Laboratory Manager Approval: Kristy E. Hrehor / 08/19/2021

QA Manager Approval: Jeffrey Moore / 08/19/2021

SM 5310C - Total Organic Carbon in Drinking Water

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1 Scope and Application

1.1 SM 5310C is the persulfate-ultraviolet oxidation method for analyzing total organic carbon. The Tekmar Fusion TOC analyzer is designed to determine the carbon content in water and other solutions. Using safe and proven UV promoted persulfate oxidation of carbonaceous material to carbon dioxide (CO2) followed by NDIR detection of the CO2 product, the Fusion is sensitive from 0.2ppbC - 4,000ppmC. To determine TOC by the NPOC method, the Fusion uses a syringe driver and 7-port valve to accurately transfer samples and reagents to the reactor. It then uses carrier gas to transfer the reaction product (CO2) from the sample either to vent or to the NDIR detector in the following sequence: 1. Removal and venting of IC and POC by acidification and sparging in the IC sparger. 2. Following IC removal, an aliquot of the sparged sample is transferred to the UV reactor and persulfate reagent is added to oxidize the organic carbon, based on the following chemical reactions: a. Free radical oxidants formed b. Excitation of organics c. Oxidation of organics. The oxidation products in Step 2 are swept into the CO2 selective NDIR detector. The exit valve from the NDIR is closed to allow the detector to become pressurized. Once the gases in the detector have reached equilibrium, the concentration of the CO2 is analyzed. The output signal is proportional to the concentration of CO2 in the carrier gas, from the oxidation of the sample.

1.2 Restricted Procedure

This procedure is restricted to use by an analyst experienced in the operation of a Tekmar Fusion TOC analyzer. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Plan for Quality Control Definitions. (see SOP reference 13.3)

2.2 Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3 Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4 Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5 Continuing Calibration Check (CCC) or Continuing Calibration Verification (CCV) – A standard used to verify that the response of the instrument has not changed since initial calibration. The CCC is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.6 Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method Blank (MBLK), MDLB (MDL Blank) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.7 MDLS (Method Detection Limit Spike) – MDLB spiked with analytes at the lowest calibration level to be used for the determination of MDL.

3 Interferences

3.1 Excessive acidification of sample, producing a reduction in pH of the persulfate solution to 1 or less, can result in sluggish and incomplete oxidation of organic carbon.

3.2 The intensity of the ultraviolet light reaching the sample matrix may be reduced by highly turbid samples or with aging of the ultraviolet source. Resulting in sluggish or incomplete oxidation.

3.3 Large organic particles or very large or complex organic molecules such as tannins, lignins, and humic acid may be oxidized slowly because persulfate oxidation is rate-limited.

3.4 Persulfate oxidation of organic molecules is slowed in samples containing significant concentrations of chloride by the preferential oxidation of chloride; at a concentration of 0.1% chloride, oxidation of organic matter may be inhibited completely.

4 Safety

4.1 Refer to the EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision. (SOP reference 13.8)

5 Apparatus and Equipment

5.1 Sample Container: 125 ml amber glass bottle with TFE-lined cap.

5.2 Auto-sampler vials, glass, 40 ml

5.3 Glassware -- Class “A” volumetric flasks, graduated cylinders, and pipettes.

5.4 Tekmar Dohrmann Fusion TOC Analyzer

5.5 Auto-Sampler

5.6 Ultra-High Purity Compressed Nitrogen Tank

5.7 Air displacement pipettes of various volumes, auto-pipettors, pipette tips in various sizes. Air displacement pipettes and auto-pipettors may also be described as mechanical pipettes.

5.7.1 Each day of use, the volume dispensed by each mechanical pipette must be verified for the specific volume for which the pipette is being used.
5.7.2 Mechanical pipette volumes are verified by measuring the weight of a volume of water dispensed by the unit. At room temperature, 1 ml of water is equal to 1g. Mechanical pipettes must be verified to be within ± 2.5 percent of the nominal volume.
5.7.3 Mechanical pipettes must be professionally calibrated every 6 months.
5.7.4 Auto-pipettors may be verified by measuring the volume dispensed with a Class "A" graduated cylinder. The volume dispensed must be within ± 2.5 percent of the nominal volume.
5.8 Auto-sampler racks, 35-position
5.9 HDPE bottles, various sizes, for storage of standards
5.10 Glass bottles, dark amber in color, for storage of reagents and standards.
5.11 Magnetic stir plate
5.12 Magnetic stir bars of various sizes
5.13 Disposable pipette tips, 101-1000 µl - Fisher PN# 02-707-507 or equivalent.
5.14 Disposable transfer pipettes:
5.14.1 Plastic - VWR® Disposable Transfer Pipets PN# 16001-190 or Fisherbrand™ Standard Disposable Transfer Pipettes PN# 13-711-7M

6 Reagents

6.1 Ultra-High Purity Compressed Nitrogen
6.2 Reagent Water:
6.2.1 Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest (Deionized, HPLC, Milli-Q water, or equivalent. Milli-Q water has a resistivity of 18.2[MΩ.cm] @ 25oC and a TOC of 50 ug/L or less).
6.3 10% Sulfuric Acid Solution:
6.3.1 Purchased from VWR, Part # BDH3358-4 or equivalent.
6.3.2 This solution is used for preservation of standards and blanks.
6.3.3 This purchased chemical is stable until expiration date on bottle or within 2 years of opening date, whichever is sooner.
6.3.4 Store at room temperature.
6.4 Phosphoric acid (21%):
6.4.1 Measure 185 ml of 85% phosphoric acid into a rinsed volumetric 2000 ml flask. Add 940 ml of reagent water. Mix well.
6.4.2 Prepare fresh every 3 weeks.
6.4.3 Store at room temperature.
6.5 10% Persulfate and 5% Phosphoric Acid Reagent Mixture:
6.5.1 Measure 125 g of 98+% sodium persulfate into a rinsed volumetric 2000 ml flask. Add 45 ml of 85% phosphoric acid. Add 1065 ml of reagent water. Insert a clean stir bar into flask. Place solution on stir plate. Wrap flask with aluminum foil to block out light. Place para-film loosely over top of flask allowing room for gas to escape. Make sure flask is centered on stir plate. Turn on stir plate and stir at low speed overnight.
6.5.2 Store reagent is an amber glass bottle and keep covered with aluminum foil.
6.5.3 Prepare fresh every 3 weeks.
6.5.4 Store at room temperature.
6.6 Primary Source (PS) Stock standard, 1000 mg organic carbon/L:

6.6.1 2.1254 grams of dried ACS grade KHP is dissolved and brought to volume in a 1000 ml volumetric flask with reagent water.
6.6.2 Keep under refrigeration.
6.6.3 Prepare every 6 months.
6.7 PS Intermediate Standard, 100 mg organic carbon/L:
6.7.1 Pipette 10 ml of 1000 mg/l of stock standard (100 mg organic carbon/L) into a 100 ml volumetric flask and bring to volume with reagent water.
6.7.2 After the standard is brought to volume with reagent water, add 1 ml of 10% sulfuric acid solution for preservation.
6.7.3 Prepare fresh every 3 months or when a new stock is made.
6.7.4 Keep under refrigeration.
6.8 Calibration standards:
6.8.1 Using the Primary Source (PS) Stock standard, 1000 mg organic carbon/L, prepare calibration standards at six concentrations in reagent water.
6.8.2 The calibration standards range from 0.50 to 9.00 mg of organic carbon/L.
6.8.3 After the standards are brought to volume with reagent water, 10 ml of 10% sulfuric acid per 1000ml of standard is added for preservation.
6.8.4 Prepare every three months or when new stock standard is made.
6.8.5 Keep standards under refrigeration.

Calibration Standards

ml of Stock Standard	Final volume (ml)	Concentration (mg/L)
0.5	1000	0.5
1	1000	1.0
2	1000	2.0
5	1000	5.0
7	1000	7.0
9	1000	9.0

6.9 0.00 mg/L Standard, ICB, CCB, MBLK, MDLB and Dilution water):
To prepare an ICB/CCB, Pipette 10ml of 10% H2SO4 into a 1L volumetric flask that already contains 1L of reagent water. This solution is stable for 28 days. The volume of the reagent may be altered as long as the final concentration remains the same.
6.9.1 The ICB/CCB/MBLK/MDLB must be poured into a sample collection bottle before it is poured into the appropriate vials. Record lot # of bottle used.
6.10 Continuing Calibration Check (CCC) 4.00 mg/L Standard:
To prepare the CCC, pipette 4 ml of the (PS) 1000 mg/L Stock standard Solution into a 1L volumetric flask. Once the standard is diluted to volume with reagent water, preserve the solution with 10 ml of 10% Sulfuric Acid Solution.
6.10.1 This solution is stable for 3 months.
6.11 Method Detection Limit Spike (MDLS) 0.50 mg/L Standard
To prepare the MDLS, pipette 0.5 ml of the (PS) 1000 mg/L Stock standard Solution into a 1L volumetric flask. Once the standard is diluted to volume with reagent water, preserve the solution with 10 ml of 10% Sulfuric Acid Solution.

6.11.1 This solution is stable for 3 months.
6.11.2 The (MDLS) 0.50 mg/L standard must be poured into a sample collection bottle before it is poured into the appropriate vial. Record lot # of bottle used.
6.12 ICV Stock Standard Solution, 1000 mg organic carbon/L or Second Source (SS)
6.12.1 The ICV stock standard is used as a second source standard.
6.12.2 This stock standard must be from a different source than the stock standard used to make the calibration standards.
6.12.3 The prepared standard is stable until expiration date on bottle or within 6 months of opening date, whichever is sooner.
6.12.4 Keep under refrigeration.
6.13 ICV Solution 5.00 mg organic carbon/L(SS)
6.13.1 A 5 ml aliquot of the ICV Stock Standard Solution (1000 mg organic carbon/L), is pipetted into a 1L volumetric flask and diluted to volume with reagent water. Once the ICV is diluted to volume with reagent water, preserve the solution using 10 ml of 10% Sulfuric Acid Solution.
6.13.2 The ICV solution must be prepared fresh every 3 months.
6.13.3 Keep under refrigeration.
6.14 Volumes and amounts of reagents, chemicals and standards may be altered if final concentrations remain the same. Sample volumes and injection amounts may be altered if required detection limits can be met and sample/reagent ratios remain the same.

7 Sample Collection

7.1 Samples are collected in 125 amber glass bottles.
7.2 1.25 ml of 10% sulfuric acid is added to the DWTOC bottles in the receiving lab, prior to sample collection in the field by the sample collectors.
7.3 Sample preservation is checked in the receiving lab at the time of receipt.
7.3.1 Sample pH is checked with disposable transfer pipettes. A clean disposable pipette is used to draw up a few drops of the sample. The drops are then placed on appropriate narrow range pH paper.
7.3.2 Never dip the test strip into the sample.
7.3.3 Sample pH must be <2.
7.3.4 If the sample pH is not <2, the collector is notified, and sample must be recollected.
7.4 Samples are cooled and stored at 0-6° C (not frozen).
7.4.1 If the sample temperature is outside the range of 0-6° C (not frozen), the collector is notified and the sample (both raw and filtered) must be recollected.
7.5 Sample holding time is 28 days.

8 Calibration

8.1 Calibration Standards
The calibration curve consists of calibration standards at the following concentrations: 0.00 mg of organic carbon/L, 0.50 mg of organic carbon/L, 1.00 mg of organic carbon/L, 2.00 mg of organic carbon/L, 5.00 mg of organic carbon/L, 7.00 mg of organic carbon/L, and 9.00 mg of organic carbon/L.
8.2 Calibration Curve
The Teledyne Tekmar Fusion TOC analyzer are calibrated every six months or as needed when the ICV does not meet acceptance criteria of +/- 10% of true

value or when new stock standard is used. Seven standards are used to calibrate the instrument and the calibration is stored in the computer. Minimum acceptable correlation coefficient is 0.995 using a linear regression. Dilute all samples with a response greater than 9.0 mg/L.

8.3 Calibration Verification

An Initial Calibration Verification standard (ICV), a Continuing Calibration Check (CCC) and an Initial Calibration Blank (ICB) must be analyzed immediately after the calibration standards.

8.3.1 The initial calibration verification standard must be prepared with a stock from a different source than the standards used in the calibration of the instrument.

8.3.1.1 The ICV value must be within 10% of its true value.

8.3.1.2 The %Drift (see calculation 11.1.) of the ICV from the true value must be within ±10%. Repeat once if it fails. If it fails the second attempt, determine the source of the problem, correct and recalibrate.

8.3.2 The ICB, CCB and MBLK values must be less than the method RL or the run will have to be repeated.

8.3.3 A CCC and a Continuing Calibration Blank (CCB) must be analyzed every 10 samples and at the end of the sample run and must meet the same criteria as the ICV and ICB respectively.

8.3.3.1 The CCC may be from the same source as the calibration standards.

8.3.3.2 If the CCC or CCB do not meet acceptance criteria, then all samples affected by the out of control CCC or CCB are to be rerun.

8.3.3.3 The CCC must be within 10% of expected value.

8.3.4 A low level calibration check standard at a concentration of 0.50 mg/L must be analyzed once per analytical run. Recovery of the standard must be ±50%.

8.3.5 A MDLS (low level spike) at the concentration of 0.50 mg/L must be analyzed with each batch to perform ongoing MDL study. All batch QC must be valid to report this result.

8.3.6 A MDLB (MDLB) must be analyzed once per analytical batch to perform an ongoing MDL study. All batch QC must be valid to report this result.

9 Quality Control

9.1 Refer to Table 14.1 for Reporting Limits (RL's), Table 14.2 for Quality Control Acceptance Criteria. Table 14.3 for Quality Control Procedures associated with this method and the Standard Operating Procedures for Control Charts and Control Limits.

9.1.1 The default control limits from SM5310C are 90 – 110% recovery for TOC for LCS recoveries. The EPD Laboratory applies LCS recovery limits to LCSDs. Note, unless specified by method, the EPD Laboratory does not validate batch quality based on LCSD recoveries.

9.1.2 By default, the EPD Laboratory sets LCS/LCSD precision control limits for this method to be 0 – 10% RPD.

9.1.3 LCS/LCSD recovery and precision limits are static by EPA/Method/EPD Lab default.

9.1.4 10% of all routine samples must be spiked. 5% of all samples must be analyzed in duplicate. This criterion will be satisfied if an MSD is analyzed with each MS resulting in 10% of samples being analyzed in duplicate. See Section 9.2 modification below. The EPD Laboratory requires recovery control limits of 90 – 110% for matrix spikes. The EPD Laboratory applies MS recovery limits to

MSDs.

9.1.5 By default, the EPD Laboratory sets default sample precision control limits to be 0 – 10% RPD.

9.1.6 MS/MSD recovery and precision limits are adjusted through the use of control charts.

9.1.7 See Administrative SOP for Control Charting and Control Limits for further details.

9.2 Batch samples in groups of 20. For each batch, analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for a minimum of 10% of routine samples.

9.2.1 For batches of 1 to 10 routine samples, one MS/MSD pair must be spiked. For batches of 11 to 20 routine samples, two MS/MSD pairs must be spiked using different samples for each pair**.**

9.3 MDL (method detection limit) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

9.3. 1 The actual MDL varies depending on instrument and matrix.

9.3. 2 The MDL must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.3.3 The Method Detection Limit Study for all analytes must be performed initially on a new instrument and performed after major instrument repairs or changes to procedures. There are two ways to perform the MDL. The first is with 7 samples and 7 blanks over 3 separate days. The second preferred way the MDL is run as a continuous format.

9.3.4 The 7 MDL samples study is performed by preparing 7 spiked vials, MDLSpike, spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDLBlank. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there may or may not be a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blanks.

9.3.5 A continuous formal MDL study is performed where one vial is spiked as an MDLSpike, at the lowest point of the calibration curve and analyzed with every batch of samples along with the method blank vial as an MDLBlank.

9.3.6 The results of the MDLBlank will be entered into Labworks using the Method Blank test code, B_TOCDW. The MDLSpike result will be entered using the MLTOCDW. The MDL Spiked Amount will be entered into the test code MATOCDW. The instrument used for the MDL and Blank analysis will be selected using the test code INSTR-TOCDW.

9.3.7 MDL study must be performed every six months and before the MDL for the instrument expires.

9.3.8 MDL data is pulled from a two year period.

10 Procedure

10.1 Procedure for the Teledyne Tekmar Fusion TOC Analyzers-TOC 08 and TOC10

10.1.1 Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.

10.1.2 Change the reagent water and make sure there is plenty of reagent remaining in the reagent bottles and that they are not expired.

10.1.3 Check the nitrogen gas tank and make sure there is at least 500 psi to do a run. The second regulator of the tank should be approximately at 90 psi (normal reading between 65 – 120 psi). The Nitrogen gas tank should always remain open so that the instrument can remain on standby.

10.1.4 Turn on the instrument (Fusion) and computer. After the instrument has been ON for at least 15 seconds, start communicating with instrument by clicking *TOC Teklink* icon on the desktop of the computer.

10.1.5 Login to the TOC *Teklink* with our Username and Password.

10.1.6 Select the instrument (Tekmar/Fusion) in the list and click the *Connect* button. The Main screen of the *TOC TekLink* will appear. In the lower right corner on the Status bar, it should say that the instrument is connected.

10.1.7 Perform maintenance checks as required and record in maintenance log.

10.1.7.1 Record Detector Abs, Tank pressure, and regulator pressure.

10.1.7.2 Check Lamp and Mist Trap decolorization.

10.1.7.3 Check Cu/Sn color.

10.1.8 In order to create a new schedule, access the *Schedule Editor Screen*. Click *Open/Schedule*, select "*Daily Blank Tray*", click OK.

10.1.9 Type in new schedule. Add rows by clicking down arrow.

10.1.10 Select the 1st position for the auto-sampler.

10.1.11 Select the sample type *"Sample"*.

10.1.12 Type in the sample ID in the name column.

10.1.13 Select the processing method "TOC Drinking Water", click OK.

10.1.14 Select the number of replicas "1" in the Reps column.

10.1.15 Before saving a schedule, make sure that *Use* column has check marks and *State* column has "Ready" in the sample line.

10.1.16 When all the samples have been entered, click on File from the main menu. Type in "T" and then the date. This will be the schedule name. Click on OK to save a new schedule in a Current Schedules folder.

10.1.17 When everything is in order, click the Ready button on the toolbar. Make sure the Status Light window at the bottom–front of the Fusion is blue. A blue Status Light indicates that the instrument detects no problems and has successfully entered the Ready mode. Note: if the instrument sits idle for 20 minutes, it will automatically switch to Standby mode. To prevent it click Hold button on the toolbar.

10.1.18 Allow the UV Lamp to warm up for at least 5 minutes.

10.1.19 Check the Detector Baseline and make sure it is between 0 – 5 Abs.

10.1.20 While the instrument is equilibrating, load the sample tray with the TOC samples that need to be analyzed. Note: The tray should have a cleaning method and blanks loaded before samples to clean instrument and allow instrument to warm up before blanking. Do not remove the sample tray from the top of the

instrument, use the Carrousel button on the toolbar to turn around the tray and load up the TOC samples.

10.1.21 LCS's and CCC's should be analyzed at 4.00 mg/l concentration.

10.1.21.1 Prepare the LCS and LCSD by pipetting 1 ml of the PS Intermediate Standard, 100 mg organic carbon/L into a 25ml volumetric flask and bring to volume with dilution water that was stored in a sample collection bottle. Record lot # of bottle used. (See Section 6.9 in SOP 3-038).

10.1.21.2 Prepare the CCC by pipetting 4 ml of the Primary Source (PS) Stock standard, 1000 mg organic carbon/L into a 1 L volumetric flask and bring to volume with reagent water. After the CCC is brought to volume with reagent water, add 10 ml of 10% sulfuric acid for preservation.

10.1.22 The matrix spike should be prepared at the 4.0 mg/l concentration. To prepare the matrix spike and matrix spike duplicate, pipette 1 ml of the PS Intermediate Standard, 100 mg organic carbon/L into a 25ml volumetric flask and bring to volume with sample chosen as the spike.

10.1.22.1 When choosing samples for matrix spikes, alternate between raw samples and filtered samples.

10.1.23 When everything is ready for analysis, click the *Start* button on the toolbar. The *Status Light* window will now turn green. A green *Status Light* indicates that the active schedule is being run and no problems are detected.

10.1.24 When the run is finished, the instrument goes to standby automatically and a pop-up *Instrument Status* window appears with the message: *"Reached the end of the schedule"*.

10.1.25 Click OK on the message icon. To print the report, click on Open from the toolbar and then click on Report. Select the report with the given name. It should be the latest report from the list.

11 Calculations

11.1 The linearized signal which is proportional to the instantaneous concentration of CO_2 is integrated and referred to stored calibration data and the carbon concentration in the sample is calculated to display carbon concentration in parts-per-million (ppm).

11.2 Mean ($\overline{X}$):

$$\overline{X} = \frac{X_1 + X_2 + \cdots X_n}{n}$$

11.2.1 Where:

$X_1 + X_2 + \cdots X_n$ = The sum of a set of values X_i, i = 1 to n

n = The number of values in the set

11.3 Standard Deviation (n – 1) (σ_{n-1}):

$$\sigma_{n-1} = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \bar{X})^2}{n-1}}$$

11.3.1 Where:

$\bar{X}$ = Mean of the values
X_i = Individual values 1 through i
n = Number of values

11.4 Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\sigma_{n-1}}{\bar{X}} * 100$$

11.4.1 Where:
σ_{n-1} = Sample Standard Deviation
$\bar{X}$ = Mean of the values

11.5 Relative Percent Difference (%RPD or RPD):

$$\%RPD = \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} * 100$$

11.5.1 Where:
$|X_1 - X_2|$ = Absolute difference between two values

$\frac{(X_1 + X_2)}{2}$ = Average of two values

11.6 Percent Drift, %Drift:

$$\%Drift = \frac{(Concentration_{Calculated} - Concentration_{Expected})}{Concentration_{Expected}} * 100$$

11.6.1 Where:

$Concentration_{Calculated}$ = Concentration calculated from result
$Concentration_{Expected}$ = Theoretical concentration of the standard

11.7 Extract Concentration:

11.7.1 The extract concentration is calculated relative to the calibration curve by the instrument software.

11.8 Percent Recovery:

11.8.1 LCS/LCSD:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.8.1.1 Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.8.2 MS/MSD:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}} - \text{Conc}_{\text{unspiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.8.2.1 Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{unspiked}}$ = Concentration found in unspiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.9 Calculation of Dilution Factors

$$C \times D = F$$

11.9.1 Where:

C = concentration from instrument in mg/L
D = dilution factor
F = final concentration in mg/L

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures, (SOP reference 13.6)

13 References

13.1 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 5-19, 5-20, Method 5310 C. Persulfate-Ultraviolet Oxidation Method (2000).
13.2 Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005.
13.3 EPD Laboratory Quality Assurance Plan, Revision 11, September 2018, or later.
13.4 GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, online revision or Continuing Demonstration of Capability SOP 6-002, online revision.

13.5 GA EPD Laboratory SOP-EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.

13.6 GA EPD Laboratory SOP-EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.7 GA EPD Laboratory SOP – Determination of Method Detection Limit, Method Detection Limit SOP 6-007, online revision.

13.8 GA EPD Laboratory Safety Plan – EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, Rev. 2, online revision.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1 RLs for Method SM 5310C

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
SM 5310C	Total Organic Carbon	0.50	mg/L

Table 14.2 Acceptance Criteria for Method SM 5310C

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
SM 5310C	Total Organic Carbon	90-110	10

Table 14.3 Summary of Calibration and QC Procedures for Method SM 5310C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SM 5310C	Total Organic Carbon	Initial calibration for all analytes	Calibration every six months or as needed.	Correlation coefficient ≥ 0.995 linear regression	Correct problem then repeat initial calibration	
		Second source calibration verification (ICV)	Once per initial calibration or quarterly, whichever is sooner.	TOC value must be within 10% of expected value	Correct problem then repeat initial calibration	

Table 14.3 Summary of Calibration and QC Procedures for Method SM 5310C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample, a method blank, a blind sample, and an MDL study. In addition, the analyst must prepare one standard.	Once per analyst	QC Acceptance Criteria Table, SOP 3-038 Appendix A and Initial Demonstration SOP (SOP Reference 13.4)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Once per analyst	QC Acceptance Criteria Table, SOP 3-038 Appendix A and Initial Demonstration SOP (SOP Reference 13.4)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Initial Calibration Blank (ICB)	Once per analytical run	TOC value <0.50 mg/L	Correct problem and repeat initial calibration	
		Method Blank (MBLK)	One per batch	TOC value must be < 0.50 mg/L	Correct problem then analyze method blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		Laboratory Control Sample (LCS/ LCSD)	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table, SOP 3-038 Appendix A	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Matrix Spike (MS/MSD)	10% of samples	QC Acceptance Criteria Table, SOP 3-038 Appendix A	Evaluate out of control event, reanalyze or flag data	
		Continuing Calibration Check (CCC)	After every 10 samples and at the end of the sample run	TOC concentration within 10% of expected value	Correct problem then reanalyze all samples associated with out of control CCC.	
SM 5310C	Total Organic Carbon	Continuing Calibration Blank (CCB)	After every 10 samples and at the end of the sample run	TOC value must be < RL	Correct problem then reanalyze all samples associated with out of control CCB.	
		Low Level standard (0.5 mg/L)	Once per analytical run.	TOC value must be ±50% of the expected value	Correct problem then repeat initial calibration.	

Table 14.3 Summary of Calibration and QC Procedures for Method SM 5310C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
		MDL Low level Spike (MDLS) 0.50 mg/L	Once per analytical batch	All batch QC must be valid	Correct problem then reanalyze the affected batch	
		MDL Blank (MDLB)	Once per analytical batch	All batch QC must be valid	Correct problem then reanalyze the affected batch	
		MDL study	Every six months or after major maintenance of the instrument	All Spiked MDLs must have a value greater than 0. Minimum Detection Limits established shall be < the RLs in Table 14.1	Re-do MDL Study	
		MDL analysis	Once per batch or as needed to acquire data points per SOP 6-007, online revision	All Spiked MDLs must have a value greater than 0. All other QC in the MDL blank and MDL sample (i.e. Surrogate Spike or Internal Standard, etc. if included) must meet established criteria	Correct problem and re-run the MDL sample or MDL blank once and initiate a corrective action. If the re-run fails a second time, do not use MDL data. Update corrective action, and use associated sample data	

Appendix A – Quality Assurance Criteria for Method SM5310C

Table A.1 Quality Assurance Criteria for Method SM5310C					
QC Type	Analyte	Accuracy(%R) LCL		UCL	Precision (%RPD)
LCS/LCSD	TOC(DW)	90	-	110	10
MS/MSD	TOC(DW	90*	-	110*	10
*MS/MSD Control limits are static by EPD Lab default. Control Chart data generated from 01/01/2019 -01/01/2021					

Updates to Previous Version:
Section 6
Section 9

Section 10
Table A.1
Table 14.3