19.4.5 Updated retention time window study requirements in section 10.
19.4.6 Added exception to SW846 requirements in section 18.
19.4.7 Updated Initial Calibration sequence example following Table 1.
- 19.5 Revision 13 (9/18/14):
19.5.1 Section 18 updated to reflect requirements for Method Blanks associated with South Carolina compliance work.
19.5.2 Returned continuous liquid/liquid extraction to Section 2.1.
- 19.6 Revision 14 (10/30/14)
19.6.1 Revision done to combine this SOP with requirements for South Carolina compliance work originally found in SOP ST-GC-0015SC, Rev 1.
19.6.2 Updated Section 10 to include instruction that forcing through zero is NOT allowed for South Carolina compliance work.
19.6.3 Updated Section 10.7 to include requirements from both 8000B and 8000C regarding timing of running retention time window studies.
19.6.4 Added calculations to Section 12.

- 19.6.5 Updated Section 12.5.3 to include instruction from both 8000B and 8000C in regards to dual column reporting requirements.
- 19.6.6 Updated Section 13.3 to include South Carolina requirements for LCS recoveries.
- 19.7 Revision 15 (12/01/2014)
 - 19.7.1 Section 4 updated to address possible interference from the DDT analogs
 - 19.7.2 Section 6 updated to include specific columns used.
 - 19.7.3 Section 10 was updated to include a note that quadratic calibrations are not allowed for South Carolina compliance work; MDL study requirements add.
 - 19.7.4 Section 13 was updated to include the 70 – 130% LCS recovery requirement for South Carolina compliance work.
 - 19.7.5 Added standard preparation and concentration information to the appendices.
- 19.8 Revision 16 (04/24/2015)
 - 19.8.1 Added IS standards to Section 7.
 - 19.8.2 Update Section 10.
 - 19.8.2.1 Changed calibration on internal Section 10.1
 - 19.8.2.2 Added IS to initial calibration, Section 10.2
 - 19.8.2.3 Updated CCV requirements, Section 10.6
 - 19.8.2.4 Updated retention time updates, Section 10.7
 - 19.8.3 Included IS standards in procedure, Section 11.
 - 19.8.4 Updated acceptance criteria for IS, and surrogates, Section 13.
 - 19.8.5 Updated Appendix 1,
- 19.9 Revision 17 (06/12/2015)
 - 19.9.1 Removed reference to CLLE extraction in Section 2.1
 - 19.9.2 Removed external standard calculations from Section 12; add internal standard calculations.
- 19.10 Revision 18 (10/23/15)
 - 19.10.1 Corrected the CCV frequency requirement in Section 10.6.3.1.

| Table 1 | | | | | | | | |
|--|---------|---------------------------|---------|---------|-----------------|---------|---------|--|
| Calibration Levels ng/ml | | | | | | | | |
| | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | |
| Aroclor 1016/1260 | 50 | 100 Reporting Limit | 250 | 500 | 1000 ICV/CCV | 2000 | 4000 | |
| The surrogate is included with all calibration mixes at the following levels | | | | | | | | |
| Decachlorobiphenyl | 2.5 | 5 | 12.5 | 25 | 50 | 100 | 200 | |

* Level 1 is optional.

Aroclors 1232, 1221, 1242, 1248, 1254, 1262 and 1268 may be quantitated within the range 100 to 4000 ng/mL

Analytical Sequence

Example Initial Calibration

Injection

| | | |
|-------|---|------------|
| 1 | Conditioning standard | |
| 2 | Solvent blank | |
| 3 | Aroclor 1221 | |
| 4 | Aroclor 1232 | |
| 5 | Aroclor 1242 | |
| 6 | Aroclor 1248 | |
| 7 | Aroclor 1262 | |
| 8 | Aroclor 1268 | |
| 9-15 | Aroclor 1254 | Levels 1-7 |
| 16 | Independent Calibration Verification (ICV) standard | |
| 17-23 | Aroclor 1016/1260 | Levels 1-7 |
| 24 | Independent Calibration Verification (ICV) standard | |
| 25-34 | Sample Injections (max.10) | |
| 35 | Aroclor 1016/1260 | Level 5 |

Appendix 1

Standards:

Surrogate Stock – 1000 ug/mL

IS Standard Stock – 100ug/mL

Aroclors Stock – 1000 ug/mL

(1221, 1232, 1242, 1248, 1254, 1016/1260, 1262, 1268

Intermediates:

Aroclor(s) Intermediate (excluding 1016/1260) – 1 mL Aroclor stock + 50 µl of Surrogate stock to 25 mL with hexane = 40 ug/mL Aroclor/0.2 ug/ml surrogate

IS Intermediate – 0.5 mL IS stock to 500 mL with hexane = 1 ug/mL

Working Standards:

1016/1260 working – 1 ml of 1016/1260 stock + 50 µ of Surrogate stock to 250 ml with hexane = 4 ug/ml

1016.1260/0.2 ug/ml surrogate

Aroclor(s) working – 5 ml of aroclor intermediate to 20 ml with hexane = 4 ug/ml aroclor/0.2 ug/ml surrogate

Calibration:
Aroclor 1016/1260 and 1254

| Calibration point | Dilution of Working Std. | Concentration (ug/ml) |
|---------------------|--------------------------|-----------------------|
| 1 | 80x | 0.05 |
| 2 | 40x | 0.10 |
| 3 | 20x | 0.20 |
| 4 | 8x | 0.50 |
| 5 | 4x | 1.00 |
| 6 | 2x | 2.00 |
| 7 | 1x | 4.00 |
| ICV (second source) | 4x | 1.00 |

Calibration:
Aroclor 1221, 1232, 1242, 1248, 1262, 1268

| Calibration point | Dilution of Working Std. | Concentration (ug/ml) |
|-------------------|--------------------------|-----------------------|
| 5 | 4x | 1.00 |



Reagent ID: 8082SurrDeca_00018

| | | | |
|-------------------|-------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/28/2015 |
| Description: | Decachlorobiphenyl Solution | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Saulters, Camme N |
| Storage Location: | Organic Prep Standards Storag | Vendor: | Ultra Scientific |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | CC-3147Z |
| Creation Date: | 08/26/2013 | Vendor Cat #: | PPS-150 |
| Container(s): | 190362 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-------------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| DCB Decachlorobiphenyl (Surr) | | | | | 1004.00000 | ug/mL |



Reagent ID: 8082 Spike_00018

| | | | |
|-------------------|-------------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 06/01/2019 |
| Description: | 8082 spike arochlor 1060/1260 (PCB) | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Saulters, Camme N |
| Storage Location: | Organic Prep Standards Storag | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A094177 |
| Creation Date: | 08/26/2013 | Vendor Cat #: | 32039 |
| Container(s): | 190372 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|----------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1016 | | | | | 1000.00000 | ug/mL |
| PCB-1260 | | | | | 1000.00000 | ug/mL |



Reagent ID: Aroclor 1254_00010

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 08/31/2016 |
| Description: | Aroclor 1254 Solution | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Ultra Scientific |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | CE-2632 |
| Creation Date: | 01/15/2013 | Vendor Cat #: | PP-352 |
| Container(s): | 74708 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1254 | | | | | 100.00000 | ug/mL |
| PCB-1254 Peak 1 | | | | | 100.00000 | ug/mL |
| PCB-1254 Peak 2 | | | | | 100.00000 | ug/mL |
| PCB-1254 Peak 3 | | | | | 100.00000 | ug/mL |
| PCB-1254 Peak 4 | | | | | 100.00000 | ug/mL |
| PCB-1254 Peak 5 | | | | | 100.00000 | ug/mL |



Reagent ID: Ar 1221_00002

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 12/31/2018 |
| Description: | Aroclor 1221 Standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A090667 |
| Creation Date: | 07/08/2013 | Vendor Cat #: | 32007 |
| Container(s): | 158536 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1221 | | | | | 1000.00000 | ug/mL |
| PCB-1221 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1221 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1221 Peak 3 | | | | | 1000.00000 | ug/mL |



Reagent ID: Ar 1268_00004

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 12/31/2019 |
| Description: | Aroclor 1268 Standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A097795 |
| Creation Date: | 06/09/2014 | Vendor Cat #: | 32410 |
| Container(s): | 381748 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1268 | | | | | 1000.00000 | ug/mL |
| PCB-1268 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1268 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1268 Peak 3 | | | | | 1000.00000 | ug/mL |
| PCB-1268 Peak 4 | | | | | 1000.00000 | ug/mL |
| PCB-1268 Peak 5 | | | | | 1000.00000 | ug/mL |



Reagent ID: Ar 1242_00002

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 11/30/2018 |
| Description: | Aroclor 1242 Standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A090182 |
| Creation Date: | 07/08/2013 | Vendor Cat #: | 32009 |
| Container(s): | 158544 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1242 | | | | | 1000.00000 | ug/mL |
| PCB-1242 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1242 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1242 Peak 3 | | | | | 1000.00000 | ug/mL |
| PCB-1242 Peak 4 | | | | | 1000.00000 | ug/mL |
| PCB-1242 Peak 5 | | | | | 1000.00000 | ug/mL |



Reagent ID: Ar 1232_00002

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 11/30/2018 |
| Description: | Aroclor 1232 | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A090290 |
| Creation Date: | 07/08/2013 | Vendor Cat #: | 32008 |
| Container(s): | 158538 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1232 | | | | | 1000.00000 | ug/mL |
| PCB-1232 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1232 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1232 Peak 3 | | | | | 1000.00000 | ug/mL |
| PCB-1232 Peak 4 | | | | | 1000.00000 | ug/mL |
| PCB-1232 Peak 5 | | | | | 1000.00000 | ug/mL |

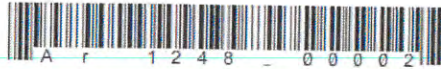


Reagent ID: Ar 1262_00002

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 06/30/2019 |
| Description: | Aroclor 1262 Standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A094073 |
| Creation Date: | 07/08/2013 | Vendor Cat #: | 32409 |
| Container(s): | 158599 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1262 | | | | | 1000.00000 | ug/mL |
| PCB-1262 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1262 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1262 Peak 3 | | | | | 1000.00000 | ug/mL |
| PCB-1262 Peak 4 | | | | | 1000.00000 | ug/mL |
| PCB-1262 Peak 5 | | | | | 1000.00000 | ug/mL |







Reagent ID: Ar 1248_00002

| | | | |
|-------------------|-----------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 04/30/2019 |
| Description: | Aroclor 1248 Standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Hunt, Joseph B |
| Storage Location: | GC and HPLC Standards Stora | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A092864 |
| Creation Date: | 07/08/2013 | Vendor Cat #: | 32010 |
| Container(s): | 158550 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| PCB-1248 | | | | | 1000.00000 | ug/mL |
| PCB-1248 Peak 1 | | | | | 1000.00000 | ug/mL |
| PCB-1248 Peak 2 | | | | | 1000.00000 | ug/mL |
| PCB-1248 Peak 3 | | | | | 1000.00000 | ug/mL |
| PCB-1248 Peak 4 | | | | | 1000.00000 | ug/mL |
| PCB-1248 Peak 5 | | | | | 1000.00000 | ug/mL |

**Title: GC/MS SEMIVOLATILES ANALYSIS
 [SW-846 8270D; EPA 625]**

| Approvals (Signature/Date): | |
|--|--|
|  Date | 11-16-15 |
| Jeff Winkler Extractable Dept. Supervisor | |
|  Date | 2015.11.15 19:45:17 -06'00' |
| Tony L. Byrd Quality Assurance Manager | |
|  Date | 2015.11.16 09:19:01 -06'00' |
| Mike Ridenhower Health & Safety Manager / Coordinator | |
|  Date | Elaine Wild cn=Elaine Wild, o, ou, email=elaine.wild@testamericainc.c om, c=US 2015.11.16 08:30:36 -06'00' |
| Elaine Wild Laboratory Director | |

This SOP was previously identified as SOP No. ST-MS-0001 Rev. 19

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.
- 1.2 This SOP is based on SW-846 Method 8000B, 8000C and 8270D and EPA method 625.
- 1.3 The following compounds are documented in the method as problematic:
 - 1.3.1 Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
 - 1.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
 - 1.3.3 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
 - 1.3.4 Hexachlorophene may not be amenable to analysis by this method.
- 1.4 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from Diphenylamine.
- 1.5 3-Methylphenol cannot be separated from 4-Methylphenol by the conditions specified in this method.
- 1.6 Phthalic acid decomposes in the gas chromatographic inlet and cannot be distinguished from Phthalic anhydride.
- 1.7 Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be reported as Azobenzene.
- 1.8 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.8.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel. Continuous liquid-liquid extraction may also be used. Solid samples are extracted with methylene chloride / acetone using sonication. Waste dilution is used for organic or unusual matrix samples. The sample extract is concentrated to a volume of 1 mL, 5 mL or 10 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.
- 2.2 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electro impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Instead of scanning everything in a retention time range, SIM looks for specific ions (qualitative and quantitative) that are placed in retention time groups. The ions used for qualitative and quantitative purposes are the same for scan and SIM analysis. SIM is not allowed for South Carolina work, as the laboratory does not hold certification there for the SIM method.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 SIM –Selected Ion Monitoring

4.0 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.
- 4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.
- 4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.
- 4.4 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

- 5.2.1 Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile, Silver Shield, or similar gloves must be used.
- 5.2.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- 5.2.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|--|------------------------|------------------------------------|--|
| Methylene Chloride | Carcinogen Irritant | 25 ppm (TWA) 125 ppm (STEL) | Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |
| TWA – Time Weighted Average | | | |
| STEL – Short Term Exposure Limit | | | |

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Gas Chromatograph/Mass Spectrometer System: HP 6890/5973 - An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source. Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in [Table 1](#) when 50 ng of the GC/MS tuning standard is injected through the GC.
- 6.1.1 Column: Restek RXI-5Sil MS, 30 meters, 0.25mm ID, 0.25 μ m df
- 6.2 Data System:
- 6.2.1 ChemStation software system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the length of the chromatographic program.
- 6.2.2 Target software system allows the searching of any GC/MS data file for ions of a specified mass and plots such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software allows integrating the abundances in any EICP for a specified time or scan-number limit. Also, for the non-target compounds with a mass spectrum that meets the required criteria, software must be available that allows for the comparison of sample spectra against the reference library spectra.
- 6.2.3 Data Library: NIST05
- 6.3 Carrier gas: Ultra high purity helium
- 6.4 Instrument columns and run conditions are posted in the instrument maintenance calendar.
- 6.5 Amber vials. Crimp top seals
- 6.6 Disposal pipettes
- 6.7 Micro syringes- 10 μ L, 250 μ L, 500 μ L, 1000 μ L. Hamilton 1700 series, Agilent Gold Standard
- 6.8 Volumetric flasks, Class A
- 6.9 Analytical Balance, capable of weighing \pm 0.01 grams.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

- 7.2 See recipes for standards and QC samples in the LIMS Reagent Log program. See Appendix 1 of this SOP
- 7.3 At a minimum, a five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to [Table 3](#) for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.
- 7.4 An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.
- 7.5 Internal Standards are added to all standards and extracts to result in 40 ng injected onto the column. SIM Analysis Internal Standards are added to all standards and extracts to result in 4 ng injected onto the column.
- 7.6 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared.
- 7.7 ICV standards, NIST traceable:
- 7.7.1 The Semivolatile ICV standard is a second source from the calibration standard, where a second viable source is available.
- 7.7.2 ICV standard is prepared and stored in the same way as calibration standards.
- 7.8 Standards are to be refrigerated at $\leq 6^{\circ}\text{C}$ when not in use. Refrigeration at less than -10°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least 6 months after opening.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are collected in amber glass, unpreserved and stored at $4 \pm 2^{\circ}\text{C}$.
- 8.3 Soil samples are refrigerated at $4 \pm 2^{\circ}\text{C}$.
- 8.4 The extraction holding time for Semivolatiles analysis in waters is 7 days.
- 8.5 The extraction holding time for Semivolatiles in soil/solid matrix is 14 days.
- 8.6 Extracts must be refrigerated at $\leq 6^{\circ}\text{C}$ and analyzed within 40 days of the beginning of the extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

- 9.2 **Method Blank (MB)**
- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
 - 9.2.2 A method blank must be prepared with every sample batch.
 - 9.2.3 DI water is used for the Method Blank.
 - 9.2.4 Sodium sulfate is used as the method blank for solid matrices.
- 9.3 **Laboratory Control Sample (LCS)**
- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 An LCS must be prepared with every sample batch.
 - 9.3.3 The LCS is comprised of sodium sulfate fortified with the target analyte(s).
- 9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**
- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.5 **Surrogate**
- 9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
 - 9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.
- 9.6 **Procedural Variations/ Nonconformance and Corrective Action**
- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Internal standard calibration is used.
- 10.1.1 Internal Standard Calibration Procedure: Internal standards are listed in [Table 5](#). Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.
 - 10.1.1.1 Compounds are assigned to the IS, generally with the closest retention time. See [Table 5](#).
- 10.2 **Instrument Tuning**
- 10.2.1 The GC/MS system must be checked to see if acceptable performance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See [Table 1](#) in this SOP.
 - 10.2.1.1 The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.
 - 10.2.1.2 **8270** - At the beginning of every twelve hour shift.
 - 10.2.1.3 **625** - At the beginning of every 24 hour shift.
 - 10.2.1.3.1 The time period begins at the moment of injection of DFTPP.
 - 10.2.2 Inject 50 ng of the GC/MS tuning standard into the GC/MS system. Obtain a background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in [Table 1](#) are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.2.3 Degradation of DDT to DDE and DDD should not exceed 20%.

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD \% DDE)}}{\text{sum of all peak areas (DDT \% DDE \% DDD)}} \times 100$$

10.2.4 Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{Tailing Factor} = BC/AB$$

Where the peak is defined as follows:

AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height.

10.3 **Initial Calibration**

- 10.3.1 Prepare calibration standards at a minimum of five concentration levels, six points for a quadratic fit, (see [Table 3](#) for suggested concentrations) for each parameter of interest. It may be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector.
- 10.3.2 Add the internal standard mixture to result in 40 ng on column. The concentrations of all analytes are listed in [Table 3](#). Add the internal standard mixture to result in 4ng on column for SIM analysis.
- 10.3.3 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. The low level standard must be at or below the reporting limit.
- 10.3.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Calibration Curves."
- 10.3.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.
- 10.3.6 A new calibration curve must be generated after major changes to the system and may be required when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., cleaning the ion source).
- 10.3.7 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).
- 10.3.8 Structural isomers that produce very similar mass spectra (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution is visibly verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification (CCV) level.
- 10.3.9 **Initial Calibration Criteria (8270D)**
- 10.3.9.1 Minimum Response Factors
- 10.3.9.2 See [Table 4](#) in this SOP for the minimum response factors. These minimum response factors are prescribed by SW method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response factor of 0.01 for compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.

- 10.3.9.2.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.
- 10.3.9.3 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.
- 10.3.9.4 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.
- 10.3.9.5 **Linear calibration using the average response factor**
- 10.3.9.5.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.
- 10.3.9.5.2 If the %RSD in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.3.9.6 **Linear calibration using a least squares regression**
- The intercept of a linear calibration at zero response (i.e. the y-intercept) must have an absolute value less than the reporting limit for each analyte. Client requirements may be tighter, please check Client Requirement Memorandum (CRM) if identified in comments.
- Note**, for analyses utilizing an internal standard the Target variable “b” does NOT equal the y-intercept. For analyses utilizing an internal standard, the Target variable “b” must be multiplied by the associated internal standard concentration to derive the concentration at the y-intercept.
- 10.3.9.6.1 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.3.9.6.2 When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve.
- 10.3.9.6.3 It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations.
- 10.3.9.6.4 The recalculated concentration of the low calibration point should be within $\pm 30\%$ of the standard's true concentration.
- 10.3.9.6.4.1 Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered “out of control” and corrective action should be taken.
- 10.3.9.7 **Linear calibration using a least squares regression, forcing thru zero**
- 10.3.9.7.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.
- 10.3.9.7.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2
- 10.3.9.7.3 For samples requiring adherence to method 8000B, forcing through zero is **NOT** allowed. This includes South Carolina compliance work.
- 10.3.9.8 **Linear calibration using a least squares regression, weighting of data points**
- 10.3.9.8.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration; for this reason it may be preferable to increase the weighting of

the lower concentration points, $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting), to improve accuracy at the low end of the curve.

10.3.9.8.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2

10.3.9.9 Non-linear calibration

10.3.9.9.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed.

10.3.9.9.2 The use of non-linear calibrations or second order regression calibrations are not allowed for South Carolina compliance samples.

10.3.9.9.3 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.9.9.3.1 These compounds are not to use non-linear calibrations:

- 1,4-Dioxane; Pyridine; n-Nitrosodimethylamine;
- 2-Fluorophenol; Aniline; Bis(2-chloroethyl)ether; Phenol-d5;
- Phenol; 2-Chlorophenol; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,2-Dichlorobenzene; Benzyl Alcohol;
- 2-Methylphenol; N-nitrosodipropylamine; Hexachloroethane; 3 and 4-Methylphenol; Nitrobenzene-d5; Nitrobenzene; Isophorone;
- 2-Nitrophenol;
- 2,4-Dimethylphenol; Bis(2-chloroethoxy) methane;
- 2,4- Dichlorophenol; 1,2,4-Trichlorobenzene; Naphthalene;
- Hexachlorobutadiene; 4-Chloro-3-Methylphenol;
- 2-Methylnaphthalene; 2,4,6-Trichlorophenol;
- 2-Fluorobiphenyl; 2,4,5-Trichlorophenol; 2-Chloronaphthalene;
- Dimethylphthalate; Acenaphthylene; Acenaphthene; Dibenzofuran;
- Diethylphthalate; Fluorene; 4-Chlorophenyl-phenylether; N-Nitrosodiphenylamine; Azobenzene; 4-Bromophenyl-phenylether;
- Hexachlorobenzene; Phenanthrene; Anthracene; Carbazole; Di-n-Butylphthalate; Fluoranthene; Pyrene; Terphenyl-d14;
- Butylbenzylphthalate; Benzo(a)Anthracene; Chrysene;
- bis(2-ethylhexyl)Phthalate; 2-Picoline;
- n-Nitrosomethylethylamine; Methyl methanesulfonate;
- n-Nitrosodiethylamine; Ethyl Methanesulfonate; Pentachloroethane;
- Acetophenone; n-Nitrosopyrrolidine;
- n-Nitrosomorpholine; O-Toluidine; n-Nitrosopiperidine; o,o,o-Triethyl-Phosphorothioate; 2,6-Dichlorophenol;
- Hexachloropropene; Benzothiazole;
- n-Nitrosodi-n-butylamine; Safrole;
- 1,2,4,5-Tetrachlorobenzene; cis-Isosafrole; trans-Isosafrole; 1,4-Dinitrobenzene; 1,3-Dinitrobenzene; Pentachlorobenzene; 1-Naphthylamine; 2-Naphthylamine; Thionazin; 5-Nitro-o-toluidine;
- Tri-n-butylphosphate; Sulfotepp; Diallate; Phorate; Phenacetin; Tris (2-chloroethyl) phosphate; 4-Aminobiphenyl; Pronamide;
- Pentachloronitrobenzene; Disulfoton; Parathion; Isodrin; Aramite;
- p- (Dimethylamino) azobenzene; Chlorobenzilate; 2-Acetylaminofluorene; 4,4'-Methylenebis (2)-Chloroaniline; 7,12-Dimethylbenz (a) anthracene;
- 3-Methylcholanthrene; Isosafrole; Octachlorostyrene;
- Methyl methacrylate;
- Ethyl methacrylate; Benzaldehyde; Caprolactam;
- 1-Methylnaphthalene; Biphenyl; Atrazine.

- 10.3.8.9.2.2 EPA Method 8000C suggests a 20% RSD limit be used when evaluating a calibration. The above compound list was constructed based on the 20% RSD criteria. TestAmerica St. Louis reserves the right to employ different calibration models when client mandated criteria are less than the 20% criteria found in method 8000C.
- 10.3.9.9.4 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.
- 10.3.9.9.5 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.3.9.9.6 Due to the nature of SIM analysis, non-linear calibrations may be used.
- 10.3.10 **625 criteria**
- 10.3.10.1 Method 625 only requires a 3 point calibration. We routinely perform a 6 point calibration; however, 3 points may be removed from the curve if necessary to meet 625 calibration criteria.
- 10.3.10.1.1 Refer to the TestAmerica corporate policy, “Calibration Curves.”
- 10.3.10.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $< 35\%$.
- 10.3.10.3 If the %RSD in the initial calibration is $> 35\%$, then calibration using a linear regression may be employed.
- 10.3.10.4 If a linear regression curve is used, the intercept of the curve at zero response must be less than \pm the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.
- 10.3.10.5 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.
- 10.3.10.6 Weighting of data points
- 10.3.10.6.1 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.
- 10.4 **Initial Calibration Verification (ICV)**
- 10.4.1 An initial calibration verification standard is a different standard source than the one used for the initial calibration.
- 10.4.2 An ICV must be performed with every initial calibration.
- 10.4.3 The ICV performance must be within $\pm 30\%$ D criteria.
- 10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
- 10.5 **Continuing Calibration Verification (CCV)**
- 10.5.1 At the start of each 12 hour period (8270) or 24 hour period (EPA 625) the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria. See [Table 1](#) in this SOP.

- 10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.
- 10.5.3 A CCV standard is analyzed every analysis tune clock immediately following the DFTPP tune.
- 10.5.3.1 **EPA 8270** – for each 12 hour tune time period
- 10.5.3.2 **EPA 625** – for each 24 hour tune time period
- 10.5.4 The CCV can be the same source or a second source from the calibration.
- 10.5.5 The internal standard response must be within 50-200 area counts (-50% to 100%) of the response in the mid level of the initial calibration. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- 10.5.6 **EPA 8270 Criteria**
- 10.5.6.1 Minimum Response Factors
- 10.5.6.2 See [Table 4](#) in this SOP for the minimum response factors. These minimum response factors are prescribed by SW-846 method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response factor of 0.01 per compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.
- 10.5.6.3 The CCV performance must be with $\pm 20\%$ D criteria.
- 10.5.6.4 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).
- 10.5.7 **EPA 625 Criteria**
- 10.5.7.1 For each target analyte %D must be $< 20\%$.
- 10.5.8 Calibration excursions are to be documented via a NCM.
- 10.6 Retention Time (RT) windows
- 10.6.1 Relative Retention Time (RRT)
- 10.6.1.1 In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.
- 10.6.1.2 The RRT is expressed as a unit-less quantity:
- $$\text{RRT} = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$
- 10.6.1.3 The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units.
- 10.6.1.4 It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus, care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.
- 10.6.1.5 If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.
- 10.6.1.6 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard and the target compounds are related to the internal

standards on the basis of the similarity of their respective chromatographic retention times (see [Table 5](#)).

10.6.2 Internal standard retention time

10.6.2.1 The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.3 Retention Time Criteria

10.6.3.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.7 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Samples are prepared following ST-OP-0002.

11.2 South Carolina requires a separate certification for SIM analysis. At this time TestAmerica St. Louis does not hold that certification. SIM analysis can not be used for South Carolina compliance samples.

11.3 South Carolina requires a separate certification for LVI (Limited Volume Extraction). At this time TestAmerica St. Louis does not hold that certification. LVI analysis can not be used for South Carolina compliance work.

11.4 All standards and extracts are allowed to warm to room temperature before injecting.

11.5 All samples must be analyzed using the same instrument conditions as the initial calibration.

11.6 Add internal standard to the extract to result in 40ng injected on column. Mix thoroughly before injection into the instrument.

11.6.1 Add internal standard to the extract to result in 4ng injected on column for SIM analysis.

11.7 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.8 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.9 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate at -10°C to -20°C (if it can be demonstrated that analytes do not fall out of solution at this temperature), protected from light in screw cap vials equipped with un-pierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 External Standard Calculations

12.1.1 See instrument software (Target/Chrom) for calculations.

12.2 Manual Integrations

12.2.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with "M" flag on the Target quantitation report. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices.

12.3 Qualitative identification

12.3.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

12.3.1.1 Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

The following analytes should be carefully reviewed:

| | | |
|------------------------------|------------------------|---------------------------|
| 1,4-Dichlorobenzene-d4 | Aniline | Bis (2-Chloroethyl) ether |
| 1,3-Dichlorobenzene | 1,4-Dichlorobenzene | 1,2-Dichlorobenzene |
| Benzyl alcohol | 2-Methylphenol | 3,4-Methylphenol |
| 2,4-Dichlorophenol | 2,4,6-Trichlorophenol | 2,4,5-Trichlorophenol |
| Phenanthrene | Anthracene | Benz (a) anthracene |
| Bis (2-ethylhexyl) phthalate | Chrysene | Di-n-octyl phthalate |
| Benzo (b) fluoranthene | Benzo (k) fluoranthene | Indeno (1,2,3-cd) pyrene |
| Benzo (g,h,i) perylene | p-Phenylenediamine | Safrole |
| Cis-Isosafrole | Trans-Isosafrole | 1,4-Dinitrobenzene |
| 1,3-Dinitrobenzene | 1-Naphthylamine | 2-Naphthylamine |
| 2,3,4,6-Tetrachlorophenol | Dinoseb | Sulfotepp |
| Diallate 1 & 2 | Methapyrilene | Aramite 1 & 2 |

12.3.2 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.3.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.3.4 The relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance should be between 20 and 80 percent.)

12.3.4.1 See [Table 2](#) for primary, secondary and tertiary ion assignments.

12.3.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.3.6 Retention time criteria for samples

12.3.6.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

12.3.6.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

12.4 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client.

- 12.4.1 TICs are done as follows:
- 12.4.1.1 The computer will give quality matches in order from most likely to least likely. In order for us to call a TIC a certain compound, the quality match must be at least 90%. However, if the next two quality matches are within (around) 10% quality match of the first choice, the compound will be identified as an unknown because it is too close to call. Unknowns are put into a group if possible (such as Unknown alkanes) but if a group is not available it will be called Unknown. A compound will be also called unknown if the top three matches are all different groups of compounds and the quality match is < 90% (ex. If the top choice is an alkane, the second choice is an alcohol, the third choice is an acid).
- 12.4.1.2 The first 30 TICs, based on abundance, will be identified in a sample, unless a different number is specified by the client. See client requirement sheet.

12.5 Dilutions

- 12.3.7 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.
- 12.5.1 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.
- 12.5.2 Samples may be diluted initially if the project reporting limits are above the laboratory's routine calibration lower limit, if there is physical evidence of matrix, or historical knowledge of the site.

12.6 Carryover

- 12.6.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
- 12.6.1.1 If a sample analyzed after a sample with high concentrations has negative results or is non-detect, carryover did not occur.
- 12.6.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.
- 12.6.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 This SOP lists requirements for the standard Quality Assurance criteria followed at TestAmerica St. Louis. If a client or program requires stricter quality controls (i.e. DoD, DOE, SC DHEC) the analyst is directed to the Client Requirement Memo for that client/project for limits.
- 13.2 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Steps taken for out-of-control situations include demonstrating that the cause of the out-of-control situation was addressed and demonstration that a return to control was obtained.
- 13.3 Method Blank
- 13.3.1 Acceptance Criteria:
- 13.3.1.1 No target analytes may be present in the method blank above the reporting limit.
- 13.3.1.2 The method blank must have acceptable surrogate recoveries.
- 13.3.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.3.1.3.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.
- 13.3.1.3.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate

recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3.1.3.3 For South Carolina compliance work, the Method Blank concentration must be below the RL.

13.4 Laboratory Control Sample (LCS)

13.4.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.1.1 For long analyte spike lists, marginal exceedances (ME) are allowed as follows:

13.4.1.2 less than 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

13.4.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

13.4.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.

13.4.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.

13.4.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.

13.4.1.7 More than 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.

13.4.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.

13.4.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

13.4.1.10 Marginal exceedance is not allowed by all programs. See Project/Program CRM for details. The use of marginal exceedances is not allowed for South Carolina Compliance samples.

13.4.2 The LCS should have acceptable surrogate recoveries.

13.4.3 Corrective Action for LCS not meeting acceptance criteria:

13.4.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.4.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.4.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prepare and re-analysis is required.

13.4.3.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prepare and re-analysis are required.

13.6 Sample result evaluation

13.6.1 Dilutions

13.6.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.6.1.2 Dilution: Sample– An NCM is created when dilutions are required.

13.6.1.3 Dilution: Surrogate(s)/spikes diluted out– An NCM is generated to document the surrogates/spikes being diluted out.

13.6.2 Carryover

- 13.6.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
- 13.6.2.2 If a sample analyzed after a sample with high concentrations is non-detect for the high concentration analyte, carryover did not occur.
- 13.6.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
- 13.6.3 Internal Standards
 - 13.6.3.1 Acceptance Criteria:
 - 13.6.3.1.1 If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, corrective action must be taken.
 - 13.6.3.1.2 If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs may require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)
 - 13.6.3.2 Corrective Action for Internal Standards not meeting acceptance criteria:
 - 13.6.3.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to analytical results. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
 - 13.6.3.2.2 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples that are non-detect for affected analytes may be reported with an NCM. Samples with positive hits above the RL for analytes associated with the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
- 13.6.4 Surrogate
 - 13.6.4.1 All Surrogates should be within established control limits for accuracy (%Recovery).
 - 13.6.4.2 Corrective Action for Surrogate not meeting acceptance criteria:
 - 13.6.4.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.
- 13.7 Insufficient Sample
 - 13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, an NCM is created and a narrative comment stating such is included in the report's Case Narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.
- 14.3 Training Qualification

- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Auto-sample vials containing Methylene Chloride are to be disposed of in the appropriate solvent vial waste accumulation container located within the GC/MS lab, for temporary storage. Once this temporary container is full or once it reaches a one-year collection time, this container must be dumped into the permanent solvent vial waste container located in the 90-day storage area, which is marked as a Type "C" waste accumulation container.
 - 16.2.1.2 Waste Methylene Chloride rinses are to be collected and disposed of within the solvent waste accumulation container located in the Organic Prep. Lab. This temporary storage container shall be dumped on a daily basis into the permanent waste accumulation container located in the 90-day storage area which is marked as a Type "D" waste drum.

17.0 REFERENCES

- 17.1 SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8000B, 8000C and 8270D.
- 17.2 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July 1, 1995, Method 625.
- 17.3 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision.
- 17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-002) and St. Louis Facility Addendum (ST-HS-0002), current revision.
- 17.5 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 17.6 TestAmerica Policy CA-T-P-002, Selection of Calibration Points

- 17.7 Associated SOPs, current revisions
 - 17.7.1 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series Methods
 - 17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.6 ST-QA-0016, IDL/MDL, LOD/LOQ Determination
 - 17.7.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

19.0 CHANGES TO PREVISION SOP REVISION

- 19.1 Table reference in Section 6.1 was corrected.
- 19.2 Y-intercept requirements added to Section 10.
- 19.3 Added requirement for 6 levels for a quadratic curve to Section 10
- 19.4 Added CLP allowance for reporting data within 10% of upper standard without dilution to Section 12
- 19.5 Clarification of criteria for TIC reporting added to Section 12.4.
- 19.6 [Table 1](#): clarified Tune criteria and added allowance of other published DFTPP Tune criteria (i.e. EPA CLP)
- 19.7 Added [Table 5](#), a listing of internal standards and associated analytes
- 19.8 Revision 13:
 - 19.8.1 Grammatical /spelling corrections
 - 19.8.2 Added SIM analysis to section 11
- 19.9 Revision 14:
 - 19.9.1 Removed QuantIMS and Clouseau references – replaced with LIMs
 - 19.9.2 Created hyperlinks to tables
 - 19.9.3 Appended LVI Calibration Levels to [Table 3](#)
 - 19.9.4 Combined fragmented [Table 5](#) into one table
 - 19.9.5 Added table of potentially mis-identifiable analytes to Section 12.3.
 - 19.9.6 Removed CLP allowance for reporting data within 10% of upper standard without dilution from Section 12.
 - 19.9.7 Revised Section 13 to remove Clouseau corrective action references and to provide specific corrective actions for non-conformances.
- 19.10 Revision 15:
 - 19.10.1 Section 3, updated SIM definition
 - 19.10.2 Section 7.5 Added SIM requirement
 - 19.10.3 Section 7.7 ICV standard 2nd source where available to acquire
 - 19.10.4 Section 10.1 corrected table references
 - 19.10.5 Section 10.2 Added % breakdown calculation and added Benzidine and pentachlorophenol requirements
 - 19.10.6 Section 10.3.8.9.2.1 removed compounds that are not to use non-linear calibration model
 - 19.10.7 Removed 12.6.1.3
 - 19.10.8 Section 13.6.2.3 removed chromatographic profile reference
- 19.11 Revision 16:

- 19.11.1 Section 1.1 Added a missing period
- 19.11.2 Section 10.2.1.2 Added a missing period
- 19.11.3 Section 10.2.2 Added a missing period
- 19.11.4 Section 10.5.2 Added a missing period
- 19.11.5 Section 12.2.1 Added a missing period
- 19.11.6 Section 13.4.3.1 Added a missing period
- 19.11.7 Section 13.4.3.4 Added a missing period
- 19.11.8 Section 10.3.8.2 Took comma out after Kepone and added the word “and”
- 19.11.9 Section 10.5.6.2 Took comma out after Kepone
- 19.11.10 Section 17.5 Changed policy number from 001 to 002
- 19.11.11 Section 2.1 Added possible concentration of 5 mL
- 19.11.12 Added Surrogates to Section 13
- 19.11.13 Table 3 – Changed calibration concentration levels
- 19.12 Revision 17:
 - 19.12.1 Added note to Section 11 that South Carolina requires certification for SIM analysis.
 - 19.12.2 Updated Section 13.4 to disallow the use of marginal exceedance for South Carolina compliance work.
 - 19.12.3 Updated Section 10.3.8 to disallow the use of non-linear or second order calibrations for South Carolina compliance work.
- 19.13 Revision 18 (11/3/14):
 - 19.13.1 Combined SOP with SOP ST-MS-0001SC Rev 1 to include the South Carolina DHEC requirements.
 - 19.13.2 Table 7 added to SOP - containing information on standards and reagents.
- 19.14 Revision 19 (12/1/14)
 - 19.14.1 Added liquid-liquid continuous extraction to Section 2.1.
 - 19.14.2 Added column used to Section 6
 - 19.14.3 Updated Section 10.3 to clarify South Carolina requirements regarding forcing through zero
 - 19.14.4 Added Section 10.7 to list MDL information
 - 19.14.5 Added Section 11.3 to disallow LVI procedure for South Carolina compliance work
 - 19.14.6 Added standard preparation and concentration information to the SOP appendix
- 19.15 Revision 20 (11/16/15)
 - 19.15.1 Added section 10.3.8 to provide a reference to 8270D isomer resolution being <50%
 - 19.15.2 Edited section 10.7.1 to remove reference to using dual column

Table 1
DFTPP Key Ions and Ion Abundance Criteria*

| Mass | Ion Abundance Criteria |
|-------------|------------------------------------|
| 51 | 30 - 60% of mass 198 |
| 68 | <2% of mass 69 |
| 70 | <2% of mass 69 |
| 127 | 40 - 60% of mass 198 |
| 197 | <1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5 - 9% of mass 198 |
| 275 | 10 - 30% of mass 198 |
| 365 | >1% of mass 198 |
| 441 | Present, but less than mass 443 |
| 442 | >40% of mass 198 |
| 443 | 17 - 23% of mass 442 |

* Tune criteria in use is a combination of 8270C and 8270D which is more stringent than either method. Alternatively, other documented tuning criteria (e.g. EPA CLP) may be used provided method performance is not adversely affected.

Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

| Primary Standard | | | |
|---|----------------|------------------|-----------------|
| Analyte | Primary | Secondary | Tertiary |
| 1,4 Dioxane | 88 | 58 | 43 |
| n-Nitrosodimethylamine | 74* | 42 | 44 |
| Pyridine | 79 | 52 | — |
| Dimethylformamide | 44 | 73 | 42 |
| Cyclohexanol | 57 | 82 | 67 |
| 2-Fluorophenol (Surrogate Standard) | 112 | 64 | 63** |
| Phenol-d5 (Surrogate Standard) | 99 | 42 | 71 |
| Aniline | 93 | 66 | 65 |
| Phenol | 94 | 65 | 66 |
| Bis(2-chloroethyl)ether | 93 | 63 | 95 |
| 2-Chlorophenol | 128 | 64 | 130 |
| 1,3-Dichlorobenzene | 146 | 148 | 111 |
| 1,4-Dichlorobenzene-d4 (Internal Standard) | 152 | 150 | 115 |
| 1,4-Dichlorobenzene | 146 | 148 | 111 |
| Benzyl Alcohol | 108 | 79 | 77 |
| 1,2-Dichlorobenzene | 146 | 148 | 111 |
| 2-Methylphenol | 108* | 107 | 79 |
| 2,2'-oxybis(1-chloropropane) ¹ | 45 | 77 | 121 |
| 3&4-Methylphenol | 107 | 108 | 79 |
| n-Nitroso-di-n-propylamine | 70 | 42 | 101 |
| Hexachloroethane | 117 | 201 | 199 |
| Nitrobenzene-d5 (Surrogate Standard) | 82 | 128 | 54 |
| Nitrobenzene | 77 | 123 | 65 |
| Isophorone | 82 | 95 | 138 |
| 2-Nitrophenol | 139 | 65 | 109 |
| 2,4-Dimethylphenol | 107* | 121 | 122 |
| Benzoic Acid | 122 | 105 | 77 |
| Bis(2-chloroethoxy)methane | 93 | 95 | 123 |

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Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

| Primary Standard | | | |
|--|----------------|------------------|-----------------|
| Analyte | Primary | Secondary | Tertiary |
| 2,4-Dichlorophenol | 162 | 164 | 98 |
| 1,2,4-Trichlorobenzene | 180 | 182 | 145 |
| Naphthalene-d8 (Internal Standard) | 136 | 68 | 54** |
| Naphthalene | 128 | 129 | 127 |
| 4-Chloroaniline | 127 | 129 | 65 |
| Hexachlorobutadiene | 225 | 223 | 227 |
| 4-Chloro-3-methylphenol | 107 | 144 | 142 |
| 2-Methylnaphthalene | 142 | 141 | — |
| Hexachlorocyclopentadiene | 237 | 235 | 272 |
| 2,4,6-Trichlorophenol | 196 | 198 | 200 |
| 2,4,5-Trichlorophenol | 196 | 198 | 200 |
| 2-Fluorobiphenyl (Surrogate Standard) | 172 | 171 | — |
| 2-Chloronaphthalene | 162 | 164 | 127 |
| 2-Nitroaniline | 65 | 92 | 138 |
| Dimethylphthalate | 163 | 194 | 164 |
| Acenaphthylene | 152 | 151 | 153 |
| 2,6-Dinitrotoluene | 165 | 63 | 89 |
| Acenaphthene-d10 (Internal Standard) | 164 | 162 | 160 |
| 3-Nitroaniline | 138 | 108 | 92 |
| Acenaphthene | 153* | 152 | 154 |
| 2,4-Dinitrophenol | 184 | 63 | 154 |
| Dibenzofuran | 168 | 139 | — |
| 4-Nitrophenol | 109* | 139 | 65 |
| 2,4-Dinitrotoluene | 165 | 63 | 89 |
| Diethylphthalate | 149 | 177 | 150 |
| Fluorene | 166 | 165 | 167 |
| 4-Chlorophenylphenylether | 204 | 206 | 141 |
| 4-Nitroaniline | 138 | 92 | 108 |
| 4,6-Dinitro-2-methylphenol | 198 | 105 | 51 |

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Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

| Primary Standard | | | |
|--|----------------|------------------|-----------------|
| Analyte | Primary | Secondary | Tertiary |
| n-Nitrosodiphenylamine | 169 | 168 | 167 |
| 2,4,6-Tribromophenol (Surrogate Standard) | 330 | 332** | 141 |
| Azobenzene | 77 | 51** | 105 |
| 4-Bromophenylphenylether | 248 | 250 | 141 |
| Hexachlorobenzene | 284 | 142 | 249 |
| Pentachlorophenol | 266 | 264 | 268 |
| Phenanthrene-d10 (Internal Standard) | 188 | 94 | 80 |
| Phenanthrene | 178 | 179 | 176 |
| Anthracene | 178 | 179 | 176 |
| Carbazole | 167 | 166 | 139 |
| Di-n-butylphthalate | 149 | 150 | 104 |
| Fluoranthene | 202 | 101 | 203 |
| Benzidine | 184 | 92 | 185 |
| Pyrene | 202 | 200 | 203 |
| Terphenyl-d14 (Surrogate Standard) | 244 | 122 | 212 |
| Butylbenzylphthalate | 149 | 91 | 206 |
| Benzo(a)Anthracene | 228 | 229 | 226 |
| Chrysene-d12 (Internal Standard) | 240 | 120 | 236 |
| 3,3'-Dichlorobenzidine | 252 | 254 | 126 |
| Chrysene | 228 | 226 | 229 |
| Bis(2-ethylhexyl)phthalate | 149 | 167 | 279 |
| Di-n-octylphthalate | 149 | 167 | 43 |
| Benzo(b)fluoranthene | 252 | 253 | 125 |
| Benzo(k)fluoranthene | 252 | 253 | 125 |
| Benzo(a)pyrene | 252 | 253 | 125 |
| Perylene-d12 (Internal Standard) | 264 | 260 | 265 |
| Indeno(1,2,3-cd)pyrene | 276 | 138 | 277 |
| Dibenz(a,h)anthracene | 278 | 139 | 279 |
| Benzo(g,h,i)perylene | 276 | 138 | 277 |

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* primary/secondary and/or tertiary ions are switched from order in Method
 ** not listed in the method

Appendix IX Standard

| Analyte | Primary | Secondary | Tertiary |
|---|----------------|------------------|-----------------|
| Methyl methacrylate | 69 | 41 | 39 |
| Ethyl methacrylate | 69 | 41 | 39 |
| 2-Picoline | 93 | 66 | 92 |
| n-Nitrosomethylethylamine | 88 | 42 | 43 |
| Methyl methanesulfonate | 80 | 79 | 65 |
| 2-Fluorophenol (Surrogate Standard) | 112 | 64 | 63** |
| n-Nitrosodiethylamine | 102 | 44 | 57 |
| Ethyl methanesulfonate | 79 | 109 | 97 |
| Benzaldehyde | 77 | 106 | 51 |
| Phenol-d5 (Surrogate Standard) | 99 | 42 | 71 |
| Pentachloroethane | 117 | 119 | 167 |
| 1,4-Dichlorobenzene-d4 (Internal Standard) | 152 | 150 | 115 |
| Acetophenone | 105 | 77 | 120 |
| n-Nitrosopyrrolidine | 100 | 41 | 42 |
| n-Nitrosomorpholine | 116 | 56 | 86 |
| o-Toluidine | 106 | 107 | — |
| Nitrobenzene-d5 (Surrogate Standard) | 82 | 128 | 54 |
| n-Nitrosopiperidine | 114 | 42 | 55 |
| O,o,o-Triethyl-Phosphorothioate | 198 | 121 | 93 |
| a,a-Dimethyl-phenethylamine | 58 | 91 | — |
| Naphthalene-d8 (Internal Standard) | 136 | 68 | 54** |
| 2,6-Dichlorophenol | 162 | 164 | 63 |
| Hexachloropropene | 213 | 215 | 211 |
| Benzothiazole | 135 | 108 | 69 |
| Caprolactam | 55 | 113 | 42 |
| p-Phenylenediamine | 108 | 80 | — |
| n-Nitrosodi-n-butylamine | 84 | 57 | 41 |
| Safrole | 162 | 104 | 77 |
| Phthalic anhydride | 104 | 76 | 50 |
| 1-methylnaphthalene | 142 | 141 | 115 |

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Appendix IX Standard

| Analyte | Primary | Secondary | Tertiary |
|--|---------|-----------|----------|
| 1,2,4,5-Tetrachlorobenzene | 216 | 214 | 218 |
| Isosafrole, cis | 162 | 104 | 131 |
| 2-Fluorobiphenyl (Surrogate Standard) | 172 | 171 | — |
| Isosafrole, trans | 162 | 104 | 131 |
| Biphenyl | 154 | 153 | 152 |
| 1,4-Dinitrobenzene | 168 | 75 | 50 |
| 1,4-Naphthoquinone | 158 | 104 | 102 |
| 1,3-Dinitrobenzene | 168 | 75 | 76 |
| Acenaphthene-d10 (Internal Standard) | 164 | 162 | 160 |
| Pentachlorobenzene | 250 | 248 | 252 |
| 1-Naphthylamine | 143 | 115 | — |
| 2-Naphthylamine | 143 | 115 | — |
| 2,3,4,6-Tetrachlorophenol | 232 | 230 | 131 |
| 5-Nitro-o-toluidine | 152 | 77 | 106 |
| Thionazin | 107 | 96 | 143 |
| 1,3,5-Trinitrobenzene | 213* | 75 | 120 |
| 2,4,6-Tribromophenol (Surrogate Standard) | 330 | 332** | 141** |
| Sulfotepp | 97 | 322 | 202 |
| Phorate | 75 | 97 | 121 |
| Phenacetin | 108 | 179 | 109 |
| Diallate 1 | 86 | 234 | 43 |
| Diallate 2 | 86 | 234 | 43 |
| Dimethoate | 87 | 93 | 125 |
| 4-Aminobiphenyl | 169 | 168 | 170 |
| Pentachloronitrobenzene | 237 | 142 | 214 |
| Phenanthrene-d10 (Internal Standard) | 188 | 94 | 80 |
| Pronamide | 173 | 175 | 145 |
| Disulfoton | 88 | 97 | 89 |
| 2-secbutyl-4,6-dinitrophenol (Dinoseb) | 211 | 163 | 147 |
| Methyl parathion | 109 | 125 | 263 |
| 4-Nitroquinoline-1-oxide | 190 | 128 | 75 |
| Parathion | 109 | 97 | 291 |
| Isodrin | 193 | 66 | 195 |

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Appendix IX Standard

| Analyte | Primary | Secondary | Tertiary |
|---|----------------|------------------|-----------------|
| Kepone | 272 | 274 | 237 |
| Methapyrilene | 97 | 58** | — |
| Octachlorostyrene | 308 | 343 | 154 |
| Terphenyl-d14 (Surrogate Standard) | 244 | 122 | 212 |
| Aramite 1 | 185 | 319 | — |
| Aramite 2 | 185 | 319 | — |
| p-(Dimethylamino)azobenzene | 120* | 225 | 77 |
| p-Chlorobenzilate | 251 | 139 | 253 |
| 3,3'-Dimethylbenzidine | 212 | 106 | — |
| 2-Acetylaminofluorene | 181 | 180 | 223 |
| Famphur | 218 | 125 | 93 |
| Chrysene-d12 (Internal Standard) | 240 | 120 | 236 |
| Hexachlorophene | 196 | 198 | 209 |
| 7,12-Dimethylbenz(a)anthracene | 256 | 241 | 120 |
| Perylene-d12 (Internal Standard) | 264 | 260 | 265 |
| 3-Methylcholanthrene | 268 | 252 | 126 |

* primary/secondary and/or tertiary ions are switched from order in Method

** not listed in the method

Table 3
Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|---|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 1,4 Dioxane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pyridine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosodimethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dimethylformamide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Cyclohexanol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Aniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Bis(2-chloroethyl)ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Chlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzyl alcohol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,2'-oxybis(1-chloropropane) ¹ | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3&4-Methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitroso-di-n-propylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachloroethane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Nitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isophorone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Nitrophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dimethylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzoic acid | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| bis(2-Chloroethoxy)methane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2,4-Trichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Naphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Chloroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorobutadiene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Table 3
Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|-----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 4-Chloro-3-methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Methylnaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorocyclopentadiene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,6-Trichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,5-Trichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Chloronaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dimethyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acenaphthylene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acenaphthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dinitrophenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| 4-Nitrophenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| Dibenzofuran | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dinitrotoluene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,6-Dinitrotoluene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Diethylphthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Chlorophenyl phenyl ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Fluorene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4,6-Dinitro-2-methylphenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| N-Nitrosodiphenylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Azobenzene ² | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Bromophenyl phenyl ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenanthrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Carbazole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Di-n-butyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Table 3
Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Benzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Butyl benzyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3,3'-Dichlorobenzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(a)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Bis(2-ethylhexyl)phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Chrysene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Di-n-octylphthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(b)fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(k)fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(a)pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Indeno(1,2,3-cd)pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dibenz(a,h)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(g,h,i)perylene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

¹ 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

³ Lower concentration standards may be analyzed on a project specific basis.

Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|---------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Methyl methacrylate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Ethyl methacrylate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Picoline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosomethylethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methyl methanesulfonate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosodiethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Ethyl methanesulfonate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzaldehyde | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|---------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Pentachloroethane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acetophenone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosopyrrolidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosomorpholine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| o-Toluidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosopiperidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| O,o,o-Triethyl-Phosphorothioate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| A,a-Dimethyl-phenethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,6-Dichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachloropropene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzothiazole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Caprolactam | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| p-Phenylenediamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosodi-n-butylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Safrole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phthalic anhydride | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1-Methylnaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2,4,5-Tetrachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isosafrole, cis | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Isosafrole, trans | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Biphenyl | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Dinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Naphthoquinone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3-Dinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1-Naphthylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Naphthylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,3,4,6-Tetrachlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 5-Nitro-o-toluidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Thionazin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3,5-Trinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Sulfotepp | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phorate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|--|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Phenacetin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Diallate 1 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Diallate 2 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Dimethoate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Aminobiphenyl | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachloronitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pronamide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Disulfoton | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-sec butyl-4,6-dinitrophenol (Dinoseb) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methyl parathion | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Nitroquinoline-1-oxide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Parathion | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isodrin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Kepone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Famphur | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methapyrilene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Octachlorostyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Aramite 1 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Aramite 2 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| p-(Dimethylamino)azobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| p-Chlorobenzilate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3,3'-Dimethylbenzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorophene | 10 | 20 | 50 | 100 | 200 | 300 | 400 | 500 | 600 |
| 2-Acetylaminofluorene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dibenz (a,j)acridine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 7,12-Dimethylbenz(a)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3-Methylcholanthrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Fluorophenol (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenol-d5 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Nitrobenzene-d5 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Fluorobiphenyl (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,6-Tribromophenol (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Terphenyl-d14 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Calibration Levels SIM Standard, ug/mL

| | | | | | | |
|------------------------|-----|-----|-----|-----|-----|------|
| Naphthalene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Acenaphthylene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Acenaphthene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Fluorene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Phenanthrene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Pyrene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Benzo(a)anthracene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Chrysene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Benzo(b)fluoranthene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Benzo(k)fluoranthene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Benzo(a)pyrene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Indeno(1,2,3-cd)pyrene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Dibenz(a,h)anthracene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Anthracene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Fluoranthene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| Benzo(g,h,i)perylene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |
| 2-Methylnaphthalene | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 | 10.0 |

Table 3

LVI Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|-------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 1,4 Dioxane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pyridine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|---|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| n-Nitrosodimethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dimethylformamide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Cyclohexanol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Aniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Bis(2-chloroethyl)ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Chlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzyl alcohol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2-Dichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,2'-oxybis(1-chloropropane) ¹ | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3&4-Methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitroso-di-n-propylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachloroethane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Nitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isophorone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Nitrophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dimethylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzoic acid | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| bis(2-Chloroethoxy)methane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2,4-Trichlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Naphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Chloroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorobutadiene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Chloro-3-methylphenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Methylnaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorocyclopentadiene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,6-Trichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,5-Trichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|-----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 2-Chloronaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dimethyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acenaphthylene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acenaphthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dinitrophenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| 4-Nitrophenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| Dibenzofuran | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4-Dinitrotoluene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,6-Dinitrotoluene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Diethylphthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Chlorophenyl phenyl ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Fluorene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Nitroaniline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4,6-Dinitro-2-methylphenol | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 100 | 120 |
| N-Nitrosodiphenylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Azobenzene ² | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Bromophenyl phenyl ether | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenanthrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Carbazole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Di-n-butyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Butyl benzyl phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3,3'-Dichlorobenzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(a)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Bis(2-ethylhexyl)phthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

| Analyte | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Chrysene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Di-n-octylphthalate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(b)fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(k)fluoranthene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(a)pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Indeno(1,2,3-cd)pyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dibenz(a,h)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzo(g,h,i)perylene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

¹2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

²Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

³Lower concentration standards may be analyzed on a project specific basis.

LVI Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|---------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Methyl methacrylate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Ethyl methacrylate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Picoline | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosomethylethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methyl methanesulfonate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosodiethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Ethyl methanesulfonate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzaldehyde | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachloroethane | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Acetophenone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosopyrrolidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosomorpholine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| o-Toluidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosopiperidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| O,o,o-Triethyl-Phosphorothioate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| A,a-Dimethyl-phenethylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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LVI Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 2,6-Dichlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachloropropene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Benzothiazole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Caprolactam | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| p-Phenylenediamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| n-Nitrosodi-n-butylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Safrole | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phthalic anhydride | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1-Methylnaphthalene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,2,4,5-Tetrachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isosafrole, cis | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Isosafrole, trans | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Biphenyl | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Dinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,4-Naphthoquinone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3-Dinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachlorobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1-Naphthylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Naphthylamine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,3,4,6-Tetrachlorophenol | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 5-Nitro-o-toluidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Thionazin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 1,3,5-Trinitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Sulfotepp | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phorate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenacetin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Diallate 1 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Diallate 2 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Dimethoate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Aminobiphenyl | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pentachloronitrobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Pronamide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Disulfoton | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

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LVI Calibration Levels, Appendix IX Standard, µg/mL

| Semivolatiles | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 | Level 8 | Level 9 |
|--|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 2-sec butyl-4,6-dinitrophenol (Dinoseb) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methyl parathion | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 4-Nitroquinoline-1-oxide | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Parathion | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Isodrin | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Kepone | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Famphur | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Methapyrilene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Octachlorostyrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Aramite 1 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| Aramite 2 | .5 | 1 | 2.5 | 5 | 10 | 15 | 20 | 25 | 30 |
| p-(Dimethylamino)azobenzene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| p-Chlorobenzilate | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3,3'-Dimethylbenzidine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Acetylaminofluorene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Dibenz (a,j)acridine | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Hexachlorophene | 10 | 20 | 50 | 100 | 200 | 300 | 400 | 500 | 600 |
| 7,12-Dimethylbenz(a)anthracene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 3-Methylcholanthrene | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Fluorophenol (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Phenol-d5 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Nitrobenzene-d5 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2-Fluorobiphenyl (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| 2,4,6-Tribromophenol (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |
| Terphenyl-d14 (Surrogate Standard) | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | 60 |

Table 4
 Minimum Response Factor Criteria

| Semivolatile Compounds | Minimum Response Factor (RF) |
|------------------------|------------------------------|
| Benzaldehyde | 0.010 |

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Table 4
Minimum Response Factor Criteria

| Semivolatile Compounds | Minimum Response Factor (RF) |
|-------------------------------|------------------------------|
| Phenol | 0.800 |
| Bis(2-chloroethyl)ether | 0.700 |
| 2-Chlorophenol | 0.800 |
| 2-Methylphenol | 0.600 |
| 2,2'-Oxybis-(1-chloropropane) | 0.010 |
| Acetophenone | 0.010 |
| 4-Methylphenol | 0.600 |
| N-Nitroso-di-n-propylamine | 0.500 |
| Hexachlorethane | 0.300 |
| Nitrobenzene | 0.200 |
| Isophorone | 0.400 |
| 2-Nitrophenol | 0.100 |
| 2,4-Dimethylphenol | 0.200 |
| Naphthalene | 0.700 |
| 4-Chloroaniline | 0.010 |
| Hexachlorobutadiene | 0.010 |
| Caprolactam | 0.010 |
| 4-Chloro-3-methylphenol | 0.200 |
| 2-Methylnaphthalene | 0.400 |
| Hexachlorocyclopentadiene | 0.050 |
| 2,4,6-Trichlorophenol | 0.200 |
| 2,4,5-Trichlorophenol | 0.200 |
| 1,1'-Biphenyl | 0.010 |
| 2-Chloronaphthalene | 0.800 |
| 2-Nitroaniline | 0.010 |
| Dimethyl phthalate | 0.010 |

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Table 4
Minimum Response Factor Criteria

| Semivolatile Compounds | Minimum Response Factor (RF) |
|-----------------------------|------------------------------|
| 2,6-Dinitrotulene | 0.200 |
| Acenaphthylene | 0.900 |
| 3-Nitroaniline | 0.010 |
| Acenaphthene | 0.900 |
| 2,4-Dinitrophenol | 0.010 |
| 4-Nitrophenol | 0.010 |
| Dibenzofuran | 0.800 |
| 2,4-Dinitrotoluene | 0.200 |
| Diethyl phthalate | 0.010 |
| 1,2,4,5-Tetrachlorobenzene | 0.010 |
| 4-Chlorophenyl-phenyl ether | 0.400 |
| Fluorene | 0.900 |
| 4-Nitroaniline | 0.010 |
| 4,6-Dinitro-2-methylphenol | 0.010 |
| N-Nitrosodiphenylamine | 0.010 |
| Hexachlorobenzene | 0.100 |
| Atrazine | 0.010 |
| Pentachlorophenol | 0.050 |
| Phenanthrene | 0.700 |
| Anthracene | 0.700 |
| Carbazole | 0.010 |
| Di-n-butyl phthalate | 0.010 |
| Fluoranthene | 0.600 |
| Pyrene | 0.600 |
| Butyl benzyl phthalate | 0.010 |
| 3,3'-Dichlorobenzidine | 0.010 |

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Table 4
Minimum Response Factor Criteria

| Semivolatile Compounds | Minimum Response Factor (RF) |
|-------------------------------|-------------------------------------|
| Benzo(a)anthracene | 0.800 |
| Chrysene | 0.700 |
| Bis-(2-ethylhexyl)phthalate | 0.010 |
| Di-n-octyl phthalate | 0.010 |
| Benzo(b)fluoranthene | 0.700 |
| Benzo(k)fluoranthene | 0.700 |
| Benzo(a)pyrene | 0.700 |
| Indeno(1,2,3-cd)pyrene | 0.500 |
| Dibenz(a,h)anthracene | 0.400 |
| Benzo(g,h,i)perylene | 0.500 |
| 2,3,4,6-Tetrachlorophenol | 0.010 |

TestAmerica St. Louis has established a default minimum response factor of 0.01 for compound not identified in this table, except for Famphur, Hexachlorophene, Kepone, Phthalic Anhydride which have a minimum response factor of 0.001.

Table 5

Semi-Volatile Internal Standards with Corresponding Analytes*

| 1,4-Dichlorobenzene-d4 | Naphthalene-d8 | Acenaphthene-d10 | Phenanthrene-d10 | Chrysene-d12 | Perylene-d12 |
|---------------------------|--------------------------------|----------------------------|-------------------------------|-------------------------------------|---------------------------------|
| 1,4-Dioxane | Acetophenone | cis-Isosafrole | 5-Nitro-o-toluidine | Benzidine | Benzo(b)fluoranthene |
| Methyl methacrylate | N-Nitrosopyrrolidine | 1,2,4,5-Tetrachlorobenzene | 4,6-Dinitro-2-methylphenol | Pyrene | Benzo(k)fluoranthene |
| Pyridine | N-Nitrosomorpholine | Hexachlorocyclopentadiene | N-Nitrosodiphenylamine | Terphenyl-d14 | 7,12-Dimethyl benz(a)anthracene |
| N-Nitrosodimethylamine | O-Toluidine | 2,4,6-Trichlorophenol | Tri-n-butyl phosphate | Aramite 1 | Hexachlorophene |
| N,N-Dimethylformamide | Nitrobenzene-d5 | 2,4,5-Trichlorophenol | Azobenzene | Kepone | Benzo(a)pyrene |
| Ethyl methacrylate | Nitrobenzene | 2-Fluorobiphenyl | Sulfotep | Aramite 2 | 3-methylcholanthrene |
| 2-Picoline | N-Nitrosopiperidine | trans-Isosafrole | Diallate 1 | p-(dimethylamino) azobenzene | Indeno (1,2,3-cd) pyrene |
| N-Nitrosomethylethylamine | Isophorone | Biphenyl | 1,3,5-Trinitrobenzene | Chlorobenzilate | Dibenz(a,h)anthracene |
| Methyl methanesulfonate | 2-Nitrophenol | 2-Chloronaphthalene | Phorate | 3,3'-Dimethylbenzidine | Benzo(g,h,i)perylene |
| 2-Fluorophenol | 2,4-Dimethylphenol | 2-Nitroaniline | 4-Bromophenyl phenyl ether | Butyl benzyl phthalate | |
| Cyclohexanol | Bis (2-chloroethoxy) methane | 1,4-Naphthoquinone | Phenacetin | 2-Acetylaminofluorene | |
| N-Nitrosodiethylamine | o,o,o-Triethylphosphorothioate | 1,4-Dinitrobenzene | Diallate 2 | Famphur | |
| Ethyl methanesulfonate | Benzoic acid | Dimethylphthalate | Hexachlorobenzene | Benzo (a) anthracene | |
| Benzaldehyde | 2,4-Dichlorophenol | 1,3-Dinitrobenzene | Dimethoate | 4,4'-methylenebis (2-Chloroaniline) | |
| Phenol-d5 | a,a-Dimethylphenethylamine | Acenaphthylene | Atrazine | 3,3'-Dichlorobenzidine | |
| Phenol | 1,2,4-Trichlorobenzene | 2,6-Dinitrotoluene | Tris(2-chloroethyl) phosphate | Chrysene | |
| Aniline | Naphthalene | 3-Nitroaniline | 4-Aminobiphenyl | Bis (2-ethylhexyl) phthalate | |
| Pentachloroethane | 4-Chloroaniline | Acenaphthene | Pentachlorophenol | Di-n-octyl phthalate | |
| Bis (2-chloroethyl) ether | 2,6-Dichlorophenol | 2,4-Dinitrophenol | Pronamide | | |
| 2-Chlorophenol | Hexachloropropene | 4-Nitrophenol | Pentachloronitrobenzene | | |
| 1,3-Dichlorobenzene | Hexachlorobutadiene | Dibenzofuran | Phenanthrene | | |

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Table 5

Semi-Volatile Internal Standards with Corresponding Analytes*

| 1,4-Dichlorobenzene-d4 | Naphthalene-d8 | Acenaphthene-d10 | Phenanthrene-d10 | Chrysene-d12 | Perylene-d12 |
|-------------------------------|---------------------------|-----------------------------|--------------------------|---------------------|---------------------|
| 1,4-Dichlorobenzene | Benzothiazole | Pentachlorobenzene | Disulfoton | | |
| 1,2-Dichlorobenzene | Caprolactam | 2,4-Dinitrotoluene | Anthracene | | |
| Benzyl alcohol | N-Nitroso-di-n-butylamine | 1-Naphthylamine | Dinoseb | | |
| 2-Methylphenol | p-Phenylenediamine | 2-Naphthylamine | Carbazole | | |
| Bis (2-chloroisopropyl) ether | 4-Chloro-3-methylphenol | 2,3,4,6-Tetrachlorophenol | Methyl parathion | | |
| 3,4-Methylphenol | Safrole | Diethylphthalate | Di-n-butyl phthalate | | |
| N-Nitroso-di-n-propylamine | 2-Methylnaphthalene | Fluorene | Parathion | | |
| Hexachloroethane | | 4-Chlorophenyl phenyl ether | 4-Nitroquinoline-1-oxide | | |
| | | Thionazin | Methapyrilene | | |
| | | 4-Nitroaniline | Isodrin | | |
| | | 2,4,6-Tribromophenol | Fluoranthene | | |

* ISTD assignment is based on instrument operating conditions and column type and may vary slightly from this listing.

Table 6a Acid Surrogates with Corresponding Analytes

| | | |
|----------------|--------------------|-----------------------|
| 2-Fluorophenol | Phenol-d5 | 2,4,6-Tribromophenol |
| none | Phenol | 2,4,6-Trichlorophenol |
| | 2-Chlorophenol | 2,4,5-Trichlorophenol |
| | 2-Methylphenol | 2,4-Dinitrophenol |
| | 3,4-Methylphenol | 4-Nitrophenol |
| | 2-Nitrophenol | 2,3,4,6- |
| | 2,4-Dimethylphenol | Tetrachlorophenol |
| | Benzoic acid | 4,6-Dinitro-2-methyl |
| | 2,4-Dichlorophenol | phenol |
| | 2,6-Dichlorophenol | Pentachlorophenol |
| | 4-Chloro-3-methyl | |
| | -phenol | |

Table 6b Base/Neutral Surrogates with Corresponding Analytes

| Nitrobenzene-d5 | 2-Fluorobiphenyl | Terphenyl-d14 |
|---------------------------|----------------------------|--------------------------|
| 1,4-Dioxane | Benzothiazole | Phenanthrene |
| Methyl methacrylate | Caprolactam | Disulfoton |
| Pyridine | N-Nitrosodi-n-butylamine | Anthracene |
| N-Nitrosodimethylamine | p-Phenylenediamine | Dinoseb |
| Dimethylformamide | Safrole | Carbazole |
| Ethyl methacrylate | 2-Methylnaphthalene | Methyl parathion |
| 2-Picoline | 1-Methylnaphthalene | Di-n-butyl phthalate |
| N-Nitrosomethylethylamine | cis-Isosafrole | Parathion |
| Methyl methanesulfonate | 1,2,4,5-Tetrachlorobenzene | 4-Nitroquinoline-1-oxide |
| Cyclohexanol | Hexachlorocyclopentadiene | Methapyrilene |
| N-Nitrosodiethylamine | trans-Isosafrole | Isodrin |
| Ethyl methanesulfonate | Biphenyl | Fluoranthene |
| Benzaldehyde | 2-Chloronaphthalene | Benzidine |
| Aniline | 2-Nitroaniline | Pyrene |
| Pentachloroethane | 1,4-Naphthoquinone | Aramite 1 & 2 |
| Bis (2-Chloroethyl) ether | 1,4-Dinitrobenzene | Kepone |
| 1,2-Dichlorobenzene | Dimethyl phthalate | p-(dimethylamino) |
| 1,3-Dichlorobenzene | 1,3-Dinitrobenzene | azobenzene |

Table 6b Base/Neutral Surrogates with Corresponding Analytes

| Nitrobenzene-d5 | 2-Fluorobiphenyl | Terphenyl-d14 |
|-----------------------------------|-------------------------|-----------------------------|
| 1,4-Dichlorobenzene | Acenaphthylene | Chlorobenzilate |
| Benzyl alcohol | 2,6-Dinitrotoluene | 3,3-Dimethylbenzidine |
| 2,2'-oxybis (1-Chloro propane) | 3-Nitroaniline | Butyl benzyl phthalate |
| Acetophenone | Acenaphthene | 2-Acetylaminofluorene |
| N-Nitrosopyrrolidine | Dibenzofuran | Famphur |
| N-Nitrosodinpropylamine | Pentachlorobenzene | Benz (a) anthracene |
| N-Nitrosomorpholine | 2,4-Dinitrotoluene | 4,4'-Methylenebis (2-Chloro |
| o-Toluidine | 1-Naphthylamine | -aniline) |
| Hexachloroethane | 2-Naphthylamine | 3,3'-Dichlorobenzidine |
| | Diethyl phthalate | Chrysene |

| | | |
|---------------------------------|------------------------------|----------------------------------|
| Nitrobenzene | Fluorene | Bis (2-ethylhexyl) phthalate |
| N-Nitrosopiperidine | 4-Chlorophenyl phenyl ether | Di-n-octyl phthalate |
| Isophorone | Thionazin | Benzo (b) fluoranthene |
| Bis (2-Chloroethoxy) Methane | 5-Nitro-o-toluidine | Benzo (k) fluoranthene |
| O,o,o-Triethylphosphoro Thioate | 4-Nitroaniline | 7,12-Dimethylbenz (a) anthracene |
| A,a-Dimethylphenethyl Amine | N-Nitrosodiphenylamine | Hexachlorophene |
| 1,2,4-Trichlorobenzene | Tri-n-butyl phthalate | Benzo (a) pyrene |
| Naphthalene | Azobenzene | 3-methylcholanthrene |
| 4-Chloroaniline | Sulfotepp | Indeno (1,2,3-cd) pyrene |
| Hexachloropropene | Diallate 1 & 2 | Dibenz (a,h) anthracene |
| Hexachlorobutadiene | 1,3,5-Trinitrobenzene | Benzo (g,h,i) perylene |
| | Phorate | |
| | 4-Bromophenyl phenyl ether | Phenacetin |
| Hexachlorobenzene | | |
| | Dimethoate | |
| | Atrazine | |
| | Tris(2-chloroethyl)phosphate | |
| | 4-Aminobiphenyl | |
| | Pronamide | |
| | | Pentachloronitrobenzene |

APPENDIX 1

Standard preparations:

SCAN Intermediates

Internal Standard at 2000 ppm (I.S.): 1 mL to 2.5 = 800 ppm

8270:

8270 Surrogate at 5000 ppm: 0.4 mL to 10 mL CH₂CL₂ = 200 ppmBenzoic Acid at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppmCyclohexanol at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppmList 1 STD 1 at 1000 ppm: 2 mL to 10 mL CH₂CL₂ = 200 ppmList 1 STD 2 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppmList 1 STD 7 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppmN,N-Dimethylformamide at 5000ppm: 0.4 mL to 10 mL CH₂CL₂ = 200 ppm

SIM Intermediate

Internal Standard at 2000 (I.S.): 0.1 mL to 2.5 mL = 80 ppm

CAL mix 5 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm200 ppm PAH intermediate: 1 mL to 10 mL CH₂CL₂ = 20 ppm8270 Surrogate at 5000 ppm: 0.04 mL to 10 mL CH₂CL₂ = 20 ppm**Working Standards Levels from 200 ppm Intermediates 8270:**

FV = 1mL for all levels, 800 ppm I.S. addition to each level of 0.05 mL = 40 ppm

Level 1: 1 ppm = 0.005 mL of 200 ppm intermediate

Level 2: 2 ppm = 0.01 mL of 200 ppm intermediate

Level 3: 5 ppm = 0.025 mL of 200 ppm intermediate

Level 4: 10 ppm = 0.05 mL of 200 ppm intermediate

Level 5: 20 ppm = 0.1 mL of 200 ppm intermediate

Level 6 (CCV): 30 ppm = 0.15 mL of 200 ppm intermediate

Level 7: 40 ppm = 0.2 mL of 200 ppm intermediate

Level 8: 50 ppm = 0.25 mL of 200 ppm intermediate

Level 9: 60 ppm = 0.3 mL of 200 ppm intermediate

ICV: 30 ppm = 0.05 of 2nd source ICV 200 ppm intermediate**Working SIM Standards Levels from 20 ppm Intermediates:**

FV = 1mL for all levels, 80 ppm I.S. addition to each level of 0.05 mL = 4 ppm

Level 1: 0.1 ppm = 0.005 mL of 20 ppm intermediate

Level 2: 0.2 ppm = 0.01 mL of 20 ppm intermediate

Level 3: 0.5 ppm = 0.025 mL of 20 ppm intermediate

Level 4 (CCV): 1 ppm = 0.05 mL of 20 ppm intermediate

Level 5: 2 ppm = 0.1 mL of 20 ppm intermediate

Level 6: 5 ppm = 0.25 mL of 20 ppm intermediate

Level 7: 10 ppm = 0.5 mL of 20 ppm intermediate

ICV: 1 ppm = 0.05 mL of 2nd source ICV 20 ppm intermediate



Reagent ID: SV IS Mix_00103

| | | | |
|-------------------|----------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 06/30/2020 |
| Description: | SV Internal Standard Mix | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Kuessner, Melissa A |
| Storage Location: | Semi-volatiles Standards Storage | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A0104707 |
| Creation Date: | 08/18/2014 | Vendor Cat #: | 31206 |
| Container(s): | 429301 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| 1,4-Dichlorobenzene-d4 | | | | | 2000.00000 | ppm |
| Acenaphthene-d10 | | | | | 2000.00000 | ppm |
| Chrysene-d12 | | | | | 2000.00000 | ppm |
| Naphthalene-d8 | | | | | 2000.00000 | ppm |
| Perylene-d12 | | | | | 2000.00000 | ppm |
| Phenanthrene-d10 | | | | | 2000.00000 | ppm |

**Reagent ID: List 1 Std 1_00005**

| | | | |
|-------------------|---------------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/13/2015 |
| Description: | 8270 List 1 Std #1 Megamix parent std | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Kuessner, Melissa A |
| Storage Location: | Semi-volatiles Standards Storage | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A0101615 |
| Creation Date: | 08/11/2014 | Vendor Cat #: | 567672 |
| Container(s): | 423987 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|------------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| 1,1'-Biphenyl | | | | | 1000.00000 | ppm |
| 1,2,4,5-Tetrachlorobenzene | | | | | 1000.00000 | ppm |
| 1,2,4-Trichlorobenzene | | | | | 1000.00000 | ppm |
| 1,2-Dichlorobenzene | | | | | 1000.00000 | ppm |
| 1,3-Dichlorobenzene | | | | | 1000.00000 | ppm |
| 1,3-Dinitrobenzene | | | | | 1000.00000 | ppm |
| 1,4-Dichlorobenzene | | | | | 1000.00000 | ppm |
| 1,4-Dioxane | | | | | 1000.00000 | ppm |
| 1-Methylnaphthalene | | | | | 1000.00000 | ppm |
| 2,2'-oxybis[1-chloropropane] | | | | | 1000.00000 | ppm |
| 2,3,4,6-Tetrachlorophenol | | | | | 1000.00000 | ppm |
| 2,4,5-Trichlorophenol | | | | | 1000.00000 | ppm |
| 2,4,6-Trichlorophenol | | | | | 1000.00000 | ppm |
| 2,4-Dichlorophenol | | | | | 1000.00000 | ppm |
| 2,4-Dimethylphenol | | | | | 1000.00000 | ppm |
| 2,4-Dinitrophenol | | | | | 2000.00000 | ppm |
| 2,4-Dinitrotoluene | | | | | 1000.00000 | ppm |
| 2,6-Dinitrotoluene | | | | | 1000.00000 | ppm |
| 2-Chloronaphthalene | | | | | 1000.00000 | ppm |
| 2-Chlorophenol | | | | | 1000.00000 | ppm |
| 2-Methylnaphthalene | | | | | 1000.00000 | ppm |
| 2-Methylphenol | | | | | 1000.00000 | ppm |
| 2-Nitroaniline | | | | | 1000.00000 | ppm |
| 2-Nitrophenol | | | | | 1000.00000 | ppm |
| 3 & 4 Methylphenol | | | | | 1000.00000 | ppm |
| 3-Nitroaniline | | | | | 1000.00000 | ppm |
| 4,6-Dinitro-2-methylphenol | | | | | 2000.00000 | ppm |

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**Reagent ID: List 1 Std 1_00005**

| | | | |
|-------------------|---------------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/13/2015 |
| Description: | 8270 List 1 Std #1 Megamix parent std | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Kuessner, Melissa A |
| Storage Location: | Semi-volatiles Standards Storage | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A0101615 |
| Creation Date: | 08/11/2014 | Vendor Cat #: | 567672 |
| Container(s): | 423987 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-----------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| 4-Bromophenyl phenyl ether | | | | | 1000.00000 | ppm |
| 4-Chloro-3-methylphenol | | | | | 1000.00000 | ppm |
| 4-Chloroaniline | | | | | 1000.00000 | ppm |
| 4-Chlorophenyl phenyl ether | | | | | 1000.00000 | ppm |
| 4-Nitroaniline | | | | | 1000.00000 | ppm |
| 4-Nitrophenol | | | | | 2000.00000 | ppm |
| Acenaphthene | | | | | 1000.00000 | ppm |
| Acenaphthylene | | | | | 1000.00000 | ppm |
| Acetophenone | | | | | 1000.00000 | ppm |
| Aniline | | | | | 1000.00000 | ppm |
| Anthracene | | | | | 1000.00000 | ppm |
| Azobenzene | | | | | 1000.00000 | ppm |
| Benzo[a]anthracene | | | | | 1000.00000 | ppm |
| Benzo[a]pyrene | | | | | 1000.00000 | ppm |
| Benzo[b]fluoranthene | | | | | 1000.00000 | ppm |
| Benzo[g,h,i]perylene | | | | | 1000.00000 | ppm |
| Benzo[k]fluoranthene | | | | | 1000.00000 | ppm |
| Benzyl alcohol | | | | | 1000.00000 | ppm |
| Bis(2-chloroethoxy)methane | | | | | 1000.00000 | ppm |
| Bis(2-chloroethyl)ether | | | | | 1000.00000 | ppm |
| Bis(2-ethylhexyl) phthalate | | | | | 1000.00000 | ppm |
| Butyl benzyl phthalate | | | | | 1000.00000 | ppm |
| Carbazole | | | | | 1000.00000 | ppm |
| Chrysene | | | | | 1000.00000 | ppm |
| Dibenz(a,h)anthracene | | | | | 1000.00000 | ppm |
| Dibenzofuran | | | | | 1000.00000 | ppm |
| Diethyl phthalate | | | | | 1000.00000 | ppm |

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**Reagent ID: List 1 Std 1_00005**

| | | | |
|-------------------|---------------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/13/2015 |
| Description: | 8270 List 1 Std #1 Megamix parent std | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Kuessner, Melissa A |
| Storage Location: | Semi-volatiles Standards Storage | Vendor: | Restek |
| Reagent Volume: | 1.000 mL | Vendor Lot #: | A0101615 |
| Creation Date: | 08/11/2014 | Vendor Cat #: | 567672 |
| Container(s): | 423987 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| Dimethyl phthalate | | | | | 1000.00000 | ppm |
| Di-n-butyl phthalate | | | | | 1000.00000 | ppm |
| Di-n-octyl phthalate | | | | | 1000.00000 | ppm |
| Fluoranthene | | | | | 1000.00000 | ppm |
| Fluorene | | | | | 1000.00000 | ppm |
| Hexachlorobenzene | | | | | 1000.00000 | ppm |
| Hexachlorobutadiene | | | | | 1000.00000 | ppm |
| Hexachlorocyclopentadiene | | | | | 1000.00000 | ppm |
| Hexachloroethane | | | | | 1000.00000 | ppm |
| Hexadecane | | | | | 1000.00000 | ppm |
| Indeno[1,2,3-cd]pyrene | | | | | 1000.00000 | ppm |
| Isophorone | | | | | 1000.00000 | ppm |
| Naphthalene | | | | | 1000.00000 | ppm |
| n-Decane | | | | | 1000.00000 | ppm |
| Nitrobenzene | | | | | 1000.00000 | ppm |
| N-Nitrosodimethylamine | | | | | 1000.00000 | ppm |
| N-Nitrosodi-n-propylamine | | | | | 1000.00000 | ppm |
| n-Octadecane | | | | | 1000.00000 | ppm |
| Pentachlorophenol | | | | | 2000.00000 | ppm |
| Phenanthrene | | | | | 1000.00000 | ppm |
| Phenol | | | | | 1000.00000 | ppm |
| Pyrene | | | | | 1000.00000 | ppm |
| Pyridine | | | | | 1000.00000 | ppm |

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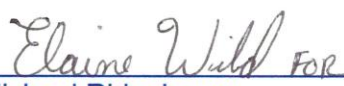
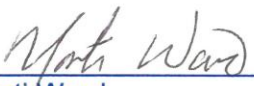
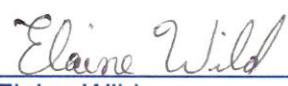
Reagent ID: 8270 Surr Std_00019

| | | | |
|-------------------|----------------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/28/2018 |
| Description: | 8270 Surrogate parent standard | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Winkler, Jeff S |
| Storage Location: | Semi-volatiles Standards Storage | Vendor: | Restek |
| Reagent Volume: | 5.000 mL | Vendor Lot #: | A093638 |
| Creation Date: | 08/28/2013 | Vendor Cat #: | 567685 |
| Container(s): | 198745 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|-------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| 2,4,6-Tribromophenol | | | | | 5000.00000 | ppm |
| 2-Fluorobiphenyl (Surr) | | | | | 5000.00000 | ppm |
| 2-Fluorophenol | | | | | 5000.00000 | ppm |
| Nitrobenzene-d5 | | | | | 5000.00000 | ppm |
| Phenol-d5 | | | | | 5000.00000 | ppm |
| Terphenyl-d14 | | | | | 5000.00000 | ppm |

**Title: ANALYSIS OF METALS BY INDUCTIVELY COUPLED
PLASMA/MASS SPECTROMETRY
[SW-846 6020; SW-846 6020A; EPA 200.8]**

| Approvals (Signature/Date): | | | |
|---|---------|--|---------|
|  | 6/22/15 |  | 6/22/15 |
| For Kristen Ely Metals Supervisor | Date | Michael Ridenhower Health & Safety Manager / Coordinator | Date |
|  | 6-22-15 |  | 6/22/15 |
| Marti Ward Quality Assurance Manager | Date | Elaine Wild Laboratory Director | Date |

This SOP was previously identified as SOP No. ST-MT-0001 Rev. 23

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1.0 SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA SW846 Method 6020, 6020A and EPA 200.8 .
- 1.2. This method is applicable to surface, and saline waters; soil and waste samples.
- 1.3. The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.
 - 1.3.1. The Technetium-99 soil preparation procedure is found in SOP ST-RC-0125, Determination of Technetium-99 Using Eichrom TEVA Resin,
- 1.4. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1. Additional elements may be amendable to this method provided the laboratory has established a MDL and the elements meets the QC requirements as prescribed in the associated preparation and analysis SOP.

2.0 SUMMARY OF METHOD

- 2.1. Sample digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produces are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole or octupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3.0 DEFINITIONS

- 3.1. See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2. EPA and SW methodology use different terminology. Our SOP references the SW 846 terminology:
 - 3.2.1. The ICV satisfies the QCS requirements found in method 200.8 .
 - 3.2.2. The LCS satisfies the requirements of the LFB found in method 200.8.
 - 3.2.3. The MS satisfies the requirements of the LFM found in method 200.8.
 - 3.2.4. The MB satisfies the requirements of the LRB found in method 200.8.
- 3.3. Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is acidified after filtration)
- 3.4. Suspended Metals: Those elements retained by a 0.45 µm filter
- 3.5. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion
- 3.6. Dilution Test: the terminology “dilution test” found in later versions of 200.8 and 6020A is referred to as a Serial Dilution in this SOP.

4.0 INTERFERENCES

- 4.1. Isobaric elemental interferences: Isobaric elemental interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

- 4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences. This may be done with either interference equations or collision cell technology.
- 4.3. Common molecular ion interferences are listed in [Table 3](#) of this SOP.
- 4.4. Matrix interferences: Internal standards are used to correct for some matrix interferences.
 - 4.4.1. Internal standards are added at a level to give approximately 100,000 - 10,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ± 50 amu of the mass of the affected analyte.
 - 4.4.2. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank.

5.0 SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
 - 5.2.1. The ICP plasma emits strong UV light, harmful to vision. Analysts must avoid looking directly at the plasma.
- 5.3. PRIMARY MATERIALS USED
 - 5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|--|---------------------------------|-----------------------------|--|
| Nitric Acid | Corrosive Oxidizer Poison | 2 ppm (TWA) 4 ppm (STEL) | Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| Hydrochloric Acid | Corrosive Poison | 5 ppm (Ceiling) | Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |
| TWA – Time Weighted Average | | | |

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|--|---------|--------------------|--------------------------------|
| STEL – Short Term Exposure Limit | | | |
| Ceiling – At no time should this exposure limit be exceeded. | | | |

6.0 EQUIPMENT AND SUPPLIES

- 6.1. PerkinElmer® ELAN 6100/ PerkinElmer® ELAN 9000 /Agilent 7500/ Agilent 7700 (all with auto samplers)
- 6.2. Helium gas: 5.5 trace analytical grade
- 6.3. Argon gas: High-purity grade (99.99%)
- 6.4. Chiller (water cooling device)
- 6.5. Peristaltic Pump
- 6.6. Calibrated automatic pipettes
- 6.7. Teflon® flasks
- 6.8. Instrument software: ELAN version 2.3.2 / ELAN version 3.3 / Mass Hunter version B.01.01.

7.0 REAGENTS AND STANDARD

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2. Concentrated nitric acid (HNO₃), trace metal grade
- 7.3. Concentrated hydrochloric acid (HCl), trace metal grade
- 7.4. DI water from the Millipore unit
 - 7.4.1. Water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Water must be shown to have a resistivity greater than or equal to 16.67 Mohm-cm.
- 7.5. Standards
 - 7.5.1. Purchased as custom multi-element mixes or as single-element solutions
 - 7.5.2. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.
 - 7.5.3. Working calibration and calibration verification solutions may be used for up to 1 week and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 2% hydrochloric and 2% nitric acid.
 - 7.5.4. Internal Standard Solution: Prepare internal standards (Au, Sc, Ge, In, Ho, Li6, Ir) when needed.
 - 7.5.5. Tuning solution: Prepare tuning solution (Be, Ba, Ce, Co, In, Pb, Li, Mg, Rh, Tl, Y) when needed.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure, ST-IP-0013.

- 8.3. Soil or waste samples are digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure, ST-IP-0002.
- 8.4. Digestate holding time is 6 months from sample collection.

9.0 QUALITY CONTROL

9.1. Batch

- 9.1.1. A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3. For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2. Method Blank (MB)

- 9.2.1. A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2. A method blank must be prepared with every sample batch.
- 9.2.3. DI water is used for the method blank associated with water batches. 2% HNO₃ is used instead of DI for gold, tantalum and palladium water batches. DI and glass beads are used with solid batches.
- 9.2.4. See Section 13 for acceptance criteria.

9.3. Laboratory Control Sample (LCS)

- 9.3.1. An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2. An LCS must be prepared with every sample batch.
- 9.3.3. DI water, spiked with the analytes of interest, is used for the LCS associated with water batches. 2% HNO₃ is used instead of DI for gold, tantalum and palladium for water batches. A commercially available solid reference material is used for the LCS associated with solid batches. Where reference material is not available, spiked glass beads comprise the LCS.
- 9.3.4. See Section 13 for acceptance criteria.

9.4. Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1. A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2. See Section 13 for acceptance criteria.

9.5. Serial Dilution

- 9.5.1. A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.
- 9.5.2. The test is performed by running a sample at a 5x (1:4) dilution.
- 9.5.3. Samples identified as field blanks cannot be used for dilution tests.
- 9.5.4. See Section 13 for acceptance criteria.

9.6. Post Digestion Spike (PDS)

- 9.6.1. A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process, with a spike concentration between 10-100 times the MDL (unless specific project/program criteria is given)
- 9.6.2. 200.8: A PDS is not applicable for this method
- 9.6.3. 6020: A PDS is analyzed with every batch
- 9.6.4. 6020A: The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed.

- 9.6.5. For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the serial dilution. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
- 9.6.6. See Section 13 for acceptance criteria.

9.7. Method of Standard Addition (MSA)

9.7.1. 6020 and 6020A

- 9.7.2. This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
- 9.7.3. MSA are not required by methods 6020 or 6020A
- 9.7.4. MSA are not considered standard batch QC and if required by the client, must appear in the sample comment section in LIMS.
- 9.7.5. MSA is required by SW846 Method 1311 when the MS/MSD recovery is less than 50%, analyte concentration is less than and within 20% of its regulatory limit.

9.8. Procedural Variations/ Nonconformance and Corrective Action

- 9.8.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

Follow the instrument start-up procedure outlined in the manufacturers operating manual.

10.1. Cone Conditioning

- 10.1.1. Aspirating a 25% tap water / 75% ICPMS rinse solution for at least 1 hour can enhance instrument performance. This procedure should be used daily after a thorough cleaning of the interface cones or the installation of new cones takes place.

10.2. Rinse Time Determination

- 10.2.1. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in this SOP it can be demonstrated that a shorter rinse time may be used.
- 10.2.1.1. To determine the appropriate rinse time, a linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for the system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

10.3. Instrument Tuning (Agilent)

10.3.1. Frequency:

- 10.3.1.1. Daily with each initial calibration

- 10.3.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Y, Tl, Co, In, and Ce.

10.3.3. Tune Criteria:

- 10.3.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.3.3.1.1. Resolution

- 10.3.3.1.1.1 6020 and 6020A: peak width must be < 0.9 amu at 10% peak height

- 10.3.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

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10.3.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.3.3.1.3. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.3.3.1.4. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.4. Instrument Tuning (Perkin Elmer)

10.4.1. Frequency:

10.4.1.1. Daily with each initial calibration

10.4.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Mg, Rh, Ce, Pb and Ba.

10.4.3. Tune Criteria:

10.4.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.4.3.1.1. Resolution

10.4.3.1.1.1 6020 and 6020A: peak width must be < 0.9 amu at 10% peak height

10.4.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.4.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.4.3.1.3. Using the Tuning Solution, an Auto-lens calibration is performed to ensure that optimum voltages are being applied to the Auto-lens. The default calibration should range from 4- 12 volts

10.4.3.1.4. Mg should be greater than 20,000 counts. Pb should be at or above 100,000 counts. Rh should be at or above 150,000. The background should be less than or equal to 30 counts. These are manufacturer recommendations, not requirements.

10.4.3.1.5. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.4.3.1.6. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.5. Initial Calibration

10.5.1. Multi-point Calibration:

10.5.1.1. A calibration curve, consisting of 3 standards and a blank, must be analyzed daily.

10.5.1.2. Calibration criteria:

10.5.1.2.1. Correlation Coefficient of ≥ 0.998

10.5.1.2.2. The low level standard in the curve must be at or below the laboratory's routine reporting limit.

10.5.1.2.2.1 If a client requested reporting limit is below the laboratory's routine reporting limit and thus below the low level verification standard, the laboratory will discuss with the client, prior to sample analysis, how to proceed with this requirement.

10.6. Initial Calibration Verification/Low Level Initial Calibration Verification/Initial Calibration Blank (ICV/LLICV/ICB)

10.6.1. ICV

10.6.1.1. Secondary source, used to verify the initial calibration accuracy.

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- 10.6.1.2. Frequency: Perform with each initial calibration
- 10.6.1.3. Criteria: $\pm 10\%$
- 10.6.1.4. Action upon failure:
 - 10.6.1.4.1. If the ICV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.6.1.4.2. For all other non-conformances, the samples must be re-analyzed.

10.6.2. **LLICV**

- 10.6.2.1. Applicable to 6020A only
- 10.6.2.2. Same source as calibration
- 10.6.2.3. Frequency: Perform with each initial calibration.
- 10.6.2.4. Criteria: $\pm 30\%$
- 10.6.2.5. Action upon failure:
 - 10.6.2.5.1. If the LLICV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.2.5.2. If the LLICV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.2.5.3. For all other non-conformances, the samples must be re-analyzed.

10.6.3. **ICB**

- 10.6.3.1. Frequency: An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.
- 10.6.3.2. Criteria: The ICB result must fall within \pm the RL from zero.
- 10.6.3.3. Action upon failure:
 - 10.6.3.3.1. If the ICB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.3.3.2. If the ICB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.3.3.3. For all other non-conformances, the samples must be re-analyzed.

10.6.4. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.

10.6.5. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7. **Continuing Calibration Verification/Low Level Continuing Calibration Verification/Continuing Calibration Blank (CCV/LLCCV/CCB)**

- 10.7.1. Calibration is monitored throughout the analytical run through the analysis of a known mid-level calibration standard.
- 10.7.2. **CCV**
 - 10.7.2.1. A CCV may be a second source or the same source as the calibration.
 - 10.7.2.2. Frequency: Analyte response factors must be verified at the beginning of each analytical

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run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run.

10.7.2.3. Criteria:

10.7.2.3.1. For **200.8**: The CCV must fall within 15% of the true value for that solution.

10.7.2.3.2. For **6020 and 6020A**: The CCV must fall within 10% of the true value for that solution.

10.7.2.4. Action upon failure:

10.7.2.4.1. If the CCV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.7.2.4.2. For all other non-conformances, the samples must be re-analyzed.

10.7.3. **LLCCV**

10.7.3.1. Applicable to 6020A only

10.7.3.2. Same source as calibration

10.7.3.3. Frequency: Perform at a minimum at the end of the run

10.7.3.4. Criteria: $\pm 30\%$

10.7.3.5. Action upon failure:

10.7.3.5.1. If the LLCCV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.3.5.2. If the LLCCV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.3.5.3. For all other non-conformances, the samples must be re-analyzed.

10.7.4. **CCB**

10.7.4.1. Frequency: A CCB is analyzed immediately following each CCV.

10.7.4.2. Criteria: The CCB result must fall within \pm RL from zero.

10.7.4.3. Action upon failure:

10.7.4.3.1. If the CCB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.4.3.2. If the CCB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.7.4.3.3. For all other non-conformances, the samples must be re-analyzed.

10.7.5. If a CCV and/or CCB has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV and/or CCB may be analyzed without any adjustments to the instrument. If this CCV and/or CCB meet criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV and/or CCB does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e., initial calibration).

10.8. **Interference Check Standard (ICSA/ICSAB)**

10.8.1. Applicable to 6020 and 6020A only

10.8.2. The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions.

10.8.3. **ICSA:**

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- 10.8.3.1. The ICSA contains only interfering elements. Refer to LIMS for the details of ICSA composition.
- 10.8.3.2. Custom multi-element ICS solutions must be used.
- 10.8.3.3. Elements known to be interferents on a required analyte must be included in the ICPMS run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICPMS runs.
- 10.8.3.4. Frequency: The ICSA must run with each initial calibration or every 12 hours whichever is shorter.
- 10.8.3.5. Criteria: ICSA results for the non-interfering elements with RL < 10 µg/L must fall within $\pm 2x$ RL from zero. ICSA results for the non-interfering elements with RL ≥ 10 µg/L must fall within $\pm 1xRL$ from zero.
- 10.8.3.6. Action upon failure:
 - 10.8.3.6.1. For interfering elements:
 - 10.8.3.6.1.1 If the ICSA fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.8.3.6.2. For non-interfering elements:
 - 10.8.3.6.2.1 If the ICSA fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.3.6.2.2 If the ICSA fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.3.6.3. For all other non-conformances, the samples must be re-analyzed.

10.8.4. ICSAB:

- 10.8.4.1. The ICSAB contains analytes and interferents.
- 10.8.4.2. Refer to [LIMS](#) for the details of ICSAB composition.
- 10.8.4.3. Custom multi-element ICS solutions must be used.
- 10.8.4.4. Frequency: The ICSAB must run with each initial calibration or every 12 hours whichever is shorter.
- 10.8.4.5. Criteria: The ICSAB results must fall within 80% – 120% of the true value.
- 10.8.4.6. Action upon failure:
 - 10.8.4.6.1. If the ICSAB fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.8.4.6.2. For all other non-conformances, the samples must be re-analyzed.

10.9. Liner Dynamic Range (LDR)

- 10.9.1. Prior to running the instrument, the upper limit of quantitation must be established for each analyte.
- 10.9.2. This upper limit is tested by running a standard containing high concentrations of the analytes against a calibration curve.
- 10.9.3. The concentration of the LDR standard is higher than the high calibration standard.
- 10.9.4. Frequency: Every 6 months
 - 10.9.4.1. When requested by client, the LDR is run daily.
- 10.9.5. Criteria: $\pm 10\%$
 - 10.9.5.1. If the LDR fails the criteria, the highest calibration standard is used as the upper limit for the

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linear range.

10.10. Calibration Sequence

Tuning Standard
Initial Calibration (3 standards plus a blank)
ICV
ICB
LLICV (for 6020A only)
ICSA*
ICSAB*
CCV
CCB
LDR (Client Specific)
CCV
CCB
10 samples (analysis runs)
CCV
CCB
10 samples (repeat every 10 analysis runs)
LLCCV (Minimum at end of run. May be analyzed more frequently)
CCV
CCB
End

* If sequence time is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.

11.0 PROCEDURE

- 11.1 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
- 11.1.4 For 200.8 analyses, dissolved samples must be digested.
- 11.2 The soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.
- 11.3 Instrument conditions, including rinse times, must be the same for all standards and samples.
- 11.4 Internal standards are introduced to the standards and sample digestates by the instrument.
- 11.5 Load autosampler with standards and digestates in accordance with the sequence given in section 10
- 11.6 Analyze samples.
- 11.7 When analysis is completed, return unused digestate to proper storage area.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2. All measurements must fall within the defined linear range where spectral interference correction factors

are valid.

- 12.2.1. Dilute and reanalyze all samples for required analytes that exceed the linear range
 - 12.2.1.1. For 200.8, any sample greater than 90% of the linear range must be diluted and re-analyzed.
- 12.2.2. Acid strength must be maintained in the dilution of samples.

12.3. The mass ions used for determination of the element of interest is given in [Table 1](#) of this SOP

12.4. Tracer Calculations

12.4.1. Tracer Recovery: The LIMS calculates the tracer recovery using the following formula:

$$\frac{\text{Measured Tracer Concentration}}{\text{Actual Tracer Concentration}} = \text{Final Recovery}$$

12.4.1.1. Tracer Criteria: The tracer recovery must fall within 30-110%.

12.4.2. Final sample concentration (corrected for tracer recovery) is determined by the LIMS using the following formula:

$$\frac{\text{Measured Sample Concentration}}{\text{Tracer Recovery}} = \text{Final Sample Concentration}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process in LIMS. The NCM process is described in SOP: ST-QA-0036.

13.2. Method Blank (MB)

- 13.2.1. Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.
- 13.2.2. Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted in the client notes.
- 13.2.3. Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.2.3.1. Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3. Laboratory Control Sample (LCS)

- 13.3.1. Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.
- 13.3.2. Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1. LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
 - 13.3.2.2. LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- 13.4.1. Analytes should be within control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.
- 13.4.2. Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1. MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration

- 13.4.2.1.1. If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.
- 13.4.2.1.2. If the excursion is due to physically evident matrix interference, the data is reported with an NCM.
- 13.4.2.1.3. In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.
- 13.4.2.1.4. When the MS/MSD recoveries and the %RPD are outside criteria, if the samples are non-homogenous, the data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.

13.5. Serial Dilution (SD)

- 13.5.1. Acceptance Criteria: The serial dilution results shall agree within $\pm 10\%$ of the undiluted sample results, if the undiluted sample results are greater than 10 times the reporting limit. There is no criterion for sample results less than 10 times the reporting limit.
- 13.5.2. Corrective Action: Serial dilution failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the serial dilution test, the analyst may re-prepare and re-analyze the samples.

13.6. Post Digestion Spike (PDS)

- 13.6.1. Method 6020 and 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD. A PDS is not required for 200.8.
- 13.6.2. **6020 Criteria:** The acceptance criteria is 75%-125%, UNLESS, other project/program criteria is given.
 - 13.6.2.1. Corrective Action: Sample must be diluted and re-analyzed to compensate for matrix effect, until the PDS is within acceptable limits.
- 13.6.3. **6020A Criteria:** The acceptance criteria is 80%-120%, with a spike concentration between 10-100 times the MDL, UNLESS, other project/program criteria is given.
 - 13.6.3.1. Corrective Action: There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with an NCM and noted in the report narrative.
- 13.6.4. The PDS is not reported in the data package unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.

13.7. Sample result evaluation

- 13.7.1. Dilutions
 - 13.7.1.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
- 13.7.2. For samples requiring dilution an NCM is created to document the reason for the dilution.
- 13.7.3. Insufficient Sample
 - 13.7.3.1. For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

13.8. Internal Recovery Standard (IS)

- 13.8.1. Criteria (for all samples and QC standards)
 - 13.8.1.1. **6020:**
 - 13.8.1.1.1. QC: 80-120%
 - 13.8.1.1.2. Samples: 30-120%
 - 13.8.1.2. **6020A:** 70%-140%
 - 13.8.1.3. **200.8:** 60%-125%

13.8.2. Action Upon Failure

- 13.8.2.1. Samples: If the criteria is not met, the sample should be diluted and re-analyzed until the

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- IS recoveries are within specified limits.
- 13.8.2.2. QC standards: If the criteria are not met, the analyst will review the data. If the sample internal standard recoveries are within control and the QC standard is within acceptable limits, it is apparent that whatever interference affected the internal standard for the QC standards has not affected the element bracketed by that internal standard based upon the criteria being met. If these specific occurrences are met then an NCM will be generated stating why the data is acceptable. Otherwise, samples linked to the QC standard will be re-analyzed

14.0 METHOD PERFORMANCE

- 14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.
- 14.2. Demonstration of Capability
- 14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3. Training Qualification
- 14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1. Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2. Waste Streams Produced by the Method
- 16.2.1. The following waste streams are produced when this method is carried out.
- 16.2.1.1. Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."
- 16.2.1.2. Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6020A
- 17.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma/Mass Spectrometry Method 200.8
- 17.3. PerkinElmer® ELAN 6100 Inductively Coupled Plasma Mass Spectrometer Hardware Guide
- 17.4. PerkinElmer® ELAN 6100 Software Kit
- 17.5. PerkinElmer® ELAN 9000 Hardware Guide
- 17.6. PerkinElmer® ELAN Version 3.0 Software Guide
- 17.7. Agilent 7500 Series MassHunter Workstation (G7200A) Operators Manual
- 17.8. Agilent 7500 Series ICP-MS Hardware Manual
- 17.9. Agilent 7700 Series ICP-MS Hardware Maintenance Manual
- 17.10. TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.11. TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.12. Associated SOPs, current revisions:
 - 17.12.1. ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS
 - 17.12.2. ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
 - 17.12.3. ST-QA-0002, Standard and Reagent Preparation
 - 17.12.4. ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.12.5. ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.12.6. ST-QA-0016, IDL/MDL Determination
 - 17.12.7. ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.12.8. ST-RC-0125, Determination of Technetium-99 Using Eichrom Resin

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1. Method 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed. Internal standards are used to monitor matrix interferences in all samples. Post spikes are done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.
- 18.2. Method 6020A requires the analysis of a Lower Limit Quantitation Check Sample (LLQC) on an as needed basis, to establish and confirm the lowest quantitation limit. TestAmerica St. Louis fills this requirement with the quarterly running of a MDL verification standard which is taken through the entire sample preparation procedure.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1. Updated formatting and spelling errors throughout SOP
- 19.2. Updated section 4.4 referring to the amount of an internal standard being used.
- 19.3. Added new instrument and gases used in section 6.0.
- 19.4. Added Lithium to section 7.0 as part of the new reagents and standards used.
- 19.5. Made reference to new instruments for calibration in section 10.0.
- 19.6. Add new list of tuning element for both instruments in section 10.5.
- 19.7. Updated the internal standard intensity throughout section 10.7 and section 10.8

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- 19.8. Added new elements to [table 2](#).
- 19.9. Rev. 18;
- 19.9.1. Added LLICV to definitions in section 3.2.
 - 19.9.2. Removed Hydrogen Peroxide from Safety Section (included in prep SOP)
 - 19.9.3. Added tuning solution to section 7.5.5.
 - 19.9.4. Updated cone conditioning solution, make up and frequency of use.
 - 19.9.5. Added clarification to tuning section 10.5.
 - 19.9.6. Added Low Level initial calibration verification standards plus criteria to section 10.0.
 - 19.9.7. Updated tables 1 and 2, added analytes, updated concentrations.
 - 19.9.8. Added method 1311 MSA requirements information to section 9.7.
 - 19.9.9. Spelling and grammatical corrections.
- 19.10. Rev. 19:
- 19.10.1. Updated [Table III](#) regarding QC Criteria limits.
- 19.11. Revision 20:
- 19.11.1. Updated section 1.3 adding reference to the Technetium-99 soil procedure.
 - 19.11.2. Added formulas for determining the Tracer Recovery and the Final tracer Corrected Concentration to section 12.5.
 - 19.11.3. Added instrument software and hardware to section 6.0.
 - 19.11.4. Updated the PDS acceptance criteria in section 9.6.
- 19.12. Rev. 21:
- 19.12.1. Removed legacy text regarding MSA from Section 18 as MSA is not required by Method 6020A.
- 19.13. Rev. 22: (8/27/2013)
- 19.13.1. Updated section 1.3 to reflect the corrected Tc-99 SOP (ST-RC-0125)
 - 19.13.2. Updated section 6. Hydrogen was removed
 - 19.13.3. Updated section 9.7, replace QuantIMS wording with TALS wording
 - 19.13.4. Updated section 10, used a more consistent format
 - 19.13.5. Updated section 12.4. Removed references to a spreadsheet, added TALS
 - 19.13.6. Updated section 13
- 19.14. Rev.23: (1/16/2015)
- 19.14.1. Made formatting and grammatical corrections
 - 19.14.2. Corrected definitions in section 3 (removed IPC and CRI, added LRB)
 - 19.14.3. Removed references to ASTM Method D5673-03
 - 19.14.4. Added equipment and software for ICPMS 9000 to section 6
 - 19.14.5. Removed the ICESA/ICSAB Table (was Table 2) and replaced references to it with instructions to look at LIMS (10.8.3.1 and 10.8.4.2)
 - 19.14.6. Updated tuning criteria for Perkin Elmer ICPMS in sections 10.5.3.1.4 & 10.5.3.1.5
 - 19.14.7. Updated section 10.8.3.5, replace $RL > 10\mu\text{g/L}$ with $RL \geq 10\mu\text{g/L}$
 - 19.14.8. Clarified criteria in section 10, 13 and 18
 - 19.14.9. Replaced "client requirement sheet" with "client notes" in section 13.2.2
 - 19.14.10. Removed reference to Marginal Exceedance in section 13.3
 - 19.14.11. Corrected IS criteria in section 13.8.1
 - 19.14.12. Updated instrument manuals in section 17
 - 19.14.13. Added affected methods to section 18
 - 19.14.14. Removed section 18.3, was same as section 18.4
 - 19.14.15. Added reference to method 6020
 - 19.14.16. Deleted references in section 9 to batch QC criteria, referenced section 13. Instrument QC criteria is in section 10.
 - 19.14.17. updated formatting, to make the wording more consistent throughout the SOP and easier to read.
 - 19.14.18. section 10: separated out the tuning for Perkin Elmer vs Agilent, for clarity.
 - 19.14.19. Added reference to tc-99 prep via ST-RC-0125
- 19.15. Rev. 24: (06/22/2015)
- 19.15.1. Added Appendix 1 MSA instructions

Table 1
ANALYTICAL ISOTOPES

| ELEMENT | 7700 Tune Step | 7500 Mass | 7700 Tune Step | 7700 Mass | 6100 Mass | 9000 Mass |
|---------|-------------------|--------------|-------------------|--------------|--------------|--------------|
| Li | 3 | 7 | 3 | 7 | N/A | N/A |
| Be | 3 | 9 | 3 | 9 | N/A | N/A |
| B | 3 | 11 | 3 | 11 | N/A | N/A |
| Na | 2 | 23 | 2 | 23 | N/A | N/A |
| Mg | 2 | 24 | 2 | 24 | N/A | N/A |
| Al | 2 | 27 | 2 | 27 | N/A | N/A |
| Si | 3 | 28 | 3 | 28 | N/A | N/A |
| P | 2 | 31 | 2 | 31 | N/A | N/A |
| S | 2 | 34 | 2 | 34 | N/A | N/A |
| K | 2 | 39 | 2 | 39 | N/A | N/A |
| Ca | 3 | 44 | 3 | 44 | N/A | N/A |
| Ti | 3 | 47 | 3 | 47 | N/A | N/A |
| V | 2 | 51 | 2 | 51 | N/A | N/A |
| Cr | 2 | 52 | 2 | 52 | N/A | N/A |
| Mn | 2 | 55 | 2 | 55 | N/A | N/A |
| Fe | 2 | 57 | 2 | 57 | N/A | N/A |
| Co | 2 | 59 | 2 | 59 | N/A | N/A |
| Ni | 2 | 60 | 2 | 60 | N/A | N/A |
| Cu | 2 | 63 | 2 | 63 | N/A | N/A |
| Zn | 2 | 66 | 2 | 66 | N/A | N/A |
| As | 2 | 75 | 2 | 75 | N/A | N/A |
| Se | 2 | 78 | 2 | 78 | N/A | N/A |
| Sr | 3 | 88 | 3 | 88 | N/A | N/A |
| Y | 2 | 89 | 2 | 89 | N/A | N/A |
| Zr | 2 | 90 | 2 | 90 | N/A | N/A |
| Nb | 2 | 93 | 2 | 93 | N/A | N/A |
| Mo | 3 | 95 | 3 | 95 | N/A | N/A |
| Ru | 2 | 101 | 2 | 101 | N/A | N/A |
| Rh | 2 | 103 | 2 | 103 | N/A | N/A |
| Pd | 2 | 105 | 2 | 105 | N/A | N/A |
| Ag | 3 | 107 | 3 | 107 | N/A | N/A |
| Cd | 3 | 111 | 3 | 111 | N/A | N/A |
| Sn | 3 | 118 | 3 | 118 | N/A | N/A |
| Sb | 3 | 121 | 3 | 121 | N/A | N/A |
| Te | 2 | 125 | 2 | 125 | N/A | N/A |
| Cs | 2 | 133 | 2 | 133 | N/A | N/A |
| Ba | 3 | 137 | 3 | 137 | N/A | N/A |
| La | 2 | 139 | 2 | 139 | N/A | N/A |
| Ce | 2 | 140 | 2 | 140 | N/A | N/A |
| Pr | 2 | 141 | 2 | 141 | N/A | N/A |
| Nd | 2 | 146 | 2 | 146 | N/A | N/A |
| Sm | 3 | 147 | 3 | 147 | N/A | N/A |
| Hf | 2 | 178 | 2 | 178 | N/A | N/A |
| Ta | 2 | 181 | 2 | 181 | N/A | N/A |
| W | 2 | 182 | 2 | 182 | N/A | N/A |
| Re | 3 | 185 | 3 | 185 | N/A | N/A |
| Pt | 2 | 195 | 2 | 195 | N/A | N/A |
| Au | 3 | 197 | 3 | 197 | N/A | N/A |
| Tl | 3 | 205 | 3 | 205 | N/A | N/A |
| Pb | 3 | 208 | 3 | 208 | N/A | N/A |
| Bi | 2 | 209 | 2 | 209 | N/A | N/A |
| Th | 3 | 232 | 3 | 232 | N/A | N/A |
| Tc | 3 | 99 | N/A | N/A | 99 | 99 |
| U | N/A | N/A | 3 | 236 | 236 | 236 |
| U | N/A | N/A | 3 | 235 | 235 | 235 |
| U | N/A | N/A | 3 | 234 | 234 | 234 |
| U | N/A | N/A | 3 | 233 | 233 | 233 |
| U | 3 | 238 | 3 | 238 | 238 | 238 |

Tune Step 2: Helium
 Tune Step 3: No Gas (argon only)

Table 2
QC Criteria

| Methods | 6020 | 6020A | 200.8 |
|-------------|---------------------------------|---------|-----------|
| Corr Coeff. | >0.998 | >0.998 | |
| Tuning Res | <0.9amu | <0.9amu | ≈ 0.75amu |
| Int Std | QC: 80-120% Samples: 30-120% | 70-140% | 60-125% |
| LCS | 80-120% | 80-120% | 85-115% |
| ICV | 90-110% | 90-110% | 90-110% |
| CCV | 90-110% | 90-110% | 85-115% |
| PDS | 75-125% | 80-120% | N/A |
| MS | 75-125% | 75-125% | 70-130% |
| LLICV | N/A | 70-130% | N/A |

Table 3

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS
BACKGROUND MOLECULAR IONS

| Molecular Ion | Mass | Element Interferences* |
|--|------|------------------------|
| NH ⁺ | 15 | |
| OH ⁺ | 17 | |
| OH ₂ ⁺ | 18 | |
| C ₂ ⁺ | 24 | |
| CN ⁺ | 26 | |
| CO ⁺ | 28 | |
| N ₂ ⁺ | 28 | |
| N ₂ H ⁺ | 29 | |
| NO ⁺ | 30 | |
| NOH ⁺ | 31 | |
| O ₂ ⁺ | 32 | |
| O ₂ H ₊ | 33 | |
| ³⁶ ArH ⁺ | 37 | |
| ³⁸ ArH ⁺ | 39 | |
| ⁴⁰ ArH ⁺ | 41 | |
| CO ₂ ⁺ | 44 | |
| CO ₂ H ⁺ | 45 | Sc |
| ArC ⁺ , ArO ⁺ | 52 | Cr |
| ArN ⁺ | 54 | Cr |
| ArNH ⁺ | 55 | Mn |
| ArO ⁺ | 56 | |
| ArOH ⁺ | 57 | |
| ⁴⁰ Ar ³⁶ Ar ⁺ | 76 | Se |
| ⁴⁰ Ar ³⁸ Ar ⁺ | 78 | Se |
| ⁴⁰ Ar ₂ ⁺ | 80 | Se |

* Method elements or internal standards affected by the molecular ions.

Table 4

MATRIX MOLECULAR IONS* No gas Mode Only

CHLORIDE

| Molecular Ion | Mass | Element Interference |
|-----------------------------|------|----------------------|
| $^{35}\text{ClO}^+$ | 51 | V |
| $^{35}\text{ClOH}^+$ | 52 | Cr |
| $^{37}\text{ClO}^+$ | 53 | Cr |
| $^{37}\text{ClOH}^+$ | 54 | Cr |
| $\text{Ar}^{35}\text{Cl}^+$ | 75 | As |
| $\text{Ar}^{37}\text{Cl}^+$ | 77 | Se |

SULFATE

| Molecular Ion | Mass | Element Interference |
|-------------------------------|------|----------------------|
| $^{32}\text{SO}^+$ | 48 | |
| $^{32}\text{SOH}^+$ | 49 | |
| $^{34}\text{SO}^+$ | 50 | V, Cr |
| $^{34}\text{SOH}^+$ | 51 | V |
| $\text{SO}_2^+, \text{S}_2^+$ | 64 | Zn |
| Ar^{32}S^+ | 72 | |
| Ar^{34}S^+ | 74 | |

PHOSPHATE

| Molecular Ion | Mass | Element Interference |
|-----------------|------|----------------------|
| PO^+ | 47 | |
| POH^+ | 48 | |
| PO_2^+ | 63 | Cu |
| ArP^+ | 71 | |

GROUP I, II METALS

| Molecular Ion | Mass | Element Interference |
|-----------------|------|----------------------|
| ArNa^+ | 63 | Cu |
| ArK^+ | 79 | |
| ArCa^+ | 80 | |

MATRIX OXIDES*

| Molecular Ion | Mass | Element Interference |
|---------------|---------|----------------------|
| TiO | 62-66 | Ni, Cu, Zn |
| ZrO | 106-112 | Ag, Cd |
| MoO | 108-116 | Cd |

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

APPENDIX 1 – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

- 1- Matrix spike recovery <50%, AND
- 2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

| | |
|----------|----------|
| Arsenic | 5mg/L |
| Barium | 100mg/L |
| Cadmium | 1 mg/L |
| Chromium | 5 mg/L |
| Lead | 5 mg/L |
| Mercury | 0.2 mg/L |
| Selenium | 1 mg/L |
| Silver | 5 mg/L |

How to run an MSA

- 1- Take 4 identical aliquots of test solution
- 2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
- 3- Perform analysis
- 4- Enter data into spreadsheet ORG-0034_TCLP_MSA
 - a. Uses unweighted least-squares linear regression curve fit
 - b. Calculates absolute value of x-intercept

Notes:

- 1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes



Reagent ID: MS A ICSAB_00170

Description: Agilent ICSAB
No. of Bottles: 1
Storage Location: Metals Standards Storage
Reagent Volume: 250.000 mL
Creation Date: 06/19/2015
Open Date:
Container(s): 663460
Comment:

Expiration Date: 06/26/2015
Laboratory: TestAmerica St. Louis
Prepared By: Buffington, Cory C
Solvent: 2% HCL 2% HN03
Solvent Lot: 661194

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|-----------------|------------------|--------------|--------------------|-------------|-------------------|
| Ag | MS CAL3 A_00006 | 02/11/2016 | 20.00000 | ug/mL | 10.00000 | ug/L |
| As | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| B | MS CAL3 A_00006 | 02/11/2016 | 200.00000 | ug/mL | 100.00000 | ug/L |
| Ba | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Be | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Cd | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Co | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Cr | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Cu | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Li | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Mn | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Ni | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Pb | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Se | MS CAL3 A_00006 | 02/11/2016 | 50.00000 | ug/mL | 25.00000 | ug/L |
| Sm | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Sr | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Th | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Tl | MS CAL3 A_00006 | 02/11/2016 | 20.00000 | ug/mL | 10.00000 | ug/L |
| U | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |



Reagent ID: MS A ICSAB_00170

Description: Agilent ICSAB
No. of Bottles: 1
Storage Location: Metals Standards Storage
Reagent Volume: 250.000 mL
Creation Date: 06/19/2015
Open Date:
Container(s): 663460
Comment:

Expiration Date: 06/26/2015
Laboratory: TestAmerica St. Louis
Prepared By: Buffington, Cory C
Solvent: 2% HCL 2% HN03
Solvent Lot: 661194

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|---------------------|------------------|--------------|--------------------|-------------|-------------------|
| V | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Zn | MS CAL3 A_00006 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Mo | MS CAL3 B_00007 | 02/11/2016 | 50.00000 | ug/mL | 2025.00000 | ug/L |
| Sb | MS CAL3 B_00007 | 02/11/2016 | 50.00000 | ug/mL | 25.00000 | ug/L |
| Sn | MS CAL3 B_00007 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Ti | MS CAL3 B_00007 | 02/11/2016 | 100.00000 | ug/mL | 2050.00000 | ug/L |
| W | MS CAL3 B_00007 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Zr | MS CAL3 B_00007 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Au | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Bi | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Ce | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Cs | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| La | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Nd | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Pd | MS CAL3 ODD A_00005 | 02/11/2016 | 10.00000 | ug/mL | 5.00000 | ug/L |
| Pr | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Pt | MS CAL3 ODD A_00005 | 02/11/2016 | 10.00000 | ug/mL | 5.00000 | ug/L |
| Re | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Rh | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |



Reagent ID: MS A ICSAB_00170

Description: Agilent ICSAB
No. of Bottles: 1
Storage Location: Metals Standards Storage
Reagent Volume: 250.000 mL
Creation Date: 06/19/2015
Open Date:
Container(s): 663460
Comment:

Expiration Date: 06/26/2015
Laboratory: TestAmerica St. Louis
Prepared By: Buffington, Cory C
Solvent: 2% HCL 2% HN03
Solvent Lot: 661194

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|----------|---------------------|------------------|--------------|--------------------|---------------|-------------------|
| Ru | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Y | MS CAL3 ODD A_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Hf | MS CAL3 ODD B_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Nb | MS CAL3 ODD B_00005 | 02/11/2016 | 50.00000 | ug/mL | 25.00000 | ug/L |
| Ta | MS CAL3 ODD B_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Te | MS CAL3 ODD B_00005 | 02/11/2016 | 100.00000 | ug/mL | 50.00000 | ug/L |
| Al | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Ca | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Chlorine | MS ICSA_00017 | 03/31/2016 | 10000.00000 | ug/mL | 1000000.00000 | ug/L |
| Fe | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| K | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Mg | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Mo | MS ICSA_00017 | 03/31/2016 | 20.00000 | ug/mL | 2025.00000 | ug/L |
| Na | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| P | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Sulfur | MS ICSA_00017 | 03/31/2016 | 1000.00000 | ug/mL | 100000.00000 | ug/L |
| Ti | MS ICSA_00017 | 03/31/2016 | 20.00000 | ug/mL | 2050.00000 | ug/L |
| Si | STD SI_00010 | 12/01/2015 | 1000.00000 | ug/mL | 500.00000 | ug/L |



Source Reagents

| Reagent | Description | Type | Expiration | Vendor | Vendor Lot # | Vendor Cat Lot # | Volume Used | Volume Units |
|------------------------|-------------|------|------------|--------------------|--------------|------------------|-------------|--------------|
| MS CAL3 A_00006 | MS CAL 3 A | ASTD | 02/11/16 | Inorganic Ventures | J2-MEB566024 | TA-CAL-3 | 0.12500 | mL |
| MS CAL3 B_00007 | CAL 3 B | ASTD | 02/11/16 | Inorganic Ventures | J2-MEB566020 | TA-CAL-3-2 | 0.12500 | mL |
| MS CAL3 ODD A_00005 | CAL 3 ODD | ASTD | 02/11/16 | Inorganic Ventures | J2-MEB566021 | TA-CAL-3-ODD | 0.12500 | mL |
| MS CAL3 ODD B_00005 | CAL 3 ODD B | ASTD | 02/11/16 | Inorganic Ventures | J2-MEB566022 | TA-CAL-3-ODD | 0.12500 | mL |
| MS ICSA_00017 | ICSA | ASTD | 03/31/16 | Inorganic Ventures | J2-MEB533111 | 6020ICS-0A | 25.00000 | mL |
| STD SI_00010 | SI 1000PPM | ASTD | 12/01/15 | INORGANIC VENTURE | G2-SI03029 | CGSI1-1 | 0.12500 | mL |



Reagent ID: MS INT STD_00006

| | | | |
|-------------------|--------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 11/01/2015 |
| Description: | INT STD | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Souris, Matthew T |
| Storage Location: | Metals Standards Storage | Vendor: | Inorganic Ventures |
| Reagent Volume: | 500.000 mL | Vendor Lot #: | H2-MEB547059 |
| Creation Date: | 08/19/2013 | Vendor Cat #: | TA-INT-STD-REV-1 |
| Open Date: | 01/08/2015 | | |
| Container(s): | 521050 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|------------------------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| Au (IS) | | | | | 100.00000 | ug/mL |
| Ge Internal Standard | | | | | 100.00000 | ug/mL |
| Ho (IS) | | | | | 100.00000 | ug/mL |
| In Internal Standard | | | | | 100.00000 | ug/mL |
| Li-6 Internal Standard | | | | | 100.00000 | ug/mL |
| Sc (IS) | | | | | 100.00000 | ug/mL |



Reagent ID: MS ICV 2_00004

| | | | |
|-------------------|--------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 11/30/2015 |
| Description: | ICV 2 - new | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Buffington, Cory C |
| Storage Location: | Metals Standards Storage | Vendor: | SPEX |
| Reagent Volume: | 250.000 mL | Vendor Lot #: | 27-169CR |
| Creation Date: | 12/08/2014 | Vendor Cat #: | ZITMO-51-250 |
| Open Date: | 12/08/2014 | | |
| Container(s): | 520114 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| Ag | | | | | 40.00000 | ug/mL |
| Ba | | | | | 200.00000 | ug/mL |
| Be | | | | | 200.00000 | ug/mL |
| Cd | | | | | 200.00000 | ug/mL |
| Co | | | | | 200.00000 | ug/mL |
| Cr | | | | | 200.00000 | ug/mL |
| Cu | | | | | 200.00000 | ug/mL |
| Li | | | | | 200.00000 | ug/mL |
| Mn | | | | | 200.00000 | ug/mL |
| Ni | | | | | 200.00000 | ug/mL |
| Pb | | | | | 200.00000 | ug/mL |
| Sm | | | | | 200.00000 | ug/mL |
| Sr | | | | | 200.00000 | ug/mL |
| Th | | | | | 200.00000 | ug/mL |
| Tl | | | | | 40.00000 | ug/mL |
| U | | | | | 200.00000 | ug/mL |
| V | | | | | 200.00000 | ug/mL |
| Zn | | | | | 200.00000 | ug/mL |
| Zr | | | | | 200.00000 | ug/mL |



Reagent ID: MS ICV 1_00004

| | | | |
|-------------------|--------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 11/30/2015 |
| Description: | ICV 1 - new | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Buffington, Cory C |
| Storage Location: | Metals Standards Storage | Vendor: | SPEX |
| Reagent Volume: | 250.000 mL | Vendor Lot #: | 27-168CR |
| Creation Date: | 12/08/2014 | Vendor Cat #: | ZITMO-50-250 |
| Open Date: | 12/08/2014 | | |
| Container(s): | 520113 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| As | | | | | 200.00000 | ug/mL |
| B | | | | | 400.00000 | ug/mL |
| Mo | | | | | 100.00000 | ug/mL |
| Sb | | | | | 100.00000 | ug/mL |
| Se | | | | | 100.00000 | ug/mL |
| Sn | | | | | 200.00000 | ug/mL |
| Ti | | | | | 200.00000 | ug/mL |
| W | | | | | 200.00000 | ug/mL |



Reagent ID: MS CAL3 A_00006

| | | | |
|-------------------|--------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/11/2016 |
| Description: | MS CAL 3 A | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Buffington, Cory C |
| Storage Location: | Metals Standards Storage | Vendor: | Inorganic Ventures |
| Reagent Volume: | 500.000 mL | Vendor Lot #: | J2-MEB566024 |
| Creation Date: | 02/11/2015 | Vendor Cat #: | TA-CAL-3 |
| Open Date: | 02/11/2015 | | |
| Container(s): | 559278 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| Ag | | | | | 20.00000 | ug/mL |
| As | | | | | 100.00000 | ug/mL |
| B | | | | | 200.00000 | ug/mL |
| Ba | | | | | 100.00000 | ug/mL |
| Be | | | | | 100.00000 | ug/mL |
| Cd | | | | | 100.00000 | ug/mL |
| Co | | | | | 100.00000 | ug/mL |
| Cr | | | | | 100.00000 | ug/mL |
| Cu | | | | | 100.00000 | ug/mL |
| Li | | | | | 100.00000 | ug/mL |
| Mn | | | | | 100.00000 | ug/mL |
| Ni | | | | | 100.00000 | ug/mL |
| Pb | | | | | 100.00000 | ug/mL |
| Se | | | | | 50.00000 | ug/mL |
| Sm | | | | | 100.00000 | ug/mL |
| Sr | | | | | 100.00000 | ug/mL |
| Th | | | | | 100.00000 | ug/mL |
| Tl | | | | | 20.00000 | ug/mL |
| U | | | | | 100.00000 | ug/mL |
| V | | | | | 100.00000 | ug/mL |
| Zn | | | | | 100.00000 | ug/mL |



Reagent ID: MS CAL 1 A_00001

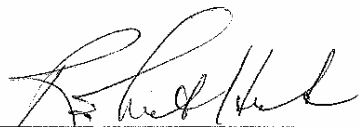
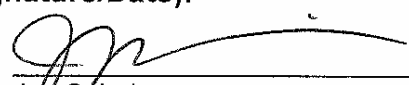
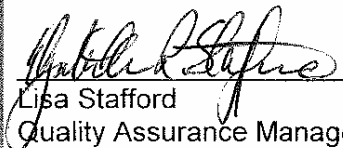

| | | | |
|-------------------|--------------------------|------------------|-----------------------|
| Type: | ASTD | Expiration Date: | 02/23/2016 |
| Description: | MS CAL 1 A | Laboratory: | TestAmerica St. Louis |
| No. of Bottles: | 1 | Prepared By: | Buffington, Cory C |
| Storage Location: | Metals Standards Storage | Vendor: | Inorganic Ventures |
| Reagent Volume: | 500.000 mL | Vendor Lot #: | J2-MEB566091 |
| Creation Date: | 02/23/2015 | Vendor Cat #: | TA-CAL-1 |
| Open Date: | 02/23/2015 | | |
| Container(s): | 567607 | | |
| Comment: | | | |

Reagent Analyte Information

| Analyte | Source ID | Source Exp. Date | Source Conc. | Source Conc. Units | Final Conc. | Final Conc. Units |
|---------|-----------|------------------|--------------|--------------------|-------------|-------------------|
| Ag | | | | | 2.00000 | ug/mL |
| As | | | | | 10.00000 | ug/mL |
| B | | | | | 50.00000 | ug/mL |
| Ba | | | | | 2.00000 | ug/mL |
| Be | | | | | 0.50000 | ug/mL |
| Cd | | | | | 0.50000 | ug/mL |
| Co | | | | | 2.00000 | ug/mL |
| Cr | | | | | 10.00000 | ug/mL |
| Cu | | | | | 1.00000 | ug/mL |
| Li | | | | | 5.00000 | ug/mL |
| Mn | | | | | 2.00000 | ug/mL |
| Ni | | | | | 5.00000 | ug/mL |
| Pb | | | | | 3.00000 | ug/mL |
| Se | | | | | 5.00000 | ug/mL |
| Sm | | | | | 10.00000 | ug/mL |
| Sr | | | | | 5.00000 | ug/mL |
| Th | | | | | 2.00000 | ug/mL |
| Tl | | | | | 2.00000 | ug/mL |
| U | | | | | 1.00000 | ug/mL |
| V | | | | | 10.00000 | ug/mL |
| Zn | | | | | 10.00000 | ug/mL |

Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

| Approvals (Signature/Date): | | | |
|---|------------------|--|----------------|
|  | <u>7/14/15</u> |  | <u>7/14/15</u> |
| Robert Hrabak Operations Manager | Date | Joe Schairer Health & Safety Manager / Coordinator | Date |
|  | <u>7/20/2015</u> |  | <u>7.20.15</u> |
| Lisa Stafford Quality Assurance Manager | Date | Crystal Pollock Laboratory Director | Date |

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| | |
|---|---|
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|---|---|

1. SCOPE AND APPLICATION

- 1.1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290 and 8290A. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.
- 1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.
- 1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).
- 1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.
- 1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

- 2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques. Sample preparation is addressed in WS-IDP-0005.
- 2.2. One to two μL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).
- 2.3. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C -labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective isotope dilution analyte or internal standard signal) and simultaneous detection of the two most abundant ions in

the molecular ion region. All other identified PCDD/PCDF congeners are identified by their RRT's based on the daily CCV standard, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

- 2.4. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).
- 3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.
- 3.3. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.
- 3.4. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.
- 3.5. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.
- 3.6. Congener: Any isomer of any homologous series.
- 3.7. Isotope Dilution Analyte: An isotope dilution analyte is a ^{13}C -labeled analog of a congener chosen from the compounds listed in Table 3. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.
- 3.8. Internal Standard: Two internal standards are used to determine the percent recoveries for the isotope dilution analytes. The ^{13}C -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated isotope dilution analytes while ^{13}C -1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-, hepta- and

octachlorinated isotope dilution analytes. ^{13}C -1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

- 3.9. Estimated Detection Limit (EDL)/ Estimated Quantitation Limit (EQL): The sample specific estimated detection limit (EDL/EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.
- 3.10. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

4. INTERFERENCES

- 4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.
- 4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.
- 4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.
- 4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.
- 4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when 2,3,7,8-TCDF on the DB-5 column is detected.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

- 5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.
- 5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.
- 5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|--|---------------------------------|--------------------------------|--|
| Acetone | Flammable | 1000 ppm-TWA | Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. |
| Hexane | Flammable Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| Iso-octane | Flammable Irritant | None established | Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath. |
| Methanol | Flammable Poison Irritant | 200 ppm-TWA | A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes. |
| Methylene Chloride | Carcinogen Irritant | 25 ppm-TWA 125 ppm-STEL | Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin. |
| Tetradecane | Irritant | None established | Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage. |
| Toluene | Flammable Poison Irritant | 200 ppm-TWA 300 ppm-Ceiling | Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

6. EQUIPMENT AND SUPPLIES

- 6.1. Preventive and routine maintenance is described in the ‘Schedule of Routine Maintenance’ in the QAM.
- 6.2. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).
 - 6.2.1. Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software and the Autospec Premiere system utilizes MassLynx version 4.1 software.
 - 6.2.2. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle

injection port is also acceptable. When using the method described in this protocol, a 2- μ L injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 μ L). 1 μ L injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

- 6.2.3. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.
- 6.2.4. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.
- 6.2.5. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.3. GC Column

- 6.3.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when 2,3,7,8-TCDF is detected.

- 6.3.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

7. REAGENTS AND STANDARDS

7.1. Solvents

- 7.1.1. High-purity, distilled-in-glass or highest available purity: methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, and acetone.

- 7.2. All calibration, daily isotope dilution analyte, daily clean up internal standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

- 7.2.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.3. Calibration Solutions

- 7.3.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/ μ L) and the highest for the octachlorinated congeners (2000 pg/ μ L).

- 7.3.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

- 7.3.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

- 7.3.4. Standards for method 8290A require storage at $\leq 6^{\circ}\text{C}$.
- 7.4. GC Column Performance Check Solution
- 7.4.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C -2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/ μL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.
- 7.4.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of $\leq 25\%$. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.
- 7.5. Field Surrogate Solution (air matrices)
- 7.5.1. This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Methods 23 and/or 0023A) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.
- 7.6. Sample Fortification Solution (Isotope dilution analyte)
- 7.6.1. This isooctane (or toluene) solution contains the nine isotope dilution analytes at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C -OCDF is not present in the solution.)
- 7.7. Internal Standard Solution
- 7.7.1. This tetradecane solution contains two internal standards (^{13}C -1,2,3,4-TCDD and ^{13}C -1,2,3,7,8,9-HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.
- 8.2. Grab and composite samples must be collected in glass containers.
- 8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.
- 8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).
- 8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.
- 8.6. With the exception of the fish tissues, which must be stored at -20°C , all samples should be stored at $4^{\circ}\text{C} \pm 2$, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.
- 8.7. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at $\leq 6^{\circ}\text{C}$.

9. QUALITY CONTROL

- 9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

- 9.1.1. The method blank must be spiked prior to extraction with the same amount of ¹³C-labeled isotope dilution analytes as added to samples.
- 9.1.2. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.
 - 9.1.2.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.
 - 9.1.2.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.
 - 9.1.2.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.
- 9.1.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.
- 9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

- 9.2.1. A LCS is deemed acceptable if control analytes are above upper control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.
- 9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.
 - 9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.
 - 9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.
 - 9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").
 - 9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.
 - 9.3.5. Analyze the MS and MSD samples as described in Section 11.

- 9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.
- 9.3.7. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.
- 9.4. Duplicates
- 9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.
- 9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.
- 9.4.2. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.
- 9.5. Surrogate/Clean Up Internal Standard
- A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up internal standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of isotope dilution analyte during both extraction and cleanup.
- 9.6. Isotope Dilution Analytes
- 9.6.1. Isotope dilution analytes must be spiked into all samples, QC samples, and included in all calibrations.
- 9.6.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine isotope dilution analytes.
- 9.6.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Isotope dilution analyte recoveries are flagged if they are outside the

recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.7. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Tuning (Mass Resolution Check)

10.2.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.2.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

- 10.2.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 292.9825 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF).
- 10.2.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile for all the descriptors. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 3) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3. Performance Checks

- 10.3.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 4 (HRCC-4) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-3 or HRCC-5 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.3.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

- 10.3.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C -HxCDF and ^{13}C -HpCDF rather than M+2

and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

10.3.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.4. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.4.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.4.2. Tune the instrument with PFK.

10.4.3. Inject 1 or 2 μL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.

10.4.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.4.3.2. The same injection volume must be used for all samples, QC, and standards.

10.4.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or 2- μL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

- 10.4.4.1. The total cycle time for data acquisition must be < 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.
- 10.4.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.
- 10.4.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).
- 10.4.4.4. The ratio of integrated ion current for the ions belonging to the ¹³C labeled isotope dilution analytes and internal standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

- 10.4.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.
- 10.4.5.1. Referring to Table 5, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate isotope dilution analytes (Table 5) and the nine RRFs for the labeled ¹³C isotope dilution analytes [RRF(m); m=18 to 26] relative to the two internal standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{IDA}}{Q_x \times A_{IDA}} \quad RRF(m) = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for unlabeled PCDDs/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

Q_{IDA} = quantity of the isotope dilution analyte injected (pg),

Q_{IS} = quantity of the internal standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Q_{IDA} , Q_{IS} , and Q_x must be the same.

10.4.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = \left(\frac{1}{5}\right) \sum_{j=1}^5 RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 5), and j is the injection number (or calibration solution number; j = 1 to 5).

10.4.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

10.4.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain ^{13}C -OCDF as an isotope dilution analyte. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of ^{13}C -OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from ^{13}C -OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C -OCDD.

10.4.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = \left(\frac{1}{t}\right) \sum_{n=1}^t RRF_n$$

Where:

k = 27 to 30, with 27 = PeCDF;

28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.4.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine isotope dilution analytes are calculated as follows:

$$RRF(m) = \frac{A_{IDA}^m \times Q_{IS}}{Q_{IDA}^m \times A_{IS}}$$

$$\overline{RRF}(m) = \left(\frac{1}{5}\right) \sum_{j=1}^5 RRF_j(m)$$

Where:

- m = 18 to 26 (congener type)
- j = 1 to 5 (injection number),
- A_{IDA}^m = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
- A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
- Q_{IDA} & Q_{IDA}^m = quantities of, respectively, the internal standard (rs) and a particular isotope dilution analyte (m) injected (pg),
- RRF(m) = relative response factor of a particular isotope dilution analyte (m) relative to an appropriate internal standard, as determined from one injection, and
- $\overline{RRF}(m)$ = calculated mean relative response factor of a particular isotope dilution analyte, as determined from the five initial calibration injections (j).

10.5. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

- 10.5.1. The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

Note: If Method 8290A criteria are required for the project then both the percent standard relative standard deviation for the mean response factors for the 17 unlabeled standards and the nine labeled reference compounds must be ≤ 20 percent.

- 10.5.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10 .

- 10.5.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.6. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

- 10.6.1. Inject 1 or 2 μL of the concentration calibration solution HRCC-4 containing 10 $\text{pg}/\mu\text{L}$ of tetrachlorinated congeners, 50 $\text{pg}/\mu\text{L}$ of penta-, hexa-, and heptachlorinated congeners, 100 $\text{pg}/\mu\text{L}$ of octachlorinated congeners, and the respective isotope dilution analyte and internal standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

10.7. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.7.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.7.1.1. The bracketing continuing calibration must be $\pm 20\%$ of the average RRF calculated from the initial calibration.

10.7.1.1.1. If the target compounds in the ending standard are less than or equal to $\pm 20\%$ of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.7.1.1.2. If the target analytes are greater than $\pm 20\%$ but less or equal to $\pm 25\%$ and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than $\pm 20\%$ but less or equal to $\pm 25\%$ and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.7.1.1.3. If the percent deviation of unlabeled compounds exceeds $\pm 25\%$, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.7.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.7.2.1. The bracketing continuing calibration must be $\pm 30\%$ of the average RRF calculated from the initial calibration.

10.7.2.1.1. If the labelled compounds in the ending standard are less than or equal to $\pm 30\%$ of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.7.2.1.2. If the isotope dilution analyte analytes are greater than $\pm 30\%$ but less or equal to $\pm 35\%$, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

- 10.7.2.1.3. If the percent deviation of labeled compounds exceeds $\pm 35\%$, reanalyze samples if adversely impacted.
- 10.7.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.
- 10.7.4. If either criteria in Sections 10.7.1 or 10.7.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.4.3 for resolution.
- 10.7.5. If the above criteria (Section 10.7) cannot be satisfied, the entire initial calibration process (Section 10.4) must be repeated.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Dilution Procedure – Simple Dilutions

Dilutions from 2X to 20X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$(\text{Concentration of the original extract}) \times (\text{amount of aliquot taken}) \times (\text{volume of diluted extract}) = \text{final concentration of dilution.}$$

Ex: 20X dilution of original 10 g/20 μL sample

$$(10 \text{ g}/20 \mu\text{L}) \times (2 \mu\text{L aliquot} + 38 \mu\text{L keeper}) = 1 \text{ g}/40 \mu\text{L FV}$$

Record the final sample concentration on the extract label.

11.3. Sample Dilution Procedure – Complex Dilutions

Complex dilution requiring respiking of IDA and IS: Dilutions greater than 20x must be done by diluting and respiking the extract with IDA's and IS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 μL final volume)

Take a 2 μL aliquot (1/10 of original sample) and add 18 μL of solvent keeper. Take a 2 μL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IDA and 20 μL IS, reduced to 20 μL FV.

Record the final sample concentration of the extract label.

11.4. Analytical Procedures

- 11.4.1. Inject a 1 or 2 μL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.
- 11.4.2. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

12. CALCULATIONS/DATA REDUCTION

12.1. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.1.1. Retention Times

- 12.1.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled isotope dilution analyte or internal standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled isotope dilution analyte or internal standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.
- 12.1.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled isotope dilution analyte present in the sample extract, the relative retention time (relative to the appropriate isotope dilution analyte) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C -OCDD as determined from the daily routine calibration results.

12.1.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.1.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.1.1.5. The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C -TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.1.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

12.1.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 4 describes the procedure to be followed for the determination of the S/N.

12.1.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12.2. For gas chromatographic peaks that have met the criteria outlined above, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{IDA}}{A_{IDA} \times W \times RRF(n)}$$

Where:

- C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,
- A_x = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,
- A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

Q_{IDA} = quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).

$RRF(n)$ = Calculated mean relative response factor for the analyte [RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1.

However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

- 12.3. Calculate the percent recovery of the nine isotope dilution analytes measured in the sample extract, using the formula:

$$\text{Isotope Dilution Analytes Percent Recovery} = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF(m)} \times 100$$

Where:

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard; the selection of the internal standard depends on the type of congeners (see Table 5, footnotes),

Q_{IDA} = Quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

Q_{IS} = Quantity, in pg, of the internal standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$RRF(m)$ = calculated mean relative response factor for the labeled isotope dilution analyte relative to the appropriate (see Table 5, footnotes) internal standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

- 12.4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

- 12.4.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

- 12.4.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.
- 12.5. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.
- 12.5.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.
- 12.5.2. Extraction of an aliquot large enough to be representative with an increased concentration of isotope dilution analyte and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.
- 12.5.3. Dilution of the original extract. Isotope dilution analyte components are re-spike at an appropriate level prior to analysis. In this case, the isotope dilution analyte recoveries are taken from the original analysis.
- 12.6. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.
- 12.7. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.
- 12.8. **Sample-Specific Estimated Detection Limit**
The sample-specific estimated detection limit (EDL) or estimated quantitation limit (EQL, 8290A) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL/EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.
- 12.8.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression for EDL/EQL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL/EQL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the isotope dilution analyte (if the congener possesses an isotope dilution analyte) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ¹³C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their isotope dilution analyte must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{\text{Specific 2,3,7,8-subst. PCDD/PCDF}} = \frac{2.5 \times H_x \times Q_{IDA}}{H_{IDA} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs. (also EQL for Method 8290A)

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{IDA} = height of one of the quantitation ions (Table 6) for the labeled isotope dilution analytes.

W, RRF (n), and Q_{IDA} retain the same meanings as defined in Section 12.2

- 12.8.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.1, calculate the “Estimated Maximum Possible Concentration” (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDLEQL can be calculated using the above formula and the height of one of the ions as appropriate.

12.9. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.10. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 10). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

12.11. Two-GC Column TEF Determination

12.11.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq 25\%$ valley.

12.11.2. For samples that have a positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result above the lower calibration limit is calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be $< 25\%$ valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.11.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.11.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.005 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 11 and the results from the routine

calibration run on the DB-5 column.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.5. GC Column Performance

13.5.1. Inject 1 or 2 μL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of < 1 second.

13.5.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of ≤ 25

percent (Figure 2),

Where:

$$\text{Valley Percent} = \left(\frac{x}{y}\right) \times 100$$

x = measured as in Figure 2 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

- 13.5.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.
- 13.5.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Autovials containing assorted solvents and extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

- 16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.
- 16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.
- 16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.
- 16.4. Compendium Method TO-9A "Determination of Polychlorinated, Polybrominated, and Brominated, Chlorinated Dibenzo-p-dioxins and Dibenzofurans in Ambient Air", EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.
- 16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.
- 16.6. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)
- 16.7. "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.
- 16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

- 17.1. Modifications from EPA 8290 and EPA 8290A
 - 17.1.1. The methods specify that 2 μ L injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 μ L injections for all performance checks, standards, QC samples, and samples.
 - 17.1.2. In Section 2.7 of Method 8290 and 8290A, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled isotope dilution analytes. All available labeled isotope dilution analytes are used; therefore, a retention time window

of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290 and 7.9 of Method 8290A.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.2. Modifications from TO-9A method

17.2.1. The ³⁷Cl-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ μL).

17.2.2. The laboratory uses 2 labeled internal standards for the quantitation of labeled isotope dilution analytes.

17.2.3. The final volume is adjusted to 20 μL in tetradecane.

17.2.4. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of the Sample Fortification and Internal Standard Solutions.

18.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

18.5. Table 5 - Concentrations of Calibration Solutions

18.6. Table 6 - Ions Monitored for PCDDs/PCDFs

18.7. Table 7 - Recommended GC Operating Conditions

18.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

18.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

18.10. Table 10 - 2,3,7,8-TCDD Equivalent Factors

18.11. Table 11 - TEF: Analyte Relative Retention Time Reference Attributes

18.12. Figure 1 - Compound Structure

18.13. Figure 2 - GC Performance Check Chromatogram on the DB-5 Column

18.14. Figure 3 - PFK Peak Profile

18.15. Figure 4 - Manual Determination of Signal-to-Noise

18.16. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-ID-0005 Revision 7.7, Effective 07/22/2015

19.1.1. Updated Copyright information on Title Page.

19.1.2. Changed Section 10.2.3 from “By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK)...”, to “By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 292.098285 (PFK)...”. Deleted the last sentence of this Section.

19.1.3. Changed Section 10.2.4 from “Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760)..” to “Documentation of the instrument resolving power must then be accomplished by recording the peak profile for all the descriptors.”

19.1.4. Sections 11.2 and 11.3 – Changed all 50x dilutions to 20x and changed example in Section 11.2 .to:

Ex. $(10 \text{ g}/20 \text{ }\mu\text{L}) \times (2 \text{ }\mu\text{L} \text{ aliquot} + 38 \text{ }\mu\text{L} \text{ keeper}) = 1 \text{ g}/40 \text{ }\mu\text{L} \text{ FV}$

19.1.5. Editorial changes.

19.2. WS-ID-0005, Revision 7.6, Effective 06/06/2014

19.2.1. Changed Section 12.11.5 from “...carbon-labeled analogs are available must fall within 0.006 units...” to “...carbon-labeled analogs are available must fall within 0.005 units....”.

19.2.2. Editorial changes.

19.3. WS-ID-0005, Revision 7.5, Effective 04/19/2013

19.3.1. Replaced all instances of ‘internal standard’ with ‘isotope dilution analyte’ and all instances of ‘recovery standard’ with ‘internal standard’ to conform with

TALS naming guidelines.

19.3.2. Editorial revisions.

19.4. WS-ID-0005, Revision 7.4, Effective 01/14/2011.

19.4.1. Editorial revisions.

19.5. WS-ID-0005, Revision 7.3, Effective 12/30/2009

19.5.1. Editorial revisions.

19.6. WS-ID-0005, Revision 7.2, Effective 11/02/2009

19.6.1. Section 6.1: Inserted "Preventive and routine maintenance is described in the 'Schedule of Routine Maintenance' in the QAM."

19.6.2. Section 12.1.2: Removed the word "presumptive" and inserted "above the lower calibration limit" after the word result.

TABLE 1

**Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based
 Method Calibration Limits (Parts per Trillion)**

| | Water | Soil Sediment Paper Pulp | Fly Ash | Human/ Fish Tissue | Adipose Tissue | Sludges, Fuel Oil | Still- Bottom | Ambient or Source Samples |
|------------------------------|--------------|---|--------------------|-----------------------------------|---------------------------|------------------------------|--------------------------|--|
| Lower MCL(a) | 0.01 | 1.0 | 2.0 | 1.0 | 2.0 | 10 | 20 | 40 |
| Upper MCL(a) | 4.0 | 400 | 400 | 400 | 400 | 2000 | 4000 | 8000 |
| Weight (g) | 1000 | 10 | 10 | 10 | 10 | 2.0 | 1.0 | 1 sample |
| | | | | | | | | |
| IDA Spiking Levels (ng) | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 4.0 |
| | | | | | | | | |
| Final Extract Volume (µL) | 20 | 20 | 20 | 20 | 20 | 20 | 20 | 20 |

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2
**Composition of the Sample Fortification
 and Internal Standard Solutions**

| Analyte | Sample Fortification Solution Concentration pg/ μ L; Solvent: Isooctane | Internal Standard Solution Concentration pg/ μ L; Solvent: Tetradecane |
|--|---|--|
| ^{13}C -2,3,7,8-TCDD | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -2,3,7,8-TCDF | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -1,2,3,4-TCDD | -- | 100 |
| ^{13}C -1,2,3,7,8-PeCDD | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -1,2,3,7,8-PeCDF | 2 ^(a) , 100 ^(c) | -- |
| | | |
| ^{13}C -1,2,3,6,7,8-HxCDD | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -1,2,3,4,7,8-HxCDF ^(d) | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -1,2,3,7,8,9-HxCDD | -- | 100 |
| | | |
| ^{37}Cl -2,3,7,8-TCDD ^{(b)(c)} | 0.8 ^(b) , 100 ^(c) | |
| | 100 ^(c) | |
| ^{13}C -2,3,4,7,8-PeCDF ^(c) | 100 ^(c) | |
| ^{13}C -1,2,3,6,7,8-HxCDF ^{(c)(d)} | 100 ^(c) | |
| ^{13}C -1,2,3,4,7,8-HxCDD ^(c) | 100 ^(c) | |
| ^{13}C -1,2,3,4,7,8,9-HpCDD ^(c) | 100 ^(c) | |
| | | |
| ^{13}C -1,2,3,4,6,7,8-HpCDD | 2 ^(a) , 100 ^(c) | -- |
| ^{13}C -1,2,3,4,6,7,8-HpCDF | 2 ^(a) , 100 ^(c) | -- |
| | | |
| ^{13}C -OCDD | 4 ^(a) , 200 ^(c) | -- |

(a) Standard 8290, 8290A, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations

(b) Method TO9 and TO9A surrogate concentrations

(c) Method 23 and Method 0023A surrogate concentrations

(d) ^{13}C -1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and ^{13}C -1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

| PCDD | PCDF |
|-------------------------|------------------------|
| 2,3,7,8-TCDD(*) | 2,3,7,8-TCDF(*) |
| 1,2,3,7,8-PeCDD(*) | 1,2,3,7,8-PeCDF(*) |
| 1,2,3,6,7,8-HxCDD(*) | 2,3,4,7,8-PeCDF |
| 1,2,3,4,7,8-HxCDD | 1,2,3,6,7,8-HxCDF |
| 1,2,3,7,8,9-HxCDD(+) | 1,2,3,7,8,9-HxCDF |
| 1,2,3,4,6,7,8-HpCDD(*) | 1,2,3,4,7,8-HxCDF(*) |
| 1,2,3,4,5,6,7,8-OCDD(*) | 2,3,4,6,7,8-HxCDF |
| | 1,2,3,4,6,7,8-HpCDF(*) |
| | 1,2,3,4,7,8,9-HpCDF |
| | 1,2,3,4,5,6,7,8-OCDF |

(*)The ¹³C -labeled analog is used as an isotope dilution analyte.

(+)The ¹³C -labeled analog is used as a internal standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

| # of Chlorine Atoms | # of Dioxin Isomers | # of 2,3,7,8 Isomers | # of Furan Isomers | # of 2,3,7,8 Isomers |
|----------------------------|----------------------------|-----------------------------|---------------------------|-----------------------------|
| 1 | 2 | --- | 4 | --- |
| 2 | 10 | --- | 16 | --- |
| 3 | 14 | --- | 28 | --- |
| 4 | 22 | 1 | 38 | 1 |
| 5 | 14 | 1 | 28 | 2 |
| 6 | 10 | 3 | 16 | 4 |
| 7 | 2 | 1 | 4 | 2 |
| 8 | 1 | 1 | 1 | 1 |
| Total | 75 | 7 | 135 | 10 |

TABLE 5

High Resolution Concentration Calibration Solutions

| RRF (n)(m) | Compound | Concentration (ng/mL) | | | | |
|---------------|--|-----------------------|-----|-----------------|-----|------|
| | | CS2 | CS3 | CS4 (ICV(6)) | CS5 | CS6 |
| | Native CDDs and CDFs | | | | | |
| 1 | 2,3,7,8-TCDD | 0.5 | 2 | 10 | 40 | 200 |
| 2 | 2,3,7,8-TCDF | 0.5 | 2 | 10 | 40 | 200 |
| 3 | 1,2,3,7,8-PeCDD | 2.5 | 10 | 50 | 200 | 1000 |
| 4 | 1,2,3,7,8-PeCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 5 | 2,3,4,7,8-PeCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 6 | 1,2,3,4,7,8-HxCDD | 2.5 | 10 | 50 | 200 | 1000 |
| 7 | 1,2,3,6,7,8-HxCDD | 2.5 | 10 | 50 | 200 | 1000 |
| 8 | 1,2,3,7,8,9-HxCDD | 2.5 | 10 | 50 | 200 | 1000 |
| 9 | 1,2,3,4,7,8-HxCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 10 | 1,2,3,6,7,8-HxCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 11 | 1,2,3,7,8,9-HxCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 12 | 2,3,4,6,7,8-HxCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 13 | 1,2,3,4,6,7,8-HpCDD | 2.5 | 10 | 50 | 200 | 1000 |
| 14 | 1,2,3,4,6,7,8-HpCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 15 | 1,2,3,4,7,8,9-HpCDF | 2.5 | 10 | 50 | 200 | 1000 |
| 16 | OCDD | 5.0 | 20 | 100 | 400 | 2000 |
| 17 | OCDF | 5.0 | 20 | 100 | 400 | 2000 |
| | Labeled CDDs and CDFs | | | | | |
| 18 | ¹³ C ₁₂ -2,3,7,8-TCDD | 100 | 100 | 100 | 100 | 100 |
| 19 | ¹³ C ₁₂ -2,3,7,8-TCDF | 100 | 100 | 100 | 100 | 100 |
| 20 | ¹³ C ₁₂ -1,2,3,7,8-PeCDD | 100 | 100 | 100 | 100 | 100 |
| 21 | ¹³ C ₁₂ -1,2,3,7,8-PeCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -2,3,4,7,8-PeCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -1,2,3,4,7,8-HxCDD | 100 | 100 | 100 | 100 | 100 |
| 22 | ¹³ C ₁₂ -1,2,3,6,7,8-HxCDD | 100 | 100 | 100 | 100 | 100 |
| 23 | ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -1,2,3,6,7,8-HxCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -1,2,3,7,8,9-HxCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -2,3,4,6,7,8-HxCDF | 100 | 100 | 100 | 100 | 100 |
| 24 | ¹³ C ₁₂ -1,2,3,4,6,7,8- HpCDD | 100 | 100 | 100 | 100 | 100 |
| 25 | ¹³ C ₁₂ -1,2,3,4,6,7,8- HpCDF | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -1,2,3,4,7,8,9- | 100 | 100 | 100 | 100 | 100 |

| RRF (n)(m) | Compound | Concentration (ng/mL) | | | | |
|---------------|--|-----------------------|-----|-----------------|-----|-----|
| | | CS2 | CS3 | CS4 (ICV(6)) | CS5 | CS6 |
| 26 | HpCDF | | | | | |
| | ¹³ C ₁₂ -OCDD | 200 | 200 | 200 | 200 | 200 |
| | Cleanup Standard/ FS | | | | | |
| | ³⁷ Cl ₄ -2,3,7,8-TCDD | 0.5 | 2 | 10 | 40 | 200 |
| | Internal Standards | | | | | |
| | ¹³ C ₁₂ -1,2,3,4-TCDD | 100 | 100 | 100 | 100 | 100 |
| | ¹³ C ₁₂ -1,2,3,7,8,9-HxCDD | 100 | 100 | 100 | 100 | 100 |

TABLE 6*
Elemental Compositions and Exact Masses of the Ions
Monitored by HR/MS for PCDD's and PCDF's

| Descriptor | Exact m/z ⁽¹⁾ | m/z Type | Elemental Composition | Substance ⁽²⁾ | |
|------------|--------------------------|----------|---|--|-------|
| 1 | 292.9825 | QC | C ₇ F ₁₁ | PFK | |
| | 303.9016 | M | C ₁₂ H ₄ ³⁵ Cl ₄ O | TCDF | |
| | 305.8987 | M+2 | C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO | TCDF | |
| | 315.9419 | M | ¹³ C ₁₂ H ₄ ³⁵ Cl ₄ O | TCDF ⁽³⁾ | |
| | 317.9389 | M+2 | ¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO | TCDF ⁽³⁾ | |
| | 319.8965 | M | C ₁₂ H ₄ ³⁵ Cl ₄ O ₂ | TCDD | |
| | 321.8936 | M+2 | C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO ₂ | TCDD | |
| | 327.8847 | M | C ₁₂ H ₄ ³⁷ Cl ₄ O ₂ | TCDD ⁽⁴⁾ | |
| | 330.9792 | Lock | C ₇ F ₁₃ | PFK | |
| | 331.9368 | M | ¹³ C ₁₂ H ₄ ³⁵ Cl ₄ O ₂ | TCDD ⁽³⁾ | |
| | 333.9339 | M+2 | ¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO ₂ | TCDD ⁽³⁾ | |
| | 339.8597 | M+2 | C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO | PeCDF | |
| | 341.8567 | M+4 | C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ ClO | PeCDF | |
| | 375.8364 | M+2 | C ₁₂ H ₄ ³⁵ Cl ₅ ³⁷ ClO | HxCDFPE | |
| | 409.7974 | M+2 | C ₁₂ H ₃ ³⁵ Cl ₆ ³⁷ ClO | HpCDFPE | |
| 2 | 330.9792 | QC | C ₇ F ₁₃ | PFK | |
| | 339.8597 | M+2 | C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO | PeCDF | |
| | 341.8567 | M+4 | C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O | PeCDF | |
| | 342.9792 | Lock | C ₈ F ₁₂ | PFK | |
| | 351.9000 | M+2 | ¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO | PeCDF | |
| | 353.8970 | M+4 | ¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO | PeCDF ⁽³⁾ | |
| | 354.9792 | Lock | C ₉ F ₁₃ | PFK | |
| | 355.8546 | M+2 | C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂ | PeCDD | |
| | 357.8516 | M+4 | C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂ | PeCDD | |
| | 366.9793 | QC | C ₉ F ₁₃ | PFK | |
| | 367.8949 | M+2 | ¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂ | PeCDD ⁽³⁾ | |
| | 369.8919 | M+4 | ¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂ | PeCDD ⁽³⁾ | |
| | 409.7974 | M+2 | C ₁₂ H ₃ ³⁵ Cl ₆ ³⁷ ClO | HpCDFPE | |
| | 3 | 373.8208 | M+2 | C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO | HxCDF |
| | | 375.8178 | M+4 | C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O | HxCDF |
| 380.9760 | | Lock | C ₈ F ₁₅ | PFK | |
| 383.8639 | | M | ¹³ C ₁₂ H ₂ ³⁵ Cl ₆ O | HxCDF ⁽³⁾ | |
| 385.8610 | | M+2 | ¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO | HxCDF ⁽³⁾ | |
| 389.8157 | | M+2 | C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂ | HxCDD | |
| 391.8127 | | M+4 | C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂ | HxCDD | |
| 392.9760 | | Lock | C ₉ F ₁₅ | PFK | |
| 401.8559 | | M+2 | ¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂ | HxCDD ⁽³⁾ | |
| 403.8529 | | M+4 | ¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂ | HxCDD ⁽³⁾ | |
| 430.9728 | | QC | C ₉ F ₁₇ | PFK | |
| 445.7550 | | M+4 | C ₁₂ H ₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O | OCDFPE | |
| 4 | | 392.9760 | QC | C ₉ F ₁₅ | PFK |
| | | 407.7818 | M+2 | C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO | HpCDF |
| | | 409.7789 | M+4 | C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O | HpCDF |

| Descriptor | Exact m/z ⁽¹⁾ | m/z Type | Elemental Composition | Substance ⁽²⁾ |
|------------|--------------------------|----------|--|--------------------------|
| | 417.8253 | M | ¹³ C ₁₂ H ³⁵ Cl ₇ O | HpCDF ⁽³⁾ |
| | 419.8220 | M+2 | ¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO | HpCDF ⁽³⁾ |
| | 423.7766 | M+2 | C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO ₂ | HpCDD |
| | 425.7737 | M+4 | C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂ | HpCDD |
| | 430.9729 | Lock | C ₉ F ₁₇ | PFK |
| | 435.8169 | M+2 | ¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO ₂ | HpCDD ⁽³⁾ |
| | 437.8140 | M+4 | ¹³ C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂ | HpCDD ⁽³⁾ |
| | 479.7165 | M+4 | C ₁₂ H ³⁵ Cl ₇ ³⁷ Cl ₂ O | NCDPE |
| 5 | 392.9760 | QC | C ₉ F ₁₅ | PFK |
| | 441.7428 | M+2 | C ₁₂ ³⁵ Cl ₇ ³⁷ ClO | OCDF |
| | 442.9728 | Lock | C ₁₀ F ₁₇ | PFK |
| | 443.7399 | M+4 | C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O | OCDF |
| | 457.7377 | M+2 | C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂ | OCDD |
| | 459.7348 | M+4 | C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂ | OCDD |
| | 469.7779 | M+2 | ¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂ | OCDD ⁽³⁾ |
| | 471.7750 | M+4 | ¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂ | OCDD ⁽³⁾ |
| | 479.7165 | M+4 | C ₁₂ Cl ₈ ³⁷ Cl ₂ O | NCDPE |
| | 513.6775 | M+4 | ¹³ C ₁₂ ³⁵ Cl ₈ ³⁷ Cl ₂ O | DCDPE |

^(a) The following nuclidic masses were used:

| | |
|-----------------------------|------------------------------|
| H = 1.007825 | O = 15.994915 |
| C = 12.000000 | ³⁵ Cl = 34.968853 |
| ¹³ C = 13.003355 | ³⁷ Cl = 36.965903 |
| F = 18.9984 | |

S = Isotope dilution analyte/internal standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes))
 Are as Follows:

Injector Temperature: 280°C
 Interface Temperature: 280°C
 Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column^(b)

| # of Chlorine Atoms | PCDD Positional Isomer | | PCDF Positional Isomer | |
|---------------------|------------------------|---------------|------------------------|-----------------|
| | Early Eluter | Late Eluter | Early Eluter | Late Eluter |
| 4 ^(a) | 1,3,6,8 | 1,2,8,9 | 1,3,6,8 | 1,2,8,9 |
| 5 | 1,2,4,6,8/1,2,4,7,9 | 1,2,3,8,9 | 1,3,4,6,8 | 1,2,3,8,9 |
| 6 | 1,2,4,6,7,9 | 1,2,3,4,6,7 | 1,2,3,4,6,8 | 1,2,3,4,8,9 |
| 7 | 1,2,3,4,6,7,9 | 1,2,3,4,6,7,8 | 1,2,3,4,6,7,8 | 1,2,3,4,6,7,8,9 |
| 8 | 1,2,3,4,6,7,8,9 | | 1,2,3,4,6,7,8,9 | |

^(a) In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, ¹³C₁₂-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- ¹³C₁₂-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are ≤ 25%.

TABLE 9**Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs**

| # of Chlorine Atoms | Ion Type | Theoretical Ratio | Control Limits | |
|---------------------|-----------|-------------------|----------------|-------|
| | | | Lower | Upper |
| 4 | M / M+2 | 0.77 | 0.65 | 0.89 |
| 5 | M+2 / M+4 | 1.55 | 1.32 | 1.78 |
| 6 | M+2 / M+4 | 1.24 | 1.05 | 1.43 |
| 6 ^(a) | M / M+2 | 0.51 | 0.43 | 0.59 |
| 7 ^(b) | M / M+2 | 0.44 | 0.37 | 0.51 |
| 7 | M+2 / M+4 | 1.04 | 0.88 | 1.20 |
| 8 | M+2 / M+4 | 0.89 | 0.76 | 1.02 |

^(a) Used only for ¹³C-HxCDF (IS)^(b) Used only for ¹³C-HpCDF (IS)**TABLE 10****2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans**

| Number | Compound(s) | TEF |
|--------|---------------------|-------|
| 1 | 2,3,7,8-TCDD | 1.00 |
| 2 | 1,2,3,7,8-PeCDD | 0.50 |
| 3 | 1,2,3,6,7,8-HxCDD | 0.10 |
| 4 | 1,2,3,7,8,9-HxCDD | 0.10 |
| 5 | 1,2,3,4,7,8-HxCDD | 0.10 |
| 6 | 1,2,3,4,6,7,8-HpCDD | 0.01 |
| 7 | OCDD | 0.001 |
| 8 | 2,3,6,7-TCDF | 0.1 |
| 9 | 1,2,3,7,8-PeCDF | 0.05 |
| 10 | 2,3,4,7,8PeCDF | 0.5 |
| 11 | 1,2,3,6,7,8-HxCDF | 0.1 |
| 12 | 1,2,3,7,8,9-HxCDF | 0.1 |
| 13 | 1,2,3,4,7,8-HxCDF | 0.1 |
| 14 | 2,3,4,6,7,8-HxCDF | 0.1 |
| 15 | 1,2,3,4,6,7,8-HpCDF | 0.01 |
| 16 | 1,2,3,4,7,8,9-HpCDF | 0.01 |
| 17 | OCDF | 0.001 |

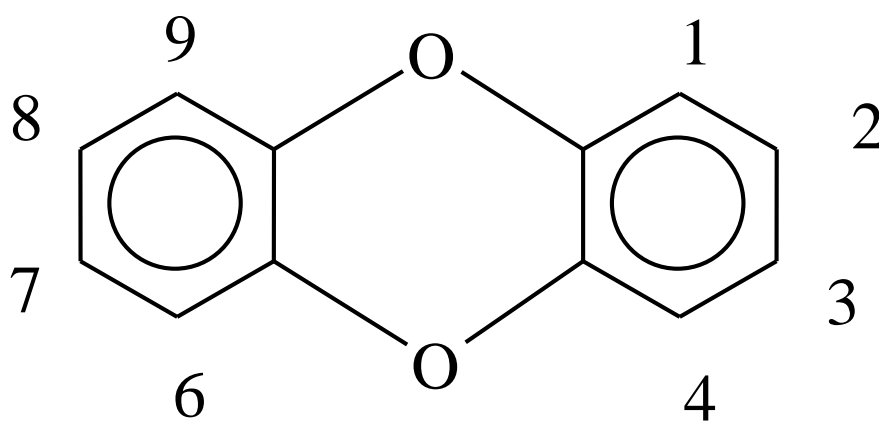
TABLE 11

**Toxicity Equivalency Factor:
 Analyte Relative Retention Time Reference Attributes**

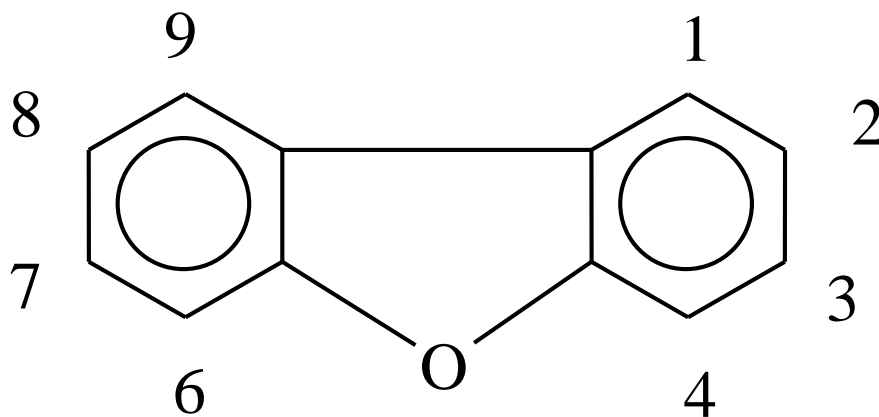
| Analyte | Analyte RRT Reference (a) |
|-------------------|--|
| 1,2,3,4,7,8-HxCDD | ¹³ C ₁₂ -1,2,3,6,7,8-HxCDD |
| 1,2,3,6,7,8-HxCDF | ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF |
| 1,2,3,7,8,9-HxCDF | ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF |
| 2,3,4,6,7,8-HxCDF | ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF |

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to ¹³C₁₂-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to ¹³C₁₂-1,2,3,4,6,7,8-HpCDF

FIGURE 1
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

FIGURE 2

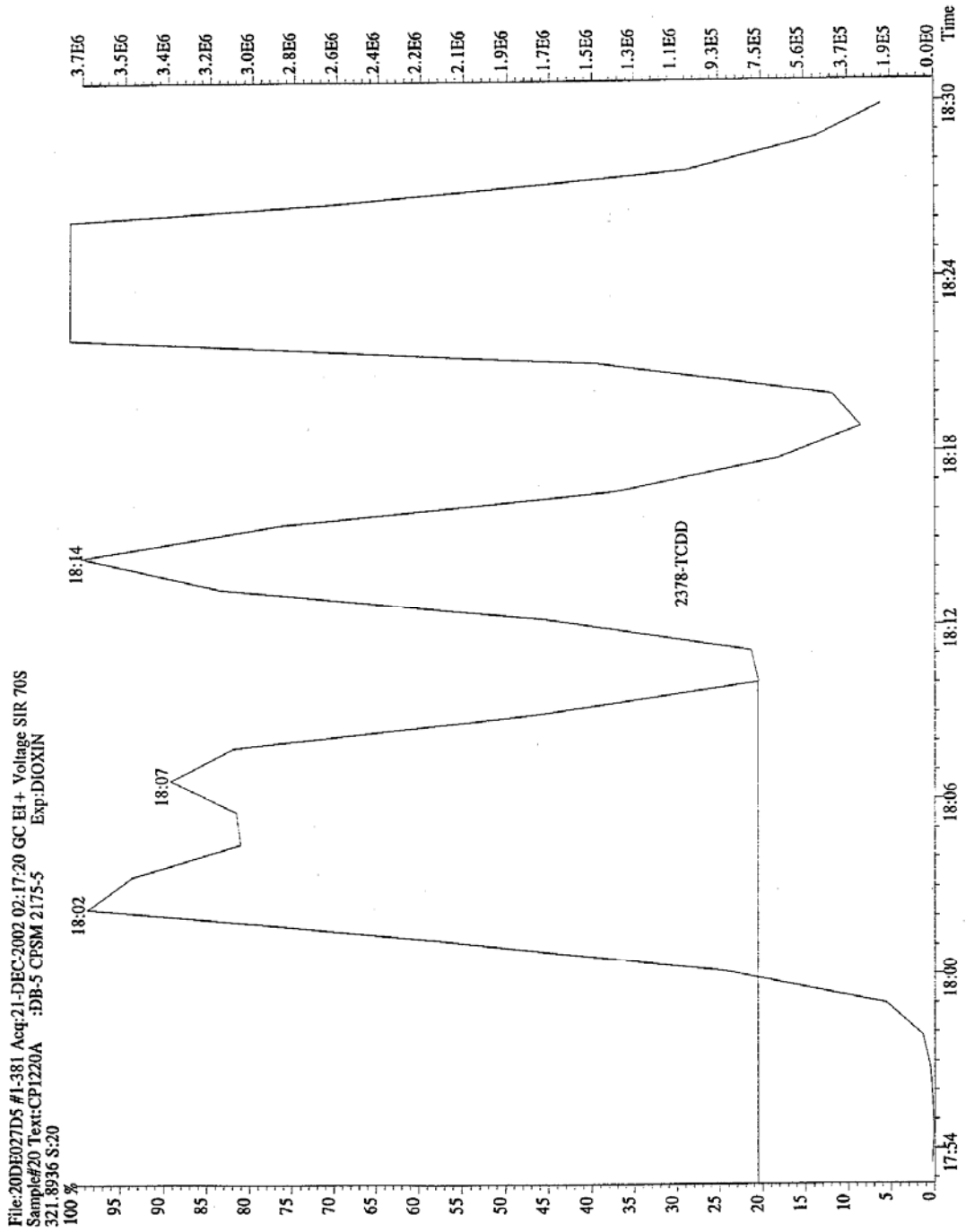


Figure 3

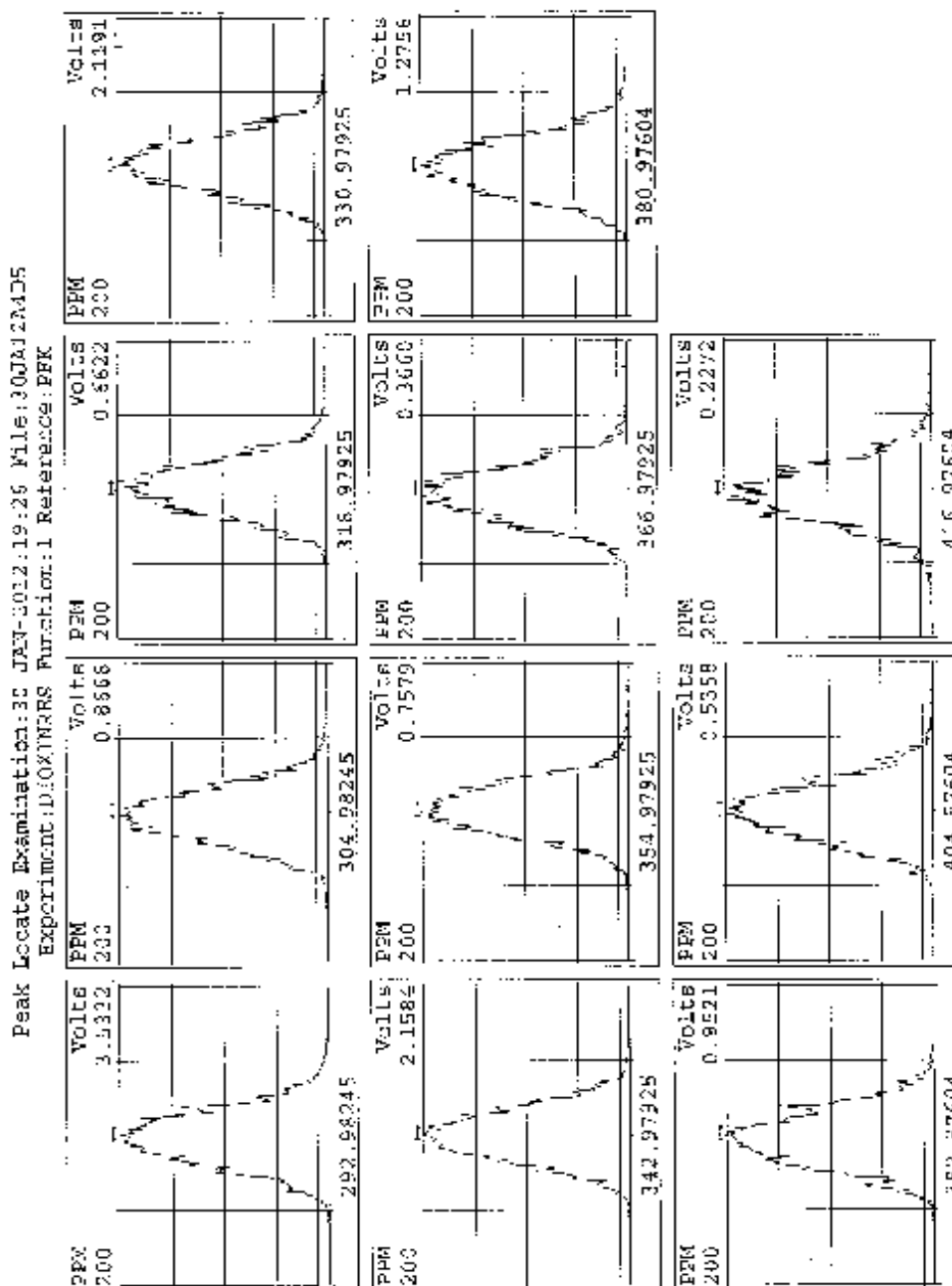
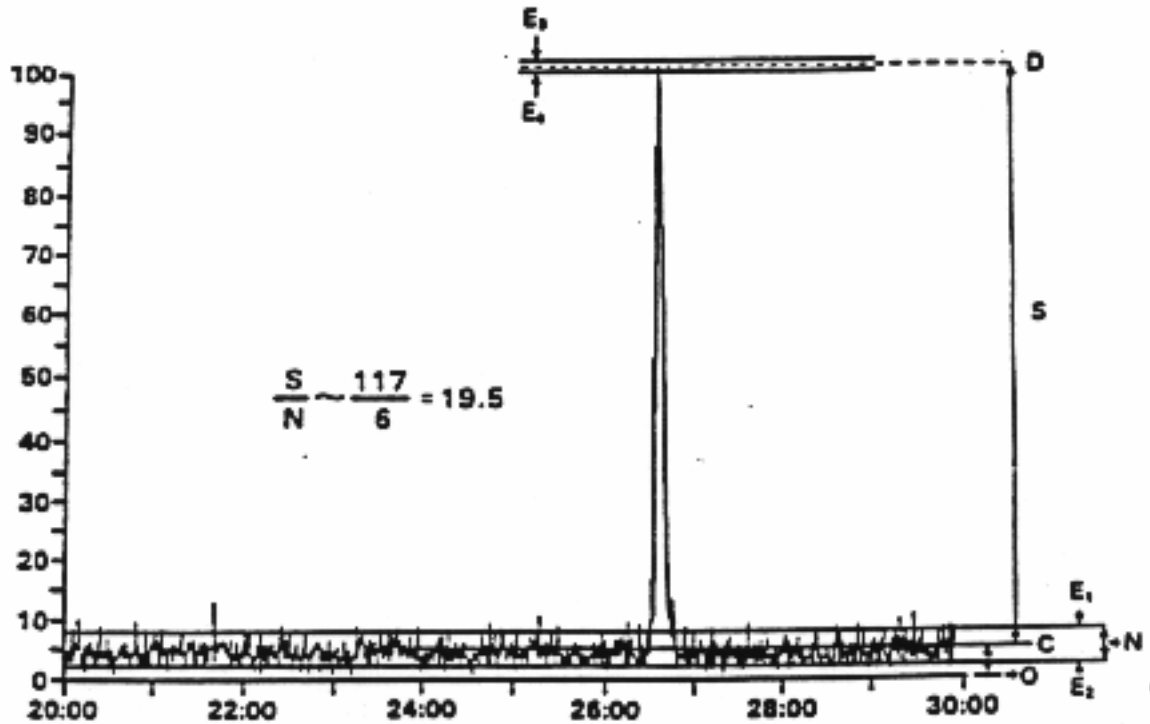


FIGURE 4



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipers and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of internal standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 μ L (either in a minivial or in a capillary tube). Inject 2 μ L of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is $25 \times 5 = 125$ pg/WTE and the positive response for the blank would be $8 \times 5 = 40$ pg). Also, report the recoveries of the isotope dilution analytes during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

WORKSHEET #15 – REFERENCE LIMITS AND EVALUATION TABLES

Matrix: Water

Analytical Method: EPA 8270C

Concentration Level (if applicable): SIM

| Analyte | Reporting Limit (µg/L) | Method Detection Limit (µg/L) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|------------------------|------------------------|-------------------------------|---------------------|------------------------|-------------------|
| 1-Methylnaphthalene | 0.200 | 0.0500 | 70-130 | 50-150 | 20 |
| 2-Methylnaphthalene | 0.200 | 0.0300 | 70-130 | 50-150 | 20 |
| Acenaphthene | 0.200 | 0.0350 | 36-98 | 50-150 | 20 |
| Acenaphthylene | 0.200 | 0.0380 | 37-97 | 50-150 | 20 |
| Anthracene | 0.200 | 0.0390 | 38-109 | 50-150 | 20 |
| Benzo(a)anthracene | 0.200 | 0.0310 | 51-111 | 50-150 | 20 |
| Benzo(a)pyrene | 0.200 | 0.0530 | 40-110 | 50-150 | 20 |
| Benzo(b)fluoranthene | 0.200 | 0.0550 | 40-122 | 50-150 | 20 |
| Benzo(g,h,i)perylene | 0.200 | 0.0400 | 37-111 | 50-150 | 20 |
| Benzo(k)fluoranthene | 0.200 | 0.0730 | 41-119 | 50-150 | 20 |
| Chrysene | 0.200 | 0.0390 | 52-107 | 50-150 | 20 |
| Dibenz(a,h)anthracene | 0.200 | 0.0460 | 36-118 | 50-150 | 20 |
| Fluoranthene | 0.200 | 0.0340 | 20-150 | 50-150 | 20 |
| Fluorene | 0.200 | 0.0320 | 21-126 | 50-150 | 20 |
| Indeno(1,2,3-cd)pyrene | 0.200 | 0.0400 | 31-130 | 50-150 | 20 |
| Naphthalene | 0.200 | 0.0680 | 34-97 | 50-150 | 20 |
| Phenanthrene | 0.200 | 0.0650 | 48-108 | 50-150 | 20 |
| Pyrene | 0.200 | 0.0370 | 43-124 | 50-150 | 20 |

Matrix: Solid

Analytical Method: EPA 8270C

Concentration Level (if applicable): SIM

| Analyte | Reporting Limit (mg/kg) | Method Detection Limit (mg/kg) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|------------------------|-------------------------|--------------------------------|---------------------|------------------------|-------------------|
| 1-Methylnaphthalene | 0.00660 | 0.00100 | 70-130 | 50-150 | 20 |
| 2-Methylnaphthalene | 0.00660 | 0.000333 | 70-130 | 50-150 | 20 |
| Acenaphthene | 0.00660 | 0.00113 | 55-92 | 48-100 | 20 |
| Acenaphthylene | 0.00660 | 0.000795 | 55-93 | 46-105 | 20 |
| Anthracene | 0.00660 | 0.000502 | 54-100 | 40-121 | 20 |
| Benzo(a)anthracene | 0.00660 | 0.000614 | 59-104 | 50-120 | 20 |
| Benzo(a)pyrene | 0.00660 | 0.000470 | 55-101 | 38-134 | 20 |
| Benzo(b)fluoranthene | 0.00660 | 0.000868 | 54-113 | 33-150 | 20 |
| Benzo(g,h,i)perylene | 0.00660 | 0.000635 | 52-95 | 20-150 | 20 |
| Benzo(k)fluoranthene | 0.00660 | 0.00113 | 50-110 | 28-147 | 20 |
| Chrysene | 0.00660 | 0.000636 | 61-99 | 51-108 | 20 |
| Dibenz(a,h)anthracene | 0.00660 | 0.00150 | 43-110 | 44-118 | 20 |
| Fluoranthene | 0.00660 | 0.000766 | 55-106 | 64-101 | 20 |
| Fluorene | 0.00660 | 0.000871 | 56-94 | 30-120 | 20 |
| Indeno(1,2,3-cd)pyrene | 0.00660 | 0.000933 | 35-121 | 47-123 | 20 |
| Naphthalene | 0.00660 | 0.00101 | 59-93 | 40-103 | 20 |
| Phenanthrene | 0.00660 | 0.000619 | 58-97 | 52-103 | 20 |
| Pyrene | 0.00660 | 0.000552 | 61-102 | 32-146 | 20 |

Matrix: Water

Analytical Method: EPA 8082A

Concentration Level (if applicable): Low

| Analyte | Reporting Limit (µg/L) | Method Detection Limit (µg/L) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|--------------|------------------------|-------------------------------|---------------------|------------------------|-------------------|
| Aroclor-1016 | 0.010 | 0.004 | 55-120 | 55-120 | 25 |
| Aroclor-1221 | 0.010 | 0.006 | | | |
| Aroclor-1232 | 0.010 | 0.006 | | | |
| Aroclor-1242 | 0.010 | 0.004 | | | |
| Aroclor-1248 | 0.010 | 0.003 | | | |
| Aroclor-1254 | 0.010 | 0.004 | | | |
| Aroclor-1260 | 0.010 | 0.003 | 55-120 | 55-120 | 25 |

Matrix: Solid

Analytical Method: EPA 8082A

Concentration Level (if applicable): Low

| Analyte | Reporting Limit (mg/kg) | Method Detection Limit (mg/kg) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|--------------|-------------------------|--------------------------------|---------------------|------------------------|-------------------|
| Aroclor-1016 | 0.00083 | 0.0004 | 50-120 | 20-120 | 30 |
| Aroclor-1221 | 0.00083 | 0.0006 | | | |
| Aroclor-1232 | 0.00083 | 0.0002 | | | |
| Aroclor-1242 | 0.00083 | 0.0003 | | | |
| Aroclor-1248 | 0.00083 | 0.0002 | | | |
| Aroclor-1254 | 0.00083 | 0.0003 | | | |
| Aroclor-1260 | 0.00083 | 0.0003 | 50-120 | 50-120 | 30 |

Matrix: Water

Analytical Group or Method: Metals/6020A

Concentration: Low

| Analyte | Reporting Limit (µg/L) | Method Detection Limit (µg/L) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|----------|------------------------|-------------------------------|---------------------|------------------------|-------------------|
| Arsenic | 10.0 | 1.18 | 80-120 | 75-125 | 20 |
| Chromium | 10.0 | 1.00 | 80-120 | 75-125 | 20 |
| Thallium | 2.00 | 0.550 | 80-120 | 75-125 | 20 |
| Lithium | 5.00 | 1.07 | 80-120 | 75-125 | 20 |

Matrix: Solid

Analytical Group or Method: Metals/6020A

Concentration: Low

| Analyte | Reporting Limit (mg/kg) | Method Detection Limit (mg/kg) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|----------|-------------------------|--------------------------------|---------------------|------------------------|-------------------|
| Arsenic | 1.00 | 0.260 | 70-131 | 75-125 | 30 |
| Lead | 0.300 | 0.100 | 73-126 | 75-125 | 30 |
| Thallium | 0.500 | 0.152 | 68-131 | 75-125 | 30 |
| Chromium | 1.00 | 0.450 | 69-130 | 75-125 | 30 |

Matrix: Water

Analytical Method: EPA 8290A

Concentration Level (if applicable): Low

| Analyte | Reporting Limit (ug/L) | Method Detection Limit (ug/L) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|---------------------|---------------------------|--|---------------------------|------------------------------|-------------------------|
| 2,3,7,8-TCDD | 0.00001 | 0.0000012 | 64-142 | 64-142 | 20 |
| 2,3,7,8-TCDF | 0.00001 | 0.000002 | 71-142 | 71-142 | 20 |
| 1,2,3,7,8-PeCDD | 0.00005 | 0.0000025 | 71-140 | 71-140 | 20 |
| 1,2,3,7,8-PeCDF | 0.00005 | 0.0000022 | 76-135 | 76-135 | 20 |
| 2,3,4,7,8-PeCDF | 0.00005 | 0.0000043 | 74-137 | 74-137 | 20 |
| 1,2,3,4,7,8-HxCDD | 0.00005 | 0.00001 | 56-146 | 56-146 | 20 |
| 1,2,3,6,7,8-HxCDD | 0.00005 | 0.0000057 | 73-144 | 73-144 | 20 |
| 1,2,3,7,8,9-HxCDD | 0.00005 | 0.0000052 | 71-151 | 71-151 | 20 |
| 1,2,3,4,7,8-HxCDF | 0.00005 | 0.0000021 | 75-131 | 75-131 | 20 |
| 1,2,3,6,7,8-HxCDF | 0.00005 | 0.0000051 | 76-133 | 76-133 | 20 |
| 1,2,3,7,8,9-HxCDF | 0.00005 | 0.0000023 | 77-142 | 77-142 | 20 |
| 2,3,4,6,7,8-HxCDF | 0.00005 | 0.0000022 | 80-137 | 80-137 | 20 |
| 1,2,3,4,6,7,8-HpCDD | 0.00005 | 0.0000094 | 78-139 | 78-139 | 20 |
| 1,2,3,4,6,7,8-HpCDF | 0.00005 | 0.0000025 | 79-133 | 79-133 | 20 |
| 1,2,3,4,7,8,9-HpCDF | 0.00005 | 0.0000038 | 83-130 | 83-130 | 20 |
| OCDD | 0.0001 | 0.000046 | 80-132 | 80-132 | 20 |
| OCDF | 0.0001 | 0.0000086 | 72-140 | 72-140 | 20 |

Matrix: Water

Analytical Group or Method: Radiochemistry

Concentration: Low

| Analyte | Reporting Limit (pCi/L) | Method Detection Limit (pCi/L) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|-------------|-------------------------|--------------------------------|---------------------|------------------------|-------------------|
| Gross Alpha | 3.0 | N/A | 73-133 | 60-140 | 40 |
| Gross Beta | 4.0 | N/A | 75-125 | 60-140 | 40 |
| Radium-226 | 1.0 | N/A | 68-137 | N/A | 40 |
| Radium-228 | 1.0 | N/A | 56-140 | N/A | 40 |
| Cesium-137 | 20.0 | N/A | 90-111 | N/A | 40 |

Matrix: Solid

Analytical Group or Method: Radiochemistry

Concentration: Low

| Analyte | Reporting Limit (pCi/g) | Method Detection Limit (pCi/g) | LCS Recovery Limits | MS/MSD Recovery Limits | MS/MSD RPD Limits |
|-------------|-------------------------|--------------------------------|---------------------|------------------------|-------------------|
| Gross Alpha | 10.0 | N/A | 44-140 | 43-123 | 40 |
| Gross Beta | 10.0 | N/A | 38-130 | 55-125 | 40 |
| Radium-226 | 1.0 | N/A | N/A | N/A | 40 |
| Radium-228 | 1.0 | N/A | N/A | N/A | 40 |
| Cesium-137 | 0.20 | N/A | 87-120 | N/A | 40 |

SAP WORKSHEET #19 CONTAINERS, VOLUME, PRESERVATION, HOLD TIME

| Matrix | Analytical Group | Analytical / Preparation Method SOP Reference ¹ | Containers (number, size, and type) | Sample volume ³ (units) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time ² (preparation / analysis) |
|--------|--------------------|--|-------------------------------------|------------------------------------|--|--|
| Soil | PAHs | SW846 3550C/8270D SIM ST-MS-0001 | 1x4oz Glass Jar | 30g | Cool ≤ 6°C | 14 days / 40 days |
| Soil | PCBs | SW846 3550C/8082A ST-GC-0015 | 1x4oz Glass Jar | 30g | Cool ≤ 6°C | 14 days / 40 days |
| Soil | Metals | SW846 3050B/6020A ST-MT-0001 | 1x2oz Glass Jar | 1g | Cool ≤ 6°C | 180 days |
| Soil | Gamma Spectroscopy | EPA 901.1 SOP ST-RD-0102 | 1x32oz Plastic or Zip Lock bag | 350g | None | None |
| Soil | Gross Alpha/Beta | SW846 9310 SOP ST-RD-0403 | 1x32oz Plastic or Zip Lock bag | 100g | None | None |
| Water | PAHs | SW846 3550C/8270D SIM ST-MS-0001 | 3x1L Amber Glass | 1L | Cool ≤ 6°C | 7 days / 40 days |
| Water | PCBs | SW846 3550C/8082A ST-GC-0015 | 3x1L Amber Glass | 1L | Cool ≤ 6°C | 7 days / 40 days |
| Water | Metals | SW846 3010A/6020A ST-MT-0001 | 1x250 mL Plastic | 50 mL | HNO ₃ to pH < 2 | 180 days |
| Water | Dioxin/Furans | SW846 8290A WS-ID-0005 | 3x1L Amber Glass | 1L | Cool ≤ 6°C | 7 days / 40 days |
| Water | Gamma Spectroscopy | EPA 901.1 SOP ST-RD-0102 | 1x1L Plastic | 1L | HNO ₃ to pH < 2 | None |
| Water | Radium-228 | EPA 904.0 SOP ST-RD-0403 | 1x1L Plastic | 500 mL | HNO ₃ to pH < 2 | None |

| Matrix | Analytical Group | Analytical / Preparation Method SOP Reference¹ | Containers (number, size, and type) | Sample volume³ (units) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time² (preparation / analysis) |
|---------------|-------------------------|--|---|---|--|---|
| Water | Radium-226 | EPA 903.0 SOP ST-RD-0403 | 1x1L Plastic | 500 mL | HNO ₃ to pH < 2 | None |
| Water | Gross Alpha/Beta | EPA 900.0 SOP ST-RD-0403 | 1x500mL Plastic | 200 mL | HNO ₃ to pH < 2 | None |

¹ Refer to the Analytical SOP References table (Worksheet #23).

² Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

³ The minimum sample size is based on analysis allowing for sufficient sample for reanalysis. Additional volume is needed for the laboratory Matrix Spike/Matrix Spike Duplicate sample analysis.

SAP WORKSHEET #23 ANALYTICAL SOP REFERENCES TABLE

| Lab SOP Number | Title, Revision Date, and/or Number ¹ | Definitive or Screening Data | Matrix and Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? (Y/N) |
|----------------|--|------------------------------|---|----------------------------------|----------------------------------|----------------------------------|
| ST-MS-0001 | GC/MS Semi-Volatile Analysis Rev.20, 11/16/15 | Definitive | Soil & Water/ PAHs | GC/MS | TestAmerica – St. Louis | N |
| ST-GC-0015 | PCB GC Analysis Rev. 18, 10/23/15 | Definitive | Soil & Water/ PCBs | GC | TestAmerica – St. Louis | N |
| ST-MT-0001 | Analysis of Metals by Inductively Coupled Plasma/ Mass Spectroscopy Rev. 24, 06/22/15 | Definitive | Soil & Water/ Metals | ICP-MS | TestAmerica – St. Louis | N |
| ST-RD-0102 | GammaVision Analysis, Rev. 13, 06/22/15 | Definitive | Soil & Water/ Gamma Spec | Gamma Spectroscopy | TestAmerica – St. Louis | N |
| ST-RD-0403 | Low Background Gas Flow Proportional Counting (GFPC) System Analysis, Rev. 16, 05/05/15 | Definitive | Soil & Water / Radium-226, Radium-228, Gross Alpha/Beta | Gas Flow Proportional Counter | TestAmerica – St. Louis | N |
| WS-ID-0005 | Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS, Rev. 7.7, 07/22/15 | Definitive | Water/ Dioxin/Furans | HRGC/HRMS | TestAmerica - Sacramento | N |

SAP WORKSHEET #24 ANALYTICAL INSTRUMENT CALIBRATION TABLE

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|--|--|---|---|---------------------------------|----------------------------|
| GC/MS | Initial Calibration (ICAL) – five-point ICAL | Initial calibration prior to sample analysis | %RSD<20% all compounds, Relative Response Factor meet method criteria | Repeat calibration | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC/MS | Second Source Calibration Verification | Once after each initial calibration | Value of second source for all analytes within ±30% of expected | Rerun ICV one time, second failure requires recalibration | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC/MS | Calibration Verification (CV) | Daily, before sample analysis, and every 12 hours of analysis time | +/- 20%D criteria for all analytes | Re-inject CV; if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC/MS | Tune Check | Prior to ICAL and prior to each 12-hour period of sample analysis | Specific ion abundance criteria of DFTPP from method | Retune instrument and verify | TestAmerica – St. Louis Analyst | ST-MS-0001 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference¹ |
|-------------------|--|---|--|---|----------------------------------|----------------------------------|
| GC/MS | Performance Check | At the beginning of each 12-hour period, prior to analysis of samples | Degradation \leq 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2 | Correct problem, then repeat performance checks | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC/MS | Retention Time window position establishment | Once per ICAL and at the beginning of the analytical sequence | Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used | N/A | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC/MS | Evaluation of Relative Retention Times (RRT) | With each sample | RRT of each reported analyte within \pm 0.06 RRT units | Correct problem, then rerun ICAL | TestAmerica – St. Louis Analyst | ST-MS-0001 |
| GC | Initial Calibration (ICAL) – five-point ICAL | Initial calibration prior to sample analysis | Mean RSD for each aroclor \leq 20% | Recalibrate | TestAmerica – St. Louis Analyst | ST-GC-0015 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|---|--|--|--|---------------------------------|----------------------------|
| GC | Second Source Calibration Verification | Once after each initial calibration | Value of second source for all analytes within $\pm 30\%$ of expected value (initial source) | Rerun ICV one time, second failure requires re-calibration | TestAmerica – St. Louis Analyst | ST-GC-0015 |
| GC | Calibration Verification (Initial [ICV] and continuing [CCV]) | ICV: Daily, before sample analysis CCV: After every 12 hours of analysis time and at the end of the analysis sequence | All analytes within $\pm 20\%$ of expected value from the ICAL | Re-inject CCV; if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate | TestAmerica – St. Louis Analyst | ST-GC-0015 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|--|---|---|---|---------------------------------|----------------------------|
| ICP-MS | Linear Dynamic Range (LDR) or high-level check standard | At initial set up and checked every 6 months high a high standard at the upper limit of the range | Within + 10% of true value | Dilute samples within the calibration range, or re-establish/verify the LDR | TestAmerica – St. Louis Analyst | ST-MT-0001 |
| ICP-MS | Tuning | Prior to ICAL | Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height | Retune instrument and verify | TestAmerica – St. Louis Analyst | ST-MT-0001 |
| ICP-MS | Initial Calibration (ICAL) – minimum one high standard and a calibration blank | Daily initial calibration prior to sample analysis | 3 standards and a blank. Correlation Coefficient of ≥ 0.998 | Recalibrate | TestAmerica – St. Louis Analyst | ST-MT-0001 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|--|--|---|--|---------------------------------|----------------------------|
| ICP-MS | Second Source Calibration Verification (ICV) | Once after each initial calibration, prior to sample analysis | Value of second source for all analyte(s) within $\pm 10\%$ of expected | Recalibrate | TestAmerica – St. Louis Analyst | ST-MT-0001 |
| ICP-MS | Continuing Calibration Verification (CCV) | After every 10 samples and at the end of the analysis sequence | All analytes within $\pm 10\%$ of expected value | Recalibrate – rerun 10 samples previous to failed CCV. | TestAmerica – St. Louis Analyst | ST-MT-0001 |
| ICP-MS | Low-level Calibration Check Standard (Low-level ICV) | Daily | All analytes within $\pm 10\%$ of expected value | Correct problem and repeat ICAL | TestAmerica – St. Louis Analyst | ST-MT-0001 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|--------------------|---|--|---|--|--------------------------------------|----------------------------|
| ICP-MS | Interference Check Solutions (ICS) | After ICAL and prior to sample analysis | <p>ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD(unless they are a verified trace impurity from one of the spike analytes)</p> <p>ICS-AB: within + 20% of true value</p> | Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples | TestAmerica – St. Louis Analyst | ST-MT-0001 |
| Gamma Spectrometer | <ol style="list-style-type: none"> 1. Energy calibration 2. FWHM calibration 3. Background | <ol style="list-style-type: none"> 1. Annual 2. Annual 3. Monthly | <p>For Energy and FWHM calibration:</p> <ul style="list-style-type: none"> • Within 0.5% or 0.1 KeV for all calibration points • Within 8% for all calibration points • Verify with second source that always contains at least Am-241, Co-60, and Cs-137 • Must be ± 10%D for each nuclide <p>For Background, acceptance criterion is 12 hours</p> | <ul style="list-style-type: none"> • Recalibrate • Instrument maintenance • Consult with Technical Director | TestAmerica – St. Louis Group Leader | ST-RD-0102 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|-------------------------------|---|--|---|--|--------------------------------------|----------------------------|
| Gas Flow Proportional Counter | <ul style="list-style-type: none"> • Plateau generation and/or verification • Discriminator setting • Initial long background count • Mass attenuated efficiency calibration • Eight source dual/single calibration curves | Annual | <ul style="list-style-type: none"> • Plot efficiencies vs masses • Calculate equation of curve – degree ≤ 3 • Remove outliers >15% deviation from theoretical values but not more than 20% of total points • Calculate coefficient of determination (R^2). R^2 must be ≥ 0.9 • Verify calibration with second source standard count – must be within 30 percent of true value and mean across all detectors <10% | <ul style="list-style-type: none"> • Recalibrate • Instrument maintenance • Consult with Technical Director | TestAmerica – St. Louis Group Leader | ST-RD-0403 |
| GC/HRMS | Tune / Mass Resolution Check (PFK) | At the beginning and the end of each 12-hour period of analysis. | Static resolving power $\geq 10,000$ (10% valley) for identified masses per method, <u>and</u> lock-mass ion between lowest and highest masses for each descriptor and level of reference compound $\leq 10\%$ full-scale deflection, per method. | Retune instrument and verify. Rerun affected samples. | Analyst, Department Manager | WS-ID-0005 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|-----------------------------|--|---|---|-----------------------------|----------------------------|
| GC/HRMS | GC Column Performance Check | Prior to ICAL and with calibration verification. | Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of \leq 25%, per method; <u>and</u> Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; <u>and</u> the difference in the absolute retention times between the last congener from one homologous series to the first congener in the next homologous series in the Window Defining Mixture must be greater than or equal to 10 seconds. | Correct problem then repeat column performance check. | Analyst, Department Manager | WS-ID-0005 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|---|--|--|------------------------------------|-----------------------------|----------------------------|
| GC/HRMS | Initial Calibration (ICAL) = Minimum five-point initial calibration for target analytes, lowest concentration standard at or below the reporting limit. | ICAL prior to sample analysis, as needed by the failure of calibration verification, and when a new lot is used as a standard source for calibration verification, internal standard or recovery standard solutions. | Ion abundance ratios in accordance with criteria in Table 8 of the method; <u>and</u> Signal/Noise ratio ≥ 10 for all target analyte ions; <u>and</u> Percent Relative Standard Deviation (RSD) $\leq 20\%$ for the response factors (RF) for all 17 unlabeled standards <u>and</u> RSD $\leq 20\%$ for the RFs for the labeled internal standards. | Correct problem, then repeat ICAL. | Analyst, Department Manager | WS-ID-0005 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person Responsible for CA | SOP Reference ¹ |
|------------|---|--|--|--|-----------------------------|----------------------------|
| GC/HRMS | Continuing Calibration Verification (CCV) | At the beginning of each 12-hour period, and at the end of each analytical sequence. | Ion abundance ratios must be in accordance with SOP; <u>and</u> RF (unlabeled standards) within \pm 20% Difference (D) of average RF from ICAL; <u>and</u> RF (labeled standards) within \pm 30%D of average RF from ICAL. | Correct problem, repeat calibration verification standard. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV. <u>End-of-run CV</u> : If the RF for unlabeled standards \leq 25% D and the RF for labeled standards \leq 35% D (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending CCV RFs differ by more than 25% D for unlabeled compounds or 35% D for labeled compounds, reanalyze samples with positive detections if necessary. | Analyst, Department Manager | WS-ID-0005 |

SAP WORKSHEET #25 ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

| Instrument/Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|-----------------------------|-----------------------------|-------------------------|----------------------------------|-------------------------|--|---|---------------------------|--|
| GCMS GC ICP-MS | Parameter Setup | Physical check | Physical check | Initially; prior to DCC | Predetermined optimum parameter settings | Reset if incorrect | Analyst | ST-MS-0001 ST-GC-0015 ST-MT-0001 |
| GC/MS | Tune Check | Instrument Performance | Conformance to instrument tuning | Initially; prior to DCC | Compliance to ion abundance criteria | Repeat tune check to rule out standard degradation or inaccurate injection. If problem persists, perform retune the instrument and repeat tune check. | Analyst | ST-MS-0001 |

| Instrument/Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|----------------------|--|---|---|--------------------------------------|--|--|--------------------|---------------|
| ICP-MS | ICS | Instrument Performance | Conformance to interference check | Prior to sample analysis | Within + 20% of expected value | Terminate analysis; reanalyze ICS to rule out standard degradation or inaccurate injection. If problem persists, perform instrument maintenance, repeat calibrations and reanalyze all associated samples. | Analyst | ST-MT-0001 |
| ICP-MS | ICB/CCB | Instrument Performance | Instrument contamination check | After every calibration verification | ICB: No analytes detected > RL; CCB: no analyte detected > 3X MDL | Determine possible source of contamination and apply appropriate measure to correct the problem. Reanalyze calibration blank and all associated samples. | Analyst | ST-MT-0001 |
| Gamma Spectrometer | 1. Clean cave; fill dewar with N ₂ 2. QA check | 1. Physical check 2. Background and source check | 1. Physical check 2. Check deviation | 1. Weekly 2. Daily | 1. Acceptable background 2. Within 3 sigma of measured population | <ul style="list-style-type: none"> · Recalibrate · Instrument maintenance · Consult with Technical Director | Analyst | ST-RD-0102 |

| Instrument/Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|-------------------------------|--|--|--|--|--|--|--------------------|---------------|
| Alpha Spectrometer | Clean planchette holders | Physical check | Physical check | Monthly | Acceptable background and calibration efficiencies | <ul style="list-style-type: none"> • Recalibrate • Instrument maintenance • Consult with Technical Director | Analyst | ST-RC-0210 |
| Gas Flow Proportional Counter | <ol style="list-style-type: none"> 1. Clean instrument 2. Inspect windows 3. QA check | <ol style="list-style-type: none"> 1. Physical check 2. Physical check 3. Background and source count | <ol style="list-style-type: none"> 1. Physical check 2. Physical check 3. Check deviation | <ol style="list-style-type: none"> 1. Daily 2. High counts and/or background 3. Daily | <ol style="list-style-type: none"> 1. None applicable 2. No physical defects 3. Within 3 sigma of 20 day population | <ul style="list-style-type: none"> • Recalibrate • Instrument maintenance • Consult with Technical Director | Analyst | ST-RD-0403 |
| HRGC/HRMS | Injection port maintenance, clean ion volume, clean source, replace filament | Instrument Performance | Conformance to interference check | As needed | Refer to Worksheet #24 | Refer to Worksheet #24 | Analyst | WS-ID-0005 |
| HRGC/HRMS | Tune instrument to maximize sensitivity and mass resolution | Instrument Performance | Conformance to interference check | Daily | Refer to Worksheet #24 | Refer to Worksheet #24 | Analyst | WS-ID-0005 |
| HRGC/HRMS | Change mechanical pump fluid | Instrument Performance | Conformance to interference check | Yearly | Refer to Worksheet #24 | Refer to Worksheet #24 | Analyst | WS-ID-0005 |

APPENDIX C
SAMPLE FIELD FORMS



ERM, Inc.
 8425 Woodfield Crossing Blvd, Suite 560-W
 Indianapolis, Indiana 46240
 (317) 706-2000

Soil Boring Log

Boring No: _____
 Page 1 of _____

Date: _____ Proj. No.: _____ Project: _____
 Client: _____ Location: _____
 Drilling Company: _____ Driller: _____
 Logged By: _____ Drilling Method: _____
 Surface Elevation: _____ Top of Casing Elevation: _____
 Total Depth: _____ Diameter: _____ Sampling Method: _____
 Comments: _____

| Depth (ft.) | PID Reading | Sample Interval | Recovery (%) | Description/Soil Classification (Color, Texture, Structures) |
|-------------|-------------|-----------------|--------------|--|
| 0.0 | | | | |
| 1.0 | | | | |
| 2.0 | | | | |
| 3.0 | | | | |
| 4.0 | | | | |
| 5.0 | | | | |
| 6.0 | | | | |
| 7.0 | | | | |
| 8.0 | | | | |
| 9.0 | | | | |
| 10.0 | | | | |
| 11.0 | | | | |
| 12.0 | | | | |
| 13.0 | | | | |
| 14.0 | | | | |
| 15.0 | | | | |
| 16.0 | | | | |
| 17.0 | | | | |
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| 22.0 | | | | |
| 23.0 | | | | |
| 24.0 | | | | |
| 25.0 | | | | |
| 26.0 | | | | |
| 27.0 | | | | |
| 28.0 | | | | |



ERM, Inc.
 11350 N. Meridian Street, Suite 320
 Carmel, Indiana 46032
 (317) 706-2000

Soil Boring Log

Boring No: _____
 Page 2 of _____

Date: _____ Proj. No.: _____ Project: _____
 Client: _____ Location: _____
 Drilling Company: _____ Driller: _____
 Logged By: _____ Drilling Method: _____
 Surface Elevation: _____ Top of Casing Elevation: _____
 Total Depth: _____ Diameter: _____ Sampling Method: _____
 Comments: _____

| Depth (ft.) | PID Reading | Blow Counts | Recovery (%) | Description/Soil Classification (Color, Texture, Structures) |
|-------------|-------------|-------------|--------------|--|
| 29.0 | | | | |
| 30.0 | | | | |
| 31.0 | | | | |
| 32.0 | | | | |
| 33.0 | | | | |
| 34.0 | | | | |
| 35.0 | | | | |
| 36.0 | | | | |
| 37.0 | | | | |
| 38.0 | | | | |
| 39.0 | | | | |
| 40.0 | | | | |
| 41.0 | | | | |
| 42.0 | | | | |
| 43.0 | | | | |
| 44.0 | | | | |
| 45.0 | | | | |
| 46.0 | | | | |
| 47.0 | | | | |
| 48.0 | | | | |
| 49.0 | | | | |
| 50.0 | | | | |
| 51.0 | | | | |
| 52.0 | | | | |
| 53.0 | | | | |
| 54.0 | | | | |
| 55.0 | | | | |
| 56.0 | | | | |
| 57.0 | | | | |





ERM, Inc.
 11350 N. Meridian Street, Suite 320
 Carmel, Indiana 46032
 (317) 706-2000

Soil Boring Log

Boring No:
 Page 3 of

Date: _____ Proj. No.: _____ Project: BMS Mt. Vernon
 Client: BMS Location: Mt. Vernon, IN
 Drilling Company: _____ Driller: _____
 Logged By: C. Burrows, D. Keagy (LPG# _____) Drilling Method: _____
 Surface Elevation: _____ Top of Casing Elevation: _____
 Total Depth: _____ Diameter: _____ Sampling Method: _____
 Comments: _____

| Depth (ft.) | PID Reading | Blow Counts | Recovery (%) | Description/Soil Classification (Color, Texture, Structures) |
|-------------|-------------|-------------|--------------|--|
| 58.0 | | | | |
| 59.0 | | | | |
| 60.0 | | | | |
| 61.0 | | | | |
| 62.0 | | | | |
| 63.0 | | | | |
| 64.0 | | | | |
| 65.0 | | | | |
| 66.0 | | | | |
| 67.0 | | | | |
| 68.0 | | | | |
| 69.0 | | | | |
| 70.0 | | | | |
| 71.0 | | | | |
| 72.0 | | | | |
| 73.0 | | | | |
| 74.0 | | | | |
| 75.0 | | | | |
| 76.0 | | | | |
| 77.0 | | | | |
| 78.0 | | | | |
| 79.0 | | | | |
| 80.0 | | | | |
| 81.0 | | | | |
| 82.0 | | | | |
| 83.0 | | | | |
| 84.0 | | | | |
| 85.0 | | | | |
| 86.0 | | | | |



INSTRUCTIONS



- 1) Choose the correct TestAmerica Facility from the pull down list by clicking on cell A1
- 2) Fill in the appropriate information for your location and phone number
- 3) Sampler - Fill in name.
- 4) Provide information on the Regulatory Program to differentiate between Drinking Water & Compliance samples.
- 5) Choose a default TAT or enter a different one if appropriate
- 6) Please indicate whether the TAT is Working or Calendar Days
- 7) In the vertical columns enter the Method/Analysis being requested
- 8) Fill out the Sample Information -- each line represents one sample
- 9) Sample Date/Time is required on all samples
- 10) In the "# of Containers" field enter the total number of bottles for each sample
- 11) Check Y or N if the sample was filtered in the field (Filtered Sample).
- 12) Note 'C' for a Composite sample; or 'G' for a Grab Sample.
- 13) The Sample name should be the one you wish to see in the final report
- 14) In the cell where the Sample Information intersects the method information please enter the number of containers submitted for the method. Alternatively simply "x" this field
- 15) In the last row of the eCOC please choose the code for the right preservative used
- 16) Note any Possible Hazards.
- 17) Use the Special Instructions field to add any special instructions to the lab
- 18) If samples are sent across the country, consider indicating the Time Zone where samples were collected
- 19) TestAmerica Terms and Conditions apply for the analysis performed on the submitted samples unless otherwise agreed upon between TestAmerica and Company

Where a purchaser (Client) places an order for laboratory, consulting or sampling services from TestAmerica Laboratories, Inc., a Delaware corporation (referred to as "TestAmerica"), TestAmerica shall provide the ordered services pursuant to these Terms and Conditions, and the related Quotation or Price Schedule, or as agreed in a negotiated contract. In the absence of a written agreement to the contrary, the Order constitutes an acceptance by the Client of TestAmerica's offer to do business under these Terms and Conditions, and an agreement to be bound by these Terms and Conditions. No contrary or additional terms and conditions expressed in a Client's document shall be deemed to become a part of the contract created upon acceptance of these Terms and Conditions, unless accepted by TestAmerica in writing.

1. ORDERS AND RECEIPT OF SAMPLES

1.1 The Client may place the Order (i.e., specify a Scope of Work) either by submitting a purchase order to TestAmerica in writing or by telephone subsequently confirmed in writing, or by negotiated contract. Whichever option the Client selects for placing the Order, the Order shall not be valid unless it contains sufficient specification to enable TestAmerica to carry out the Client's requirements. In particular, samples must be accompanied by: a) adequate instruction on type of analysis requested, and b) complete written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law. Where any samples which were not accompanied by the required disclosure, cause interruptions in the lab's ability to process work due to contamination of instruments or work areas, the Client will be responsible for the costs of clean up and recovery.

1.2 The Client shall provide one week's advance notice of the sample delivery schedule, or any changes to the schedule, whenever possible. Upon timely delivery of samples, TestAmerica will use its best efforts to meet mutually agreed turnaround times. All turnaround times will be calculated from the point in time when TestAmerica has determined that it can proceed with defined work following receipt, inspection of samples, and resolution of any discrepancies in Chain-of-Custody forms and project guidance regarding work to be done (Sample Delivery Acceptance). In the event of any changes in the sample delivery schedule by the Client, prior to Sample Delivery Acceptance, TestAmerica reserves the right to modify its turnaround time commitment, to change the date upon which TestAmerica will accept samples, or refuse Sample Delivery Acceptance for the affected samples.

1.3 TestAmerica reserves the right, exercisable at any time, to refuse or revoke Sample Delivery Acceptance for any sample which in the sole judgment of TestAmerica: a) is of unsuitable volume; b) may pose a risk or become unsuitable for handling, transport, or processing for any health, safety, environmental or other reason, whether or not due to the presence in the sample of any hazardous substance and whether or not such presence has been disclosed to TestAmerica by the Client; or c) holding times cannot be met, due to passage of more than 48 hours from the time of sampling or 1/2 the holding time for the requested test, whichever is less.

1.4 Prior to Sample Delivery Acceptance, the entire risk of loss or damage to samples remains with the Client, except where TestAmerica provides courier services. In no event will TestAmerica have any responsibility or liability for the action or inaction of any carrier shipping or delivering any sample to or from TestAmerica's premises. Client is responsible to assure that any sample containing any hazardous substance which is to be delivered to TestAmerica's premises will be packaged, labeled, transported and delivered properly and in accordance with applicable laws.

2. PAYMENT TERMS

2.1 Services performed by TestAmerica will be in accordance with prices quoted and later confirmed in writing or as stated in the Price Schedule. Quoted prices do not include sales tax. Applicable sales tax will be added to invoices where required by law. Where requested services on a group of samples received and logged in together at the laboratory total less than \$200, there will be a minimum transaction charge of \$200 for the sample group, or as shown on any related quote from TestAmerica. An Environmental Management Fee of 5% of the invoice value will also be applied, at TestAmerica's discretion.

2.2 Invoices may be submitted to Client upon completion of any sample delivery group. Billing corrections must be requested within 30 days of invoice date. Payment in advance is required for all Clients except those whose credit has been established with TestAmerica. For Clients with approved credit, payment terms are net 30 days from the date of invoice by TestAmerica. All overdue payments are subject to an additional interest and service charge of one and one half percent (1.5%) (or the maximum rate permissible by law, whichever is lesser) per month or portion thereof from the due date until the date of payment. All fees are charged or billed directly to the Client. The billing of a third party will not be accepted without a statement, signed by the third party, that acknowledges and accepts payment responsibility.

2.3 TestAmerica may suspend work and withhold delivery of data under this order at any time in the event Client fails to make timely payment of its invoices. Client shall be responsible for all costs and expenses of collection including reasonable attorney's fees. TestAmerica reserves the right to refuse to proceed with work at any time based upon an unfavorable Client credit report.

3. CHANGE ORDERS, TERMINATION

3.1 Changes to the Scope of Work, price, or result delivery date may be initiated by TestAmerica after Sample Delivery Acceptance due to any condition which conflicts with analytical, QA or other protocols warranted in these Terms and Conditions. TestAmerica will not proceed with such changes until an agreement with the Client is reached on the amount of any cost, schedule change or technical change to the Scope of Work, and such agreement is documented in writing.

3.2 Changes to the Scope of Work, including but not limited to increasing or decreasing the work, changing test and analysis specification, or acceleration in the performance of the work may be initiated by the Client after sample delivery acceptance. Such a change will be documented in writing and may result in a change in cost and turnaround time commitment. TestAmerica's acceptance of such changes is contingent upon technical feasibility and operational capacity.

3.3 Suspension or termination of all or any part of the work may be initiated by the Client. TestAmerica will be compensated consistent with Section 2 of these Terms and Conditions. TestAmerica will complete all work in progress and be paid in full for all work completed.

4. WARRANTIES AND LIABILITY

4.1 Where applicable, TestAmerica will use analytical methodologies which are in substantial conformity with published test methods. TestAmerica has implemented these methods in its Laboratory Quality Manuals and referenced Standard Operating Procedures and where the nature or composition of the sample requires it, TestAmerica reserves the right to deviate from these methodologies as necessary or appropriate, based on the reasonable judgment of TestAmerica, which deviations, if any, will be made on a basis consistent with recognized standards of the industry and/or TestAmerica's Laboratory Quality Manuals. Client may request that TestAmerica perform according to a mutually agreed Quality Assurance Project Plan (QAPP). In the event that samples arrive prior to agreement on a QAPP, TestAmerica will proceed with analyses under its standard Quality Manuals then in effect, and TestAmerica will not be responsible for any resampling or other charges if work must be repeated to comply with a subsequently finalized QAPP.

4.2 TestAmerica shall start preparation and/or analysis within holding times provided that Sample Delivery Acceptance occurs within 48 hours of sampling or 1/2 of the holding time for the test, whichever is less. Where resolution of inconsistencies leading to Sample Delivery Acceptance does not occur within this period, TestAmerica will use its best efforts to meet holding times and will proceed with the work provided that, in TestAmerica's judgment, the chain-of-custody or definition of the Scope of Work provide sufficient guidance. Reanalysis of samples to comply with TestAmerica's Quality Manuals will be deemed to have met holding times provided the initial analysis was performed within the applicable holding time. Where reanalysis demonstrates that sample matrix interference is the cause of failure to meet any Quality Manual requirements, the warranty will be deemed to have been met.

4.3 TestAmerica warrants that it possesses and maintains all licenses and certifications which are required to perform services under these Terms and Conditions provided that such requirements are specified in writing to TestAmerica prior to Sample Delivery Acceptance. TestAmerica will notify the Client in writing of any decertification or revocation of any license, or notice of either, which affects work in progress.

4.4 The warranty obligations set forth in Sections 4.1, 4.2 and 4.3 are the sole and exclusive warranties given by TestAmerica in connection with any services performed by TestAmerica or any Results generated from such services, and TestAmerica gives and makes NO OTHER REPRESENTATION OR WARRANTY OF ANY KIND, EXPRESS OR IMPLIED. No representative of TestAmerica is authorized to give or make any other representation or warranty or modify this warranty in any way.

4.5 Client's sole and exclusive remedy for the breach of warranty in connection with any services performed by TestAmerica, will be limited to repeating any services performed, contingent on the Client's providing, at the request of TestAmerica and at the Client's expense, additional sample(s) if necessary. Any reanalysis requested by the Client generating Results consistent with the original Results will be at the Client's expense. If resampling is necessary, TestAmerica's liability for resampling costs will be limited to actual cost or one hundred and fifty dollars (\$150) per sample, whichever is less.

4.6 TestAmerica's liability for any and all causes of action arising hereunder, whether based in contract, tort, warranty, negligence or otherwise, shall be limited to the lesser amount of compensation for the services performed or \$100,000. All claims, including those for negligence, shall be deemed waived unless suit thereon is filed within one year after TestAmerica's completion of the services. Under no circumstances, whether arising in contract, tort (including negligence), or otherwise, shall TestAmerica be responsible for loss of use, loss of profits, or for any special, indirect, incidental or consequential damages occasioned by the services performed or by application or use of the reports prepared.

4.7 In no event shall TestAmerica have any responsibility or liability to the Client for any failure or delay in performance by TestAmerica which results, directly or indirectly, in whole or in part, from any cause or circumstance beyond the reasonable control of TestAmerica. Such causes and circumstances

directly or indirectly, in whole or in part, from any cause or circumstance beyond the reasonable control of TestAmerica. Such causes and circumstances shall include, but not be limited to, acts of God, acts of Client, acts or orders of any governmental authority, strikes or other labor disputes, natural disasters, accidents, wars, civil disturbances, equipment breakdown, matrix interference or unknown highly contaminated samples that impact instrument operation, unavailability of supplies from usual suppliers, difficulties or delays in transportation, mail or delivery services, or any other cause beyond TestAmerica's reasonable control.

5. RESULTS, WORK PRODUCT

5.1 Data or information provided to TestAmerica or generated by services performed under this agreement shall only become the property of the Client upon receipt in full by TestAmerica of payment for the whole Order. Ownership of any analytical method, QA/QC protocols, software programs or equipment developed by TestAmerica for performance of work will be retained by TestAmerica, and Client shall not disclose such information to any third party.

5.2 Data and sample materials provided by Client or at Client's request, and the result obtained by TestAmerica shall be held in confidence (unless such information is generally available to the public or is in the public domain or Client has failed to pay TestAmerica for all services rendered or is otherwise in breach of these Terms and Conditions), subject to any disclosure required by law or legal process.

5.3 Should the Results delivered by TestAmerica be used by the Client or Client's client, even though subsequently determined not to meet the warranties described in these Terms and Conditions, then the compensation will be adjusted based upon mutual agreement. In no case shall the Client unreasonably withhold TestAmerica's right to independently defend its data.

5.4 TestAmerica reserves the right to perform the services at any laboratory in the TestAmerica network, unless the Client has specified a particular location for the work. In addition, TestAmerica reserves the right to subcontract services ordered by the Client to another laboratory or laboratories, if, in TestAmerica's sole judgment, it is reasonably necessary, appropriate or advisable to do so. TestAmerica will in no way be liable for any subcontracted services (outside the TestAmerica network) except for work performed at laboratories which have been audited and approved by TestAmerica.

5.5 TestAmerica shall dispose of the Client's samples 30 days after the analytical report is issued, unless instructed to store them for an alternate period of time or to return such samples to the Client, in a manner consistent with U.S. Environmental Protection Agency regulations or other applicable federal, state or local requirements. Any samples for projects that are canceled or not accepted, or for which return was requested, will be returned to the Client at his own expense. TestAmerica reserves the right to return to the Client any sample or unused portion of a sample that is not within TestAmerica's permitted capability or the capabilities of TestAmerica's designated waste disposal vendor(s). ALL DIOXIN, MIXED WASTE, AND RADIOACTIVE SAMPLES WILL BE RETURNED TO THE CLIENT, unless prior arrangements for disposal are made.

5.6 Unless a different time period is agreed to in any order under these Terms and Conditions, TestAmerica agrees to retain all records for five (5) years.

5.7 In the event that TestAmerica is required to respond to legal process related to services for Client, Client agrees to reimburse TestAmerica for hourly charges for personnel involved in the response and attorney fees reasonably incurred in obtaining advice concerning the response, preparation to testify, and appearances related to the legal process, travel and all reasonable expenses associated with the litigation.

6. INSURANCE

6.1 TestAmerica shall maintain in force during the performance of services under these Terms and Conditions, Workers' Compensation and Employer's Liability Insurance in accordance with the laws of the states having jurisdiction over TestAmerica's employees who are engaged in the performance of the work. TestAmerica shall also maintain during such period, Comprehensive General and Contractual Liability (limit of \$1,000,000 per occurrence/\$2,000,000 aggregate), Comprehensive Automobile Liability, owned and hired, (\$1,000,000 combined single limit), and Professional/Pollution Liability Insurance (limit of \$5,000,000 per occurrence/aggregate).

7. AUDIT

7.1 Upon prior notice to TestAmerica, the Client may audit and inspect TestAmerica's records and accounts covering reimbursable costs related to work done for the Client, for a period of two (2) years after completion of the work. The purpose of any such audit shall be only for verification of such costs, and TestAmerica shall not be required to provide access to cost records where prices are expressed as fixed fees or published unit prices.

8. MISCELLANEOUS PROVISIONS

8.1 These Terms and Conditions, together with any additions or revisions which may be agreed to in writing by TestAmerica, embody the whole agreement of the parties and provide the only remedies available. There are no promises, terms, conditions, understandings, obligations or agreements other than those contained herein, and these Terms and Conditions shall supersede all previous communications, representations, or agreements, either verbal or written, between the Client and TestAmerica. These Terms and Conditions, and any transactions or agreements to which they apply, shall be governed both as to interpretation and performance by the laws of the state where TestAmerica's services are performed.

8.2 The invalidity or unenforceability, in whole or in part of any provision, term or condition hereof shall not affect in any way the validity or enforceability of the remainder to these Terms and Conditions, the intent of the parties being that the provisions be severable. The section headings of these Terms and Conditions are intended solely for convenient reference and shall not define, limit or affect in any way these Terms and Conditions or their interpretations. No waiver by either party of any provision, term or condition hereof or of any obligation of the other party hereunder shall constitute a waiver of any subsequent breach or other obligation.

8.3 The obligations, liabilities, and remedies of the parties, as provided herein, are exclusive and in lieu of any others available at law or in equity. Indemnifications, releases from liability and limitations of liability shall apply, notwithstanding the fault, negligence or strict liability of the party to be indemnified, released, or whose liability is limited, except to the extent of sole negligence or willful misconduct.

| <u>Name</u> | <u>Address 1</u> | <u>Address 2</u> | <u>City</u> | <u>State</u> | <u>Zip</u> | <u>Phone</u> | <u>Fax</u> |
|---------------------------------|-----------------------------------|---------------------|-----------------------------|--------------|------------|--------------|--------------|
| CHOOSE A LOCATION | USE DROP DOWN MENU | TO PICK YOUR | LABORATORY / Service Center | | | | |
| TestAmerica Albany | 25 Kraft Ave. | | Albany | NY | 12205-5464 | 518.438.8140 | 518.438.8150 |
| TestAmerica Anchorage | 2000 W International Airport Road | Suite A10 | Anchorage | AK | 99502-1117 | 907.563.9200 | 907.563.9210 |
| TestAmerica Atlanta | 6500 McDonough Dr | Suite C-10 | Norcross | GA | 30093-1233 | 678.966.9991 | |
| TestAmerica Baltimore | 7526 Connelley Drive | Suite F | Hanover | MD | 21076-1670 | 410.766.2516 | 410.766.2368 |
| TestAmerica Baton Rouge | 6113 Benefit Dr | | Baton Rouge | LA | 70809-4247 | 225.755.8200 | 225.755.3080 |
| TestAmerica Beaumont | 6310 Rothway Street | | Houston | TX | 77040-5062 | 713.690.4444 | |
| TestAmerica Boston | 240 Bear Hill Road | Suite 104 | Waltham | MA | 02451-1039 | 781.466.6900 | 781.466.6901 |
| TestAmerica Buffalo | 10 Hazelwood Drive | | Amherst | NY | 14228-2223 | 716.691.2600 | 716.691.7991 |
| TestAmerica Burlington | 30 Community Drive | Suite 11 | South Burlington | VT | 05403-6809 | 802.660.1990 | 802.660.1919 |
| TestAmerica Cambridge | 1340 Oxford Ave. | | Cambridge | OH | 43725-3012 | 740.630.0016 | |
| TestAmerica Canton | 4101 Shuffel Street NW | | North Canton | OH | 44720-6900 | 330.497.9396 | 330.497.0772 |
| TestAmerica Cedar Falls | 704 Enterprise Drive | | Cedar Falls | IA | 50613-6907 | 319.277.2401 | 319.277.2425 |
| TestAmerica Charleston | 1436-A North Point Ln | | Mt. Pleasant | SC | 29464-4615 | 843.849.6550 | |
| TestAmerica Charlotte | 2838-B Queen City Drive | | Charlotte | NC | 28208-2738 | 704.392.1164 | |
| TestAmerica Chicago | 2417 Bond Street | | University Park | IL | 60484-3101 | 708.534.5200 | 708.534.5211 |
| TestAmerica Cincinnati | 11416 Reading Rd | | Cincinnati | OH | 45241-2247 | 513.733.5700 | |
| TestAmerica Columbus | 961 Checkrein Avenue | | Columbus | OH | 43229-1106 | 614.310.4818 | |
| TestAmerica Corpus Christi | 1733 N. Padre Island Drive | | Corpus Christi | TX | 78408-2329 | 361.289.2673 | 361.289.2471 |
| TestAmerica Dallas / Fort Worth | 3226 Commander Drive | | Carrollton | TX | 75006-2507 | 214.218.1894 | |
| TestAmerica Davenport | 321 8th Street | Unit B | Bettendorf | IA | 52722-4711 | 563.323.7944 | |
| TestAmerica Dayton | 4738 Gateway Circle | | Dayton | OH | 45440-1724 | 937.294.6856 | |
| TestAmerica Denver | 4955 Yarrow Street | | Arvada | CO | 80002-4517 | 303.736.0100 | 303.431.7171 |
| TestAmerica Des Moines | 2175 NW 86th Street | Suite 3 | Clive | IA | 50325-5500 | 515.619.5100 | 515.619.5101 |
| TestAmerica Edison | 777 New Durham Road | | Edison | NJ | 08817-2859 | 732.549.3900 | 732.549.3679 |
| TestAmerica Ft. Lauderdale | 6301 NW 5th Way | Suite 1410A | Ft. Lauderdale | FL | 33309-6131 | 954.809.5580 | 954.776.8485 |
| TestAmerica Honolulu | 4429 Malaai Street | Suite 104 | Honolulu | HI | 96818-3158 | 808.486.5227 | 808.486.2456 |
| TestAmerica Houston | 6310 Rothway Street | | Houston | TX | 77040-5062 | 713.690.4444 | 713.690.5646 |
| TestAmerica Irvine | 17461 Derian Avenue | Suite 100 | Irvine | CA | 92614-5843 | 949.261.1022 | 949.260.3299 |
| TestAmerica Jacksonville | 8933 Western Way | Suite 1 | Jacksonville | FL | 32256-0372 | 904.728.8547 | |
| TestAmerica King Of Prussia | 1008 W. Ninth Avenue | | King of Prussia | PA | 19406-1216 | 610.337.9992 | 610.337.9939 |
| TestAmerica Knoxville | 5815 Middlebrook Pike | | Knoxville | TN | 37921-5947 | 865.291.3000 | 865.584.4315 |
| TestAmerica Las Vegas | 6100 Mountain Vista | #160 | Henderson | NV | 89014-2040 | 702.429.1264 | |
| TestAmerica Michigan | 10448 Citation Drive | Suite 200 | Brighton | MI | 48116-6561 | 810.229.2763 | |
| TestAmerica Minneapolis | 7600 West 27th St | Unit 236 | St. Louis Park | MN | 55426-3100 | 952.922.2777 | |
| TestAmerica Mobile | 826 Lakeside Drive | Suite D | Mobile | AL | 36693-5118 | 251.666.6633 | 251.666.6696 |
| TestAmerica Nashville | 2960 Foster Creighton Drive | | Nashville | TN | 37204-3719 | 615.726.0177 | 615.726.3404 |
| TestAmerica New York City | 47-32 32nd Place | Suite 1141 | Long Island City | NY | 11101-2425 | 347.507.0579 | |
| TestAmerica North Seattle | 19515 North Creek Pkwy N | Suite 100 | Bothell | WA | 98011-8200 | 253.922.2310 | |
| TestAmerica Northwest Chicago | 453 N. York Street | | Elmhurst | IL | 60126-2003 | 630.758.0262 | |
| TestAmerica Northwest Indiana | 1581 East 93rd Avenue | | Merrillville | IN | 46410-6483 | 219.252.7570 | |
| TestAmerica Orlando | 6220 Hazeltine National Drive | Suite 114 | Orlando | FL | 32822-5145 | 407.851.2560 | 407.856.0886 |
| TestAmerica Pensacola | 3355 McLemore Drive | | Pensacola | FL | 32514-7045 | 850.474.1001 | 850.474.4789 |
| TestAmerica Phoenix | 4625 East Cotton Center Boulevard | Suite 189 | Phoenix | AZ | 85040-4807 | 602.437.3340 | 602.454.9303 |
| TestAmerica Pittsburgh | 301 Alpha Drive | RIDC Park | Pittsburgh | PA | 15238-2907 | 412.963.7058 | 412.963.2468 |
| TestAmerica Pleasanton | 1220 Quarry Lane | | Pleasanton | CA | 94566-4756 | 925.484.1919 | 925.600.3002 |
| TestAmerica Portland | 9405 SW Nimbus Avenue | | Beaverton | OR | 97008-7145 | 503.906.9200 | 503.906.9210 |
| TestAmerica Richland | 2800 George Washington Way | | Richland | WA | 99354-1613 | 509.375.3131 | 509.375.5590 |
| TestAmerica Sacramento | 880 Riverside Parkway | | West Sacramento | CA | 95605-1500 | 916.373.5600 | 303.467.7248 |
| TestAmerica San Antonio | 1951 NW Loop 410 | Building 11 Door 11 | San Antonio | TX | 78216-2333 | 361.563.1039 | |
| TestAmerica Savannah | 5102 LaRoche Avenue | | Savannah | GA | 31404-6019 | 912.354.7858 | 912.352.0165 |
| TestAmerica Seattle | 5755 8th Street East | | Tacoma | WA | 98424-1317 | 253.922.2310 | 253.922.5047 |

| | | | | | | | |
|----------------------------|---------------------------|-----------|----------------|----|------------|--------------|--------------|
| TestAmerica Shelton | 12 Progress Drive | | Shelton | CT | 06484-6216 | 203.929.8140 | |
| TestAmerica South Jersey | 3000 Lincoln Drive East | Suite A | Marlton | NJ | 08053-1500 | 856.334.1030 | |
| TestAmerica Spokane | 11922 E 1st Avenue | | Spokane | WA | 99206-5302 | 509.924.9200 | 509.924.9290 |
| TestAmerica St. Louis | 13715 Rider Trail North | | Earth City | MO | 63045-1205 | 314.298.8566 | 314.298.8757 |
| TestAmerica Syracuse | 118 Boss Rd | | Syracuse | NY | 13211-2217 | 315.431.0171 | |
| TestAmerica Tallahassee | 2846 Industrial Plaza Dr. | | Tallahassee | FL | 32301-3539 | 850.878.3994 | 850.878.9504 |
| TestAmerica Tampa | 6712 Benjamin Road | Suite 100 | Tampa | FL | 33634-4403 | 813.885.7427 | 813.885.7049 |
| TestAmerica Virginia Beach | 5135 Cleveland St | | Virginia Beach | VA | 23462-6501 | 757.671.1291 | |
| TestAmerica Westfield | 501 Southampton Road | | Westfield | MA | 01085-1592 | 413.572.4000 | 413.572.3707 |



ERM, Inc.
 8425 Woodfield Crossing Blvd, Ste. 560-W
 Indianapolis, Indiana 46240
 (317) 706-2000

Well Construction Diagram

Well Identification: _____

Client: _____
 Project: _____
 Proj. No.: _____
 ERM Geologist: _____
 Drilling Company: _____
 Driller: _____
 Drilling Method: _____

Date Installed: _____
 Date Developed: _____
 Development Method: _____
 Water Removed During Development: _____
 Static Water Level Depth/Elevation: _____
 Top of Casing Elevation: _____
 Ground Elevation: _____

