

Sulfur Determination in Coal and Coke

LECO Corporation; Saint Joseph, Michigan USA

Instrument: S832 Series

Introduction

Total sulfur value is an important characterization measurement for coal and coke fuel materials and is considered to be part of the ultimate analysis parameters (determination of carbon, hydrogen, nitrogen, sulfur, and ash with oxygen calculation by difference). The total sulfur value is a primary parameter used for assessing the quality of a fuel, calculating coal and coke preparation and cleaning processes, and the emissions potential of the fuel.

Sample Preparation

A representative, uniform sample is required. Samples should be prepared in accordance to ASTM D2013. Coal and coke reference materials such as those offered by LECO and NIST are properly prepared. Samples are analyzed on an as-determined basis. A separate portion of the sample is analyzed for moisture content in accordance with ASTM D3173, ASTM D7582, or ISO 11722 for calculations to dry basis.

Method Reference

ASTM D4239 – Sulfur (Method A)

Accessories 528-203 Ceramic Boat

Calibration Samples

LECO coal and coke reference materials, NIST, or other suitable coal, coke, and pure compounds per ASTM methods.

Method Parameters*

Furnace Temperature	1350°C
Lance On Delay	20 seconds
Manual Analysis Mode	Single Sample
Nominal Blank Mass	1.0000 g

Element Parameters	Sulfur
Wait for Baseline Stability	Yes
Starting Baseline	2 seconds
Use Comparator	Yes
Comparator	0.30
Minimum Integration Time	90 seconds
Maximum Integration Time	360 seconds

Range Select**	Auto
Range Lower Limit**	800
Range Upper Limit**	950

Automatically Started Analysis	
Auto Detect Data Missed Time	3 seconds
Autostart Level	0.01 V

Manually Started Analysis	
Integration Delay	0 seconds

*Refer to S832 Operator's Instruction Manual for Method Parameter definitions.

**Applicable to S832DR only.



Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing a minimum of three ~0.25 g coal or coke samples.
3. Determine instrument blank.
 - a. Login a minimum of three blanks.
 - b. Place the ceramic boat in front of the furnace entrance or the appropriate autoloader position.
 - c. Initiate the analysis by pressing the Analyze button.
 - d. For manual systems, load the sample into the furnace and press the analyze button when prompted by the software.
 - e. Repeat steps 3b through 3d a minimum of three times.
 - f. Set the Blank according to the procedure outlined in the operator's instruction manual.
4. Instrument calibration/drift correction.
 - a. Login a minimum of three Standard reps for each calibration/drift reference material to be used for calibration/drift.
 - b. Weigh ~0.25 g of a calibration/drift reference material into the ceramic boat and enter the mass and reference material identification into the standard login.
 - c. Place the ceramic boat in front of the furnace entrance or the appropriate autoloader position.
 - d. Initiate the analysis by pressing the Analyze button.
 - e. For manual systems, load the sample into the furnace and press the analyze button when prompted by the software.
 - f. Repeat steps 4b through 4e a minimum of three times for each calibration/drift reference material.
 - g. Calibrate/drift correct by following the procedure in the operator's instruction manual.
5. Sample Analysis
 - a. Login a Sample with a desired number of reps.
 - b. Weigh ~0.25 g of sample into the ceramic boat and enter the mass and sample identification into the sample login.
 - c. Place the ceramic boat in front of the furnace entrance or the appropriate autoloader position.
 - d. Initiate the analysis by pressing the Analyze button.
 - e. For manual systems, load the sample into the furnace and press the analyze button when prompted by the software.
 - f. Repeat steps 5b through 5e as necessary.

The LECO logo, consisting of the word 'LECO' in a stylized, bold, sans-serif font. The 'L' and 'E' are connected, and the 'O' has a globe icon inside it. The logo is white and set against a green background.

Organic Application Note

Typical Results*

S832

Standard	Mass (g)	Sulfur (%)
NIST 1632d	0.2532	1.49
Coal	0.2411	1.48
Sulfur %	0.2438	1.46
1.46 ±0.07	0.2570	1.48
	0.2556	1.48
Avg.		1.48
Std. Dev.		0.01

502-684	0.2429	5.69
Petroleum Coke	0.2588	5.71
Sulfur %	0.2433	5.71
5.60 ±0.13	0.2599	5.67
	0.2509	5.70
Avg.		5.70
Std. Dev.		0.02

502-845	0.2483	0.66
QAR Coal	0.2406	0.67
Sulfur %	0.2500	0.67
0.66 ±0.03	0.2457	0.67
	0.2582	0.67
Avg.		0.67
Std. Dev.		0.003

NIST 2693	0.2488	0.47
Bituminous Coal	0.2487	0.47
Sulfur %	0.2521	0.47
0.46 ±0.01	0.2584	0.47
	0.2484	0.47
Avg.		0.47
Std. Dev.		0.001

502-672	0.2402	2.11
Coal	0.2415	2.12
Sulfur %	0.2499	2.11
2.16 ±0.09	0.2525	2.12
	0.2454	2.12
Avg.		2.11
Std. Dev.		0.01

S832DR

Standard	Mass (g)	Sulfur (%)
NIST 1632d	0.2442	1.50
Coal	0.2489	1.50
Sulfur %	0.2443	1.50
1.46 ±0.07	0.2423	1.50
	0.2534	1.49
Avg.		1.50
Std. Dev.		0.01

502-684	0.2527	5.64
Petroleum Coke	0.2572	5.63
Sulfur %	0.2503	5.68
5.60 ±0.13	0.2579	5.67
	0.2489	5.68
Avg.		5.66
Std. Dev.		0.02

502-845	0.2480	0.65
QAR Coal	0.2472	0.65
Sulfur %	0.2542	0.64
0.66 ±0.03	0.2460	0.65
	0.2481	0.65
Avg.		0.65
Std. Dev.		0.003

NIST 2693	0.2535	0.46
Bituminous Coal	0.2552	0.45
Sulfur %	0.2553	0.46
0.46 ±0.01	0.2423	0.46
	0.2453	0.46
Avg.		0.46
Std. Dev.		0.003

502-672	0.2476	2.13
Coal	0.2514	2.11
Sulfur %	0.2494	2.11
2.16 ±0.09	0.2542	2.12
	0.2531	2.14
Avg.		2.12
Std. Dev.		0.01

*Results dry basis (moisture corrected) using a linear force through origin calibration. LECO Reference Materials were used for the calibrations.