Organic Application Note

Sulfur Determination in Cement, Limestone, Lime, Coal Fly Ash, Shale, Ore, and Similar-Type Material

Instrument
LECO SC144-Series

Accessories
529-204 Ceramic Crucibles, 502-321 Com-Cat® Accelerator

Calibration Samples
NIST SRM 2690 & 2691 Coal Fly Ash, NIST SRM 886 Gold Ore, NIST Portland Cement Series, LECO 502-096 Coal Fly Ash, or other suitable reference materials

Sample Weight
0.1 to 0.25 g* **Use ~ 0.1 g for refractory samples such as Portland cement.*

Furnace Temperature
1400°C

NOTE: Normal operating temperature is 1350°C, refer to the operator's instruction manual for information for modifying the furnace temperature.

Method Parameters

Minimum Integration Time:	60 Seconds
Maximum Integration Time:	400 Seconds
Comparator Terminate Level:	2.00%

Procedure
1. Prepare instrument as outlined in operator's instruction manual.
2. Condition instrument by analyzing ~0.3 gram coal samples until the sulfur readings are stable.
3. Determine Blank.
   a. Enter 1.0000 gram into weight stack using Blank in the Name column.
   b. Add ~1 gram of 502-321 Com-Cat® into a 529-204 Crucible.
   c. Initiate the Analysis sequence, when Load Furnace message appears on the display, slide crucible into combustion tube until it reaches crucible stop. Remove crucible at the end of the analysis.
   Note: Allow the instrument to purge for 1-2 minutes prior to starting the next analysis.
   d. Repeat steps 3a through 3c at least three times and enter blank following the procedure outlined in the operator's instruction manual.
4. Calibrate (refer to operator's instruction manual for details on calibration procedures).
   a. Weigh ~0.1 to 0.25 gram of calibration sample into a 529-204 Crucible and enter weight into weight stack.
   b. Add ~1 gram of 502-321 Com-Cat® to the crucible and thoroughly mix with the sample.
   c. Initiate the Analysis sequence, when Load Furnace message appears on the display, slide crucible into combustion tube until it reaches crucible stop. Remove crucible at the end of the analysis.
   Note: Allow the instrument to purge for 1-2 minutes prior to starting the next analysis.
   d. Repeat steps 4a through 4c at least three times for each calibration sample that is to be used for calibration.
   e. Calibrate the instrument following the calibration instructions found in the operator's instruction manual.
5. Analyze unknown samples following the procedure outlined in steps 4a through 4c.

SC144-Series

Typical Results

Sample	Weight g	% S
NIST	0.2134	0.152
SRM 2690	0.2103	0.154
Coal Fly Ash	0.2069	0.154
@ 0.15% S	0.2066	0.150
	0.2035	0.154
	***X* =**	***0.153***
	***s* =**	***0.002***
NIST	0.2061	0.837
SRM 2691	0.2052	0.821
Coal Fly Ash	0.2111	0.832
@ 0.83% S	0.2099	0.832
	0.2057	0.835
	***X* =**	***0.831***
	***s* =**	***0.006***
NIST	0.1010	1.24
SRM 1888	0.1023	1.20
Portland	0.1039	1.23
Cement	***X* =**	***1.22***
@ 1.26% S	***s* =**	***0.02***
NIST	0.1528	1.47
SRM 886	0.1405	1.46
Gold Ore	0.1411	1.49
@ 1.466% S	***X* =**	***1.47***
	***s* =**	***0.015***

Delivering the Right Results

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