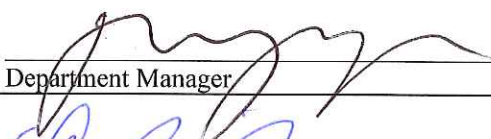

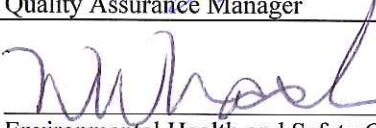
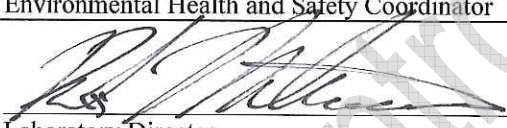


**FACILITY SOP ATTACHMENT**

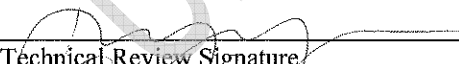
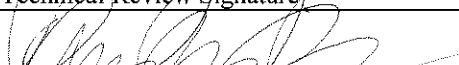
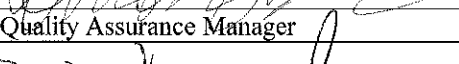
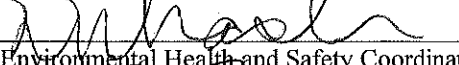
<b>SOP NUMBER: IR-WET-TOC, Rev 2 (9/28/2012)</b>	<b>CHANGE FORM ID: CF2</b>
<b>SOP TITLE: SM 5310B / EPA METHOD SW 9060A (TOTAL ORGANIC CARBON)</b>	
<p><b>REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary):</b></p> <ol style="list-style-type: none"> <li>1) Specify how replicate analyses are processed and reported</li> <li>2) Add evaluation of replicate RSD for samples</li> <li>3) Specify balance calibration criteria for solids</li> </ol>	
<p><b>CHANGE OR ADDITION (Use additional sheets if necessary):</b>  <b>Change 10.6.1 5010B to 10.6.1 Replicate Readings</b></p> <p><u>Currently:</u>      The TOC instrument is set-up to take duplicate readings from each aliquot in the sequence. The average of these readings is taken as the final result. However, if the standard deviation (based on response) of these two readings is &gt;200% or the coefficient of variation (CV) is &gt;2%, a third replicate readings is automatically performed. If the RSD and CV of this replicate and one of the previous two replicates meet acceptance criteria, they are averaged as the final result and the unused replicate is considered a statistical outlier and is automatically eliminated. In some situations a fourth replicate may also be run in the same manner. These calculations are performed by the instrument and cannot be modified. Repeatability may deteriorate due to the presence of suspended solids in samples and the instrument's ability to keep them uniformly distributed in the sample prior to introduction into the combustion tube.</p> <p><u>Revised:</u>      Replicate readings are determined from each sample aliquot in the sequence. For SM5310B, two replicates are measured. For EPA 9060A, four replicates are measured. The average of these readings is taken as the final result. The following rules are applied in evaluating the final result:</p> <ul style="list-style-type: none"> <li>• If the standard deviation (based on response) of these replicate readings is &gt;200% <b>or</b> the coefficient of variation (CV) is &gt;2%, an additional replicate readings is automatically performed. If the RSD and CV of this replicate and all but one of the initial replicates meet acceptance criteria, they are averaged as the final result and the unused replicate is considered a statistical outlier. In some situations a second additional replicate may also be run in the same manner. These calculations are performed by the instrument.</li> <li>• If replicate results straddle the required reporting limit (RL or MDL), the sample must be re-analyzed. If the replicates still straddle the required limit, the average is reported with an NCM. Averaged results are reported based on "final concentration" results with non-detect results included as zeroes in the average calculation.</li> <li>• If the Relative Standard Deviation (RSD or "SD Conc" in instrument software) of replicate readings does not meet acceptance of <math>\leq 20\%</math> (regardless of CV), the sample must be re-analyzed.</li> <li>• Repeatability may deteriorate due to the presence of suspended solids in samples and the instrument's ability to keep them uniformly distributed in the sample prior to introduction into the combustion tube.</li> </ul> <p><b>Remove 10.6.2</b>  <del>The TOC instrument is set up to take quadruplicate readings (4 injections drawn for one vial). The instrument will perform at least four (4) injections to at most six (6); the instrument will calculate the final result under</del></p>	

**FACILITY SOP ATTACHMENT**

<b>SOP NUMBER: IR-WET-TOC, Rev 2 (9/28/2012)</b>		<b>CHANGE FORM ID: CF2</b>	
<p><del>the same conditions (SD/CV) as specified for 5310B. Note: For all calibration and batch QC samples (MB, LCS), quadruplicate analysis are not required.</del></p> <p><b>Add to 11.2</b>          NOTE: The analytical balance must be verified on the day of use with masses that bracket the sample weight measured</p>			
<b>Prepared By: D. Dawes and Y. Zakhrabov</b>			
<b>APPROVED BY:</b>			
 Department Manager		9/17/13 Date	
 Quality Assurance Manager		9-17-2013 Date	
 Environmental Health and Safety Coordinator		09/16/2013 Date	
 Laboratory Director		09/16/2013 Date	

Control Copy Number \_\_\_\_\_

## FACILITY SOP ATTACHMENT

<b>SOP NUMBER: IR-WET-TOC, Rev 2 (9/28/2012)</b>		<b>CHANGE FORM ID: CF1</b>	
<b>SOP TITLE: SM 5310B / EPA METHOD SW 9060A (TOTAL ORGANIC CARBON)</b>			
<b>REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary):</b>			
Update of procedure for analysis of wipes following DI Leach preparation			
<b>CHANGE OR ADDITION (Use additional sheets if necessary):</b>			
<b>Section 10.3.2 Wipes</b>			
<u>Currently reads:</u>			
When a wipe sample is to be analyzed, place the wipe in a 40 ml VOA vial and fill with 40.0mL of reagent grade water			
<ul style="list-style-type: none"> <li>• Preserve the vial with 1:1 HCl to a pH &lt; 2.</li> <li>• Shake the 40 ml VOA vial for approximately 20-30 seconds or place on a shaker plate for approximately 5 minutes.</li> <li>• Filter to remove any particulates. This prevents the particulates from clogging the TOC Analyzer</li> </ul>			
<u>Revise to read:</u>			
When a wipe sample is to be analyzed, place the wipe in a <b>4-oz (or equivalent) glass jar and add 50.0mL</b> of reagent grade water.			
<ul style="list-style-type: none"> <li>• Preserve the vial with 1:1 HCl to a pH &lt; 2.</li> <li>• Shake the jar for approximately 20-30 seconds or place on a shaker plate for approximately 5 minutes.</li> <li>• Filter to remove any particulates. This prevents the particulates from clogging the TOC Analyzer</li> <li>• Prepare a Method Blank and LCS/LCSD in the same manner using laboratory-grade filter paper as the wipe.</li> <li>• An LCSD is prepared and analyzed unless sufficient wipes are supplied to prepare and analyze a sample duplicate</li> <li>• NOTE: A wipe must be prepared intact and in its entirety. Preparing on a portion of a wipe or otherwise splitting a wipe is NOT appropriate.</li> </ul>			
<b>Prepared By: D. Dawes</b>			
<b>APPROVED BY:</b>			
 _____ Technical Review Signature		_____ Date 8/14/13	
 _____ Quality Assurance Manager		_____ Date 8-14-2013	
 _____ Environmental Health and Safety Coordinator		_____ Date 08/14/2013	
 _____ Laboratory Director		_____ Date 08/14/2013	

**Title: Total and Dissolved Organic Carbon  
 EPA Method 9060A & SM 5310B**

<b>Approvals (Signature/Date):</b>			
	9/25/12		09/25/2012
Tung Nguyen Technical Manager	Date	William Nash Health & Safety Coordinator	Date
	9/24/12		10/2/12
David Dawes Quality Assurance Manager	Date	Fred Haley Laboratory Director	Date

**Copyright Information:**

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

**THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:**

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED

<b>Facility Distribution No.</b> _____	<b>Distributed To:</b> _____
--	------------------------------

## 1.0 SCOPE AND APPLICATION

- 1.1 Methods 9060A and 5310B are used to determine the concentration of organic carbon and dissolved organic carbon in water and solid samples. Although commonly referred to as Total Organic Carbon (TOC), the methodology described actually measures Non-Purgeable Organic Carbon as the process used to remove inorganic carbon also removes any volatile organics.
- 1.2 These methods can be used to analyze for organic carbon in drinking, surface and saline water, and in domestic and industrial wastes and solids. For the determination of Dissolved Organic Carbon (DOC), samples must be filtered prior to preservation. The nominal reporting limit for waters is 1 mg/L and for soils, 5000 mg/Kg.
- 1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in "Validation of Methods" in the Quality Assurance Manual

## 2.0 SUMMARY OF METHOD

An acidified and sparged aliquot of aqueous sample is introduced to a heated combustion tube filled with oxidation catalyst. (For solids, the sample is acidified and dried.) Organic carbon present in the sample is converted to carbon dioxide (CO<sub>2</sub>) by catalytic combustion. Chemical detection is measured by a Non-Dispersive Infrared analyzer (NDIR).

## 3.0 DEFINITIONS

- 3.1 Carbon-containing compounds may exist in various forms that can be measured by carbonaceous analyzers. These include:
- Soluble, nonvolatile organic carbon: e.g., natural sugars.
  - Soluble, volatile organic carbon: e.g., mercaptans, alkanes and alcohols.
  - Insoluble, partially volatile carbon: e.g., oils.
  - Insoluble, particulate carbonaceous materials: e.g., cellulose fibers
  - Inorganic carbon (IC): carbonate, bicarbonate and dissolved CO<sub>2</sub>.
  - Total carbon (TC): summation of Total Organic Carbon (TOC) and IC.
  - Total Organic Carbon (TOC): all carbon atoms covalently bonded in organic molecules. It is the summation of Volatile Organic Carbon (VOC) and Non-Purgeable Organic Carbon. (In practice, VOC is negligible in many ground and surface waters).
  - Dissolved Organic Carbon (DOC): all TOC compounds that can be filtered through a 0.2µm membrane filter prior to acid preservation.
- 3.2 Fractional Organic Carbon (FOC) may be reported for solid samples as a percentage of grams of carbon divided by the dry weight of the sample in grams.
- 3.3 There are no additional specific definitions associated with this test. See the laboratory QA manual and methods 9060A and Standard Methods 5310B for general definitions.

## 4.0 INTERFERENCES

Inorganic Carbon can be an interference under the conditions of this test. It can be eliminated by acidifying and subsequently purging samples with an inert gas. This purging will, however, remove any traces of volatile organic carbon.

Water samples with high chloride concentrations of 1000ppm or above should be diluted to avoid interferences.

## 5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

### 5.1 Specific Safety Concerns or Requirements

Personal Protective Equipment Required: Safety Glasses, Labcoat, Nitrile Gloves  
The auto sampler has a probe that is sharp; use caution not to stick yourself.

The furnace is very hot and can cause severe burns if touched.

### 5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Hydrogen phthalate	Irritant	None Listed	Harmful if swallowed. Maybe harmful if inhaled. Use with adequate ventilation. Avoid contact with eyes, skin, and clothes. Inhalation may cause irritation. Contact with skin may cause irritation. Contact with eyes may cause irritation, redness and pain. Ingestion may cause nausea, vomiting and diarrhea
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

## 6.0 EQUIPMENT AND SUPPLIES

### 6.1 Instrumentation

- 6.1.1 Shimadzu TOC-5000A Analyzer with ASI-5000A autosampler (7mL vials)
- 6.1.2 Shimadzu TOC-VCSH Analyzer with SA-V autosampler (24mL vials)
- 6.1.3 OI Analytical TOC-Soil (for soil analysis)

**6.2 Supplies**

- 6.2.1 Porcelain sample boats
- 6.2.2 Muffle furnace, ~600±50°C
- 6.2.3 Centrifuge
- 6.2.4 Filtration device (0.2µ)
- 6.2.5 Graduated cylinders, Class A
- 6.2.6 Volumetric flasks, class A
- 6.2.7 1 L plastic bottles with lids
- 6.2.8 50 ml , 10mL glass sampling tubes
- 6.2.9 Volumetric pipettes
- 6.2.10 Analytical balance
- 6.2.11 Parafilm

**7.0 REAGENTS AND STANDARDS**

All purchased and prepared reagents and standards must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory.

**7.1 Reagents**

- 7.1.1 Laboratory Reagent Grade Water (as referred to as “NanoPure” or “UltraPure”)
- 7.1.2 Hydrochloric Acid (HCl), 2N
- 7.1.3 Hydrochloric Acid (HCl), 1:1
- 7.1.4 Potassium hydrogen phthalate (KHP), primary standard grade (or 1000ppm carbon standard from an outside vendor)
- 7.1.5 Glucose, anhydrous -- ACS grade
- 7.1.6 Fructose, crystalline --USP grade
- 7.1.7 Glass wool -- OI Analytical (or equivalent), 99.99% silicon dioxide
- 7.1.8 Oxygen gas (O<sub>2</sub>), UHP grade
- 7.1.9 Compressed air, Ultra Zero grade

**7.2 Standards**

- 7.2.1 Carbon standard (1000 ppm) - LabChem or equivalent (second source)

**8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE**

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber Glass bottle	250 mLs	HCl, pH < 2; Cool >0 to 6°C	28 Days	40 CFR Part 136.3
Soils	Sleeve or Jar	100 grams	Cool >0 to 6°C	28 Days	N/A

**9.0 QUALITY CONTROL**

## **Sample QC**

The following quality control samples are prepared with each batch of samples. Each of these QC samples may be re-analyzed once if it doesn't pass, in order to verify that the failure wasn't due to a physical or mechanical problem.

### **9.1.1 Method Blank (MB)**

Prepare and analyze a method blank (MB) for each matrix and with every batch of 20 samples, or less. Check that there are no analytes detected at or above the reporting limit. If the method blank shows contamination, re-prepare all samples in the batch unless:

- The samples are ND (Flag the data and write an NCM).
- The sample result is > 10x the blank level (Flag the data and write an NCM).

### **9.1.2 Laboratory Control Sample (LCS)**

Prepare and analyze a primary source laboratory control sample (LCS) for every batch of 20 samples or less. The LCS recovery must be within laboratory acceptance limits (see attachment 1). If the LCS is outside of these limits, re-prepare the whole batch and/or re-calibrate the system unless:

- The LCS recovery is above the upper limit and samples are ND. Flag the data and write an NCM.

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract. When an LCS/LCSD pair is analyzed, the RPD must also be within acceptance limits. If not, the problem must be identified and corrected before samples can be reported.

### **9.1.3 Matrix Spike and Matrix Spike Duplicate.**

The sample for MS/MSD is randomly selected, unless specifically requested by a client. Prepare and analyze a matrix spike (MS) and a matrix spike (MSD) duplicate for each matrix and with every batch of 20 samples, or less. The recovery and relative percent difference must be within laboratory acceptance limits (see attachment 1), unless there is matrix interference. In the case of matrix interference, the data should be flagged and an NCM written.

## **9.2 Instrument QC**

The following instrument QC samples are run with each analytical sequence. Each of these QC samples may be re-analyzed once if it does not pass, in order to verify the failure wasn't due to a physical or mechanical problem. Re-analysis must be performed before any batch QC or client samples are analyzed

### **9.2.1 Initial Calibration Verification (ICV)**

Immediately after the initial calibration, analyze the Secondary source standard. Verify that that its response is within  $\pm 10\%$  of the true value.

- If not, re-prepare and re-analyze the ICV standard once
- If the ICV is still out of control, re-calibrate the system

### **9.2.2 Continuing Calibration Verification (CCV)**



Analyze a CCV standard after every 10 sample aliquots or less and at the end of the sequence. The recovery must be within  $\pm 10\%$  of the true value. If the CCV is outside recovery limits, re-prepare/re-analyze once. If it is still out the instrument must be recalibrated and samples not bracketed by acceptable CCVs be reanalyzed unless:

- if the CCV is out high, any ND samples may be reported with a qualifier.

Follow every CCV with a CCB (calibration blank). The CCB must read less than the method reporting limit. If the CCB is above the reporting limit, re-prepare/re-analyze once. If it is still out, the instrument must be recalibrated and samples not bracketed by acceptable CCBs be reanalyzed unless:

- if the CCB is out high, any ND samples may be reported with a qualifier.

### **9.2.3 Calibration Acceptance Summary**

Prepare a calibration curve by plotting the response (peak area) of at least a 3 standards and a blank against the corresponding carbon concentrations. The resulting correlation coefficient (r) must be  $\geq 0.995$ . If this criteria is not met:

- re-evaluate the curve without the highest calibration standard (60 ppm) removed or
- re-prepare the calibration standards and repeat the calibration.

If continuing the use of a previous calibration curve, the calibration must be verified on each day of use by analyzing a calibration blank and a midpoint calibration standard. If the following criteria are not met, a new calibration curve must be prepared:

- The calibration blank result must be less than the reporting limit.
- The midpoint recovery must be within  $\pm 10\%$  of the true value

## **10.0 PROCEDURE FOR AQUEOUS SAMPLES**

### **10.1 Reagent Preparation**

#### **10.1.1 Hydrochloric Acid, 2N**

Prepare a 2N Hydrochloric acid by adding 16.6mL of concentrated ACS reagent grade HCl to laboratory Reagent Grade Water, and bring up to a total volume of 100mL. Assign a shelf life of six months from the date of preparation.

### **10.2 Standard Preparation**

#### **10.2.1 Primary Standard Preparation (1000ppm Carbon)**

Dissolve 0.2128 g of Potassium Hydrogen Phthalate (KHP) in reagent grade water, and bring to volume in a 100ml volumetric flask. Use this prepared standard for the calibration and midpoint verification standards. Refrigerate at  $>0$  to  $6^{\circ}\text{C}$ . Shelf life is six months from preparation date.

Alternately, a 1000ppm carbon standard can be purchased. The certificate of analysis must be kept on file. Storage conditions and shelf life must be in accordance with vendor recommendations (If not provided, follow the storage conditions for the laboratory-prepared standard.)

#### **10.2.2 Secondary Standard Preparation (1000ppm Carbon)**

Dissolve 0.2128 g of KHP (from a different vendor than the primary standard) in reagent grade water, and bring to volume in a 100ml volumetric flask. Use this prepared standard for the calibration verification. Refrigerate at  $>0$  to  $6^{\circ}\text{C}$ . Shelf life is six months from preparation date.

Alternately, a 1000ppm carbon standard can be purchased. The certificate of analysis must be kept on file. Storage conditions and shelf life must be in accordance with vendor recommendations (If not provided, follow the storage conditions for the laboratory-prepared standard.)

### 10.2.3 Working Standard Preparation

Prepare the working standards from the appropriate stock standard (1000 ppm KHP) by pipetting the volumes below to 100 mL volumetric flasks partially filled with reagent grade water. Add 1.0 mL of 2N HCL to all standard flasks and bring to volume. All working calibration standards must be used on the day of preparation.

Standard	Conc. (mg/L Carbon)	mL of 1000 ppm C (KHP)	mL 2N HCL	Final Vol (mL)	Matrix
Cal0 (MB/CCB)	0	0	1.0	100	Reagent Grade Water
Cal1	1.0	0.1	1.0	100	Reagent Grade Water
Cal2	10.0	1.0	1.0	100	Reagent Grade Water
Cal3	40.0	4.0	1.0	100	Reagent Grade Water
Cal4 (optional)	60.0	6.0	1.0	100	Reagent Grade Water
ICV*	10.0	1.0*	1.0	100	Reagent Grade Water
Midpoint	20.0	2.0	1.0	100	Reagent Grade Water
CCV/LCS	10.0	1.0	1.0	100	Reagent Grade Water
MS/MSD	5.0	0.25	--	50	Source Sample

\*The ICV must be from a different source than that used for the primary standard (if purchased)

### 10.3 Sample Preparation

#### 10.3.1 Aqueous samples

Test the pH of all water samples using pH paper. (Do not allow paper to come in contact with sample directly; use a disposable pipette.) Record the pH measurement on the sample table print out from the instrument. If not properly preserved to pH < 2, add a few drops of 1:1 HCL and re-test the pH to verify the sample is properly preserved. Notify the Project Manager immediately and initiate an NCM if the sample was improperly preserved. Record HCL lot number.

**NOTE 1:** Water samples that contain particulates should be filtered to prevent clogging the TOC Analyzer. When samples are filtered, a filtered blank must also be prepared and analyzed. An NCM must be written to indicate filtration was performed due to sample matrix.

**NOTE 2:** Samples that have solids that may not clog the instrument but which do not readily remain in suspension while on the instrument awaiting analysis, must be analyzed on an instrument configured such that:

- the injected aliquot is taken from the vial immediately after sparging
- the instrument is configured with a stirrer to keep the solid suspended prior to sampling

#### 10.3.2 Wipes

When a wipe sample is to be analyzed, place the wipe in a 40 ml VOA vial and fill with 40.0mL of reagent grade water

- Preserve the vial with 1:1 HCL to a pH < 2.
- Shake the 40 ml VOA vial for approximately 20-30 seconds or place on a shaker plate for approximately 5 minutes.
- Filter to remove any particulates. This prevents the particulates from clogging the TOC Analyzer.

Wrap all sample tubes with parafilm to prevent the CO<sub>2</sub> and O<sub>2</sub> from getting into the sample.

#### **10.4 Instrument Initialization**

**10.4.1** Turn on the compressed air.

**10.4.2** Turn on the TOC analyzer and the autosampler. Turn on the gas and adjust the baseline to zero by turning the baseline adjustment until the baseline is stable.

**10.4.3** Adjust the compressed air flow to 150 ml/min.

**10.4.4** Adjust the sparging flow to 100mL/min.

**10.4.5** Set the autosampler to:

- NPOC - nonpurgable organic carbon
- Purging time - 3 minutes
- Washing - 4 times
- Injection - 2-4 for 5310B; 4-6 for 9060A

Recall the calibration file (if the old calibration remains valid) or load a new calibration curve sequence.

#### **10.5 Calibration**

For a new calibration, load the calibration standards in ascending concentration order followed by the ICV. Verify all calibration criteria are met before proceeding.

To continue using a previous calibration, recall the calibration file, analyze an ICV, ICB, CCV and midpoint standard. Verify acceptance criteria are met before proceeding.

#### **10.6 Sample Analysis**

A typical daily run sequence is listed below:

Instrument Blank
ICV
ICB
CCV high (30ppm)
Midpoint (20ppm)
LCS/CCVL (10ppm)
MB
10 samples (including MS/MSD)
CCV / CCB
10 samples
CCV / CCB

##### **10.6.1 5310B**

The TOC instrument is set-up to take duplicate readings from each aliquot in the sequence. The average of these readings is taken as the final result. However, if the standard deviation (based on response) of these two readings is >200% or the coefficient of variation (CV) is >2%, a third replicate readings is automatically performed. If the RSD and CV of this replicate and one of the previous two replicates meet acceptance criteria, they are averaged as the final result and the unused replicate is considered a statistical outlier and is automatically eliminated. In some situations a fourth replicate may also be run in the same manner. These calculations are performed by the instrument and cannot be modified. Repeatability may deteriorate due to the

presence of suspended solids in samples and the instrument's ability to keep them uniformly distributed in the sample prior to introduction into the combustion tube.

### 10.6.2 EPA 9060A

The TOC instrument is set-up to take quadruplicate readings (4 injections drawn for one vial). The instrument will perform at least four (4) injections to at most six (6); the instrument will calculate the final result under the same conditions (SD/CV) as specified for 5310B. Note: For all calibration and batch QC samples (MB, LCS), quadruplicate analysis are not required.

### 10.6.3 Dissolved Organic Carbon (DOC)

If dissolved organic carbon (DOC) is to be determined, filter the unpreserved sample through a 0.2um membrane filter and preserve to pH<2 with 1:1 HCl. Analyze a filtered blank and a filtered LCS for each batch of samples. Verify that the blank does not contain any DOC above the reporting limit.

## 10.7 Preventative Maintenance

The following preventative maintenance should be performed at the frequency indicated:

Frequency	Action
Daily consumable checks	Check that gas tank pressure is >500psi prior to start of run
	Verify that the carrier gas pressure on the instrument is 200 kPa
	Verify that the gas flow rate is 150 mL/min
	Verify that there is sufficient acid reagent for the day's run
	Check the water level in the drain vessel--must be within 10mm of the overflow tube
	Verify that the water level in the humidifier is above the "low" mark
	Verify that the water in the rinse bottle is above the 2L mark
Daily instrument condition checks	Zero point detection
	Regeneration of the TC catalyst
	Regeneration of the IC solution
	Residue removal
As needed	Replace the halogen scrubber (when the front of the discoloration band reaches the top of the halogen scrubber)
	Replace the UHP air cylinder when the is less than ~200 kPa
	Replace the syringe

Record all performed maintenance in the instrument maintenance logbook.

If an instrument is unusable or has limitation to its use, it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

## 11.0 PROCEDURE FOR SOLID SAMPLES

### 11.1 Standard preparation

#### 11.1.1 Primary stock Calibration Standard (25,000 ppm Carbon)

Prepare the 2.5% Carbon Calibration stock standard by diluting 6.25 g **Glucose** into 100 ml reagent grade water. Shelf life is six months when refrigerated at >0 to 6°C.

**11.1.2 Secondary stock Calibration Standard (50,000 ppm Carbon)**

Prepare the 50,000 ppm Carbon Verification stock solution by diluting 12.50g **Fructose** into 100 ml reagent grade water. Shelf life is six months when refrigerated at >0 to 6°C.

**11.1.3 Working Standards**

Prepare the fresh calibration standards from the appropriate stock source by pipetting the volumes below to the combustion furnace boats containing 100 mg of glass wool and loading them into the Solid Sample Module.

Standard	Std Conc. (mg/kg) <sup>1</sup>	Stock Source	uL of Stock Std.
Cal0	0	Primary	0
Cal1	5,000	Primary	20
Cal2	12,500	Primary	50
Cal3	40,000	Primary	160
ICV	10,000	Secondary	20
Midpoint	20,000	Primary	80
LCS/CCV	10,000	Primary	40

<sup>1</sup> Concentration based on total mg Carbon in 100mg of sample

**11.2 Sample Preparation**

Weigh 100 mg of a well mixed sample into the TOC combustion boat.

For Method Blank (MB), weigh out 100 mg of a glass wool.

For MS/MSD samples, add 80 uL of the **Primary** Stock Standard for a spike level of 20,000ppm (mg/Kg) into 100 mg of a well mixed sample.

Add 1 drop of 2N Hydrochloric acid or enough to dampen sample and remove interferences. Process all QC in the same manner as regular samples.

Note: if bubbles form on the sample after the addition of acid, a few more drops of acid may be needed to thoroughly remove interferences.

Dry in an oven for 5-10 minutes at 105±5°C.

**11.3 Instrument Initialization**

- Soak sample boats in 2N HCL for at least 1 hour. Wash the boats with soap and water and then dry them at 550°C for at least 1 hour
- Turn on the compressed oxygen supply (The combustion gas pressure should be 2.0 kgf/cm<sup>2</sup>.)
- Turn on the TOC analyzer, the Solid Sample Module (SSM), and the gas
- Set the instrument to SSM (2)

- Set the furnace to 900°C
- Create sequence and save files as “Rundate” e.g. 090101

#### 11.4 Calibration

For a new calibration, load the calibration standards in ascending concentration order followed by the ICV. Verify all calibration criteria are met before proceeding.

To continue using a previous calibration, recall the calibration file, analyze a calibration blank and midpoint standard. Verify acceptance criteria are met before proceeding.

Insert the calibration standard-containing boats on to the instrument. Allow the combustion furnace heat to 900°C prior to analysis.

#### 11.5 Sample Analysis

Insert the sample boat into the Solid Sample Module. Let the combustion furnace heat to 900°C for analysis.

For high level samples, use a smaller sample aliquot.

Instrument Blank
Calibration sequence
ICV
ICB
Midpoint
LCS
MB
(no greater than) 10 determinations*
CCV/CCB
(no greater than) 10 determinations
CCV/CCB

\*Including MS and MSD determinations in quadruplicate

#### 11.6 Preventative Maintenance

After analyzing soil samples, turn the TOC soil furnace back to stand-by temperature (36°C). **Turn off the oxygen gas.**

### 12.0 CALCULATIONS / DATA REDUCTION

#### 12.1 Accuracy

$$\frac{\text{ICV} / \text{CCV}, \text{LCS \% Recovery}}{\text{known concentration}} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

#### 12.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

### 12.3 Calibration

$$y = mx + b \qquad C = \frac{(y - b)}{m}$$

Where y is the instrument response (area)

m is the slope

x is the concentration

b is the y-intercept

C is the raw sample concentration (instrument reading)

### 12.4 Concentration

- Water: TOC (mg/L) = C x D
- Soil: TOC (mg/Kg) = C x S/100

Where:

C = instrument reading (ppm)

D = Dilution Factor

S= Sample weight used (mg)

### 12.5 Procedure for report of FOC (Fractional Organic Carbon) by combustion and NDIR detection.

FOC may be reported for solid samples as a percentage of grams of carbon divided by the dry weight of the sample in grams. More simply,

$$\% \text{FOC} = \frac{\text{TOC}(\text{mg} / \text{kg})}{10,000} \times \frac{1}{\text{percent\_solid}}$$

where TOC (mg/kg) is determined in quadruplicate by Method 9060A

percent\_solid is the fractional weight percentage of sample dried at 103-105°C

Example: a sample with a TOC result of 25,000 mg/kg and a percent solid result of 85% will yield an FOC result of  $(25,000) \div (10,000) \times 1/0.85 = 2.94\%$

### 12.6 Reporting soil sample quadruplicate replicates when sample is heterogeneous.

If at least one replicate is significantly different from the average (e.g. a magnitude difference for higher levels or several times the RL for concentrations near the RL), the sample is reanalyzed in quadruplicate. If the re-analysis also has one or more replicates significantly different from the average, then the sample result will be reported from the average of all four but will be qualified with an NCM stating "Sample Cannot be adequately homogenized; reported result is an estimate."

## 13.0 **METHOD PERFORMANCE**

### 13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure as described in laboratory's SOP, IR-QA-

MDL. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are performed annually.

### **13.2 Demonstration of Capabilities**

Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples. An IDOC consists of 4 consecutive LCS samples at 1 to 4 times the RL with an average recovery and RSD within the in-house statistical limits. An on-going DOC must be performed annually. An ODOC can be 4 consecutive LCSs at mid level or a passing PT.

### **13.3 Training Requirements**

The analyst must have documented training, including reading of the SOP and source methods, conducted by the department manager, senior chemist, or other analyst with training documentation and a passing DOC.

### **14.0 POLLUTION CONTROL**

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in the "Waste Management and Pollution Prevention" section of the Corporate Environmental Health and Safety Manual (CW-E-M-001).

### **15.0 WASTE MANAGEMENT**

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the laboratory's Waste Disposal SOP (IR-EHS-WASTE). The following waste streams are produced when this method is carried out:

**Hydrochloric acid waste.** This waste is generated from TOC analysis. Waste bulked with the acid waste stream.

**Unused standards.** Standards are collected in the satellite waste container for one of the waste streams of the method. Pour the standard into the appropriate satellite container, rinse the original container, and collect the rinsate in the satellite container. The original container can be placed in the regular trash.

**Non-Hazardous waste** is disposed of by pouring the samples water that have been extracted into the sink, measuring the pH and neutralizing the water using soda ash, and then draining the neutralized contents into the sewer system.

### **16.0 REFERENCES / CROSS-REFERENCES**

- 16.1** EPA Method 9060A, Revision 1, November 2004
- 16.2** Standard Method 5310 B, 20<sup>th</sup> Edition 1998

### **17.0 METHOD MODIFICATIONS**



Item	Methods 9060A & 5310B	Modification
1	9060 Sec. 5.1 SM 5310B Sec 3.a	Reagent water is not boiled before use to remove dissolved CO <sub>2</sub> . Provided the method blank is below the RL, boiling is not necessary.
2	9060 Sec. 4.1	Samples are homogenized in accordance with the laboratory Subsampling SOP.
3	9060 Sec.7.6	Quadruplicate analyses by EPA 9060A are reported as an average rather than average and range, unless specifically requested by the client.
	9060A	For soil analysis, CCV/CCB frequency has been changed from every 10 to every 12 determinations.
4	9060 Sec. 8.3	Calibration verification is performed every 10 samples rather than every 15.
5	9060 Sec. 8.4	An MS/MSD is prepared every 20 samples rather than an MS every 10.

## 18.0 ATTACHMENTS

- 18.1 **Attachment 1:** Analysis Information
- 18.2 **Attachment 2:** Calibration Review Checklist
- 18.3 **Attachment 3:** Data Review Checklist
- 18.4 **Attachment 4:** Datatypes

## 19.0 REVISION HISTORY

### 19.1 **Revision 0, dated 17 December 2007**

- Integration for TestAmerica and STL operations
- This revision supersedes 415\_1.SOP, revision 9 (06/18/07)

### 19.2 **Revision 1, dated 17 February 2010**

- This revision supersedes IR-WET-TOC, revision 0 (12/17/07)
- Changes made in accordance with "Template\_Method SOP Format\_R2-1"
- Addition of table for method modifications
- Revision of Standard Preparation for Solid Samples
- Addition of procedure for report of FOC (Fractional Organic Carbon) by combustion and NDIR detection.
- Clarification of how soil sample quadruplicate replicates are analyzed and reported when sample is heterogeneous.
- Addition of Calibration Review Checklist.
- Revision of Demonstration of Capabilities section.
- Prepared by LH

### 19.3 **Revision 2, dated 28 September 2012**

- This revision supersedes IR-WET-TOC, revision 1 (02/17/10)
- Removed references to 415.1

- Addition of tables for working standard preparation
- Added note regarding samples with settleable solids
- Specification of grade of gases used
- Revised sequence of analysis
- Updated data review checklist
- Added Datatypes and attachment
- Prepared by YZ and DD

Uncontrolled Document

**Attachment 1  
Analysis Information**

		<b>TestAmerica Irvine</b>				<b>08/21/2012</b>			
<b>Analytical Method Information</b>									
<b>Analyte</b>	<b>MDL</b>	<b>Reporting Limit</b>	<b>Surrogate %R</b>	<b>Duplicate RPD</b>	<b>Matrix Spike %R</b>	<b>Matrix Spike RPD</b>	<b>Blank Spike / LCS %R</b>	<b>Blank Spike / LCS RPD</b>	
<b>TOC Water (EPA 9060A/SM5310B)</b>									
<b>Preservation:4 C, HCL</b>									
<b>Container:250 mL Glass w/ HCl</b>									
<b>Amount Required:120 ml</b>									
<b>Hold Time:28 days</b>									
Total Organic Carbon	0.50	1.0 mg/l			80 - 120	20	90 - 110	20	
<b>TOC - Soil (EPA 9060A)</b>									
<b>Preservation:4 C, Cool</b>									
<b>Container:4 oz Jar/BrassSleeve</b>									
<b>Amount Required:100 grams</b>									
<b>Hold Time:28 days</b>									
Total Organic Carbon	2200	5000 mg/kg			70 - 130	30	90 - 110	20	

		<b>TestAmerica Irvine</b>				<b>12/13/2007</b>			
<b>Analytical Method Information</b>									
<b>Analyte</b>	<b>MDL</b>	<b>Reporting Limit</b>	<b>Surrogate %R</b>	<b>Duplicate RPD</b>	<b>Matrix Spike %R</b>	<b>Matrix Spike RPD</b>	<b>Blank Spike / LCS %R</b>	<b>Blank Spike / LCS RPD</b>	
<b>TOC - Dissolved in Water (EPA 9060A/SM5310B)</b>									
<b>Preservation:Filtration + HCl, pH&lt;2, 4 C</b>									
<b>Container:250 mL Glass</b>									
<b>Amount Required:120 ml</b>									
<b>Hold Time:28 days</b>									
Dissolved Organic Carbon	0.50	1.0 mg/l			80 - 120	20	90 - 110	20	

**Attachment 2**  
**Calibration Review Checklist**

**CALIBRATION CHECKLIST**  
Total Organic Carbon – SW 9060 / SM5310 B & C

Analyst: _____	2 <sup>nd</sup> Level Review: _____
Analysis Date: _____	Date: _____
Instrument ID: _____	Calibration File #: _____

Analyst Rev    2<sup>nd</sup> Level Rev

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

**Calibration:**

As needed  
Coefficient of linearity  $r^2 \geq 0.995$

2<sup>nd</sup> source ICV :    %REC = 90 – 110

Calibration date and file checked for completeness and accuracy:

- Correct date, analyst's name and calibration file
- Correct instrument parameters and calculation method
- Peak shape and baseline acceptable
- Calibration summary and raw data match for calibration levels and area counts

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Attachment 3  
 Data Review Checklist**

**DAILY REVIEW CHECKLIST  
 Total Organic Carbon – SW 9060 / SM5310 B&C**

Analyst: _____	2 <sup>nd</sup> Level Review: _____
Analysis Date: _____	Date: _____
Instrument ID: _____	Calibration File # _____
QC Batches: _____	

**Analyst Rev    2<sup>nd</sup> Level Rev**

_____	_____	New sequence file created for each day of analysis
_____	_____	<b>Mid-Point (MP)/CCV/LCS Standard:</b> %R = 90 - 110
_____	_____	<b>ICV Standard:</b> %R = 90 - 110
_____	_____	<b>ICB/MB (Instrument Blank/Method Blank):</b> < Reporting Limit
_____	_____	<b>LCS:</b> every batch of 20 samples or less
_____	_____	%REC (Water matrix): 90 - 110
_____	_____	%REC (Soil matrix): 90 - 110
_____	_____	<b>MS/MSD:</b> every batch of 20 samples or less
_____	_____	%REC (Water matrix): 80 - 120
_____	_____	%REC (Soil matrix): 70 - 130
_____	_____	<b>CCV:</b> every 10 aliquots/end of run for waters; every 12 determinations for soils
_____	_____	%R = 90 - 110
_____	_____	<b>CCB:</b> After every CCV
_____	_____	-RL (or 0.1x sample concentration)
_____	_____	<b>EPA 9060:</b> Quadruplicate analysis, four (4) determinations are analyzed per sample
_____	_____	For all aqueous samples, pH is checked and recorded on instrument sample table printout
_____	_____	<b>NCM required:</b> Yes: _____    No: _____
_____	_____	All samples checked for dilutions, accuracy, precision, linear range, transcription errors and bracketed between compliant CCV/CCB

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**Attachment 4**  
**Datatypes**

Method Code	Level	Datatype Description	Value to Enter	Units
9060 & 5310B	BATCH	Analysis comment	If needed	N/A
9060 & 5310B	ANALYSIS	Batch Comment	If needed	N/A
9060 & 5310B	ANALYSIS	Final weight/volume of sample	Enter weight/volume	mL
9060 & 5310B	BATCH	Lot # of hydrochloric acid	[Specify lot or TALS ID]	N/A
9060 & 5310B	BATCH	HCl Vendor	N/A	N/A
9060 & 5310B	ANALYSIS	Initial weight/volume of sample	Enter weight/volume	mL
9060 & 5310B	BATCH	Lot # of Phosphoric Acid	N/A	N/A
9060 & 5310B	BATCH	Sodium Persulfate Reagent ID Number	N/A	N/A

Uncontrolled Document