Carbon, organic, suspended, wet oxidation
(O-7100-83)

Parameter	*Code*
Carbon, organic, suspended (mg/L as C)	00689

1. Application

This method is suitable for the analysis of suspended organic carbon (SOC) constituents found in natural waters, brines, and waste waters. The method is not suitable for the determination of volatile organic constituents.

2. Summary of method

The sample is collected on a silver filter, acidified, purged to remove inorganic 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 will 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.

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,* 14.7 *N* (85 percent), reagent grade.

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

6. Procedure

6.1 Carefully remove the silver filter from its container with forceps and coil it into a roll about 1/8inch in diameter using a steel or glass mandrill.

6.2 Drop the coiled filter into a precombusted glass ampule. Add 0.5 mL of 14.7 *N* (85 percent) phosphoric acid and 8 mL reagent water.

6.3 Repeat steps 6.1 and 6.2 using the blank filter supplied by field personnel.

6.4 Cover the top of the ampule with aluminum foil and heat on a steam bath for 12 to 24 h.

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). Pipet 10.0 mL of each standard into precombusted glass ampules containing 0.5 mL of 14.7 N phosphoric acid.

6.6 Introduce 0.2 g potassium persulfate (a dipper calibrated to deliver 0.2 g may be used) and rinse down any solid adhering to the neck of the ampule with 2 mL of reagent water.

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

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

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

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

6.11 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 Calculate the concentration of suspended organic carbon in the original water sample from the equation

where

S = apparent carbon concentration of sample, in mg/L,

B = apparent carbon concentration of blank, in mg/L,

V = number of liters of water filtered (in field).

8. Report

Report suspended organic carbon concentrations (SOC) 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 suspended organic carbon will be greater than that reported for dissolved organic carbon (method O-1100).

Selected references

Menzel, D.W., and Vacaro, F.F., 1964, The measurement of dissolved organic and particulate carbon in seawater: Limnology and Oceanography, v. 9, p. 138—142.

Oceanography International Corporation, 1970, The total carbon system operating manual: College Station, Tex., 51 p.

$$\text{Carbon, suspended organic (mg/L)} = \frac{S-B}{V},$$