

METHOD 9060

TOTAL ORGANIC CARBON

1.0 SCOPE AND APPLICATION

1.1 Method 9060 is used to determine the concentration of organic carbon in ground water, surface and saline waters, and domestic and industrial wastes. Some restrictions are noted in Sections 2.0 and 3.0.

1.2 Method 9060 is most applicable to measurement of organic carbon above 1 mg/L.

2.0 SUMMARY OF METHOD

2.1 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO₂) by either catalytic combustion or wet chemical oxidation. The CO₂ formed is then either measured directly by an infrared detector or converted to methane (CH₄) and measured by a flame ionization detector. The amount of CO₂ or CH₄ in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.2 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of preliminary sample treatment as well as the instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by Method 9060 are:

1. Soluble, nonvolatile organic carbon: e.g., natural sugars.
2. Soluble, volatile organic carbon: e.g., mercaptans, alkanes, low molecular weight alcohols.
3. Insoluble, partially volatile carbon: e.g., low molecular weight oils.
4. Insoluble, particulate carbonaceous materials: e.g., cellulose fibers.
5. Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g., oily matter adsorbed on silt particles.

2.3 Carbonate and bicarbonate are inorganic forms of carbon and must be separated from the total organic carbon value. Depending on the instrument manufacturer's instructions, this separation can be accomplished by either a simple mathematical subtraction, or by removing the carbonate and bicarbonate by converting them to CO₂ with degassing prior to analysis.

3.0 INTERFERENCES

3.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

3.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle which may be included in the sample.

3.3 Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

4.2 Apparatus for total and dissolved organic carbon:

4.2.1 Several companies manufacture analyzers for measuring carbonaceous material in liquid samples. The most appropriate system should be selected based on consideration of the types of samples to be analyzed, the expected concentration range, and the forms of carbon to be measured.

4.2.2 No specific analyzer is recommended as superior. If the technique of chemical oxidation is used, the laboratory must be certain that the instrument is capable of achieving good carbon recoveries in samples containing particulates.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities, and should be boiled and cooled to remove CO₂.

5.2 Potassium hydrogen phthalate, stock solution, 1,000 mg/L carbon: Dissolve 0.2128 g of potassium hydrogen phthalate (primary standard grade) in Type II water and dilute to 100.0 mL.

NOTE: Sodium oxalate and acetic acid are not recommended as stock solutions.

5.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with Type II water.

5.4 Carbonate-bicarbonate, stock solution, 1,000 mg/L carbon: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100-mL volumetric flask. Dissolve with Type II water.

5.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to Step 5.3.

NOTE: This standard is not required by some instruments.

5.6 Blank solution: Use the same Type II water as was used to prepare the standard solutions.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

NOTE: A brief study performed in the EPA Laboratory indicated that Type II water stored in new, 1-qt cubitainers did not show any increase in organic carbon after 2 weeks' exposure.

6.3 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between sample collection and the start of analysis should be minimized. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

6.4 In instances where analysis cannot be performed within 2 hr from time of sampling, the sample is acidified (pH \leq 2) with HCl or H₂SO₄.

7.0 PROCEDURE

7.1 Homogenize the sample in a blender.

NOTE: To avoid erroneously high results, inorganic carbon must be accounted for. The preferred method is to measure total carbon and inorganic carbon and to obtain the organic carbon by subtraction. If this is not possible, follow Steps 7.2 and 7.3 prior to analysis; however, volatile organic carbon may be lost.

7.2 Lower the pH of the sample to 2.

7.3 Purge the sample with nitrogen for 10 min.

7.4 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

7.5 For calibration of the instrument, a series of standards should be used that encompasses the expected concentration range of the samples.

7.6 Quadruplicate analysis is required. Report both the average and the range.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Verify calibration with an independently prepared check standard every 15 samples.

8.4 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 415.1 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th ed., p. 532, Method 505 (1975).

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