

Organic Application Note

Sulfur in Cement, Fly Ash, Limestone, Soil,[®] and Ore

Instrument

TruSpec S Module

Sample Preparation

A representative, uniform sample is required. Samples should be ground to pass a No. 100 (150 micron) sieve. Samples should be dried to a constant weight.



Accessories

528-203 Ceramic Boat, 502-321 Com-Cat™

Calibration Samples

NIST, or other suitable cement, fly ash, ore, or limestone reference materials.

Method Parameters*

External Sulfur Analysis Parameters

Furnace Temperature	1450°C
Analysis Stabilize Comparator	2
Manual Load Baseline Delay Time (seconds)	5
Baseline Time (seconds)	1
Auto Detect Data Missed Time (seconds)	5
Endline Time (seconds)	1
Minimum Analysis Time (seconds)	120
Comparator Level	0.30%
Conversion Factor	1.00
Significant Digits	5

System Configuration

Gas Conservation Timeout (minutes)	15
Auto Increment Sample Name	Disable
Nominal Mass	1.0000
Lance Delay Time (seconds)	20
Lance Limits	50000

*Refer to TruSpec/Sulfur Module Operator's Instruction Manual for Method Parameter definitions.

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing a three to five ~0.25 g coal samples.
3. Determine blank.
 - a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
 - b. Add ~1 g of 502-321 Com-Cat™ into a 528-203 Ceramic Boat.

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- c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on the display, slide ceramic boat into the combustion tube until it reaches boat stop. Alternately, place ceramic boat onto the appropriate position of carousel if equipped with autoloader system.
- d. Repeat steps 3a through 3c a minimum of three times.
- e. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate/Drift Correct.
 - a. Weigh ~0.1 to 0.35 g of calibration sample into a 528-203 Ceramic Boat, enter mass and sample identification into Sample Login (F3).
 - b. Add ~1 g of 502-321 Com-Cat™ to the ceramic boat and thoroughly mix with the sample.
 - c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches boat stop.
 - d. Repeat steps 4a through 4c a minimum of three times for each calibration/drift sample used.
 - e. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
5. Analyze Samples.
 - a. Weigh ~0.1 to 0.35 g sample into 528-203 Ceramic Boat and enter mass and identification information into Sample Login (F3).
 - b. Add ~1 g of 502-321 Com-Cat™ to the ceramic boat and thoroughly mix with the sample.
 - c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches boat stop. Alternately, place crucible onto the appropriate position of carousel if equipped with auto-loader system.

Typical Results*

Sample	Mass g	% S	Sample	Mass g	% S
NIST	0.2126	0.150	LECO	0.2485	0.747
SRM	0.2156	0.152	502-318	0.2434	0.722
2690	0.2063	0.152	Ore Tailing	0.2590	0.727
Fly Ash	0.2060	0.146	0.73 % S	0.2583	0.725
0.15 %S	0.2066	0.148		0.2454	0.748
		X = 0.150			X = 0.734
		s = 0.002			s = 0.013

Sample	Mass g	% S
LECO	0.2058	1.37
502-319	0.2112	1.37
Ore Tailing	0.2143	1.36
1.36 % S	0.2126	1.35
	0.2026	1.35
		X = 1.36
		s = 0.01
		s = 0.004

* Results based on multipoint, multi-standard calibration using NIST and LECO soil, ore, cement, and fly ash reference materials.



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