PROFESSIONAL SERVICES CONTRACT

Contract #00000000000000000000024360

This Contract ("this Contract"), entered into by and between the Indiana Department of Environmental Management (the "State") and Microbac Laboratories, Inc. (the "Contractor"), is executed pursuant to the terms and conditions set forth herein. In consideration of those mutual undertakings and covenants, the parties agree as follows:

1. Duties of Contractor.

The Contractor shall provide the following services relative to this Contract in accordance with the attached **Exhibit A, Scope of Work; Exhibit C, Schedule of Project Tasks, Exhibit D, Special Conditions and Exhibit E, Laboratory Request Form.**

2. Consideration.

The Contractor will be paid in accordance with the attached **Exhibit B, Total Estimated Project Expense Budget** for performing the duties set forth above. Total remuneration under this Contract shall not exceed $164,000.00.

3. Term. This Contract shall be effective for a period of four (4) years. It shall commence on **January 1, 2018** and shall remain in effect for four years.

4. Access to Records. The Contractor and its subcontractors, if any, shall maintain all books, documents, papers, accounting records, and other evidence pertaining to all costs incurred under this Contract. They shall make such materials available at their respective offices at all reasonable times during this Contract, and for three (3) years from the date of final payment under this Contract, for inspection by the State or its authorized designees. Copies shall be furnished at no cost to the State if requested.

5. Assignment; Successors.

The Contractor binds its successors and assignees to all the terms and conditions of this Contract. The Contractor shall not assign or subcontract the whole or any part of this Contract without the State's prior written consent. The Contractor may assign its right to receive payments to such third parties as the Contractor may desire without the prior written consent of the State, provided that the Contractor gives written notice (including evidence of such assignment) to the State thirty (30) days in advance of any payment so assigned. The assignment shall cover all unpaid amounts under this Contract and shall not be made to more than one party.

6. Assignment of Antitrust Claims.

As part of the consideration for the award of this Contract, the Contractor assigns to the State all right, title and interest in and to any claims the Contractor now has, or may acquire, under state or federal antitrust laws relating to the products or services which are the subject of this Contract.

7. Audits.

The Contractor acknowledges that it may be required to submit to an audit of funds paid through this Contract. Any such audit shall be conducted in accordance with IC §5-11-1, et seq., and audit guidelines specified by the State.

The State considers the Contractor to be a "Contractor" under 2 C.F.R. 200.330 for purposes of this Contract. However, if it is determined that the Contractor is a "subrecipient" and if required by applicable provisions of 2 C.F.R. 200 (Uniform Administrative Requirements, Cost Principles, and Audit Requirements), Contractor shall arrange for a financial and compliance audit, which complies with 2 C.F.R. 200.500 et seq.

8. Authority to Bind Contractor.

The signatory for the Contractor represents that he/she has been duly authorized to execute this Contract on behalf of the Contractor and has obtained all necessary or applicable approvals to make this Contract fully binding upon the Contractor when his/her signature is affixed, and accepted by the State.

9. Changes in Work.

The Contractor shall not commence any additional work or change the scope of the work until authorized in writing by the State. The Contractor shall make no claim for additional compensation in the absence of a prior written approval and amendment executed by all signatories hereto. This Contract may only be amended, supplemented or modified by a written document executed in the same manner as this Contract.

10. Compliance with Laws.

A. The Contractor shall comply with all applicable federal, state, and local laws, rules, regulations, and ordinances, and all provisions required thereby to be included herein are hereby incorporated by reference. The enactment or modification of any applicable state or federal statute or the promulgation of rules or regulations thereunder after execution of this Contract shall be reviewed by the State and the Contractor to determine whether the provisions of this Contract require formal modification.

B. The Contractor and its agents shall abide by all ethical requirements that apply to persons who have a business relationship with the State as set forth in IC §4-2-6, et seq., IC §4-2-7, et seq. and the regulations promulgated thereunder. **If the Contractor has knowledge, or would have acquired knowledge with reasonable inquiry, that a state officer, employee, or special state appointee, as those terms are defined in IC 4-2-6-1, has a financial interest in the Contract, the Contractor shall ensure compliance with the disclosure requirements in IC 4-2-6-10.5 prior to the execution of this contract.** If the Contractor is not familiar with these ethical requirements, the Contractor should refer any questions to the Indiana State Ethics Commission, or visit the Inspector General's website at http://www.in.gov/ig/. If the Contractor or its agents violate any applicable ethical standards, the State may, in its sole discretion, terminate this Contract immediately upon notice to the Contractor. In addition, the Contractor may be subject to penalties under IC §§4-2-6, 4-2-7, 35-44.1-1-4, and under any other applicable laws.

C. The Contractor certifies by entering into this Contract that neither it nor its principal(s) is presently in arrears in payment of taxes, permit fees or other statutory, regulatory or judicially required payments to the State of Indiana. The Contractor agrees that any payments currently due to the State of Indiana may be withheld from payments due to the Contractor. Additionally, further work or payments may be withheld, delayed, or denied and/or this Contract suspended until the Contractor is current in its payments and has submitted proof of such payment to the State.

D. The Contractor warrants that it has no current, pending or outstanding criminal, civil, or enforcement actions initiated by the State, and agrees that it will immediately notify the State of any such actions. During the term of such actions, the Contractor agrees that the State may delay, withhold, or deny work under any supplement, amendment, change order or other contractual device issued pursuant to this Contract.

E. If a valid dispute exists as to the Contractor's liability or guilt in any action initiated by the State or its agencies, and the State decides to delay, withhold, or deny work to the Contractor, the Contractor may request

that it be allowed to continue, or receive work, without delay. The Contractor must submit, in writing, a request for review to the Indiana Department of Administration (IDOA) following the procedures for disputes outlined herein. A determination by IDOA shall be binding on the parties. Any payments that the State may delay, withhold, deny, or apply under this section shall not be subject to penalty or interest, except as permitted by IC §5-17-5.

F. The Contractor warrants that the Contractor and its subcontractors, if any, shall obtain and maintain all required permits, licenses, registrations, and approvals, and shall comply with all health, safety, and environmental statutes, rules, or regulations in the performance of work activities for the State. Failure to do so may be deemed a material breach of this Contract and grounds for immediate termination and denial of further work with the State.

G. The Contractor affirms that, if it is an entity described in IC Title 23, it is properly registered and owes no outstanding reports to the Indiana Secretary of State.

H. As required by IC §5-22-3-7:

(1) The Contractor and any principals of the Contractor certify that:

(A) the Contractor, except for de minimis and nonsystematic violations, has not violated the terms of:

(i) IC §24-4.7 [Telephone Solicitation Of Consumers];

(ii) IC §24-5-12 [Telephone Solicitations]; or

(iii) IC §24-5-14 [Regulation of Automatic Dialing Machines];

in the previous three hundred sixty-five (365) days, even if IC §24-4.7 is preempted by federal law; and

(B) the Contractor will not violate the terms of IC §24-4.7 for the duration of the Contract, even if IC §24-4.7 is preempted by federal law.

(2) The Contractor and any principals of the Contractor certify that an affiliate or principal of the Contractor and any agent acting on behalf of the Contractor or on behalf of an affiliate or principal of the Contractor, except for de minimis and nonsystematic violations,

(A) has not violated the terms of IC §24-4.7 in the previous three hundred sixty-five (365) days, even if IC §24-4.7 is preempted by federal law; and

(B) will not violate the terms of IC §24-4.7 for the duration of the Contract, even if IC §24-4.7 is preempted by federal law.

11. Condition of Payment.

All services provided by the Contractor under this Contract must be performed to the State's reasonable satisfaction, as determined at the discretion of the undersigned State representative and in accordance with all applicable federal, state, local laws, ordinances, rules and regulations. The State shall not be required to pay for work found to be unsatisfactory, inconsistent with this Contract or performed in violation of any federal, state or local statute, ordinance, rule or regulation.

12. Confidentiality of State Information.

The Contractor understands and agrees that data, materials, and information disclosed to the Contractor may contain confidential and protected information. The Contractor covenants that data, material, and information gathered, based upon or disclosed to the Contractor for the purpose of this Contract will not be disclosed to or discussed with third parties without the prior written consent of the State.

The parties acknowledge that the services to be performed by Contractor for the State under this Contract may require or allow access to data, materials, and information containing Social Security numbers maintained by the State in its computer system or other records. In addition to the covenant made above in this section and pursuant to 10 IAC 5-3-1(4), the Contractor and the State agree to comply with the provisions of IC §4-1-10 and IC §4-1-11. If any Social Security number(s) is/are disclosed by Contractor, Contractor agrees to pay the cost of the notice of disclosure of a breach of the security of the system in addition to any other claims and expenses for which it is liable under the terms of this contract.

13. Continuity of Services. Deleted by agreement of the Parties

14. Debarment and Suspension.

A. The Contractor certifies by entering into this Contract that neither it nor its principals nor any of its subcontractors are presently debarred, suspended, proposed for debarment, declared ineligible or voluntarily excluded from entering into this Contract by any federal agency or by any department, agency or political subdivision of the State of Indiana. The term "principal" for purposes of this Contract means an officer, director, owner, partner, key employee or other person with primary management or supervisory responsibilities, or a person who has a critical influence on or substantive control over the operations of the Contractor.

B. The Contractor certifies that it has verified the state and federal suspension and debarment status for all subcontractors receiving funds under this Contract and shall be solely responsible for any recoupment, penalties or costs that might arise from use of a suspended or debarred subcontractor. The Contractor shall immediately notify the State if any subcontractor becomes debarred or suspended, and shall, at the State's request, take all steps required by the State to terminate its contractual relationship with the subcontractor for work to be performed under this Contract.

15. Default by State. Deleted by Agreement of the Parties.

16. Disputes.

A. Should any disputes arise with respect to this Contract, the Contractor and the State agree to act immediately to resolve such disputes. Time is of the essence in the resolution of disputes.

B. The Contractor agrees that, the existence of a dispute notwithstanding, it will continue without delay to carry out all of its responsibilities under this Contract that are not affected by the dispute. Should the Contractor fail to continue to perform its responsibilities regarding all non-disputed work, without delay, any additional costs incurred by the State or the Contractor as a result of such failure to proceed shall be borne by the Contractor, and the Contractor shall make no claim against the State for such costs.

C. If the parties are unable to resolve a contract dispute between them after good faith attempts to do so, a dissatisfied party shall submit the dispute to the Commissioner of the Indiana Department of Administration for resolution. The dissatisfied party shall give written notice to the Commissioner and the other party. The notice shall include: (1) a description of the disputed issues, (2) the efforts made to resolve the dispute, and (3) a proposed resolution. The Commissioner shall promptly issue a Notice setting out documents and materials to be submitted to the Commissioner in order to resolve the dispute; the Notice may also afford the parties the opportunity to make presentations and enter into further negotiations. Within thirty (30) business days of the conclusion of the final presentations, the Commissioner shall issue a written decision and furnish it to both parties. The Commissioner's decision shall be the final and conclusive administrative decision unless either party serves on the Commissioner and the other party, within ten (10) business days after receipt of the Commissioner's decision, a written request for reconsideration and modification of the written decision.

If the Commissioner does not modify the written decision within thirty (30) business days, either party may take such other action helpful to resolving the dispute, including submitting the dispute to an Indiana court of competent jurisdiction. If the parties accept the Commissioner's decision, it may be memorialized as a written Amendment to this Contract if appropriate.

D. The State may withhold payments on disputed items pending resolution of the dispute. The unintentional nonpayment by the State to the Contractor of one or more invoices not in dispute in accordance with the terms of this Contract will not be cause for the Contractor to terminate this Contract, and the Contractor may bring suit to collect these amounts without following the disputes procedure contained herein.

E. With the written approval of the Commissioner of the Indiana Department of Administration, the parties may agree to forego the process described in subdivision C. relating to submission of the dispute to the Commissioner.

F. This paragraph shall not be construed to abrogate provisions of Ind. Code 4-6-2-11 in situations where dispute resolution efforts lead to a compromise of claims in favor of the State as described in that statute. In particular, releases or settlement agreements involving releases of legal claims or potential legal claims of the state should be processed consistent with Ind. Code 4-6-2-11, which requires approval of the Governor and Attorney General.

17. Drug-Free Workplace Certification.

As required by Executive Order No. 90-5 dated April 12, 1990, issued by the Governor of Indiana, the Contractor hereby covenants and agrees to make a good faith effort to provide and maintain a drug-free workplace. The Contractor will give written notice to the State within ten (10) days after receiving actual notice that the Contractor, or an employee of the Contractor in the State of Indiana, has been convicted of a criminal drug violation occurring in the workplace. False certification or violation of this certification may result in sanctions including, but not limited to, suspension of contract payments, termination of this Contract and/or debarment of contracting opportunities with the State for up to three (3) years.

In addition to the provisions of the above paragraph, if the total amount set forth in this Contract is in excess of $25,000.00, the Contractor certifies and agrees that it will provide a drug-free workplace by:

A. Publishing and providing to all of its employees a statement notifying them that the unlawful manufacture, distribution, dispensing, possession or use of a controlled substance is prohibited in the Contractor's workplace, and specifying the actions that will be taken against employees for violations of such prohibition;

B. Establishing a drug-free awareness program to inform its employees of: (1) the dangers of drug abuse in the workplace; (2) the Contractor's policy of maintaining a drug-free workplace; (3) any available drug counseling, rehabilitation and employee assistance programs; and (4) the penalties that may be imposed upon an employee for drug abuse violations occurring in the workplace;

C. Notifying all employees in the statement required by subparagraph (A) above that as a condition of continued employment, the employee will: (1) abide by the terms of the statement; and (2) notify the Contractor of any criminal drug statute conviction for a violation occurring in the workplace no later than five (5) days after such conviction;

D. Notifying the State in writing within ten (10) days after receiving notice from an employee under subdivision (C)(2) above, or otherwise receiving actual notice of such conviction;

E. Within thirty (30) days after receiving notice under subdivision (C)(2) above of a conviction, imposing the following sanctions or remedial measures on any employee who is convicted of drug abuse violations occurring in the workplace: (1) taking appropriate personnel action against the employee, up to and including termination; or (2) requiring such employee to satisfactorily participate in a drug abuse assistance or rehabilitation program approved for such purposes by a federal, state or local health, law enforcement, or other appropriate agency; and

F. Making a good faith effort to maintain a drug-free workplace through the implementation of subparagraphs (A) through (E) above.

18. Employment Eligibility Verification.

As required by IC §22-5-1.7, the Contractor swears or affirms under the penalties of perjury that the Contractor does not knowingly employ an unauthorized alien. The Contractor further agrees that:

A. The Contractor shall enroll in and verify the work eligibility status of all his/her/its newly hired employees through the E-Verify program as defined in IC §22-5-1.7-3. The Contractor is not required to participate should the E-Verify program cease to exist. Additionally, the Contractor is not required to participate if the Contractor is self-employed and does not employ any employees.

B. The Contractor shall not knowingly employ or contract with an unauthorized alien. The Contractor shall not retain an employee or contract with a person that the Contractor subsequently learns is an unauthorized alien.

C. The Contractor shall require his/her/its subcontractors, who perform work under this Contract, to certify to the Contractor that the subcontractor does not knowingly employ or contract with an unauthorized alien and that the subcontractor has enrolled and is participating in the E-Verify program. The Contractor agrees to maintain this certification throughout the duration of the term of a contract with a subcontractor.

The State may terminate for default if the Contractor fails to cure a breach of this provision no later than thirty (30) days after being notified by the State.

19. Employment Option.

If the State determines that it would be in the State's best interest to hire an employee of the Contractor, the Contractor will release the selected employee from any non-competition agreements that may be in effect. This release will be at no cost to the State or the employee.

20. Force Majeure.

In the event that either party is unable to perform any of its obligations under this Contract or to enjoy any of its benefits because of natural disaster or decrees of governmental bodies not the fault of the affected party (hereinafter referred to as a "Force Majeure Event"), the party who has been so affected shall immediately or as soon as is reasonably possible under the circumstances give notice to the other party and shall do everything possible to resume performance. Upon receipt of such notice, all obligations under this Contract shall be immediately suspended. If the period of nonperformance exceeds thirty (30) days from the receipt of notice of the Force Majeure Event, the party whose ability to perform has not been so affected may, by giving written notice, terminate this Contract.

21. Funding Cancellation.

When the Director of the State Budget Agency makes a written determination that funds are not appropriated or otherwise available to support continuation of performance of this Contract, this Contract shall be canceled. A determination by the Director of State Budget Agency that funds are not appropriated or otherwise available to support continuation of performance shall be final and conclusive.

22. Governing Law.

This Contract shall be governed, construed, and enforced in accordance with the laws of the State of Indiana, without regard to its conflict of laws rules. Suit, if any, must be brought in the State of Indiana.

23. HIPAA Compliance.

If this Contract involves services, activities or products subject to the Health Insurance Portability and Accountability Act of 1996 (HIPAA), the Contractor covenants that it will appropriately safeguard Protected Health Information (defined in 45 CFR 160.103), and agrees that it is subject to, and shall comply with, the provisions of 45 CFR 164 Subpart E regarding use and disclosure of Protected Health Information.

24. Indemnification.

The Contractor agrees to indemnify, defend, and hold harmless the State, its agents, officials, and employees from all third party claims and suits including court costs, attorney's fees, and other expenses caused by any act or omission of the Contractor and/or its subcontractors, if any, in the performance of this Contract. The State shall not provide such indemnification to the Contractor.

25. Independent Contractor; Workers' Compensation Insurance.

The Contractor is performing as an independent entity under this Contract. No part of this Contract shall be construed to represent the creation of an employment, agency, partnership or joint venture agreement between the parties. Neither party will assume liability for any injury (including death) to any persons, or damage to any property, arising out of the acts or omissions of the agents, employees or subcontractors of the other party. The Contractor shall provide all necessary unemployment and workers' compensation insurance for the Contractor's employees, and shall provide the State with a Certificate of Insurance evidencing such coverage prior to starting work under this Contract.

26. Information Technology Enterprise Architecture Requirements.

If the Contractor provides any information technology related products or services to the State, the Contractor shall comply with all IOT standards, policies and guidelines, which are online at http://iot.in.gov/architecture/. The Contractor specifically agrees that all hardware, software and services provided to or purchased by the State shall be compatible with the principles and goals contained in the electronic and information technology accessibility standards adopted under Section 508 of the Federal Rehabilitation Act of 1973 (29 U.S.C. 794d) and IC §4-13.1-3. Any deviation from these architecture requirements must be approved in writing by IOT in advance. The State may terminate this Contract for default if the Contractor fails to cure a breach of this provision within a reasonable time.

27. Insurance.

A. The Contractor and their subcontractors (if any) shall secure and keep in force during the term of this Contract the following insurance coverages (if applicable) covering the Contractor for any and all claims of any nature which may in any manner arise out of or result from Contractor's performance under this Contract:

1. Commercial general liability, including contractual coverage, and products or completed operations coverage (if applicable), with minimum liability limits not less than $700,000 per person and $1,000,000 per occurrence unless additional coverage is required by the State. The State is to be named as an additional insured on a primary, non-contributory basis for any liability arising directly or indirectly under or in connection with this Contract.

2. Automobile liability for owned, non-owned and hired autos with minimum liability limits of $100,000,000 per person and $1,000,000 per occurrence. The State is to be named as an additional insured on a primary, non-contributory basis.

3. Errors and Omissions liability with minimum liability limits of $1,000,000 per claim and in the aggregate. Coverage for the benefit of the State shall continue for a period of two (2) years after the date of service provided under this Contract.

4. Fiduciary Liability is required if the Contractor is responsible for the management and oversight of various employee benefit plans and programs such as pensions, profit-sharing and savings, among others. These contractors face potential claims for mismanagement brought by plan members. Limits should be no less than $1,000,000 per cause of action or per occurrence.

5. Valuable Papers coverage, available under an Inland Marine policy, is required when any plans, drawings, media, data, records, reports, billings and other documents are produced or used under this agreement. Insurance must have limits sufficient to pay for the re-creation and reconstruction of such records.

6. The Contractor shall secure the appropriate Surety or Fidelity Bond(s) as required by the state department served or by applicable statute.

7. Excess (Umbrella Liability) policy for no less than $4,000,000 will cover claims over the basic policy limits for commercial general liability and automobile liability policies.

8. The Contractor shall provide proof of such insurance coverage by tendering to the undersigned State representative a certificate of insurance prior to the commencement of this Contract and proof of workers' compensation coverage meeting all statutory requirements of IC §22-3-2. In addition, proof of an "all states endorsement" covering claims occurring outside the State is required if any of the services provided under this Contract involve work outside of Indiana.

B. The Contractor's insurance coverage must meet the following additional requirements:

1. The insurer must have a certificate of authority or other appropriate authorization to operate in the state in which the policy was issued.

2. Any deductible or self-insured retention amount or other similar obligation under the insurance policies shall be the sole obligation of the Contractor.

3. The State will be defended, indemnified and held harmless to the full extent of any coverage actually secured by the Contractor in excess of the minimum requirements set forth above. The duty to indemnify the State under this Contract shall not be limited by the insurance required in this Contract.

4. The insurance required in this Contract, through a policy or endorsement(s), shall include a provision that the policy and endorsements may not be canceled or modified without thirty (30) days' prior written notice to the undersigned State agency.

5. The Contractor waives and agrees to require their insurer to waive their rights of subrogation against the State of Indiana.

C. Failure to provide insurance as required in this Contract may be deemed a material breach of contract entitling the State to immediately terminate this Contract. The Contractor shall furnish a certificate of insurance and all endorsements to the State before the commencement of this Contract.

28. Key Person(s).

A. If both parties have designated that certain individual(s) are essential to the services offered, the parties agree that should such individual(s) leave their employment during the term of this Contract for whatever reason, the State shall have the right to terminate this Contract upon thirty (30) days' prior written notice.

B. In the event that the Contractor is an individual, that individual shall be considered a key person and, as such, essential to this Contract. Substitution of another for the Contractor shall not be permitted without express written consent of the State.

Nothing in sections A and B, above shall be construed to prevent the Contractor from using the services of others to perform tasks ancillary to those tasks which directly require the expertise of the key person. Examples of such ancillary tasks include secretarial, clerical, and common labor duties. The Contractor shall, at all times, remain responsible for the performance of all necessary tasks, whether performed by a key person or others.

Key person(s) to this Contract is/are if applicable, listed in **Exhibit D.**

29. Licensing Standards.

The Contractor, its employees and subcontractors shall comply with all applicable licensing standards, certification standards, accrediting standards and any other laws, rules, or regulations governing services to be provided by the Contractor pursuant to this Contract. The State will not pay the Contractor for any services performed when the Contractor, its employees or subcontractors are not in compliance with such applicable standards, laws, rules, or regulations. If any license, certification or accreditation expires or is revoked, or any disciplinary action is taken against an applicable license, certification, or accreditation, the Contractor shall notify the State immediately and the State, at its option, may immediately terminate this Contract.

30. Merger & Modification.

This Contract constitutes the entire agreement between the parties. No understandings, agreements, or representations, oral or written, not specified within this Contract will be valid provisions of this Contract. This Contract may not be modified, supplemented, or amended, except by written agreement signed by all necessary parties.

31. Minority and Women's Business Enterprises Compliance.

Award of this Contract was based, in part, on the MBE/WBE participation plan. The following certified MBE or WBE subcontractors will be participating in this Contract:

MBE/WBE PHONE COMPANY NAME SCOPE OF PRODUCTS and/or SERVICES UTILIZATION
DATE PERCENT
Contract was not awarded based on a MBE/WBE participation plan.

A copy of each subcontractor agreement must be submitted to IDOA's MBE/WBE Division within thirty (30) days of the effective date of this Contract. Failure to provide a copy of any subcontractor agreement will be deemed a violation of the rules governing MBE/WBE procurement, and may result in sanctions allowable under 25 IAC 5-7-8. Failure to provide any subcontractor agreement may also be considered a material breach of this Contract. The Contractor must obtain approval from IDOA's MBE/WBE Division before changing the participation plan submitted in connection with this Contract.
The Contractor shall report payments made to MBE/WBE Division subcontractors under this Contract on a monthly basis. Monthly reports shall be made using the online audit tool, commonly referred to as "Pay Audit." MBE/WBE Division subcontractor payments shall also be reported to the Division as reasonably requested and in a format to be determined by Division.

32. Nondiscrimination.

Pursuant to the Indiana Civil Rights Law, specifically including IC §22-9-1-10, and in keeping with the purposes of the federal Civil Rights Act of 1964, the Age Discrimination in Employment Act, and the Americans with Disabilities Act, the Contractor covenants that it shall not discriminate against any employee or applicant for employment relating to this Contract with respect to the hire, tenure, terms, conditions or privileges of employment or any matter directly or indirectly related to employment, because of the employee's or applicant's race, color, national origin, religion, sex, age, disability, ancestry, status as a veteran, or any other characteristic protected by federal, state, or local law ("Protected Characteristics"). Contractor certifies compliance with applicable federal laws, regulations, and executive orders prohibiting discrimination based on the Protected Characteristics in the provision of services. Breach of this paragraph may be regarded as a material breach of this Contract, but nothing in this paragraph shall be construed to imply or establish an employment relationship between the State and any applicant or employee of the Contractor or any subcontractor.

The State is a recipient of federal funds, and therefore, where applicable, Contractor and any subcontractors shall comply with requisite affirmative action requirements, including reporting, pursuant to 41 CFR Chapter 60, as amended, and Section 202 of Executive Order 11246 as amended by Executive Order 13672.

33. Notice to Parties.

Whenever any notice, statement or other communication is required under this Contract, it shall be sent by first class mail or via an established courier/delivery service to the addresses listed in **Exhibit D.**

As required by IC §4-13-2-14.8, payments to the Contractor shall be made via electronic funds transfer in accordance with instructions filed by the Contractor with the Indiana Auditor of State.

34. Order of Precedence; Incorporation by Reference.

Any inconsistency or ambiguity in this Contract shall be resolved by giving precedence in the following order: (1) this Contract, (2) attachments prepared by the State, (3) RFP# 109, (4) Contractor's response to RFP# 109, and (5) attachments prepared by the Contractor. All attachments, and all documents referred to in this paragraph, are hereby incorporated fully by reference.

35. Ownership of Documents and Materials.

A. All documents, records, programs, applications, data, algorithms, film, tape, articles, memoranda, and other materials (the "Materials") not developed or licensed by the Contractor prior to execution of this Contract, but specifically developed under this Contract shall be considered "work for hire" and the Contractor hereby transfers and assigns any ownership claims to the State so that all Materials will be the property of the State. If ownership interest in the Materials cannot be assigned to the State, the Contractor grants the State a non-exclusive, non-cancelable, perpetual, worldwide royalty-free license to use the Materials and to use, modify, copy and create derivative works of the Materials.

B. Use of the Materials, other than related to contract performance by the Contractor, without the prior written consent of the State, is prohibited. During the performance of this Contract, the Contractor shall be responsible for any loss of or damage to the Materials developed for or supplied by the State and used to develop or assist in the services provided while the Materials are in the possession of the Contractor. Any loss or damage thereto shall be restored at the Contractor's expense. The Contractor shall provide the State full, immediate, and unrestricted access to the Materials and to Contractor's work product during the term of this Contract.

36. Payments.

A. All payments shall be made thirty five (35) days in arrears in conformance with State fiscal policies and procedures and, as required by IC §4-13-2-14.8, the direct deposit by electronic funds transfer to the financial institution designated by the Contractor in writing unless a specific waiver has been obtained from the Indiana Auditor of State. No payments will be made in advance of receipt of the goods or services that are the subject of this Contract except as permitted by IC §4-13-2-20.

B. The State Budget Agency and the Contractor acknowledge that if the Contractor is being paid in advance for the maintenance of equipment and/ or software. Pursuant to IC §4-13-2-20(b)(14), Contractor agrees that if it fails to perform the maintenance required under this Contract, upon receipt of written notice from the State, it shall promptly refund the consideration paid, pro-rated through the date of non-performance.

37. Penalties/Interest/Attorney's Fees.

The State will in good faith perform its required obligations hereunder and does not agree to pay any penalties, liquidated damages, interest or attorney's fees, except as permitted by Indiana law, in part, IC §5-17-5, IC §34-54-8, IC §34-13-1 and IC § 34-52-2-3.

Notwithstanding the provisions contained in IC §5-17-5, any liability resulting from the State's failure to make prompt payment shall be based solely on the amount of funding originating from the State and shall not be based on funding from federal or other sources.

38. Progress Reports.

The Contractor shall submit progress reports to the State upon request. The report shall be written. The progress reports shall serve the purpose of assuring the State that work is progressing in line with the schedule, and that completion can be reasonably assured on the scheduled date.

39. Public Record.

The Contractor acknowledges that the State will not treat this Contract as containing confidential information, and will post this Contract on its website as required by Executive Order 05-07. Use by the public of the information contained in this Contract shall not be considered an act of the State.

40. Renewal Option.

This Contract may be renewed under the same terms and conditions, subject to the approval of the Commissioner of the Department of Administration and the State Budget Director in compliance with IC §5-22-17-4. The term of the renewed contract may not be longer than the term of the original Contract.

41. Severability.

The invalidity of any section, subsection, clause or provision of this Contract shall not affect the validity of the remaining sections, subsections, clauses or provisions of this Contract.

42. Substantial Performance.

This Contract shall be deemed to be substantially performed only when fully performed according to its terms and conditions and any written amendments or supplements.

43. Taxes.

The State is exempt from most state and local taxes and many federal taxes. The State will not be responsible for any taxes levied on the Contractor as a result of this Contract.

44. Termination for Convenience.

This Contract may be terminated, in whole or in part, by the State, which shall include and is not limited to IDOA and the State Budget Agency whenever, for any reason, the State determines that such termination is in its best interest. Termination of services shall be effected by delivery to the Contractor of a Termination Notice at least thirty (30) days prior to the termination effective date, specifying the extent to which performance of services under such termination becomes effective. The Contractor shall be compensated for services properly rendered prior to the effective date of termination. The State will not be liable for services performed after the effective date of termination. The Contractor shall be compensated for services herein provided but in no case shall total payment made to the Contractor exceed the original contract price or shall any price increase be allowed on individual line items if canceled only in part prior to the original termination date. For the purposes of this paragraph, the parties stipulate and agree that IDOA. shall be deemed to be a party to this agreement with authority to terminate the same for convenience when such termination is determined by the Commissioner of IDOA to be in the best interests of the State.

45. Termination for Default.

A. With the provision of thirty (30) days' notice to the Contractor, the State may terminate this Contract in whole or in part if the Contractor fails to:

1. Correct or cure any breach of this Contract; the time to correct or cure the breach may be extended beyond thirty (30) days if the State determines progress is being made and the extension is agreed to by the parties;
2. Deliver the supplies or perform the services within the time specified in this Contract or any extension;
3. Make progress so as to endanger performance of this Contract; or
4. Perform any of the other provisions of this Contract.

B. If the State terminates this Contract in whole or in part, it may acquire, under the terms and in the manner the State considers appropriate, supplies or services similar to those terminated, and the Contractor will be liable to the State for any excess costs for those supplies or services. However, the Contractor shall continue the work not terminated.

C. The State shall pay the contract price for completed supplies delivered and services accepted. The Contractor and the State shall agree on the amount of payment for manufacturing materials delivered and accepted and for the protection and preservation of the property. Failure to agree will be a dispute under the Disputes clause. The State may withhold from these amounts any sum the State determines to be necessary to protect the State against loss because of outstanding liens or claims of former lien holders.

D. The rights and remedies of the State in this clause are in addition to any other rights and remedies provided by law or equity or under this Contract.

46. Travel.

No expenses for travel will be reimbursed unless specifically permitted under the scope of services or consideration provisions. Expenditures made by the Contractor for travel will be reimbursed at the current rate paid by the State and in accordance with the State Travel Policies and Procedures as specified in the current Financial Management Circular. Out-of-state travel requests must be reviewed by the State for availability of funds and for appropriateness per Circular guidelines.

47. Indiana Veteran's Business Enterprise Compliance.

Award of this Contract was based, in part, on the Indiana Veteran's Business Enterprise ("IVBE") participation plan. The following IVBE subcontractors will be participating in this Contract:

VBE	PHONE	COMPANY NAME	SCOPE OF PRODUCTS and/or SERVICES
	UTILIZATION	DATE	PERCENT

Award of this Contract was not based on an IVBE participation plan.

A copy of each subcontractor agreement shall be submitted to IDOA within thirty (30) days of the request. Failure to provide any subcontractor agreement may also be considered a material breach of this Contract. The Contractor must obtain approval from IDOA before changing the IVBE participation plan submitted in connection with this Contract.

The Contractor shall report payments made to IVBE subcontractors under this Contract on a monthly basis. Monthly reports shall be made using the online audit tool, commonly referred to as "Pay Audit." IVBE subcontractor payments shall also be reported to IDOA as reasonably requested and in a format to be determined by IDOA.

48. Waiver of Rights.

No right conferred on either party under this Contract shall be deemed waived, and no breach of this Contract excused, unless such waiver is in writing and signed by the party claimed to have waived such right. Neither the State's review, approval or acceptance of, nor payment for, the services required under this Contract shall be construed to operate as a waiver of any rights under this Contract or of any cause of action arising out of the performance of this Contract, and the Contractor shall be and remain liable to the State in accordance with applicable law for all damages to the State caused by the Contractor's negligent performance of any of the services furnished under this Contract.

49. Work Standards.

The Contractor shall execute its responsibilities by following and applying at all times the highest professional and technical guidelines and standards. If the State becomes dissatisfied with the work product of

or the working relationship with those individuals assigned to work on this Contract, the State may request in writing the replacement of any or all such individuals, and the Contractor shall grant such request.

50. State Boilerplate Affirmation Clause.

I swear or affirm under the penalties of perjury that I have not altered, modified, changed or deleted the State's Boilerplate contract clauses (as contained in the 2016 OAG/ IDOA *Professional Services Contract Manual)* in any way except for the following clauses which are named below:

1. Duties of the Contractor – Modified to attach Exhibits regarding Contractor duties.
2. Consideration – Modified to attach Exhibit of Project Expense Budget.
13. Continuity of Service – Deleted.
15. Default by State – Deleted.
27. Insurance – Modified by Agreement of the Parties.
28. Key Person – Specified in the attached Exhibit D.
33. Notice to Parties – Specified in the attached Exhibit D
38. Progress Reports – Modified to require written form.

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Non-Collusion and Acceptance

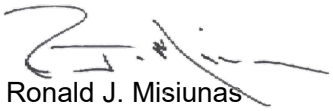
The undersigned attests, subject to the penalties for perjury, that the undersigned is the Contractor, or that the undersigned is the properly authorized representative, agent, member or officer of the Contractor. Further, to the undersigned's knowledge, neither the undersigned nor any other member, employee, representative, agent or officer of the Contractor, directly or indirectly, has entered into or been offered any sum of money or other consideration for the execution of this Contract other than that which appears upon the face hereof**.**
Furthermore, if the undersigned has knowledge that a state officer, employee, or special state appointee, as those terms are defined in IC 4-2-6-1, has a financial interest in the Contract, the Contractor attests to compliance with the disclosure requirements in IC 4-2-6-10.5.

Agreement to Use Electronic Signatures

I agree, and it is my intent, to sign this Contract by accessing State of Indiana Supplier Portal using the secure password assigned to me and by electronically submitting this Contract to the State of Indiana. I understand that my signing and submitting this Contract in this fashion is the legal equivalent of having placed my handwritten signature on the submitted Contract and this affirmation. I understand and agree that by electronically signing and submitting this Contract in this fashion I am affirming to the truth of the information contained therein. I understand that this Contract will not become binding on the State until it has been approved by the Department of Administration, the State Budget Agency, and the Office of the Attorney General, which approvals will be posted on the Active Contracts Database:
https://hr85.gmis.in.gov/psp/pa91prd/EMPLOYEE/EMPL/h/?tab=PAPP_GUEST

In Witness Whereof, Contractor and the State have, through their duly authorized representatives, entered into this Contract. The parties, having read and understood the foregoing terms of this Contract, do by their respective signatures dated below agree to the terms thereof.

MICROBAC LABORATORIES, INC

By: 
Ronald J. Misiunas

Title: Director of Laboratory Services

Date: 12/21/17

Indiana Department of Environmental Management

By:

Title:

Date: Brian Rockensuess Digitally signed by Brian Rockensuess Date: 2017.12.21 13:50:51 -05'00'

Electronically Approved by: Department of Administration By: (for) Jessica Robertson, Commissioner *Refer to Electronic Approval History found after the final page of the Executed Contract for details.*	
Electronically Approved by: State Budget Agency By: (for) Jason D.Dudich, Director *Refer to Electronic Approval History found after the final page of the Executed Contract for details.*	Electronically Approved as to Form and Legality: Office of the Attorney General By: (for) Curtis T. Hill, Jr., Attorney General *Refer to Electronic Approval History found after the final page of the Executed Contract for details.*

EXHIBIT A

SECTION I

SCOPE OF WORK

EXHIBIT A
Scope of Work

The Contractor shall provide laboratory analytical services to support the regulatory activities of the State and to meet State program needs related to the areas of sampling and analysis. The Contractor shall perform the following tasks relative to performing analytical laboratory services and submit documentation of sample analyses.

Task A. General Services and Responsibilities

1. The Contractor shall provide analytical laboratory services and reporting for the analysis of samples submitted by the Indiana Department of Environmental Management (IDEM), Office of Land Quality (OLQ).

2. The Contractor shall perform laboratory analysis on samples collected and submitted by IDEM OLQ staff for the SW-846 protocol and the SW-846 Special Analytical Services (SAS) provided in the Technical Specifications, Section III, "Protocol Analyte Lists".

3. The Contractor shall maintain a current Quality System capable of demonstrating that the data have a specified degree of reliability as described under the Technical Specifications, Section XI, "General Technical Requirements", A. Quality System.

4. The Contractor shall validate each method used and each analysis performed through this Quality System.

5. The Contractor shall participate in a Proficiency Testing (PT) sample program that incorporates all analytes in this contract, with the exception of the US EPA Office of Water Methods, available from commercial PT sample providers. Within thirty (30) days of request by the State, the Contractor shall report the results of all PT studies for which the Contractor has participated in during the term of the contract.

6. The Contractor shall maintain a sufficient number and type of functional analytical instruments as listed in the Technical Specifications, Section XI, "General Technical Requirements", B. Instrumentation.

7. The Contractor shall notify the IDEM/OLQ Quality Assurance Officer in the event of a merger, reorganization or major modification for possible audit.

8. The Contractor shall make laboratory services available 8AM to 5PM (ET) Monday through Friday except for holidays. This includes sample containers deliveries to the State and samples delivered to the laboratory. The Contractor shall make arrangements with the State for any pick-up outside of these hours.

Task B. Personnel Availability

1. The Contractor shall provide qualified personnel that meet the minimum qualifications and requirements as specified in the Technical Specifications, Section X, "Personnel Requirements".

2. The Contractor shall make personnel available for enforcement actions or litigation at no additional cost to the State.

3. The Contractor shall notify the State in writing of any personnel changes within thirty (30) days of the change.

Task C. Sample Containers, Preservatives and Holding Times

1. The Contractor shall provide USEPA-approved containers for all samples to be analyzed at the laboratory unless notified differently at the time of scheduling. The Contractor will be notified at the time of scheduling a sampling event, the number of container sets required for a sampling event plus coolers. The Contractor shall deliver these items by the requested due date. The technical specifications pertaining to the SW-846 protocol and the SW-846 Special Analytical Services (SAS) are listed in Technical Specifications, Section IV. "Sample Containers, Preservatives, and Holding Times" A. Containers and Preservations – Mandatory Specifications.

2. The Contractor shall provide sufficient containers so that one (1) matrix spike/matrix spike duplicate (MS/MSD) set can be measured per every twenty (20) samples per matrix, for all requested analytes.

3. The Contractor shall provide an aqueous trip blank, if volatile organic analysis (VOA) is requested for any sample matrix, consisting of two (2) VOA vials filled with organic-free reagent water and preserved as specified in the Technical Specifications, Section IV, Table 1. "Sample Containers, Preservatives and Holding Times".

4. The Contractor shall provide an overnight or one-day courier service to transport samples from the sampling location to the laboratory and shall be included in the cost of the analyses.

Task D. Analytical Protocols and Methods

1. The Contractor shall receive samples in sample delivery groups called cases. All sample identification numbers listed on the chain-of-custody (COC) form accompanying a particular sample delivery group will be considered one case. The Contractor shall ensure that the analyses requested on the COC form and the request provided during the sample setup on the Laboratory Request Form agree. The Laboratory Request form is in Exhibit E and serves to document the requested services. The Contractor shall contact the IDEM/OLQ Quality Assurance Officer (QAO) if there are any discrepancies.

2. The Contractor shall perform analysis by the method specified within the SW-846 protocol and SW-846 Special Analytical Services (SAS) when requested by the State at the time of analysis setup. See Technical Specifications, Section III. "Protocol Analyte Lists" for details. The Contractor shall obtain approval to substitute alternative methods or equivalent methodology from the IDEM/OLQ QAO.

3. The Contractor shall provide the State two (2) days for sample collection and delivery to the laboratory as sample holding times begin at the time of sample collection. Holding times are specified in the Technical Specifications, Section IV, Table 1. "Sample Containers, Preservatives, and Holding Times".

4. The Contractor shall perform matrix spikes and matrix spike duplicates (MS/MSD) at the frequency specified in the Technical Specifications, Section XII "Analytical and QA/QC Requirements" for the SW-846 protocol. The Contractor will not be paid for "batch" MS/MSDs that are not from the IDEM/OLQ sample delivery group.

5. The Contractor shall contact the IDEM/OLQ QAO if a case is delivered to the laboratory that has no samples designated for MS/MSD analysis. The Contractor shall not spike identified field blanks.

6. The Contractor shall not bill for all other samples analyzed for QA/QC purposes such as method blanks, calibration standards, standard reference materials, laboratory control samples, quality control check samples.

Task E. Reporting

1. The Contractor shall report results for each individual case in a separate analytical report. The report shall be provided on a compact disc (CD) and as specified in the Technical Specifications, Section V, "Reporting Requirements".

2. The Contractor shall report all analytes listed for a particular analysis type, method or method grouping as specified in the Technical Specifications, Section III. "Protocol Analyte Lists", for the SW-846 protocol and the SW-846 Special Analytical Services (SAS). The Contractor shall not modify any methods unless approved by IDEM/OLQ QAO.

3. The Contractor shall provide results via fax or e-mail when requested by the IDEM/OLQ QAO.

4. The Contractor shall retain all documentation for three (3) years after the expiration of this contract.

5. The Contractor shall provide internal chain-of-custody (COC) documentation when requested by the IDEM/OLQ QAO.

6. The Contractor shall provide all documentation as specified in the Technical Specifications, Section V. "Reporting Requirements" and Section VI." Deliverables List" for the SW-846 protocol and the SW-846 Special Analytical Services (SAS).

7. The Contractor shall analyze samples and submit a completed written analytical report in the standard turnaround time of thirty (30) days as specified in Technical Specifications, Section VII. "Turnaround Times for Delivery of Full Analytical Reports".

Task F. Invoicing and Payment Reduction

1. The Contractor shall provide itemized invoices as specified in the Technical Specifications, Section IX. "Payment for Analytical Services".

2. The Contractor shall receive payment for services when all required data and documentation has been received. Penalties shall apply for late delivery as specified in the Technical Specifications, Section IX. "Payment for Analytical Services".

3. The Contractor shall, upon written work assignment by the State, provide pricing on SW-846 Special Analytical Services (SAS) and Additional Analytical Services as detailed in the Technical Specifications, Section III. "Protocol Analyte Lists", C. and D.

EXHIBIT A

SECTION II

TECHNICAL SPECIFICATIONS
FOR
ANALYTICAL SERVICES

STATE OF INDIANA
DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
OFFICE OF LAND QUALITY

TECHNICAL SPECIFICATIONS FOR ANALYTICAL SERVICES
IDEM OFFICE OF LAND QUALITY (OLQ)

TABLE OF CONTENTS

TECHNICAL SPECIFICATIONS FOR ANALYTICAL SERVICES

I. OVERVIEW

These Technical Specifications contain detailed functional requirements for analytical services for the State of Indiana, Department of Environmental Management, Office of Land Quality (IDEM OLQ). Specifications and clarifications are provided for technical aspects of contract compliance.

All services and specifications contained herein are to be considered mandatory. If there is a conflict between the technical specifications stated in this document and the required analytical method, the criteria specified in this document take precedence.

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II. DEFINITIONS OF TERMS AND ACRONYMS

TERM	DEFINITION
AA (or AAS)	Atomic Absorption Spectroscopy
Accuracy	The closeness of agreement between an observed value and an accepted reference value.
ACS	American Chemical Society
Aliquot	A measured portion of a field sample taken for analysis.
Analytical Spike	A known quantity of target analyte added to an aliquot of sample prior to analysis but after digestion or extraction. (Also called post digestion spike)
Attachment II	RCRA Subtitle D List of Hazardous Inorganic and Organic Constituents: 40 CFR 258, Attachment II
Attachment VIII	RCRA Subtitle C Hazardous Constituents List: 40 CFR 261, Attachment VIII
Attachment IX	RCRA Subtitle C Groundwater Monitoring List: 40 CFR 264, Attachment IX.
Aqueous Sample	Samples consisting of drinking water, ground water, surface water, water-based waste, or dilute aqueous solutions.
ASTM	American Society for Testing and Materials.
Batch	A group of samples of the same matrix from the same site, not to exceed 20, and which are processed as a unit at the laboratory. If the total number of samples of a particular matrix from a site number more than 20, each group of 20 or fewer samples is treated as a separate batch.
Bias	The deviation, due to matrix effects, of the measured value of an analyte from the "true" value. In the laboratory, this is determined from the difference between the measured value of the analyte and the known spiked amount.
Blank	See Equipment Blank, Field Blank, Method Blank, and Trip Blank.
BOD	Biochemical Oxygen Demand.
Breakdown	A measure of the decomposition of certain analytes into by-products. For purposes of this RFP, it specifically refers to decomposition of DDT and Endrin during gas chromatographic analysis.
BNA	Base-Neutral-Acid Extractables. (class of semivolatile organic compounds)
Case	A finite number of samples collected over a given time period from a particular site. Also referred to as a sample set or sample delivery group.
CCC (SW-846)	Calibration Check Compound (Used in GC/MS analysis of volatile and semivolatile organic compounds)
CCC (EPA Water Analysis Methods)	Continuing Calibration Check (Used in GC/ECD analysis of derivatized semivolatile organic acid compounds)
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980, as amended. (Superfund)
CF	Calibration Factor
Chain of Custody	In a chemical sampling situation the maintenance of the sampled material by providing documentation of the control, transfer, and analysis of the sample

TERM	DEFINITION
CLP	Contract Laboratory Program – (laboratory specifications, analytical methods, and QA/QC protocols commonly utilized for Superfund activities)
COC	Chemical of Concern: Contaminant (target analyte) at a site undergoing remediation or closure.
COD	Chemical Oxygen Demand
Co-located	Separate samples collected from the same location or source, as closely as possible to the same point in space and time. They are stored in separate containers and analyzed separately to document the variability of the sampling process and matrix effect on recoveries (for groundwater, two samples gathered in sequence from separate bailer quantities from the same well).
Control Sample	A QC sample introduced into a data collection process to monitor the performance of the system
CVAA	Cold Vapor Atomic Absorption
D001	Ignitability - as defined in 40 CFR 261.21 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic)
D002	Corrosivity - as defined in 40 CFR 261.22 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic)
D003	Reactivity - as defined in 40 CFR 261.23 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic)
Data Quality Objective Process	Strategic planning approach used to prepare for a data collection activity. It provides a systematic procedure for defining the criteria that a data collection design should satisfy, including the tolerable level of decision error. (See Guidance for the Data Quality Objectives Process: EPA/240/B-06/001 February 2006.)
Data Quality Objectives (DQOs)	Qualitative and quantitative statements that clarify the overall objective of a data collection activity by defining the criteria that the project should satisfy. (This is distinct from quality control measurements such as precision and bias. It also does not refer to levels of data documentation or volume of "data deliverables.")
Dissolved Metals	Digestion and analysis for metals of a filtered aqueous sample.
Duplicate	See Field Duplicate, Laboratory Duplicate, Matrix Duplicate, and Matrix Spike Duplicate.
Dry Weight	The mass of a soil sample or an object when dried
ECD	Electron Capture Detector
Eh	Oxidation-Reduction Potential
Elutriate Test	See USACE Modified Elutriate Test.
Enforcement Level Reporting	Project Deliverables plus raw data plus internal laboratory chain-of-custody and other documentation specified at the time of the analytical request.
EPA	United States Environmental Protection Agency
EPH	Extractable Petroleum Hydrocarbons
Equipment Blank	A sample of analyte-free reagent water that has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling at the next location. This blank is used to document whether decontamination is adequate.

TERM	DEFINITION
Estimated Quantitation Limit (EQL)	Estimated Quantitation Limit (Used in SW-846: Test Methods for Evaluation Solid Waste – Formerly called "Practical Quantitation Limit (PQL)."") The lowest concentration that can be reliably achieved in a given matrix within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL for aqueous samples and low concentration soils. The EQL for concentrated wastes, high concentration soils, and samples requiring cleanup will be the MDL multiplied by a higher factor, frequently 500 or 670. These factors are indicated in the Tables at the end of applicable SW-846 methods.
FID	Flame Ionization Detector
Field Blank	Analyte-free reagent water taken to the sampling site, then analyzed by the laboratory for the same parameters as the investigative samples to check for procedural contamination of samples. Also see Trip Blank, Equipment Blank, etc.
Field Duplicate	Samples collected from the same location or source. The sample is then split in the field or lab and stored in separate containers. They are analyzed by the same procedures separately to document the variability of the sampling process and matrix effect on recoveries (for groundwater two samples gathered from the same bailer quantity from the same well).
Field Reagent Blank (FRB)	EPA Water Analysis Methods: An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. (Called Trip Blank in SW-846 and CLP.)
Fraction of Organic Carbon (f_{oc})	The fraction of organic carbon in the soils is the total mass of organic carbon divided by a unit of mass of soils.
Full QA/QC	Level IV Deliverables
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption Spectroscopy
Hazardous Constituent	Compound or element designated as a constituent of hazardous waste in the RCRA Subtitle C program and listed in 40 CFR 261, Attachment VIII.
Hazardous Substance	Compound or element listed in CERCLA (Superfund) Hazardous Substance List, 40 CFR 302.4
Hazardous Waste	Material (solid waste) listed as a hazardous waste by the RCRA Subtitle C program by meeting any of the criteria stated in 40 CFR 261.11.
Holding Time	Elapsed time, expressed in days, from the date of sampling until the date of analysis
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP (or ICAP)	Inductively Coupled Plasma – Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
ICS -	Interference Check Sample
IDEM	Indiana Department of Environmental Management
IR	Infrared Spectroscopy

TERM	DEFINITION
Laboratory Control Sample (LCS)	A known matrix or laboratory blank spiked with known quantities of the target analytes used to document laboratory performance. (Also known as Laboratory Fortified Blank, Ongoing Precision and Recovery Standard, or "DI Spike.")
Laboratory Fortified Blank (LFB)	Laboratory Control Sample (EPA Water Analysis Methods)
Laboratory Fortified Sample Matrix (LFM)	Matrix Spike
Laboratory Project Manager (LPM)	Laboratory contact person for IDEM staff.
Laboratory Reagent Blank	Method Blank. (EPA Water Analysis Methods)
LCMRL	Lowest Concentration Minimum Reporting Level
Level IV Deliverables	See Project Deliverables with Raw Data
Matrix Spike	An aliquot of sample spiked with a known concentration of all target analytes. The spiking occurs prior to sample preparation and analysis. The matrix spike is used to document the bias of a method for the spiked analytes in a given sample matrix.
Matrix Spike Duplicates	Laboratory duplicates (split samples) spiked with identical concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. Matrix spike duplicates are used to document the precision and bias of a method for the spike analytes in a given sample matrix.
Maximum Contaminant Level (MCL)	Maximum concentration of a contaminant allowed in drinking water systems by the National Primary and Secondary Drinking Water regulations at 40 CFR 141. Reporting limits required by this Request for Proposal (RFP) have been set to meet primary Maximum Contaminant Levels
Method Blank	An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing and is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. (Also known as preparation blank, reagent blank, laboratory reagent blank, and laboratory blank.)
Method Detection Limit (MDL)	The minimum concentration of a substance 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 type containing the analyte. The procedure for determining the MDL is found at 40 CFR 136, Attachment B.
MS	Matrix Spike Mass Spectrometry or Mass Spectrometer
MS/SCAN	The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.
MS/SIM	The GC is coupled to a MS programmed to acquire data for only specified ions and to disregard all others, selected ion monitoring (SIM). This is performed using SIM coupled to retention time discriminators. The MS/SIM analysis provides quantitative results for selected constituents of the sample as programmed by the user.

TERM	DEFINITION
MSA	Method of Standard Additions as described in SW-846 Method 7000A (Update III), SW-846 Method 7010 (Update IVA), or various EPA Water Analysis Methods, such as Method 200.7, Revision 5.0, or Method 1639.
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSD	Matrix Spike Duplicate
NA or N/A	Not applicable
Neutral Leaching Method (Neutral Leachate)	The leaching procedure extraction as specified for SW-846 Method 1311, the Toxic Characteristic Leaching Procedure (TCLP), except using reagent water instead of acidic extraction fluids 1 or 2. (Used for characterizing certain types of non-hazardous waste. See 329 IAC 10-9.)
NIST	National Institute of Standards Testing
Non-aqueous samples	Samples consisting of soil, sediment, sludge, oil, solid waste, or highly concentrated water-based waste.
Notice of Data Availability (NODA)	Publication in the Federal Register used to officially release finalized updates to SW-846. Formerly, SW-846 updates were formally promulgated. Replacement of promulgation with NODA began with Update IVA on May 8, 1998.
OLQ	Office of Land Quality
Ongoing Precision and Recovery Standard (OPR)	Laboratory Control Sample. (EPA Water Analysis Methods)
Organic-Free Reagent Water	For analysis of organic analytes: water prepared so that interferents or contaminants are observed at the method detection limit of the compounds of interest. Methods of preparation include passing tap water through a carbon filter containing about one pound of activated carbon or using a water purification system to generate organic-free deionized water.
OSWER	USEPA Office of Solid Waste and Emergency Response
PAH	Polynuclear Aromatic Hydrocarbon(s). (Also called PNAs.)
PCB	Polychlorinated Biphenyl Compound(s)
Petroleum Analysis	Total petroleum hydrocarbons determination: Identification of petroleum fuel contamination (gasoline, kerosene, diesel) using Gas Chromatography with a Flame Ionization Detector or a Photoionization Detector. Also, analysis of heavy oils using an Infrared Spectrophotometer.
PFOA	Perfluorooctanic Acid
PFOS	Perfluorooctanesulfonic acid
PID	Photoionization Detector
ppb	Parts per billion (usually µg/kg (solids) or µg/L (aqueous))
ppm	Parts per million (usually mg/kg (solids) or mg/L (aqueous))
ppq	Parts per quadrillion (usually picograms/liter (pg/L))
ppt	Parts per trillion (usually nanograms/liter (ng/L))
PQL	Practical Quantatation Limit
PNA	Polynuclear Aromatic Hydrocarbon(s). (Usually called PAHs.)
Precision	The agreement among a set of replicate measurements without consideration of the "true" or accurate value. The variability between measurements of the same material for the same analyte.
Preparation Blank	Method blank
Project	Single or multiple data collection activities (or remediation activities) that are related through the same planning sequence.

TERM	DEFINITION
Project Deliverables	All laboratory results listed in the Deliverables List for the analysis requested except raw data. See Deliverables List in Attachment I, Section VI.
Project Deliverables with Raw Data	All laboratory results listed in the Deliverables List for the analysis requested including raw data for field samples, field and laboratory QC samples. See Deliverables List in Section VI.
Protocol	A broad category of analytical methods for which the source is one or more USEPA methods manuals developed for a specific regulatory program. For this RFP the applicable Protocols are: SW-846 - from the RCRA program in the USEPA Office of Solid Waste and Emergency Response (OSWER) EPA Drinking Water Methods from the USEPA Office of Ground Water and Drinking Water in the Office of Water
Purgeable Compounds	Volatile Organic Compounds
QAO	Quality Assurance Officer. IDEM OLQ senior chemist responsible for all QA/QC and technical aspects of the laboratory services contract and managing the OLQ sampling and analysis program.
QA/QC	Quality Assurance/Quality Control
QCS	Quality Control Sample or Quality Control Check Sample (EPA Water Analysis Methods)
Quality Assurance (QA)	The management procedures and controls used to ensure data quality through the sampling and analysis process.
Quality Assurance Project Plan (QAPP)	An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives (DQOs) for a specific data collection activity.
Quality Control (QC)	The day-to-day operational measures used in the field during sampling and in the laboratory during analysis to ensure data quality.
Quality Control Check Sample (Quality Control Sample)	A sample containing all or a subset of the target analytes at known concentrations. It is used to check laboratory performance with test materials prepared external to the normal preparation process. The QCS is obtained from an external source or prepared with standards from a different source than the calibration standards.
Raw Data	All laboratory-generated documentation contributing to the final reported results. Includes initial calibration records, daily and continuing calibration records, calibration curves, bench sheets, lab worksheets, strip chart recordings, sample preparation records, run lists, record of dilutions, instrument numerical printouts, instrument peak printouts, chromatograms, second column confirmations, tuning criteria and results, spectra, and quantitation reports. (See Deliverables List.)
Remediation Closure Guidance (RCG)	Describes selected approaches to investigation and risk-based closure of contaminated or potentially contaminated sites.
RCRA	The Resource Conservation and Recovery Act of 1976, as amended. RCRA Subtitle C addresses hazardous waste. RCRA Subtitle D addresses non-hazardous solid waste.
Reagent Blank	Method Blank
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

TERM	DEFINITION
Reagent Water	Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.
Reference Material	A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.
Relative Percent Difference (RPD)	An estimate of precision used when only two samples are available. It is calculated by subtracting one replicate measurement from the other, dividing the difference by the mean of the two measurements, and multiplying by 100.
Relative Standard Deviation (RSD)	An estimate of precision calculated by multiplying the standard deviation of replicate measurements by 100 and dividing by the mean. Also known as the coefficient of variation (CV).
Replicate	One sample split into two or more samples in the laboratory and analyzed separately with identical procedures.
RF	Response Factor
RISC	Risk Integrated System of Closure. One version of IDEM OLQ guidance for a risk assessment-based approach to remediation and closure.
RL	Reporting Limit. The reporting limits listed in the Protocol Analyte Lists are required target quantitation limits, except for sample matrices in which they cannot technically be attained (dilutions, matrix interference, etc.)
RRF	Relative Response Factor
RT	Retention Time
SAS	Special Analytical Services - Non-routine analyses
S.C.	Specific Conductance
SIM	See MS/SIM
% Solids	Total percent solids, as determined in a 103°C to 105°C oven.
SOP(s)	Standard Operating Procedure(s)
SPCC	System Performance Check Compound (Used in SW-846 GC/MS analysis of volatile and semivolatile organic compounds)
Split Samples	Aliquots of sample taken from the same container and analyzed independently, usually after mixing or compositing, and used to document precision.
SPLP	Synthetic Precipitation Leaching Procedure: SW-846 Method 1312.
Standard Addition	The practice of adding a known amount of an analyte to a sample immediately prior to analysis and typically used to evaluate interferences.
Surrogate (Surrogate Standard)	An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. (Called System Monitoring Compound in CLP volatile analysis.)
SVOA	Semivolatile Organics Analysis.
SVOC	Semivolatile Organic Compound(s): Organic compounds amenable to analysis by extraction with solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.
SW-846	Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods: SW-846, Third Edition Nov. 1986, and all subsequent updates or amendments. (Currently includes Final Updates I, II, IIA, IIB, III, IVA and IVB.)
System Monitoring Compound	Surrogate. (CLP Statement of Work for Organic Analysis)

TERM	DEFINITION
TCLP	Toxic Characteristic Leaching Procedure: SW-846 Method 1311. (Used to characterize RCRA Subtitle C waste codes D004 – D043.)
TIC	Tentatively Identified Compound. In GC/MS analysis, compounds that are not included in the calibration standard mixture(s) but are identified by the mass spectral library with a reasonable degree of certainty.
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
Total Metals	Digestion and analysis for metals of an unfiltered aqueous sample.
TOX	Total Organic Halides
TPH	Total Petroleum Hydrocarbons. (See Petroleum Analysis)
Trip Blank	A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples but may be used for all analytes of interest. (Called Field Reagent Blank in EPA Water Analysis methods).
TRPH	Total Recoverable Petroleum Hydrocarbons. Extraction and analysis of non-volatile petroleum fractions. Not applicable to gasoline-range petroleum samples.
USACE	United States Army Corps of Engineers
USACE Modified Elutriate Test	USACE-developed method to model quality of effluent discharged from confined dredged material disposal areas. The test uses surface water and sediment from the site under investigation, and the resulting effluent is analyzed for water quality. (The test is included in the document, "Interim Guidance for Predicting Quality of Effluent Discharged from Confined Dredged Material Disposal Areas--Test Procedures" (June 1985), available from the USACE web site as document EEDP-04-2 at: http://www.wes.army.mil)
USEPA	United States Environmental Protection Agency
VOA	Volatile Organics Analysis
VOCs	Volatile Organic Compounds: Organic compounds amenable to the purge-and-trap procedure. (Also called purgeable compounds.)
VPH	Volatile Petroleum Hydrocarbons
Wet Weight	The amount of the chemical found in subsequent analysis is expressed as the weight of chemical divided by the total weight, including any water present

III. PROTOCOL ANALYTE LISTS

INTRODUCTION

Analyte and Method Requirements

The Contractor shall adhere to the method requirements on the following pages which include the Analyte Lists for each of the Protocols.

SW-846 and Drinking Water. Each Protocol consists of several Analyte Groups. After the required Analyte Groups within each Protocol, lists of Special Analytical Services (SAS) Analyte Groups are provided. A Contractor is not required to have the capability to perform the SAS analyses in order to bid on a particular protocol.

When a particular Analyte Group is requested for analysis, all analytes listed in that Group must be run and reported unless the Contractor is instructed to omit certain analytes. For Analyte Groups that have one Required Method listed, analysis must be performed by that method. For Analyte Groups in which several Acceptable Methods are listed, any of the listed methods appropriate to the sample matrix and required Reporting Limit (RL) may be selected.

The Reporting Limits (RLs) listed for each analyte in a particular matrix must be met unless it is technically impossible to do so. In such cases, a sufficient technical explanation must be provided in the Case Narrative accompanying the data package.

When method numbers listed in the Acceptable Methods column are italicized (e.g. *8015C*), it means that the compound of interest is not specifically listed in that method's analyte list, but that the compound is amenable to analysis by that method.

A. SW-846 Protocol

Unless otherwise noted in the Analyte List, all methods listed for the SW-846 Protocol are from *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods,* Third edition and update 1 (August 31, 1993) and updates:

1. **Updates**: **II** (January 1995), **IIA** (January 1994), **IIB** (April 1995), **III** (June 1997), **IIIA** (May 1999), **IIIB** (June 2005), **IVA** and **IVB** (January 2008), **Methods Innovation Rule** (MIR) (June 2005), **IV** (January 2008), and Update V (August 2015).
2. Also referenced in the SW-846 Protocol General Chemistry Analyte Lists are:
   - **a.** *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100, August 1993,
   - **b.** *Methods for Chemical Analysis of Water and Wastes*, EPA/600/R-79/020, revised March 1983, and
   - **c.** *Standard Methods for the Examination of Water and Wastewater,* 19th edition, 1995.

Contractors awarded contracts for the SW-846 Protocol shall incorporate the most recent method revisions and newly developed methods from the SW-846 Updates listed above and from subsequent Updates as they become available. Information regarding SW-846 Updates and downloads of all SW-846 methods can be obtained from the USEPA website at https://www.epa.gov/hw-sw846/sw-846-compendium

B. EPA Drinking Water Protocol

The EPA Office of Water Method Compendia from which the specified methods are drawn is indicated in the footnotes at the bottom of each page of the Drinking Water Protocol Analyte Lists. The compendia referenced include:

1. *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA/600/4-90/020 - July 1999; Supplement II, August 1992: Supplement III, August 1995.
2. *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater, Revision A* EPA-821-B-98-016 - July 1998;
3. *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater –Volume I* - EPA-821-R-93-010-A August 1993, Revision 1;
4. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Anaylsis and Sampling Procedures; Final Rule, (2012).

Contractors awarded contracts for the Drinking Water Protocol shall keep up-to-date with method revisions, technical notes and newly developed methods as they become available. Information regarding Office of Water analytical methodology, including sources for obtaining the methods, is available on the EPA website, https://www.epa.gov/dwanalyticalmethods/approved-drinking-water-analytical-methods. Some, but not all, drinking water methods are available for download from links to this site.

C. Special Analytical Services (SAS)

The SW-846 and Drinking Water Protocols include potential SAS Analyte Lists with suggested analytical methodology and quantitation limits. The State may request additional analyses not listed. Any SAS requested will be specified at the time of sampling set up as to analytical methodology and quantitation limits required. Contractors are not required to bid on or perform SAS analyses.

D. Additional Analytical Services

The Additional Analytical Services includes analysis of air samples using TO-15, analysis of soil and sediment for Fraction Organic Carbon (foc), analysis of aqueous, soil, and sediment samples for Total Petroleum Hydrocarbons (TPH), and analysis of aqueous samples for PFOA and PFOS. Specifications will be made at the time of sampling set up as to analytical methodology and quantitation limits required.

SW-846 PROTOCOL

SW-846 Metals

Metals: Acceptable Sample Preparation Methods			
Method No.	**Sample Matrix**	**Procedure**	**Comments**
3005A	Aqueous (ground and surface water only)	Acid Digestion for ICP analysis	*Total Recoverable* and *Dissolved* Metals
3010A	Aqueous (including leaching procedure extracts)	Acid Digestion for ICP analysis	Total Metals
3020A	Aqueous (including leaching procedure extracts	Acid Digestion for GFAA analysis	Total Metals
3031	Oils and Oily Wastes	Acid Digestion for ICP analysis	Total Metals
3040A	Crude Oil and Virgin Oils, Greases and Waxes	Solvent Dissolution for ICP	Total Metals
3050B	Soils, Sediments, and Sludges	Acid Digestion for ICP and GFAA	Total *Available* Metals
3051A	Soils, Sediments, and Sludges	Microwave-Assisted Acid Digestion for ICP and GFAA	Total *Available* Metals
3052	Siliceous and Organically-Based Matrices (i.e., all except aqueous)	Microwave Assisted Acid Digestion	Total Metals
3060A	Soils, Sediments, and Sludges	**Alkaline** Digestion for **Cr^{6+}**	Hexavalent chromium only

TOTAL METALS – Group A: RCRA Metals										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Arsenic[1]	7440-38-2	10.0	µg/L	1.0	mg/kg	6010C[1]	6020A	7010[2a]	7062	7063
Barium	7440-39-3	50.	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Cadmium[1]	7440-43-9	05.0	µg/L	0.50	mg/kg	6010C[1]	6020A	7010[2a]		
Chromium (all forms)	7440-47-3	10.	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Lead[1]	7439-92-1	15.	µg/L	5.0	mg/kg	6010C[1]	6020A	7010[2a]		
Mercury	7439-97-6	0.20	µg/L	0.20	mg/kg	7470A	7471B	7472	7473	6020A[3]
Selenium[1]	7782-49-2	50.	µg/L	1.0	mg/kg	6010C[1]	6020A	7010[2a]	7741A	7742
Silver	7440-22-4	10.	µg/L	3.0	mg/kg	6010C	6020A	7010[2a]		

TOTAL METALS – Group B: Additional CERCLA Metals and non-RCRA Metals with Primary MCLs										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Antimony[1]	7440-36-0	6.0	µg/L	3.0	mg/kg	6010C[1]	6020A	7010[2a]	7062	
Beryllium	7440-41-7	4.0	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Cobalt	7440-48-4	5.0	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Copper	7440-50-8	5.0	µg/L	2.5	mg/kg	6010C	6020A	7010[2a]		
Nickel	7440-02-0	10.	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Thallium[1]	7440-28-0	2.0	µg/L	1.0	mg/kg	6010C[1]	6020A	7010[2a]		
Vanadium	7440-62-2	10.	µg/L	5.0	mg/kg	6010C	6020A	7010[2a]		
Zinc	7440-66-6	20.	µg/L	2.0	mg/kg	6010C	6020A	7010[2a]		

[1]In order to achieve detection limits below the required reporting limit and MCL, Method 6010C will not be allowed for these analytes in aqueous samples unless the instrument is modified for trace analysis (e.g., with axially-oriented torch).

[2a]Method 7010 is found in SW-846 Update IVA, which appeared as a NODA in the *Federal Register* on May 8, 1998 (63 *FR* 25430). It incorporates graphite furnace atomic absorption (GFAA) analysis for all non-derivatized metals into one method. It replaces Methods 7041, 7060A, 7131A, 7191, 7740, etc.

[3]If Method 6020A is proposed for mercury analysis, Method Detection Limit studies must be submitted that demonstrate required reporting limits can be achieved in matrix(es) proposed.

TOTAL METALS – Group C: Indicator and Water Quality Metals										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Aluminum	7429-90-5	200.	µg/L	100.	mg/kg	6010C	6020A			
Calcium	7440-70-2	500.	µg/L	100.	mg/kg	6010C	6020A			
Iron	7439-89-6	100.	µg/L	100.	mg/kg	6010C	6020A	7010[2b]		
Magnesium	7439-95-4	100.	µg/L	10.	mg/kg	6010C	6020A			
Manganese	7439-96-5	5.0	µg/L	2.0	mg/kg	6010C	6020A	7010[2b]		
Potassium	7440-09-7	100.	µg/L	100.	mg/kg	6010C	6020A			
Sodium	7440-23-5	500.	µg/L	100.	mg/kg	6010C	6020A			

TOTAL METALS - Group D: Hexavalent Chromium										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Cr^{6+} Aqueous samples[4]	18540-29-9	0.3	µg/L	N/A	N/A	7195	7196A	7197	7198	7199
Cr^{6+}-Non-aqueous[5]	18540-29-9	N/A	N/A	20.	mg/kg	**Digestion by 3060A[4]**, then: 7196A or 7199				

TOTAL METALS - Group E: Metals, other										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Suggested Methods**				
		RL	**units**	**RL**	**units**					
Boron	7440-42-8	50.	µg/L	100.	mg/kg	6010C				
Lithium	7439-93-2	50.	µg/L	1.0	mg/kg	6010C				
Molybdenum	7439-98-7	50.	µg/L	1.0	mg/kg	6010C				
Strontium	7440-24-6	50.	µg/L	5.0	mg/kg	6010C				
Tin	7440-31-5	200.	µg/L	2.0	mg/kg	6010C				
Titanium	7440-32-6	50.	µg/L	2.0	mg/kg	6010C				

[2b]Method 7010 is found in SW-846 Update IVA, which appeared as a NODA in the *Federal Register* on May 8, 1998 (63 *FR* 25430). It incorporates graphite furnace atomic absorption (GFAA) analysis for all non-derivatized metals into one method. It replaces Methods 7041, 7060A, 7131A, 7191, 7740, etc.

[4]If aqueous samples are preserved with 1 mL 50% NaOH solution (per 125 mL sample), holding time for hexavalent chromium is increased from 24 hours to 30 days. See EPA Office of Water Method 1669, sections 2.10, 7.3, and 8.4.

[5]SW-846 Method 3060A (alkaline digestion) is **required** for digestion of soils, sludges, sediments, and solid wastes that will be analyzed for hexavalent chromium. The State will not pay for hexavalent chromium analyses if acid digestions are performed.This Rl does anot apply to all acceptable methods listed, and may only be needed for select drinking water metho samples.

SW-846 PROTOCOL
General Chemistry

GENERAL CHEMISTRY – Group A: Cyanide and Sulfide										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Total Cyanide	57-12-5	0.0050	mg/L	1.0	mg/kg	9012A,	9010B (or 9013) plus 9014 or 9213			
Free Cyanide[6]	57-12-5	0.2	mg/L	1.0	mg/kg	9014	9213			
Amenable Cyanide	57-12-5	0.0050	mg/L	1.0	mg/kg	9012A,	9010B (or 9013) plus 9014 or 9213			
Total Sulfide	18496-25-8	1.0	mg/L	25.	mg/kg	9031,	9030B plus 9034 or 9215, 9031/9215			

GENERAL CHEMISTRY – Group B: Solid Waste Standard List										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Ammonia-N[6]	14798-03-9	0.10	mg/L	N/A	N/A	350.1	1689	1690		
Chloride	16887-00-6	2.0	mg/L	20.	mg/kg	9056A	9212	9250	9251	9253
Nitrate-Nitrite[6]	14797-55-8/ 14797-65-0	0.010	mg/L	1.	mg/kg	9056A	353.2	1685	1686	
pH	N/A	± 0.1	units	± 0.1	units	9040B	9045C	(9041A-only if necessary)		
Specific Conductance	N/A	5.0	µS/cm	N/A	N/A	9050A				
Sulfate	14808-79-8	5.0	mg/L	100.	mg/kg	9056A	9035	9036	9038	
Residue, Filterable[6] (TDS)	N/A	20.	mg/L	N/A	N/A	2540C				
Residue, Total[6] (TS)	N/A	20.	mg/L	N/A	N/A	2540B				

GENERAL CHEMISTRY – Group C: Additional Ground Water Indicator Parameters										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Alkalinity[7,8a] (as $CaCO_3$)	N/A	5.0	mg/L	N/A	N/A	310.2	2320	2320B		
Bicarbonate[8a] (as $CaCO_3$)	71-52-3	10.	mg/L	N/A	N/A	2320				
Carbonate[8a] (as $CaCO_3$)	3812-32-6	10.	mg/L	N/A	N/A	2320				
Eh[8a] (Oxidation-Reduction Potential)	N/A	0	Volts	N/A	N/A	2580				
Fluoride[7]	16984-48-8	0.10	mg/L	N/A	N/A	9056A	9214			
Hardness[7] (as $CaCO_3$)	N/A	1.0	mg/L	N/A	N/A	130.1				
Oxygen, Total Dissolved[7]	7782-44-7	1.0	mg/L	N/A	N/A	4500·O·G	4500·O·C			
Phosphorus, Total[7] (as PO_4^{3-})	14265-44-2	0.5	mg/L	130	mg/kg	9056A				
Turbidity[7]	N/A	0.5	NTU	N/A	N/A	180.1				

[6]Determination of **free cyanide** is for Risk Integrated System of Closure (**RISC**)/Remediation Closure Guide (**RCG**).

[7]The reference for method 180.1, Rev. 2.0; 350.1, Rev 2.0, 351.2, Rev. 2.0; 353.2, Rev. 2.0, 365.1, Rev. 2.0; and 420.4, Rev. 1.0 is *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100, August 1993. The reference for methods130.1, 310.2, 365.3, and 365.4 is *Methods for Chemical Analysis of Water and Wastes*, EPA/600/R-79/020, revised March 1983. Methods 1685, 1686, 1689 and 1690 are USEPA Office of Water "stand alone methods." (Methods 9056A, and 9214 are SW-846 methods.)

[8a]The reference for methods 2320, 2540B, 2540C, 2580, 4500·O·C, and 4500·O·G is *Standard Methods for the Examination of Water and Wastewater*, 20th edition, 1998.

SW-846 PROTOCOL - General Chemistry

GENERAL CHEMISTRY – Group D: Non-Specific Organic Determinations										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Oil and Grease (HEM)	N/A	5.0	mg/L	10.	mg/kg	9070	9071B			
Total Organic Carbon (TOC)	N/A	3.0	mg/L	500.	mg/kg	9060				
Total Organic Halides (TOX)	N/A	0.050	mg/L	500.	mg/kg	9020B	9022			
Phenolics, Total[8b]	64743-03-9	0.010	mg/L	0. 5	mg/kg	9065	9066	9067	420.1-420.4	
Surfactants (MBAS)[8b]	N/A	0.1	mg/L	N/A	N/A	425.1				
Chemical Oxygen Demand (COD)[8b,9]	N/A	5.0	mg/L	N/A	mg/kg	410.3	410.4	5220		
Biochemical Oxygen Demand (total) (BOD_5)[8b,9]	N/A	5.0	mg/L	N/A	N/A	5210B				
Carbonaceous Biochemical Oxygen Demand ($CBOD_5$)[9]	N/A	5.0	mg/L	N/A	N/A	5210B				

GENERAL CHEMISTRY – Group E-1: Feedlot Runoff and Manure Spill Characterization								
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**		
		RL	**units**	**RL**	**units**			
E. coli[9]	N/A	1.	MPN/100mL or CFU/100mL	10.	MPN/g or CFU/g	9222G 9223B		**Must provide count**
Fecal Coliform[8b,9]	N/A	1.		10.		1680	9222D	**Must provide count**
Total Coliform[9]	N/A	1.		10.		9132	9222B	**Must provide count**

GENERAL CHEMISTRY – Group E-2: Feedlot Runoff and Manure Spill Characterization										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
Nitrogen-Ammonia[8b]	14798-03-9	0.10	mg/L	N/A	N/A	350.1	1689	1690		
Nitrogen-Nitrate[8b]	14797-55-8	0.20	mg/L	1.0	mg/kg	9056A	9210	352.1	1686	300.1
Nitrogen-Nitrite[8b]	14797-65-0	0.20	mg/L	1.0	mg/kg	9056A	354.1	300.0	300.1	
Nitrogen-Total Kjeldahl[8b] (TKN)	7727-37-9	0.50	mg/L	130.	mg/kg	351.1	351.2	1687 1688		
Phosphorus, Total[8b]	7723-14-0	0.5	mg/L	10.	mg/kg	7580	6010C	365.1 365.3 365.4		
Residue, Non-Filterable[8b,9] (TSS)	N/A	5.0	mg/L	N/A	N/A	2540D				

GENERAL CHEMISTRY – Group F General Chemistry – Other/Miscellaneous: Physical Testing										
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Acceptable Methods**				
		RL	**units**	**RL**	**units**					
% Solids[8b]	N/A	N/A	N/A	1.	%	1684				
Total Solids/Residue, total[8b]	N/A	10.	mg/L	20	mg/kg	1684				
Volatile Solids/Residue, volatile[8b]	N/A	10.	mg/L	20	mg/kg	1684	160.4			
Paint Filter Test	N/A	N/A	N/A	N/A	N/A	9095A				

[8b] The reference for method 350.1, Rev 2.0, 351.2, Rev. 2.0; 353.2, Rev. 2.0, 365.1, Rev. 2.0; and 420.4, Rev. 1.0 is *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100, August 1993. The reference for methods 160.4, 410.3, 420.1, 420.2, and 420.3 is *Methods for Chemical Analysis of Water and Wastes*, EPA/600/R-79/020, revised March 1983. Methods 300.0, 300.1, 1680, 1684, 1686, 1687, 1688, 1689 and 1690 are USEPA Office of Water "stand alone methods." (Methods 6010C, 7580, 9056A, 9065, 9066, 9067and 9210 are SW-846 methods.)

[9] The reference for methods 2540D, 5210B, 5220, 9221E, 9222B, 9222D, 9222G, and 9223B is *Standard Methods for the Examination of Water and Wastewater*, 19th edition, 1995. (Method 9132 is an SW-846 method.)

SW-846 PROTOCOL
Volatile Organic Analysis (VOA)

VOA: Acceptable Sample Preparation and Introduction Methods			
Method No.	**Sample Matrix**	**Procedure**	**Comments**
5000	All	General Guidance	**Required.** Analyst **must** be familiar with.
3585	Oily Wastes	Solvent Dilution	Introduction to GC by direct injection
5021	Soils, Sediments, Solid Waste	Automated Headspace	---
5030B	Aqueous	Purge-and-Trap	---
5031	All	Azeotropic Distillation	Alternative sample introduction technique for water-soluble "poor purgers"
5032	Biota, Tissue, Oils	Vacuum Distillation	---
5035-A	Soils, Sediments, Solid Waste	Closed System Purge-and Trap (Modified)	
5035A	Soils, Sediments, Solid Waste	Heated Purge-and-Trap	Solid samples that have not been collected and stored properly for performance of 5021 should be analyzed using the soil procedure in Method 5030A in SW-846 Final Update I, July 1992.

VOA – Group A: OLQ Standard Volatiles List[10b]							
Analyte	**CAS Number**	**Aqueous** *25 mL purge*		**Soil/Sediment** (low concentration)		**Acceptable Methods**[10a]	
		RL	**units**	**RL**	**units**		
Acetone	67-64-1	20.	µg/L	50.	µg/kg	**8260B**	8015C
Acrolein	107-02-6	**20**	µg/L	40	µg/kg	**8260B**	8015C
Benzene	71-43-2	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
Bromodichloromethane	75-27-4	1.	µg/L	5.	µg/kg	**8260B**	8021B
Bromoform	75-25-2	1.	µg/L	5.	µg/kg	**8260B**	8021B
Bromomethane	74-83-9	2.	µg/L	10.	µg/kg	**8260B**	8021B
n-Butanol	71-36-3	1000	µg/L	1000	µg/kg	**8260B**	8015C
2-Butanone (MEK)	78-93-3	20.	µg/L	50.	µg/kg	**8260B**	8015C
n-Butylbenzene	104-51-8	1.	µg/L	5.	µg/kg	**8260B**	
sec-Butylbenzene	135-98-8	1.	µg/L	5.	µg/kg	**8260B**	
tert-Butylbenzene	98-06-6	1.	µg/L	5.	µg/kg	**8260B**	
Carbon disulfide	75-15-0	20.	µg/L	50.	µg/kg	**8260B**	
Carbon tetrachloride	56-23-5	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
Chlorobenzene	108-90-7	1.	µg/L	5.	µg/kg	**8260B**	8021B
Chloroethane	75-00-3	2.	µg/L	10.	µg/kg	**8260B**	8021B
Chloroform	67-66-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
Chloromethane	74-87-3	2.	µg/L	10.	µg/kg	**8260B**	8021B
1,2-Dibromoethane (EDB)	106-93-4	1.	µg/L	5.	µg/kg	**8260B**	8021B
Dibromomethane	74-95-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,2-Dichlorobenzene	95-50-1	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,3-Dichlorobenzene	541-73-1	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,4-Dichlorobenzene	106-46-7	1.	µg/L	5.	µg/kg	**8260B**	8021B
Dichlorodifluoromethane	75-71-8	2	µg/L	10.	µg/kg	**8260B**	8021B
1,1-Dichloroethane	75-34-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,2-Dichloroethane	107-06-2	**1**.	µg/L	5.	µg/kg	**8260B**	8021B
1,1-Dichloroethene	75-35-4	1.	µg/L	5.	µg/kg	**8260B**	8021B
cis-1,2-Dichloroethene	156-59-2	1.	µg/L	5.	µg/kg	**8260B**	8021B
trans-1,2-Dichloroethene	156-60-5	1.	µg/L	5.	µg/kg	**8260B**	8021B

[10a] **8260B** is the preferred Method for **SW-846 Protocol VOA Group A**, the full OLQ Standard Volatiles List.
[10b] Reporting limits that are **bolded** are important in order to meet or come as close as possible to RISC/RCG levels.

VOA – Group A: OLQ Standard Volatiles List, continued[10b]							
Analyte	**CAS Number**	**Aqueous** *25 mL purge*		**Soil/Sediment** (low concentration)		**Acceptable Methods**[10a]	
		RL	**units**	**RL**	**units**		
1,2-Dichloropropane	78-87-5	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
cis-1,3-Dichloropropene	10061-01-5	1.	µg/L	5.	µg/kg	**8260B**	8021B
trans-1,3-Dichloropropene	10061-02-6	1.	µg/L	5.	µg/kg	**8260B**	8021B
Ethylbenzene	100-41-4	1.	µg/L	5.	µg/kg	**8260B**	8021B
2-Hexanone (MBK)	591-78-6	20.	µg/L	50.	µg/kg	**8260B**	*8015C*
Isopropylbenzene	98-82-8	1.	µg/L	5.	µg/kg	**8260B**	8021B
p-Isopropylbenzene	99-87-6	1.	µg/L	5.	µg/kg	**8260B**	
Methyl-t-butyl ether (MTBE)	1634-04-4	20.	µg/L	50.	µg/kg	**8260B**	*8021B*
Methylene chloride	75-09-2	5.	µg/L	50.	µg/kg	**8260B**	8021B
4-Methyl-2-pentanone (MIBK)	108-10-1	20.	µg/L	50.	µg/kg	**8260B**	8015C
Naphthalene	91-20-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
n-Propylbenzene	103-65-1	1.	µg/L	5.	µg/kg	**8260B**	
Styrene	100-42-5	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,1,1,2-Tetrachloroethane	630-20-6	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,1,2,2-Tetrachloroethane	79-34-5	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
Tetrachloroethene	127-18-4	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
Toluene	108-88-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,2,4-Trichlorobenzene	120-82-1	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,1,1-Trichloroethane	71-55-6	1.	µg/L	5.	µg/kg	**8260B**	8021B
1,1,2-Trichloroethane	79-00-5	1.	µg/L	5.	µg/kg	**8260B**	8021B
Trichloroethene	79-01-6	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
Trichlorofluoromethane	75-69-4	2	µg/L	10	µg/kg	**8260B**	8021B
1,2,4 Trimethylbenzene	95-63-6	1.	µg/L	5.	µg/kg	**8260B**	
1,3,5 Trimethylbenzene	108-67-8	1.	µg/L	5.	µg/kg	**8260B**	
Vinyl acetate	108-05-4	20.	µg/L	50.	µg/kg	**8260B**	
Vinyl chloride	75-01-4	**1.**	µg/L	5.	µg/kg	**8260B**	8021B
o-Xylene	95-47-6	1.	µg/L	5.	µg/kg	**8260B**	8021B
m-Xylene	108-38-3	1.	µg/L	5.	µg/kg	**8260B**	8021B
p-Xylene	106-42-3	1.	µg/L	5.	µg/kg	**8260B**	8021B

VOA – Group B: BTEX and MTBE							
Analyte	**CAS Number**	**Aqueous** *25 mL purge*		**Soil/Sediment** (low concentration)		**Acceptable Methods**	
		RL	**units**	**RL**	**units**		
Benzene	71-43-2	1.	µg/L	5.	µg/kg	**8021B**	8260B
Ethylbenzene	100-41-4	1.	µg/L	5.	µg/kg	**8021B**	8260B
Toluene	108-88-3	1.	µg/L	5.	µg/kg	**8021B**	8260B
Xylenes (total o-, m-, and p-)	1330-20-7	1.	µg/L	5.	µg/kg	**8021B**	8260B
Methyl-t-butyl-ether (MTBE)	1634-04-4	10.	µg/L	50.	µg/kg	***8021B***	8260B

VOA – Group C: Antifreeze/Coolant							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Acceptable Methods**	
		RL	**units**	**RL**	**units**		
Ethylene glycol	107-21-1	500.	µg/L	500.	µg/kg	**8015C**	8430
Propylene glycol	107-21-1	500.	µg/L	500.	µg/kg	***8015C***	*8430*

[10a] **8260B** is the preferred Method for **SW-846 ProtocolVOA Group A**, the full OLQ Standard Volatiles List.
[10b] Reporting limits that are **bolded** are important in order to meet or come as close as possible to RISC/RCG levels.

SW-846 PROTOCOL
Semivolatile Organic Analysis (SVOA) and Nonvolatile Organic Analysis (NVOA)

SVOA & NVOA: Acceptable Sample Preparation Methods			
Method No.	**Sample Matrix**	**Procedure**	**Comments**
3500C	All	General Guidance	**Required.** Analyst **must** be familiar with.
3510	Aqueous	Separatory Funnel Liquid-Liquid Extraction	For any semivolatile or nonvolatile organic analytes
3520	Aqueous	Continuous Liquid-Liquid Extraction	For any semivolatile or nonvolatile organic analytes
3535A (Update IVB)	Aqueous	Solid Phase Extraction (SPE)	For any semivolatile or nonvolatile organic analytes
3540	Soils, Sediments, Solid Waste	Soxhlet Extraction	For any semivolatile or nonvolatile organic analytes
3541	Soils, Sediments, Solid Waste	Automated Soxhlet Extraction	For any semivolatile or nonvolatile organic analytes
3545A (Update IVB)	Soils, Sediments, Solid Waste	Pressurized Fluid Extraction (PFE)	For any semivolatile or nonvolatile organic analytes
3546	Soils, Sediments, Solid Waste	Microwave Extraction	For any semivolatile or nonvolatile organic analytes
3550C	Soils, Sediments, Solid Waste	Ultrasonic Extraction	For any semivolatile or nonvolatile organic analytes
3560	Soils, Sediments, Solid Waste	Supercritical Fluid Extraction (SFE)	For semivolatile petroleum hydrocarbons
3561	Soils, Sediments, Solid Waste	Supercritical Fluid Extraction (SFE)	For polynuclear aromatic hydrocarbons (PAHs)
3562	Soils, Sediments, Solid Waste	Supercritical Fluid Extraction (SFE)	For polychlorinated biphenyl compounds (PCBs) and organochlorine pesticides
3580A	Semivolatile and Nonvolatile Oily Wastes	Solvent Dilution	For non-aqueous, non-water soluble organic samples

SVOA – Group A: OLQ Standard Semivolatiles List							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment** (low concentration)		**Acceptable Methods[11a]**	
		RL	**units**	**RL**	**units**		
Acenaphthene	83-32-9	10	µg/L	660	µg/kg	**8270D**	8410
Acenaphthylene	208-96-8	10	µg/L	660	µg/kg	**8270D**	8410
Anthracene	120-12-7	10	µg/L	660	µg/kg	**8270D**	8410
Benzo[a]anthracene[12a]	56-55-3	10	µg/L	660	µg/kg	**8270D**	8410
Benzo[b]fluoranthene[12a]	205-99-2	10	µg/L	660	µg/kg	**8270D**	*8410*
Benzo[k]fluoranthene[12a]	207-08-9	10	µg/L	660	µg/kg	**8270D**	*8410*
Benzoic acid	65-85-0	50	µg/L	3300	µg/kg	**8270D**	8410
Benzo[g,h,i]perylene	191-24-2	10	µg/L	660	µg/kg	**8270D**	*8410*
Benzo[a]pyrene[12a]	50-32-8	10	µg/L	660	µg/kg	**8270D**	8410
Benzyl alcohol	100-54-6	20	µg/L	1300	µg/kg	**8270D**	*8410*
Bis(2-chloroethoxy)methane	111-91-1	10	µg/L	660	µg/kg	**8270D**	8410

[11a] **8270D** (analysis by GC/MS) is the preferred Method for **SVOA Group A**, the OLQ Standard Semivolatiles List.

[12a] The 8270D aqueous detection limits do not meet **RISC/RCG** levels. The soil detection limit for benzo[a]pyrene does not meet the **RISC/RCG** level. These PAH analytes should be analyzed by Method 8310 if they are COCs for the affected matrix (es) at sites pursuing **RISC/RCG** levels. (See **SVOA – Group C.)**

SVOA – Group A: OLQ GC/MS Standard Semivolatiles List, continued							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment** (low concentration)		**Acceptable Methods[11b]**	
		RL	**units**	**RL**	**units**		
Bis(2-chloroethyl)ether[13]	111-44-4	10	µg/L	660	µg/kg	**8270D**	8410
Bis(2-chloroisopropyl)ether[13]	108-60-1	10	µg/L	660	µg/kg	**8270D**	8410
Bis(2-ethylhexyl)phthalate[14]	117-81-7	10	µg/L	660	µg/kg	**8270D**	8410
4-Bromophenyl phenyl ether	101-55-3	10	µg/L	660	µg/kg	**8270D**	8410
Butyl benzyl phthalate	85-68-7	10	µg/L	660	µg/kg	**8270D**	8410
Carbazole	86-74-8	10	µg/L	660	µg/kg	***8270D***	*8410*
4-Chloroaniline	106-47-8	20	µg/L	1300	µg/kg	**8270D**	8410
4-Chloro-3-methylphenol	59-50-7	20	µg/L	1300	µg/kg	**8270D**	8410
2-Chloronaphthalene	91-58-7	10	µg/L	660	µg/kg	**8270D**	8410
2-Chlorophenol	95-57-8	10	µg/L	660	µg/kg	**8270D**	8410
4-Chlorophenyl phenyl ether	7005-72-3	10	µg/L	660	µg/kg	**8270D**	8410
Chrysene[12b]	218-01-9	10	µg/L	660	µg/kg	**8270D**	8410
Dibenzo[a,h]anthracene[12b]	53-70-3	10	µg/L	660	µg/kg	**8270D**	*8410*
Dibenzofuran	132-64-9	10	µg/L	660	µg/kg	**8270D**	8410
Di-n-butylphthalate	84-74-2	10	µg/L	660	µg/kg	**8270D**	8410
3,3'-Dichlorobenzidine[15]	91-94-1	20	µg/L	1300	µg/kg	**8270D**	*8410*
2,4-Dichlorophenol	120-83-2	10	µg/L	660	µg/kg	**8270D**	8410
Diethyl phthalate	84-66-2	10	µg/L	660	µg/kg	**8270D**	8410
2,4-Dimethylphenol	105-67-9	10	µg/L	660	µg/kg	**8270D**	*8410*
Dimethyl phthalate	131-11-3	10	µg/L	660	µg/kg	**8270D**	8410
4,6-Dinitro-2-methylphenol	534-52-1	50	µg/L	3300	µg/kg	**8270D**	8410
2,4-Dinitrophenol	51-25-5	50	µg/L	3300	µg/kg	**8270D**	8410
2,4-Dinitrotoluene[16a]	121-14-2	10	µg/L	660	µg/kg	**8270D**	8410
2,6-Dinitrotoluene[16a]	606-20-2	10	µg/L	660	µg/kg	**8270D**	8410
Di-n-octyl phthalate	117-84-0	10	µg/L	660	µg/kg	**8270D**	8410
Fluoranthene	206-44-0	10	µg/L	660	µg/kg	**8270D**	8410
Fluorene	86-73-7	10	µg/L	660	µg/kg	**8270D**	8410
Hexachlorobenzene[17]	118-74-1	10	µg/L	660	µg/kg	**8270D**	8410
Hexachlorobutadiene[17]	87-68-3	10	µg/L	660	µg/kg	**8270D**	8410
1-Methylnaphthalene	90-12-0	10	µg/L	660	µg/kg	**8270D**	
2-Methylnaphthalene	91-57-6	10	µg/L	660	µg/kg	**8270D**	

[11b] **8270D** (analysis by GC/MS) is the preferred Method for **SVOA Group A**, the OLQ Standard Semivolatiles List.

[12b] The 8270D aqueous detection limits do not meet **RISC/RCG** levels. The soil detection limit for dibenz[a,h] anthracene does not meet the **RISC/RCG** levels. These PAH analytes should be analyzed by Method 8310 if they are COCs for the affected matrix (es) at sites pursuing **RISC/RCG** levels. (See **SVOA – Group C.)**

[13] The 8270D soil detection limits do not meet **RISC/RCG** levels. The aqueous detection limits do not meet **RISC/RCG** levels, and the aqueous limit for bis(2-chloroethyl)ether also does not meet the **RISC/RCG** levels. These haloether analytes should be analyzed by alternate methods if they are COCs at sites pursuing **RISC/RCG** levels. Method 8410 is recommended for soils 8111 for water.

[14] The 8270D aqueous detection limit does not meet the **RISC /RCG** levels. This phthalate ester analyte should be analyzed by Method 8061A if it is a ground water COC at a site pursuing **RISC/RCG** levels.

[15] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. This analyte should be analyzed by an alternate method if it is a COC at a site pursuing **RISC/RCG** levels. Method 8325 is recommended for water and EPA Method 1625C for soils.

[16a] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. These nitroaromatic compounds should be analyzed by other methods if they are COCs at sites pursuing **RISC/RCG** levels. Method 8410 is recommended for soils and 8330 for water.

[17] The 8270D aqueous detection limits for both compounds do not meet **RISC/RCG** levels, and the aqueous limit for hexachlorobenzene does not meet the **RISC/RCG** levels. Hexachloro-1,3-butadiene should be analyzed by Method 8121 if it is a ground water COC at a site pursuing **RISC/RCG** levels. If hexachlorobenzene is a ground water COC at site pursuing **RISC/RCG** levels, it should be analyzed by Method 8121, or by Method 8081B (See **SVOA – Group C.**)

SVOA – Group A: OLQ GC/MS Standard Semivolatiles List, continued							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment** (low concentration)		**Acceptable Methods[11c]**	
		RL	**units**	**RL**	**units**		
Hexachlorocyclopentadiene	77-47-4	10.	µg/L	660	µg/kg	**8270D**	8410
Hexachloroethane	67-72-1	10.	µg/L	660	µg/kg	**8270D**	8410
Indeno[1,2,3-cd]pyrene[12c]	193-39-5	10.	µg/L	660	µg/kg	**8270D**	*8410*
Isophorone	78-59-1	10.	µg/L	660	µg/kg	**8270D**	8410
2-Methylnaphthalene	91-57-6	10.	µg/L	660	µg/kg	**8270D**	8410
2-Methylphenol *(o-cresol)*	95-48-7	10.	µg/L	660	µg/kg	**8270D**	8410
3-Methylphenol *(m-cresol)*	108-39-4	10.	µg/L	660	µg/kg	**8270D**	*8410*
4-Methylphenol *(p-cresol)*	106-44-5	10.	µg/L	660	µg/kg	**8270D**	8410
Naphthalene	91-20-3	18.	µg/L	18000.	µg/kg	**8270D**	8410
2-Nitroaniline[18]	88-74-4	50.	µg/L	3300	µg/kg	**8270D**	8410
3-Nitroaniline	99-09-2	50.	µg/L	3300	µg/kg	**8270D**	8410
4-Nitroaniline	100-01-6	20.	µg/L	1300	µg/kg	**8270D**	8410
Nitrobenzene[16b]	98-95-3	10.	µg/L	660	µg/kg	**8270D**	8410
2-Nitrophenol	88-75-5	10.	µg/L	660	µg/kg	**8270D**	8410
4-Nitrophenol	100-02-7	50.	µg/L	3300	µg/kg	**8270D**	8410
N-Nitrosodiphenylamine	86-30-6	10.	µg/L	660	µg/kg	**8270D**	8410
N-Nitroso-di-n-propylamine[19]	621-64-7	10.	µg/L	660	µg/kg	**8270D**	8410
Pentachlorophenol[20]	87-86-5	50.	µg/L	3300	µg/kg	**8270D**	8410
Phenanthrene	85-01-8	10.	µg/L	660	µg/kg	**8270D**	8410
Phenol	108-95-2	10.	µg/L	660	µg/kg	**8270D**	8410
Pyrene	129-00-0	10.	µg/L	660	µg/kg	**8270D**	8410
1,2,4-Trichlorobenzene	120-82-1	10.	µg/L	660	µg/kg	**8270D**	8410
2,4,5-Trichlorophenol	95-95-4	10.	µg/L	660	µg/kg	**8270D**	8410
2,4,6-Trichlorophenol	88-06-2	10.	µg/L	660	µg/kg	**8270D**	8410

SVOA – Group B: Polychlorinated Biphenyl Compounds (PCBs) as Aroclors								
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment** (low concentration)		**Wipes**		**Required Method**
		RL	**units**	**RL**	**units**	**RL**	**units**	
Aroclor 1016	12674-11-2	0.5	µg/L	1800	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1221	11104-28-2	0.5	µg/L	1800	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1232	11141-16-5	0.5	µg/L	1800	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1242	53469-21-9	0.5	µg/L	1800	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1248	12672-29-6	0.5	µg/L	1800.	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1254	11097-69-1	**0.5**	µg/L	1800.	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1260	11096-82-5	**0.5**	µg/L	1800.	µg/kg	10.	µg /100 cm^2	8082A
Aroclor 1262	37324-23-5	**0.5**	µg/L	1800.	µg/kg	10.	µg /100 cm^2	8082A

[11c] **8270D** (analysis by GC/MS) is the preferred Method for **SVOA Group A**, the OLQ Standard Semivolatiles List.

[12c] The 8270D aqueous detection limit does not meet **RISC/RCG** levels. This PAH analyte should be analyzed by Method 8310 if it is a ground water chemical of concern (COC) at a site pursuing **RISC/RCG** levels. (See **SVOA – Group C.**)

[16b] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. These nitroaromatic compounds should be analyzed by other methods if they are COCs at sites pursuing **RISC/RCG** levels. Method 8410 is recommended for soils and Method 8330 is recommended for water. Nitrobenzene in soil at **industrial** sites may also be analyzed by Method 8330.

[18] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. This compound should be analyzed by another method if a COC at sites pursuing **RISC/RCG** levels. Method 8410 is recommended for soils, and Method 8131 is recommended for water.

[19] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. This compound should be analyzed by another method if a COC at sites pursuing **RISC/RCG** levels. Method 8410 is recommended for soils, and Method 8070A is recommended for water.

[20] The 8270D aqueous and soil detection limits do not meet **RISC/RCG** levels. This chlorinated herbicide should be analyzed by Method 8151A if a ground water COC, and by Method 8151A or Method 8410 if a soil COC, at sites pursuing **RISC/RCG** levels.

SVOA – Group C: Polynuclear Aromatic Hydrocarbons by HPLC (Meets RISC/RCG Levels)[21,22]							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment** (low concentration)		**Required Method**	
		RL	**units**	**RL**	**units**		
Acenaphthene	83-32-9	20.	µg/L	20000.	µg/kg	8310	
Acenaphthylene	208-96-8	25.	µg/L	25000.	µg/kg	8310	
Anthracene	120-12-7	6.6	µg/L	6600.	µg/kg	8310	
Benzo[a]anthracene	56-55-3	0.13	µg/L	200.	µg/kg	8310	
Benzo[b]fluoranthene	205-99-2	0.18	µg/L	200.	µg/kg	8310	
Benzo[k]fluoranthene	207-08-9	0.17	µg/L	200.	µg/kg	8310	
Benzo[g,h,i]perylene	191-24-2	1.0	µg/L	1000.	µg/kg	8310	
Benzo[a]pyrene[21]	50-32-8	**0.20**	µg/L	230.	µg/kg	8310	
Chrysene	218-01-9	1.5	µg/L	1500.	µg/kg	8310	
Dibenzo(a,h)anthracene[21]	53-70-3	**0.15**	µg/L	300.	µg/kg	8310	
Fluoranthene	206-44-0	2.1	µg/L	2500.	µg/kg	8310	
Fluorene	86-73-7	2.5	µg/L	2500.	µg/kg	8310	
Indeno(1,2,3-cd)pyrene[21]	193-39-5	**0.022**	µg/L	430.	µg/kg	8310	
1-Methyl-naphthalene	90-12-0	18.	µg/L	18000	µg/kg	8310	
2-Methyl-naphthalene	91-57-6	18.	µg/L	18000	µg/kg	8310	
Naphthalene[22]	91-20-3	18.	µg/L	18000.	µg/kg	8310	
Phenanthrene	85-01-8	10.	µg/L	10000.	µg/kg	8310	
Pyrene	129-00-0	2.7	µg/L	2700.	µg/kg	8310	

[21]Reporting limits that are **bolded** are lower than the standard SW-846 Method 8310 Estimated Quantitation Limits (EQLs) for those analytes. They are either the Method 8310 MDL, or the MDL times a factor less than the standard SW-846 matrix factor (10 for ground water and 670 for low level soils). The bolded reporting limits are necessary for samples from sites pursuing **RISC/RCG** levels.

[22]The Method 8310 detection limits for naphthalene do not meet **RISC/RCG** values. If naphthalene is a chemical of concern (COC) at a site pursuing residential closure, it must be analyzed by Method 8260B or 8021B.

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SVOA – Group D: Organochlorine Pesticides by GC/ECD (Meets RISC/RCG Levels)[23]							
Analyte	CAS Number	Aqueous		Soil/Sediment		Required Method	
		RL	units	RL	units		
Aldrin	309-00-2	**0.04**	µg/L	**50**	µg/kg	8081B	
α-BHC (α-HCH)	319-84-6	**0.10**	µg/L	**5.**	µg/kg	8081B	
β-BHC (β-HCH)	319-85-7	0.25	µg/L	**5**.	µg/kg	8081B	
γ-BHC (Lindane, γ-HCH)	58-89-9	**0.05**	µg/L	**5.**	µg/kg	8081B	
δ-BHC (δ-HCH)	319-86-8	0.25	µg/L	20.	µg/kg	8081B	
α-Chlordane	5103-71-9	0.40	µg/L	25.	µg/kg	8081B	
γ-Chlordane	5103-74-2	0.40	µg/L	25.	µg/kg	8081B	
Chlordane-*not otherwise specified*	57-74-9	0.40	µg/L	25.	µg/kg	8081B	
4,4'-DDD	72-54-8	0.35	µg/L	50.	µg/kg	8081B	
4,4'-DDE	72-55-9	0.25	µg/L	50.	µg/kg	8081B	
4,4'-DDT	50-29-3	0.25	µg/L	50.	µg/kg	8081B	
Dieldrin	60-57-1	**0.04**	µg/L	**2.**	µg/kg	8081B	
Endosulfan I (α-endosulfan)	959-98-8	1.0	µg/L	50.	µg/kg	8081B	
Endosulfan II (β-endosulfan)	33213-65-9	1.0	µg/L	50.	µg/kg	8081B	
Endosulfan sulfate	1031-07-8	1.0	µg/L	25.	µg/kg	8081B	
Endrin	72-20-8	0.40	µg/L	10.	µg/kg	8081B	
Endrin aldehyde	7421-93-4	0.50	µg/L	10.	µg/kg	8081B	
Endrin ketone	53494-70-5	0.50	µg/L	10.	µg/kg	8081B	
Heptachlor	76-44-8	**0.20**	µg/L	30.	µg/kg	8081B	
Methoxychlor	72-43-5	0.90	µg/L	60.	µg/kg	8081B	
Toxaphene	8001-35-2	0.90	µg/L	60.	µg/kg	8081B	

[23]Reporting limits that are **bolded** are lower than the standard SW-846 Method 8081B Estimated Quantitation Limits (EQLs) for those analytes. They are either the Method 8081 (09/94) MDL, or the MDL times a factor less than the standard SW-846 matrix factor (10 for ground water and 670 for low level soils). The bolded reporting limits are necessary for samples from sites pursuing **RISC/RCG** levels.

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SW-846 PROTOCOL
Petroleum Analysis

Petroleum Analysis: Acceptable Sample Preparation and Introduction Methods		
Analyte	**Acceptable Methods**	**Comments**
TPH Gasoline Range Organics	5000 **(required)** plus one of: 5021, 5030, 5032, 3585, or other technique, as appropriate	See Acceptable Sample Preparation and Introduction Methods for VOA, above
TPH Diesel Range Organics and TPH Heavy Oil Range Organics/Extended Range Organics (ERO) – GC/FID	3500C **(required)** plus one of: 3510, 3520, 3535A, 3540, 3541, 3545A, 3546, 3550C, 3580A, or other technique, as appropriate	See Acceptable Sample Preparation Methods for SVOA and NVOA, above
TRPH in Water – Gravimetry	Extraction with n-hexane	Included in determinative method
TRPH in Soils, Sediments, and Sludges – IR	3560 or 9071B	SFE or extraction with n-hexane

Petroleum – Group A: Total Petroleum Hydrocarbons (TPH) or Total Recoverable Petroleum Hydrocarbons (TRPH) by GC/FID							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Acceptable Methods[24]**	
		RL	**units**	**RL**	**units**		
TPH Gasoline Range Organics (GRO) C_5-C_{10}	N/A	0.10	mg/L	1.0	mg/kg	8015C	8260B
TPH Diesel Range Organics (DRO) >C_{10}–C_{28}	N/A	0.10	mg/L	3.0	mg/kg	8015C	8270D
Extended Range Organics (ERO) >C_{10}-C_{36}	N/A	0.10	mg/L	3.0	mg/kg	8015C	8270D
TPH Heavy Oil Range Organics (TRPH)	N/A	0.50	mg/L	5.0	mg/kg	8015C	

Petroleum - Group B: Total Recoverable Petroleum Hydrocarbons (TRPH) by Infrared Spectrophotometry (soils and sediments) or Gravimetry (water)							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Acceptable Methods**	
		RL	**units**	**RL**	**units**	Prep	Analysis
TRPH in Sludge, Sediment, and Soil as n-Hexane Extractable Material (HEM) *or* by Supercritical Fluid Extraction (SFE)	N/A	N/A	N/A	10.	mg/kg	9071B or 3560	8440
TRPH in Water as HEM	N/A	5.0	mg/L	N/A	N/A	N/A	1664A/9070 [25]

[24]Section 7.7.2 of Method 8015C provides instructions for quantitation of GRO and DRO total petroleum hydrocarbons.

[25]As of Draft Update IVB of SW-846, Method 9070 has been replaced by a reference to EPA Office of Water "stand alone" Method 1664A, Publication No. EPA-821-R-98-002.

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SW-846 PROTOCOL
RCRA Characteristics of Hazardous Waste

RCRA Characteristics Group A– 40 CFR 261 Characteristics: Ignitability, Corrosivity, and Reactivity								
Waste Code	**RCRA Characteristic of HW**	**Parameter to be Measured**	**Aqueous**		**Other Matrices**		**Required Methods[27]**	
			RL or Range	**units**	**RL or Range**	**units**		
D001	Ignitability	Flash Point	N/A	N/A	N/A	Deg. F	1010	1020A
D002	Corrosivity	pH	1.0-14.0	pH units	1.0-14.0	pH units	9040B	9045C
D002	Corrosivity	Corrosivity to Steel	N/A	mm/yr.	N/A	mm/yr.	1110	
D003	Reactivity	Reactive Cyanide	25.	mg/L	25.	mg/kg	7.3.3.2	
D003	Reactivity	Reactive Sulfide	25.	mg/L	250.	mg/kg	7.3.4.2	

RCRA Characteristics Group B– 40 CFR 261 Characteristic of Toxicity: Toxic Characteristic Leaching Procedure (TCLP) – SW-846 Method 1311								
Waste Code	**Parameter to be Measured**	**Required Leaching Method[27]**	**Leachate**				**Acceptable Determinative Methods** (on Leachate)	
			RL	**units**	**Regulatory Limit**	**units**		
D004	Arsenic[26]	1311	0.50	mg/L	5.0	mg/L	6010C	6020A
D005	Barium	1311	10.	mg/L	100.0	mg/L	6010C	6020A
D006	Cadmium[26]	1311	0.10	mg/L	1.0	mg/L	6010C	6020A
D007	Chromium	1311	0.50	mg/L	5.0	mg/L	6010C	6020A
D008	Lead[26]	1311	0.50	mg/L	5.0	mg/L	6010C	6020A
D009	Mercury	1311	0.020	mg/L	0.2	mg/L	6020A	7470A 7472
D010	Selenium[26]	1311	0.10	mg/L	1.0	mg/L	6010C	6020A
D011	Silver	1311	0.50	mg/L	5.0	mg/L	6010C	6020A
D012	Endrin	1311	0.002	mg/L	0.02	mg/L	8081B	
D013	Lindane (γ-HCH)	1311	0.040	mg/L	0.4	mg/L	8081B	
D014	Methoxychlor	1311	1.0	mg/L	10.0	mg/L	8081B	
D015	Toxaphene	1311	0.050	mg/L	0.5	mg/L	8081B	
D016	2,4-D	1311	1.0	mg/L	10.0	mg/L	8151A	8321B
D017	2,4,5-TP (Silvex)	1311	0.10	mg/L	1.0	mg/L	8151A	8321B
D018	Benzene	1311	0.050	mg/L	0.5	mg/L	8260B	8021B
D019	Carbon Tetrachloride	1311	0.050	mg/L	0.5	mg/L	8260B	8021B
D020	Chlordane	1311	0.0030	mg/L	0.03	mg/L	8081B	
D021	Chlorobenzene	1311	10.	mg/L	100.0	mg/L	8260B	8021B
D022	Chloroform	1311	0.060	mg/L	6.0	mg/L	8260B	8021B
D023	o-Cresol	1311	5.0	mg/L	200.0	mg/L	8270D	8041A 8410
D024	m-Cresol	1311	5.0	mg/L	200.0	mg/L	8270D	8041A *8410*
D025	p-Cresol	1311	5.0	mg/L	200.0	mg/L	8270D	8041A 8410
D026	Cresol (total o-,m-,p-)	1311	20.0	mg/L	200.0	mg/L	8270D	8041A 8410
D027	1,4-Dichlorobenzene	1311	0.75	mg/L	7.5	mg/L	8260B	8021B 8270D
D028	1,2-Dichloroethane	1311	0.05	mg/L	0.5	mg/L	8260B	8021B
D029	1,1-Dichloroethene	1311	0.070	mg/L	0.7	mg/L	8260B	8021B
D030	2,4-Dinitrotoluene	1311	0.010	mg/L	0.13	mg/L	8270D	8091 8410

[26]Method 6010C will not be allowed for these analytes in aqueous samples unless the required reporting limits can be met.
[27]These methods may not be substituted. RCRA characteristic testing currently does not allow for the use of PBMS.

SW-846 PROTOCOL – RCRA CHARACTERISTICS OF HAZARDOUS WASTE

RCRA Characteristics Group B– 40 CFR 261 Characteristic of Toxicity: Toxic Characteristic Leaching Procedure (TCLP) – SW-846 Method 1311, continued								
Waste Code	**Parameter to be Measured**	**Required Leaching Method**[27]	**Leachate**				**Acceptable Determinative Methods** (on Leachate)	
			RL	**units**	**Regulatory Limit**	**units**		
D031	Heptachlor (and heptachlor epoxide)	1311	0.0008	mg/L	0.008	mg/L	8081B	
D032	Hexachlorobenzene	1311	0.010	mg/L	0.13	mg/L	8270D	8081B 8410
D033	Hexachlorobutadiene	1311	0.05	mg/L	0.5	mg/L	8270D	8260B 8021B
D034	Hexachloroethane	1311	0.30	mg/L	3.0	mg/L	8270D	8260B 8121
D035	Methyl ethyl ketone	1311	20.0	mg/L	200.0	mg/L	8260B	8015C
D036	Nitrobenzene	1311	0.20	mg/L	2.0	mg/L	8270D	8091 8410
D037	Pentachlorophenol	1311	10.	mg/L	100.0	mg/L	8270D	8041A 8151A
D038	Pyridine	1311	0.50	mg/L	5.0	mg/L	8270D	8260B 8015C
D039	Tetrachloroethylene	1311	0.070	mg/L	0.7	mg/L	8260B	8021B
D040	Trichloroethylene	1311	0.050	mg/L	0.5	mg/L	8260B	8021B
D041	2,4,5-Trichlorophenol	1311	40.	mg/L	400.0	mg/L	8270D	8041A 8410
D042	2,4,6-Trichlorophenol	1311	0.2	mg/L	2.0	mg/L	8270D	8041A 8410
D043	Vinyl chloride	1311	0.020	mg/L	0.2	mg/L	8260B	8021B

[27]These methods may not be substituted. RCRA characteristic testing currently does not allow for the use of PBMS.

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ADDITIONAL ANALYTICAL SERVICES

Additional Analytical Service – Petroleum: Total Petroleum Hydrocarbons (TPH) Fractionation						
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Acceptable Methods**
		RL	**units**	**RL**	**units**	
Gasoline Range Organics (GRO) Fractionation	N/A	0.25	mg/L	1.0	mg/kg	Washington Department of Ecology **VPH**
Diesel Range Organics (DRO) Fractionation	N/A	0.050	mg/L	5.0	mg/kg	Washington Department of Ecology **EPH**

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air		
Collection Units	**Acceptable Methods**	**Comments**
One or Six Liter Summa Canister	TO – 15	Total Analyte List & canister provided at time of request
One or Six Liter Summa Canister	TO – 15 SIM	Specific Analytes to be Provided at Time of Request

Fraction of Organic Carbon – Soils/Sediments						
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Acceptable Methods**
		RL	**units**	**RL**	**units**	
Fraction Organic Carbon (prep)	N/A	N/A	N/A	N/A	N/A	ASTM D421
Fraction Organic Carbon	N/A	N/A	N/A	N/A	%	Walkley Black
Soil Drying and #10 Sieving	NA	NA	NA	NA	NA	As Requested

Analyte[28]	**CAS Number**	**Aqueous**		**Acceptable Methods**
		LCMRL	**units**	
PFOA	335-67-1	2.	ng/L	537
PFOS	1763-23-1	2.	ng/L	537

[28]To account for linear and branched isomers of PFOA, EPA recommends that integration and quantitation of real-world drinking water samples include peaks that represent both linear and branched isomers. Since there is currently no certified quantitative PFOA standard that contains both linear and branched isomers that can be used to quantitate in the traditional manner, EPA recommends that until such standard is available, labs use the following approach:
• Calibrate instrumentation using a certified quantitative standard containing only the linear isomer.
• Identify the branched isomers by analyzing a "*qualitative/semi-quantitative"* PFOA mixed standard that includes both linear and branched isomers (Wellington Laboratories, cat#: T-PFOA or equivalent) and compare retention times and tandem mass spectrometry transitions.
• Quantitate PFOA by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration with the linear-isomer quantitative standard.

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SAS-GENERAL CHEMISTRY

SW-846 SAS Group A: GENERAL CHEMISTRY – Miscellaneous										
Analyte	CAS Number	Aqueous		Non-Aqueous		Acceptable Methods[29]				
		RL	units	RL	units					
Extractable Organic Halides in Solids	N/A	N/A	N/A	10.	mg/kg	9023				
Purgeable Organic Halides (POX)	N/A	0.005	mg/L	N/A	N/A	9021				
Specific Oxygen Uptake Rate in Biosolids (SOUR)[29]	N/A	N/A	N/A	1.0	(mg/g)/hr.	1683				
Ignitability of Solids	N/A	N/A	N/A	N/A	mm/sec	1030				
Oxidizing Solids	N/A	N/A	N/A	N/A	sec.	1040				
Substances Likely to Spontaneously Combust	N/A	N/A	N/A	N/A	N/A	1050				
Liquid Release Test	N/A	N/A	N/A	N/A	N/A	9096				
Settleable Matter (residue)[29]	N/A	0.2	Ml/L/hr.	N/A	N/A	2540 F[30]				
Bromide	24959-67-9	0.1	mg/L	N/A	N/A	9056	9211			
Chlorine, total residual[29]	7782-50-5	0.2	mg/L	N/A	N/A	4500 CL[30]				
Total Chlorine (new & used oil)	7782-50-5	N/A	N/A	200.	mg/kg	9075	9076			
Orthophosphate[29]	N/A	5.0	mg/L	1.0	N/A	9056	365.1	365.3		
Silica (SiO_2)[29]	7631-86-9	0.50	mg/L	50.	mg/kg	6010C	370.1			
Sulfite[29]	14265-45-3	2.0	mg/L	N/A	N/A	377.1				

SAS - RADIONUCLIDES

SW-846 SAS Group B: RADIONUCLIDES – Miscellaneous								
Analyte	CAS Number	Aqueous		Non-Aqueous		Acceptable Methods[29]		
		RL	units	RL	units			
Gross Alpha Radiation	12587-46-1	1.	pCi/L	N/A	N/A	9310	900.0	
Gross Beta Radiation	12587-47-2	4.	pCi/L	N/A	N/A	9310	900.0	
Radium-223	15623-45-7	1.	pCi/L	N/A	N/A	9315	903.0	
Radium-224	13233-32-4	1.	pCi/L	N/A	N/A	9315	903.0	
Radium-226	13982-63-3	1.	pCi/L	N/A	N/A	9315	903.0	903.1
Radium-228	15262-20-1	1.	pCi/L	N/A	N/A	9320	904.0	
Cesium-134	13967-70-9	10.	pCi/L	N/A	N/A	901.0		
Cesium-137	10045-97-3	10.	pCi/L	N/A	N/A	901.0		
Iodine-131	10043-66-0	1.	pCi/L	N/A	N/A	902.0		
Radon-222	14859-67-7	60.	pCi/L	N/A	N/A	903.1		
Gamma Emitting Radionuclides	N/A	10.	pCi/L	N/A	N/A	901.1		
Strontium-89	14158-27-1	10.	pCi/L	N/A	N/A	905.0		
Strontium-90	10098-97-2	2.	pCi/L	N/A	N/A	905.0		
Tritium	10028-17-8	1000.	pCi/L	N/A	N/A	906.0		
Uranium-234	13966-29-5		pCi/L	N/A	N/A	908.0		
Uranium-238	7440-61-1		pCi/L	N/A	N/A	908.0		

[29]General Chemistry: The reference for methods 365.1, Rev. 2.0 is *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 August 1993. The reference for method 365.3 is *Methods for Chemical Analysis of Water and Wastes*, EPA/600/R-79/020, revised March 1983. Method 1683 is a USEPA Office of Water "stand alone methods." (Methods 6010C and 9056 are SW-846 methods.)
Radionuclides: The reference for Methods 900.0, 901.0, 901.1, 902.0, 903.1, 905.0, 906.0, and 908.0 are *Prescribed Procedures for Measurement of Radioactivity in Drinking Water* , EPA 600 4-80-032 – August 1980. (Methods 9310, 9315, and 9320 are SW-846 methods.)

[30]The reference for methods 2540 F and 4500 CL is *Standard Methods for the Examination of Water and Wastewater*, 20th edition, 1998.

SAS – VOLATILE ORGANIC ANALYSIS

SW-846 SAS Group C: Supplemental VOLATILES List For Attachment VIII, Attachment IX, and Table 2										
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods**	
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**		
Acetonitrile	75-05-8	20.	µg/L	50.	µg/kg	X	X	X	8260B	8015C
Acrylonitrile	107-13-1	**1.**	µg/L	50.	µg/kg	X	X	X	8260B	8015C
Allyl alcohol	107-18-6	20.	µg/L	50.	µg/kg	X			8260B	8015C
Allyl chloride	107-05-1	5.	µg/L	5.	µg/kg	X	X	X	8260B	8021B
Benzyl chloride	100-44-7	5.	µg/L	5.	µg/kg	X			8260B	8021B
Bromoacetone	598-31-2	5.	µg/L	5.	µg/kg	X			8260B	8021B
Bromochloromethane	74-97-5	5.	µg/L	5.	µg/kg			X	8260B	8021B
Chloral hydrate	302-17-0	50.	µg/L	50.	µg/kg	X			8260B	
Chloroacetaldehyde	107-20-0	50.	µg/L	50.	µg/kg	X			*8021B*	*8260B*
Chloromethyl methyl ether	107-30-2	50.	µg/L	50.	µg/kg	X			8021B	
Chloroprene	126-99-8	5.	µg/L	5.	µg/kg	X	X	X	8260B	8021B
3-Chloropropionitrile	542-76-7	5.	µg/L	5.	µg/kg	X			8260B	
Crotonaldehyde	123-73-9	20.	µg/L	50.	µg/kg	X			8260B	8015C
Dibromochloromethane	124-48-1	**1.**	µg/L	5.	µg/kg		X	X	8260B	8021B
trans-1,4-Dichloro-2-butene	110-57-6	**1**.	µg/L	**5.**	µg/kg	X	X	X	8260B	
1,3-Dichloropropane	142-28-9	5.	µg/L	5.	µg/kg	X		X	8260B	8021B
2,2-Dichloropropane	590-20-7	5.	µg/L	5.	µg/kg	X		X	8260B	8021B
1,3-Dichloro-2-propanol	96-23-1	50.	µg/L	50.	µg/kg	X			8260B	8021B
1,1-Dichloropropene	563-58-6	5.	µg/L	5.	µg/kg	X		X	8260B	8021B
1,4-Dioxane	123-91-1	**5.**	µg/L	50.	µg/kg		X		8260B	8015C
Ethyl methacrylate	97-63-2	5.	µg/L	5.	µg/kg	X	X	X	8260B	
Iodomethane	74-88-4	5.	µg/L	5.	µg/kg	X	X	X	8260B	*8021B*
Isobutyl alcohol	78-83-1	20.	µg/L	50.	µg/kg	X	X	X	8260B	8015C
Methacrylonitrile	126-98-7	**1.**	µg/L	50.	µg/kg	X	X	X	8260B	
Methyl methacrylate	80-62-6	5.	µg/L	5.	µg/kg	X	X	X	8260B	
Paraldehyde	123-63-7	50.	µg/L	50.	µg/kg	X			8260B	8015C
Pentachloroethane	76-01-7	5.	µg/L	5.	µg/kg	X	X		8260B	
Propionitrile (ethyl cyanide)	107-12-0	20.	µg/L	50.	µg/kg	X	X	X	8260B	8015C
1,2,3-Trichloropropane	96-18-4	**1.**	µg/L	**5.**	µg/kg	X	X	X	8260B	8021B

SAS – SEMIVOLATILE ORGANIC ANALYSIS

SW-846 SAS Group D: Organophosphorus Pesticides and Herbicides for Attachment VIII, Attachment IX, and Table 2										
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods**	
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**		
Dimethoate	60-51-5	20.	µg/L	1300.	µg/kg	X	X	X	8141B	8270D 8321B
Disulfoton	298-04-4	1.5	µg/L	1000.	µg/kg	X	X	X	8141B	8321B
Famphur	52-85-7	20.	µg/L	1300.	µg/kg	X	X	X	8141B	8321B
Parathion, ethyl	56-38-2	20.	µg/L	1300.	µg/kg	X	X	X	8141B	8270D
Parathion, methyl	298-00-0	10.	µg/L	660.	µg/kg	X	X	X	8141B	8321B

SW-846 SAS Group D: Organophosphorus Pesticides and Herbicides for Attachment VIII, Attachment IX, and Table 2										
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods**	
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**		
Phorate	298-02-2	6.	µg/L	400.	µg/kg	X	X	X	8141B	8321B
Tetraethyl dithiopyrophosphate *(Sulfotepp)*	3689-24-5	20.	µg/L	1300.	µg/kg	X	X		8141B	8270D
Tetraethyl pyrophosphate *(TEPP)*	107-49-3	40.	µg/L	2600.	µg/kg	X			8141B	8270D

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SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS Group E: Additional Organophosphorus Pesticides, Herbicides, and Industrial Chemicals											
Analyte	CAS Number	Aqueous		Soil/Sediment		Attachment/Table			Suggested Methods	
		RL	units	RL	units	VIII	IX	2		
Aspon	3244-90-4	20.	µg/L	1300.	µg/kg				8141B	
Atrazine *(Aatrex)*	1912-24-9	0.3	µg/L	200.	µg/kg				8141B	
Azinphos-methyl	86-50-0	50.	µg/L	3300.	µg/kg				8141B	8270D
Azinphos-ethyl	2642-71-9	20.	µg/L	1300.	µg/kg				8141B	
Bolstar *(Sulprofos)*	35400-43-2	4.	µg/L	300.	µg/kg				8141B	
Carbophenothion	786-19-6	20.	µg/L	1300.	µg/kg				8141B	8270D
Chlorfenvinphos	470-90-6	20.	µg/L	1300.	µg/kg				8141B	8270D
Chlorpyrifos *(Dursban)*	2921-88-2	10.	µg/L	660.	µg/kg				8141B	
Chlorpyrifos methyl	5598-13-0	20.	µg/L	1300.	µg/kg				8141B	
Coumaphos	56-72-4	30.	µg/L	2000.	µg/kg				8141B	8270D
Crotoxyphos	7700-17-6	20.	µg/L	1300.	µg/kg				8141B	8270D
Demeton-O	8065-48-3	1.2	µg/L	1000.	µg/kg				8141B	8270D
Demeton-S	8065-48-3	1.2	µg/L	1000.	µg/kg				8141B	8270D
Diazinon	333-41-5	10.	µg/L	660.	µg/kg				8141B	
Dichlorofenthion	97-17-6	20.	µg/L	1300.	µg/kg				8141B	
Dichlorvos (DDVP)	62-73-7	2.	µg/L	1300.	µg/kg				8141B	8321B
Dicrotophos	141-66-2	20.	µg/L	1300.	µg/kg				8141B	8270D
Dioxathion	78-34-2	30.	µg/L	2000.	µg/kg				8141B	8270D
EPN	2104-64-5	20.	µg/L	1300.	µg/kg				8141B	8270D
Ethion	563-12-2	20.	µg/L	1300.	µg/kg				8141B	8270D
Ethoprop	13194-48-4	10.	µg/L	660.	µg/kg				8141B	
Fenitrothion	122-14-5	20.	µg/L	1300.	µg/kg				8141B	
Fensulfothion	115-90-2	20.	µg/L	1300.	µg/kg				8141B	8321B
Fenthion	55-38-9	20.	µg/L	1300.	µg/kg				8141B	8270D
Fonophos	944-22-9	10.	µg/L	660.	µg/kg				8141B	
Hexamethylphosphoramide *(HMPA*)	680-31-9	20.	µg/L	1300.	µg/kg				8141B	8270D
Leptophos	21609-90-5	20.	µg/L	1300.	µg/kg				8141B	8270D
Malathion	121-75-5	20.	µg/L	1300.	µg/kg				8141B	8270D
Merphos	150-50-5	1.2	µg/L	660.	µg/kg				8141B	8321B
Mevinphos	7786-34-7	20.	µg/L	1300.	µg/kg				8141B	8270D
Phosmet	732-11-6	30.	µg/L	2000.	µg/kg				8141B	8270D
Phosphamidon	13171-21-6	20.	µg/L	1300.	µg/kg				8141B	8270D
Ronnel	299-84-3	20.	µg/L	1300.	µg/kg				8141B	
Simazine *(Princep)*	122-34-9	5.	µg/L	330.	µg/kg				8141B	
Stirophos *(Tetrachlorvinphos)*	22248-79-9	2.	µg/L	1300.	µg/kg				8141B	8270D
Terbufos *(Counter)*	13071-79-9	0.9	µg/L	660.	µg/kg				8141B	8270D
Thionazine *(Zinophos)*	297-97-2	20.	µg/L	1300.	µg/kg				8141B	8270D
Tokuthion *(Prothiofos)*	34643-46-4	5.	µg/L	330.	µg/kg				8141B	
Trichlorfon	52-68-6	20.	µg/L	1300.	µg/kg				8141B	8321B
Trichloronate	327-98-0	5.	µg/L	330.	µg/kg				8141B	
Tri-o-cresyl phosphate *(TOCP*)	78-30-8	20.	µg/L	1300.	µg/kg				8141B	

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS Group F: Additional Organochlorine Pesticides										
Analyte	CAS Number	Aqueous		Soil/Sediment		Attachment/Table			Suggested Methods	
		RL	units	RL	units	VIII	IX	2		
Alachlor *(Lasso)*	15972-60-8	0.8	µg/L	50.	µg/kg				8081B	
Captafol	2425-06-1	20.	µg/L	1300.	µg/kg				8081B	8270D
Carbophenthion	786-19-6	10.	µg/L	660.	µg/kg				8081B	8270D
Chloroneb	2675-77-6	1.	µg/L	50.	µg/kg				8081B	
Chloropropylate	5836-10-2	1.	µg/L	50.	µg/kg				8081B	
Chlorothalonil	1897-45-6	1.	µg/L	50.	µg/kg				8081B	
DCPA *(Dacthal)*	1861-32-1	10.	µg/L	660.	µg/kg				8081B	
Dichlone	117-80-6	N/A	µg/L	1300.	µg/kg				8081B	8270D
Dicofol	115-32-2	0.1	µg/L	50.	µg/kg				8081B	
Etridiazole	2593-15-9	1.	µg/L	50.	µg/kg				8081B	
Mirex	2385-85-5	0.3	µg/L	20.	µg/kg				8081B	
Nitrofen	1836-75-5	20.	µg/L	1300.	µg/kg				8081B	8270D
Permethrin (cis + trans)	52645-53-1	10.	µg/L	6600.	µg/kg				8081B	

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS Group G: Chlorinated Herbicides										
Analyte	CAS Number	Aqueous		Soil/Sediment		Attachment/Table			Suggested Methods	
		RL	units	RL	units	VIII	IX	2		
Acifluorfen	50594-66-6	1.	µg/L	100.	µg/kg				8151A	
Bentazon	25057-89-0-	10.	µg/L	1000.	µg/kg				8151A	
Chloramben	133-90-4	10.	µg/L	1000.	µg/kg				8151A	
2,4-D	94-75-7	10.	µg/L	1000.	µg/kg	X	X	X	8151A	8321B
2,4-DB	94-82-6	10.	µg/L	1000.	µg/kg				8151A	8321B
DCPA diacid *(Dacthal diacid)*	2136-79-0	10.	µg/L	1000.	µg/kg				8151A	
Dalapon	75-99-0	20.	µg/L	2000.	µg/kg				8151A	8321B
Dicamba	1918-00-9	10.	µg/L	1000.	µg/kg				8151A	8321B
3,5-Dichlorobenzoic acid	51-36-5	10.	µg/L	1000.	µg/kg				8151A	
Dichloroprop	120-36-5	10.	µg/L	1000.	µg/kg				8151A	8321B
Dinoseb	88-85-7	2.	µg/L	1000.	µg/kg	X	X	X	8151A	8321B 8041A
5-Hydroxydicamba	7600-50-2	10.	µg/L	1000.	µg/kg				8151A	
2-Methyl-4-chlophenoxyacetic acid *(MCPA)*	94-74-6	2.	µg/L	1000.	µg/kg				8151A	8321B
2-(2-Methyl-4-chlophenoxy)-propionic acid *(MCPP)*	93-65-2	4.	µg/L	1000.	µg/kg				8151A	8321B
Pentachlorophenol *(PCP)*	87-86-5	0.5	µg/L	100..	µg/kg	X	X	X	8151A	8410 8041A
Picloram	1918-02-1	10.	µg/L	1000.	µg/kg				8151A	
2,4,5-T	93-76-5	10.	µg/L	1000.	µg/kg	X	X	X	8151A	8321B
2,4,5-TP *(Silvex)*	93-72-1	10.	µg/L	1000.	µg/kg	X	X	X	8151A	8321B

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS Group H: Supplemental **SEMIVOLATILES** List For Attachment VIII, Attachment IX, and Table 2										
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods**	
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**		
Acetophenone	98-86-2	10	µg/L	660	µg/kg	X	X	X	8270D	
2-Acetylaminofluorene (2-AAF)	53-96-3	20	µg/L	1300	µg/kg	X	X	X	8270D	
1-Acetyl-2-thiourea	591-08-2	1000	µg/L	66000	µg/kg	X			8270D	
Acrylamide	79-06-1	0.5	µg/L	50	µg/L	X			8032A	8316
4-Aminobiphenyl	92-67-1	20	µg/L	1300	µg/kg	X	X	X	8270D	
Aniline	62-53-3	10	µg/L	660	µg/kg	X	X		8270D	8131
Aramite	140-57-8	20	µg/L	1300	µg/kg	X	X		8270D	
Benz[c]acridine	225-51-4	100	µg/L	6600	µg/kg	X			*8270D*	*8310*
Benzal chloride	98-87-3	1	µg/L	5	µg/kg	X			8121	
Benzidine	92-87-5	20	µg/L	1300	µg/kg	X			8270D	8325
Benzo[j]fluoranthene	205-82-3	10	µg/L	660	µg/kg	X			*8270D*	*8310*
p-Benzoquinone	106-51-4	10	µg/L	660	µg/kg	X			8270D	
Benzotrichloride	98-07-7	1	µg/L	5	µg/kg	X			8121	
Dibenz[a,h]acridine	226-36-8	10	µg/L	660	µg/kg	X			*8270D*	*8310*
Dibenz[a,j]acridine	224-42-0	10	µg/L	660	µg/kg	X			8270D	*8310*
Dibenzo[a,e]pyrene	192-65-4	10	µg/L	660	µg/kg	X			8270D	*8310*
Dibenzo[a,h]pyrene	189-64-0	10	µg/L	660	µg/kg	X			*8270D*	*8310*
Dibenzo[a,i]pyrene	189-55-9	10	µg/L	660	µg/kg	X			*8270D*	*8310*
2,6-Dichlorophenol	87-65-0	10	µg/L	660	µg/kg				8270D	8041
Diethylstilbestrol	56-53-1	20	µg/L	1300	µg/kg	X			8270D	
3,3'-Dimethoxybenzidine	119-90-4	100	µg/L	660	µg/kg	X			8270D	8325
Dimethylaminoazobenzene	60-11-7	10	µg/L	660	µg/kg	X	X	X	8270D	
7,12-Dimethylbenz[a]anthracene	57-97-6	10	µg/L	660	µg/kg	X	X	X	8270D	*8310*
3,3'-Dimethylbenzidine	119-93-7	10	µg/L	660	µg/kg	X	X	X	8270D	8325
α,α-Dimethylphenethylamine	122-09-8	50	µg/L	3300	µg/kg	X	X		8270D	
1,3-Dinitrobenzene	99-65-0	20	µg/L	1300	µg/kg		X	X	8270D	
Dinitrobenzene, total	25154-54-5	40	µg/L	2600	µg/kg	X			8270D	
Diphenylamine	122-39-4	50	µg/L	3300	µg/kg	X	X	X	8270D	
1,2-Diphenylhydrazine	122-66-7	50	µg/L	3300	µg/kg	X			8270D	
Ethyl carbamate	51-79-6	50	µg/L	3300	µg/kg	X			8270D	
Ethyl methanesulfonate	62-50-0	20	µg/L	1300	µg/kg		X	X	8270D	
Fluchloralin	33245-39-5	20	µg/L	1300	µg/kg				8270D	
Hexachlorophene	70-30-4	50	µg/L	3300	µg/kg	X	X		8270D	
Hexachloropropene	1888-71-7	10	µg/L	660	µg/kg	X	X	X	8270D	
Isosafrole	120-58-1	10	µg/L	660	µg/kg	X	X	X	8270D	
Kepone	143-50-0	20	µg/L	1300	µg/kg	X	X	X	8270D	
Maleic anhydride	108-31-6	N/A	µg/L	3300	µg/kg	X			8270D	
Methapyrilene	91-80-5	100	µg/L	6600	µg/kg	X	X	X	8270D	
3-Methylcholanthrene	56-49-5	10	µg/L	660	µg/kg	X	X	X	8270D	
4,4'-Methylenebis(2-chloroaniline)	101-14-4	N/A	µg/L	3300	µg/kg	X			8270D	
Methyl methanesulfonate	66-27-3	10	µg/L	660	µg/kg	X	X	X	8270D	
1,4-Naphthoquinone	130-15-4	10	µg/L	660	µg/kg	X	X	X	8270D	8091
1-Naphthylamine	134-32-7	10	µg/L	660	µg/kg	X	X	X	8270D	

SW-846 SAS Group H: Supplemental **SEMIVOLATILES** List For Attachment VIII, Attachment IX, and Table 2,continued										
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods**	
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**		
2-Naphthylamine	91-59-8	10	µg/L	660	µg/kg	X	X	X	8270D	
Nicotine	54-11-5	20	µg/L	1300	µg/kg	X			8270D	
5-Nitro-o-toluidine	99-55-8	10	µg/L	660	µg/kg	X	X	X	8270D	
4-Nitroquinoline-1-oxide	56-57-5	40	µg/L	2600	µg/kg		X		8270D	
N-Nitrosodi-n-butylamine	924-16-3	10	µg/L	660	µg/kg	X	X	X	8270D	
N-Nitrosodiethylamine	55-18-5	20	µg/L	1300	µg/kg	X	X	X	8270D	
N-Nitrosodimethylamine	62-75-9	20	µg/L	1300	µg/kg	X	X	X	8270D	
N-Nitrosomethylethylamine	10595-95-6	20	µg/L	1300	µg/kg	X	X	X	8270D	
N-Nitrosomorpholine	59-89-2	40	µg/L	2600	µg/kg	X	X		8270D	
N-Nitrosopiperidine	100-75-4	20	µg/L	1300	µg/kg	X	X	X	8270D	
N-Nitrosopyrrolidine	930-55-2	40	µg/L	2600	µg/kg	X	X	X	8270D	
Octamethyl pyrophosphoramide	152-16-9	200	µg/L	13000	µg/kg	X			8270D	
Pentachlorobenzene	608-93-5	10	µg/L	660	µg/kg	X	X	X	8270D	8121
Phenacetin	62-44-2	20	µg/L	1300	µg/kg	X	X	X	8270D	
1,4-Phenylenediamine	106-50-3	10	µg/L	660	µg/kg	X	X	X	8270D	
Phthalic anhydride	85-44-9	100	µg/L	6600	µg/kg	X			8270D	
2-Picoline	109-06-8	20	µg/L	1300	µg/kg	X	X		8270D	
Pronamide	23950-58-5	10	µg/L	660	µg/kg	X	X	X	8270D	
Propylthiouracil	51-52-5	100	µg/L	6600	µg/kg	X			8270D	
Pyridine	110-86-1	20	µg/L	1300	µg/kg	X	X		8270D	
Resorcinol	108-46-3	100	µg/L	6600	µg/kg	X			8270D	
Safrole	94-59-7	10	µg/L	660	µg/kg	X	X	X	8270D	
Sulfallate	95-06-7	10	µg/L	660	µg/kg	X			8270D	
1,2,4,5-Tetrachlorobenzene	95-94-3	10	µg/L	660	µg/kg	X	X	X	8270D	8121
2,3,4,6-Tetrachlorophenol	58-90-2	10	µg/L	660	µg/kg	X	X	X	8270D	8041
Thiophenol	108-98-5	20	µg/L	1300	µg/kg	X			8270D	
Toluene diisocyanate	584-84-9	50	µg/L	3300	µg/kg	X			8270D	
o-Toluidine	95-53-4	10	µg/L	660	µg/kg	X	X	X	8270D	8015B
1,3,5-Trinitrobenzene	99-35-4	10	µg/L	660	µg/kg	X	X	X	8270D	8330A
O,O,O-Triethyl phosphorothioate	126-68-1	200	µg/L	13000	µg/kg	X	X	X	8270D	

SW-846 SAS Group I: Attachment VIII Thiocarbate Pesticides											
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/Table**			**Suggested Methods[31]**		
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**			
Butylate *(Sutan+)*	2008-41-5	10	µg/L	660	µg/kg	X			507	634	525.2
Cycloate *(Ro-Neet)*	1134-23-2	200	µg/L	1300	µg/kg	X			507	634	525.2
EPTC *(Eradicane)*	759-94-4	10	µg/L	660	µg/kg	X			507	634	525.2
Molinate *(Ordram)*	2212-67-1	10	µg/L	660	µg/kg	X			507	634	525.2
Pebulate *(Tillam)*	1114-71-2	200	µg/L	1300	µg/kg	X			507	634	525.2
Vernolate *(Vernam)*	1929-77-7	200	µg/L	1300	µg/kg	X			507	634	525.2

[31]The source for Methods 507, Revision 2.1, and 525.2, Revision 2.0 is *Methods for the Determination of Organic Compounds in Drinking Water - Supplement III* -EPA/600/R-95/131 - August 1995. The source for Method 634 is *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater –Volume I* - EPA-821-R-93-010-A August 1993, Revision 1.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS - Group J: Polychlorinated Biphenyl Compounds (PCBs) as Individual Congeners									
Analyte	CAS Number	IUPAC PCB No.	Aqueous		Soil/Sediment		Wipes		Required Method[32]
			RL	units	RL	units	RL	units	
2-Chlorobiphenyl	2051-60-7	1	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,3-Dichlorobiphenyl	16605-91-7	5	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',5-Trichlorobiphenyl	37680-65-2	18	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,4',5-Trichlorobiphenyl	16606-02-3	31	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206	0.05	µg/L	5.	µg/kg	1.	µg /100 cm^2	8082A

[32]When the sample matrix is biosolids, EPA Method 1668a (EPA 821/R-97-001 1997 or NTIS PB98-149213) may be used.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS - Group K: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs)								
Analyte	CAS Number	Aqueous		Soil/Sediment/ Paper Pulp		Acceptable Methods[33]	
		RL	units	RL	units		
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	0.050	ng/L *(ppt)*	5.0	ng/kg *(ppt)*	8290A	1613B
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0	0.050	ng/L *(ppt)*	5.0	ng/kg *(ppt)*	8290A	1613B

[33]Method 1613B is a USEPA Office of Water "Stand Alone" Method: "Tetra- hrough Octa-Chlorinated Dioxins and Furans by Isotope Dilution High Resolution Gas Chromatography/ High Resolution Mass Spectrometry," EPA 821-B-94-005 - Revision B (October 1994).

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS - Group K: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs), cont'd.							
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment/ Paper Pulp**		**Acceptable Methods[34]**	
		RL	**units**	**RL**	**units**		
Total Tetrachlorodibenzo-p-dioxin (TCDD)	41903-57-5	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
Total Pentachlorodibenzo-p-dioxin (PeCDD)	36088-22-9	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
Total Hexachlorodibenzo-p-dioxin (HxCDD)	34465-46-8	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
Total Heptachlorodibenzo-p-dioxin (HpCDD)	37871-00-4	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4	0.010	ng/L *(ppt)*	1.0	ng/kg *(ppt)*	8290A	1613B
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3	0.025	ng/L *(ppt)*	2.5	ng/kg *(ppt)*	8290A	1613B

[34]Method 1613B is a USEPA Office of Water "Stand Alone" Method: "Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution High Resolution Gas Chromatography/ High Resolution Mass Spectrometry," EPA 821-B-94-005 - Revision B (October 1994).

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS - Group L: N-Methyl-Carbamate Pesticides and Industrial Compounds											
Analyte	**CAS Number**	**Aqueous**		**Soil/Sediment**		**Attachment/ Table**			**Acceptable Methods**		
		RL	**units**	**RL**	**units**	**VIII**	**IX**	**2**			
Aldicarb (Temik)	116-06-3	20	µg/L	1300	µg/kg	X			8318A	8321B	
Aldicarb sulfone	1646-88-4	20	µg/L	1300	µg/kg	X			8318A	8321B	
Aldicarb sulfoxide	1646-87-3	20	µg/L	1300	µg/kg				8318A	8321B	
Bendiocarb	22781-23-3	200	µg/L	50000	µg/kg	X			8318A	8321B	
Carbaryl *(Sevin)*	63-25-2	10	µg/L	660	µg/kg	X			8318A	8321B	8270D
Carbofuran *(Furadan)*	1563-66-2	10	µg/L	660	µg/kg	X			8318A	8321B	8270D
m-Cumenyl methylcarbamate	64-00-6	100	µg/L	6600	µg/kg	X			8318A		
Dioxacarb	6988-21-2	20	µg/L	1300	µg/kg				8318A		
Formetanate hydrochloride	23422-53-9	20	µg/L	1300	µg/kg	X			8318A		
3-Hydroxycarbofuran	16655-82-6	30	µg/L	2000	µg/kg				8318A	8321B	
Methiocarb *(Mesurol)*	2032-65-7	30	µg/L	2000	µg/kg	X			8318A	8321B	
Methomyl *(Lannate)*	16752-77-5	20	µg/L	1300	µg/kg	X			8318A	8321B	
Metolcarb	1129-41-5	20	µg/L	1300	µg/kg	X			8318A	8321B	
Mexacarbate	315-18-4	20	µg/L	1300	µg/kg	X			8318A	8321B	8270D
Oxamyl	23135-22-0	20	µg/L	1300	µg/kg	X			8318A	8321B	
Promecarb	2631-37-0	25	µg/L	1700	µg/kg	X			8318A		
Propoxur *(Baygon)*	114-26-1	20	µg/L	1300	µg/kg	X			8318A	8321B	
Thiodicarb	59669-26-0	20	µg/L	1300	µg/kg	X			8318A		

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SAS Group M: Full-Range IR Scan on Unknown Semivolatile or Nonvolatile Organic Compound Material						
Analyte	**CAS Number**	**Aqueous**		**Non-Aqueous**		**Suggested Methods**
		RL	**units**	**RL**	**units**	
Unknown Semivolatile Organic or Nonvolatile Organic Material	N/A	10.	mg/L	10.	mg/kg	Extraction (if applicable) and Full-Range IR scan, using full scanning wavelength. (Similar to TRPH except without identification/ quantification of TPH.) Deliverable is complete IR scan without interpretation.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

SW-846 SAS - Group N: Additional Pesticides and Other Solvent-Extractable Nonvolatile Compounds											
Analyte	CAS Number	Aqueous		Soil/Sediment		Attachment/ Table			Suggested Methods		
		RL	units	RL	units	VIII	IX	2			
Aminocarb	2032-59-9	10.	µg/L	660.	µg/kg				8321B		
Asulam	3337-71-1	200.	µg/L	13000.	µg/kg				8321B		
Barban	101-27-9	200.	µg/L	13000.	µg/kg	X			8321B	8270D	
Benomyl	17804-35-2	200.	µg/L	13000.	µg/kg	X			8321B		
Bromacil	314-40-9	10.	µg/L	660.	µg/kg				8321B		
Butylate	2008-41-5	10.	µg/L	660.	µg/kg	X			8321B		634
Caffeine	58-08-2	40.	µg/L	2600.	µg/kg				8321B	*8270D*	
Carbendazim	10605-21-7	100.	µg/L	6600.	µg/kg	X			8321B		
Chloropropham	101-21-3	200.	µg/L	13000.	µg/kg				8321B		
Chloroxuron	1982-47-4	200.	µg/L	13000.	µg/kg				8321B		
Diuron	330-54-1	5.	µg/L	660.	µg/kg				8321B		
Fenuron	101-42-8	5.	µg/L	660.	µg/kg				8321B		
Fluometuron	2164-17-2	40.	µg/L	2600.	µg/kg				8321B		
Linuron	330-55-2	5.	µg/L	660.	µg/kg				8321B		
Monuron	150-68-5	5.	µg/L	660.	µg/kg				8321B		
Monocrotophos	6923-22-4	40.	µg/L	2600.	µg/kg				8321B	8270D	8141B
Naled	300-76-5	20.	µg/L	1300.	µg/kg				8321B	8270D	8141B
Neburon	555-37-3	5.	µg/L	660.	µg/kg				8321B		
Propachlor	1918-16-7	40.	µg/L	2600.	µg/kg				8321B		
Propham	122-42-9	50.	µg/L	3300.	µg/kg	X			8321B		
Siduron	1982-49-6	5.	µg/L	660.	µg/kg				8321B		
Strychnine	57-24-9	40	µg/L	2600	µg/kg	X			8321B	8270D	
Tebuthiuron	34014-18-1	200.	µg/L	13000.	µg/kg				8321B		
Thiofanox	39196-18-4	5.	µg/L	660.	µg/kg	X			8321B		
Tris(2,3-dibromopropyl) phosphate	126-72-7	200.	µg/L	13000	µg/kg	X			8321B	8270D	

SAS – LEACHING PROCEDURES

SW-846 SAS – Group O Synthetic Precipitation Leaching Procedure (SPLP) – SW-846 Method 1312					
Extraction Procedure	**Applicable Analytes**	**Extraction Fluid Required**	**Required Leaching Method**	**Acceptable Determinative Methods** (on Leachate)	
Nonvolatile (bottle) extraction	SVOCs, pesticides, herbicides, explosives, other semivolatile and nonvolatile organic compounds, metals, and other inorganic analytes	#1 (pH 4.20 ± 0.05). Prepared from 60/40 weight % mixture of sulfuric and nitric acid	1312	See Metals, SVOA, and General Chemistry Analyte Groups above.	
				To be based on site-specific COCs determined and specified at time of analytical request.	
Zero headspace extraction (ZHE)	VOCs	#3 (Reagent water)	1312	8260B	8021B/8015C
				Specific analyte list will be specified at time of analytical request.	
Nonvolatile (bottle) extraction	Cyanide	#3 (Reagent water)	1312	9014	9213
				Assume determination is **free** cyanide unless notified differently at time of analytical request.	

SAS – LEACHING PROCEDURES, continued

SW-846 SAS – Group P: Neutral Leaching Method for Industrial Wastes[35]					
Applicable Waste Stream	**Extraction Procedure**	**Required Analytes**	**Extraction Fluid Required**	**Required Leaching Method**	**Acceptable Determinative Methods** (on Leachate)
▪ Coal Ash ▪ Flue Gas Desulfurization Byproducts	Nonvolatile (bottle) extraction	Barium Chloride Cyanide* Fluoride pH Sodium Sulfate Sulfide* Total dissolved solids **Total*	Deionized water	▪ SW-846 Method 1311, substituting reagent water for extraction fluid *or* SW-846 Method 1312, using fluid #3 (reagent water)	See Metals and General Chemistry Analyte Groups above.
▪ Foundry Waste	Nonvolatile (bottle) extraction	Chloride Copper Cyanide* Fluoride Iron Manganese Nickel Phenols pH Sodium Sulfate Sulfide* Total dissolved solids Zinc **Total*	Deionized water	▪ SW-846 Method 1311, substituting reagent water for extraction fluid *or* SW-846 Method 1312, using fluid #3 (reagent water)	See Metals and General Chemistry Analyte Groups above.
▪ Other Industrial Wastes	Nonvolatile (bottle) and/or ZHE extraction	Site-specific analytes to be specified at time of analytical request.	To be specified at time of analytical request.	To be specified at time of analytical request.	See appropriate Analyte Group(s) above.

[35] See 329 IAC 10-9 in Indiana Solid Waste Regulations.

SAS – LEACHING PROCEDURES, continued

SW-846 SAS – Group Q: USACE Modified Elutriate [36]				
Extraction Procedure	**Applicable Analytes**	**Extraction Fluid Required**	**Required Leaching Method[36]**	**Acceptable Determinative Methods** (on Leachate)
Elutriate Test on dredged sediment	SVOCs, pesticides, herbicides, PCBs, explosives, other semivolatile and nonvolatile organic compounds, metals, and other inorganic analytes, USACE column settling tests[36] (Site-specific analytes will be communicated at time of analytical request.)	Effluent water collected from dredged sediment storage site being evaluated (with bubble aeration)	USACE document no.: EEDP-04-2	See Metals, SVOA, and General Chemistry Analyte Groups above. To be based on site-specific COCs determined and communicated at time of analytical request.

[36] Test procedures are included in the document, "Interim Guidance for Predicting Quality of Effluent Discharged from Confined Dredged Material Dis http://el.erdc.usace.army.mil/dots/pdfs/mpd86-1.pdf posal Areas--Test Procedures" (June 1985), available from the USACE web site as document number EEDP-04-2.

USEPA OFFICE OF WATER DRINKING WATER (Drinking Water) PROTOCOL

Drinking Water Volatile Organic Compounds

Drinking Water – Volatiles Group A: Drinking Water 524.2 VOCs List[37]							
Analyte	**CAS Number**	**Aqueous**				**Acceptable[37,38] Methods**	
		RL	**units**	**RL**	**units**		
Acetone	67-64-1	1.	µg/L	N/A	N/A	**524.2**	
Benzene	71-43-2	0.2	µg/L	N/A	N/A	**524.2**	502.2
Bromobenzene	108-86-1	0.5	µg/L	N/A	N/A	**524.2**	502.2
Bromochloromethane	74-97-5	0.5	µg/L	N/A	N/A	**524.2**	502.2
Bromodichloromethane	75-27-4	0.4	µg/L	N/A	N/A	**524.2**	502.2
Bromoform	75-25-2	0.6	µg/L	N/A	N/A	**524.2**	502.2
Bromomethane	74-83-9	1.	µg/L	N/A	N/A	**524.2**	502.2
2-Butanone (MEK)	78-93-3	1.	µg/L	N/A	N/A	**524.2**	
n-Butylbenzene	104-51-8	1.	µg/L	N/A	N/A	**524.2**	502.2
sec-Butylbenzene	135-98-8	1.	µg/L	N/A	N/A	**524.2**	502.2
tert-Butylbenzene	98-06-6	1.	µg/L	N/A	N/A	**524.2**	502.2
Carbon disulfide	75-15-0	1.	µg/L	N/A	N/A	**524.2**	
Carbon tetrachloride	56-23-5	**1.**	µg/L	N/A	N/A	**524.2**	502.2
Chlorobenzene	108-90-7	0.5	µg/L	N/A	N/A	**524.2**	502.2
Chloroethane	75-00-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
Chloroform	67-66-3	0.2	µg/L	N/A	N/A	**524.2**	502.2
Chloromethane	74-87-3	0.7	µg/L	N/A	N/A	**524.2**	502.2
2-Chlorotoluene	95-49-8	0.5	µg/L	N/A	N/A	**524.2**	502.2
4-Chlorotoluene	106-43-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
Dibromochloromethane	124-48-1	**0.1**	µg/L	N/A	N/A	**524.2**	502.2
1,2-Dibromo-3-Chloropropane	96-12-8	**0.05**	µg/L	N/A	N/A	**524.2**	502.2
1,2-Dibromoethane (EDB)	106-93-4	**0.1**	µg/L	N/A	N/A	**524.2**	502.2
Dibromomethane	74-95-3	1.	µg/L	N/A	N/A	**524.2**	502.2
1,2-Dichlorobenzene	95-50-1	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,3-Dichlorobenzene	541-73-1	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,4-Dichlorobenzene	106-46-7	0.2	µg/L	N/A	N/A	**524.2**	502.2
Dichlorodifluoromethane	75-71-8	1.	µg/L	N/A	N/A	**524.2**	502.2
1,1-Dichloroethane	75-34-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2-Dichloroethane	107-06-2	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1-Dichloroethene	75-35-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
cis-1,2-Dichloroethene	156-59-2	0.5	µg/L	N/A	N/A	**524.2**	502.2
trans-1,2-Dichloroethene	156-60-5	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2-Dichloropropane	78-87-5	0.1	µg/L	N/A	N/A	**524.2**	502.2
1,3-Dichloropropane	142-28-9	0.5	µg/L	N/A	N/A	**524.2**	502.2

[37]Method 524.2, Revision 4.1 is the preferred method for **Drinking Water Protocol Group A**, the drinking water volatile organics list.
[38]The reference for Methods 524.2, Revision 4.1; 502.2, Revision 2.1; and 551.1, Revision 1.0, is *Methods for the Determination of Organic* Compounds in Drinking Water, Supplement III, -EPA/600/R-95/131 - August 1995.

DRINKING WATER PROTOCOL – VOCs

Drinking Water – Volatiles Group A: Drinking Water 524.2 List[39] continued							
Analyte	**CAS Number**	**Aqueous**				**Acceptable**[40] **Methods**	
		RL	**units**	**RL**	**units**		
2,2-Dichloropropane	590-20-7	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1-Dichloropropene	563-58-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
cis-1,3-Dichloropropene	10061-01-5	0.1	µg/L	N/A	N/A	**524.2**	502.2
trans-1,3-Dichloropropene	10061-02-6	0.1	µg/L	N/A	N/A	**524.2**	502.2
Ethylbenzene	100-41-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
Hexachlorobutadiene	87-68-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
2-Hexanone (MBK)	591-78-6	1.	µg/L	N/A	N/A	**524.2**	
Isopropylbenzene	98-82-8	1.	µg/L	N/A	N/A	**524.2**	502.2
Methyl-t-butyl ether (MTBE)	1634-04-4	0.5	µg/L	N/A	N/A	**524.2**	*502.2*
Methylene chloride	75-09-2	0.5	µg/L	N/A	N/A	**524.2**	502.2
4-Methyl-2-pentanone (MIBK)	108-10-1	1.	µg/L	N/A	N/A	**524.2**	
Naphthalene	91-20-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
2-Nitropropane	79-46-9	0.5	µg/L	N/A	N/A	**524.2**	
n-Propylbenzene	103-65-1	1.	µg/L	N/A	N/A	**524.2**	502.2
Styrene	100-42-5	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1,1,2-Tetrachloroethane	630-20-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1,2,2-Tetrachloroethane	79-34-5	0.5	µg/L	N/A	N/A	**524.2**	502.2
Tetrachloroethene	127-18-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
Toluene	108-88-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2,3-Trichlorobenzene	87-61-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2,4-Trichlorobenzene	120-82-1	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1,1-Trichloroethane	71-55-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,1,2-Trichloroethane	79-00-5	0.5	µg/L	N/A	N/A	**524.2**	502.2
Trichloroethene	79-01-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
Trichlorofluoromethane	75-69-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2,3-Trichloropropane	96-18-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
1,2,4-Trimethylbenzene	95-63-6	1.	µg/L	N/A	N/A	**524.2**	502.2
1,3,5-Trimethylbenzene	108-67-8	1.	µg/L	N/A	N/A	**524.2**	502.2
Vinyl chloride	75-01-4	0.5	µg/L	N/A	N/A	**524.2**	502.2
o-Xylene	95-47-6	0.5	µg/L	N/A	N/A	**524.2**	502.2
m-Xylene	108-38-3	0.5	µg/L	N/A	N/A	**524.2**	502.2
p-Xylene	106-42-3	0.5	µg/L	N/A	N/A	**524.2**	502.2

[39]Method 524.2, Revision 4.1 is the preferred method for **Drinking Water Protocol VolatilesGroup A**, the drinking water volatile organics list.

[40]The reference for Methods 524.2, Revision 4.1; 502.2, Revision 2.1; and 551.1, Revision 1.0, is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III,* -EPA/600/R-95/131 - August 1995**.**

DRINKING WATER PROTOCOL
Semivolatile Organic Compounds

Drinking Water – Semivolatiles Group A: Method 525.2 SVOC Extractables List[41]								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[41,42]**		
		RL	**units**	**RL**	**units**			
Acenaphthylene	208-96-8	1.	µg/L	N/A	N/A	**525.2**	550.1	
Anthracene	120-12-7	1.	µg/L	N/A	N/A	**525.2**	550.1	
Benzo[a]anthracene	56-55-3	0.5	µg/L	N/A	N/A	**525.2**	550.1	
Benzo[b]fluoranthene	205-99-2	0.2	µg/L	N/A	N/A	**525.2**	550.1	
Benzo[k]fluoranthene	207-08-9	0.5	µg/L	N/A	N/A	**525.2**	550.1	
Benzo[g,h,i]perylene	191-24-2	2.	µg/L	N/A	N/A	**525.2**	550.1	
Benzo[a]pyrene	50-32-8	**0.2**	µg/L	N/A	N/A	**525.2**	550.1	
Butyl benzyl phthalate	85-68-7	10.	µg/L	N/A	N/A	**525.2**	506	
Chrysene	218-01-9	1.	µg/L	N/A	N/A	**525.2**	550.1	
Dibenzo[a,h]anthracene	53-70-3	**0.1**	µg/L	N/A	N/A	**525.2**	550.1	
Di-n-butylphthalate	84-74-2	10.	µg/L	N/A	N/A	**525.2**	506	
Diethyl phthalate	84-66-2	10.	µg/L	N/A	N/A	**525.2**	506	
Di(2-ethylhexyl)adipate	103-23-1	10.	µg/L	N/A	N/A	**525.2**	506	
Di(2-ethylhexyl)phthalate	117-81-7	**1.**	µg/L	N/A	N/A	**525.2**	525.1	506
Dimethyl phthalate	131-11-3	10.	µg/L	N/A	N/A	**525.2**	506	
2,4-Dinitrotoluene	121-14-2	0.5.	µg/L	N/A	N/A	**525.2**	609	
2,6-Dinitrotoluene	606-20-2	0.5	µg/L	N/A	N/A	**525.2**	609	
Fluorene	86-73-7	10.	µg/L	N/A	N/A	**525.2**	550.1	
Hexachlorobenzene	118-74-1	0.2	µg/L	N/A	N/A	**525.2**	505	508.1
Hexachlorocyclopentadiene	77-47-4	5.	µg/L	N/A	N/A	**525.2**	505	508.1
Indeno[1,2,3-cd]pyrene	193-39-5	**0.02**	µg/L	N/A	N/A	**525.2**	550.1	
Isophorone	78-59-1	10.	µg/L	N/A	N/A	**525.2**	609	
Pentachlorophenol	87-86-5	0.2	µg/L	N/A	N/A	**525.2**	515.1	515.2
Phenanthrene	85-01-8	1.	µg/L	N/A	N/A	**525.2**	550.1	
Pyrene	129-00-0	10.	µg/L	N/A	N/A	**525.2**	550.1	

Drinking Water – Semivolatiles Group B: Aroclors List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[42]**		
		RL	**units**	**RL**	**units**			
Aroclor 1016	12674-11-2	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1221	11104-28-2	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1232	11141-16-5	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1242	53469-21-9	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1248	12672-29-6	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1254	11097-69-1	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505
Aroclor 1260	11096-82-5	**0.5**	µg/L	N/A	N/A	**525.2**	508.1	505

[41]Method 525.2, Revision 2.0, is the preferred method for **Drinking Water Protocol Semivolatile Group A**.

[42]The reference for Methods 525.2, Revision 2.0; 505, Revision 2.1; 506, Revision 1.1; 508.1, Revision 2.0; 515.1, Revision 4.1; and 515.2, Revision 1.1; is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III*, -EPA/600/R-95/131 - August 1995. The reference for Method 550.1 is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA/600/4-90/020 - July 1990 The reference for Method 609 is Code of Federal Regulations (CFR) 40, Parts 136 - Revised as of July 1, 1995, Exhibit E to Part 136 –Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

DRINKING WATER PROTOCOL
Special Analytical Services (SAS)

SAS – VOLATILE ORGANIC ANALYSIS

Drinking Water SAS Group A: Additional Volatile Organic Compounds (not on 524.2 list)								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[43]**		
		RL	**units**	**RL**	**units**			
Acrolein	107-02-8	**0.7**	µg/L	N/A	N/A	**603**		
n-Butanol	71-36-3	500.	µg/L	N/A	N/A	**1666**		
Vinyl acetate	108-05-4	50.	µg/L	N/A	N/A	**1624C**		

SAS – SEMIVOLATILE ORGANIC ANALYSIS

Drinking Water SAS Group B: Method 525.2 Organochlorine Pesticides List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[43]**		
		RL	**units**	**RL**	**units**			
Alachlor *(Lasso)*	15972-60-8	1.	µg/L	N/A	N/A	**525.2**	508.1	505
Aldrin	309-00-2	**0.05**	µg/L	N/A	N/A	**525.2**	508.1	505
Atrazine *(Aatrex)*	1912-24-9	**0.3**	µg/L	N/A	N/A	**525.2**	508.1	507
α-Chlordane	5103-71-9	0.2	µg/L	N/A	N/A	**525.2**	508.1	505
γ-Chlordane	5103-74-2	0.2	µg/L	N/A	N/A	**525.2**	508.1	505
Chlorneb	2675-77-6	1.	µg/L	N/A	N/A	**525.2**	508.1	
Chlorobenzilate	510-15-6	0.25	µg/L	N/A	N/A	**525.2**	508.1	
Chlorothalonil	1897-45-6	0.5	µg/L	N/A	N/A	**525.2**	508.1	
Dacthal (DCPA)	1861-32-1	2.	µg/L	N/A	N/A	**525.2**	508.1	
4,4'-DDD	72-54-8	0.5	µg/L	N/A	N/A	**525.2**	508.1	
4,4'-DDE	72-55-9	0.5	µg/L	N/A	N/A	**525.2**	508.1	
4,4'-DDT	50-29-3	0.5	µg/L	N/A	N/A	**525.2**	508.1	
Dieldrin	60-57-1	**0.05**	µg/L	N/A	N/A	**525.2**	508.1	505
Endosulfan I	959-98-8	10.	µg/L	N/A	N/A	**525.2**	508.1	
Endosulfan II	33123-65-9	10.	µg/L	N/A	N/A	**525.2**	508.1	
Endosulfan sulfate	1031-07-8	10.	µg/L	N/A	N/A	**525.2**	508.1	
Endrin	72-20-8	1.	µg/L	N/A	N/A	**525.2**	508.1	505
Endrin aldehyde	7421-93-4	1.	µg/L	N/A	N/A	**525.2**	508.1	
Etridiazole	2593-15-9	2.	µg/L	N/A	N/A	**525.2**	508.1	
α-HCH *(α-BHC)*	319-84-6	**0.1**	µg/L	N/A	N/A	**525.2**	508.1	
β-HCH *(β-BHC)*	319-85-7	**0.4**	µg/L	N/A	N/A	**525.2**	508.1	
δ-HCH *(δ-BHC)*	319-86-7	0.2	µg/L	N/A	N/A	**525.2**	508.1	
γ-HCH *(Lindane, γ-BHC)*	58-89-9	**0.2**	µg/L	N/A	N/A	**525.2**	508.1	505
Heptachlor	76-44-8	**0.2**	µg/L	N/A	N/A	**525.2**	508.1	505
Heptachlor epoxide	1024-57-3	**0.2.**	µg/L	N/A	N/A	**525.2**	508.1	505
Methoxychlor	72-43-5	1.	µg/L	N/A	N/A	**525.2**	508.1	505
cis-Nonachlor	5103-73-1	1.	µg/L	N/A	N/A			**505**
trans-Nonachlor	39765-80-5	0.2	µg/L	N/A	N/A	**525.2**	508.1	505
cis-Permethrin	54774-45-7	10.	µg/L	N/A	N/A	**525.2**	508.1	
trans-Permethrin	51877-74-8	10.	µg/L	N/A	N/A	**525.2**	508.1	
Simazine *(Princep)*	122-34-9	0.5	µg/L	N/A	N/A	**525.2**	508.1	507
Toxaphene	8001-35-2	1.	µg/L	N/A	N/A	**525.2**	508.1	505

[43]The reference for Methods 525.2, Revision 2.0; 505, Revision 2.1; 507, Revision 2.1; and 508.1, Revision 2.0; is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III*, EPA/600/R-95/131 - August 1995. The reference for Method 550.1 is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA/600/4-90/020 - July 1990 The reference for Method 603 is Code of Federal Regulations (CFR) 40, Parts 136 - Revised as of July 1, 1995, Exhibit E to Part 136 –Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. The reference for Method 1666 is *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater*, Revision A EPA-821-B-98-016 - July 1998. Method 1624C is a USEPA Office of Water "Stand Alone" method.

Drinking Water – SAS Group C: Method 525.2 Nitrogen/Phosphorus Pesticides List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[44]**		
		RL	**units**	**RL**	**units**			
Ametryn	834-12-8	5.	µg/L	N/A	N/A	**525.2**	507	
Atroton	1610-17-9	5.	µg/L	N/A	N/A	**525.2**	507	
Bromacil	314-40-9	5.	µg/L	N/A	N/A	**525.2**	507	
Butachlor	23184-66-9	5.	µg/L	N/A	N/A	**525.2**	507	508.1
Butylate *(Sutan Plus)*	2008-41-5	5.	µg/L	N/A	N/A	**525.2**	507	634
Carboxin	5234-68-4	1.	µg/L	N/A	N/A	**525.2**	507	
Chlorpropham	101-21-3	2.	µg/L	N/A	N/A	**525.2**	507	
Chlorpyrifos *(Dursban)*	2921-88-2	5.	µg/L	N/A	N/A	**525.2**	*508.1*	622
Cyanazine *(Bladex)*	21725-46-2	0.2	µg/L	N/A	N/A	**525.2**	508.1	
Cycloate	1134-23-2	1.	µg/L	N/A	N/A	**525.2**	507	634
Diazinon	331-41-5	3.	µg/L	N/A	N/A	**525.2**	507	
Dichlorvos	62-73-7	3.	µg/L	N/A	N/A	**525.2**	507	
Diphenamid	957-51-7	10.	µg/L	N/A	N/A	**525.2**	507	
Disulfoton	298-04-4	1.	µg/L	N/A	N/A	**525.2**	507	
Disulfoton sulfone	2497-06-5	10.	µg/L	N/A	N/A	**525.2**	507	
Disulfoton sulfoxide	2497-07-6	10.	µg/L	N/A	N/A	**525.2**	507	
EPTC *(Eradicane)*	759-94-4	10.	µg/L	N/A	N/A	**525.2**	507	634
Ethoprop	13194-48-4	1.	µg/L	N/A	N/A	**525.2**	507	
Fenamiphos	22224-92-6	5.	µg/L	N/A	N/A	**525.2**	507	
Fenarimol	60168-88-9	10.	µg/L	N/A	N/A	**525.2**	507	
Fluridone	59756-60-4	10.	µg/L	N/A	N/A	**525.2**	507	
Hexazinone	51235-04-2	10.	µg/L	N/A	N/A	**525.2**		
Merphos	150-50-5	1.	µg/L	N/A	N/A	**525.2**	507	
Methyl paraoxon	950-35-6	10.	µg/L	N/A	N/A	**525.2**	507	
Metolachlor *(Dual)*	51218-45-2	10.	µg/L	N/A	N/A	**525.2**	507	508.1
Metribuzin *(Lexone)*	21087-64-9	10.	µg/L	N/A	N/A	**525.2**	507	508.1
Mevinphos	7786-34-7	10.	µg/L	N/A	N/A	**525.2**	507	
MGK 264	113-48-4	5.	µg/L	N/A	N/A	**525.2**	507	
Molinate *(Ordram)*	2212-67-1	1.	µg/L	N/A	N/A	**525.2**	507	634
Napropamide	15299-99-7	10.	µg/L	N/A	N/A	**525.2**	507	
Norflurazon	27314-13-2	10.	µg/L	N/A	N/A	**525.2**	507	
Pebulate *(Tillam)*`	1114-71-2	10.	µg/L	N/A	N/A	**525.2**	507	634
Prometon *(Pramitol)*	1610-18-0	10.	µg/L	N/A	N/A	**525.2**	507	
Prometryn *(Caparol)*	7287-19-6	10.	µg/L	N/A	N/A	**525.2**	507	
Pronamide	23950-58-5	10.	µg/L	N/A	N/A	**525.2**	507	
Propachlor	1918-16-7	10.	µg/L	N/A	N/A	**525.2**	508.1	
Propazine *(Milogard)*	139-40-2	10.	µg/L	N/A	N/A	**525.2**	507	
Simetryn	1014-70-6	1.	µg/L	N/A	N/A	**525.2**	507	
Stirofos	22248-79-9	1.	µg/L	N/A	N/A	**525.2**	507	
Tebuthiuron	34014-18-1	10.	µg/L	N/A	N/A	**525.2**	507	
Terbacil	5902-51-2	10.	µg/L	N/A	N/A	**525.2**	507	
Terbufos *(Counter)*	13071-79-9	0.5	µg/L	N/A	N/A	**525.2**	507	
Terbutryn	886-50-0	3.	µg/L	N/A	N/A	**525.2**	507	
Triademefon	43121-43-3	3.	µg/L	N/A	N/A	**525.2**	507	
Tricyclazole	41814-78-2	20.	µg/L	N/A	N/A	**525.2**	507	
Trifluralin *(Treflan)*	1582-09-8	1.	µg/L	N/A	N/A	**525.2**	508.1	
Vernolate *(Vernam)*	1929-77-7	1.	µg/L	N/A	N/A	**525.2**	507	508.1

[44] The reference for Methods 525.2, 507, and 508.1 is Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, -EPA/600/R-95/131 - August 1995. The reference for Method 622 is Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater –Volume I - EPA-821--93-010-A August 1993, Revision 1.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

Drinking Water SAS Group D: Method 525.2 PCB Congeners List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[45]**		
		RL	**units**	**RL**	**units**			
2-Chlorobiphenyl	2051-60-7	1.	µg/L	N/A	N/A	**525.2**		
2,3-Dichlorobiphenyl	16605-91-7	1.	µg/L	N/A	N/A	**525.2**		
2,2',3,3',4,4',6-Heptachlorobiphenyl	52663-71-5	0.5	µg/L	N/A	N/A	**525.2**		
2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4	0.5	µg/L	N/A	N/A	**525.2**		
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	50186-71-8	0.5	µg/L	N/A	N/A	**525.2**		
2,2',3',4,6-Pentachlorobiphenyl	60233-25-2	1.	µg/L	N/A	N/A	**525.2**		
2,2',4,4'-Tetrachlorobiphenyl	2437-79-8	0.5	µg/L	N/A	N/A	**525.2**		
2,4,5-Trichlorobiphenyl	15862-07-4	0.5	µg/L	N/A	N/A	**525.2**		

Drinking Water – SAS Group E: Semivolatile Organic Compound Extractables List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[45]**		
		RL	**units**	**RL**	**units**			
Benzoic acid	65-85-0	10.	µg/L	N/A	N/A	**1625C**		
Benzyl alcohol	100-51-6	50.	µg/L	N/A	N/A	**1625C**		
Bis(2-chloroethyl)ether	111-44-4	**0.3**	µg/L	N/A	N/A	1625C	**611**	
Bis(2-chloroisopropyl)ether	108-60-1	**4.**	µg/L	N/A	N/A	1625C	**611**	
Bis(2-ethylhexyl)phthalate	117-81-7	**6.**	µg/L	N/A	N/A	1625C	**506**	525.2
Butyl benzyl phthalate	85-68-7	10.	µg/L	N/A	N/A	**1625C**	506	525.2
Carbazole	86-74-8	20.	µg/L	N/A	N/A	**1625C**		
p-Chloroaniline	106-47-8	20.	µg/L	N/A	N/A	**1625C**		
2-Chlorophenol	95-57-8	10.	µg/L	N/A	N/A	**1625C**	604	625
3,3'-Dichlorobenzidine	91-94-1	1.	µg/L	N/A	N/A	1625C	**605**	
2,4-Dichlorophenol	120-83-2	10.	µg/L	N/A	N/A	**1625C**	604	625
Diethyl phthalate	84-66-2	10.	µg/L	N/A	N/A	**1625C**	506	525.2
2,4-Dimethylphenol	105-67-9	10.	µg/L	N/A	N/A	**1625C**	604	625
Dimethyl phthalate	131-11-3	10.	µg/L	N/A	N/A	**1625C**	506	525.2
2,4-Dinitrophenol	51-28-5	50.	µg/L	N/A	N/A	**1625C**	506	525.2
Dinitrotoluene (mixed isomers)	25321-14-16	1.	µg/L	N/A	N/A	1625C	609	525.2
Di-n-octyl phthalate	117-84-0	10.	µg/L	N/A	N/A	**1625C**	506	625
Hexachlorobenzene	118-74-1	**1.**	µg/L	N/A	N/A	1625C	**508.1**	**525.2**
Hexachlorocyclopentadiene	77-47-4	5.	µg/L	N/A	N/A	**1625C**	508.1	525.2
Isophorone	78-59-1	10.	µg/L	N/A	N/A	**1625C**	609	525.2
2-Methylphenol *(o-cresol)*	95-48-7	10.	µg/L	N/A	N/A	**1625C**		
3-Methylphenol *(m-cresol)*	108-39-4	10.	µg/L	N/A	N/A	***1625C***		
4-Methylphenol *(p-cresol)*	106-44-5	10.	µg/L	N/A	N/A	**1625C**		
2-Nitroaniline	88-74-4	**2.**	µg/L	N/A	N/A	**1625C**		
N-Nitrosodiphenylamine	86-30-6	10.	µg/L	N/A	N/A	**1625C**	607	625
N-Nitroso-di-n-propylamine	621-64-7	**0.5**	µg/L	N/A	N/A	1625C	**607**	625
Pentachlorophenol	87-86-5	**0.2**	µg/L	N/A	N/A	**515.2**	515.1	525.2
Phenol	108-95-2	10.	µg/L	N/A	N/A	**1625C**	604	625
2,4,5-Trichlorophenol	95-95-4	10.	µg/L	N/A	N/A	**1625C**	*604*	1653
2,4,6-Trichlorophenol	88-06-2	10.	µg/L	N/A	N/A	**1625C**	604	1653

[45]The reference for Methods 525.2, 506, 507, 515.1, and 515.2 is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III*, EPA/600/R-95/131 - August 1995. The reference for Methods 604, 605, 609, 611, and 625 is Code of Federal Regulations (CFR) 40, Parts 136 - Revised as of July 1, 1995, Exhibit E to Part 136 –*Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*. Methods 1625C and 1653 are USEPA Office of Water "Stand Alone" methods.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

Drinking Water – SAS Group F: Polynuclear Aromatic Hydrocarbons List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[46]**		
		RL	**units**	**RL**	**units**			
Acenaphthene	83-32-9	10.	µg/L	N/A	N/A	**550.1**	1625C	
Anthracene	120-12-7	10.	µg/L	N/A	N/A	**550.1**	1625C	525.2
Benzo[a]anthracene	56-55-3	0.5	µg/L	N/A	N/A	**550.1**		525.2
Benzo[b]fluoranthene	205-99-2	0.2	µg/L	N/A	N/A	**550.1**		525.2
Benzo[k]fluoranthene	207-08-9	0.5	µg/L	N/A	N/A	**550.1**		525.2
Benzo[g,h,i]perylene	191-24-2	2.	µg/L	N/A	N/A	**550.1**		525.2
Benzo[a]pyrene	50-32-8	**0.2**	µg/L	N/A	N/A	**550.1**		525.2
Chrysene	218-01-9	0.8	µg/L	N/A	N/A	**550.1**		525.2
Dibenz[a,h]anthracene	53-70-3	0.6	µg/L	N/A	N/A	**550.1**		525.2
Fluoranthene	206-44-0	10.	µg/L	N/A	N/A	**550.1**	1625C	
Fluorene	86-73-7	10.	µg/L	N/A	N/A	**550.1**	1625C	525.2
Indeno[1,2,3-cd]pyrene	193-39-5	**0.02**	µg/L	N/A	N/A	**550.1**		525.2
Pyrene	129-00-0	10.	µg/L	N/A	N/A	**550.1**	1625C	525.2

Drinking Water – SAS Group G: Pesticides List								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[46]**		
		RL	**units**	**RL**	**units**			
Aldrin	309-00-2	**0.05**	µg/L	N/A	N/A	**525.2**	508.1	505
Chlordane	57-74-9	0.2	µg/L	N/A	N/A	**525.2**	508.1	505
4,4'-DDD	72-54-8	0.5	µg/L	N/A	N/A	**525.2**	508.1	
4,4'-DDE	72-55-9	0.5	µg/L	N/A	N/A	**525.2**	508.1	
4,4'-DDT	50-29-3	0.5	µg/L	N/A	N/A	**525.2**	508.1	
Dieldrin	60-57-1	**0.05**.	µg/L	N/A	N/A	**525.2**	508.1	505
Endosulfan	115-29-7	10.	µg/L	N/A	N/A	**525.2**	508.1	
Endrin	72-20-8	1.	µg/L	N/A	N/A	**525.2**	508.1	505
α-HCH *(α-BHC)*	319-84-6	**0.1**	µg/L	N/A	N/A	**525.2**	508.1	
β-HCH *(β-BHC)*	319-85-7	**0.4**	µg/L	N/A	N/A	**525.2**	508.1	
γ-HCH *(Lindane, γ-BHC)*	58-89-9	**0.2**	µg/L	N/A	N/A	**525.2**	508.1	505
Heptachlor	76-44-8	**0.2**	µg/L	N/A	N/A	**525.2**	508.1	505
Heptachlor epoxide	1024-57-3	**0.2.**	µg/L	N/A	N/A	**525.2**	508.1	505
Methoxychlor	72-43-5	1.	µg/L	N/A	N/A	**525.2**	508.1	505
Toxaphene	8001-35-2	1.	µg/L	N/A	N/A	**525.2**	508.1	505

[46]The reference for Methods 525.2, Revision 2.0; 505, Revision 2.1; and 508.1, Revision 2.0; is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III,* -EPA/600/R-95/131 - August 1995. The reference for Method 550.1 is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I,* EPA/600/4-90/020 - July 1990. Method 1625C is a USEPA Office of Water "Stand Alone" method.

SAS – SEMIVOLATILE ORGANIC ANALYSIS, continued

Drinking Water – SAS Group H: Chlorinated Acid Pesticides and Herbicides								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[47]**		
		RL	**units**	**RL**	**units**			
Acifluorfen	50594-66-6	1.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Bentazon	25057-89-0	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Chloramben	133-90-4	1.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
2,4-D	94-75-7	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Dalapon	75-99-0	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
2,4-DB	94-82-6	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Dicamba	1918-00-9	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
3,5-Dichlorobenzoic Acid	51-36-5	10.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Dichlorprop	120-36-5	5.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
Dinoseb	88-85-7	2.	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
5-Hydroxydicamba	7600-50-2	1.	µg/L	N/A	N/A	**515.1**	515.3 555	
4-Nitrophenol	100-02-7	10.	µg/L	N/A	N/A	**515.1**	515.3 555	
Picloram	1918-02-1	50	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
2,4,5-T	93-76-5	50	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	
2,4,5-TP *(Silvex)*	93-72-1	50	µg/L	N/A	N/A	**515.1**	515.2 515.3 555	

Drinking Water – SAS Group I: N-Methylcarbamoyloxime and N-Methyl-Carbamate Pesticides								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[47]**		
		RL	**units**	**RL**	**units**			
Aldicarb	116-06-3	3.	µg/L	N/A	N/A	**531.1**		
Aldicarb Sulfone	1646-88-4	3.	µg/L	N/A	N/A	**531.1**		
Aldicarb Sulfoxide	1646-87-3	3.	µg/L	N/A	N/A	**531.1**		
Baygon *(Propoxur)*	114-26-1	10.	µg/L	N/A	N/A	**531.1**	632	
Carbaryl *(Sevin)*	63-25-2	10.	µg/L	N/A	N/A	**531.1**	632	
Carbofuran *(Furadan)*	1563-66-2	10.	µg/L	N/A	N/A	**531.1**	632	
3-Hydroxycarbofuran	16655-82-6	5.	µg/L	N/A	N/A	**531.1**		
Methiocarb	2032-65-7	5.	µg/L	N/A	N/A	**531.1**	632	
Methomyl	16752-65-7	10.	µg/L	N/A	N/A	**531.1**	632	
Oxamyl	23135-22-0	10.	µg/L	N/A	N/A	**531.1**	632	

Drinking Water – SAS Group J: Additional Pesticides and Herbicides, Miscellaneous								
Analyte	**CAS Number**	**Aqueous**				**Acceptable Methods[47]**		
		RL	**units**	**RL**	**units**			
Diquat	85-00-7	8.	µg/L	N/A	N/A	**549.1**	549.2	
Paraquat	1910-42-5	10.	µg/L	N/A	N/A	**549.1**	549.2	
Endothall	145-73-3	10.	µg/L	N/A	N/A	**548.1**		
Ethafluralin *(Sonalan)*	55283-68-6	1.	µg/L	N/A	N/A	**627**	1656	
Profluralin *(Tolban)*	26399-36-0	10.	µg/L	N/A	N/A	**627**		
Pendimethalin	40487-42-1	10.	µg/L	N/A	N/A	**1656**		
Fluchloralin *(Basalin)*	33245-39-5	1.	µg/L	N/A	N/A	**646**		
Glyphosate	1071-83-6	100.	µg/L	N/A	N/A	**547**		

[47]The reference for Methods 515.1, Revision 4.1; 515.2, Revision 1.1; and 531.1, Revision 3.1, is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement III*, -EPA/600/R-95/131 - August 1995. The reference for Method 547 is *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA/600/4-90/020 - July 1990. The reference for Methods 548.1, Revision 1.0, and 549.1, Revision 1.0, is *Methods for the Determination of Organic Compounds in Drinking Water Supplement II*, EPA/600/R-92/129 - August 1992. The reference for Methods 627, 646, and 1656 is *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater –Volume I* - EPA-821-R-93-010-A August 1993, Revision 1. Methods 515.3, Revision 1.0, and 549.2, Revision 1.0, are USEPA Office of Water "Stand Alone" methods.

IV. SAMPLE CONTAINERS, PRESERVATIVES AND HOLDING TIMES

A. Containers and Preservatives - Mandatory Specifications

The following specifications apply to all protocols.

1. The Contractor shall provide sample containers prepared with appropriate chemical preservatives for all sample delivery groups, unless instructed otherwise at time of analytical request.

2. The Contractor shall provide prepared, pre-labeled sample containers. Information on the label must include bottle lot number, identification of chemical preservative (if applicable), and lot number of chemical preservative.

3. The Contractor shall provide prepared sample containers that must reach the State so as to allow timely sample collection by State personnel. For Contractors in the greater Indianapolis area, State staff may pick up prepared containers directly from the Contractor. For Contractors located outside of the greater Indianapolis area, prepared containers must be delivered or shipped to the State at the Contractor's expense.

4. The Contractor shall provide sample containers and preservatives that are not contaminated and not capable of reacting with the samples. Documentation indicating that the container lot has passed all QA/QC requirements must be provided by the bottle vendor to the bottle purchaser with each container lot. **A copy of this documentation must be included with each group of containers prepared for the State.**

5. The Contractor shall provide containers and preservatives of sufficient size and quantity to provide adequate sample volume for the required analyses, including QC samples (i.e., matrix spike/matrix spike duplicates and laboratory duplicates). For more information on the numbers and types of containers and preservatives required for the individual tests, see TABLE 1 on the following page. Amounts may vary depending on laboratory requirements.

6. If sufficient sample volume in one container is available for multiple analytes requested; and container, preservative, and holding time requirements are identical for those analytes; then only one container need be furnished for those analytes. For example, if pH, total solids, total dissolved solids, chloride, sulfate, nitrate, nitrite, and alkalinity are all requested to be run on the same water sample, only one 1-liter bottle need be furnished to obtain that water sample (not eight separate bottles).

7. The holding times listed in TABLE 1 are from time of sampling. The Contractor will be responsible for ensuring that sample holding times indicated in TABLE 1 are met. The sampling date on the chain-of-custody must be noted. The State must be given two (2) days for collection and delivery of the samples to the Contractor.

TABLE 1

SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIME REQUIREMENTS

ANALYTES	SOILS, SEDIMENTS, WASTES			AQUEOUS SAMPLES		
	Containers	**Preservatives**	**Holding Times**	**Containers**	**Preservatives**	**Holding Times**
Pathogens						
Coliform, fecal and total	NA			1-1 L P, G	Ice (4^0C)	6 hours
Fecal Streptococci	NA			1-1 L P, G	Ice (4^0C)	6 hours
Cryptosporidium + Giardia	NA			1-1 L P, G	Ice (4^0C)	
Metals						
Total Metals (except Hg and Cr^{+6})	500 mL G	none	6 months	1-1 L P, G	HNO_3 to pH<2	6 months
Dissolved Metals (except Hg and Cr^{+6})	500 mL G	none	6 months	1-1 L P, G	*Filter*, HNO_3 to pH<2	6 months
Suspended Metals (except Hg and Cr^{+6})	500 mL G	none	6 months	1-1 L P, G	**Filter**	6 months
Total Mercury (Hg)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	HNO_3 to pH<2+Ice (4^0C)	28 days
Dissolved Mercury (Hg)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	*Filter*, HNO_3 to pH<2 +Ice (4^0C)	28 days
Chromium, Hexavalent (Cr^{+6})	500 mL G	Ice (4^0C)	24 days to extr; 7 days to analysis	1-250 mL P	Ice (4^0C) **(See EPA Method 1669, Sec. 8.4.5)*	24 hrs *
General Chemistry						
Acidity	NA			1-1 L P, G	Ice (4^0C)	14 days
Alkalinity	NA			1-1 L P, G	Ice (4^0C)	14 days
Ammonia NH_3	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Biochemical Oxygen Demand	NA			1-1 L P, G	Ice (4^0C)	2 days
Biological Oxygen Demand, Carbonaceous	NA			1-1 L P, G	Ice (4^0C)	2 days
Bromide (Br^-)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)	28 days
Carbonate or Bicarbonate	NA			1-1 L P, G	Ice (4^0C)	14 days
Chemical Oxygen Demand COD	NA			1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Chloride (Cl^-)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)	28 days
Chlorine, Residual	NA			1-500 mL P	Ice (4^0C)	Immediately
Cyanide (CN^-)	500 mL G	Ice (4^0C)	14 days	1-1 L P, G	1 ml 50% NaOH +Ice (4^0C)	14 days
Fluoride	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)	28 days
Hardness	NA			1-1 L P, G	Ice (4^0C)+H_2SO_4	6 months

Table 1-Continued

ANALYTES	SOILS, SEDIMENTS, WASTES			AQUEOUS SAMPLES		
	Containers	Preservatives	Holding Times	Containers	Preservatives	Holding Times
General Chemistry (continued)						
Kjeldahl Nitrogen, Total (TKN)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Nitrates (NO_3^{2-})	500 mL G	Ice (4^0C)	2 days	1-1 L P, G	Ice (4^0C)	2 days
Nitrites (NO_2^-)	500 mL G	Ice (4^0C)	2 days	1-1 L P, G	Ice (4^0C)	2 days
Nitrates & Nitrites (NO_3^{2-}+NO_2^-)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Oil and Grease	NA			1-1 L G	Ice (4^0C)+H_2SO_4 to pH<2	28days
Orthophosphate, Dissolved	NA			1-1 L P, G	filter, Ice (4^0C)	2 days
pH	500 mL G	Ice (4^0C)	Immediately	1-1 L P, G	Ice (4^0C)	Immediately
Phenols	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Phosphorus, Total	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Radiochemistry (except Tritium, Radon, I)	NA			2-1L P	HNO_3 to pH<2	6 months
Radiochemistry – Tritium	NA			1-100mL G	none	6 months
Radiochemistry – Radon	NA			3-40 mL G	none	4 days
Radiochemistry – Iodine-131	NA			1-1L P, G	NaOH to pH>8	16 days
Residue, Settleable	NA			1-1 L P, G	Ice (4^0C)	2 days
Residue, Volatile	NA			1-1 L P, G	Ice (4^0C)	7 days
Silica, Dissolved	NA			1-1 L P, G	Ice (4^0C)	28 days
Specific Conductivity	NA			1-1 L P, G	Ice (4^0C)	28 days
Sulfate ($S0_4^{2-}$)	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)	28 days
Sulfide (S^{2-})	500 mL G	Ice (4^0C)	7 days	1-1 L P, G	Zinc Acetate/NaOH	7 days
Sulfite	NA			1-125mL P	Ice (4^0C)	Immediately
Surfactants	NA			1-1 L P, G	Ice (4^0C)	2 days
Total Suspended Solids (TSS)	NA			1-1 L P, G	Ice (4^0C)	7 days
Total Dissolved Solids (TDS)	NA			1-1 L P, G	Ice (4^0C)	2 days
Total Solids (TS) (Total Residue)	NA			1-1 L P, G	Ice (4^0C)	28 days
Total Organic Carbon TOC	500 mL G	Ice (4^0C)	28 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	28 days
Total Organic Halides TOX	500 mL G	Ice (4^0C)	7 days	1-1 L P, G	Ice (4^0C)+H_2SO_4	7 days
Turbidity	NA			1-1 L P, G	Ice (4^0C)	28 days

Table 1 - Continued

ANALYTES	SOILS, SEDIMENTS, WASTES			AQUEOUS SAMPLES		
	Containers	**Preservatives**	**Holding Times**	**Containers**	**Preservatives**	**Holding Times**
Organic Analysis						
Volatile Organic Compounds (VOCs) – Aqueous (except Acrolein and Acrylonitrile)	NA			3-40 mL G	Ice (4^0C)+ H_2SO_4 or HCl to pH<2	14 days
Acrolein and Acrylonitrile – Aqueous	NA			3-40 mL G	Ice (4^0C) and adjust pH to 4-5	14 days
Volatile Organic Compounds (VOCs) – Low Concentration Soil/Sediment	TerraCore, En Core Samplers or VOA vials	Ice (4^0C)+ See 5035A	See Method 5035A	NA		
Volatile Organic Compounds (VOCs) – High Concentration Soil/Sediment/Waste	2-120 mL G	Ice (4^0C)	14 days	NA		
Semivolatile Organic Compounds (SVOCs)	500 mL G	Ice (4^0C)	Extraction 14 days; 40 days analysis	2-1 L Glass	Ice (4^0C)	Extraction 7 days; 40 days to analysis
Pesticides & PCB	500 mL G	Ice (4^0C)	Extraction 14 days; 40 days analysis	1-1 L Glass	Ice (4^0C)	Extraction 7 days; 40 days to analysis
Petroleum Analysis						
Gasoline (GRO)	5035A	5035A	5035A	3-40 glass vials	Ice (4^0C)+2 drops HCL	14 days
Diesel (DRO)	500 mL Glass	Ice (4^0C)	Extraction 14 days; 40 days to analysis	1-1 L Glass	Ice (4^0C)	Extraction 7 days; 40 days to analysis
Oil	500 mL Glass	Ice (4^0C)	Extraction 14 days; 40 days to analysis	1-1 L Glass	Ice (4^0C)	Extraction 7 days; 40 days to analysis
Hazardous Waste Characteristics						
TCLP Metals, VOCs, SVOCs, Pesticides	Same as non-TCLP		Extraction: Same as non-TCLP; plus same as non-TCLP to analysis	Same as non-TCLP		Same as non-TCLP to Extraction and again to analysis
Reactivity (Cyanide and Sulfide)	Same as cyanide and sulfide above		Same as CN and S above	Same as cyanide and sulfide above		Same as CN and S
Ignitability (Flash Point: non-aqueous liquid waste)	250 mL Glass	Ice (4^0C)	As soon as possible	NA		
Corrosivity (pH)	Same as pH above		Same as pH above	Same as pH above		Same as pH above
Corrosivity (Corrosivity to Steel: aqueous and non-aqueous liquid wastes)	2-1 L Glass	none	As soon as possible	2-1 L Glass	none	As soon as possible

V. REPORTING REQUIREMENTS

Analytical Reports

The State will submit samples to the Contractor in sample delivery groups called cases. All State sample identification numbers listed on the chain-of-custody form(s) accompanying a particular sample delivery group will be considered one case. The Contractor shall report results for each individual case in a separate analytical report. Analytical reports must be delivered to the following address:

Indiana Department of Environmental Management
OLQ Chemistry Services Section, Room N1101
100 North Senate Avenue
Indianapolis, IN 46204

Each analytical report will be reviewed by the State upon receipt by the State. This review will include an assessment of compliance with these Technical Specifications.

Report content:

All analytical reports produced for the State **must** include the requirements identified in the Deliverables List in Section V1. (The requirement for deliverables is provided in Section VI, Deliverables List. The elements required are provided in the following Deliverables List Section.) All data must be considered to be "enforcement level" unless the Contractor is instructed differently at the time of the analytical request. The only exception to this is that internal laboratory chain-of-custody shall not be required unless it is specified at the time of the analytical request. Analytical reports provided for this Contract must include the following elements:

a. Sample identification information;
b. Summary of final analytical results for all requested analytes;
Note: Target analytes detected above the detection limit but below the quantitation limit should be reported but flagged as estimated.
c. Signed original chain-of custody form (external);
d. Case narrative explaining all QA/QC or analytical problems encountered, deviations from standard method procedures or requirements (and reason(s) for deviations), and corrective actions taken;
e. Complete QA/QC result summaries and documentation for each analysis; and
f. Raw data for all sample analyses, QC analyses, and calibrations.

The documentation comprising these elements must contain all items listed in the Deliverables List for each type of analysis performed on each sample in the group. In special situations additional documentation may be requested.

Report format:

Reports provided for this Contract must be presented in an organized, clear and understandable format. Reports must be provided on a CD, in Word format, and pages must be sequentially numbered. Data must be presented in the following order:

a. Metals
b. General chemistry
c. Volatile Organic Compounds or BTEX
d. Semivolatile Organic Compounds

e. PAHs
f. PCBs
g. Pesticides
h. Other Semivolatile Organic and Nonvolatile Organic Compounds
i. Special Analytical Services

With regard to QC result summaries, the State considers the Contract Laboratory Program (CLP) -like report forms to be representative of a "clear and understandable format." Forms similar to these are to be used for reporting QC summaries. Reference to these forms are provided in Section VIII. Data **must** be provided in the sequence described in the Deliverable List.

Electronic Document Submittals for Laboratory Case Narrative

The Contractor shall submit electronic document files that are no larger than 75MB. When splitting larger files into 75MB sections, a document identification page should be included as the first page of each subsequent section. The document identification page should include the document title, date, and section (i.e. Part X of Y). The Contractor may compress the electronic document files using the Zip file format (.zip) to reduce the file size.

File names for electronic documents must not include any symbols, i.e.:

- Exclamation point (!)
- Pound sign (#)
- Dollar sign ($)
- Percent (%)
- Ampersand (&)
- Asterisk (*)
- Single quote/apostrophe (‘) or double quotes (“)
- At symbol (@)
- Slash (/) or backslash (\)

Reports should be submitted as Portable Document Format (.pdf) files, version 8.0 or lower. Acceptable file formats for images are .PDF and .TIF. Data files should be formated according to OLQ Electronic Data File Submittals Guidelines. Embedded images or graphics must not be included in documents (non-embedded images and graphics are acceptable).

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Electronic Submittals for Monitoring and Sampling Data

The electronic copy of sampling results should be formatted as an ASCII, tab-delimited text file and contain the facility's name and ID (Federal or State regulatory ID). Field parameters and analytical results must include the fields listed in the electronic data format below. All fields are required unless noted otherwise.

1. SamplingDate: Month, day and year (mm/dd/yyyy). Value should be formatted as a date if possible.
2. SamplePointName: Names of monitoring well, piezometer, soil boring, leachate well, surface water collection point, etc.
3. SampleID: ID of the individual sample collected from a sample point. Required for sample points with multiple sampling horizons such as soil borings, geoprobe samples, surface water samples, or wells with multiple screens within a single riser.
4. LaboratorySampleID: ID assigned to the sample by the laboratory.
5. SampleHorizonName: Name of the individual, depth-dependent sample collected from a sample point. Required for sample points with multiple sampling horizons such as soil borings, geoprobe samples, surface water samples, or wells with multiple screens within a single riser.
6. SampleCollectionElevation: Elevation of the collected sample in feet above Mean Sea Level or Depth below a fixed measurement point. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for sample locations with multiple samples such as soil borings, geoprobe samples, surface water samples, and sediment samples. Optional for fixed depth sample points such as permanent wells.
7. SoilSampleTop: Upper elevation in feet above Mean Sea Level or Depth below a fixed measurement point of the top of the soil sample interval. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for subsurface soil samples.
8. SoilSampleBottom: Lower elevation in feet above Mean Sea Level or Depth below a fixed measurement point of the bottom of the soil sample interval. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for subsurface soil samples.
9. SampleType: Regular, duplicate(s), trip blank(s), equipment blank(s), field blank(s), verification re-sample(s) and replicate(s).
10. SpeciesName (analysis): Chloride, sodium, ammonia, etc. The order of constituents is not critical. However, it is best to reflect the order that is on the laboratory-data sheets and keep all field data grouped together. Metals should indicate the "dissolved" phase or the "total" phase.
11. Concentration (results): The entry MUST be a number. Please do not enter text such as "NA", "ND", or "<".
12. ConcentrationUnits: mg/L, ug/L, mg/Kg, ug/Kg, SU (standard units) for pH, degrees Celsius (oC), or degrees Fahrenheit (oF) for temperature, and umhos/cm for specific conductance.
13. Detected: Yes or no
14. DetectionLimit
15. AnalyticalMethods

16. EstimatedValue: Indicate "Yes" if the reported concentration is an estimated value. If the value recorded was not estimated, enter "No". If a concentration is estimated, use the "Comment" field to explain why the concentration was estimated.
17. Comment: Analytical lab and/or field personnel comments regarding the reported results.
18. SampleMedium: Ground water, Leachate, Surface water, Soil, Sediments, Air, Waste, Sludge or Solids, Container (drum, barrel), or Soil Gas.
19. ProgramArea: Regulatory program for which the sample was collected (e.g. VRP, Solid Waste, Hazardous Waste, LUST, UST, DERP, etc.).

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VI. DELIVERABLES LIST

Quality Assurance/Quality Control Documentation Required for All Protocols

The Deliverables List applies to all protocols. Although the QA/QC requirements in the analytical methods for the two protocols may appear different, the requirements are similar. For example, the two methodologies require that each batch of samples have an aliquot of reagent water that is spiked with analytes of concern and carried through the preparation and analysis process. Whether it is called a Laboratory Control Sample (LCS), Laboratory Fortified Blank (LFB), QC Check Sample, or DI Spike, the principle is the same. An effort has been made to accommodate the terminology of the two protocols in the Deliverables List.

1. General Requirements

The Contractor shall submit the following documentation with all analytical data reported. This is applicable to all sample matrices and all types of analysis.

A. Completed external chain-of-custody form
B. Date and time of receipt at the laboratory
C. Condition of samples upon receipt at the laboratory
E.g.: Temperature of cooler (thermometer reading or presence of ice); condition of bottles (cracked? broken? leaking?); condition of samples (pH reading; preserved? Air bubbles present?)
D. Facility sample identification or number *(e.g., well no.)*
E. Laboratory sample numbers corresponding to facility sample identification
F. Sample preparation, extraction, cleanup, or digestion method(s) and date(s)
G. Analytical method (name, number, and source) and date of analysis
H. Final analytical results
I. Case narrative:
To include deviations from standard analytical or preparatory procedure(s); quality control problems encountered--whether stemming from system, instrumentation, analyst error, or sample matrix; corrective measures taken; if corrective measures as called for in the method were not taken; results of corrective measures taken; etc.
J. Only when requested (for enforcement cases): Completed internal chain-of-custody form.

2. Requirements by Analysis Type

The laboratory documentation listed below must be provided based on the analytical method(s) used:

A. Metals and General Chemistry Analysis Deliverables

B. Organic Analysis Deliverables: GC/MS
C. Organic Analysis Deliverables: GC and HPLC
D. Organic Analysis Deliverables: Pesticides and PCBs

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A. METALS AND GENERAL CHEMISTRY ANALYSIS DELIVERABLES

TOTAL AND DISSOLVED METALS by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), ICP/MS, or Atomic Absorption Spectroscopy (AA) and GENERAL CHEMISTRY ANALYSES

- Method/sample quantitation limits

- Instrument detection limits:

- Calibration records and results:
  *Initial calibration:
  --- Calibration curve established for each metal
  ICP: A blank plus at least one calibration standard (containing all target analytes) with a minimum of two replicate exposures
  AA: (graphite furnace and flame emission) A blank plus at least three standards
  CVAA: (mercury by cold vapor AA) A blank plus at least five standards
  General Chemistry Analysis: A blank plus at least three standards
  Additional requirement for cyanide analyses: a mid-range standard must be distilled and analyzed with results compared to curve for undistilled standards.
  Correlation coefficient of at least 0.995 for each curve (or calibration is repeated)
  Concentrations and responses for each standard and blank (numeric)
  Graphical plot of calibration curve (AA analysis)
  --- Date and time of initial calibration
  If not the same day as analysis, provide explanation. If this is allowed by analytical method, cite section of method.
  *Initial and continuing calibration verification (ICV and CCV) (mid-level standard results and % recovery; CCV to be run every ten samples)

- Blank results
  --- Initial and continuing calibration blank results
  --- Method (preparation) blank results

- Matrix spike (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix) Results to be reported include: sample number of sample spiked, sample concentration for analyte, concentration of spike added, results and % Recovery)

- Matrix spike duplicate or laboratory duplicate at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Analyte concentrations and Relative Percent Difference [RPD]; if matrix spike duplicate, also report %Recovery)

- Laboratory control sample (QC standard or lab-fortified blank: results and %Recovery)

- **Additional deliverables for ICP and ICP/MS analysis:**
  --- Interference check sample (results and % recovery)
  --- Serial dilution results (five-fold analysis) for methods requiring
  --- ICP Linear Range
  --- Interelement correction factors

METALS AND GENERAL CHEMISTRY DELIVERABLES, continued

- **Additional deliverables for ICP/MS analysis:**
  ---ICP/MS Tuning criteria and results
  ---ICP/MS Internal standard intensities for samples, dilutions, calibration blanks, and check standards
  ---Serial dilution analysis results (Method 6020A requires)
  ---Post-digestion spike analysis results (Method 6020A requires)

- **Additional deliverables for AA Method of Standard Addition (MSA)** (if used):
  ---Data and results for MSA, including concentrations of standard added

- **Raw data**: To include instrument numerical printouts, instrument peak printouts (all AA and general inorganic, where applicable), lab worksheets, strip chart recordings, sample preparation records, record of dilutions, and instrumental print outs (or manual worksheets) for initial and continuing calibration runs.

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B. ORGANIC ANALYSIS DELIVERABLES: GC/MS

VOLATILE ORGANIC ANALYSIS (VOA) and SEMIVOLATILE ORGANIC ANALYSIS (SVOA) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

- Tuning criteria and results for:
  ---VOA: Bromofluorobenzene (BFB) or
  ---SVOA: Decafluorotriphenylphosphine (DFTPP)

- Initial calibration data and results:
  --- Calibration standards containing all target analytes run at five concentrations
  --- Retention time (RT) for each target compound in the calibration standards
  --- Response factors (RFs) for each target compound in the calibration standards
  --- Average RF for each compound
  --- Percent relative standard deviation (RSD) for the RFs for the five concentrations of each calibration standard
  --- Date and time of injection
  --- Total ion chromatogram

- Initial and Continuing Calibration Verification data and results (beginning of run and every twelve hours:
  --- RF for each compound in the 50 ppb standard
  --- Percent Difference for RF in 12-hour standard as compared to average RF from initial calibration for each compound
  --- Date and time of injection

- Method blank summary sheet with results, including detections

- Detection/quantitation limit for each compound

- Internal standards summary documented by:
  ---area of primary peak and respective RT for each standard from the 12-hour standard
  ---area of primary peak and respective RT for each standard from each sample
  ---upper and lower acceptance limits clearly defined

- Surrogate (System Monitoring Compound) results (concentration of surrogate spikes added, measured concentrations, and % Recoveries of all surrogates) for each sample

- Matrix Spike/Matrix Spike Duplicate (MS/MSD) results (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix) Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

  OR For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MSD **Note: Check with OLQ QA Officer before substituting laboratory duplicates for the MSD.**

- Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including In-house control criteria when outside the State required control criteria.)

ORGANIC ANALYSIS – VOA AND SVOA BY GC/MS continued

- **Raw Data** for each sample, field duplicate, blank, matrix spike, and matrix spike duplicate including:
  --- total ion chromatogram (indicating surrogates, internal standards, and target compounds detected).
  --- individual mass spectra for target analytes **and tentatively identified compounds (TICs, other non-target analytes) detected in each sample and blank (and reference/library search spectra detected analytes that TICs are compared to).**
  --- quantitation reports (to include identification of internal and surrogate standards, scan number, area, retention time, concentration of target analytes detected, dilution factors, and date and time of injection).
  --- total ion chromatograms and quantitation reports for initial and daily calibrations.

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C. ORGANIC ANALYSIS DELIVERABLES: GC AND HPLC

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS, SEMIVOLATILE ORGANIC COMPOUNDS, AND NONVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY (GC) AND HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) Using Method-Specified Detectors (FID, PID, HECD, UV, etc.) **(Excludes PCBs and Pesticides: See Section D.)**

- Initial Calibration, data and results documented by:
  Either an external standard calibration procedure or an internal standard calibration procedure may be used. Calibration factors (CFs) as defined in SW-846 Method 8000A (July 1992) may be reported in place of response factors.
  --- Calibration standards containing all target analytes run at five concentrations
  --- Calibration chromatograms
  --- Response factors (RFs) or CFs for each target compound in the calibration standards
  --- Average RF (or average CF) for each compound
  --- Percent relative standard deviation (%RSD) for the RFs (or CFs) for the five concentrations of each calibration standard
  --- Date and time of injection (or introduction by purge-and-trap)

- Retention Time (RT) Summary to include:
  --- RT measured for each target compound from three separate injections over a 72-hour period
  --- Mean and standard deviations of the three RTs measured (over the 72-hour period)
  --- RT window for each target compound (mean ± three standard deviations)
  --- Date and time of injections (or introduction by purge-and-trap)

- Initial and Continuing Calibration Verification (ICV and CCV) documented by:
  Note*: An instrument blank, a QC reference sample ("check sample"), and a midrange calibration standard must be injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.)*
  --- Chromatograms for midpoint standard and blank
  --- RT for each analyte (or major peak(s) of each multicomponent analyte, if applicable) in the midrange standard and comparison to daily RT window
  --- Percent Difference (%D) between calculated concentration and nominal ("true") concentration of each target analyte in the QC reference sample
  --- %D between RF or CF of each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard

- Method of sample introduction (direct injection or purge-and-trap)

- Detection/quantitation limit for each compound

- Method blank summary and chromatograms

- Surrogate recoveries for samples, blanks, and spikes

ORGANIC ANALYSIS – GC AND HPLC DELIVERABLES (except PCBs and Pesticides), continued

- Matrix spike/matrix spike duplicate (MS/MSD) analysis (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix) to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

  OR For medium to high concentration soil and waste samples, laboratory duplicates may be used for the MSD **Note: Check with OLQ QA Officer before substituting laboratory duplicates for the MSD.**

- Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including In-house control criteria when outside the State required control criteria.)

- **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, quantitation reports, chromatograms, prep and cleanup records; and chromatograms and quantitation reports from initial and daily calibrations.

- Include confirmation 1 by GC/MS or on second GC column, if required by determinative method or if interference is suspected. Include results and raw data.

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D. ORGANIC ANALYSIS DELIVERABLES: PESTICIDES AND PCBS

QUALITY ASSURANCE/QUALITY CONTROL INFORMATION FOR ANALYSIS OF PESTICIDES and PCBs by Gas Chromatography (GC) with Electron Capture Detector (ECD) or Electrolytic Conductivity Detector (ELCD or HECD)

- Initial Calibration (Include listing of calibration sequence)
  An external standard calibration procedure is preferred, but an internal standard procedure may be substituted. If internal standard procedure is used, report Response Factors (RFs) for each compound at each calibration standard concentration, mean RF, and RF %RSD instead of Calibration Factors (CFs).

  *For Single Component Analytes, initial calibration is documented by:
  --- Five-point calibration preferred; minimum of three-point calibration required.
  --- Calibration chromatograms must be provided.
  --- Retention Time (RT) Summary to include:
  RT measured for each target compound and surrogate at each standard concentration from three-point or five-point calibration
  OR RT measured for each target compound from three separate injections over a 72-hour period
  Mean RT for each target compound and surrogate (mean of three to five RTs from calibration
  OR mean of three RTs measured from injections over a 72-hour period)
  RT window for each target compound and surrogate
  --- Calibration Factor (CF) Summary to include:
  CF calculated for each target compound and surrogate at each standard concentration
  Mean CF for each target compound and surrogate
  % Relative Standard Deviation (%RSD) of the CFs at each standard concentration for each compound
  --- % Breakdown of endrin and % breakdown of DDT
  --- Date and time of injection

  *For multicomponent analytes, initial calibration is documented by:
  --- Three-point or five-point calibration using mixture of Aroclors 1016 and 1260
  --- A "one-point calibration" using a midrange standard must be run for all target multicomponent compounds
  --- Calibration chromatograms must be provided.
  --- Retention Time (RT) Summary:
  For Aroclors 1016 and 1260:
  --- RT measured for at least one major peak at each standard concentration from the three-point or five-point calibration (same peak(s) at each concentration)
  OR RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection)
  -- Mean RT for the chosen major peak(s)
  -- RT window for the chosen major peak(s)

ORGANIC ANALYSIS – PESTICIDES AND PCBs DELIVERABLES, continued

- Initial and Continuing Calibration Verification (ICV and CCV) documented by:
  Note*: An instrument blank, a QC reference sample ("check sample"), and a midrange calibration standard is injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.) For PCBs only Aroclors 1016 and 1260 need be injected unless there are specific known target PCBs at the site. If so, all targeted PCBs must be injected.*

  --- Chromatograms for midpoint standard and blank
  --- Absolute RT for each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard (and comparison to RT window established at calibration)
  --- Percent Difference (%D) between calculated concentration and nominal ("true") concentration of each target analyte in the QC reference sample
  --- %D between RF or CF of each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard
  For multicomponent analytes run at midrange concentration only:
  -- RT measured for three to five major peaks from one-point calibration run
  OR RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection)
  -- Mean RT for the chosen major peak(s)
  -- RT window for the chosen major peak(s)
  --- Calibration Factor (CF) Summary to include:
  CF calculated for each target compound (total area of all peaks used for quantitation) at each standard concentration (or from each of three injections)
  OR CF calculated for three to five major peaks of each target compound from calibration run of midpoint standard
  Mean CF for each target compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only)
  % Relative Standard Deviation (%RSD) of the CFs for each compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only)
  --- % Breakdown of endrin and % breakdown of DDT
  --- Date and time of injection

- Method blank summary and chromatograms

- Detection/quantitation limit for each compound (in each sample)

- Surrogate recoveries for samples, blanks, and spikes

- Matrix spike/matrix spike duplicate (MS/MSD) analysis (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.
  OR For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MSD **Note: Check with OLQ QA Officer before substituting laboratory duplicates for the MSD.**

- Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including in-house control criteria when outside the State required control criteria.)

ORGANIC ANALYSIS – PESTICIDES and PCBs DELIVERABLES, continued

- **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, preparatory records, and chromatograms

- Confirmation of detection **required**: on second GC column *OR* by GC/MS
  --- Chromatograms for samples, blanks, spikes, and standards for confirmation run on second column must be provided.
  --- <u>If confirmation is done by Gas Chromatography/Mass Spectroscopy (GC/MS), the following information (relevant to GC/MS analysis) must also be provided:</u>
  Tuning criteria and results (instrument performance check)
  Calibration records (including total ion chromatogram)
  Chromatograms for samples and method blank
  QC reference sample for detected compounds
  Mass spectra for samples, QC reference sample, and blank, including reference spectra for detected compounds

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VII. TURN AROUND TIMES FOR DELIVERY OF LEVEL IV ANALYTICAL REPORTS

The Contractor shall use a standard turn around time of 30 days. If a turn around time for delivery is not explicitly stated at the time of the analytical request, a 30-day turnaround may be assumed. Occasionally shorter turn around times of 14 days, 7 days, or 48 hours will be requested. Delivery of the complete, fully documented analytical report by the turn around time requested is extremely important to the State for decision-making purposes.

Meeting the turn around time means that the fully documented, Level IV (Full QA/QC) report is delivered by the requested deadline. Paper, fax, or electronic delivery of results without full documentation does **not** constitute meeting the turn around time. The only exception is when a 48-hour turn around is requested. The 48-hour turn around requires that preliminary results be transmitted to the State within 48 hours of sample receipt. The complete, fully documented analytical report is then due within 7 calendar days of the preliminary report.

Financial penalties will be assessed for analytical reports not delivered by the turnaround time. See Section IX, **Payment for Analytical Services**, for details.

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VIII. EXAMPLE QC SUMMARY REPORTING FORMS

The Contractor shall use the referenced documents as the QC summary forms described in Section **V, Deliverables List:**

EPA Contract Laboratory Program Statement of Work for Organic Superfund Methods Multi-Media, Multi-Concentration SOM02.4 October 2016

and

EPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods Multi-Media, Multi-Concentration ISM02.4 October 2016

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IX. PAYMENT FOR ANALYTICAL SERVICES

A. Invoices

The Contractor shall remit an original invoice to the following address:

Laboratory Services Coordinator, OLQ Operations
Indiana Department of Environmental Management
100 North Senate Avenue,
Indianapolis, IN 46204-2251

One invoice must be used for billing all analytical charges for a particular sample delivery group (case). I.e., there must be only one invoice per sample delivery group and only one sample delivery group per invoice.

Invoices for analytical charges must include the following information:

1. Date of invoice,
2. The State's Purchase Order (P.O.) Number (if applicable),
3. The State's Sample Numbers. These will be "LQ numbers" in the format LQ*nnnn*, where "*nnnn*:" represents a sequential four-digit number.
4. Itemization of charges. Charges must be itemized by sample number and analysis type by the cost matrix identification, e.g. Aqueous: Total Metals Group A…, General Chemistry Group F, 'Non-Aqueous…
5. Total charges for the invoice, and
6. A copy of the chain-of-custody for the sample delivery group being billed must be included. (The original chain-of-custody must be included in the analytical report.)

In addition, charges for Special Analytical Services and Additional Analytical Services must be billed on separate invoices form analytical chages for field samples.

B. Payment Approval

The Contractor shall not receive approval for payment until all required data and documentation for the Deliverables List, including raw data package, have been received. Approval of payment for the full amount is contingent on the technical adequacy and timeliness of the analytical report.

1. **Penalties for late delivery of analytical reports**
   It is the responsibility of the Contractor's project manager or quality assurance officer to notify the State if the requested turn around time cannot be met for any reason. Financial penalties will be assessed for analytical reports not delivered by the turnaround time requested, **unless**:

   - The delay is due to circumstances beyond the Contractor's control, **and**
   - The Contractor has notified the State of the reasons for the delay in advance.

a. Amount of penalty

Financial penalties for late delivery will consist of deduction of 5% of the total invoice amount per week that the complete report is late. This applies to all turn around times requested, including 48-hour and 7-day turn arounds. For 48-hour turn around requests, the following criteria will be applied:

b. 48-Hour turn around times

(1) When the preliminary results are received within 48 hours:

The complete analytical report (Deliverables List with raw data) is due within 7 calendar days of transmission of the preliminary report. **Penalties will be assessed for analytical reports for 48-hour turn around requests that are not received within 7 calendar days of the preliminary report.** Such penalties will consist of deduction of 5% of the total invoice amount per week that the complete report is late. These penalties will be waived only under circumstances beyond the Contractor's control and the State has been notified.

(2) When the preliminary results are received 7 or more days after submission to the Contractor (and the State has not been consulted):

If the results will not be received until 7 or more days after sample submittal, the complete Level IV (full QA/QC) analytical report is also due at the time the results arrive. **In effect, the promised 48-hour turn around has become a 7-day turnaround.** Accordingly, the State will adjust the 48-hour charges on the invoice to reflect 7-day rates. If, in addition, the complete Deliverables List with raw data package is not received within 7 calendar days after the analytical results have been received, (i.e., 14 days after sample submittal), late penalties will be assessed in addition to reducing charges to the 7-day rate.

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X. PERSONNEL REQUIREMENTS

A. General Requirements for Contractor's Staff

The Contractor shall, at all times during the performance of the contract, have adequate personnel to ensure that the State receives data that meet the terms and conditions of the Contract, and these Technical Specifications.

All Contractor's staff working on State projects shall have the necessary education, training, technical knowledge, and experience for their assigned functions.

All of the Contractor's staff working on State projects shall be responsible for complying with all QA/QC requirements that pertain to their organizational/technical function. Each such technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures, and records management.

B. Specific Requirements for Key Personnel

1. The Contractor shall designate key personnel who will be responsible for work undertaken under the State laboratory services contract. Resumes of key personnel must be supplied to the State. All key personnel designations are subject to State approval. The positions to be designated include the following:

- **a. Technical Director(s)**
- **b. Quality Assurance Officer**
- **c. Project Manager** for the State Contract
- **d. Laboratory Information Management System (LIMS) Administrator**
- **e. Laboratory Supervisors or Group Leaders (**responsible for the testing areas applicable to the contract)
- **f. Lead Analyst** (for each testing area applicable to the contract)

2. The Technical Director, Quality Assurance Officer, Project Manager, and Laboratory Supervisors must have the qualifications:

- **a.** A bachelors degree in the chemical, environmental, biological, or physical sciences or engineering;
- **b.** A minimum of two years of experience in the appropriate area of environmental analysis for which they are responsible.

3. The LIMS Administrator must have the following qualifications:

- **a.** A bachelors degree in the computer sciences, or in one of the sciences listed in **2.a.**, above, or in engineering; and
- **b.** A minimum of two years of experience in the area of computer science, preferably in a LIMs environment.

4. Each lead analyst shall have the following qualifications:

a. **Identify each lead analyst, the areas of responsibility per analyst, and the level of education for the assigned testing area deemed appropriate by the Technical Director.** Preferably, this will be, at a minimum, an associates degree in the chemical, environmental, biological, or physical sciences or engineering technology;

b. Documented Demonstrations of Capability for all analyses and procedures in their area of responsibility; and

c. A minimum of one year of experience in the analyses that they will perform for the State contract. At the Technical Director's discretion, and with the State's approval, a shorter period experience may be appropriate for areas which do not revolve heavily on interpretation of data (e.g., extraction/digestion).

C. Specific Requirements for All Analysts Performing Work for the State Contract

All chemists and technicians performing analysis for the State must be fully trained in the procedures to which they are assigned. This training must be documented in the Contractor's training records as mandated by the Contractor's Quality System. The training records must be available for inspection by State auditors.

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XI. GENERAL TECHNICAL REQUIREMENTS

A. Quality System

The Contractor shall maintain a documented Quality System (previously known as Quality Assurance/Quality Control Program) capable of demonstrating that the data have a specified degree of reliability. Such a Quality System must be patterned after systems outlined in such publications as:

- NELAC Standard: Approved June 5, 2003, Effective July 1, 2003 and NELAC Standard: Field Sampling and Measurement Organization Sector Adopted May 1, 2007, Updated November 10, 2010
- SW-846, 3rd edition, Chapter One; "Quality Control"(July 1992), Chapter Nine, "Sampling Plan," and Chapter Ten, "Sampling Methods";
- USEPA Contract Laboratory Program Statements of Work, Exhibit E, "Quality Assurance/Quality Control Requirements," and Exhibit F, Chain-of-Custody, Document Control, and Written Standard Operating Procedures"; or
- ISO/DIS 17025, *General Requirements for the Competence of Testing and Calibration Laboratories* (December 1999).

The Contractor shall validate each method used and each analysis performed through this Quality System.

B. Instrumentation

The Contractor shall have a sufficient number and type of functional analytical instruments and a computer system capable to meet all the terms and conditions of the Contract.

1. Analytical instrumentation must include the following at a minimum.
Identify instruments in each category as a, then b, etc.

- **a.** 1 Inductively Coupled Plasma Emission Spectrometer (ICP) or 1 ICP-Mass Spectrometer (ICP-MS) system;
- **b.** 1 ICP modified for trace analysis (e.g., axially-oriented torch), 1 ICP-MS, or 1 Graphite Furnace Atomic Absorption (GFAA) spectrometer;
- **c.** 1 mercury analyzer, 1 Cold Vapor Atomic Absorption (CVAA) spectrometer, or 1 ICP-MS capable of meeting required mercury detection limits;
- **d.** 2 Gas Chromatograph/Mass Spectrometer (GC/MS) systems;
- **e.** 1 Gas Chromatograph/Electron Capture Detector (GC/ECD) with a dual column system (for confirmation), or 2 GC/ECDs with single column systems;
- **f.** 1 Gas Chromatograph/Flame Ionization Detector (GC/FID) with a dual column system (for confirmation), or 2 GC/FIDs with single column systems; and
- **g.** 1 High Performance Liquid Chromatograph (HPLC) with UV and Fluorescence detectors.

2. The following additional instrumentation is recommended but not required:
Identify instruments in each category as a, then b, etc.

- **a.** 2 additional GCs with PID, ELCD, and/or NPD detectors;
- **b.** 1 or more automated inorganic analyzers;
- **c.** 1 ion chromatograph; and
- **d.** Additional instruments of the required types listed in item **1.**, above

C. Facilities

The Contractor shall maintain a facility suitable for the receipt, storage, analysis and delivery of analytical reports (full Deliverables List) meeting all terms and conditions of the contract. **The Contrator shall provide a drawing detailing the areas of the lab for the above activities.**

D. Demonstration of Capability

Before performing work for this Contract, the Contractor shall perform a Demonstration of Capability (DOC) for each combination of preparatory and determinative method that will be used. The DOC is analogous to the "Initial Demonstration of Proficiency" in SW-846 terminology and the: Initial Demonstration of Capability," "Initial Demonstration of Accuracy and Precision," and "Initial Demonstration of Performance" in EPA Office of Water methods. DOC procedures can be found in Attachment C of the NELAC standard for Quality Systems (June 2000) and in the Quality Control sections of the EPA Office of Water analytical methods and SW-846 *organic* analytical methods. In SW-846 methods, the Quality Control section is usually Section 8.0. In EPA Office of Water Methods, the Quality Control Section is usually Section 9.0. (500 and 1600 series) and Section 8.0 (600 series). This demonstration must be made available to the State reviewers when visiting the Contractor's laboratory.

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XII. ANALYTICAL AND QA/QC REQUIREMENTS

SW-846 PROTOCOL

A. INORGANIC AND GENERAL ANALYSIS

Metals and General Chemistry: QA/QC Measures Required (See Detailed Instructions Below.)		
Source	**Title**	**Comments**
SW-846 Chapter One, especially Sections 4 & 5	"Quality Control": especially, Laboratory Operations and Definitions	Use for all analyses: States general QA/QC requirements and provides resource for Method QA/QC measures
SW-846 Chapter Three	"Inorganic Analytes" - Definitions	General information for Metals methods
SW-846 Preparation Method used	See Protocol Analyte Lists , SW-846 Protocol, SW-846 Metals, Metals: Acceptable Sample Preparation Methods	All QA/QC measures in applicable method are required.
SW-846 Determinative Method run	6010C, 7010, etc. See SW-846 Protocol Analyte List	All QA/QC measures in applicable method required.
Deliverables List (from these Technical Specifications)	"Metals and General Chemistry Analysis Deliverables," Deliverables List 2A	QA/QC Documentation Required
Detailed Specifications Below	"A. Inorganic and General Analysis"	Control criteria and corrective action requirements.

1. **Holding Times and Preservatives:**
   The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, based on sample characteristics.

2. **Instrument Detection Limit Determination:**
   The Contractor shall establish the instrument detection limits (IDL) before any field samples are analyzed. The IDLs must meet the specified requirements in the analytical methods. The instrument detection limits should be determined by following the instrument manufacturer's recommendations and the individual method requirements. (See also SW-846 Chapter Three, Method 6010C, and Method 7000A. For each case, the relevant IDLs must be reported on the QC Report. If multiple instruments are used for the analysis of an element or compound within a case, the IDLs for each instrument must be reported.

3. **Method Detection Limit Determinations for Each Matrix**
   The Contractor shall perform and document Method Detection Limit Studies for each analytical method run. A separate detection limit study must be performed for each sample matrix type analyzed and for each instrument used to run a particular method. To determine an MDL for a given analyte in a given matrix, see SW-846 Chapter One, Definitions Section, and Attachment B to 40 CFR Part 136.

4. Initial Calibration

The Contractor shall calibrate all insturments daily or, if the analysis is not run on a daily basis, each time the instrument is set up. Guidelines for instrument calibrations are given in the individual analytical methods.

a. For atomic absorption (AA) systems:

Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. For best results, calibration standards must be prepared fresh each time a batch of samples is analyzed. Low calibration standards must be prepared fresh each time an analysis is to be performed and discarded after each use. Prepare a calibration blank and minimum of three calibration standards in graduated amounts in the appropriate range (linear part of the curve). One atomic absorption calibration standard must be at the reporting limit (RL).

The calibration standards must be prepared using the same type of acid or combination of acids as was used in sample preparation. The concentration of acid(s) in the standards must be at the same concentration as will result in the samples following processing. Beginning with the blank and working toward the highest standard, aspirate or inject the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure a reliable average reading for each solution. Calibration standards for furnace procedures must be prepared as described on the individual sheets for that metal. Linear calibration curves are always required. *The correlation coefficient of the line of the calibration curve must be equal to or greater than 0.995.*

b. For cyanide, mercury, and other inorganics analyses:

Follow the calibration procedures outlined in the analytical method. One calibration standard must be at the RL. Calibration curves must be linear. *The correlation coefficient of the line of the calibration curve must be equal to or greater than 0.995.*

c. For ICP systems

Calibrate the instrument according to instrument manufacturer's recommended procedures.

If an ICP/MS system is used, the mass spectrometer must be tuned to ensure that mass calibration and resolution are within required specifications. This must be done in addition to calibration of the ICP. Also, internal standards corresponding to each analyte must be added to all field samples, quality control samples, and calibration standards.

5. Initial and Continuing Calibration Verification

The Contractor shall verify and document the accuracy of the initial calibration for every analyte by the analysis of an initial calibration verification solution after instrument calibration has been performed with the curve provided. To ensure calibration accuracy throughout the analytical run, a continuing calibration verification standard must be run at periodic intervals.

a. Initial Calibration Verification

The accuracy of the initial calibration is verified by the analysis of at least a calibration blank and a calibration check standard, often referred to as an initial calibration verification standard or solution (ICV). The ICV must be made from a reference material or other independent standard material at or near the mid-range of the calibration curve. An independent standard is defined as a standard composed of the analytes from a different source than that used in the standards for the initial calibration. The initial calibration verifications must be analyzed at the beginning of analysis.

The initial calibration verification standard must be run under the same conditions used for analysis. For ICP analysis, the initial calibration verification solution must be run at each wavelength used in the analysis of the sample. A certified standard analyte solution must be used. If a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range. The measured concentration of the ICV must be within the percentage of its true value indicated in TABLE 2 for the curve to be considered valid. When measurements exceed the control limits of TABLE 2, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

For cyanide analyses in which the analytical method calls for distillation of samples, it is also recommended that at least two standards (a high and a low) be distilled and compared to similar values for undistilled standards on the curve to ensure that the distillation technique is reliable. If distilled standards do not agree within ± 10% of the undistilled standards, the analyst must find the cause of the apparent error before proceeding.

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b. Continuing Calibration Verification

If more than 10 samples per day are analyzed, the working standard curve must be verified by measuring satisfactorily a mid-range standard or reference standard after every 10 samples, or 1 per sample set whenever the sample set is less than 10. Every effort must be made to analyze the State samples as a set. If samples other than the State's are prepared for analysis in a set with the State samples, these samples are to be regarded as part of the 1 in 10 frequency. One continuing calibration verification standard must also be performed for each analyte at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be at or near the mid-range levels of the calibration curve. The standard must be prepared from one of the following solutions:

NIST Standards, or
A laboratory-prepared standard solution.

The same continuing calibration standard must be used throughout the analysis runs for a case (i.e., set or batch) of samples received. A log of spiking solutions, preparation, and sources must be maintained.

If the deviation of the continuing calibration verification is greater than the control limits specified in TABLE 2, the instrument must be recalibrated and the preceding 10 samples reanalyzed for the analytes affected. Information regarding the continuing verification of calibration must be recorded and reported.

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TABLE 2
Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses

Analytical Method	Inorganic Species	% of True Value (EPA Set)	
		Low Limit	High Limit
ICP/AA (except cold vapor)	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide/Sulfide	85	115
Other	General Inorganic & Wet Chemistry	90	110

6. Calibration Blank Analysis

The Contractor shall analyze a calibration (or instrument) blank each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10% during the run directly after the continuing calibration standard is analyzed. The results for the calibration blank solution must be recorded and reported. Blank results are to be reported whether "negative" or "positive". If the absolute value of the blank result is greater than the EQL, terminate analysis, correct the problem, and recalibrate.

7. Method (Preparation) Blank Analysis

The Contractor shall perform at least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample matrix preparation procedure (i.e., one each for water, solids, sludges, oils, etc.) for each case. The blank must be prepared and analyzed with every 10 samples received or with each batch (a group of samples prepared at the same time) of samples digested, whichever is more frequent. The first 10 samples of a case are to be assigned to preparation blank one, and the second 10 samples to preparation blank two, etc. Each data package must contain the results of all the preparation blank analyses associated with the samples in that case. The method blank must be taken through the entire procedure step by step, including all of the reagents and solvents in the quantity required by the method.

This blank is to be reported for each case (i.e., set) and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner.

a. If the absolute value of the blank is less than the RL, no corrective action is required.

b. If the absolute value of the blank is above the RL, the analysis for all samples affected (i.e., all samples prepared with the blank) must be repeated.

8. Spiked Sample Analysis (Matrix Spike)

The Contractor shall add the spike before the digestion and prior to any distillation steps (e.g., cyanide analysis). The spiked sample analysis or matrix spike (MS) is designed to provide information (TABLE 3) about the effect of the sample matrix on the digestion and measurement methodology. At least one spiked sample analysis must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) and concentration (e.g., low, medium) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 0.1% or the sample concentration is ≥ four times the spike concentration.

The matrix spike is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the case of field samples.

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

Analyte spiking levels must reflect the concentration range expected to be measured in the field samples for that analyte. If no estimate of the concentration in the field samples is available, the analyte must be spiked at a concentration between the detection limit and the middle of the linear dynamic range (i.e. mid-range) of the calibration curve for each element analyzed. If the mid-range approach is used, and after samples are analyzed measured concentrations are substantially higher or lower than mid-range for any analyte (i.e., near the EQL or near the upper limit of the linear dynamic range), an additional sample spiked at the concentration observed in the field samples must be analyzed.

If two analytical methods are used to obtain the reported values for the same element for a case of samples (e.g., ICP, GFAA), spike samples must be analyzed by each method used. **Samples identified as field blanks shall not be used for spiked sample analysis**.

The % Recovery (%R) for each component must be calculated and reported in the QC report. Individual component percent recoveries are calculated as follows:

$$\%R = \frac{SSR - SR}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the detection limit, use SR = 0 for purposes of calculating % Recovery. If the spike recovery is not within the limits shown in TABLE 4, or not within the documented historical acceptance limits for the analyte in that matrix, all samples associated with that spiked sample must be reanalyzed.

TABLE 3
Recommended Concentration Levels for Spiked Sample Analysis

	For Standard ICP and Flame AA		For Trace ICP and Furnace AA		Other Analysis Types	
Analyte	Aqueous µg/L	Solid mg/kg	Aqueous µg/L	Solid mg/kg	Aqueous µg/L	Solid mg/kg

9. Matrix Spike Duplicate Sample Analysis

The Contractor shall perform at least one matrix spike duplicate sample (MSD), prepared identically to the spiked sample for each analyte, on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) and concentration (e.g., low, medium)-for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 0.1% or the sample concentrations are ≥ four times the spike concentration

Along with the matrix spike, the matrix spike duplicate is a measure of the bias and variability attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample and spiked duplicate must be from the same project as the case of field samples. If two analytical methods are used to obtain the reported values for the same element for a case of samples (e.g., ICP, GFAA), duplicate samples must be run by each method used. **Samples identified as field blanks shall not be used for matrix spike duplicate sample analysis**.

The MSD % Recoveries and the relative percent differences (RPD) between the MS and MSD for each analyte must be calculated and reported in the QC report. The relative percent differences for each component are calculated as follows:

$$RPD = \frac{|D_1 + D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = Value for First Duplicate (MS % Rec.)
D_2 = Value for Second Duplicate. (MSD % Rec.)

If the matrix spike duplicate % Recovery or RPD are outside the control limits shown in TABLE 4 or outside the documented historical acceptance limits for the analyte in that matrix, the analysis must be repeated for all samples associated with that matrix spike duplicate. When the RPD is large, redigestion is also required.

TABLE 4
Matrix Spike/Matrix Spike Duplicate Control Limits for Inorganic Analyses

Inorganic Species	% of True *(Spiked)* Value *(%R)*		Relative Percent Difference (RPD)
	Low Limit	High Limit	
Metals	75*****	125*****	20
Mercury	75	125	20
Cyanide/Sulfide	75	125	20
General Chemistry	75*****	125*****	20

***In aqueous matrices %R control limits must be set at 80% - 120%.**

10. Duplicate Sample Analysis

The Contractor shall analyze one duplicate sample for each matrix type (e.g., water, sludges, soil) and concentration (e.g., low, medium) for each case of samples, or for each 10 samples received, whichever is more frequent, when a laboratory or matrix duplicate sample is required. The results must not be averaged; results of each replicate must be reported. **Samples identified as field blanks shall not be used for duplicate sample analysis.**

The RPD for each analyte detected must be calculated and reported in the QC report. The RPD is calculated in the same way for matrix duplicates and laboratory replicates as is indicated above for matrix spike duplicates. If the RPD exceeds the control limits listed in TABLE 5, data must be qualified as estimated.

TABLE 5
Control Limits for Laboratory Duplicate Sample Analysis RPD

Concentration of Analyte in Sample & Laboratory Duplicate	Aqueous Samples	Soil, Sludge, Sediment, Oil, & Waste Samples
Both results Less than (<) 5 X EQL	± EQL value	± 2 X EQL value
Both results Greater than (>) 5 X EQL	± 20 %	□□□□□± 35%
One result < EQL, one result > EQL	± EQL value	± 2 X EQL value

11. Laboratory Control Sample Analysis (LCS)

The Contractor shall analyze aqueous and solid laboratory quality control samples for each analyte using the same sample preparation and analytical methods employed for the samples received. One aqueous LCS must be analyzed for every 10 samples received, or for each batch[48] of samples digested, whichever is more frequent. Each data package must contain the results of all the LCS analyses associated with the samples in that case. For cyanide analysis, the distilled mid-range calibration standard may be used as the aqueous LCS. An aqueous LCS is not required for mercury analysis. All aqueous LCS results will be reported in terms of true concentrations with percent recovery as calculated by:

$$\%R = (Observed/True) \times 100$$

Where "observed" is the measured concentration. If the % recovery for the aqueous LCS falls outside the control limits of TABLE 6, the analysis must be terminated, the problems corrected, and the previous samples associated with that LCS reanalyzed (i.e., previous 10 samples or the batch of samples from the case).

TABLE 6
Laboratory Control Sample (LCS) Control Limits for Inorganic Analyses

Inorganic Species	% of True *(Spiked)* Value *(%R)*	
	Low Limit	High Limit
Metals	80	120
Mercury	80	120
Cyanide/Sulfide	80	120
General Chemistry	80	120

[48] A certified standard analyte solution must be used.

12. Serial Dilution Analysis and Post-Digestion Spike Analysis (Spike Recovery Test) for Metals Analysis by ICP and ICP/MS

Note: The State contract requires the serial dilution analysis for <u>all</u> ICP analysis, including Method 6010C.

The Contractor shall analyze and report the results of the Serial Dilution Analysis prior to reporting concentration data for the analyte of interest. The Serial Dilution Analysis must be performed on each group of samples of a similar matrix type (e.g., water, soil) and each concentration (e.g., low, medium) for each case of samples, or for each 10 samples received, whichever is more frequent. **Samples identified as field blanks shall not be used for serial dilution analysis**.

If the analyte concentration is sufficiently high (at least 25 times the estimated detection limit), an analysis of a five-fold (1+4) dilution must be performed. The diluted result must agree with the undiluted result within a % Difference (% D) of 10 after correction for dilution. Agreement within 10% between the concentration of the undiluted sample and five times the concentration of the diluted sample indicates the absence of chemical or physical interferences.

The % Difference is calculated as:

$$\%D = \frac{|I - S|}{I} x100$$

Where: I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading x 5)

For standard ICP: If the % Difference for serial dilution analysis and the original sample does not meet the control limits of TABLE 7, a spike recovery test (post-digestion spike) must be performed to confirm the interference problem. If the spike recovery does not meet the control limits of TABLE 9, all samples in the batch must be analyzed by the method of standard additions.

If all the samples in the batch have analyte concentrations less than 10 times the estimated detection limits, serial dilution analysis must not be performed. Instead, the spike recovery test (analytical spike) must be run. If the spike recovery does not meet the control limits of TABLE 8, all samples in the batch must be analyzed by the method of standard additions.

For ICP/MS: Both the serial dilution analysis **<u>and</u>** the post digestion spike analysis must be run for each analytical batch. (See Method 6020B.)

Serial dilution and spike recovery test results must be reported in the analytical report.

TABLE 7
Serial Dilution Control Limits for Inorganic Analyses

Inorganic Species	% Difference, Dilution vs. Original Determination
Metals	10
Mercury	
Hexavalent Chromium	10

TABLE 8
Spike Recovery Test Control Limits for Inorganic Analyses

Inorganic Species	% Recovery of Post-Digestion Spike	
	Low Limit	High Limit
Metals	85	115
Mercury	85	115
Cyanide/Sulfide	85	115
General Chemistry	85	115

13. Method of Standard Additions (MSA)

The Contractor shall perform analysis by the method of standard additions when matrix interference is indicated This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses 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.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_s of a standard analyte solution of concentration C. To the second aliquot (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_x = \frac{S_B V_B C_S}{(S_A - S_B) V_X}$$

Where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S must be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 must be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 must be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain this intercept concentration.

14. ICP Interference Check Sample Analysis (ICS)

The Contractor shall analyze and report the results for an ICP Interference Check Sample at the beginning and end of each sample analysis run, but not before the initial calibration verification to verify inter-element and background correction factors. The interference check solution must be prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The ICS for standard ICP must be spiked with the elements of interest, particularly those with known interferences, at 0.5 to 1 mg/L. ICP/MS analysis will require analysis and checking of a greater number of interferents. For ICP/MS, it is recommended that the elements of interest be spiked at 0.02 mg/L. Chlorine is an interferent in ICP/MS analysis. The use of chlorine-containing compounds in reagents for sample preparation and analysis must be avoided.

In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero. Therefore, spiked concentrations must be high enough to ensure measurability. If the particular instrument will display over-correction as a negative number, this spiking procedure will not be necessary. Suggested components and concentrations for preparation of the ICS are provided in TABLE 9A-Standard ICP for standard ICP systems and in TABLE 9B-ICP/MS for ICP/MS systems. Other analytes of interest or interferents must be added as necessary to meet project-specific requirements or sample-specific characteristics.

Results for the check sample analysis must fall within the control limits indicated in TABLE 10 (± 20 % of the true value) for the analytes included in the Interference Check Sample. Results of all Interference Check Sample analyses for all ICP parameters must be recorded and reported in the QC report.

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TABLE 9A – Standard ICP

Suggested Interferent and Analyte Elemental Concentrations for Preparation of *Standard ICP* Interference Check Sample

Analyte	**Conc.,** mg/L	**Interferent**	**Conc.,** mg/L
Barium	0.5	Aluminum	500
Beryllium	0.5	Calcium	500
Cadmium	1.0	Iron	200
Cobalt	0.5	Magnesium	500
Chromium	0.5		
Copper	0.5		
Manganese	0.5		
Nickel	1.0		
Lead	1.0		
Vanadium	0.5		
Zinc	1.0		

TABLE 9B - ICP/MS

Suggested Interferent and Analyte Elemental Concentrations for Preparation of *ICP/MS* Interference Check Sample

Analyte	**Conc.,** mg/L	**Interferent**	**Conc.,** mg/L
Arsenic	0.0200	Aluminum	100.0
Cadmium	0.0200	Calcium	100.0
Chromium	0.0200	Iron	100.0
Cobalt	0.0200	Magnesium	100.0
Copper	0.0200	Sodium	100.0
Manganese	0.0200	Phosphorus	100.0
Nickel	0.0200	Potassium	100.0
Silver	0.0200	Sulfur	100.0
Zinc	0.0200	Carbon	200.0
		Chlorine	1000.0
		Molybdenum	2.0
		Titanium	2.0

TABLE 10
Interference Check Sample Control Limits for Inorganic Analyses

Inorganic Species	% Recovery of Analyte True Value	
	Low Limit	High Limit
Metals	80	120

15. ICP Linear Range Analysis

a. Low Level Check Standard

The Contractor shall analyze and report an ICP standard at two times the EQL at the beginning and end of each sample analysis run, or at a minimum of twice per hour working shift, whichever is more frequent, to verify linearity near the EQL for ICP analysis. This standard must be run for all elements analyzed by ICP, recorded, and reported in the QC report. No specific control criteria have been established for the low level check standard (TABLE 11). The analyst and end-user of the data must use professional judgment to qualify data as needed, based on the information provided by the low-level check standard results.

TABLE 11
ICP Linear Range Low Level Check Standard
Control Limits for Inorganic Analyses

Inorganic Species	% Recovery of Analyte True Value
Metals	No specific control criteria established.

b. High Level Check Standard

The Contractor shall analyze and report a quarterly linear range verification check standard for each element (TABLE 12) to verify linearity near the upper end of the linear dynamic range. The concentration of the upper level check standard must be selected based on upper limit establishment procedure described in A. 14, ICP Interference Check Sample Analysis (ICS) of Section XII, Analytical and QA/QC Requirements. The standard must be analyzed during a routine analytical run. The analytically determined concentration of this standard must be within ±5 % of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported.

TABLE 12
ICP Linear Range High Level Check Standard Control Limits for Inorganic Analyses

Inorganic Species	% Recovery of Analyte True Value	
	Low Limit	High Limit
Metals	95	105

c. Establishment of Upper Limit of Linear Range

The Contractor shall establish the upper limit of the linear dynamic range for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these must be near the upper limit of the range. The ranges which may be used for the analysis of samples must be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made must be documented and kept on file. The upper range limit must be an observed signal no more than 10% below the level extrapolated from lower standards. New dynamic ranges must be determined whenever there is a significant change in instrument response.

16. Additional QA/QC Requirements for ICP/MS Analysis

The Contractor shall adhere to the following ICP/MS analysis additional QA/QC measures:

a. Instrument Tuning

Prior to calibration and analysis, the mass spectrometer must be tuned. A solution containing elements representing all of the mass regions of interest (for example, 10 □g/L each of Li, Co, In, and Tl) must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability.

(1) Verification of Thermal Stability: The analyst must follow the instructions provided by the instrument manufacturer. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. This must be verified by analyzing a tuning solution at least four times with relative standard deviations of < 5% for the analytes contained in the tuning solution (TABLE 13).

NOTE: Precautions must be taken to protect the channel electron multiplier from high ion currents. The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. *During this time period, response factors are constantly changing, which invalidates the calibration curve, causes instability, and invalidates sample analyses.*

TABLE 13
ICP/MS Tuning Control Limits to Verify Thermal Stability for Inorganic Analyses

Inorganic Species	Relative Standard Deviation (RSD) for Four Analyses of Analytes in Tuning Solution
Metals	5%

(2) Mass Calibration and Resolution Checks in the Mass Regions of Interest: The mass calibration and resolution parameters are required criteria that must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 AMU from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be less than 0.9 amu full width at 10 percent peak height.

b. Internal Standards (IS)

An appropriate internal standard is required for each analyte determined by ICP-MS. The internal standards aid in quantitation of detected analytes and in identifying when physical or chemical interferences are present in samples.

Generally, an internal standard must be no more than 50 amu removed from the analyte. Recommended internal standards are ^{6}Lithium, ^{45}Scandium, ^{89}Yttriurm, ^{103}Rhodium, ^{115}Indium, ^{159}Terbium, ^{165}Holmium, and ^{209}Bismuth. Other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standards. The lithium internal standard must have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards. The internal standards must be added to the calibration standards, calibration blanks, and preparation blanks as well as to samples and duplicates.

(1) IS Peak Intensities - Field Samples**.** The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed (TABLE 14). The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal-standard intensities fall within the prescribed window.

TABLE 14
ICP/MS Linear Range Peak Intensity Control Limits for Field Samples

Inorganic Species	% of Internal Standard Peak Intensity in Initial Calibration Standard	
	Low Limit	High Limit
Metals	30	120

(2) IS Peak Intensities - QC Samples**.** The intensity levels of the internal standards for the calibration blank and instrument check standard must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution (TABLE 15). If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

TABLE 15
ICP/MS Linear Range Peak Intensity Control Limits for QC Samples[49]

Inorganic Species	% of Internal Standard Peak Intensity in Initial Calibration Standard[49]	
	Low Limit	High Limit
Metals	80	120

c. Rinse Blank
In addition to preparation and calibration blanks, ICP/MS analysis requires a third type of blank, a rinse blank. The rinse blank consists of 1 to 2 percent HNO_3 (volume/volume) in reagent water. It is used to flush the system between each sample analysis.

17. Inorganic Corrective Actions
The Contractor shall find and correct the problem whenever an analytical procedure is "out-of-control,"[49]; also, the analysis must be repeated (which may require redigestion) for all affected samples. It must be noted that for MS/MSD and method blanks, all affected samples would include any sample that was prepared in the same batch with the out-of-control MS/MSD or blank. The analytical procedure is out-of-control:

a. Whenever the absolute value of the method blank results exceeds the detection limit;

b. Whenever matrix spikes, surrogates, laboratory control samples, reference standards, or other laboratory fortified samples results fall outside control limits;

c. Whenever matrix spike duplicates, laboratory duplicates, or matrix duplicate samples results fall outside control limits;

d. Whenever the ICP interference check sample or spike recovery check sample results fall outside control limits;

e. Whenever the ICP serial dilution analysis falls outside control limits.

f. Whenever the intensity of any ICP/MS internal standard in a field sample falls outside the control limits of 30 and 120 percent of the intensity of that internal standard in the initial calibration standard; [49] or

[49]ICP/MS samples out-of-control for internal standard intensity must be diluted fivefold (1+4) prior to reanalysis. Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery), and the sample results are all below detection limits, then reanalysis is not required. However, the contractor must make every effort to correct the problem for future analyses.

g. Whenever the intensity of the ICP/MS internal standards for the calibration blank and instrument check standard fall outside the control limits of ± 20 percent of the intensity level of the internal standard of the original calibration solution**.**

When the control limits are met, reanalysis of "out-of-control" samples must be performed for analytical requirements to be confirmed.[50] If the reanalysis is within control limits, only the results of the reanalysis must be reported. If QC control/criteria following redigestion and reanalysis still fall outside acceptance limits, then the Contractor must submit the data from both analyses. Distinguish between the initial analysis and reanalysis on all data deliverables.

It must be noted that the above is contingent upon the initial and continuing calibrations being in control. There are no exceptions to meeting the criteria for calibration.

B. QUALITY ASSURANCE/QUALITY CONTROL TCLP EXTRACT ANALYSIS

The Contractor shall follow all control criteria specified in Method 1311 for sample handling, preparation, extraction, and analysis.

The following QC measures refer to the performance of determinative analyses on the extract generated according to the specifications of SW-846 Method 1311.

1. TCLP Inorganic Parameters QA/QC Requirements:

a. A three- point calibration curve must have a correlation coefficient (**r**) of 0.995 or greater. The curve must define the range of the instrument. One point must be at or near the detection limit and one point at the mid-range of the curve.

b. A calibration verification sample must be analyzed for every ten samples or one per set if the set contains less than ten samples. The control criterion is a percent recovery between 90% and 110%.

c. A calibration blank and a method blank must be analyzed for every ten samples or one per set if the set contains less than ten samples.

d. A duplicate sample must be analyzed for every ten samples or one per set if the set contains less than ten samples. The RPD control criterion is 20%.

2. TCLP Organic Parameters QA/QC Requirements:

a. An initial calibration is required as provided in A.4., Initial Calibration, and A.5., Initial and Continuing Calibration Verification, of Section XII, Analytical and QA/QC Requirements.

b. A calibration verification sample as required in A. 4. Initial Calibration, and A. 5. Initial and Continuing Calibration Verification, of Section XII, Analytical and QA/QC Requirements, must be analyzed every 12 hours, or one per sample set if analyzed in less than 12 hours.

c. A method blank is to be analyzed for every ten samples or one per sample set if the set contains less than ten samples.

d. For TCLP analysis, a duplicate sample must be analyzed for every 10 samples or one per set if the set contains less than 10 samples.

[50] Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The

acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst's best judgement. Resampling may be necessary in some cases.

C. VOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

VOA: QA/QC Measures Required (See Detailed Requirements below.)		
Source	**Title**	**Comments**
SW-846 Chapter One, especially Sections 4 & 5	"Quality Control": especially, Laboratory Operations and Definitions	Use for all analyses: States general QA/QC requirements and provides resource for Method QA/QC measures
SW-846 Method 5000 (and subsequent updates), especially Section 8.0	"Sample Preparation for Volatile Organic Compounds," Quality Control	Use for **all** VOA.
SW-846 Preparation Method used	3585, 5021, 5030B, 5031, 5032, or *5035A* as appropriate. (See Protocol Analyte List, VOA: Acceptable Sample Preparation & Introduction Methods)	All QA/QC measures in applicable method are required.
SW-846 Method 8000B (and subsequent updates), especially Sections 7.0 and 8.0	"Determinative Chromatographic Separations": especially sections on Procedure and Quality Control	Use for **all** chromatographic analyses: Detailed procedural information and summary of QA/QC requirements
SW-846 Determinative Method run	8260B, 8021B, 8015C as appropriate	All QA/QC measures in applicable method required.
Deliverables List (from these Technical Specifications)	"VOA and SVOA by GC/MS" See Deliverables List 2B and 2C	QA/QC Documentation Required
Detailed Specifications Below	"C. Volatile Organic Analysis by Gas Chromatography/Mass Spectrometry"	Control criteria and corrective action requirements.

TheContractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements based on sample characteristics.

1. Instrument Tuning

The Contractor shall hardware-tune each GC/MS system for accurate mass assignment, sensitivity, and resolution using the compound specified in the analytical method. The tuning criteria specified in the method must be met, prior to the initial calibration procedure. Tuning must be repeated every 12 hours while analysis continues. Analyses must not begin until the criteria specified in the method are met. All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

For volatile organic analysis, tuning is accomplished using a 5-50 ng injection or purging of 4-bromofluorobenzene (BFB), i.e., a 2-□L injection of the BFB standard. (Note that if a more sensitive mass spectrometer is used to achieve lower detection levels, a BFB standard more dilute than the usual 25 ng/µL concentration may be required.) Recommended tuning criteria are listed in TABLE 16.

2. Initial Calibration

The Contractor shall perform and document initial calibration for each instrument used to analyze samples. Initial calibration of volatile organic target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples but must not exceed the working range of the GC/MS system. Project DQOs requiring very low detection limits (e.g. risk assessment) may require specialized calibration and analytical procedures, such as preparation of lower concentration standards to 25 μL volume (and purging 25 μL sample) rather than using 5 μL volumes.[51]

TABLE 16
Recommended BFB Tuning Criteria for VOC Analysis

m/z	Required Intensity (relative abundance)	
50		15 to 40% of m/z 95
75		30 to 60% of m/z 95
95		**Base peak, 100% relative abundance**
96		5 to 9% of m/z 95
173		Less than 2% of m/z 174
174		Greater than 50% of m/z 95
175		5 to 9% of m/z 174
176		Greater than 95% but less than 101% of m/z 174
177		5 to 9% of m/z 176

If an analyte saturates at the highest standard concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity consistent with the project DQOs, the Contractor must document it in the report narrative. In this instance, the Contractor must calculate the results based on a four-point initial calibration *for the specific analyte* that saturates.

The target analytes are quantitated through the calculation of a response factor (RF). A RF is a measure of the relative instrument response of a target analyte as compared to the instrument response of its internal standard. It is calculated as the ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample:

[51] If project DQOs required detection limits lower than 1 part per billion (ppb), it may be necessary to use selective ion monitoring (SIM) techniques or, for aqueous samples, to follow GC/MS Method 524.2 procedures. Another alternative would to be perform analysis by a GC method that does not use MS detection.

$$RF = \frac{A_S x C_{IS}}{A_{IS} x C_S}$$

Where: A_s = Peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard

The internal standard selected for quantitation (i.e., calculation of the response factor) of a particular target analyte must be the internal standard that has a retention time closest to the analyte being measured. The target analytes must be quantitated using the base peak ion (most intense ion, also referred to as primary ion) from the appropriate internal standard. If there are sample interferences with the primary ion, the next most intense ion must be used as the quantitation ion. If this occurs, document the reasons in the report narrative.

Initial calibration of a GC/MS system is performed upon installation of an instrument, prior to beginning analysis of a sample case for an environmental project, whenever corrective action is taken on the system which may change or affect the initial calibration criteria (ion source cleaning or repair, column replacement, etc.), or if the continuing calibration (calibration verification) acceptance criteria have not been met.

Validation of Initial Calibration
A system performance check must be made and documented for the initial calibration to be considered valid. The following criteria must be met:

(1) The mean response factors (RFs) for the volatile System Performance Check Compounds (SPCCs) must be no lower than the minima indicated in TABLE 17. Specific compounds that are especially susceptible to certain analytical problems were selected to be the SPCCs. They are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

(2) The relative standard deviation (RSD) of the response factors for each individual volatile Calibration Check Compound (CCC) must be less than or equal to 30%. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may indicate system leaks or reactive sites in the column. The CCCs are listed in TABLE 17.

(3) The RSD of the response factors for all other target analytes must be less than or equal to 15%.[52]

(4) Retention times must be evaluated for all target analytes. The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units.

(5) Good GC performance must be indicated on the total ion chromatogram. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are unusually broad, or if there is poor resolution between peaks, corrective action is required before analysis can begin.

[52]SW-846 Method 8260B requires a 15% RSD. (See SW-846 3rd ed. Final Update III page 8260B-18.) Water method 524.2 and the CLP GC/MS VOA procedure specify a linearity criterion of 20% RSD.

(6) Adequate MS sensitivity must be demonstrated by the calibration data generated. The GC/MS identification software must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make good tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required.

The RSD is calculated from the mean and standard deviation of the response factors for the five concentration measurements of each analyte:

$$RSD = \frac{SD}{\overline{RF}} x100$$

Where: $\overline{RF}$ = mean RF for that compound from the initial calibration at 5 concentrations
SD = Standard Deviation for that 5 RFs for the compound from the initial calibration

The standard deviation is calculated as a sample standard deviation (not a population standard deviation):

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

Where: RF_i = RF for each of the 5 calibration standards from the initial calibration for that compound
$\overline{RF}$ = mean RF of the 5 concentrations from the initial calibration for that compound
n = Number of calibration standards (e.g. 5)

The criteria listed in TABLE 17 must be met for the initial calibration to be valid. Only after these criteria are met can sample analysis begin:

If the minimum mean response factor criterion for any SPCC is not met, the system must be evaluated and corrective action must be taken before beginning or continuing sample analysis.

If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

If the RSD of any non-CCC analyte is greater than 15%, or the average is greater than 15% a new initial calibration must be performed.[53]

[53]Alternatively, rather than reattempting linear calibration, it may be appropriate to use a non-linear calibration model. The non-linear option must be reserved for appropriate circumstances, such as the need to achieve low detection limits. Non-linear calibration may not be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

3. Calibration Verification

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification consists of the following three steps that must be performed at the beginning of each 12-hour analytical shift. A minimum of one calibration verification must be reported per sample set, even if the set is completed in fewer than twelve hours of analysis time. The calibration verification steps include:

a. BFB is analyzed and results compared to the criteria in the method (or TABLE 16) to verify mass calibration and tuning. The criteria must be met prior to further analysis.

b. A calibration verification standard at a concentration near the midpoint of the calibration range is analyzed and assessed for the following criteria. The calibration standard must contain all target compounds, surrogates, and internal standards.

c. A method blank must be analyzed after the calibration standard to assure that the total system (introduction device, transfer lines, and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to ensure that the contamination is not a result of carryover from standards or samples.

TABLE 17
Initial Calibration Criteria for VOC Analysis

Analyte Type	Compound	Minimum Mean RF	Maximum RSD
SPCC	Chloromethane	0.10	15%
SPCC	1,1-Dichloroethane	0.10	15%
SPCC	Bromoform	0.10	15%
SPCC	Chlorobenzene	0.30	15%
SPCC	1,1,2,2-Tetrachloroethane	0.30	15%
CCC	1,1-Dichloroethene	-	30%
CCC	Chloroform	-	30%
CCC	1,2-Dichloropropane	-	30%
CCC	Toluene	-	30%
CCC	Ethylbenzene	-	30%
CCC	Vinyl chloride	-	30%
ALL OTHER TARGET ANALYTES OR THE AVERAGE		-	15%

Table 17 Continued Additional Calibration Criteria Applicable to All Compounds (Target and QC)	
RT Evaluation	Agreement within ± 0.06 relative retention time units for RTs of each target analyte among the 5 calibration standards.
GC Performance	Symmetrical peaks, minimum tailing, good resolution
MS Sensitivity	99% (minimum) target compound peaks recognized and identified in appropriate retention time window

(1) System performance check. Each SPCC in the calibration verification standard must meet the minimum response factor listed in TABLE 17. If the minimum response factors are not met, the system must be evaluated and corrective action taken before beginning or continuing sample analysis.

(2) Calibration validation The response factors for the CCCs in the calibration verification standard are compared to the mean response factors determined in the initial calibration through a percent difference (%D) calculation.[54] The %D is calculated as follows:

$$\%D = \frac{RF_V - \overline{RF}}{\overline{RF}} x100$$

Where: RF_V = the response factor for the verification standard, and

$\overline{RF}$ = the mean response factor from the initial calibration.

The %D criteria must meet the criteria in TABLE 18 for the initial calibration to be considered valid. If the CCCs are not in or added to the list of target analytes for the project, the %D criteria must be applied to all analytes.

If the criteria in TABLE 18 are not met for any one compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

(3) Calibration Standard Internal Standard Check Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time for any internal standard in the calibration verification standard must not change by more than 30 seconds from the RTs of the internal standards in the mid-range concentration standard of the most recent initial calibration sequence. The peak area counts for the internal standards in the calibration verification standard must change by less than a factor of 2 (-50% to +100%) from the area counts for the internal standard peaks in the mid-range concentration standard of the most recent initial calibration sequence.

[54]RF %D is calculated when the calibration model used is average response factor. If a non-linear regression fit model is used, percent drift is calculated instead. See SW-846 Method 8000B.

If either of these criteria are not met, the mass spectrometer must be inspected for malfunctions, and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Corrections must be documented in the case narrative. Internal standard RT and area count data must be reported for both analyses (before and after corrective action).

TABLE 18
Response Factor %D Calibration Verification Criteria for VOC Analysis

Analyte Type	Compound	Maximum %D
CCC	**1,1-Dichloroethene**	**20**
CCC	**Chloroform**	**20**
CCC	**1,2-Dichloropropane**	**20**
CCC	**Toluene**	**20**
CCC	**Ethylbenzene**	**20**
CCC	**Vinyl chloride**	**20**
Alternatively, if CCCs are not in analyte list: **ALL TARGET ANALYTES**		**20**

4. Blanks

The Contractor shall use the organic-free sample to meet the specific method requirements. A method blank is an organic-free sample (water or soil as appropriate) taken through the entire preparatory and analytical procedure step by step, including all the reagents and solvents in the quantity required by the method. Prior to being subjected to the method procedure, interferents must not be observed in the water at the method detection limit of the compounds of interest.

a. Frequency

For volatile organic compounds analyzed by the purge-and-trap method, the preparation is equivalent to the analysis. Therefore, one purge-and-trap method blank must be analyzed with each group of samples analyzed on the same instrument during the same analytical shift. At a minimum, this frequency must be one method blank per 12-hour shift per instrument.

b. Control Criteria

Analysis of a volatile method blank must meet the following criteria:

(1) Methylene chloride, acetone, toluene, and 2-butanone (common laboratory contaminants) must be present at a concentration no greater than 5 times the estimated quantitation limit (EQL).

(2) Concentrations of target analytes observed in the method blank must be no higher than the highest of:

(a) The Contractor's MDL for the analyte;
(b) 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or
(c) 5% of the measured concentration in the sample.

(3) Failure of control criteria. If any laboratory method blank exceeds these criteria, the Contractor must take corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented. All samples processed with the contaminated method blank must be re-extracted/repurged and reanalyzed**.**

(4) Results and reporting. The Contractor must report results of all volatile method blank analyses. However, the Contractor must not subtract the results of the method blank from those of any associated samples.

5. Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Unspiked Duplicates)
The Contractor shall analyze at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, unspiked duplicates should be run.**

In matrix spike/matrix spike duplicate analysis, predetermined quantities of stock solutions of target analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected and used to assess bias due to sample matrix effects. The relative percent difference (RPD) between the split samples is calculated and used to assess analytical precision. If unspiked duplicates are analyzed, the RPD of detected analytes in the unspiked split samples is used to assess precision.

a. Matrix Spike
The matrix spike analysis is designed to provide information about the effect of the sample matrix on the preparation and measurement methodology. The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples.

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

(1) Selection of sample to be spiked. For many projects, the State will select the sample to be spiked based on site conditions. If the State does not designate a specific sample for spiking, the Contractor must contact the State.

(2) Compounds to be spiked. The State requires that the MS/MSD be spiked with **all** requested target analytes in order to accurately interpret matrix effects on sample results.

(3) Spike concentrations. The concentration of the stock spiking solution and the final concentration of the spike in the sample will be specified in the individual methods of analysis and generally must be followed. However, the concentration may require adjustment to meet project DQOs. For example, if a method modification or a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

(4) Control limits. Recommended control limits for the MS (and MSD, if applicable) % Recovery are listed in TABLE 19. The % Recovery for each component is calculated as follows. When the concentration of the spiked analyte is less than the detection limit in the unspiked sample, use SR = 0 for purposes of calculating % R:

$$\%R = \frac{(SSR - SR)}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result (prior to spiking)
SA = Spike Added

b. MS/MSD or Unspiked Matrix Duplicate Pair

At least one MSD or one unspiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. Recommended RPD control limits are listed in TABLE 19.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = %R Value for First Duplicate (unspiked sample or MS)
D_2 = %R Value for Second Duplicate (unspiked dup. or MSD)

TABLE 19
Recommended MS/MSD and Matrix Duplicate Control Criteria for VOC Analysis

Matrix:	Water		Other Matrices	
Compound	**MS/MSD Spike %Recovery**	**MS/MSD or Duplicate RPD**	**MS/MSD Spike %Recovery**	**MS/MSD or Duplicate RPD**
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21
ALL OTHER ANALYTES	70-130	20	60-140	30

6. Analysis of Surrogates

The Contractor shall use the following recommended surrogates for GC/MS analysis of VOCs: toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. Every blank, standard, and environmental sample (including matrix spike/matrix spike duplicate and matrix duplicate samples) must be spiked with surrogate compounds prior to purging or extraction.

Surrogates must be spiked into samples as directed in the appropriate analytical methods. The concentration of the surrogate spiking solution and final concentration of surrogate in the samples must be appropriate to the project DQOs. For example, if a more sensitive mass spectrometer or method modification is used to achieve lower detection limits, a spiking solution more dilute than the usual 5-25 µg/mL and a final surrogate concentrations lower than 50 µg/L may be required.

a. Control criteria for surrogate recoveries

Required control criteria for volatile surrogate recoveries are listed in TABLE 20.

Surrogate recoveries are calculated as:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

TABLE 20
Required Surrogate Spike Control Criteria for VOC Analysis

Matrix:	Water	Soil & Other Matrices
Compound	**Surrogate Spike %Recovery**	**Surrogate Spike %Recovery**
Toluene-d_8	88-110	81-117
4-Bromofluorobenzene	86-115	74-121
1,2-Dichloroethane-d_4	76-114	80-120
Dibromofluoromethane	86-118	80-120

b. Corrective actions for surrogate recovery problems:

The Contractor shall take the actions listed below if recovery of any surrogate compound is outside of the surrogate recovery limits required in TABLE 20.

(1) Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc. Examine chromatograms for interfering peaks and integrated peak areas. Also, check instrument performance.

(2) If the above steps fail to identify the problem, then reanalyze the sample or extract.

(3) If, after the above steps are followed, surrogate recoveries still do not meet control criteria and the sample was a soil extracted with methanol, then re-extract and reanalyze the sample.

(4) If re-extraction and/or reanalysis of the sample does not solve the problem (i.e., surrogate recoveries are outside the requirements for both analyses), then submit the surrogate spike recovery data and the sample data from <u>both</u> analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. (See **Section E, Corrective Action for Organic Analysis by GC/MS**, for additional information.)

c. Dilution of surrogate response:

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses must be provided to the data user.

Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

7. Internal Standards

The Contractor shall spike all samples (including matrix spike/matrix spike duplicate and matrix duplicate samples), standards, and blanks with the internal standards.

a. Choosing internal standards

The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Depending on the project target analytes, sample matrix, the technique used for introduction of the compounds into the GC/MS system (e.g., purge-and-trap, direct injection, closed-system vacuum distillation, or equilibrium head space), it may be appropriate to use other compounds as internal standards. Other compounds may be used as long as they have retention times similar to the target compounds being detected by GC/MS. The compounds chosen as internal standards must permit most components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards.

b. Control criteria for internal standards

Area counts of the internal standard peaks in the samples (environmental and QC) must be within 50-200% of the area of the corresponding peak in the 12-hour calibration verification standard. The retention times for each internal standard in the sample must not vary by more than 30 seconds. If these criteria are not met, the analysis of all affected samples must be repeated.

c. Assignment of internal standards for quantitation

The internal standard selected for quantitation of a particular target compound must be the internal standard that has a retention time closest to the retention time of the analyte being measured. TABLE 21 lists the possible assignment of target compounds to the recommended internal standards for quantitation.

8. Laboratory Control Sample

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. The LCS consists of an aliquot of an organic free (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the all the target analytes at the same concentrations as the matrix spike, and the % recoveries are calculated. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported. TABLE 22 lists required % Recovery values for LCS analyses.

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TABLE 21
Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Fluorobenzene	**1,4-Difluorobenzene-d4**	**Chlorobenzene-d_5**
Acetone Acetonitrile Acrolein Acrylonitrile Bromochloromethane Bromomethane Carbon disulfide Chloroethane Chloroform Chloromethane Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethane 1,2-Dichloroethane-d_4 *(surr.)* 1,1-Dichloroethene cis-1,2-Dichloroethene trans-1,2-Dichloroethene 2,2-Dichloropropane Iodomethane Methylene chloride Trichlorofluoromethane Vinyl chloride	Benzene Bromodichloromethane Bromoform 2-Butanone Carbon tetrachloride Chlorodibromomethane 2-Chloroethyl vinyl ether Dibromomethane 1,2-Dichloropropane 1,3-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Vinyl acetate	Bromobenzene 4-Bromofluorobenzene *(surr.)* Chlorobenzene 2-Chlorotoluene 4-Chlorotoluene Ethylbenzene Ethyl methacrylate 2-Hexanone Isopropylbenzene 4-Methyl-2-pentanone n-Propylbenzene Styrene 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene Toluene-d_8 *(surr.)* 1,2,3-Trichloropropane Xylenes

TABLE 22
Required Laboratory Control Sample %R Criteria for Organic Analysis

Matrix:	**Water**	**Soil & Other Matrices**
Compound	**LCS %Recovery**	**LCS %Recovery**
ALL TARGET ANALYTES	70-130	60-140

D. SEMIVOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

SVOA by GC/MS: QA/QC Measures Required (See Detailed Instructions Below)

Source	Title	Comments
SW-846 Chapter One, especially Sections 4 & 5	"Quality Control": especially, Laboratory Operations and Definitions	Use for **all** analyses: States general QA/QC requirements and provides resource for Method QA/QC measures
SW-846 Method 3500C (and subsequent updates)	"Organic Extraction and Sample Preparation"	Use for **all** SVOA and NVOA. All QA/QC measures required.
SW-846 Preparation Method used	(See TABLE, Protocol Analyte List SW-846 Protocol, Semivolatile Organic Analysis (SVOA) and Nonvolatile Organic Analysis (NVOA))	All QA/QC measures in applicable method are required.
SW-846 Method 8000B (and subsequent updates), especially Sections 7.0 and 8.0	"Determinative Chromatographic Separations": especially sections on Procedure and Quality Control	Use for all chromatographic analyses: Detailed procedural information and summary of QA/QC requirements
SW-846 Determinative Method run	Method 8270D	All QA/QC measures in applicable method required.
Deliverables List (from these Technical Specifications)	"VOA and SVOA by GC/MS," Deliverables List 2B and 2C	QA/QC Documentation Required
Detailed Specifications Below	"D. Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry"	Control criteria and corrective action requirements.

The Contractor shall extract the samples prior to analysis. Based on sample matrix characteristics, follow requirements in appropriate preparation techniques (including sample cleanup if applicable). Preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Times Requirements, must be adhered to based on sample characteristics. Holding time requirements for both samples and extracts must be met.

1. **Instrument Tuning**

   The Contractor shall hardware-tune each GC/MS system for accurate mass assignment, sensitivity, and resolution using the compound specified in the analytical method. The tuning criteria specified in the method must be met prior to the initial calibration procedure. Tuning must be repeated every 12 hours while analysis continues. Analyses must not begin until the criteria specified in the method are met. All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a decafluorotriphenylphosphine (DFTPP) analysis must use identical mass spectrometer instrument conditions.

   For semivolatile organic analysis, a 50 ng injection of DFTPP is used. (Note that if a more sensitive mass spectrometer is used to achieve lower detection levels, a DFTPP solution more dilute than the usual 50 ng/µL concentration may be required.) Recommended tuning criteria are listed in TABLE 23, below.

2. Initial Calibration

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. Initial calibration of volatile organic target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared. The remaining standards must

TABLE 23
Recommended DFTPP Tuning Criteria for SVOC Analysis

Mass	Ion Abundance Criteria	
51		30-80% of mass 198
68 70		< 2% of mass 69 < 2% of mass 69
127		10-80% of mass 198
197 **198** 199		< 1% of mass 198 **Base peak, or > 50%o of mass 442** 5-9% of mass 198
275		10-60% of Base Peak
365		> 1% of mass 198
441 442 443		Present but > than mass 442 Base Peak, or >50%of mass 198 15-24% of mass 442

correspond to the range of concentrations found in typical samples but must not exceed the working range of the GC/MS system. Project DQOs requiring detection limits below the normal range of electron impact mass spectrometry (e.g. risk assessment) may require specialized calibration and analytical procedures. For example, the use of selective ion monitoring (SIM) is acceptable. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

If an analyte saturates at the highest standard concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity consistent with the project DQOs, the Contractor shall document it in the report narrative. In this instance, the Contractor shall calculate the results based on a four-point initial calibration *for the specific analyte* that saturates.

The target analytes are quantitated through the calculation of a response factor (RF). A RF is a measure of the relative instrument response of a target analyte as compared to the instrument response of its internal standard. It is calculated as the ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample:

$$RF = \frac{A_S x C_{IS}}{A_{IS} x C_S}$$

Where: A_s = Peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard

The internal standard selected for quantitation (i.e., calculation of the response factor) of a particular target analyte must be the internal standard that has a retention time closest to the analyte being measured. The target analytes must be quantitated using the base peak ion (most intense ion, also referred to as primary ion) from the appropriate internal standard. If there are sample interferences with the primary ion, the next most intense ion must be used as the quantitation ion. If this occurs, document the reasons in the case narrative.

Initial calibration of a GC/MS system is performed upon installation of an instrument, prior to beginning analysis of a sample case for an environmental project, whenever corrective action is taken on the system which may change or affect the initial calibration criteria (ion source cleaning or repair, column replacement, etc.), or if the continuing calibration (calibration verification) acceptance criteria have not been met.

Validation of Initial Calibration

A system performance check must be made and documented for the initial calibration to be considered valid. The following criteria must be met:

(1) The mean response factors (RFs) for the volatile System Performance Check Compounds (SPCCs) must be no lower than the minima indicated in TABLE 24. Specific compounds that are especially susceptible to certain analytical problems were selected to be the SPCCs. They are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

(2) The relative standard deviation (RSD) of the response factors for each individual volatile Calibration Check Compound (CCC) must be less than or equal to 30%. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may indicate system leaks or reactive sites in the column. The CCCs are listed in TABLE 24.

(3) The RSD of the response factors for all other target analytes must be less than or equal to 15%.[55]

(4) Retention times must be evaluated for all target analytes. The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units.

(5) Good GC performance must be indicated on the total ion chromatogram. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are unusually broad, or if there is poor resolution between peaks, corrective action is required before analysis can begin.

[55] SW-846 Method 8270C requires a 15% RSD, except that the criterion is 30% for CCCs. (See SW-846 3rd edition. Final Update III page 8270C-16.) Water method 525.2 specifies a linearity criterion of 30% RSD for all analytes.

(6) Adequate MS sensitivity must be demonstrated by the calibration data generated. The GC/MS identification software must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make good tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required. The RSD is calculated from the mean and standard deviation of the response factors for the five concentration measurements of each analyte:

$$RSD = \frac{SD}{\overline{RF}} x100$$

Where: $\overline{RF}$ = mean RF for that compound from the initial calibration at 5 concentrations

SD = Standard Deviation for that 5 RFs for the compound from the initial calibration

The standard deviation is calculated as a sample standard deviation (not a population standard deviation):

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

Where: RF_i = RF for each of the 5 calibration standards from the initial calibration for that compound

$\overline{RF}$ = mean RF of the 5 concentrations from the initial calibration for that compound

n = Number of calibration standards (e.g. 5)

The criteria listed in TABLE 24 must be met for the initial calibration to be valid. Only after these criteria are met can sample analysis begin

If the minimum mean response factor criterion for any SPCC is not met, the system must be evaluated and corrective action must be taken before beginning or continuing sample analysis. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the initial calibration procedure.

If the RSD of any non-CCC analyte is greater than 15%, a new initial calibration must be performed.[56]

[56] Alternatively, rather than reattempting linear calibration, it may be appropriate to use a non-linear calibration model. The non-linear option must be reserved for appropriate circumstances, such as the need to achieve low detection limits. Non-linear calibration may not be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance. See SW-846, Method 8000B, Section 7.5, (3rd edition, December, 1996).

3. Calibration Verification

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification consists of three steps that must be performed at the beginning of each 12-hour analytical shift. A minimum of one calibration verification must be reported per sample set, even if the set is completed in fewer than twelve hours of analysis time.

The calibration verification steps include:

a. DFTPP is analyzed and results compared to the criteria in the method (or TABLE 23*)* to verify mass calibration and tuning. The criteria must be met prior to further analysis.

b. A calibration verification standard at a concentration near the midpoint of the calibration range is analyzed and assessed for the following criteria. The calibration standard must contain all target compounds, surrogates, and internal standards**.**

2 System performance check. Each SPCC in the calibration verification standard must meet the minimum response factor listed in TABLE 24. If the minimum response factors are not met, the system must be evaluated and corrective action taken before beginning or continuing sample analysis.

3 Calibration validation: The response factors for the CCCs in the calibration verification standard are compared to the mean response factors determined in the initial calibration through a percent difference (%D) calculation.[57] The %D is calculated as follows:

$$\%D = \frac{RF_V - \overline{RF}}{\overline{RF}} x100$$

Where: RF_V = the response factor for the verification standard, and

$\overline{RF}$ = the mean response factor from the initial calibration.

The %D criteria must meet the criteria in TABLE 25 for the initial calibration to be considered valid. If the CCCs are not in or added to the list of target analytes for the project, the %D criteria must be applied to all analytes.

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[57] RF %D is calculated when the calibration model used is average response factor. If a non-linear regression fit model is used, percent drift is calculated instead. See SW-846 Method 8000B.

c. Calibration Standard Internal Standard Check

Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time for any internal standard in the calibration verification standard must not change by more than 30 seconds from the RTs of the internal standards in the mid-range concentration standard of the most recent initial calibration sequence. The peak area counts for the internal standards in the calibration verification standard must change by less than a factor of 2 (-50% to +100%) from the area counts for the internal standard peaks in the mid-range concentration standard of the most recent initial calibration sequence.

If either of these criteria are not met, the mass spectrometer must be inspected for malfunctions, and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Corrections must be documented in the case narrative. Internal standard RT and area count data must be reported for both analyses (before and after corrective action).

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TABLE 24
Initial Calibration Criteria for SVOC Analysis

Analyte Type*	Compound	Minimum Mean RF	Maximum RSD
B/N **SPCC**	N-Nitroso-di-n-propylamine	0.050	15%
B/N **SPCC**	Hexachlorocyclopentadiene	0.050	15%
Acid **SPCC**	2,4-Dinitrophenol	0.050	15%
Acid **SPCC**	4-Nitrophenol	0.050	15%
B/N **CCC**	Acenaphthene		30%
B/N **CCC**	1,4-Dichlorobenzene		30%
B/N **CCC**	Hexachlorobutadiene		30%
B/N **CCC**	Diphenylamine		30%
B/N **CCC**	Di-n-octyl phthalate		30%
B/N **CCC**	Fluoranthene		30%
B/N **CCC**	Benzo(a)pyrene		30%
Acid **CCC**	4-Chloro-3-methylphenol		30%
Acid **CCC**	2,4-Dichlorophenol		30%
Acid **CCC**	2-Nitrophenol		30%
Acid **CCC**	Phenol		30%
Acid **CCC**	Pentachlorophenol		30%
Acid **CCC**	2,4,6-Trichlorophenol		30%
ALL OTHER BNA TARGET ANALYTES			15%

*B/N denotes base/neutral fraction compound.
Acid denotes acid fraction compound.
BNA denotes base, neutral, and acid compounds.

Additional Calibration Criteria Applicable to All BNA Compounds (Target and QC)	
RT Evaluation	Agreement within ± 0.06 relative retention time units for RTs of each target analyte among the 5 calibration standards.
GC Performance	Symmetrical peaks, minimum tailing, good resolution. **Anthracene and phenanthrene must be separated by baseline. Benzo[a]anthracene and chrysene must be separated by a valley whose height is less than 25% of the average peak height of these two compounds.**
MS Sensitivity	99% (minimum) target compound peaks recognized and identified in appropriate retention time window

TABLE 25
Response Factor %D Calibration Verification Criteria for SVOC Analysis

Analyte Type	Compound	Maximum %D
CCC	**All Semivolatile CCCs** (Base/Neutral and Acid)	20
Alternatively, if CCCs are not in analyte list: **ALL TARGET ANALYTES**		20

If the criteria in TABLE 25 are not met for any one required compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

4. Blanks

The Contractor shall use the organic-free sample to meet the specific method requirements. A method blank is an organic-free sample (water or soil as appropriate) taken through the entire preparatory and analytical procedure step by step, including all the reagents and solvents in the quantity required by the method. Prior to being subjected to the method procedure, interferents must not be observed in the water at the method detection limit of the compounds of interest.

a. Frequency

One method blank must be extracted and analyzed with each group of samples analyzed on the same instrument during the same analytical shift. At a minimum, this frequency must be one method blank per 12-hour shift per instrument. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

b. Control Criteria

Analysis of a semivolatile method blank must meet the following criteria:

(1) The phthalate esters on the target analyte list (which are common laboratory contaminants in the analysis of semivolatile organic compounds) must be present at a concentration no greater than 5 times the estimated quantitation limit (EQL).

(2) Concentrations of target analytes observed in the method blank must be no higher than the highest of:

(a) The Contractor's MDL for the analyte;
(b) 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or
(c) 5% of the measured concentration in the sample.

(3) Failure of control criteria. If any laboratory method blank exceeds these criteria, the Contractor shall take corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented. All samples processed with the contaminated method blank must be re-extracted/repurged and reanalyzed.

(4) Results and reporting. The Contractor shall report results of all volatile method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

5. Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Unspiked Duplicates)

The Contractor shall analyze at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, unspiked duplicates should be run.

In matrix spike/matrix spike duplicate analysis, predetermined quantities of stock solutions of target analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected and used to assess bias due to sample matrix effects. The relative percent difference (RPD) between the split samples is calculated and used to assess analytical precision. If unspiked duplicates are analyzed, the RPD of detected analytes in the unspiked split samples is used to assess precision.

a. Matrix Spike

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the preparation and measurement methodology. The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples.

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

(1) Selection of sample to be spiked. For many projects, the State will select the sample to be spiked based on site conditions. If the State does not designate a specific sample for spiking, the Contractor shall contact the State. **However, samples identified as field blanks shall not be spiked.**

(2) Compounds to be spiked. The State requires that the MS/MSD be spiked with **all** requested target analytes in order to accurately interpret matrix effects on sample results.

At a minimum, the matrix spike must include the following compounds:

Base/Neutrals	Acids
1, 2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

(3) Spike concentrations. The concentration of the stock spiking solution and the final concentration of the spike in the sample will be specified in the individual methods of analysis and generally must be followed. However, the concentration may require adjustment to meet project DQOs. For example, if a method modification or a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

(4) Control limits. Recommended control limits for the MS (and MSD, if applicable) % Recovery are listed in TABLE 26. The % Recovery for each component is calculated as follows. When the concentration of the spiked analyte is less than the detection limit in the unspiked sample, use SR = 0 for purposes of calculating % R:

$$\%R = \frac{(SSR - SR)}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result (prior to spiking)
SA = Spike Added

b. MS/MSD or Unspiked Matrix Duplicate Pair

At least one MSD or one unspiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. Recommended RPD control limits are listed in TABLE 26.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = %R Value for First Duplicate (unspiked sample or MS)
D_2 = %R Value for Second Duplicate (unspiked dup. or MSD)

6. Analysis of Surrogates

The Contractor shall use the following recommended surrogates for GC/MS analysis of SVOCs: phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. Other compounds may be used as surrogates as necessary or appropriate to meet project objectives. Every blank, standard, and environmental sample (including matrix spike/matrix spike duplicate and matrix duplicate samples) must be spiked with surrogate compounds prior to extraction or processing.

Surrogates shall be spiked into samples as directed in the appropriate extraction method. The concentration of the surrogate spiking solution and final concentration of surrogate in the sample extracts shall be appropriate to the project DQOs. Surrogate concentrations in the sample extracts must generally either be near the middle of the calibration range or approximately ten times the quantitation limit of the surrogate. If a more sensitive mass spectrometer or method modification is used to achieve lower detection limits, a spiking solution more dilute than the usual 100-200 µg/mL may be required.

TABLE 26
Recommended MS/MSD and Matrix Duplicate Control Criteria for SVOC Analysis

Matrix:	**Water**		**Other Matrices**	
Compound	**MS/MSD Spike % R**	**MS/MSD or Duplicate RPD**	**MS/MSD Spike % R**	**MS/MSD or Duplicate RPD**
Phenol	12-110	42	26-100	42
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-100	28	28-104	28
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-100	28	38-107	28
4-Chloro-3-methylphenol	23-100	42	26-103	42
Acenaphthene	46-118	31	31-137	31
4-Nitrophenol	10-100	50	11-114	50
2,4-Dinitrotoluene	24-100	38	28-100	47
Pentachlorophenol	9-103	50	17-109	50
Pyrene	26-127	31	35-142	36
ALL OTHER B/N ANALYTES	25-125	35	25-140	40
ALL OTHER ACID ANALYTES	10-125	50	10-125	50

If the surrogate quantitation limit is unknown, the average quantitation limit of method target analytes may be used to estimate a surrogate quantitation limit. Determine the appropriate surrogate concentration for the blank extracts after all extraction, cleanup, and concentration steps.

a. Control criteria for surrogates

Surrogate spike recoveries must not fall outside the control limits listed in TABLE 27.

Surrogate recoveries are calculated as:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \text{ X } 100$$

TABLE 27
Required Surrogate Spike Control Criteria for SVOC Analysis

Matrix:	**Water**	**Soil & Other Matrices**
Compound	**Surrogate Spike %Recovery**	**Surrogate Spike %Recovery**
Nitrobenzene-d_5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d_{14}	33-141	18-137
Phenol-d_6	10-110	24-113
2-Fluorophenol	21-110	25-121
2,4,6-Tribromophenol	10-123	19-122

b. Corrective action for surrogate recoveries:

The Contractor shall take corrective action if either of the following conditions exists during the analysis of environmental samples for semivolatile parameters:

Recovery of any one surrogate compound in <u>either</u> the base-neutral or the acid fraction is below 10%, or

Recoveries of two surrogate compounds in <u>either</u> the base-neutral or the acid fraction are outside the surrogate spike recovery limits.

If either of these conditions occur, the Contractor shall take the following corrective actions:

(1) Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc. Examine chromatograms for interfering peaks and integrated peak areas. Also check instrument performance.

(2) If the above steps fail to identify the problem, and if control limits or DQOs have not been met, then reanalyze the extract.

(3) If after reanalysis of the extract, surrogate recoveries still do not meet control criteria, and if DQOs have not been met, then re-extract and reanalyze the sample.

(4) If re-extraction and reanalysis of the sample does not solve the problem (i.e., surrogate recoveries are outside the requirements for both analyses), then submit the surrogate spike recovery data and the sample data from <u>both</u> analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. (See **Section E, Corrective Action for Organic Analysis by GC/MS**, for additional information.)

c. Dilution of surrogate response

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses must be reported to the State.

Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

7. Internal Standards

The Contractor shall spike all samples (including matrix spike/matrix spike duplicate and matrix duplicate samples), standards, and blanks with the internal standards.

a. Choosing internal standards

The recommended internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. Depending on the project target analytes, it may be appropriate to use other compounds as internal standards. Other compounds may be used as long as they permit most components of interest in a chromatogram to have retention times of 0.80 - 1.20 relative to one of the internal standards.

b. Control criteria for internal standards

Area counts of the internal standard peaks in the samples (environmental and QC) must be within 50-200% of the area of the corresponding peak in the 12-hour calibration verification standard. The retention times for each internal standard in the sample must not vary by more than 30 seconds. If these criteria are not met, the analysis of all affected samples must be repeated.

c. Assignment of internal standards for quantitation

The internal standard selected for quantitation of a particular target compound must be the internal standard that has a retention time closest to the retention time of the analyte being measured. TABLE 29 lists the possible assignment of target compounds to the recommended internal standards for quantitation.

8. Laboratory Control Sample

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. The LCS consists of an aliquot of an organic free (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the all the target analytes at the same concentrations as the matrix spike, and the % recoveries are calculated. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846 Method 8000B, Section 8.5.5.) If re-extraction and reanalysis of the sample does not solve the problem and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. [58] Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.

TABLE 28
Recommended Laboratory Control Sample %R Criteria for Organic Analysis[59]

Matrix:	**Water**	**Soil & Other Matrices**
Compound	**LCS %Recovery**	**LCS %Recovery**
ALL TARGET ANALYTES	70-130	60-140

The range for LCS recoveries provided in TABLE 28 may not be achievable for some semivolatile target analytes, in which case the acceptance tables provided at the end of the method or the Contractor's historical recoveries may provide more realistic ranges. LCS percentage recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 28 are to be supported by the Contractor's historical data which are also provided in the report.

[58] Resampling may be required in some cases. If reanalysis is performed past the holding time, both analysis results must be reported.

[59] Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

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TABLE 29
Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
Aniline Benzyl alcohol Bis(2-chloroethyl)ether Bis(2-chloroisopropyl)ether 2-Chlorophenol 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Ethyl methanesulfonate 2-Fluorophenol *(surr.)* Hexachloroethane Methyl methanesulfonate 2-Methylphenol 4-Methylphenol N-Nitrosodimethylamine N-Nitroso-di-n-propylamine Phenol Phenol-d_6 *(surr.)* 2-Picoline	Acetophenone Benzoic acid Bis(2-chloroethoxy)methane 4-Chloroaniline 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2,6-Dichlorophenol □,□-Dimethylphenethylamine 2,4-Dimethylphenol Hexachlorobutadiene Isophorone 2-Methylnaphthalene Naphthalene Nitrobenzene Nitrobenzene-d_8 *(surr.)* 2-Nitrophenol N-Nitrosodi-n-butylamine N-Nitrosopiperidine 1,2,4-Trichlorobenzene	Acenaphthene Acenaphthylene 1-Chloronaphthalene 2-Chloronaphthalene 4-Chlorophenyl phenyl ether Dibenzofuran Diethyl phthalate Dimethyl phthalate 2,4-Dinitrophenol 2,4-Dinitrotoluene 2,6-Dinitrotoluene Fluorene 2-Fluorobiphenyl *(surr.)* Hexachlorocyclopentadiene 1-Naphthylamine 2-Naphthylamine 2-Nitroaniline 3-Nitroaniline 4-Nitroaniline 4-Nitrophenol Pentachlorobenzene 1,2,4,5-Tetrachlorobenzene 2,3,4,6-Tetrachlorophenol 2,4,6-Tribromophenol *(surr)* 2,4,5-Trichlorophenol 2,4,5-Trichlorophenol	4-Aminobiphenyl Anthracene 4-Bromophenyl phenyl ether Di-n-butyl phthalate 4,6-Dinitro-2-methylphenol Diphenylamine Fluoranthene Hexachlorobenzene N-Nitrosodiphenylamine Pentachlorophenol Pentachloronitrobenzene Phenacetin Phenanthrene Pronamide	Benzidine Benzo(a)anthracene Bis(2-ethylhexyl)phthalate Butyl benzyl phthalate Chrysene 3,3'-Dichlorobenzidine p-Dimethylaminoazobenzene Pyrene Terphenyl-d_{14} *(surr.)* Di-n-octyl phthalate Indeno(1,2,3-cd)pyrene 3-Methylcholanthrene	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Benzo(a)pyrene Dibenz(a,j)acridine Dibenz(a,h)anthracene 7,12-Dimethylbenz[a]anthracene Di-n-octyl phthalate Indeno(1,2,3-cd)pyrene 3-Methylcholanthrene

E. CORRECTIVE ACTION for Organic Analysis by GC/MS (VOCs and SVOCs)

The Contractor shall find and correct the problem whenever an analytical procedure is "out-of-control" (fails to meet control criteria); also, the analysis must be repeated (which may require re-extraction) for all affected samples. The analytical procedure is out-of-control when any one or more of the following conditions occurs:

a. **Whenever the *tuning results* do not meet control criteria: STOP! The instrument must be retuned and recalibrated before proceeding with analysis!** (See Section **C.1.** (VOCs) and Section **D.1.** (SVOCs) for details.)

b. **Whenever the *initial calibration results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis!** (See Section **C.2.** (VOCs) and Section **D.2.** (SVOCs) for details.)

c. **Whenever the *calibration verification results* (CCC and SPCC) do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis!** (See Section **C.3.** (VOCs) and Section **D.3.** (SVOCs) for details.)

d. Whenever the method blank results exceed the detection limit. (See Section **C.4.** (VOCs) and Section **D.4.** (SVOCs) for details.)

e. Whenever matrix spikes, surrogates, internal standards, or other laboratory fortified sample results fail to meet control criteria.[60] (See Sections **C.5.**, **C.6., C.7.**, and **C.8.** (VOCs) and Sections **D.5.**, **D.6., D.7**., and **D.8.** (SVOCs) for details.)

f. Whenever matrix spike duplicate or matrix duplicate results fall outside control limits. (See Section **C.5**. (VOCs) and Section **D.5.** (SVOCs) for details.); or

g. Whenever the chromatographic performance or mass spec sensitivity is poor (e.g., rising baseline, peak broadening, tailing, poor resolution, etc.).

When the out-of-control conditions listed in items **c.** through **g.** above occur, re-extraction (if applicable) and reanalysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and method blank failure, the affected samples would include all field samples prepared or purged with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.[61]

[60]The corrective action for internal standards does not require re-extraction of samples affected by out-of-control results. However, reanalysis of the affected sample is required. (See Sections **C.7 and D7., Internal Standards.**)

[61]The State's position on holding times for reanalysis of out-of-control results is that it would be preferred that sample analysis be performed within holding times, but if that is not possible, reanalysis, based on analytical requirements, may still need to be performed for analytical obligations to be considered met. The acceptance of results analyzed beyond holding time requirements will be predicated on DQO and threshold requirements along with analyst's best judgment.

F. PESTICIDES AND PCBs ANALYSES by Gas Chromatography/Electron Capture Detector

Pesticides and PCBs by GC/ECD: QA/QC Measures Required (See Detailed Instructions Below		
Source	**Title**	**Comments**
SW-846 Chapter One, especially Sections 4 & 5	"Quality Control": especially, Laboratory Operations and Definitions	Use for **all** analyses: States general QA/QC requirements and provides resource for Method QA/QC measures
SW-846 Method 3500C (and subsequent updates)	"Organic Extraction and Sample Preparation"	Use for **all** SVOA and NVOA. All QA/QC measures required.
SW-846 Preparation Method used	As appropriate. (See Protocol Analyte Lists, SW-846, (Semi-Volatile Organic Analysis (SVOA) and Non-Volatile Organic Analysis (NVOA))	All QA/QC measures in applicable method are required.
SW-846 Method 8000B (and subsequent updates), especially Sections 7.0 and 8.0	"Determinative Chromatographic Separations": especially sections on Procedure and Quality Control	Use for all chromatographic analyses: Detailed procedural information and summary of QA/QC requirements
SW-846 Determinative Method run	8081B or 8082A as appropriate	All QA/QC measures in applicable method required.
Deliverables List (from these Technical Specifications)	"Analysis of Pesticides and PCBS by GC with ECD or ELCD Detector," (See Deliverables List 2D)	QA/QC Documentation Required
Detailed Specifications Below	"F. Analysis of Pesticides and PCBs by Gas Chromatography / ECD"	Control criteria and corrective action requirements.

Although most State requests for GC/ECD analysis will be regarding PCBs and organichlorine pesticides, GC/ECD can also be used to analyze other types of halogenated hydrocarbon and chlorinated herbicides. The principles in this section can be used as guidance for such compounds, substituting appropriate surrogates and internal standards.

1. General Requirements and Considerations

The Contractor shall adhere to the following requirements:

a. Extraction and cleanup: Samples must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. Most samples will require cleanup of extracts before determinative analysis to remove phthalate esters, sulfur, and other non-target interferents.

b. Holding times and preservatives: Preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, must be followed based on sample characteristics. Holding time requirements for both samples and extracts must be adhered to.

d. Compound identification: Compound identification based on single-column analysis must be confirmed on a second column, or must be supported by at least one other qualitative technique. GC/MS may be used as qualitative confirmation *if sensitivity permits (i.e.,* GC/MS may be used if the detected compound is present in high enough concentration to be detectable by standard GC/MS, or if a more sensitive GC/MS system or method modification is utilized to achieve low enough detection limits.)

e. Multicomponent analytes: When samples contain more than one target analyte that is a multicomponent mixture (e.g., Chlordane, Aroclors), a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multicomponent analytes that have been subjected to environmental degradation (weathering) or degradation by treatment technologies. Such weathered multicomponent mixtures may have significant differences in peak patterns than those of standard extracts.

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2. Initial Calibration

The Contractor shall use an external standard calibration procedure for analysis of pesticides and Aroclors because of the sensitivity of the electron capture detector.[62] Surrogates and, if applicable, internal standards must be present in the calibration standards at the same concentration as the sample extracts.

a. Calibration Standards

(1) Single-component analytes (including individual PCB congeners): Calibration standards for single-component analytes may be prepared separately for each analyte or as an analyte mixture. If there are a large number of target analytes (e.g., the full analyte list for SW-846 Method 8081A), and standard mixtures are used, it is recommended that the target analytes be divided between two separate calibration mixtures. This will minimize potential resolution and quantitation problems and allow determination of DDT and Endrin breakdown.

For each surrogate and analyte of interest, prepare calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. One of the external standards must be at a concentration near, but above, the method detection limit, and one must be at or near the midrange of the curve. The other concentrations must correspond to the expected range of concentrations found in real samples or must define the working range of the detector. For each analyte, at least one of the calibration standards must correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

(2) Chlordane, Toxaphene, and similar multi-component analytes (other than Aroclors): Separate external calibration standards are required for each multi-component target analyte. Standard mixtures must not be used.[63]

Once the linear range has been established for the instrument and column for which the analysis is being performed, a single-point calibration may be used for multi-component analytes (unless a three-point or five-point calibration is necessary to meet the DQOs for a specific project).[64] A single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column.

[62]Exception: Internal standard calibration is recommended when PCBs are to be determined as individual congeners.
[63]The exception to this is Aroclors 1016 and 1260. See Section 2.3.a.3, Aroclors, below.
[64]This does not apply to Aroclors. See Section 2.3. Aroclors, below.

(3) Aroclors

(a) When all seven Aroclors are target analytes as part of a standard analyte list: A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures (i.e., 1221, 1232, 1242, 1248, and 1254). As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations is sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors.

Such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260 that are present in a sample. However, this standard cannot be used to identify or quantitate Aroclors other than 1016 or 1260.

Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with an appropriate solvent. The concentrations must correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector.

Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described above have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors can also be used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors. The concentrations must correspond to the mid-point of the linear range of the detector.

(b) When specific site-related Aroclors are target analytes: In situations where only a few Aroclors are of interest for a specific project, a five-point initial calibration of each site-related Aroclor must be run. In this case, the 1016/1260 mixture and the pattern recognition standards described in **Section F (3) (a)**, above, need not be run. Prepare the standards as indicated in **Section F (2) (a).**

b. Calibration Process (External Standard Procedure)[65]

Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1-3 □L injections). Tabulate peak height or area responses against the mass injected. In the case of multi-component analytes, a minimum of 3 peaks (and preferably 5 peaks) must be chosen for each multi-component analyte and responses for each of these peaks tabulated. The results are used to prepare a calibration curve or to calculate a calibration factor (CF) for each analyte. The CF is defined as the ratio of the detector response to the amount (mass) injected. It can be calculated for each analyte at each standard concentration as follows:

$$CF = \frac{\text{Total Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$ [66]

[65] If an internal standard calibration procedure is used (e.g., for PCB congeners) peak responses are tabulated against concentrations rather than mass. A response factor is calculated instead of a calibration factor. See **Section F. 2.** and SW-846 Method 8000B for the RF calculation and linearity determination.

[66] For multi-component pesticides and Aroclors, a separate CF is calculated for each characteristic peak in the mixture. Each multicomponent analyte will have 3-5 CFs for each concentration of calibration statndard.

c. Initial Calibration Control Criteria

The mean and standard deviation of the calibration factors across the five concentrations for each analyte are calculated; from these the relative standard deviation for each analyte is calculated:

$$RSD = \frac{SD}{\overline{CF}} x100$$

If the relative standard deviation (RSD) of the calibration factor is ≤20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve. If linearity through the origin cannot be assumed (i.e., the criteria in TABLE 30 cannot be met), the analysis must be stopped and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean calibration factor. (See SW-846 Method 8000B, December 1996.)[67]

TABLE 30
Initial Calibration CF RSD Criteria for GC Analysis

Compound	RSD for Standard CFs across all concentrations
EACH TARGET ANALYTE	≤ 20 %

A new calibration curve (or calibration factor) must be prepared whenever a new column or detector is installed. The initial calibration data, calibration factors, and RSDs calculated must be reported with the analysis results.

3. Establishment of Retention Time Windows

The Contractor shall adhere to the following requirements:

a. Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multi-component analytes over the course of a 72-hour period.

b. Record the retention time for each single component analyte and surrogate to three decimal places.

c. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks and calculate the mean and standard deviation for each of those peaks.

d. If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

e. 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. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

[67] SW-846 Method 8000B, Section 7.5, (3rd edition Final Update III, December 1996) provides criteria for linear as well as for non-linear alibration models. A linear calibration curve is preferred. In some situations it may be appropriate to use a non-linear calibration model. The non-linear option must be reserved for appropriate circumstances, such as the need to achieve low detection limits. Non-linear calibration may not be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

f. The Contractor shall establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

g. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each GC column and instrument. New retention time windows must be established whenever a new GC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

4. Calibration Verification

The Contractor shall verify the calibration relationship established during the initial calibration by injecting a calibration verification standard at periodic intervals:

a. At the beginning of each 12-hour analytical shift, prior to conducting sample analyses. Analysts must alternate the use of high and low concentration mixtures of single-component analytes and multi-component analytes for calibration verification.

b. A calibration verification standard must also be injected at intervals of, at a minimum, once every 20 samples and at the end of the analysis sequence. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring re-injection when QC limits are exceeded).

c. Calibration verification control criteria

(1) The calibration factor for each analyte must not exceed a ± 15 percent difference from the mean calibration factor calculated for the initial calibration. The percent difference is calculated as:

$$\%D = \frac{CF_V - \overline{CF_i}}{\overline{CF_i}} x100$$

Where: CF_V = the calibration factor from the analysis of the calibration verification standard, and

$\overline{CF_i}$ = the mean calibration factor calculated for the initial calibration

(2) The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 31 are not met for any analyte, then corrective action must be taken prior to continuing analysis of samples. If attempts to correct the problem are unsuccessful, a new initial calibration must be performed. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed.

The Contractor shall report the results from the calibration verifications.

TABLE 31

Calibration Verification Control Criteria for GC Analysis

Compound	Calibration Factor % D	Retention Time
ALL TARGET ANALYTES	± 15	In Window (established with initial calibration midlevel standard RT)

Additional Calibration Criteria Applicable to All Compounds (Target and QC)	
GC Performance	Symmetrical peaks, minimum tailing, good resolution

5. Degradation

The Contractor shall check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin as DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with buildup of high boiling residue from sample injection, or when the injector contains metal fittings. Presence of the degradation products of 4,4□-DDT (4,4'-DDE and 4,4'-DDD) and Endrin (Endrin ketone or Endrin aldehyde) indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding further with calibration. Calculate percent breakdown as follows:

$$\%\text{Breakdown of DDT} = \frac{\text{Sum of degradation peak areas (DDD + DDE)}}{\text{Sum of all peak areas (DDT + DDD + DDE)}} x100$$

$$\%\text{Breakdown of Endrin} = \frac{\text{Sum of degradation peak areas (aldehyde + ketone)}}{\text{Sum of all peak areas (Endrin + aldehyde + ketone)}} x100$$

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour analytical shift. Injector maintenance and recalibration must be completed if the breakdown exceeds the criteria in TABLE 32 for either compound. The Contractor shall report the results from the degradation/ breakdown calculations.

TABLE 32
Degradation Control Criteria for GC Analysis of Pesticides

Compound	% Breakdown Criteria
4,4'-DDT	≤15%
Endrin	≤ 15%

6. Blanks

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples. A method blank is an organic-free sample (water or soil as appropriate) carried through all stages of the sample preparation and measurement steps. The organic-free sample used must meet the specific method requirements. Prior to being subjected to the method procedure, interferents must not be observed at the method detection limit of the compounds of interest.

a. Frequency. One method blank must be extracted and analyzed with each group of samples analyzed on the same instrument during the same analytical shift. At a minimum, this frequency must be one method blank per 12-hour shift per instrument. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

b. Control Criteria. Analysis of a method blank for analysis of pesticides, PCBs, and other semivolatile organic compounds by GC/ECD must meet the following criteria:

(1) Interferences by phthalate esters introduced during sample preparation can cause a major problem in analysis of pesticides, PCBs, and other semivolatile organic compounds. The phthalate esters on the target analyte list must be present at a concentration no greater than 5 times the pratical quantitation limit (PQL).

(2) Concentrations of target analytes observed in the method blank must be no higher than the highest of:

a. The Contractor's MDL for the analyte**,**
b. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit), or
c. 5% of the measured concentration in the sample.

c. Failure of control criteria. If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the above control criteria), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

If method blank contamination cannot be attributable to carryover, the Contractor shall take corrective action. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

d. Results and reporting. The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

7. Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Unspiked Duplicates)
The Contractor shall spike at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, unspiked duplicates should be run.**

In matrix spike/matrix spike duplicate analysis, predetermined quantities of stock solutions of target analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected and used to assess bias due to sample matrix effects. The relative percent difference (RPD) between the split samples is calculated and used to assess analytical precision. If unspiked duplicates are analyzed, the RPD of detected analytes in the unspiked split samples is used to assess precision.

a. Matrix Spike

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the preparation and measurement methodology. The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

(1) Compounds to be spiked: Matrix spiking solutions must be prepared from compounds that are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all single-component target analytes in order to accurately interpret matrix effects on sample results.

(a) Pesticides analysis: At a minimum, the matrix spike must contain γ-BHC (Lindane), Heptachlor, Aldrin, Dieldrin, Endrin, and 4, 4'-DDT.

(b) PCBs Analysis: When samples are known or expected to contain specific Aroclors or PCB congeners, the target Aroclors or congeners must be spiked. If samples are not expected to contain target analytes, the Aroclor 1016/1260 mixture (or, at a minimum Aroclor 1260) must be spiked.

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

(2) Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the regulatory limit, health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

(3) Control limits. Recommended control limits for the MS (and MSD, if applicable) minimum spiked compounds' % Recovery are listed in TABLE 33. The % Recovery for each component is calculated as follows. When the concentration of the spiked analyte is less than the detection limit in the unspiked sample, use SR = 0 for purposes of calculating % R**:**

$$\%R = \frac{(SSR - SR)}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result (prior to spiking)
SA = Spike Added

b. MS/MSD or Unspiked Matrix Duplicate Pair

At least one MSD or one unspiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. Recommended RPD control limits are listed in TABLE 33.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = %R Value for First Duplicate (unspiked sample or MS)
D_2 = %R Value for Second Duplicate (unspiked dup. or MSD)

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TABLE 33

Recommended MS/MSD and Matrix Duplicate Control Criteria for GC/ECD Analysis

Matrix:	Water		Other Matrices	
Compound	**MS/MSD Spike % Recovery**	**MS/MSD or Duplicate RPD**	**MS/MSD Spike % Recovery**	**MS/MSD or Duplicate RPD**
□-BHC (Lindane)	56-123	**15**	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	**18**	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50
Aroclor 1016/1260	56-103	20	40-140	50
ALL OTHER ANALYTES	40-130	30	30-140	50

8. Surrogate Standards

The Contractor shall monitor the performance of the method using surrogate compounds. Surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards. The following compounds are recommended as possible surrogates:

a. Pesticides analysis: Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) have been found to be a useful pair of surrogates for both single-column and dual-column instrument configurations. However, if the chromatographic conditions of a dual-column configuration cannot be adjusted to preclude co-elution of a target analyte with either DCB or TCMX, another compound such as 4-Chloro-3-nitrobenzotrifluoride may be used.

b. PCBs as Aroclors: The recommended surrogate is decachlorobiphenyl. Tetrachloro-m-xylene may be used in addition to DCB.

c. PCB congeners: When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as an internal standard and cannot also be used as a surrogate. The use of tetrachloro-m-xylene is recommended.

Surrogate recoveries should not exceed the control limits listed in TABLE 34. Proceed with corrective action when % Recovery for either surrogate is outside of the control limits. The required control limits are listed in TABLE 34. Surrogate recoveries are calculated as follows:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} x100$$

TABLE 34

Required Control Limits for GC/ECD Surrogate % Recovery

Compounds	Aqueous Samples % Recovery	Soil, Sludge, Sediment, Oil, & Waste Samples % Recovery
Decachlorobiphenyl	30-150	30-150
Tetrachloro-m-xylene	30-150	30-150
4-Chloro-3-nitrobenzotrifluoride, other	30-150	30-150

9. Internal Standards

The Contractor shall use an internal standard when individual PCB congeners are to be determined. The use of an internal standard when pesticides or Aroclors are to be determined is optional but can be beneficial, especially when low concentrations are being analyzed. Compounds to use as internal standards are recommended in the analytical methods. Recommended internal standards for certain analyte types are listed below:

a. PCB congeners: The recommended internal standard is decachlorobiphenyl. It is added to each sample extract and calibration standard prior to analysis.

b. Aroclors: An internal standard is not usually used when PCBs are determined as Aroclors.

c. Organochlorine pesticides: 1-Bromo-2-nitrobenzene is suggested as an internal standard for dual-column analysis and can also be used for single-column analysis. Pentachloronitrobenzene is recommended for single-column analysis when it is not a target analyte.

d. Control criteria for internal standards. Whenever quantitation is accomplished using an internal standard, internal standard data must be evaluated for acceptance. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

10. Confirmation of Target Analyte Identification

The Contractor shall confirm each positive tentative analysis in one of the following ways listed below. Tentative identification of single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Identification of multi-component analytes is based on retention time windows established for three to five major peaks (i.e., components of the mixture). The confirmation results must be reported:

a. Confirmation on a second GC column of dissimilar stationary phase:

(1) Single-column analysis: When confirmation is made on a second column, the second analysis must meet all the QC criteria described, just as is required for the primary analysis. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis.

(2) Dual-column analysis: When simultaneous analyses are performed from a single injection (using a dual column/dual detector system with columns of different polarities), identification and confirmation is incorporated in a single run. In this case, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, target analyte identification has been confirmed.

b. Confirmation by GC/MS analysis.

GC/MS confirmation may be used in conjunction with either single- or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan GC/MS will normally require a concentration of approximately 10 ng/µL in the final extract for each target analyte. Ion trap or selective ion monitoring (SIM) will normally require a concentration of approximately 1 ng/µL. The following requirements apply to confirmation by GC/MS:

(1) The GC/MS must be calibrated for the specific target analytes being confirmed

(2) GC/MS may not be used for confirmation when concentrations are below 1 ng/µL in the extract.

(3) GC/MS confirmation must be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated blank from the GC/ECD analysis.

(4) A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that the target analytes identified by GC/ECD can be confirmed by GC/MS.

11. Laboratory Control Sample

The Contractror shall include a Laboratory Control Sample (LCS) with each analytical batch. The LCS consists of an aliquot of an organic free (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same target analytes at the same concentrations as the matrix spike, and the % recoveries are calculated. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix.

TABLE 35
Recommended Laboratory Control Sample %R Criteria for Organic Analysis

Matrix:	**Water**	**Soil & Other Matrices**
Compound	**LCS %Recovery**	**LCS %Recovery**
ALL TARGET ANALYTES	70-130	60-140

The range for LCS recoveries provided in TABLE 35 may not be achievable for the pesticide and PCB target analytes, in which case the acceptance tables provided at the end of the method or the Contractor's historical recoveries may provide more realistic ranges. LCS percent recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 35 are to be supported by the Contractor's historical data which are to be provided in the report.

12. Corrective Action for Organic Analysis by GC/ECD

The Contractor shall find and correct the problem whenever an analytical procedure is "out-of-control" (fails to meet control criteria); also the analysis[68] must be repeated for all affected samples. The analytical procedure is out-of-control when any one or more of the following conditions occurs:

a. **Whenever the *initial calibration results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis! (**See Section **F.3.c.** for details.)

b. **Whenever the *calibration verification results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis!** (See Section **F.4.c**. for details.)

c. Whenever the method blank results exceed the control criteria. (See section **F.6.** for details.)

d. Whenever matrix spikes, surrogates, internal standards, laboratory control samples or other laboratory fortified sample results fall outside control limits. (See Sections **F.7**. **F.8**., **F.9.**, and **F.11** for details.)

e. Whenever matrix spike duplicate or matrix duplicate results fall outside control limits. (See Section **F.7.** for details.)

f. Whenever the chromatographic performance is poor (e.g., rising baseline, peak broadening, tailing, poor resolution, etc.).

g. Degradation of DDT or Endrin is outside control limits. (See Section **F.5.** for details.)

When the "out-of-control" conditions listed in items **c.** through **g.** above occur, re-extraction and reanalysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and blank failure the affected samples would include all field samples prepared with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.[69]

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000B, Section 8.5.5.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

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[68] Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

[69] Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. The State's position on holding times or reanalysis of out-of-control results is the following: it would be preferred that sample analysis be done within holding times. However, if that is not possible, reanalysis based on analytical requirements may still need to be performed for analytical obligations to be considered achieved. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results for samples analyzed beyond holding time requirements will be predicated on DQO and threshold requirements. Resampling may be required in some cases.

G. Volatile and Semivolatile Organic Analysis Including Petroleum Hydrocarbons by Gas Chromatography with Method-Specified Detectors (other than MS or ECD) (FID, PID, HECD, etc.)

VOCs and SVOCs by GC: QA/QC Measures Required (See Detailed Instructions Below)		
Source	**Title**	**Comments**
SW-846 Chapter One, especially Sections 4 & 5	"Quality Control": especially, Laboratory Operations and Definitions	Use for **all** analyses: States general QA/QC requirements and provides resource for Method QA/QC measures
SW-846 Method 5000 (and subsequent updates), especially Section 8.0	"Sample Preparation for Volatile Organic Compounds," Quality Control	Use for **all** VOA. All QA/QC measures required.
SW-846 Method 3500C (and subsequent updates)	"Organic Extraction and Sample Preparation"	Use for **all** SVOA and NVOA. All QA/QC measures required.
SW-846 Preparation Method used	As appropriate. (See TABLES, Protocol Analytes Lists, SW-846 Protocol, Petroleum Analysis)	All QA/QC measures in applicable method are required.
SW-846 Method 8000B (and subsequent updates), especially Sections 7.0 and 8.0	"Determinative Chromatographic Separations": especially sections on Procedure and Quality Control	Use for all chromatographic analyses: Detailed procedural information and summary of QA/QC requirements
SW-846 Determinative Method run	As appropriate; See Protocol Analyte List	All QA/QC measures in applicable method required.
Deliverables List (from these Technical Specifications)	"Analysis of Volatile and Semivolatile Organic Compounds by GC Using Method-Specified Detectors (FID, PID, ECD, UV, etc.," See Deliverables List 2C	QA/QC Documentation Required
Detailed Specifications Below	"G. Analysis of VOCs and SVOCs by Gas Chromatography "	Control criteria and corrective action requirements.

1. General Requirements and Considerations

The Contractor shall adhere to the following requirements:

a. Holding times and preservatives: Holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, must be followed based on sample characteristics. Holding time requirements for both samples and sample extracts (when applicable) must be adhered to.

b. SVOC extraction and cleanup: Samples to be analyzed for SVOCs must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. To achieve maximum sensitivity, the extract must be concentrated to 1 mL. If interferences prevent proper detection of the analytes of interest, extracts may undergo silica gel column cleanup prior to analysis. Additional cleanup steps may be required for some samples.

c. Total Petroleum Hydrocarbons by GC/FID are to be measured by Method 8015C using fused silica capillary columns and following the instructions for analysis and quantitation of petroleum hydrocarbons (GRO, DRO, and ERO). An external calibration procedure is to be used. TRPH for motor oils can be similarly determined, using the instructions for GRO and DRO, but employing a higher boiling range standard and making other appropriate adjustments.

2. Initial Calibration

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. GC calibration may be accomplished through either an internal or an external standard calibration procedure. Initial calibration must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be found in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration near, but above, the MDL. If data will be compared to risk-based human health or ecological protective levels, this low standard concentration for each analyte must be as low as or lower than the risk-based level to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples or must define the working range of the system.

a. External standard calibration procedure

(1) External Standard Calibration for Analysis of Single-Component Analytes

Prepare calibration standards at a minimum of five concentration levels for each analyte by dilution of stock standards with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the mass of analyte injected. The results can be used to prepare a calibration curve for each compound.

Alternatively, the ratio of detector response to mass of analyte injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the CF is a constant over the working range (i.e., the relative standard deviation (RSD) is 20%), linearity through the origin can be assumed. The average calibration factor can be used in place of a calibration curve to determine sample concentrations. The CF is calculated as follows:

$$CF = \frac{\text{Total Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

(2) External Standard Calibration for Total Petroleum Hydrocarbons by GC/FID

The standard used for TPH calibration must correspond with the distillation range of the type of petroleum being analyzed, with a separate standard for each fuel type. For purposes of this contract, range by carbon number is defined as:

(a) Gasoline: C4 – C12,
(b) Jet Fuel and Kerosene: C9 – C16,
(c) Diesel Fuel: C9 – C22,
(d) Motor Oil: C18 – C26, and
(e) Tar: C26 and greater.

As for single-component analytes, the standard must be run at a minimum of five concentrations, and a CF calculated for each concentration. If the CF is a constant over the working range (i.e., the RSD is ±20% using the equation above), linearity through the origin can be assumed. Then the average calibration factor can be used in place of a calibration curve to determine sample concentrations. The CF for petroleum fuels is calculated as:

$$CF = \frac{\text{Total Peak Area within the Retention Time Range}}{\text{Mass of the Compound Injected (in nanograms)}}$$

b. Internal standard calibration procedure for single-analyte components

Prepare calibration standards at a minimum of five concentration levels for each analyte by adding volumes of one or more stock standard solutions to a volumetric flask. To each calibration standard add a known amount of one or more internal standards, and dilute to volume with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 μL injections). Tabulate peak area or height responses against the concentration for each compound and internal standard. Calculate the response factor for each compound at each concentration. If the RF value is constant over the working range (the RSD is ≤20%), linearity through the origin can be assumed and the average RF can be used to calculate sample concentrations. The RF is calculated using the following equation:

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

Where: A_s = Peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard

c. Initial Calibration Control Criteria

Calculate the RSD for each analyte across all concentrations using the mean and standard deviation of the CFs or RFs:

$$RSD = \frac{SD}{\overline{CF}} x100 \qquad \text{or} \qquad RSD = \frac{SD}{\overline{RF}} x100$$

The RSD criteria in TABLE 36 must be met for linearity through the origin to be assumed using the CF or RF approach. If linearity through the origin cannot be assumed, the analysis must be stopped and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean CF for the external calibration procedure or the mean RF for the internal standard procedure. (See SW-846 Method 8000B, December 1996.)[70]

A new calibration curve (or calibration factor or response factor) must be prepared whenever a new column or detector is installed. The initial calibration data (and curve if used), calibration or response factors, and RSDs must be reported with the analysis results.

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[70]SW-846 Method 8000B, Section 7.5, (3rd edition Final Update III, December 1996) provides criteria for linear as well as for non-linear calibration models. Linear calibration curves are preferred. At times, it may be appropriate to use a non-linear calibration model. The non-linear option must be reserved for appropriate circumstances, such as the need to achieve low detection limits. Non-linear calibration may not be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

TABLE 36
Initial Calibration RSD Criteria for Assumption of Linearity in GC Analysis

Compound	External Calibration	Internal Calibration
	RSD for **Calibration Factors** across all concentrations	**RSD** for **Response Factors** across all concentrations
EACH TARGET ANALYTE	≤ 20 %	≤ 20 %

3. Establishment of Retention Time Windows

The Contractor shall identiy single-component target analytes based on retention time windows. GRO, DRO and ERO are distinguished based on the ranges of retention times for characteristic components in each type of fuel.

a. Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all standards (or standard mixtures) over the course of a 72-hour period. Serial injections over a period of less than 72 hours may result in retention time windows that are too tight (narrow).

b. Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007). (Recording retention times to three decimal places rather than only two must minimize the instances in which the standard deviation is calculated as zero.)

c. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

d. If the standard deviation of the retention times for a target compound is 0.000 (i.e., there is no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

e. The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

f. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

g. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each GC column and instrument. New retention time windows must be established whenever a new GC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

4. Calibration Verification

The Contractor shall verify the initial calibration and retention times at the beginning of each 12-hour work shift, at a minimum. Additional analyses of the verification standard(s) throughout a 12-hour shift are strongly recommended, especially for samples that contain visible concentrations of oily material. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring re-injection when QC limits are exceeded).

a. When **individual target analytes** are being analyzed, verification is accomplished by the analysis of one or more calibration standards (normally mid-concentration) that contain all of the target analytes and surrogates. If external standard calibration procedures are used, the midpoint calibration verification standard must also be injected at intervals during the 12-hour analytical shift.

b. When **petroleum hydrocarbons** are being analyzed, verification is accomplished by the measurement of the fuel standard **and** the hydrocarbon retention time standard.

c. Calibration verification control criteria

(1) Response criteria:[71] If an external standard calibration technique is used, the calibration factor for each single-component analyte, GRO, jet fuel, and kerosene should not exceed ≤ 15 %Difference from the mean calibration factor calculated for the initial calibration. DRO and motor oil should not exceed ≤ 20 %Difference. If an internal standard calibration technique is used for single-component analytes, the response factor for each analyte should not exceed a ≤ 15 percent difference from the mean response factor calculated for the initial calibration. The percent difference is calculated:

$$\%D = \frac{CF_v - \overline{CF_i}}{\overline{CF_i}} x100 \qquad \textbf{or} \qquad \%D = \frac{RF_v - \overline{RF_i}}{\overline{RF_i}} x100$$

Where: CF_v *and* RF_v are the calibration factor and response factor (whichever applies) from the analysis of the calibration verification standard,

And $\overline{CF_i}$ *and* $\overline{RF_i}$ are the mean calibration factor and the mean response factor from the initial calibration.

(2) The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 37 are not met for any analyte during calibration verification, then corrective action must be taken prior to continuing with analysis of samples. If attempts to correct the response %Difference problem are unsuccessful, a new initial calibration must be performed. If attempts to correct the retention time window problem are unsuccessful, new RT windows must be determined. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed. The contractor must report the results from the calibration verifications.

[71] If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are 85-115%. % Drift is calculated as: $\%Drift = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}}$ X 100%

TABLE 37

Calibration Verification Control Criteria for GC Analysis

Compound	Calibration Factor % D or Response Factor % D	Retention Time
SINGLE COMPONENT ANALYTES	≤ 15 one or more calibration standards	In Window (established with initial calibration midlevel standard RT)
GRO, JET FUEL, KEROSENE	≤ 15 fuel standard and hydrocarbon retention time standard	In Range (established with initial calibration standard RT)
DIESEL FUEL AND MOTOR OIL	≤ 20 fuel standard and hydrocarbon retention time standard	In Range (established with initial calibration standard RT)

Additional Calibration Criteria Applicable to All Compounds (Target and QC)	
GC Performance	Symmetrical peaks, minimum tailing, good resolution

5. Blanks

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples. Prior to being subjected to the method procedure, interferents must not be observed at the method detection limit of the compounds of interest.

a. Frequency

Method blanks must be prepared at a frequency of at least 5%. That is, at least one method blank must be extracted and analyzed with each group of up to 20 samples analyzed on the same instrument during the same analytical shift. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

b. Control Criteria

Concentrations of target analytes observed in the method blank must be no higher than the highest of:

(1) The Contractor's quantitation limit for the analyte,
(2) 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit), or
(3) 5% of the measured concentration in the sample.

c. Failure of control criteria.

If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the above control criteria), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

If method blank contamination cannot be attributable to carryover, the Contractor shall take corrective action. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

d. Results and reporting

The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

6. Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Unspiked Duplicates)

The Contractor shall analyze at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less**. **If the sample concentration exceeds this level, unspiked duplicates should be run.**

In matrix spike/matrix spike duplicate analysis, predetermined quantities of stock solutions of target analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected and used to assess bias due to sample matrix effects. The relative percent difference (RPD) between the split samples is calculated and used to assess analytical precision. If unspiked duplicates are analyzed, the RPD of detected analytes in the unspiked split samples is used to assess precision.

a. Matrix Spike

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the preparation and measurement methodology. The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

(1) Compounds to be spiked: Matrix spiking solutions must be prepared from compounds that are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all target analytes in order to accurately interpret matrix effects on sample results. The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

(2) Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

(3) Calculations and Control limits. The Contractor shall develop its own in-house acceptance criteria for spike recoveries. Recommended minimum % Recovery control limits for the spiked compounds in the MS (and MSD, if applicable) are listed in TABLE 38. The % Recovery for each component is calculated as follows. When the concentration of the spiked analyte is less than the detection limit in the unspiked sample, use SR = 0 for purposes of calculating % R:

$$\%R = \frac{(SSR - SR)}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result (prior to spiking)
SA = Spike Added

b. MS/MSD or Unspiked Matrix Duplicate Pair

At least one MSD or one unspiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. The Contractor shall develop its own in-house acceptance criteria for duplicate RPD. Recommended RPD control limits are listed in TABLE 38. The RPD is calculated as:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = %R Value for First Duplicate (unspiked sample or MS)
D_2 = %R Value for Second Duplicate (unspiked dup. or MSD)

TABLE 38

Recommended MS/MSD and Matrix Duplicate Control Criteria for GC Analysis

Matrix:	Water		Other Matrices	
Compound	**MS/MSD Spike % Recovery**	**MS/MSD or Duplicate RPD**	**MS/MSD Spike % Recovery**	**MS/MSD or Duplicate RPD**
ALL TARGET ANALYTES	70-130	20	60-140	40

7. Surrogate Standards

The Contractror shall monitor the performance of the method using at least one surrogate compound. The surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards.

Surrogate recoveries must not exceed the control limits listed in the analytical method or developed by the Contractor. Proceed with corrective action when the % Recovery for any surrogate does not meet control limits. Surrogate recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

Surrogate recoveries must be reported. Include the in-house historical surrogate recoveries in the report.

8. Control Criteria for Internal Standards

The Contractor shall evaluate internal standard data for acceptance whenever quantitation is accomplished using an internal standard. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

9. Confirmation of Target Analyte Identification

The Contractor shall confirm each <u>positive</u> tentative analysis of a single-component analyte in one of the following ways listed below. Tentative identification of a single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Identification of petroleum hydrocarbons is based on retention time patterns and this confirmation need be run only if analytical interferences are evident. The confirmation results must be reported:

a. Confirmation on a second GC column of dissimilar stationary phase:

(1) <u>Single-column analysis</u>: When confirmation is made on a second column, the second analysis must meet all the QC criteria described, just as is required for the primary analysis. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis.

(2) Dual-column analysis: When simultaneous analyses are performed from a single injection (using a dual column/dual detector system with columns of different polarities), identification and confirmation is incorporated in a single run. In this case, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, target analyte identification has been confirmed.

b. Confirmation by GC/MS analysis.

GC/MS confirmation may be used in conjunction with either single- or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan GC/MS will normally require a concentration of approximately 10 ng/µL in the final extract for each target analyte. Ion trap or selective ion monitoring (SIM) will normally require a concentration of approximately 1 ng/µL. The following requirements apply to confirmation by GC/MS:

(1) The GC/MS must be calibrated for the specific target analytes being confirmed

(2) GC/MS may not be used for confirmation when concentrations are below 1 ng/µL in the extract.

(3) GC/MS confirmation must be accomplished by analyzing the same extract that is used for GC analysis and the extract of the associated blank from the GC analysis.

(4) A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that the target analytes identified by GC can be confirmed by GC/MS.

When confirmation is made by a second analysis, that analysis must meet all of the QC criteria required for the first analysis. The confirmation results must be reported.

10. Laboratory Control Sample

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. The LCS consists of an aliquot of an organic free (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same target analytes at the same concentrations as the matrix spike, and the % recoveries are calculated. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix.

TABLE 39
Recommended Laboratory Control Sample %R Criteria for GC Analysis

Matrix:	Water	Soil & Other Matrices
Compound	**LCS %Recovery**	**LCS %Recovery**
ALL TARGET ANALYTES	70-130	60-140

The range for LCS recoveries provided in TABLE 39 may not be achievable for non-purgeable and semivolatile range target analytes, in which case the acceptance tables at the end of the method or the Contractor's historical recoveries may provide more realistic ranges. LCS percent recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 39 are to be supported by historical data which are to be provided in the report.

11. Corrective Action for Organic Analysis by GC

The Contractor shall find and correct the problem whenever an analytical procedure is "out-of-control" (fails to meet control criteria); also the analysis must be repeated[72] for all affected samples. The analytical procedure is out-of-control when any one or more of the following conditions occurs:

a. **Whenever the *initial calibration results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis! (**See Section **G.2.c.** for details.)

b. **Whenever the *calibration verification results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis!** (See section **G.4.** for details.)

c. Whenever the method blank results exceed the control criteria. (See section **G.5.** for details.**)**

d. Whenever matrix spikes, surrogates, internal standards, laboratory control samples or other laboratory fortified sample results fall outside control limits. (See Sections **G.6., G.7., G.8., and G.10.** for details.)

e. Whenever matrix spike duplicate or matrix duplicate results fall outside control limits. (See Section **G.6.** for details.)

f. Whenever the chromatographic performance is poor (e.g., rising baseline, peak broadening, tailing, poor resolution, etc.).

When the "out-of-control" conditions listed in items (**c.**) through (**f.**) above occur, re-extraction and reanalysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and blank failure the affected samples would include all field samples prepared with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.[73]

[72] Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

[73] Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. The State's position on holding times for reanalysis of out-of-control results is the following: It would be preferred that sample analysis be done within holding times. However, if that is not possible, reanalysis based on analytical requirements may still need to be performed for analytical obligations to be considered achieved. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results for samples analyzed beyond holding time requirements will be predicated on DQO and threshold requirements. Resampling may be required in some cases.

If QC results from the re-extraction and reanalysis ~~is~~ are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000B, Section 8.5.5.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

12. Additional Notes for Petroleum Hydrocarbon Analysis by GC

The Contractor shall adhere to the following requirements:

a. Unresolved peaks (the petroleum "hump"):

Diesel fuel, gasoline, and other petroleum products contain a large number of compounds that will produce well-resolved peaks in a GC/FID chromatogram. However, they also contain many other components that cannot be chromatographically resolved. This unresolved mixture results in the "hump" in the chromatogram that is characteristic of petroleum. While the resolved peaks are important for the identification of the specific fuel type, the area of the unresolved mixture contributes a significant amount of the total area response and should be included in the calculation of results.

b. When sample appears to be a particular type of hydrocarbon but sample response does not fall within appropriate retention time range:

When the retention time for the detected hydrocarbons in a sample does not fall within the appropriate retention time window, the following action should be taken:

(1) Run a reagent blank, and

(2) Dilute the sample to minimize matrix interferences and reanalyze.

(a) If the sample response falls within the retention time window after dilution, report only the second run.

(b) If the sample response still falls outside the retention time window, use professional judgment:

(i) Tentatively identify the detected material
(ii) Report both runs
(iii) Flag the results as tentatively identified.

c. When sample "saturates" (causes a full deflection response)

When a highly contaminated sample causes a saturated, full deflection peak, the following action should be taken:

(1) Run a reagent blank to prevent carry-over**.** See Section G.5.
(2) Dilute the sample and reanalyze.

H. Semivolatile and Non-Volatile Organic Compound Analysis by High Performance Liquid Chromatography

HPLC can be used for analysis of many semivolatile, nonvolatile, and some volatile organic compounds. The State will mainly request HPLC for analysis of polynuclear aromatic hydrocarbons (PAHs) and other compounds for which human health and ecological risk-based protective levels are lower than can be achieved by standard full scan GC/MS. This section will focus on analysis of PAHs for use in risk assessment. Guidance in this section refers to HPLC using non-MS detection: specifically, fluorescence and/or UV detectors. HPLC/MS techniques utilize different criteria than presented here. Refer to the analytical method for guidance.

1. General Requirements and Considerations

The Contractor shall adhere to the following requirements:

a. Extraction and cleanup: Samples must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. To achieve maximum sensitivity, the extract must be concentrated to 1 mL. If interferences prevent proper detection of the analytes of interest, extracts may undergo silica gel column cleanup prior to analysis. Additional cleanup steps may be required by some samples.

b. Interference considerations**:** The sensitivity of the HPLC technique usually depends on the level of interferences rather than instrumental limitations. When interferences are present, the level of sensitivity will be lower. Non-target PAHs present in the sample matrix may pose significant interference problems.

c. Holding times and preservatives: Preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, must be followed based on sample characteristics. Holding time requirements for both samples and sample extracts must be adhered to.

d. Detection: It is recommended that a combination of fluorescence and UV detectors be used. UV detection is applicable to a wide range of analytes and is less sensitive to RT fluctuation than fluorescence. However, UV does not provide sufficient sensitivity to quantitate some PAHs at sub-ppb concentrations, and hence to meet risk-based health protective levels, particularly for carcinogens. Fluorescence provides improved sensitivity, but not all target compounds fluoresce (e.g., acenaphthylene). An UV detector or an UV-Visible diode array detector (DAD) coupled to a fluorescence detector maximizes both sensitivity and selectivity. For compounds that fluoresce and for which UV detection can provide sufficient sensitivity, obtaining spectra from both detectors provides the additional advantage of combining identification and confirmation of target analytes in a single analysis.

e. Confirmation of compound identification: Compound identification by HPLC using non-MS detection must be supported by at least one additional qualitative technique unless the composition of the sample matrix has been well established by prior analyses.

2. Initial Calibration

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. HPLC calibration may be accomplished through either an internal or external standard calibration procedure. However, it may be difficult to find compounds for use as internal standards that can be chromatographically resolved from the target analytes.

Initial calibration must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be found in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration near, but above, the MDL. If data will be compared to risk-based human health or ecological protective levels, this low standard concentration for each analyte must be as low as or lower than the risk-based level to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples or must define the working range of the HPLC system.

a. External standard calibration procedure

Prepare calibration standards at a minimum of five concentration levels for each analyte by dilution of stock standards with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the mass of analyte injected. The results can be used to prepare a calibration curve for each compound.

Alternatively, the ratio of detector response to mass of analyte injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the CF is a constant over the working range (i.e., the relative standard deviation (RSD) is ±20%), linearity through the origin can be assumed. Then the average calibration factor can be used in place of a calibration curve to determine sample concentrations. The CF is calculated as follows:

$$CF = \frac{\text{Total Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

b. Internal standard calibration procedure

If an internal standard calibration procedure is used, a known constant amount of one or more internal standards is added to each calibration standard and each sample prior to analysis. Compounds selected for use as internal standards must be similar in analytical behavior to the compounds of interest, but must not be expected to be present in the samples. The analyst must demonstrate that the measurement of the internal standards is not affected by method or matrix interferences and that the internal standard can be chromatographically resolved from the target compounds.

Possible choices for internal standards might include brominated, fluorinated, or stable isotopically labeled PAH analogs. 4, 4'-difluorobiphenyl is a possible internal standard candidate for early eluting compounds determined by UV adsorbance. A different compound would need to be chosen for the higher molecular weight, fluorescent analytes.

Prepare calibration standards at a minimum of five concentration levels for each analyte by adding volumes of one or more stock standard solutions to a volumetric flask. To each calibration standard add a known amount of one or more internal standards, and dilute to volume with an appropriate

solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the concentration for each compound and internal standard. Calculate the response factor for each compound at each concentration. If the RF value is constant over the working range (the RSD is 20%), linearity through the origin can be assumed and the average RF can be used to calculate sample concentrations.

The RF is calculated using the following equation:

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

Where: A_s = Peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard

c. Initial Calibration Control Criteria

Calculate the RSD for each analyte across all concentrations using the mean and standard deviation of the CFs or RFs:

$$RSD = \frac{SD}{\overline{CF}} x100 \qquad \text{or} \qquad RSD = \frac{SD}{\overline{RF}} x100$$

The RSD criteria in TABLE 40 must be met for linearity through the origin to be assumed using the CF or RF approach. If linearity through the origin cannot be assumed, the analysis must be stopped and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean CF for the external calibration procedure or the mean RF for the internal standard procedure. (See SW-846 Method 8000B, December 1996.)[74]

A new calibration curve (or calibration factor or response factor) must be prepared whenever a new column or detector is installed. The initial calibration data (and curve if used), calibration or response factors, and RSDs must be reported with the analysis results.

[74]SW-846 Method 8000B, Section 7.5, (3rd edition Final Update III, December 1996) provides criteria for linear as well as for non-linear calibration models. Linear calibration curves are preferred. At times, it may be appropriate to use a non-linear calibration model. The non-linear option must be reserved for appropriate circumstances, such as the need to achieve low detection limits. Non-linear calibration may not be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance

TABLE 40
Initial Calibration RSD Criteria for Assumption of Linearity in HPLC Analysis

Compound	External Calibration	Internal Calibration
	RSD for **Calibration Factors** across all concentrations	**RSD** for **Response Factors** across all concentrations

Compound	External Calibration	Internal Calibration
	RSD for **Calibration Factors** across all concentrations	**RSD** for **Response Factors** across all concentrations
EACH TARGET ANALYTE	≤ 20 %	≤ 20 %

3. Establishment of Retention Time Windows

The Contractor shall adhere to the following requirements:

a. Before establishing windows, make sure the HPLC system is within optimum operating conditions. Make three injections of all standards (or standard mixtures) over the course of a 72-hour period.

b. Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007).

c. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

d. If the standard deviation of the retention times for a target compound is 0.000 (i.e., there is no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

e. The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

f. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

g. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each HPLC column and instrument. New retention time windows must be established whenever a new HPLC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

4. Calibration Verification

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals:

a. A calibration verification standard must be injected at the beginning of each 12-hour analytical shift, prior to conducting sample analyses.

b. If external standard calibration procedures are used, the midpoint calibration verification standard must also be injected at intervals during the 12-hour analytical shift. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring re-injection when QC limits are exceeded

c. Calibration verification control criteria

(1) Response criteria:[75] If an external standard calibration technique is used, the calibration factor for each analyte should not exceed a ±15 percent difference from the mean calibration factor calculated for the initial calibration. If an internal standard calibration technique is used, the response factor for each analyte should not exceed a ±15 percent difference from the mean response factor calculated for the initial calibration. The percent difference is calculated:

$$\%D = \frac{CF_v - \overline{CF_i}}{\overline{CF_i}} x100 \qquad \textbf{or} \qquad \%D = \frac{RF_v - \overline{RF_i}}{\overline{RF_i}} x100$$

where: CF_v *and* RF_v are the calibration factor and response factor (whichever applies) from the analysis of the calibration verification standard;

and $\overline{CF_i}$ *and* $\overline{RF_i}$ are the mean calibration factor and the mean response factor from the initial calibration.

(2) The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 41 are not met for any analyte during calibration verification, then corrective action must be taken prior to continuing with analysis of samples. If attempts to correct the response % Difference problem are unsuccessful, a new initial calibration must be performed. If attempts to correct the retention time window problem are unsuccessful, new RT windows must be determined. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed. The Contractor shall report the results from the calibration verifications.

TABLE 41
Calibration Verification Control Criteria for HPLC Analysis

Compound	Calibration Factor % D or Response Factor % D	Retention Time
ALL TARGET ANALYTES	±15	In Window (established with initial calibration midlevel standard RT)

Additional Calibration Criteria Applicable to All Compounds (Target and QC)	
GC Per	Symmetrical peaks, minimum tailing, good resolution

[75] If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are ±15%. % Drift is calculated as: $\%Drift = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}}$

5. Blanks

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples. Prior to

being subjected to the method procedure, interferents must not be observed at the quantitation limit of the compounds of interest.

a. Frequency

Method blanks must be prepared at a frequency of at least 5%. That is, at least one method blank must be extracted and analyzed with each group of up to 20 samples analyzed on the same instrument during the same analytical shift. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

b. Control Criteria

Concentrations of target analytes observed in the method blank must be no higher than the highest of:

(1) The Contractor's MDL for the analyte;

(2) 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or

(3) 5% of the measured concentration in the sample**.**

c. Failure of control criteria

If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the control criteria listed above), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

The Contractor shall take corrective action if method blank contamination cannot be attributable to carryover. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

d. Results and reporting

The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

6. Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Unspiked Duplicates)

The Contractor shall analyze at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less**. **If the sample concentration exceeds this level, unspiked duplicates should be run.**

In matrix spike/matrix spike duplicate analysis, predetermined quantities of stock solutions of target analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into

duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected and used to assess bias due to sample matrix effects. The relative percent difference (RPD) between the split samples is calculated and used to assess analytical precision. If unspiked duplicates are analyzed, the RPD of detected analytes in the unspiked split samples is used to assess precision.

a. Matrix Spike

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the preparation and measurement methodology. The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 0.1% (1000 pm).

Please note: MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

(1) Compounds to be spiked. Matrix spiking solutions must be prepared from compounds which are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all target analytes in order to accurately interpret matrix effects on sample results. The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

(2) Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

(3) Calculations and Control limits. The Contractor shall develop its own in-house acceptance criteria for spike recoveries. Recommended control limits for the MS (and MSD, if applicable) minimum spiked compounds' % Recovery are listed in TABLE 47. The % Recovery for each component is calculated as follows. When the concentration of the spiked analyte is less than the detection limit in the unspiked sample, use SR = 0 for purposes of calculating % R:

$$\%R = \frac{(SSR - SR)}{SA} x100$$

Where: SSR = Spiked Sample Result
SR = Sample Result (prior to spiking)
SA = Spike Added

b. MS/MSD or Unspiked Matrix Duplicate Pair

The Contractor shall perform at least one MSD or one unspiked duplicate on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. The Contractor shall develop its own in-house acceptance criteria for duplicate RPD. Recommended RPD control limits are listed in TABLE 42. The RPD is calculated as:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x100$$

Where: D_1 = %R Value for First Duplicate (unspiked sample or MS)
D_2 = %R Value for Second Duplicate (unspiked dup. or MSD)

TABLE 42
Recommended MS/MSD and Matrix Duplicate Control Criteria for HPLC Analysis

Matrix:	Water		Other Matrices	
Compound	MS/MSD Spike % Recovery	MS/MSD or Duplicate RPD	MS/MSD Spike % Recovery	MS/MSD or Duplicate RPD
ALL TARGET ANALYTES	70-130	20	60-140	40

7. Surrogate Standards

The performance of the method must be monitored using at least one surrogate compound. The surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards. Decafluorobiphenyl is recommended for use as the surrogate compound for PAH analysis. Additional PAH compounds may be used as surrogates if they are not expected to be present in the sample. Deuterated analogs of target analytes must not be used as surrogates for HPLC analysis due to coelution problems.

Surrogate recoveries must not exceed the control limits listed in TABLE 43. Proceed with corrective action when the % Recovery for any surrogate does not meet control limits. Surrogate recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \text{ x } 100$$

TABLE 43
Required Control Limits for Surrogate % Recovery in HPLC Analysis of PAHs

Compounds	Aqueous Samples % Recovery	Soil, Sludge, Sediment, Oil, & Waste Samples % Recovery
Decachlorobiphenyl	30-150	30-150
other compounds	30-150	30-150

8. Control Criteria for Internal Standards

Whenever quantitation is accomplished using an internal standard, internal standard data must be evaluated for acceptance. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

9. Confirmation of Target Analyte Identification

Tentative identification of single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Compound identification by HPLC using non-MS detection must be supported by at least one additional qualitative technique. Some possible methods for confirmation of positive tentative analysis include:

a. HPLC data from two different detectors (e.g., UV and fluorescence),

b. HPLC/UV data at two different wavelengths, or

c. Analysis on a second column with a dissimilar stationary phase.

Use of UV-Visible diode array detection may provide confirmation data from a single analysis if the Contractor can demonstrate this ability for typical sample extracts (not just standards) by comparison to another recognized confirmation technique.

Standard GC/MS techniques (e.g., SW-846 Method 8270D, unmodified) are not recommended for confirmation of carcinogenic PAHs due to insufficient sensitivity to achieve detection limits below risk-based human health and ecological protective levels. However, standard GC/MS is acceptable if concentrations of preliminarily identified target analytes are sufficiently high (e.g., > 660 µg/kg in solid matrices).

When confirmation is made by a second analysis, that analysis must meet all of the QC criteria required for the first analysis. The confirmation results must be reported.

10. Laboratory Control Sample

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch (Table 44). The LCS consists of an aliquot of an organic free (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same target analytes at the same concentrations as the matrix spike, and the % recoveries are calculated. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported.

TABLE 44
Required Laboratory Control Sample %R Criteria for Organic Analysis

Matrix:	Water	Soil & Other Matrices
Compound	**LCS %Recovery**	**LCS %Recovery**
ALL TARGET ANALYTES	70-130	60-140

11. Corrective Action for Organic Analysis by HPLC

The Contractor shall find and correct the problem whenever an analytical procedure is" out-of-control" (fails to meet control criteria); also the analysis must be repeated[76] for all affected samples. The analytical procedure is out-of-control when any one or more of the following conditions occurs:

a. **Whenever the *initial calibration results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis! (**See Section **H.2.** for details.)

b. **Whenever the *calibration verification results* do not meet control criteria: STOP! The instrument must be recalibrated before proceeding with analysis!** (See section **H.4.** for details.)

c. Whenever the method blank results exceed the control criteria. (See section **H.5.** for details.**)**

d. Whenever matrix spikes, surrogates, internal standards, laboratory control samples or other laboratory fortified sample results fall outside control limits. (See Sections **H.6., H.7., H.8., and H.10.** for details.)

e. Whenever matrix spike duplicate or matrix duplicate results fall outside control limits. (See Section **H.6.** for details.)

f. Whenever the chromatographic performance is poor (e.g., rising baseline, peak broadening, tailing, poor resolution, etc.).

When the "out-of-control" conditions listed in items (**c.**) through (**f.**) above occur, re-extraction and reanalysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and blank failure the affected samples would include all field samples prepared with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.[77]

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000B, Section 8.5.5.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

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[76] Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

[77] Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. The State's position on holding times for reanalysis of out-of-control results is the following: It would be preferred that sample analysis be done within holding times. However, if that is not possible, reanalysis based on analytical requirements may still need to be performed for analytical obligations to be considered achieved. If reanalysis is performed past the holding time, both analysis results must be reported.

USEPA DRINKING WATER PROTOCOL

The Contractor shall follow the same general guidance as is listed under the SW-846 Protocol, **except:**

1. Make the following substitutions in terminology:

Where SW-846 says:	**Replace with USEPA Office of Water term:**
Method Blank	Laboratory Reagent Blank (LRB)
Laboratory Control Sample (LCS)	Laboratory Fortified Blank (LFB)
Matrix Spike	Laboratory Fortified Sample Matrix (LFM)
Quantitation Limit, PQL, or Reporting Limit	Method Detection Limit (MDL)

2. Use any numerical control criteria provided in the Office of Water analytical methods instead of the recommended criteria supplied in the TABLES in these Technical Specifications. E.g.: Substitute the tuning criteria in TABLE 3 of Method 524.2, Revision 4.1, for the tuning criteria in TABLE 16 of these Technical Specifications.

3. Perform all QA/QC measures listed in the Analytical and QA/QC Requirements section of these Technical Specifications—even if the Office of Water Method does not explicitly require them. Provide all items on the Deliverables List in these Technical Specifications for the appropriate analysis--even if the Office of Water Method does not explicitly require them.

E.g.: The Drinking Water Methods (500 series) frequently do not require a Laboratory Fortified Sample Matrix (matrix spike) unless the QC criteria for internal standards and surrogates are not met. Run and report the matrix spike and a matrix spike duplicate in any case. **The State requires site-specific MS/MSD analysis for every batch unless sample concentrations exceed 4x the spike concentration or 1000 ppm.**

EXHIBIT B

SECTION I

PAYMENT FOR LABORATORY SERVICES

EXHIBIT B
Section I
Payment for Laboratory Services

Task	Cost
Task A. General Services and Responsibilities	
A.1. The Contractor shall provide analytical laboratory services and report the results to IDEM, Office of Land Quality.	Included in Cost Matrix Sheet Pricing
A.2. The Contractor shall perform laboratory analysis according to the protocols.	Included in Cost Matrix Sheet Pricing
A.3. The Contractor shall maintain a Quality System.	Included in Cost Matrix Sheet Pricing
A.4. The Contractor shall validate each method through the Quality System.	Included in Cost Matrix Sheet Pricing
A.5. The Contractor shall participate in a Proficiency Testing Program and submit the results within 30 days of request.	Included in Cost Matrix Sheet Pricing
A.6. The Contractor shall maintain a sufficient number of analytical instruments.	Included in Cost Matrix Sheet Pricing
A.7. The Contractor shall notify IDEM in the event of organizational changes.	Included in Cost Matrix Sheet Pricing
A.8. The Contractor shall make laboratory services available 8AM to 5PM (ET).	Included in Cost Matrix Sheet Pricing
Task B. Personnel Availability	
B.1. The Contractor shall provide qualified personnel.	Included in Cost Matrix Sheet Pricing
B.2. The Contractor shall make personnel available for enforcement or litigation.	Included in Cost Matrix Sheet Pricing
B.3. The Contractor shall notify the State of any personnel changes.	Included in Cost Matrix Sheet Pricing

Task C. Sample Containers, Trip Blank And Preservatives

C.1. The Contractor shall provide USEPA approved sample containers	Included in Cost Matrix Sheet Pricing
C.2. The Contractor shall provide containers for matrix spike/matrix spike duplicate (MS/MSD) measurement.	Included in Cost Matrix Sheet Pricing
C.3. The Contractor shall prepare containers for aqueous samples with required chemical preservatives.	Included in Cost Matrix Sheet Pricing
C.4. The Contractor shall provide overnight or one-day courier service.	Included in Cost Matrix Sheet Pricing

Task D. Analytical Protocals and Methods

D.1. The Contractor shall ensure that the COC form matches the laboratory request form.	Included in Cost Matrix Sheet Pricing
D.2. The Contractor shall perform analyses specified within the protocol.	Included in Cost Matrix Sheet Pricing
D.3. The Contractor shall give the State two (2) days to conduct the sampling.	Included in Cost Matrix Sheet Pricing
D.4. The Contractor shall perform matrix spike/ matrix spike duplicates (MS/MSD) at the specified frequency.	Included in Cost Matrix Sheet Pricing
D.5. The Contractor shall contact IDEM should no samples be identified for MS/MSD.	Included in Cost Matrix Sheet Pricing
D.6. The Contractor shall not bill for other samples analyzed for QA/QC purposes.	Included in Cost Matrix Sheet Pricing

Task E. Reporting

E.1. The Contractor shall report each case separately and on a compact disc.	Included in Cost Matrix Sheet Pricing
E.2. The Contractor shall report all analytes.	Included in Cost Matrix Sheet Pricing

E.3.	The Contractor shall provide results via fax or email when requested.	Included in Cost Matrix Sheet Pricing
E.4.	The Contractor shall retain documentation for three (3) years.	Included in Cost Matrix Sheet Pricing
E.5.	The Contractor shall provide internal chain-of-custody when requested by the State.	Included in Cost Matrix Sheet Pricing
E.6.	The Contractor shall provide all documentation.	Included in Cost Matrix Sheet Pricing
E.7.	The Contractor shall analyze and submit a report in the standard turn-around-time of thirty (30) days.	Included in Cost Matrix Shaeet Pricing

Task F. Invoicing and Payment Reduction

F.1.	The Contractor shall provide itemized invoices.	The Contractor shall be paid according to the pricing list in the Cost Matrix Sheets for 30-day, 14-day, 7-day and 48-hour TATs found in Exhibit B, Section II. Any other additional charges shall not be paid.
F.2.	The Contractor shall receive payment for services when all data and documentation has been received by the State.	The Contractor shall be paid according to the pricing list in the Cost Matrix Sheets for 30-day, 14-day, 7-day and 48-hour TATs found in Exhibit B, Section II. Any other additional charges shall not be paid
F.3.	The Contractor shall provide pricing on Special Analytical Services when requested by the State.	Upon request

EXHIBIT B

SECTION II

COST MATRIX SHEETS

SW-846 Protocol

AQUEOUS Sample Pricing

TOTAL METALS Group A				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Arsenic	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Barium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Cadmium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Chromium (total)	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Lead	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Mercury	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Selenium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Silver	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Preparation Cost	**$ 9.00**	**$ 10.00**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 60.30**	**$ 86.95**	**$ 120.60**	**$ 150.75**

TOTAL METALS Group B				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Antimony	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Beryllium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Cobalt	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Copper	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Nickel	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Thallium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Vanadium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Zinc	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Preparation Cost	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 52.20**	**$ 78.30**	**$ 104.40**	**$ 130.50**

TOTAL METALS Group C				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Aluminum	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Calcium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Iron	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Magnesium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Manganese	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Potassium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Sodium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Preparation Cost	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 46.80**	**$ 70.20**	**$ 93.60**	**$ 117.00**

TOTAL METALS Group D				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Hexavalent Chromium	**$ 20.00**	**$ 30.00**	**$ 40.00**	**$ 50.00**
TOTAL	**$ 20.00**	**$ 30.00**	**$ 40.00**	**$ 50.00**

TOTAL METALS Group E				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Boron	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Lithium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Molybdenum	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Strontium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Tin	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Titanium	**$ 5.40**	**$ 8.10**	**$ 10.80**	**$ 13.50**
Preparation Cost	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 41.40**	**$ 62.10**	**$ 82.80**	**$ 103.50**

General Chemistry Group A				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Total Cyanide	**$ 15.30**	**$ 22.95**	**$ 30.60**	**$ 38.25**
Free Cyanide	**$ 15.30**	**$ 22.95**	**$ 30.60**	**$ 38.25**
Amenable Cyanide	**$ 30.60**	**$ 45.90**	**$ 61.20**	**$ 76.50**
Total Sulfide	**$ 15.30**	**$ 22.95**	**$ 30.60**	**$ 38.25**
TOTAL	**$ 76.50**	**$ 114.75**	**$ 153.00**	**$ 191.25**

General Chemistry Group B				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Ammonia-N	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Chloride	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
Nitrate-Nitrite	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
pH	**$ 7.20**	**$ 10.80**	**$ 14.40**	**$ 18.00**
Specific Conductance	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
Sulfate	**$ 16.20**	**$ 24.30**	**$ 32.40**	**$ 40.50**
Total Dissolved Solids	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
Total Solids	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 90.00**	**$ 135.00**	**$ 180.00**	**$ 225.00**

General Chemistry Group C				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Alkalinity	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Bicarbonate	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Carbonate	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Eh	**$ 7.20**	**$ 10.80**	**$ 14.40**	**$ 18.00**
Fluoride	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Hardness	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Oxygen, Dissolved	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Total Phosphorus (PO4)	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Turbidity	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
TOTAL	**$ 130.50**	**$ 195.75**	**$ 261.00**	**$ 326.25**

General Chemistry Group D				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Oil and Grease(HEM)	**$ 27.00**	**$ 40.50**	**$ 54.00**	**$ 67.50**
Total Organic Carbon	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Total Organic Halides	**$ 112.50**	**$ 168.75**	**$ 225.00**	**$ 281.25**
Phenolics, Total	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Surfactants (MBAS)	**$ 67.50**	**$ 101.25**	**$ 135.00**	**$ 168.75**
Chemical Oxygen Demand	**$ 19.80**	**$ 29.70**	**$ 39.60**	**$ 49.50**
BOD5	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
CBOD5	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
TOTAL	**$ 307.80**	**$ 461.70**	**$ 615.60**	**$ 769.50**

General Chemistry Group E-1				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
E. coli	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
Fecal Coliform	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
Total Coliform	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
Nitrogen-Ammonia	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
TOTAL	**$ 81.00**	**$ 121.50**	**$ 162.00**	**$ 202.50**

General Chemistry Group E-2				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Nitrogen-Ammonia	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Nitrogen-Nitrate	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Nitrogen-Nitrite	**$ 13.50**	**$ 20.25**	**$ 27.00**	**$ 33.75**
Nitrogen-Total Kjeldahl	**$ 22.50**	**$ 33.75**	**$ 45.00**	**$ 56.25**
Phosphorus, Total	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
Total Suspended Solids	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 90.00**	**$ 135.00**	**$ 180.00**	**$ 225.00**

GENERAL CHEMISTRY Group F				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
% Solids	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
Total Solids	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
Volatile Solids	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
Paint Filter Test	**$ 9.00**	**$ 13.50**	**$ 18.00**	**$ 22.50**
TOTAL	**$ 39.60**	**$ 59.40**	**$ 79.20**	**$ 99.00**

VOA Group A: OLQ STANDARD VOLATILES LIST				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Volatile Organics List	**$ 85.50**	**$ 128.25**	**$ 171.00**	**$ 213.75**
TOTAL	**$ 85.50**	**$ 128.25**	**$ 171.00**	**$ 213.75**

VOA Group B: BTEX and MTBE				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
BTEX and MTBE	**$ 31.50**	**$ 47.25**	**$ 63.00**	**$ 78.75**
TOTAL	**$ 31.50**	**$ 47.25**	**$ 63.00**	**$ 78.75**

VOA Group C: Antifreeze/Coolant				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Ethylene & Propylene Glycol	**$ 49.50**	**$ 74.25**	**$ 99.00**	**$ 123.75**
TOTAL	**$ 49.50**	**$ 74.25**	**$ 99.00**	**$ 123.75**

SVOA Group A: OLQ STANDARD SEMIVOLATILES LIST				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Semivolatile Organics List	**$ 144.00**	**$ 216.00**	**$ 288.00**	**$ 360.00**
TOTAL	**$ 144.00**	**$ 216.00**	**$ 288.00**	**$ 360.00**

SVOA Group B: Polychlorinated Biphenyl Compounds (PCBs) as AROCLORS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Aroclors	**$ 54.00**	**$ 81.00**	**$ 108.00**	**$ 135.00**
TOTAL	**$ 54.00**	**$ 81.00**	**$ 108.00**	**$ 135.00**

SVOA Group C: Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
PAHs	**$ 72.00**	**$ 108.00**	**$ 144.00**	**$ 180.00**
TOTAL	**$ 72.00**	**$ 108.00**	**$ 144.00**	**$ 180.00**

SVOA Group D: Organochlorine Pesticides by GC/ECD				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Organochlorine Pesticides	**$ 72.00**	**$ 108.00**	**$ 144.00**	**$ 180.00**
TOTAL	**$ 72.00**	**$ 108.00**	**$ 144.00**	**$ 180.00**

PETROLEUM Group A – TPH/TRPH by GC/FID				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Gasoline Range	**$ 27.00**	**$ 40.50**	**$ 54.00**	**$ 67.50**
Diesel Range	**$ 45.00**	**$ 67.50**	**$ 90.00**	**$ 112.50**
Heavy Oil Range	**$ 49.50**	**$ 74.25**	**$ 99.00**	**$ 123.75**
TOTAL	**$ 121.50**	**$ 182.25**	**$ 243.00**	**$ 303.75**

PETROLEUM Group B – TRPH as HEM by Gravimetry				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
TRPH as HEM	**$ 36.00**	**$ 54.00**	**$ 72.00**	**$ 90.00**
TOTAL	**$ 36.00**	**$ 54.00**	**$ 72.00**	**$ 90.00**

RCRA CHARACTERISTICS of HAZARDOUS WASTE				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Corrosivity (pH)	**$ 10.80**	**$ 16.20**	**$ 21.60**	**$ 27.00**
Corrosivity to Steel	**$ 67.50**	**$ 101.25**	**$ 135.00**	**$ 168.75**
Reactive Cyanide	**$ 19.80**	**$ 29.70**	**$ 39.60**	**$ 49.50**
Reactive Sulfide	**$ 19.80**	**$ 29.70**	**$ 39.60**	**$ 49.50**

Ignitability (Flashpoint) **Value**	**$ 27.00**	**$ 40.50**	**$ 54.00**	**$ 67.50**
TOTAL	**$ 144.90**	**$ 217.35**	**$ 289.80**	**$ 362.25**

TCLP: RCRA CHARACTERISTIC of TOXICITY				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
TCLP – Metals only	**$ 85.50**	**$ 128.25**	**$ 171.00**	**$ 213.75**
TCLP – VOCs only	**$ 117.00**	**$ 175.50**	**$ 234.00**	**$ 292.50**
TCLP – SVOCs only	**$ 162.00**	**$ 243.00**	**$ 324.00**	**$ 405.00**
TCLP – Pesticides only	**$ 103.50**	**$ 155.25**	**$ 207.00**	**$ 258.75**
TCLP – **all analytes:**	**$ 450.00**	**$ 675.00**	**$ 900.00**	**$ 1,125.00**
TOTAL	**$ 918.00**	**$ 1,377.00**	**$ 1,836.00**	**$ 2,295.00**

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EPA Drinking Water Protocol AQUEOUS Sample Pricing

VOLATILE ORGANIC COMPOUNDS Group A: 524.2 LIST				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SEMIVOLATILE ORGANIC COMPOUNDS Group A: 525.2 LIST Extractables List				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound **VALUE**	$ -	$ -	$ -	$ -
Entire List **VALUE**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SEMIVOLATILE ORGANIC COMPOUNDS Group B: AROCLORS LIST				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Aroclors List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Total Bid Amount (30 day)	$ -

SAS- Aqueous				
SAS Group A – SAS GENERAL CHEMISTRY				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Purgeable Organic Halides (POX)	$ -	$ -	$ -	$ -
Settleable Matter (residue)	$ -	$ -	$ -	$ -
Bromide	$ -	$ -	$ -	$ -
Chlorine, total residual	$ -	$ -	$ -	$ -
Orthophosphate	$ -	$ -	$ -	$ -
Silica	$ 13.50	$ 20.25	$ 27.00	$ -
Sulfite	$ -	$ -	$ -	$ -
TOTAL	$ 13.50	$ 20.25	$ 27.00	$ -

SAS Group B – RADIONUCLIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Gross Alpha Radiation **Value**	$ -	$ -	$ -	$ -
Gross Beta Radiation **Value**	$ -	$ -	$ -	$ -
Radium-226 **Value**	$ -	$ -	$ -	□□
Radium-228 **Value**	$ -	$ -	$ -	□□
Radon-222 **Value**	$ -	$ -	$ -	□□
Gamma Emitting Radionuclides **Value**	$ -	$ -	$ -	□□
Strontium-89 **Value**	$ -	$ -	$ -	□□
Tritium **Value**	$ -	$ -	$ -	□□
Uranium-234 **Value**	$ -	$ -	$ -	□□
TOTAL	$ -	$ -	$ -	$ -

SAS Group C – SUPPLEMENTAL VOLATILE ORGANIC COMPOUNDS (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Supplemental VOCs –entire list	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group D– ORGANOPHOSPHORUS PESTICIDES and HERBICIDES (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group E– ORGANOPHOSPHORUS PESTICIDES and HERBICIDES (Other)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group F– Additional ORGANOCHLORINE PESTICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Organochlorine compounds list	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group G– CHLORINATED HERBICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Chlorinated Herbicides list	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group H – SUPPLEMENTAL SEMIVOLATILE ORGANIC COMPOUNDS (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**

One compound	$ -	$ -	$ -	$ -
Entire Semivolatiles list	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group I – THIOCARBATE PESTICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Thiocarbate Pesticides List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group J – PCB CONGENERS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound **Value**	$ -	$ -	$ -	$ -
PCB Congeners List **Value**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group K – POLYCHLORINATED DIBENZODIOXINS AND DIBENZOFURANS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound **Value**	$ -	$ -	$ -	
Entire List **Value**	$ -	$ -	$ -	
TOTAL	$ -	$ -	$ -	

SAS Group L – N-METHYL CARBAMATE PESTICIDES and INDUSTRIAL COMPOUNDS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound **Value**	$ -	$ -	$ -	$ -
Entire List **Value**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group M – Full-Range IR Scan on Unknown				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound **Value**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	

				$ -

SAS Group N – Additional Pesticides and Other				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound **Value**	$ -	$ -	$ -	$ -
Entire List **Value**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group O – Synthetic Precipitation Leaching Procedure (SPLP)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Metals (in Leachate)	$ -	$ -	$ -	$ -
Cyanide	$ -	$ -	$ -	$ -
Other Gen Chemistry	$ -	$ -	$ -	$ -
VOCs	$ -	$ -	$ -	$ -
SVOCs	$ -	$ -	$ -	$ -
Pesticides	$ -	$ -	$ -	$ -
PAHs	$ -	$ -	$ -	$ -
Leaching Procedure	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group P – Neutral Leaching Method for Industrial Waste				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Leaching Procedure	$ -	$ -	$ -	$ -
Coal Ash – Leach plus analyses	$ -	$ -	$ -	$ -
Foundry Waste – Leach plus analyses	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group Q- USACE Modified Elutriate Test				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**

Elutriate Test on Dredged Sediment	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS- Non Aqueous				
SAS Group A – SAS GENERAL CHEMISTRY				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Purgeable Organic Halides (POX)	$ -	$ -	$ -	$ -
Ignitability of Solids	$ -	$ -	$ -	$ -
Spontaneously Combustible Mat'l	$ -	$ -	$ -	$ -
Liquid Release Test	$ -	$ -	$ -	$ -
Settleable Matter (residue)	$ -	$ -	$ -	$ -
Bromide	$ -	$ -	$ -	$ -
Chlorine, total residual	$ -	$ -	$ -	$ -
Total Chlorine in Oils	$ -	$ -	$ -	$ -
Orthophosphate	$ -	$ -	$ -	$ -
Silica	$ -	$ -	$ -	$ -
Sulfite	$ -	$ -	$ -	$ -
Oxidizing Solids **Value**	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group B – RADIONUCLIDES	ALL VALUES			
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Gross Alpha Radiation	$ -	$ -	$ -	$ -
Gross Beta Radiation	$ -	$ -	$ -	$ -
Radium-223	$ -			
Radium-224	$ -			
Radium-226	$ -	$ -	$ -	
Radium-228	$ -	$ -	$ -	
Cesium-134	$ -			
Cesium-137	$ -			
Iodine-131	$ -			
Radon-222	$ -	$ -	$ -	
Gamma Emitting Radionuclides	$ -	$ -	$ -	
Strontium-89	$ -	$ -	$ -	
Strontium-90	$ -			
Tritium	$ -			
Uranium-234	$ -	$ -	$ -	
Uranium-238	$ -			
TOTAL	$ -	$ -	$ -	$ -

SAS Group C – SUPPLEMENTAL VOLATILE ORGANIC COMPOUNDS (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Supplemental VOCs –entire list	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group D– ORGANOPHOSPHORUS PESTICIDES and HERBICIDES (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group E– ORGANOPHOSPHORUS PESTICIDES and HERBICIDES (Other)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -

Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group F– Additional ORGANOCHLORINE PESTICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Organochlorine compounds list	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group G– CHLORINATED HERBICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Chlorinated Herbicides list	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group H–SUPPLEMENTAL SEMI-VOLATILE ORGANIC COMPOUNDS (Appendices)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Entire Semivolatiles list	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group I – THIOCARBATE PESTICIDES				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
Thiocarbate Pesticides List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group J – PCB CONGENERS **ALL VALUES**				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	$ -	$ -	$ -	$ -
PCB Congeners List	$ -	$ -	$ -	$ -

TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group K – POLYCHLORINATED DIBENZODIOXINS AND DIBENZOFURANS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	
Entire List	**$ -**	**$ -**	**$ -**	
TOTAL	**$ -**	**$ -**	**$ -**	

SAS Group L – N-METHYL CARBAMATE PESTICIDES and INDUSTRIAL COMPOUNDS				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group M – IR SCAN – UNKNOWN ORGANIC MATERIAL (Semivolatile or Nonvolatile)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
IR Scan of Unknown	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group N – Additional Pesticides and Nonvolatile Compounds				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

SAS Group O – Synthetic Precipitation Leaching Procedure (SPLP)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Metals (in Leachate)	**$ -**	**$ -**	**$ -**	**$ -**
Cyanide	**$ -**	**$ -**	**$ -**	**$ -**
Other General Chemistry	**$ -**	**$ -**	**$ -**	**$ -**
VOCs	**$ -**	**$ -**	**$ -**	

				$ -
SVOCs	$ -	$ -	$ -	$ -
Pesticides	$ -	$ -	$ -	$ -
PAHs	$ -	$ -	$ -	$ -
Leaching Procedure	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group P – Neutral Leaching Method for Industrial Waste				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Leaching Procedure	$ -	$ -	$ -	$ -
Coal Ash – Leach plus analyses	$ -	$ -	$ -	$ -
Foundry Waste – Leach plus analyses	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS Group Q-USACE Modified Elutriate Test				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Elutriate Test on Dredged Sediment	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

SAS- EPA Drinking Water Protocol				
Drinking Water SAS Group A: Additional VOLATILE Compounds (not on 524.2 list)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Three Compounds	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group B: Method 525.2 Organochlorine Pesticides List Values				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group C: Method 525.2 Nitrogen/Phosphorus Pesticides List Values				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group D: Method 525.2 PCB Congeners List				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group E: SVOC EXTRACTABLES LIST				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group F: Polynuclear Aromatic Hydrocarbons List				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	$ -

Drinking Water SAS Group G: Pesticides List				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	$ -	$ -	$ -	$ -
Entire List	$ -	$ -	$ -	$ -

TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

Drinking Water SAS Group H: Chlorinated Acid Pesticides and Herbicides				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

Drinking Water SAS Group I: N-Methylcarbamoyloxime and N-Methyl-Carbamate Pesticides				
ANALYTE Value	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

Drinking Water SAS Group J: Additional Pesticides and Herbicides, Miscellaneous				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
One Compound	**$ -**	**$ -**	**$ -**	**$ -**
Entire List	**$ -**	**$ -**	**$ -**	**$ -**
TOTAL	**$ -**	**$ -**	**$ -**	**$ -**

Additional Analytical Services

TPH Washington Dept. of Ecology Methods				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
VPH - Fractions **Value**	**$ 63.00**	**$ 94.50**	**$ 126.00**	**$ 157.50**
EPH - Fractions Value	**$ 81.00**	**$ 121.50**	**$ 162.00**	**$ 202.50**
TOTAL	**$ 144.00**	**$ 216.00**	**$ 288.00**	**$ 360.00**

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Six Liter Summa TO-15 analysis	**$ 121.50**	**$ 182.25**	**$ 243.00**	**$ 303.75**
Six Liter Summa TO-15 SIM analysis	**$ 139.50**	**$ 209.25**	**$ 279.00**	**$ 348.75**
6 Liter Summa, Flow controller, Vacuum/Pressure gauges **Value**	**$ 105.00**	**$ 157.50**	**$ 210.00**	**$ 262.50**
TOTAL	**$ 366.00**	**$ 549.00**	**$ 732.00**	**$ 915.00**

Fraction Organic Carbon				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
Soils by Walkley -Black	**$ 36.00**	**$ 54.00**	**$ 72.00**	**$ 90.00**
Soil Drying and #10 Sieving	**$**	**$**	**$**	**$ -**
Soils by ASTM D 2974 C	**$ 18.00**	**$ 27.00**	**$ 36.00**	**$ 45.00**
TOTAL	**$ 54.00**	**$ 81.00**	**$ 108.00**	

Analysis of Aqueous Samples for Perfluorooctanoic Acid (PFOA) and Perfluorooctanane Sulfonate (PFOS)				
ANALYTE	**30 DAY**	**14 DAY**	**7 DAY**	**48 HOURS**
PFOA		$ -	$ -	$ -
PFOS	$ -	$ -	$ -	$ -
TOTAL	$ -	$ -	$ -	

EXHIBIT B

SECTION III

RATE SCHEDULE

Microbac Laboratories, Inc.
Rate Schedule
Exhibit B, Section III

RATE SCHEDULE	
Classification	**Rate/Hourly**
President	**$ 150.00**
Vice President	**$ 125.00**
Lab Director	**$ 125.00**
Senior Programmer	**$ 125.00**
QA Officer	**$ 90.00**
QA Coordinator	**$ 90.00**
Senior Chemist	**$ 75.00**
Laboratory Group	**$ 75.00**
Leader/Supervisor	**$ 75.00**
Project Manager	**$ 75.00**
All Other Service Positions	**$ 75.00**

Travel/Lodging/Per Diem

Expenditures made by the Contractor for travel will be reimbursed by the State at the current rate paid by the State of Indiana. Travel expenses can only be reimbursed in accordance with State Travel Policies and Procedures as specified in Financial Management circular #2003-1

Mileage	$0.44 per mile
Lodging	$89.00 + applicable taxes per night
Per Diem	$26.00 per day

Indiana does not recognize, engage in, or accept cost plus contractor costs. IC 5-22-1 et seq.

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EXHIBIT C
Schedule for Project Tasks

The associated time periods and tasks are necessary for the project are as follows:

TASK	**TIME PERIOD**
Task A. General Services and Responsibilities	Ongoing
Task B. Personnel Availability	Ongoing
Task C. Sample Containers, Preservatives and assignment Holding Times	Dependent upon sampling
Task D. Analytical Protocols and Methods	According to the requested Turn-Around-Time (TAT)
Task E. Reporting	According to the requested Turn-Around-Time (TAT)

EXHIBIT D
SPECIAL CONDITIONS

In addition to the terms and conditions set forth herein, the parties agree to abide by the following special conditions:

Notice to Parties.

Whenever any notice, statement or other communication is required under this Contract, it shall be sent to the following addresses:

Notices to State:

Chief, Finance and Operations Section
Office of Land Quality, IDEM
100 North Senate Avenue
MC 66-30, IGCN 1101
Indianapolis, IN 46204-2251

Notices to Contractor:

Ron Misiunas, Director, Laboratory Services
Microbac Laboratories, Inc
250 West 84th Drive
Merrillville, IN 46410
219-798-9367 mobile
219-472-4555 direct
219-769-8378 office/lab
Ron.misiunas@microbac.com

Payments to Contractor:

As required by IC 4-13-2-14.8, payments to the Contractor shall be made via electronics funds transfer in accordance with the instructions filed by the Contractor with the Indiana Auditor of State.

EXHIBIT E

Laboratory Request
Form

Laboratory Request

Date:	Sample Numbers: OL - OL
From:	
To:	
IDEM Project Manager:	

As identified by the provisions of RFP Scope of Work, IDEM/OLQ is requesting a sample setup. Please submit an electronic data package as requested in an e-mail on November 15, 2006.

SCOPE OF WORK REQUESTED

Please provide the State with a copy of the cleanliness certification with each group of containers provided. Please supply enough sample containers for MS/MSD analysis.

Data Quality:	As Required By RFP	Protocol:
Matrix Type:		Dedicated Equipment? Yes

Analysis:									
Samples:									
Duplicates:									
Trip Blanks:									
Equipment Blanks:									
Total analysis:									

Projected Sample Date(s):	Projected Date(s) to Lab:	Turnaround Time: 30 Days	Bottles:	Will arrive by:
			Date:	

Electronic Approval History

	User ID	Approver Name	Datetime	Description
1	K302180	Diller,Kimberly Michele	12/21/2017 4:09:43PM	Agency Fiscal Approval
2	S004382	Redding,Sandra D	01/04/2018 2:00:31PM	IDOA Legal Approval
3	J292897	Dant,Joseph N.	01/09/2018 11:30:39AM	SBA Approval
4	M338811	Skarbeck,Molly H	01/09/2018 12:07:40PM	Attorney General Approval
5	S210690	Gard,Susan W	01/09/2018 3:50:38PM	Attorney General Approval