ABBT-B
IN0006062582

TOTAL ORGANIC CARBON (TOC)
(Soils and Solids, Boat Method)

Submitted by: Scott Anderson

Gregory A. Busch - QA Officer — 1-8-97
Approval / Title — Date

Harry A. Klingle - Lab Director — 1-8-97
Approval - Title — Date

Barbara A. Shrake - Lab Supervisor — 1-10-97
Approval - Title — Date

PURPOSE: This method is applicable to the determination of total organic carbon (TOC) in soils, solids, and matrices not soluble in water. This procedure is applicable only to samples that can be acidified and dried without significant loss of organic substances.

All forms of carbon in a sample are converted to carbon dioxide (CO_2) by catalytic combustion in a high temperature, oxygen rich atmosphere. The CO_2 formed is measured directly by an infrared detector. The amount of CO_2 is directly proportional to the concentration of total carbon (TC) in the sample.

Inorganic carbon (IC) is removed by drying the sample in a 100 oC oven, acidifying and mixing well, and drying again. The TC concentration of the prepared sample is taken to be the TOC concentration.

Test	- O401.5 Total Organic Carbon
Method Detection Limit	- 100 mg/Kg
Referenced Procedures	- USEPA SW846-9060 (Modified)

POLICY: It is the policy of Heritage Laboratories to have written Standard Operating Procedures for all analytical procedures.

I. Personnel

All laboratory personnel assigned to do this test will be trained by the Group Leader or their designee. This will be documented on group training forms. The requirements for passing training are:

BLA01 - Result $\leq$ 100 mg/Kg

CCV - Recovery 80-120%

Calibration Correlation $\geq$ 0.995

II. Safety

When working in the laboratory, wear protective equipment including safety glasses, lab coat, and gloves. Safe laboratory practices should be used at all times.

All acids used in this method are corrosive. Skin contact can produce deep burns. Flush affected areas immediately with copious amounts of cold water.

The furnace and combustion tube operate at 900 oC. Use extreme caution when performing any maintenance in this area of the instrument.

III. Equipment

A. TOC Analyzer - Shimadzu TOC-5000A and SSM-5000 or equivalent
B. Oxygen gas cylinder - regulated to 40 psi

IV. Materials

A. Reagents - All reagents are ACS reagent grade and entered into the appropriate logbook.

1. 10,000 mg/Kg TOC Stock Solution (Dextrose)
   Weigh 2.501g Dextrose into a 100 mL volumetric flask. Dilute to 100 mL with millipore water, and preserve with H_2SO_4.

2. Phosphoric Acid 1 N
   Dilute 20.75 mL of conc. H_3PO_4 to a total volume of 250 mL with millipore water.

3. TOC in Soil Standard
   Purchased from ERA or equivalent.

V. Sample Requirement

Containers	- Amber glass containers, with Teflon-lined lids
Preservatives	- refrigeration to 4 oC
Regulatory Holding Time	- 28 days
Recommended Holding Time	- 28 days

VI. Procedure

A. Instrument Set Up

1. Set total carbon furnace to 900 °C.
2. Set carrier gas flow to ~0.5 L/min.
3. Choose appropriate calibration curve.
4. Follow all instructions from manufacturer's manual.

B. Instrument Calibration

1. Perform calibration curve using the 10,000 ppm Carbon standard.
2. The curve is generated as ug Carbon vs. peak area.
3. Use the following volumes and true values:

Volume Std, uL	True Value, ug
0	0
25	250
100	1000
500	5000

C. Sample Preparation

1. Transfer approximately 10 g of sample to a clean glass jar.
2. Dry sample overnight in a 103 to 105 °C oven.
3. Homogenize sample by breaking up any clumps, and reducing the particle size by grinding.
4. Add enough 1 N Phosphoric Acid to completely cover the dried sample. Mix thoroughly.
5. Dry acidified sample overnight in a 103 to 105 °C oven.
6. Homogenize sample by breaking up any clumps, and reducing the particle size by grinding.

D. Analysis Procedure

1. Weigh a maximum of 1.0 g of prepared sample into a tared clean dry sample boat. Record weight and enter into instrument.
2. Place sample boat into analyzer and close hatch.
3. Start analyzer and push boat into fumace.
4. When analysis is finished pull boat out of furnace.

E. Quality Assurance / Quality Control

Quality control procedures described by the HES/CLO Comprehensive Quality Assurance Plan have been implemented to assure that the precision, accuracy, and completeness of data are known and documented. QAQC is routinely performed on all types of sample matrices. This QAQC includes analytical blanks and standards, duplicates, matrix spikes and matrix spike duplicates, where applicable.

Due to the lack of a carbon free solid matrix, all standards except spikes are injected onto clean dry quartz wool, with 0.999g entered as the sample weight.

Acceptance criteria for the following QAQC are determined from in-house statistical limits.

QAQC samples done per TOC run as follows:

BLA01
- Burn an empty boat
- beginning and end of run
- one per ten samples
- result $\leq$ 100 mg/Kg

LCS01
- ERA TOC in Soil Standard or equivalent
-one per run

CCV
- 10,000 mg/L TOC Standard (Dextrose)
- use 100 uL for a true value of 1000 mg/Kg
- one per ten samples, end of run

CDL01
- 10,000 mg/L TOC Standard (Dextrose)
- use 30 uL for a true value of 300 mg/Kg
- one per run

SPI01/DPS01- 10,000 mg/L TOC Standard (Dextrose)
- use 100 uL of standard
- one per ten samples, or one per run

All Total Organic Carbon data generated is reviewed by the group leader or designee for completeness and accuracy before the samples are reported. The reviewer is responsible for making sure that all relevant information has been completed. No one can review their own data. Each runsheet is initialed and dated by the reviewer.

E. Interferences / Troubleshooting

Samples containing volatile organic compounds will give low results. Due the oven drying involved in the sample preparation, many volatile compounds will evaporate before analysis.

The combustion tube operates at a high temperature under a high oxygen atmosphere. Any samples containing high levels of gasoline or other flammable materials may be an explosion hazard. These samples should be analyzed with the utmost caution.

F. Clean Up and Disposal

Clean combustion boats by removing any residual residue and rinsing with 1 N HCl and with DI water. Dry boats in a 105 °C oven.

G. Calculations

To determine spike true value:

$$TV\ mg/Kg = \frac{(\text{Vol Std, uL}) \times (10,000\ mg/L) \times (0.001)}{(\text{Weight Sample, g})}$$

VII. Documentation

All analytical information is recorded on bench sheets, and in the appropriate logbooks.

VIII. Appendix

A. Example bench sheet

IX. Disclaimer

This Standard Operating Procedure has been prepared for the sole use of HES/CLO, and may not be specifically applicable to the activities of other organizations.

• Appendix A. Example Benchsheet

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Reference No. O401.0, O401.1, G410.1, G401.2 2986

TOTAL ORGANIC CARBON

Analyst KMW 1313
Start Date 12-27-96 Start Time 11:45
End Date 12-27-96 End Time 17:30

Recorder LDF
Date 12-30-96

Reviewer PSA 2146
Date 12/30/96

HEADER	QAQC	SAMPLE	TEST CODE	TV	SPIKE	RESULT	%REC	RPD	DL	UNITS	DILUTION	COMMENTS
1	BLK1	BLANK				34.1			100	mg/kg		
2	LCS1	X289		6250		7210	115.4					
3	CDL1	CDL		300		309	102.7					
5		A396049	0401.5.0			960						LTD-N-95-5.5
4	SIS1	A396049		960	1050	1970	96.2					LTD-N-94-2.0
4	DPS1	A396049		960	1050	1930	97.6	9.4				LTD-N-94-4.0
6		A396059				610						LTD-N-94-5.5
7		A396060				U						LTD-N-93-2.0
8		A396061				2300						LTD-N-93-4.0
9		A396063				U						LTD-N-93-5.5
10		A396064				U						LTD-N-90-2.0
11		A396065				1400						
12		A396067				U						
13	BLK1	BLANK				334						
14	CCV	CCV		1000		1160	116.0					