Inorganic Application Note

Carbon and Sulfur in Lime

Instrument

CS-200, CS-300, CS-400 and CS-444 Series Carbon and Sulfur Determinators

Calibration Standard

NIST or other suitable standards
Suggestions: LECO 502-319 @ 1.36% C and 1.53% S, NIST SRM 2690 Fly Ash @ 0.15% S

Accessories

LECO 528-018 Ceramic Crucibles (preheated), LECO 773-579 Metal Scoop, 763-266 LECOCEL or 763-263 LECOCEL III, and 502-231 or 501-077 Iron Chip Accelerators (for carbon-only instruments substitute 501-263 Copper Accelerator for LECOCEL or LECOCEL III)

Sample Weight

~0.20 to 0.25 grams

Sample Preparation

Sample should be uniform powder.

Instrument Settings

Power Level:	maximum
Pre-analyze Purge*:	5 seconds
Pre-analyze Delay*:	10 seconds
Carbon Minimum Time-out:	50 seconds
Carbon Comparator Level:	1.00%
Sulfur Minimum Time-out:	60 seconds
Sulfur Comparator Level:	1.00%

**For improved precision at low carbon levels (<0.1%), a pre-analyze purge of 10 and delay of 25 seconds is recommended.*

Note

Samples containing water of hydration (crystallization) or hydroxides could experience diminished sulfur recovery.

Carbon/Sulfur

Method

1. Preheat ceramic crucibles in a muffle or tube furnace at 1200 to 1300°C for not less than 15 minutes or at 950 to 1050°C for not less than 40 minutes. Remove the crucibles from the furnace, cool for 1 to 2 minutes, and place in a desiccator for storage. If the crucibles are not used within four hours, they should be rebaked.
2. Determine the blank.
   a. Enter 1.000 g weight into weight stack.
   b. Add one level 773-579 Metal Scoop of LECOCEL followed by one level metal scoop of iron chip accelerator to a preheated crucible.
   c. Place crucible on furnace pedestal and analyze.
   d. Repeat steps 2a through 2c a minimum of five times.
   e. Enter blank following routine outlined in operator's instruction manual.
3. Calibrate.
   a. Weigh ~0.20 to 0.25 g calibration standard into the center of the preheated crucible. Enter the weight into the weight stack.
   b. Add one level 773-579 Metal Scoop of LECOCEL followed by one level metal scoop of iron chip accelerator covering the calibration sample.
   c. Place crucible on furnace pedestal and analyze.
   d. Repeat steps 3a through 3c a minimum of five times and calibrate the instrument following the calibration procedure as outlined in the operator's instruction manual.
   e. Verify the calibration by analyzing the calibration standard again. It should fall within the expected tolerance. If not repeat steps 3a through 3e.
4. Analyze samples.
   a. Weigh ~0.20 to 0.25 g sample into the center of the crucible. Enter the weight into the weight stack.
   b. Add one level 773-579 Metal Scoop of LECOCEL followed by one level metal scoop of iron chip accelerator covering the sample.
   c. Place crucible on furnace pedestal and analyze.

Typical Results

	Weight (g)	Carbon (%)	Sulfur (%)
LECO 502-319	0.2056	1.36	1.53
Ore	0.2035	1.36	1.53
@1.36% C,	0.2024	1.36	1.53
1.53% S	0.2175	1.36	1.54
	0.2130	1.36	1.52
NIST 2690	0.1966	0.393	0.151
Fly Ash	0.2072	0.392	0.150
@ 0.15% S	0.2000	0.386	0.150
Carbon is	0.2019	0.383	0.149
not certified	0.1960	0.383	0.151

	Weight (g)	Carbon (%)	Sulfur (%)
Quicklime	0.1951	1.914	0.0168
Sample A	0.2120	1.842	0.0174
	0.2120	1.910	0.0161
Quicklime	0.2020	0.606	0.0220
Sample B	0.1993	0.653	0.0213
	0.2072	0.595	0.0218
Dolomitic Lime	0.2171	0.209	0.0249
Sample A	0.2067	0.235	0.0240
	0.1951	0.213	0.0232
Dolomitic Lime	0.2217	0.0952	0.0149
Sample B	0.2040	0.0918	0.0151
	0.2173	0.0914	0.0152

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