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Carbon (Total, Organic, and Inorganic)

1. Application

This method covers the determination of total carbon (TC), organic carbon (OC) and inorganic carbon (IC) concentrations in soil, plant tissues and manures by dry combustion using a LECO CNS-2000 analyzer.

The LECO CNS-2000 Carbon, Nitrogen and Sulfur Analyzer is a non-dispersive, infrared, microcomputer based instrument, designed to measure the total carbon, nitrogen, and sulfur content in a wide variety of materials (soil, plant tissue, fertilizers, meat products, dairy products, seeds, food, resins, and environmental wastes) in a nominal 200 mg sample weight.

2. Summary of Method

Total carbon and organic carbon contents of a sample are determined in two separate combustion conditions/profiles. The first combustion profile will maximize the recovery of TC while the second profile will minimize the decomposition of carbonate C and maximize the recovery of OC. The two main variables of these profiles are the furnace temperatures and the oxygen flow rate. The furnace temperatures are set at 1350°C and 900°C for the TC and OC profiles, respectively. IC is calculated as the difference between the TC and OC values.

Although SPAL uses 900°C for the determination of OC, temperatures between 375 and 1000°C are found in the literature. Based on this, SPAL could accommodate specific requests from clients to run OC samples at a specific temperature.

In some cases hydrochloric acid has been used for the decomposition of carbonates. However, this treatment generates Cl gases that can damage the infrared detector and requires the use of scrubbing substrates in the system to prevent damage. Therefore, if a client needs to determine organic and inorganic carbon (by difference) the first option given is the OC at 900°C, the second option is OC at a temperature defined by the client, and as a last option (and therefore more expensive) the use of hydrochloric acid (by the client or by SPAL) to remove carbonates previous to the OC determination.

3. Safety

3.1 Each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should be made available to all personnel involved in the chemical analysis.

3.2 Follow the manufacturer's recommendation for safe operation of the instrument.

3.3 Secure compressed gas cylinders and use the proper gas regulators.

3.4 Sample boats being unloaded from the furnace are extremely hot - do not handle them until they cool down.

4. Interferences

4.1 Fineness of the ground sample affects sample combustion and thus analysis results. All samples should be ground to pass an 18 mesh sieve (1-mm) or finer.

4.2 Sample boats will be contaminated with inorganic carbonates (IC) following the analysis for organic carbon. To remove the carbonates place the contaminated boats in a muffle furnace at 1000°C for one hour or run the boats as blanks at 1350°C in the Leco CNS-2000

5. Sample Collection, Preservation and Handling

5.1 Soil and plant samples are dried at 55°C and 65°C, respectively. The dried soil sample is then ground to pass a 12 mesh screen and the plant tissue is ground to pass a 2 mm screen.

5.2 Acid-digested or acid-treated samples should not be run on the Leco CNS-2000

5.3 The Leco COM-CAT combustion accelerator can be used to insure complete combustion when large samples are used or when total sulfur determination is required

6. Apparatus and Materials

6.1 Scale 0.0001 g

6.2 Leco CNS-2000 Carbon, Nitrogen and Sulfur Analyzer

6.3 Autoloader Assembly with 49-position sample rack

6.4 Printer

6.5 Sample spatula

7. Reagents

7.1 COM-CAT combustion catalyst (Tungsten Tri-oxide, Leco 501-426)

7.2 Anhydron (Anhydrous Mg perchlorate, Leco 501-171)

7.3 Lecosorb (Sodium hydroxide, Leco 502-174)

7.4 Sulfamethazine (Leco 502-304), EDTA (Leco 502-092) or soil standards (Leco 502-309, 502-308)

7.5 Glass wool

7.6 UHP helium gas

7.7 UHP oxygen gas

7.8 Compressed air (low water content)

8. Methods

8.1 Operate instrument according to manufacturer's instructions. The following are generalized instructions:

8.1.1 Turn furnace on (or take off standby).

8.1.2 Turn gas regulators to desired flow rate (40 psi).

8.1.3 Select the appropriate method for either TC or OC.

8.1.4 Wait until the furnace has stabilized at the set temperature.

8.1.5 Test for leaks in the helium lines, ballast tank and combustion system.

8.1.6 Define the standard by entering the appropriate carbon content of the pure primary standard.

8.1.7 Include ten blanks and three dried (or desiccated pure) primary standards at the beginning of each run to calculate the calibration factor for determining carbon (to correct for drift).

8.1.8 Weigh out 0.15 to 2.0g of dried soil or plant tissue, respectively, in a clean sample ceramic crucible (boat). 1.0g of COM-CAT accelerator can be added to the boats before the sample is weighed. Weights are automatically transferred to the microprocessor by pressing the print button on the scale pad.

8.1.9 Transfer the weighed samples to the 49-position sample rack and load the rack onto the autoloader.

8.1.10 Run the samples.

9. Calculations

The inorganic carbon content is calculated as the difference between total carbon and organic carbon as follows:

$$\% \text{ IC} = \% \text{ TC} - \% \text{ OC}$$

10. Quality Control

10.1 The method's analysis range (lower limit is based on 3x standard deviation of the blank) is 0.02 - 200 mg carbon. Analysis precision is RSD 0.4%.

10.2 At least 10 blanks must be analyzed daily before each run and the blank with the value closest to zero should be selected and used for blank correction. Three to five standards should be analyzed and the one with the value closest to the real value of the standard should be used to correct for drift in the calibration curve.

10.3 At least one standard of the same material as the samples should be run with every ten unknowns and at the end of each run to verify calibration.

11. Reporting

11.1 Data is reported as %C for soil and plant tissue on a dry weight basis.

11.2 The detection limit is 0.020 mg C.

12. References

12.1 Leco Corporation. 2002. CNS-2000 elemental analyzer. CNS-2000 instruction manual.

12.2 Organic Carbon Dry Combustion method, Leco CN-2000, FP 2000, or CNS-2000 (JD Schwab, Personal communications).

12.3 Islam, K.R. and R.R. Weil. 1998. A rapid microwave method for colorimetric measurement of soil organic carbon. *Commu- soil-sci-plant-anal.* 29 (15/16):2269-2284.

12.4 Matejovic, I. 1997. Determination of carbon and nitrogen in samples of various soils by dry combustion. *Commu-soil- sci-plant-anal.* 28(17/18):1499-1511.

12.5 Leco Corporation 2003. Organic application note. Carbon, nitrogen and sulfur testing in soil/plant tissue.