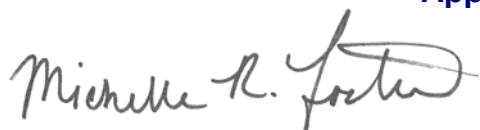


**Title: Total Organic Carbon
Dissolved Organic Carbon
Method: 9060A and SM5310C**

Once printed, this is considered an uncontrolled document.

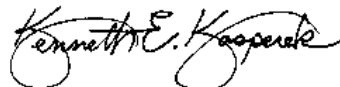
Approvals (Signature/Date):



2/23/18

Michelle Foster
Department Manager

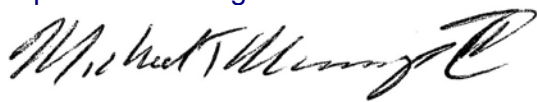
Date



2/23/18

Kenneth Kasperek
Laboratory Director

Date



2/23/18

Michael Moss crop
Quality Assurance Manager

Date

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Facility Distribution No. _____

Distributed To: _____

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP was derived from Standard Method 5310C and EPA Method 9060A. Applicable matrices are aqueous samples, soils, or wastes. The standard reporting limit is 1.0 mg/L.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 10 in the Quality Assurance Manual.

2.0 Summary of Method

- 2.1** This method is applicable to all aqueous samples including ground, drinking, surface, and saline waters, as well as domestic and industrial wastes.
- 2.2** Solids and wastes may be analyzed for leachable Total Organic Carbon after first generating a leachate using the ASTM Shake Extraction Procedure.
- 2.3** Total Organic Carbon (TOC) is determined by heated persulfate-oxidation. Each form of carbon is ultimately measured as carbon dioxide (CO₂) by a nondispersive infrared detector (NDIR) that has been calibrated to directly display the mass of CO₂ detected.
- 2.4** TOC is determined by measuring the carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of Total Inorganic Carbon (TIC), sodium persulfate, a strong oxidizer, is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide.
- 2.5** 9060A total and dissolved methods both require 4 replicates per sample. SM5310C total and dissolved require 2 replicates per sample.
- 2.6** Dissolved Organic Carbon (DOC): 9060A DOC and 5310C DOC must be filtered through a 0.45 µm filter, if not already field filtered.

3.0 Definitions

All definitions are consistent with those described in Appendix 2 of the Glossary contained in TestAmerica Buffalo's Quality Manual.

- 3.1** Organic carbon: carbon present in the form of organic carbon-based compounds.
- 3.2** Inorganic carbon: carbon present in the form of inorganic compounds; usually carbon dioxide.

4.0 Interferences

Inorganic halides in samples compete with the organics for persulfate. The Model 1030 is able to analyze samples with up to 30 mg of chlorine without any modification. When samples contain over 30 mg of chlorine, additional persulfate reagent, increased TOC reaction time and/or a halide scrubber option may be necessary.

5.0 **Safety**

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 **Specific Safety Concerns or Requirements**

Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

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 Safety Data Sheets (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material ¹	Hazards	Exposure Limit ²	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm- ceiling	Inhalation of vapors will cause irritation of the nasal and respiratory system. 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.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Persulfate	Oxidizer Corrosive	0.1 Mg/M3- TWA as Persulfate	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause 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:**

- Sample vials (44-mL vials with caps and septa).
- Volumetric flasks
- Class A pipettes
- Eppendorf Pipettes
- Graduated cylinder

6.1 **Instrumentation:** O-I-Analytical Carbon Analyzer (Model 1030) with corresponding auto sampler.

7.0 **Reagents and Standards:**

7.1 Carbon-free Milli-Q water used for initial calibration blanks (ICB), method blanks (MB), continuing calibration blanks (CCB), to make all stock solutions, and all dilutions that are needed.

7.2 1000ppm Stock Standard KHP purchased from two separate vendors.

7.2.1 One vendor's stock solution is used to make the calibration and laboratory control sample (LCS) and matrix spike (MS)/matrix spike duplicate (MSD). The second source vendor's stock solution is used for the initial calibration verification (ICV) and continuing calibration verification (CCV) solutions.

7.2.2 60.0ppm is used for the ICV, CCV and LCS. To make the 60.0 ppm, add 30.0 mL of 1000ppm and dilute to final volume of 500mL using metals free Milli-Q H₂O.

7.2.3 For the MS/ MSD (44mL), remove 880 μ L of sample and discard. Then add 880 μ L 1000 ppm KHP. For a 40 mL dilution, 800 μ L of 1000 mg/L KHP is added, after first removing 800 μ L of sample.

- FOR 5310C: When insufficient sample volume is provided for the MS/MSD: 40ml of sample is added to 2mL of 1000 ppm KHP and 3 drops concentrated hydrochloric acid and then diluted to 80 mL with metals free MilliQ DI H₂O in a graduated cylinder. Cover with parafilm and invert the graduated cylinder three times to homogenize. Pour into 2 Vials. This must be entered into TALS as a 2x dilution with 1 mL of spike per vial.

7.3 Inorganic Carbon (carbonate-bicarbonate) stock standard, 1000 mg/L, purchased commercially to be used for a Total Inorganic Carbon efficiency check as follows:

- One sample per matrix and per QC batch must be spiked with inorganic carbon to determine the efficiency of the inorganic carbon removal process. To perform this check, split a sample into two 40 mL portions and spike one portion with 800 μ L of 1000 ppm Inorganic Carbon standard. Alternatively, if using duplicate client provided 44 mL volumes, spike one volume with 880 μ L of 1000ppm Inorganic Carbon

standard. Analyze both samples and compare the TOC results; they should agree. If the % RPD between the spiked portion and the un-spiked portion is greater than 20%, adjust sample volume, pH, purge gas flow rate and/or purge time as necessary to obtain complete removal of inorganic carbon.

7.4 Sodium Persulfate (20%)

- To make: add 400g of $\text{Na}_2\text{S}_2\text{O}_8$ to carbon free Milli-Q water to create a final reagent volume of 2 liters. Prolonged stirring _{may} be necessary; **do not heat**. The shelf life for this solution is approximately three weeks. Solution made according to OI Operations Manual.
- This solution can also be purchased from OI Analytical

7.5 Phosphoric Acid (5%)

- To make: prepare a 5% phosphoric acid solution by adding 118.0 ml of reagent grade (85%) H_3PO_4 to carbon free Milli-Q water to create a total final volume of 2 liters. (**Caution; this reaction is exothermic**). Solution made according to OI Operations Manual.
- This solution can also be purchased from OI Analytical.

7.6 Hydrochloric Acid

- TOC methods 5310 and 9060A are preserved methods. Hydrochloric acid is used to preserve the samples. The pH of the samples must be ≤ 2 .

7.7 UHP Nitrogen tanks (carrier gas).

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
Water	Vials with caps and septa	40 mL	HCL with a pH <2 and zero headspace; Cool 0-6°C	28 Days	40 CFR 136.3 SW846 3 rd edition Standard Methods 5310
Soil	Glass wide jar	4oz	Cool 0-6°C	28 days to extract. 28 days after extraction	N/A
Waste	Variable	Variable	Cool 0-6°C	28 Days. 28 days after extraction	N/A

9.0 **Quality Control**

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

The requirements for the 5310C method are:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< 1.0 mg/L (RL)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	90-110%
Matrix Spike (MS) ¹	1 in 20 or fewer samples	Statistical Limits ³
MS Duplicate (MSD) ¹	1 in 20 or fewer samples	Statistical Limits ³
Continuing Calibration Verification (CCV)	1 in 10 or fewer samples	90-110%
Continuing Calibration Blank (CCB)	1 in 10 or fewer samples	< 1.0 mg/L (RL)

The requirements for the 9060A method are:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< 1.0 mg/L (RL)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	90-110%
Matrix Spike (MS) ¹	1 in 10 or fewer samples	Statistical Limits ³
Matrix Duplicate (DU) or MS Duplicate (MSD) ¹	1 in 10 or fewer samples	<20% RPD Statistical Limits ³
Continuing Calibration Verification (CCV)	1 in 10 or fewer samples	90-110%
Continuing Calibration Blank (CCB)	1 in 10 or fewer samples	< 1.0 mg/L (RL)

¹ The samples selected for MS/MSD and DU are random, unless specifically requested by a client.

² Analytical and QC samples (MB, LCS, MS/MSD)

³ Statistical control limits are updated annually and are updated into LIMS.

10.0 **Procedure**

10.1 **Sample Preparation**

10.1.1 It is recommended that 5 reagent blanks be analyzed at the beginning of each analytical run as an instrument clean up. This should be done as part of the daily start-up of each instrument. Check instrument calibrations to verify they have been calibrated within the last three months and that all calibration requirements have been met (see section 10.2).

10.1.2 TOC Wheel Template: Each sample wheel should only contain samples for one method (either 9060 OR 5310). The worksheet should include the appropriate amounts of samples, CCVs, CCBs, MBs, LCSs, spikes, spike duplicates, and duplicates that are required per method, see section 9.1 for method QC requirements.

10.1.3 Prepare sample wheel following the TOC Wheel Template, filling the positions with the appropriate QC/samples (see section 7 for QC preparation).

10.1.4 When preparing the samples, be sure to homogenize. Samples with historical values of less than 100mg/L should be analyzed without a dilution unless the sample matrix has apparent issues (color, visible oils,

cloudiness, etc). Check each sample's pH prior to analysis and adjust if necessary to a $\text{pH} \leq 2$ with HCL. If historical data is greater than 100mg/L then samples must be diluted accordingly and adjust the pH to ≤ 2 with HCL. Be sure to spike the samples chosen to spike (see Section 7.2).

- 10.1.5** If a dissolved method is required and status on the backlog is at wait, samples must be filtered with a 0.45 μm filter and batched off in TALS with the appropriate filtration batch method.

10.2 Instrument Prep/Analysis:

- 10.2.1** If instrument is on stand by, prime the lines. The lines can be primed by pressing *maintenance* on the main screen, then *syringe pump*, then *prime lines*. Make sure the option in the drop down menu is selected to prime ALL lines.
- 10.2.2** If on gas saver mode, the instrument must be initialized prior to priming the lines. Initialize the instrument by pressing *configuration*, *system*, *automated start up/ shutdown*, and *wake up now*.
- 10.2.3** Check that reagents, water, and waste containers are full.
- 10.2.4** To enter the wheel sample order, go to the TOC instrument software and on the control screen choose *Editor*. Under *Sequence*, open the master template that fits the method you are analyzing. Scan the samples into template, remembering to make sure dilutions are entered and QC types are added as suffixes. Then save the sequence as the date you are loading it. Then in *Monitor* under the *Sequence* tab, choose *Load Active Sequence* and load the sequence just built.
- 10.2.5** Place wheel onto the instrument to be ran. Be sure to have a rinse cup in the middle of the wheel. Line up the wheel as it should be placed on to the instrument.
- 10.2.6** Start the instrument by pressing the play button (consult the instrument software manual for details). Both chamber heating core temperatures must be lower than 76° C or the instrument will display an error message. If the temperature has not reached the setpoint, prime the lines again (see section 10.2.1).
- 10.2.7** Once analysis is complete and the instrument is once again in standby, prepare to transfer data to the LIMS. First, open the *Shortcut to LIMS Data* folder on the TOC instruments desktop. Then proceed by finding the file that you need to transfer, for example *TOC-P*. Right click on the file and select rename file. Rename it by the instrument letter and the date you are transferring it, for example P01282016. Copy this file and paste it into the corresponding instrument folder that is located on the TOC instrument desktop. (TOC-A = TOC 1, TOC-E = TOC 2 and TOC-P = TOC 3). Open TOC Aurora software. In the import path, change the instrument number at the end of the command line. For example, for TOC-A, the import path should read: \\CORPTALSAPP07\480-BF-RawData\Wetchem\TOC1. For the raw data file, select the "... " box. Go to the desktop and choose your instrument and find the data file that you

just pasted in to the folder. Then hit *transfer file*. Once you receive the message that file has been transferred, close this window.

- 10.2.8** To print a hard copy of the data, open TOC Launch Pad located on the TOC desktop. Go to the *PC Applications* tab and double click on *Reporter*. Log in using *toc* as both the username and password, and then select your instrument. Once open, right click on the date at the top and choose *add samples to workspace*. Then click on the *TOC* tab on the right side of the screen. Click on the printer on the top left of the screen. Hit *print report* and this will send the data to the default wet chemistry printer. It is now time to set up the daily blank cleaning sequence from section 10.1.1.

- 10.3 Calibration:** Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented. The acceptable curve type is a linear curve.

Prior to analysis of the initial calibration, go into *edit calibration* and save a copy of the curve name that you are about to analyze (for example, *01202016-04202016*). As the calibration is running, the points will fill in the empty calibration that you just saved. You then need to go into *edit sequence* and select the calibration template. In the calibration template you will need to change the method to the calibration you just saved by double clicking on the method, selecting the method for the new calibration and by checking the box to apply to all samples. Hit Ok. To change the method to the one you just selected, hit the method box at the top of the column and that should change all line to the method that you just made. Save the sequence as the same file as the calibration.

To prepare the calibration use the table below. You will also need three water blanks.

concentration	Amount of of 1000ppm first source standard	ml of MilliQ DI water
0 ppm	0.0mL	100ml
1 ppm	100µL	99.9ml
5 ppm	500µL	99.5ml
10 ppm	1.0mL	99.0ml
50 ppm	5.0mL	95.0ml
100ppm	10.0mL	90.0ml

- Acceptance criteria for the calibration curve.
 - Correlation coefficient (R value) > 0.995.
 - RSD's for the Calibration points must be below 20%.

Step	Standards	Type	Contol Limit	Frequency
ICV	60PPM	LINEAR	90-110%	Immediately following calibration.
ICB	Milli-Q water	LINEAR	<1.0 mg/l	Immediately following calibration.

- ICV – 6mL of second source 1000ppm TOC standard diluted to 100mL with Milli-Q di water.
- ICB- Milli-Q water

11.0 Calculations

11.1 Leachable TOC samples and soil QC are calculated in TALS:

$$\frac{\text{TOC result (mg/l)}}{\text{Weight of the sample (g)}} \times \text{final volume (soil and de ionized water) (mL)}$$

Then dry weight correct this calculated result. Result is divided by Dry Weight.

$$11.2 \quad \text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$11.3 \quad \text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{Spike amount}} \times 100$$

$$11.4 \quad \text{Precision (RPD)} = \frac{\left| x_1 - x_2 \right|}{\left(\frac{x_1 + x_2}{2} \right)} \times 100$$

$$11.5 \quad \text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

$$11.6 \quad \text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. MDLs must be run annually for each instrument.

12.2 Demonstration of Capabilities

Analysts must perform their initial demonstration of capabilities (IDOCs) for each individual method:

12.2.1 This requires quadruplicate analysis of a mid-level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.5 Demonstration of capabilities must be done annually to show continuing demonstration of capability.

12.3 Training Requirements

- The supervisor has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.
- The following analyst validation information is maintained for this method in the laboratory QA files:
 - The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
 - The analyst must read and understand this SOP.
 - The analyst must read and understand the Method used as reference for this SOP.

- The analyst must complete a DOC or successfully analyze PT samples annually.
- The analyst must complete the TA Quality Assurance Training.

13.0 **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).

14.0 **Waste Management:**

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

The following waste streams are produced when this method is carried out.

-Acidic waste from the auto-analyzer must be disposed of in the "A" waste container.

15.0 **References / Cross-References**

- 15.1 Standard Methods for the Examination of Water and Wastewater, 22nd Edition SM 5310 C- 2011.
- 15.2 Method 415.1, "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Revised March 1983
- 15.3 Method 9060A, SW-846, Fourth Edition, 11/04.

16.0 **Method Modifications:**

Item	Method	Modification
1	9060A SM5310	Reagents in section 7.4 and 7.5 made according to instrument(OI analytical) operational manual

17.0 **Attachments**

- 17.1 Raw data-9060
- 17.2 Raw data-5310
- 17.3 TOC Wheel Worksheet
- 17.4 Calibration worksheet
- 17.5 Calibration raw data
- 17.6 Wet Chemistry Data Review Sheet

18.0 **Revision History**

- Revision 7, dated 23 February 2018
 - Re-formatted and re-wrote section 7.2
 - Re-formatted 7.3-7.6
 - Delete 10.1.2 (how to run backlog), adjust all of the corresponding numbers up
 - Section 10, condensed
 - Added 10.1.5
 - Added 10.2-instrument prep bringing all of the instrument instructions under one section
 - 10.2.6 updated number on the referenced section
 - 10.3 chart: fixed typo in the units
 - Update attachment 17.1
 - Added attachment 17.2
 - Update attachment 17.3
 - Update attachment 17.4
 - Added attachment 17.5
 - Updated attachment 17.6
- Revision 6, dated August 17, 2016
 - Changed Standard Method references from 5310D to 5310C
 - Updated Quality Assurance Manager, signature added
 - Added section 7.3 regarding performing a Total Inorganic Carbon efficiency check
- Revision 5, dated February 8, 2016
 - Changed Department Manager
 - Section 5.2 changed sulfuric acid to hydrochloric acid in table.
 - Section 7.2 added ms/msd instructions for when no ms/msd volume is available.
 - Section 7.2 added more instructions to the preparation of 5310 ms/msd.
 - Section 9.1. added (CCB)
 - Section 9.1 changed (MBLK) to (MB)
 - Section 9.1 changed (md) to (DU)
 - Section 10.1 Change the lay out of the section to provide step by step details on preparing sample wheels and retrieving data.
 - Section 10 added the running of backlog in TALs.
 - Section 10 added instructions as to data transfer and printing from instrument software.
 - Section 10.2 changes ICB control limit from <.5 to <1.0
 - Section 10.2 removed CCVL
 - Section 10.2 how calibration is made added table
 - Attachment 17.3 updated
 - Added attachment 17.4 – calibration documentation
 - Updated section 9.0
- Revision 4, dates 19 March 2015
 - Updated Title – Removed 9060 reference and all reference to this method.
 - Section 7.7 was changed from sulfuric acid to hydrochloric acid.
 - Added 9060_Dill and SM5310D_DOC methods.
 - Updated QA charts
 - Updated procedure

- Removed total inorganic carbon from SOP
 - Updated IDOCs section
 - Updated Department Manager
 - Changed Lab Director, signature added
- Revision 3, dated 28 August, 2013
 - Updated Method in Title with Update IV method
 - Updated QA Manager and signature
 - Added Update 4 reference to section 15.0
- Revision 2, dated 28 December, 2011
 - Updated QA manager and Department manager name and signature.
 - Added pH requirement for all samples post analysis in Section 10.0.
- Revision 1, dated 31 December 2009
 - Updated Sodium Persulfate reagent, 15% to 20%
 - Updated attachments
- Revision 0, dated 19 December, 2007
 - Integration for TestAmerica and STL operations.
 - Updated attachments
 - Added pH check and documentation to section 10.0

Attachment 17.1: 9060 raw data

OI Corporation TOC Reporter
Instrument ID: E616730030 Wet-Chemistry
User ID: toc Name: Total Organic Carbon
Title: Mr Dept: OIC-TOC

12-21-2017 05:56 |

Results - Workspace View - TOC

SP#	Sample ID	Date/Time Analysed	Rep #	Area (Counts)	Mass (ugC)	Conc (PPM)
2	Rinse	12/20/2017 5:12:54PM	1	1548	1.204	0.601
		12/20/2017 5:18:42PM	2	1587	1.227	0.614
		12/20/2017 5:26:10PM	3	1553	1.207	0.603
		12/20/2017 5:33:11PM	4	1578	1.222	0.611
			Avg	1566	1.210	0.607
			Std. Dev	19		
			%RSD	1.20		
3	CCV	12/20/2017 5:42:17PM	1	185803	113.129	56.565
		12/20/2017 5:49:05PM	2	194912	118.676	59.338
		12/20/2017 5:55:41PM	3	203108	123.666	61.833
		12/20/2017 6:02:25PM	4	200190	121.889	60.945
			Avg	196003	119.340	59.670
			Std. Dev	7599		
			%RSD	3.88		
4	CCB	12/20/2017 6:11:58PM	1	1743	1.061	0.531
		12/20/2017 6:19:02PM	2	1786	1.088	0.544
		12/20/2017 6:25:19PM	3	1493	0.909	0.455
		12/20/2017 6:32:17PM	4	1655	1.007	0.504
			Avg	1669	1.020	0.509
			Std. Dev	130		
			%RSD	7.78		
5	MB	12/20/2017 6:41:42PM	1	1662	1.012	0.505
		12/20/2017 6:48:24PM	2	1639	0.998	0.499
		12/20/2017 6:54:49PM	3	1462	0.890	0.445
		12/20/2017 7:01:54PM	4	1555	0.947	0.473
			Avg	1579	0.960	0.480
			Std. Dev	91		
			%RSD	5.75		
6	LCS	12/20/2017 7:11:14PM	1	194189	118.235	59.118
		12/20/2017 7:18:00PM	2	198404	120.802	60.401
		12/20/2017 7:24:34PM	3	201091	122.438	61.219
		12/20/2017 7:31:25PM	4	203757	124.061	62.031
			Avg	199360	121.380	60.692
			Std. Dev	4082		
			%RSD	2.05		
7	128753-a-3^2 20mL → 40mL	12/20/2017 7:40:58PM	1	223160	136.136	68.068
		12/20/2017 7:47:47PM	2	230560	140.642	70.320
		12/20/2017 7:54:20PM	3	227907	139.027	69.513
		12/20/2017 8:01:17PM	4	225625	137.637	68.818

Results - Workspace View - TOC

Page 1 of 10

Denotes First F

Attachment 17.2: 5310 raw data

OI Corporation TOC Reporter
Instrument ID: P410730479 Wet-Chemistry

User ID: toc Name: Total Organic Carbon
Title: Mr Dept: OIC-TOC

01-11-2018 07:31,

Results - Workspace View - TOC

SP#	Sample ID	Date/Time Analysed	Rep #	Area (Counts)	Mass (ugC)	Conc (PPM)
2	Rinse	1/10/2018 9:25:01AM	1	2766	0.792	0.396
		1/10/2018 9:30:07AM	2	2483	0.648	0.324
			Avg	2624	0.720	0.360
			Std. Dev	200		
			%RSD	7.63		
3	CCV	1/10/2018 9:39:28AM	1	245914	124.759	62.380
		1/10/2018 9:45:19AM	2	257888	130.834	65.417
			Avg	251901	127.800	63.899
			Std. Dev	8467		
			%RSD	3.36		
4	CCB	1/10/2018 9:54:39AM	1	2592	1.315	0.657
		1/10/2018 10:00:46AM	2	2686	1.363	0.681
			Avg	2639	1.340	0.669
			Std. Dev	67		
			%RSD	2.53		
5	MB	1/10/2018 10:09:48AM	1	2260	1.147	0.573
		1/10/2018 10:15:44AM	2	2383	1.209	0.605
			Avg	2322	1.180	0.589
			Std. Dev	87		
			%RSD	3.75		
6	LCS	1/10/2018 10:25:11AM	1	248974	126.312	63.156
		1/10/2018 10:31:01AM	2	256736	130.250	65.125
			Avg	252855	128.280	64.141
			Std. Dev	5489		
			%RSD	2.17		
7	480-129857-B-1	1/10/2018 10:40:23AM	1	9349	4.132	2.066
		1/10/2018 10:46:23AM	2	9419	4.167	2.084
			Avg	9384	4.150	2.075
			Std. Dev	49		
			%RSD	0.52		
8	480-129857-B-2	1/10/2018 10:55:49AM	1	10348	4.639	2.319
		1/10/2018 11:01:37AM	2	10386	4.658	2.329
			Avg	10367	4.650	2.324
			Std. Dev	27		
			%RSD	0.26		
9	480-129856-B-1	1/10/2018 11:10:38AM	1	1232	0.014	0.007
		1/10/2018 11:16:16AM	2	1086	0.000	0.000

Attachment 17.3: TOC Wheel WorksheetBATCH # _____
Instrument: _____SRF # _____
File Name: _____

TOC wheel Worksheet									
	Sample ID	Dil.	Rep.	pH		Sample ID	Dil	Rep.	pH
1	Rinse			45					
2	CCV			46					
3	CCB			47					
4	MB			48					
5	LCS			49					
6				50	CCV				
7				51	CCB				
8				52	MB				
9				53	LCS				
10				54					
11				55					
12				56					
13				57					
14	CCV			58					
15	CCB			59					
16				60					
17				61					
18				62	CCV				
19				63	CCB				
20				64					
21				65					
22				66					
23				67					
24				68					
25				69					
26	CCV			70					
27	CCB			71					
28	MB			72					
29	LCS			73					
30				74	CCV				
31				75	CCB				
32				76	MB				
33				77	LCS				
34				78					
35				79					
36				80					
37				81					
38	CCV			82					
39	CCB			83					
40				84					
41				85					
42				86	CCV				
43				87	CCB				
44									

Date- _____
Analyst- _____

All PH <2 _____

Solutions: _____

BF-WC-TOCWW-016
Rev.0 3/18/2015

Attachment 17.4: Calibration worksheet

BATCH # _____
Instrument: _____

SRF # _____
File Name: _____

TOC Calibration wheel Worksheet			
	Sample ID	Dil.	Rep.
1	Rinse	N/A	
2	Blank	N/A	
3	1.0 ppm	N/A	
4	5.0 ppm	N/A	
5	10 ppm	N/A	
6	50 ppm	N/A	
7	100 ppm	N/A	
8	ICV	N/A	
9	ICB	N/A	

Method Name: _____

Solutions:

ICV (60ppm) : _____

RICCA / AQUA (1000PPM) : _____

CPI CAL (1000ppm) : _____

Phosphoric acid solution: _____

Sodium per sulfate solution: _____

Date- _____
Analyst- _____

BF-WC-TOCCAL-017
Rev. 0 6/12/2015
TOC Calibration Worksheet

Attachment 17.5: Calibration raw data

OI Corporation TOC Reporter
Instrument ID: E616730030 Wet - Chemistry

12-20-2017 12:36

User ID: toc	Name: Total Organic Carbon
Title: Mr	Dept: OIC-TOC

Calibration - Quick View -TOC

Revision: 1-12202017-03202018 - Dec 20, 2017; 08-32-32 AM
Modified By: toc
Date Created: 2017/12/20; 08:34 AM
Last Modified: 2017/12/20; 08:34 AM
Last Calibrated: 2017/12/20; 11:32 AM

RF(ugC/k-cnt): 0.6089
R2: 0.9997
Reagent Blank(cts): 660
Offset Area(cts): -429
Offset Mass(ugC): 0.26

Std #	Conc (PPM)	Volume (mL)	# Reps	Area	Std. Dev	%RSD	Date Analy
RW	0.000	2.000	2	1,873	227	12.14	2017-12-20;
1	1.000	2.000	2	4,684	211	4.51	2017-12-20;
2	5.000	2.000	2	15,487	301	1.94	2017-12-20;
3	10.000	2.000	2	30,260	66	0.22	2017-12-20;
4	50.000	2.000	2	160,437	693	0.43	2017-12-20;
5	100.000	2.000	2	329,959	579	0.18	2017-12-20;

Attachment 17.6: Wet Chemistry Data Review Sheet

BF-WC-DRC-003
Rev. 2 07/27/2016



Data Review Coversheet—Wet Chemistry Dept

Analysis: _____

Method: _____

Date of Analytical Run: _____

Analyst/Primary Reviewer: _____

Date of Primary Review: _____

Secondary Reviewer: _____

Date of Secondary Review: _____

Prep Batch Number					
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Analytical Batch Number					
--------------------------------	--	--	--	--	--

Criteria for QC	1 st Level	2 nd Level	n/a	Notes/Comments
Does the calibration meet method requirements? Low point at or below RL, minimum number of calibration points met per SOP, $r > \text{or} = 0.995$, pH meter slope 90-110% (if applicable)				
Was Data <div style="text-align: right;"> Imported _____ Manually Entered _____ Balance Interface Used _____ </div>				
Were the ICV, CCV and LCS within acceptable limits for QC recovery?				
Were the ICB, CCB and MB all <RL?				
Was there a CCV/CCB combination run after every 10 samples or less?				
Was there an LCS run with every batch of 20 samples or less?				
Was there a DU, MS or MSD run with every batch of 20 samples or less?				
Were all MS/MSD results within acceptable limits for QC recovery?				
Were all DU or MSD RPDs within acceptable limits for QC recovery?				
Were the raw data points for samples within the working curve range, or if not were the samples diluted to bring them within this range?				
Are dilution factors all present and correct?				
Do all entries match raw data?				
Were there any holding time violations in this batch?				NOTE! The PM and QA Manager must be notified by email <i>immediately</i> of any holding time violations!!
Are all errors crossed out with single line, initialed and dated?				
Were any NCMs needed in the batch?				NCM #s:
Data scanned in?				