Designation: D7573 – 09 (Reapproved 2017)

Standard Test Method for
Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection[1]

This standard is issued under the fixed designation D7573; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of total carbon (TC), inorganic carbon (IC), total organic carbon (TOC), dissolved organic carbon (DOC), and non-purgable organic carbon (NPOC) in water, wastewater, and seawater in the range from 0.5 mg/L to 4000 mg/L of carbon. Higher levels may be determined by sample dilution. The sample is injected onto a quartz bed heated at 680ºC. The sample converts into a gaseous phase and forced through a layer of catalyst ensuring conversion of all carbon containing compounds to CO_2. A non-dispersive infrared (NDIR) detector measures the resulting CO_2.

1.2 For TOC and DOC analysis a portion of the sample is injected to determine TC or dissolved carbon (DC). A portion of the sample is then acidified and purged to remove the IC. The purged inorganic carbon is measured as TIC, or DIC. TOC or DOC is calculated by subtracting the inorganic fraction from the total carbon:

$$\mathrm{TOC} = \mathrm{TC} - \mathrm{IC}$$

1.3 For NPOC analysis a portion of sample is acidified and purged to remove IC. The purged sample is then injected to determine NPOC.

1.4 This test method was used successfully with reagent water spiked with potassium hydrogen phthalate, sucrose, nicotinic acid, benzoquinone, sodium dodecyl benzene sulfonate, urea, acetic acid, and humic acid. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.5 This test method is applicable only to carbonaceous matter in the sample that can be introduced into the reaction zone. The syringe needle or injector opening size generally limits the maximum size of particles that can be so introduced.

1.6 In addition to laboratory analyses, this test method may be applied to stream monitoring.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)[3]
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
D4129 Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *inorganic carbon (IC), n*—carbon in the form of carbon dioxide, carbonate ion, or bicarbonate ion.

3.2.2 *total organic carbon (TOC), n*—carbon in the form of organic compounds.

3.2.3 *non-purgable organic carbon (NPOC), n*—carbon measured in a sample after acidification and sparging to remove inorganic carbon.

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[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

3.2.4 *total carbon (TC), n*—the sum of IC and TOC.

3.2.5 *dissolved organic carbon (DOC), n*—carbon determined on filtered samples.

3.2.6 *purgable organic carbon (POC), n*—carbon that purges from acidified samples, also known as volatile organic compounds (VOC).

3.2.7 *refractory material, n*—that which cannot be oxidized completely under the test method conditions.

4. Summary of Test Method

4.1 *Fundamentals*—Carbon can occur in water as an inorganic and organic compound. This test method can be used to make independent measurements of IC, NPOC, and TC, and can also determine OC by the difference of TC and IC. DOC is determined on samples that have been filtered through a 0.45-µm filter.

4.2 TOC and DOC procedures require that IC has been removed from the sample before it is analyzed for organic carbon content. The sample free of IC is injected into the TOC instrument where all carbon is converted to CO_2 and measured by the detector. Failure of the method to remove all IC prior to analysis for organic carbon will result in significant error. A diagram of suitable apparatus is given in Fig. 1.

5. Significance and Use

5.1 This test method is used for determination of the carbon content of water from a variety of natural, domestic, and industrial sources. In its most common form, this test method is used to measure organic carbon as a means of monitoring organic pollutants in industrial wastewater. These measurements are also used in monitoring waste treatment processes.

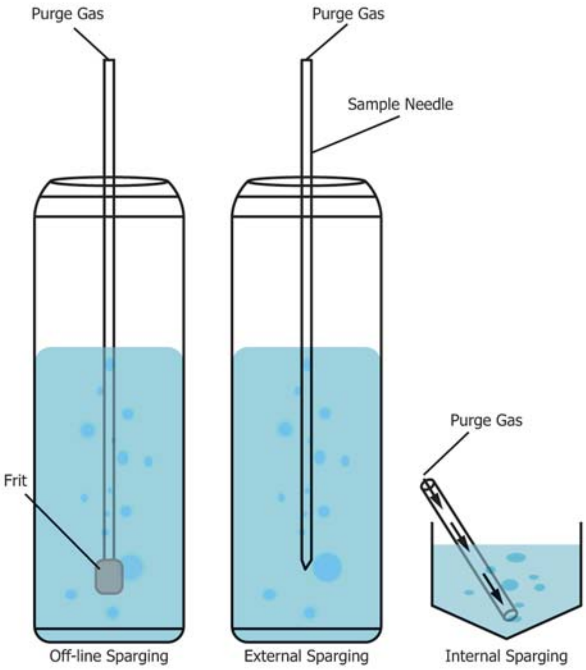

FIG. 1 TIC Removal

5.2 The relationship of TOC to other water quality parameters such as chemical oxygen demand (COD) and total oxygen demand (TOD) is described in the literature.[4]

6. Interferences and Limitations

6.1 The oxidation of dissolved carbon to CO_2 is brought about at high temperatures (680°C) in the presence of oxygen. A catalyst promotes the oxidation process and the resulting carbon dioxide is measured by a non-dispersive infrared detector (NDIR). Suspended and refractory materials are completely oxidized under these conditions.

6.2 Acid preservation can precipitate some compounds, such as humic acids, removing them from solution and causing erroneously low results.

6.3 Homogenizing or sparging of a sample, or both, may cause loss of purgable organic compounds, thus yielding a value lower than the true TOC level. (For this reason, such measurements are sometimes known as NPOC). The extent and significance of such losses must be evaluated on an individual basis. Comparison of the difference, if any, between NPOC and TOC by subtraction represents POC lost during sparging.

6.4 If POC is important then TOC must be measured by subtraction:

$$TOC = TC - TIC$$

6.5 Note that error will be introduced when the method of difference is used to derive a relatively small level from two large levels. For example, a ground water high in IC and low in TOC will give a poorer TOC value as (TC – IC) than by direct measurement as NPOC.

6.6 Samples containing high levels (>1 ppm) of surfactant may lose TOC by foaming.

6.7 Elemental carbon may not be completely combusted at 680ºC; however, it is not generally found in water samples. Elemental carbon does not form during the catalytic oxidation of water samples.

6.8 Inorganics dissolved in the sample are not volatilized into gas and remain on the catalyst or quartz shard surfaces. High amounts of solids eventually react with the quartz surfaces causing devitrification, or solidify in the catalyst bed decreasing flow rates. Limit sample volume injected to reduce the amount of soluble salts and to reduce cooling of the reaction chamber. Buildup of salts; reduction of flow rate, or large injection volumes could result in peak splitting.

6.9 Carbon in reagent water and reagent blanks can be reduced to a minimum, and consistent value, but cannot be completely eliminated. Analyzing low-level TOC (less than 1.0 mg/L) bears special consideration requiring that the same water used to set the baseline be used to prepare the calibration standards.

6.10 Atmospheric carbon dioxide absorbs into reagent water increasing its inorganic carbon content with time. The small

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[4] *Handbook for Monitoring Industrial Wastewater*, Section 5.3, U.S. Environment Protection Agency, August 1973, pp. 5–12.

levels of CO_2 absorbed into reagent water can cause considerable inaccuracies in low-level TIC analysis. For instance, a 40-milliliter vial of reagent water containing no detectable TIC was analyzed to contain 160 µg/LTIC after 1 hour of exposure to ambient air.

6.11 Trace organics in the atmosphere can be absorbed into reagent water increasing its organic carbon content with time. The small levels of organics absorbed into reagent water can cause considerable inaccuracies in low-level (<1 mg/L) TOC measurements.

7. Apparatus

NOTE 1—See also Fig. 2.

7.1 *Sampling Devices*—Manually operated or automatically operated sampling valves, or syringes are typically used with this method. Sampling devices with inside diameters as small as 0.15 mm may be used with samples containing little or no particulate matter. Larger inside dimensions, such as 0.2 mm, will be required for samples with particulate matter.

7.2 *Apparatus for Carbon Determination*—This instrument consists of reagent and sample introduction mechanism, a gas-sparged reaction vessel for TIC removal, the high temperature combustion chamber with catalyst, a gas demister or dryer and halogen trap, an optional CO_2 trap, a CO_2-specific infrared detector, a control system, and a display. Fig. 1 shows a diagram of such an arrangement.[5]

7.2.1 Reaction vessel consists of TIC removal and the combustion chamber.

7.2.1.1 *TIC Removal*—Sparging requires an inert vessel with a capacity of at least double the sample size with provision for sparging with 50 to 200 mL/min of carbon-free gas. This procedure should remove essentially all IC in 2 to 10 min, depending on design and can be at room temperature or at elevated temperatures (≤70°C) to promote CO_2 removal. Verify that heated sparging does not remove >5 % of the NPOC. Fig. 1 illustrates three different options for TIC removal.

7.2.1.2 *Combustion Chamber*—A heated catalyst contained in a quartz tube, may contain quartz wool, quarts shards, or other items to protect the catalyst from dissolved salts to extend its life.

7.2.2 *Gas Conditioning*—The gas passing from the reactor is dried, and the CO_2 produced is either trapped and later

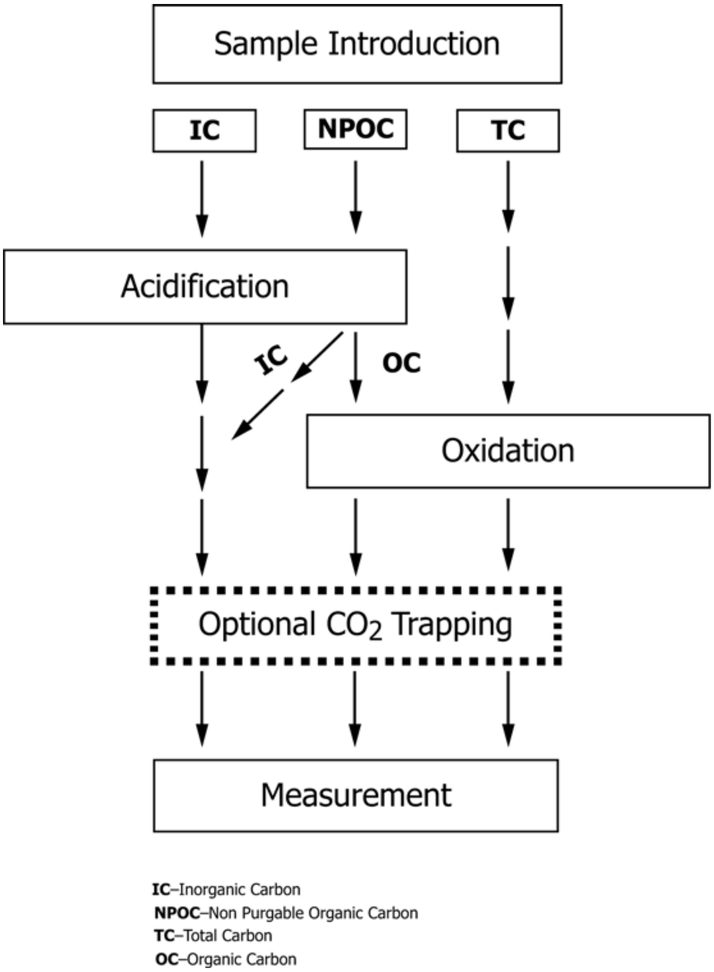

FIG. 2 Diagram of Apparatus

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[5] The sole source of supply of the apparatus known to the committee at this time is the OI Analytical Aurora 1030C and 1020. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

released to the detector, or routed directly to the detector through a halogen-removing scrubber.

7.2.3 *Detector*—The CO_2 in the gas stream is detected by a CO_2-specific NDIR detector.

7.2.4 *Detector Response*—Integrated area unless CO_2 is collected and desorbed from a CO_2 specific trap. Area integration accurately quantifies carbon content in the event of split or overlapping peaks that result from furnace cooling or variable combustion rates of different organic molecules contained in a sample.

7.2.5 *Presentation of Results*—The NDIR detector output is related to stored calibration data and then displayed as milligrams of carbon per liter.

7.3 *Low TOC Sample Containers*—Analysis of TOC below 10 ppm requires the use of sample bottles and vials certified as low TOC. This avoids variable contribution of TOC and is especially important when analyzing TOC below 1 ppm.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[6] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I or Type II. The indicated specification does not actually specify inorganic carbon or organic carbon levels but is recommended that NPOC be ≤ 0.05 mg/L. Higher levels can affect the results of this test method, especially at progressively lower levels of the carbon content in the samples to be measured. Where inorganic carbon in reagent water is significant, CO_2-free water may be prepared from reagent water by acidifying to pH 2, then sparging with fritted-glass sparger using CO_2-free gas (time will depend on volume and gas flow rate, and should be determined by test). Alternatively, if the carbon contribution of the reagent water is known accurately, its effect may be allowed for in preparation of standards and other solutions. CO_2-free water should be protected from atmospheric contamination. Glass containers are required for storage of water and standard solutions. It is recommended that the same reagent water be used in preparation of all standards and blanks per calibration.

8.3 *Acid*—Acid is used for sample preservation and TIC removal. Follow the manufacturers suggestions for acid and acid concentration used for TIC removal. Do not use nitric acid.

8.4 *Organic Carbon, Stock Calibration Standard Solution (1000 mg/L)*—Weigh 2.128 grams of anhydrous potassium hydrogen phthalate ($KHC_8H_4O_4$) previously dried for two hours at 120ºC and quantitatively transfer to a 1000-milliliter volumetric flask containing about 500 milliliters of reagent water. Stir to dissolve and add 1 milliliter of concentrated hydrochloric acid (HCl), dilute to the mark with reagent water and mix. Transfer to an amber glass reagent bottle and cap for storage. This stock solution, or dilutions of it, is used to calibrate and test performance of the carbon analyzer.

8.5 *Organic Carbon, Stock Calibration Verification Solution (1000 mg/L)*—Weigh 2.377 grams of sucrose ($C_{12}H_{22}O_{11}$) and quantitatively transfer to a 1000-milliliter volumetric flask containing about 500 milliliters of reagent water. Stir to dissolve and add 1 milliliter of concentrated hydrochloric acid (HCl), dilute to the mark with reagent water and mix. Transfer to an amber glass reagent bottle and cap for storage. This solution, or dilutions of it, is used to verify calibration accuracy and test performance of the carbon analyzer.

8.6 *Inorganic Carbon, Stock Calibration Standard Solution (1000 mg/L)*—Weigh 8.826 grams of anhydrous sodium carbonate (Na_2CO_3) previously dried at 120ºC for two hours and transfer to a 1000-milliliter volumetric flask containing about 500 milliliters of reagent water. Mix to dissolve, dilute to the mark, and mix.

8.7 *Inorganic Carbon, IC Test Solution (Alkalinity 834 mg $CaCO_3$/L)*—Dilute 10 milliliters of the inorganic carbon stock solution (Section 8.6) to 100 milliliters with reagent water. Use this solution to verify IC removal.

8.8 *Calibration Solutions*—TC, IC

8.8.1 *Organic Carbon Calibration Solutions*—At least 4 calibration concentrations and a calibration blank (CB) are used to prepare an initial calibration curve. Standards are prepared to cover the concentration of interest from the organic carbon stock calibration solution. Calibration standards are prepared in reagent water and preserved to pH <2 with concentrated HCl. Filtration of these standards for determination of dissolved organic carbon is unnecessary. These standards may be used for TC and NPOC calibrations. For NPOC the standards are sparged, just like samples, to remove the IC fraction. The calibration blank (CB) is a 0.0 mg C/L standard that contains the carbon contributed to the calibration standards by the reagent water. The CB is stored and treated the same as the calibration standards. These standards, if stored in the dark, are stable for about 30 days and may be used to recalibrate the instrument within the 30-day period. The CB stored with and prepared from the same reagent water as the calibration standards must be used as the blank when recalibrating the instrument. See Table 1 for calibration standard preparation.

8.8.2 *Inorganic Carbon Calibration Solutions*—At least 4 calibration concentrations and a CB are used to prepare an initial calibration curve. For best results, do not prepare inorganic carbon and organic carbon standards as mixed solutions. (These standards are not necessary for NPOC). See Table 1 for calibration standard preparation.

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[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Preparation of Calibration Curve Standards for Organic Carbon and Inorganic Carbon

Cal Level	Concentration of Stock (mg/L)	Volume of Stock (mL)	Final Volume of Calibration Standard (mL)	Final Concentration of Calibrations Standard (mg/L)
Calibration Blank (CB)	- - -	0.0	1000	0.0
1	1000	0.5	1000	0.5
2	1000	1.0	1000	1.0
3	1000	10.0	1000	10.0
4	1000	25.0	1000	25.0
5	1000	50.0	1000	50.0
6	1000	100	1000	100
7	1000	250	1000	250

8.9 *Gas Supply*—High-purity oxygen free of CO_2 and of organic matter is required. Use gas purity as specified by the equipment manufacturer. The use of oxygen is preferred.

9. Sampling and Sample Preservation

9.1 Collect the sample in accordance with Guide D1192 and Practices D3370.

9.2 To preserve samples for this analysis, store samples in low TOC glass at 6°C. To aid preservation, acidify the samples to a pH ≤2 with HCL, or the acid specified by the instrument manufacturer. It should be noted that acidification enhances loss of inorganic carbon. If the purgable organic fraction is important, fill the sample bottles to overflowing with a minimum of turbulence and cap them using a fluoropolymer-lined cap, without headspace. The sample must be analyzed within 28 days of collection.

9.3 DOC must be filtered in the field or in the laboratory within 48 hours of collection and prior to acidification and analysis. After filtration, the sample is acidified to a pH ≤2 and stored at 2–6ºC. The sample must be analyzed within 28 days of collection.

9.4 For monitoring of waters containing solids or immiscible liquids that are to be injected into the heated reaction zone, use a mechanical homogenizer or ultrasonic disintegrator. Filtering or screening may be necessary after homogenization to reject particle sizes that are too large for injection. Volatile organics may be lost. See 6.3.

NOTE 2—For greater accuracy and precision filter high particulate samples through quartz fiber filters and analyze the aqueous DOC and the particulate carbon separately.

9.5 For wastewater streams where carbon concentrations are greater than the desired range of instrument operation, dilute the samples as necessary.

10. Instrument Operation

10.1 Follow the manufacturer's instructions for instrument warm-up, gas flows, and liquid flows.

11. Calibration

11.1 Prior to calibration, monitor the background carbon dioxide levels for at least 30 minutes or until the background signal reaches the manufacturer's recommended level. Adjust instrument temperatures, flow rates, and reagent settings according to the manufacturer's instructions. Perform multiple injections of reagent water until three consecutive injections fall within 20 % of their mean.

11.2 A new calibration curve is generated when fresh standards are made, or when continuing calibrations verification standards (CCV) fall outside of quality control (QC) Limits. Use a CB and at least four calibration standards to span the expected concentration range of the samples to be analyzed. For example, if samples are between 25 and 250 mg/L, only calibration standards 4–7 would need to be used. The lowest calibration standard must be at the minimum reporting level (MRL). Sparge the calibration standards to measure IC or NPOC. Do not sparge to measure TC. Inject standards from low to high in triplicate to calibrate the instrument. Triplicate measurements should be within 5 % of their mean. Be careful not to extend the concentration range so high that "injection memory" causes analytical error.

11.3 The instrument generated calibration curve must have an r2 ≥ 0.993 before proceeding with analysis of samples. Ideally the r2 should be ≥0.9995. After the instrument has been calibrated, verify the calibration with an initial calibration verification standard (ICV) prepared at the midpoint of the curve. The ICV should be within 10 % of its known value.

11.4 Record the data from the calibration in an instrument log or laboratory notebook. This calibration serves as a historical record of instrument performance. Compare subsequent calibrations, recording the data each time. If the slope of a calibration changes significantly such that QC criteria cannot be met, consult the instrument manual or laboratory SOP for corrective action, which may include replacement of the combustion tube, quartz shards, catalyst, or combinations thereof.

12. Procedure

12.1 Mix or blend each sample thoroughly and carry out any necessary dilution to bring the carbon content within range of the instrument. Since some high molecular weight organic compounds may precipitate upon cooling, warm the sample room temperature prior to analysis. If acid-preserved samples contain humic acids (brownish to dark brown color) they may have precipitated humic acid on the sides of the sample bottle or formed a floc that settles to the bottom of the sample container. Treat samples suspected to contain humic acids as follows:

12.1.1 Split a well-mixed sample into two portions.

12.1.2 Keep one portion at pH ≤2 and adjust the other to pH 5–7 with 10N NaOH to increase the solubility of the humic acid.

12.1.3 Cap, and allow both portions to sit at room temperature for ½ hour.

12.1.4 Analyze TIC and TC on both splits and report the subtracted TOC values along with the pH.

12.1.4.1 If the sample is high in IC preventing accurate TOC by subtraction, purge the acid preserved fraction to remove the IC, add CO_2-free 10N NaOH to pH 5–7, and analyze TC.

12.2 If inorganic carbon is to be measured directly, inject the sample into the analyzer under appropriate conditions according to the manufacturer's instructions and as determined by analysis of the IC Test solution. All TIC should be effectively removed from solution and measured by the analyzer.

12.3 If inorganic carbon is to be removed by sparging prior to sample introduction, acidify to approximately pH 2 with acid (if not already done) and sparge with an appropriate flow of gas for the appropriate time as determined by analysis of the IC test solution. Samples with high alkali content or buffer capacity may require larger amounts of acid, or a more concentrated acid. In such cases, incorporate this dilution into the calculation. If incomplete sparging of CO_2 from IC is suspected, sparge and analyze the sample and then repeat the procedure until appropriate conditions are established. In difficult conditions, use of a heated-glass sparger (≤70°C) may help.

12.4 To measure TOC as NPOC, inject an appropriate volume of the sample into the analyzer. If external sparging is required to remove IC, inject a sparged sample for the NPOC measurement. See 6.4.

12.5 To measure TC, inject an appropriate volume of unsparged sample.

13. Calculation

13.1 Read carbon values directly from a digital display or printer, or both.

13.2 Do not use automatic outlier removal procedures supplied with most software packages.

14. Quality Assurance/Quality Control

14.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test.

14.2 *Calibration and Calibration Verification*—See Section 11.

14.3 *Analyst Performance Check*—If a laboratory has not performed the test before or if there has been a major change in the measurement system, for example, new analyst, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory capability.

14.3.1 Analyze four replicates of a standard solution prepared from a certified reference material containing a concentration of analyte similar to that expected in test samples and within the range of 0.5 to 4000 mg/L. Each replicate must be taken through the complete analytical test method including any sample preservation and pretreatment steps.

14.3.2 Calculate the mean and standard deviation of these values and compare to the acceptable ranges of precision and bias that may be calculated by the user using the precision and bias relationships listed in Section 15. This study should be repeated until the single operator precision and the mean recovery are within acceptable limits. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

14.4 *Laboratory Control Sample (LCS)*—To ensure that the test method is in control, analyze an LCS at the beginning and ending of a sequence of samples. If large numbers of samples are analyzed in a single day, analyze the LCS after every 20 samples. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The value obtained for the LCS should be within 15 % of the true value. If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

14.5 *Inorganic Carbon Test Solution (IC Test Solution)*—To ensure that inorganic carbon has been completely removed from solution prior to measurement of NPOC, or to ensure accurate measurement of IC, analyze the IC test solution for NPOC in at least four portions at the same volume as samples to be analyzed. The NPOC result must be less than the required MRL. TIC and TOC matrix spikes may also be used to verify complete TIC removal in selected matrices.

14.6 *Reagent Water Blank*—Each time new standards or control samples are prepared the reagent water must be analyzed also to account for the carbon content of the reagent water. Follow the manufacturer's instructions on handling blanks used for QC analysis.

14.7 *Matrix Spike (MS)*—To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each set of samples being analyzed by spiking an aliquot of the sample with a known concentration of analyte and taking it through the complete analytical method.

14.7.1 The spike concentration plus the background concentration of the analyte must not exceed the upper limit of the method. The spike must produce a concentration in the spiked sample 2 to 5 times the background concentration, or 10 to 50 times the detection limit of the test method, whichever is greater.

14.7.2 Calculate the percent recovery of the spike (P) using the following formula:

TABLE 2 MDL Calculation

	0.519
	0.484
	0.505
	0.502
	0.494
	0.443
	0.542
STDDEV	0.030762
MDL	0.096592
ML	0.307162

TABLE 3 Percent Recovery From Various Compounds

Compound	10 ppm Carbon	1 ppm Carbon
Potassium Hydrogen Phthalate	100 % NPOC	101 % NPOC
Sucrose	98 % NPOC	Not tested
Benzoquinone	101 % NPOC (RSD = 0.73 %)	Not tested
Benzoic Acid	101 % NPOC (RSD = 1.85 %)	Not tested
Humic Acid	79 % NPOC / 96 % TC	Not tested
Oxalic Acid	108 % NPOC / 99 % TC	Not tested
Nicotinic Acid	97 % NPOC	98 % NPOC
Sodium Dodecyl Benzene Sulfonate	62 % NPOC / 98 % TC	Not tested
Acetic Acid	Not tested	97 % NPOC
Urea	Not tested	100 % NPOC
Cellulose (100 ppm)	70–103 % NPOC	Not Tested
Potassium Hydrogen Phthalate (5 ppm in pH 2 Sulfuric Acid)	93–95 % NPOC (RSD = 1.55 %)	Not Tested

$$P = 100\left[A\left(V_s + V\right) - B\,V_s\right]/C\,V \qquad (1)$$

where:

A = analyte concentration (mg/L) in spiked sample,
B = analyte concentration (mg/L) in unspiked sample,
C = concentration (mg/L) of analyte in spiking solution,
V_s = volume (mL) of sample used, and
V = volume (mL) added with spike.

14.7.3 The percent recovery of the spike shall fall within limits to be specified in advance by the user, or within 80–120 % if no values are specified. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: *(1)* the matrix interference must be removed, *(2)* all samples in the batch must be analyzed by a test method not affected by the matrix interference, or *(3)* the results must be qualified with an indication that the spiked sample did not fall within the performance criteria of the test method.

14.8 *Duplicate:*

14.8.1 To check the precision of sample analyses, analyze a sample in duplicate with each sequence of samples to be analyzed.

14.8.2 Calculate the standard deviation of the duplicate values and compare to the single operator precision in the collaborative study using an F test. Refer to 6.4.4 of Practice D5847 for information on applying the F test.

14.8.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

14.9 *Independent Reference Material (IRM)*—In order to verify the quantitative value produced by the test method, analyze an IRM submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the reference material should be in the range of the test method. The value obtained must fall within the control limits specified by the outside source.

15. Precision and Bias[7]

15.1 This method was evaluated and validated in a single laboratory to meet the requirements of a not fully validated standard, subject to full validation within five years of its initial publication, as described in Practice D2777 – 06.

15.2 The method detection limit is shown in Table 2 and accuracy and precision data for various compounds is shown in Table 3.

16. Keywords

16.1 carbon; carbon dioxide; high temperature catalytic oxidation; organic carbon; total carbon

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1180. Contact ASTM Customer Service at service@astm.org.

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