Carbon, inorganic plus organic, total in bottom material, dry weight, induction furnace (O-5101-83)

Parameter	*Code*
Carbon, total in bottom material dry weight (g/kg as C)	00693

1. Application

This method is suitable for the determination of total carbon in bottom material at concentrations of 0.1 g/kg and above.

2. Summary of method

A sample is oxidized, in the presence of oxygen and a catalyst, in a crucible in an induction furnace. The carbon dioxide evolved is measured by thermal conductivity.

3. Interferences

Sulfur, halides, and water vapor can interfere but are eliminated by traps. Carbon impurities in the reagents can interfere.

4. Apparatus

4.1 *Carbon determinator,* Leco WR-12, or equivalent, consisting of an oxygen purification system, an induction furnace, dust traps, moisture traps, a halide trap, a sulfur trap, a catalytic furnace, and thermistor cells.

4.2 *Combustion crucibles,* carbon-free, disposable, Leco 528-018, or equivalent.

4.3 *Dryer-balance,* consisting of a Mettler LP 15 dryer, a Mettler PC 440 balance, and a Mettler GC 301 application input device, or equivalents.

4.4 *Glass reagent scoops,* Leco 501-032, or equivalent.

4.5 *Grinder,* Torsion MG 2 electric mortar grinder, or equivalent.

4.6 *Sieves*, U.S. No. 18 (2-mm opening) and U.S. No. 60 (0.25-mm opening).

5. Reagents

5.1 *Carbon standards,* 1 g steel rings containing known amounts of carbon C.

5.1.1 High standard, ca. 0.8 percent C, Leco 501-506.

5.1.2 Low standard, ca. 0.1 percent C, Leco 501-502.

5.2 *Catalytic furnace reagent,* platinum on silica, Leco 501-587.

5.3 *Combustion accelerators*

5.3.1 Copper accelerator, Leco 501-263, or equivalent.

5.3.2 Iron chip accelerator, Leco 501-077, or equivalent.

5.4 *Oxygen,* commercial grade.

5.5 *Sodium carbonate,* anhydrous, reagent grade.

5.6 *Trap reagents*

5.6.1 Anhydrone, Leco 501-171, or equivalent, for moisture trap.

5.6.2 Antimony, Leco 769-608, or equivalent, for halide trap.

5.6.3 Ascarite, Leco 183-001, or equivalent, for oxygen purification system.

5.6.4 Manganese dioxide, Leco 501-587, or equivalent, for sulfur trap.

6. Procedure

6.1 Sieve the wet sample through a 2-mm sieve. Place the sieved sample in an aluminum pan that has been fired at 550°C for 1 h and dry the material at 40°C overnight.

6.2 Grind the dried material in an electric grinder, to a powder that passes a No. 60 sieve.

6.3 To determine the percent of dry material, the initial powder must be weighed before drying. Dry an accurately weighed amount of the powder (approximately 1 g) in a combination dryer-balance at 105°C to a constant weight. Determine the percent of dry material.

6.4 Follow the instructions in the manufacturer's manual to leak-test, blank, and calibrate the carbon determinator.

6.5 Place a new combustion crucible on an analytical balance and add 1 g (maximum) of the sample (obtained in step 6.2). Record the sample weight to three significant figures.

6.6 Add a scoopful of iron chip accelerator, spreading it evenly over the sample.

6.7 Add a scoopful of copper accelerator in a similar manner.

6.8 Combust the sample in the determinator and record the reading of percent carbon for the sample.

6.9 Repeat the determination using a smaller sample, if the reading is higher than 5.000 percent C.

7. Calculations

7.1 Calculate the weight of dry bottom material in the sample from the equation

$$W = \frac{S \times D}{100}$$

where

W = weight of dry bottom material in sample, in g,

S = weight of sample, from step 6.5, in g, and

D = percent of dry material in sample, from step 6.3.

7.2 Calculate the concentration of carbon in the bottom material from the equation

$$C(g/kg) = \frac{10 \times DVM \times C_S}{W},$$

where

DVM = direct readout of percent carbon for sample weight as set on compensator,

W = weight of dry bottom material in sample, obtained in step 7.1, in g, and

C_3 = compensator setting, in g.

8. Report

Report total carbon concentrations in bottom material as follows: less than 1 g/kg, one decimal; 1 g/kg and above, two significant figures.

9. Precision

Single-operator precision data for carbon steel standards for each standard (10 replicates each) is as.follows:

Carbon steel (percent)	*Mean determined (percent)*	*Relative standard deviation (percent)*
0.0440----------	0.0442	3.6
0.166----------	0.166	2.1
0.941----------	0.943	0.86

Selected reference

Leco Corporation, Instruction manual, WR-12, Carbon determinator, model 761-100, 1976: 3000 Lakeview Ave., St. Joseph, MI 49085.