

Organic Application Note

Sulfur in Coal and Coke

Instrument

TruSpec S Module

Sample Preparation

A representative, uniform sample is required. Samples should be prepared in accordance to ASTM D 2013. Coal and coke reference materials such as those offered by LECO and NIST are properly prepared.

Accessories

528-203 Ceramic Boat

Calibration Samples

NIST, LECO coal and coke reference materials, or other suitable coal and coke reference materials.

Method Parameters*

External Sulfur Analysis Parameters

Furnace Temperature	1350°C
Analysis Stabilize Comparator	2
Manual Load Baseline Delay Time (seconds)	5
Baseline Time (seconds)	1
Auto Detect Data Missed Time (seconds)	3
Minimum Analysis Time (seconds)	60
Comparator Level	0.30 %
Endline Time (seconds)	1
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 sample name.
 - b. Initiate the Analysis Sequence (F5), when "Load Sample into Furnace" message appears on the display, slide an empty (clean) 528-203 Ceramic Boat into the combustion tube until it reaches boat stop. Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.



TruSpec[®]

- c. Repeat steps 3a and 3b a minimum of three times.
- d. Set blank following the procedure outlined in operator's instruction manual.
4. Calibrate/Drift Correct.
 - a. Weigh ~0.1 to 0.35 g calibration sample into 528-203 Ceramic Boat and enter mass and identification information into Sample Login (F3).
 - b. Initiate the Analysis Sequence (F5). When "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches the boat stop. Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.
 - c. Repeat steps 4a and 4b a minimum of three times for each calibration/drift sample used.
 - d. 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 sample identification into Sample Login (F3).
 - b. Initiate the Analysis Sequence (F5). When "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches the boat stop. Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.

NOTE: Sulfur results for coal and coke samples and reference materials are typically reported on a Dry Basis. Therefore, either the materials must be dried prior to analysis or the moisture content determined and entered during the Sample Login procedure. Samples can be dried at 105°C for one hour prior to analysis.

Typical Results*

Sample	Mass g	% S	Sample	Mass g	% S
NIST	0.2533	0.504	LECO	0.2092	1.31
2682b	0.2532	0.500	502-452	0.2020	1.28
Coal	0.2538	0.495	Coal	0.2041	1.30
0.4917% S	0.2538	0.495	1.30% S	0.2504	1.31
		X = 0.498			X = 1.30
		s = 0.004			s = 0.01

Sample	Mass g	% S	Sample	Mass g	% S
NIST	0.2529	0.585	NIST	0.2527	4.71
SRM	0.2542	0.589	2685b	0.2525	4.69
2775	0.2536	0.583	Coal	0.2533	4.74
Foundry	0.2547	0.581	4.73% S	0.2521	4.72
Coke		X = 0.585			X = 4.72
0.5816% S		s = 0.001			s = 0.02

*Results based on multipoint calibration using NIST and LECO coal and coke reference materials.



LECO Corporation • 3000 Lakeview Ave. • St. Joseph, MI 49085-2396
Phone 800-292-6141 • Fax 269-982-8977
info@leco.com • www.leco.com • ISO-9001:2000 No. FM 24045