



Chapter R

Total carbon by combustion

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U.S. Geological Survey Open File Report 02-223-R

Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey

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Accepted: 1/27/93

Revised: 1/15/02

Principle

Total carbon in geologic materials is determined using an automated carbon analyzer (Jackson and others, 1987). A weighed sample (approximately 0.20 g) is combusted in an oxygen atmosphere at 1350°C to oxidize carbon (C) to carbon dioxide (CO₂). Moisture and dust are removed and the carbon dioxide gas is measured by a solid state infrared detector.

Interferences

High concentrations of fluorine and molybdenum will interfere with the detection of CO₂ by coating the cell walls and the detector of the carbon analyzer. Samples suspected to contain molybdenum in the range of 0.2 to 1 percent are analyzed using a reduced sample weight and a halogen trap is installed in the flow system when high concentrations of fluorine are present in the samples. A problem may be encountered due to abnormally rapid combustion of organic-rich materials. This problem can be corrected by the addition of a retardant (COM-AID) to the sample.

Scope

The operating range for total carbon is from 0.05 percent to about 50 percent. Approximately 40 samples can be analyzed in a day.

Apparatus

- Carbon analyzer, LECO Model CR-412
- Ceramic combustion sample boats
- Tank of high purity oxygen, with regulator

Reagents

- COM-AID, combustion retardant
- Anhydrous, magnesium perchlorate, Mg(ClO₄)₂
- Distributing organization and reference material samples are:
 - LECO Corporation: 0.98, 5.05, 12.0, 42.0, and 47 percent total carbon
 - U.S. Geological Survey: GXR-4, GXR-3, GXR-2, and SDO-1
 - Canadian Certified Reference Materials Project (CCRMP): MGR-1, SO-4, SO-1, SO-3, and, STSD-2.
 - National Research Council Canada: BCSS-1

Safety precautions

The major danger in this procedure are thermal burns which can potentially be sustained when the operator removes the very hot sample boats from the furnace. Care must be exercised in removing the boats with tongs. Avoid contact of the hot boats with combustible materials. The use of a lab coat, safety glasses, and protective shoes are strongly recommended while operating the instrument. Toxic gases may be produced during sample combustion, therefore, the instrument should be used under a working exhaust hood or vented to one. See the *CHP* and *MSDS* for further information.

Procedure

(Additional details are in the manual provided by the LECO Corporation.)

1. Replace the left anhydrous tube with the right anhydrous tube. Replace the right side with a fresh anhydrous tube. This procedure must be followed daily when using the instrument.
2. Begin by pressing anywhere on the monitor, now press the "MAINTENANCE" key on the monitor, and then the "AMBIENT MONITOR" key. This will provide a list showing the current status of the various systems parameters. The furnace temperature will gradually rise to 1350°C. Turn on the necessary fume hoods.
3. Open valve on oxygen cylinder and set second stage regulator to 40 psi.
4. Turn on the printer.
5. Calibrate the balance.
6. Conditioning the instrument for analyses:
 - a. First run 3 to 4 blanks. (Use spent boats for this purpose.)
 - b. Go to "FRONT PANEL" by pressing "ESC" twice. Press "ANALYZE".
 - c. Press "Select ID Code", select the blank code. Press "ESC". Press "Manual Weight", enter 0.3500g, press "Enter" 3 times, then press "Manual Weight" again.
 - d. To begin, place the boat next to the viewing window, press the "ANALYZE" key.
 - e. Wait until the message center displays "Load Sample Now" and then slide the boat into the furnace until it touches the boat stop.
 - f. Close the viewing window and immediately press "Start integration".
 - g. After analysis is complete, remove the combustion boat from the furnace CAREFULLY.
 - h. Complete the other blanks.
 - i. Now run 3 to 5 conditioning samples by randomly picking samples from the jobs to be analyzed to determine the concentration range for the samples. (Use spent boats for this purpose.)
 - j. Press "Select ID Code", select the appropriate code to identify the sample. Press "ESC".
 - k. Weigh approximately 0.200 g of the sample material. (Use a smaller amount, approximately 0.100g for samples with high Carbon content (greater than 10%). Add COM-AID, (combustion retardant), to the samples with high Carbon content only.)
 - l. After weighing the sample, press "Print" on the balance, then spread the sample evenly by rocking the boat back and forth.
 - m. To begin analysis, place boat next to the viewing window, press the "ANALYZE" key.
 - n. Wait until the message center displays "Load Sample Now" and then slide the boat into the furnace until it touches the boat stop
 - o. Close the viewing window and immediately press "Start integration".
 - p. After analysis is complete, the total Carbon content will be displayed and printed.
 - q. Remove the combustion boat from the furnace CAREFULLY.
 - r. Continue with the analysis (steps j. through q.) until all the samples are complete
7. Calibration procedure:
 - a. A wide variety of standards are used depending upon the concentration range of the unknown samples used for conditioning the instrument and the type of material being analyzed.
 - b. Press the "Select ID Code", select the appropriate standard. Press "ESC".
 - c. Weigh out 5 aliquots of the calibration standard. Use new boats for this purpose.
 - d. Continue with steps k. through r. above.
 - e. After the 5 standards are analyzed, press "ESC", then "Calibrate", and then "Standard Calibrate". Choose the 5 analyzed standards for the calibration curve. Press "ESC", press "Yes" for the calibration curve. Press "ESC". The calibration information will be printed.
 - f. Go to "FRONT PANEL". Press "Method", select the appropriate calibration standard. This information will be printed. Press "ESC".
8. Sample analysis: (Reference standards and samples)
 - a. Analyze one or two reference standards using new boats. The reference standard is a check for the calibration curve. Continue as described above in *Conditioning the instrument for analyses*: (steps j. through r.).

- b. Analyze all the samples using new boats and continue as described above in *Conditioning the instrument for analyses*: (steps j. through r.). If the percent carbon of an unknown is higher than the calibration standard, take a smaller weight and repeat analysis or use a higher calibration curve just for the samples with higher Carbon contents.
- c. Run one duplicate sample for each job at the end of the day.

Calculation

Calculations are performed by the instrument's microprocessor, and the percent carbon is reported for each sample.

Assignment of uncertainty

Table 1 shows the analytical results for total carbon in selected reference materials, duplicate samples, and method blanks by combustion.

Table 1.—Analytical performance summary for total carbon (percent)

[A= Terashima, 1979; B=Govindaraju, 1989; remaining *pv* from Potts and others, 1992; carb=carbonate carbon]. See page ix of the introduction to this Methods Manual for an explanation of the abbreviations used in the analytical performance summary tables.

Reference	Description	n	Mean	s	pv		% RSD	% R
GXR-1	jasperoid	10	0.187	0.005	0.16	? carb	3	125
MRG-1	gabbro	24	0.342	0.008	0.293		2	117
STSD-2	stream sediment	23	1.73	0.02	1.60		0.9	108
BCSS-1	marine sediment	24	2.19	0.02	2.19		1	100
SRM 2704	river sediment	20	3.39	0.03	3.35		0.9	101
SO-3	soil	10	6.74	0.03	6.66		0.5	101
SDO-1	shale	32	9.94	0.05	9.95	B	0.5	100
SRM 88b	dolomitic limestone	10	12.69	0.03	12.65	cv carb	0.2	100
SGR-1	shale	14	27.5	0.3	27.03	A	1	102

Table 1.—Continued—Duplicate samples results

Duplicate samples	k	n	Mean	s	% RSD	Concentration range	No. of < (total)	No. of < (pairs)
	66	2	4.84	0.05	1	0.23 to 49.0	0	0

Table 1.—Continued--Method blank results 3s values are considered the lower limit of detection (LOD), and 5s values are considered the lower limit of determination (LLD)

Method blank	n	Mean	s	3s	5s
	30	-0.02	0.01	0.03	0.05

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