

Instrument: SC832DR

Determination of Sulfur and Carbon in Graphite and Carbon-Based Battery Materials

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Introduction

The electrical conductivity of an anode material is closely linked to its Carbon content and structural purity. In particular, high-purity graphite (typically with a Carbon content exceeding 99.95 %) is preferred due to its superior electronic conductivity and consistent performance characteristics. Impurities within the graphite matrix, especially Sulfur, can significantly degrade conductivity and overall battery efficiency. Sulfur is commonly present in raw mined graphite and must be effectively removed during the refining process to ensure material quality. As such, precise determination of both Carbon and Sulfur content is essential for quality control, as these parameters directly influence the electrochemical performance and reliability of Carbon-based anode materials in lithium-ion batteries.

Instrument Model and Configuration

The LECO SC832DR is a macro combustion Sulfur and Carbon determinator. The instrument utilizes a pure Oxygen environment in a high-temperature, horizontal, ceramic combustion furnace designed to handle macro sample masses. A weighed sample is combusted, and the combustion gases are swept from the furnace and passed through anhydrous for the removal of moisture. The combustion gases are then carried to non-dispersive infrared (NDIR) cells for the detection of Sulfur (as SO₂) and Carbon (as CO₂).

NDIR cells function on the principle that SO₂ and CO₂ absorb infrared (IR) energy at unique wavelengths within the IR spectrum. Incident IR energy at these wavelengths is absorbed as the analyte gases pass through the IR absorption cells, with the absorption being dependent upon the path length of the cell. The Dual Range (DR) SC832 model has a wider Sulfur range due to the use of both a short and long path length IR cell for Sulfur determination. This allows for the measurement of both high and low range Sulfur signals. The SC832DR software automatically selects which cell to use for optimum Sulfur determination.

Sample Preparation

Samples should be a uniform, representative powder or granular material.

Note: Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all reference materials and samples.



Accessories

528-203 Ceramic Combustion Boats*, 761-929 Crucible Tongs, and 501-614 Spatula

*For optimal precision, combustion boats should be baked in a muffle furnace at 1,000 °C for a minimum of one hour prior to use. Once the combustion boats have cooled, they should be transferred to a desiccator for storage until use. If the combustion boats are not used within twenty-four hours, they should be re-baked. After baking, handle combustion boats with clean tongs only; do not use fingers.

Reference Materials

LCRM[®], LRM[®], NIST, or other suitable reference materials.

Note: Refer to reference material certificates for sample preparation instructions.

General Parameters

Furnace Temperature	1450 °C
Lance On Delay	20 s
Manual Loading Model	Single Sample
Nominal Mass	1.0000 g
Cooling Delay	0 s

Element Parameters

Parameter	Sulfur	Carbon
Wait for Baseline Stability	Yes	Yes
Starting Baseline	2 s	2 s
Use Comparator	Yes	Yes
Comparator Level	0.30 %	1.00 %
Minimum Integration Time	120 s	180 s
Maximum Integration Time	250 s	360 s
Range Select	Auto	--
Range Lower Limit	800	--
Range Upper Limit	950	--

Automatically Started Analyses

Auto Detect Data Missed Time	3 s	3 s
Low Cell Autostart Level	0.010 V	--
High Cell Autostart Level	0.010 V	--
Autostart Level	--	0.010 V

Manually Started Analyses

Integration Delay	0 s	0 s
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Note: Refer to the 832 Series Operator's Instruction Manual for parameter definitions.

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system.
 - a. From the Analysis Screen, use the Login Bar to add three sample replicates.
 - b. Weigh ~0.1 g of a similar matrix (graphite or Carbon-based anode material) into a pre-baked 528-203 Ceramic Combustion Boat and spread the material evenly within the boat.
 - c. Enter the sample mass and identification information into the Analysis Screen.
 - d. Place the combustion boat containing the sample in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
 - e. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the combustion boat containing the sample into the furnace and press the Analyze button.
 - f. Perform steps 2b through 2d (or 2b through 2e for manual loading systems) three times.
 - g. For auto-loading systems, initiate the analysis sequence.
3. Determine the blank.
 - a. From the Analysis Screen, use the Login Bar to add five or more blank replicates.
 - b. Place a pre-baked 528-203 Ceramic Combustion Boat in front of the furnace entrance (for manual loading systems) or place the required number of combustion boats (minimum of five) into the appropriate positions in the autoloader.
 - c. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the combustion boat into the furnace and press the Analyze button.
 - d. For manual loading systems, perform steps 3b through 3c a minimum of five times.
 - e. For auto-loading systems, initiate the analysis sequence.
 - f. Set the blank following the procedure outlined in the operator's instruction manual.

Note: The standard deviation of the last three blanks should be less than or equal to 0.001 % (10 ppm) for both Sulfur and Carbon. Additional blanks beyond the recommended five may be required to achieve the recommended precision.
4. Calibrate or drift correct.
 - a. From the Analysis Screen, use the Login Bar to add the desired number of calibration/drift replicates (minimum of five).
 - b. Weigh an appropriate mass of a suitable reference material into a pre-baked 528-203 Ceramic Combustion Boat and spread the material evenly within the boat.
 - c. Enter the sample mass and identification information into the Analysis Screen.
 - d. Place the combustion boat containing the reference material in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
 - e. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the combustion boat containing the sample into the furnace and press the Analyze button.
 - f. Perform steps 4b through 4d (or 4b through 4e for manual loading systems) a minimum of five times for each calibration/drift sample used.
 - g. For auto-loading systems, initiate the analysis sequence.
 - h. Calibrate or drift correct the instrument following the procedure outlined in the operator's instruction manual.
 - i. Verify the calibration/drift correction by analyzing an appropriate mass of another/different suitable reference material, following steps 4b through 4g and confirm that the results are within the acceptable tolerance range.
5. Analyze the samples.
 - a. From the Analysis Screen, use the Login Bar to add the desired number of sample replicates.
 - b. Weigh ~0.1 g of the sample into a pre-baked 528-203 Ceramic Combustion Boat and spread the material evenly within the boat.
 - c. Enter the sample mass and identification information into the Analysis Screen.
 - d. Place the combustion boat containing the sample in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
 - e. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the combustion boat containing the sample into the furnace and press the Analyze button.
 - f. Perform steps 5b through 5d (or 5b through 5e for manual loading systems) for each sample to be analyzed.
 - g. For auto-loading systems, initiate the analysis sequence.

Typical Results

Data was generated utilizing a linear, force through origin calibration using (~0.125 g) of LECO 502-683 (Lot 23344) LCRM Metallurgical Coke (89.1 % C) for Carbon calibration, and (~0.150 g) of LECO 502-672 (Lot 19345) LCRM Coal (1.93 % S) for Sulfur calibration. The Carbon calibration was verified using (~0.075 g) of LECO 502-683 (Lot 23344) LCRM Metallurgical Coke (89.1 % C). The Sulfur calibration was verified using (~0.075 g) of LECO 502-675 (Lot 20219) LCRM Coal (0.223 % S).

Samples	Mass (g)	Sulfur (%)	Carbon (%)
SiO_x/C Composite Silicon-Based Anode Powder	0.1009	0.014	96.9
MSE Supplies (PO0197)	0.1005	0.014	97.2
	0.0998	0.013	97.0
	0.1000	0.015	96.8
	0.0996	0.013	96.6
	\bar{x} =	0.014	96.9
	s =	0.001	0.2
Hard Carbon Powder	0.1007	0.008	93.7
MSE Supplies (PO0199)	0.1000	0.009	93.6
	0.0997	0.008	93.4
	0.1004	0.009	93.4
	0.1005	0.010	93.0
	\bar{x} =	0.009	93.4
	s =	0.001	0.3
Medium Purity Graphite	0.1002	<0.004**	99.4
BAM (S009)	0.1010	<0.004**	99.1
	0.1008	<0.004**	99.7
	0.1007	<0.004**	99.5
	0.1003	<0.004**	99.5
	\bar{x} =	--	99.5
	s =	--	0.2
Graphite Powder	0.1005	0.018	100.4
Thermo Fisher Scientific (385031000)	0.0995	0.022	99.6
	0.0995	0.018	100.3
	0.0997	0.017	100.0
	0.1005	0.018	99.6
	\bar{x} =	0.019	100.0
	s =	0.002	0.4
	0.1003	2.31	78.1
Conductive Expanded Graphite	0.1002	2.22	78.4
(TIMCAL Timrex® BNB90) Powder	0.1006	2.26	78.1
MSE Supplies (PO5036)	0.0999	2.28	78.4
	0.1003	2.26	77.8
	\bar{x} =	2.27	78.1
	s =	0.03	0.3
Silicon Carbon Alloy (950)	0.1027	0.025	58.2
MSE Supplies (PO8157)	0.1027	0.026	58.5
	0.1021	0.027	58.7
	0.1023	0.027	58.6
	0.1023	0.027	58.8
	\bar{x} =	0.026	58.5
	s =	0.001	0.2

**Results were below the lower method detection limit.

\bar{x} = Sample Mean; s = Sample Standard Deviation

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