

Carbon, organic, total, wet oxidation (O-3100-83)

Parameter	Code
Carbon, organic, total (mg/L as C)-----	00680

1. Application

This method is suitable for the analysis of water, water-suspended-sediment mixtures, brines, and waste waters containing at least 0.1 mg/L of total organic carbon (TOC). The method is not suitable for the determination of volatile organic constituents.

2. Summary of method

The sample is acidified, purged to remove inorganic forms of carbon, and oxidized with persulfate in an autoclave at 116-130°C. The resultant carbon dioxide is measured by nondispersive infrared spectrometry.

3. Interferences

3.1 Inorganic forms of carbon usually present in most waters are readily converted to carbon dioxide when acidified but will interfere if the sample is not purged adequately. Purgeable organic compounds are lost during this step.

3.2 Water samples containing large concentrations of reducing agents interfere by decomposing the persulfate oxidant.

4. Apparatus

4.1 *Ampules*, precombusted, 10 mL, glass, Oceanography International, or equivalent.

4.2 *Ampule purging and sealing unit*, Oceanography International, or equivalent.

4.3 *Autoclave*, Oceanography International 0512AU, or equivalent.

4.4 *Carbon analyzer*, Oceanography International, or equivalent.

4.5 *Homogenizer*, Willems Polytron PT10ST, Brinkman Instruments, or equivalent.

5. Reagents

Carbon-free reagent water is required. All reagents should be analyzed to determine carbon content, and any reagent that yields a significant blank value should be rejected.

5.1 *Carbon standard solution*, 1.00 mL = 1.00 mg C (carbon): Dissolve 2.1254 g potassium hydrogen phthalate (primary standard grade, dried at 105°C for 1 h in reagent water and dilute to 1,000 mL. Store at 4°C.

5.2 *Phosphoric acid solution*, 1.2 N: Add 83 mL H₃PO₄ (85 percent) to reagent water and dilute to 1 L with reagent water. Store in a tightly stoppered glass bottle.

5.3 *Potassium persulfate*, reagent grade, granular: Finely divided forms of this reagent should be avoided.

6. Procedure

6.1 Add 0.2 g potassium persulfate (a dipper calibrated to deliver 0.2 g may be used) and 0.5 mL of 1.2 N phosphoric acid solution to precombusted ampules.

6.2 Homogenize the sample until it is uniformly suspended. Rinse the homogenizer with distilled water after each use.

6.3 Pipet (class A pipets are recommended) a volume of water sample (10.0 mL maximum) into an ampule and adjust the volume to 10 mL with reagent water.

6.4 Prepare one reagent blank (10 mL reagent water plus acid and oxidant) for every 15 to 20 water samples.

6.5 Prepare standards to cover the range 0.1 to 40 mg C/L by dilution of the carbon standard solution (1.00 mL = 1.00 mg C).

6.6 Immediately place the filled ampules on the purging and sealing unit and purge them at 60 mL/min for 6 min with purified oxygen.

6.7 Seal the ampules according to instructions in the manufacturer's manual.

6.8 Place the sealed samples, blanks, and a set of standards in ampule racks, place the racks in an autoclave, and digest for 4 h at 116-130°C.

6.9 Set the sensitivity range of the carbon analyzer by adjusting the zero and span controls in accordance with instructions in the manufacturer's manual.

6.10 Break the combusted ampules in the cutter assembly of the carbon analyzer, sweep the carbon dioxide into the infrared cell with nitrogen gas, and record the area of each carbon dioxide peak. CAUTION: Combusted ampules are under positive pressure and should be handled with care to prevent them from exploding.

7. Calculations

7.1 Prepare an analytical curve by plotting the peak area of each standard versus the concentration (mg/L) of the organic carbon standards.

7.2 The relationship between peak area and carbon concentration is curvilinear. Operating curves must be defined each day the samples are analyzed.

7.3 Determine the concentration of total organic carbon in each sample by comparing its peak area with the analytical curve.

7.4 Multiply the result of step 7.3 by the appropriate dilution factor.

8. Report

Report total organic carbon concentration (TOC) as follows: less than 10 mg/L, one decimal; 10 mg/L and above, two significant figures.

9. Precision

It is estimated that the percent relative standard deviation for total organic carbon will be greater than that reported for dissolved organic carbon (method O-1100).

Selected references

- Fredericks, A.D., 1968, Concentration of organic carbon in the Gulf of Mexico: Office of Naval Research, Report 68-27T, 65 p.
- Oceanography International Corporation, 1970, The total carbon system operating manual: College Station, Tex., 51 p.