Application Note

Instrument: S832/S832DR

Determination of Sulfur in Carbon Black

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Determination of total sulfur in carbon black is an important tool in the rubber manufacturing process. It provides manufacturers with the information they need to determine if the material specifications of a product meet their needs, and aids in calculating the sulfur content of the finished product. Higher sulfur levels in rubber produces a stronger product, but it also causes the product to become more brittle. Adding a filler, such as carbon black, to the rubber can reduce its brittleness and create a more durable rubber product. Knowing the sulfur content of the filler being used provides manufacturers with the ability to add the correct ratio of filler-to-sulfur in the rubber, creating the optimal finished product.

The LECO S832 is a macro combustion sulfur determinator that utilizes a pure oxygen environment in a high-temperature horizontal ceramic combustion furnace designed to handle macro sample masses. The combustion gases are swept from the furnace and are carried to a non-dispersive infrared (NDIR) cell for the detection of sulfur (as SO_2).

Instrument Model and Configuration

NDIR cells function based on the principle that CO_2 and SO_2 absorb infrared (IR) energy at unique wavelengths within the IR spectrum. Incident IR energy at these wavelengths is absorbed as the gases pass through IR absorption cells, with the absorption being dependent upon the path length of the cell. The Dual Range (DR) sulfur 832 model has a wider sulfur range, due to the use of both a short and long path length IR cell. This provides for the measurement of both high and low range sulfur signals. The software automatically selects which cell to use for optimum sulfur determination using the S832DR.

Method Reference

ASTM D1619 Sulfur (Method A)

Sample Preparation

A representative, uniform sample is required, and should be prepared following ASTM D1619. Carbon black should be dried for a minimum of 1 hour @ 125 ±5 °C, then stored in a desiccator until the time of analysis, within 24 hours.

Note: Coal Certified Reference Materials (CRM) should be prepared according to the certificate provided with the material. Coal samples should be analyzed as received and corrected for moisture. A moisture correction value must be determined by oven drying a separate 1 g sample of the coal @ 107 ±3 °C for one hour, in a manner that is consistent with ASTM D7582 or equivalent procedure. The moisture value of the coal should be determined within 24 hours of analysis for an accurate moisture correction.

Accessories

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

**Note: For optimal precision, ceramic combustion boats should be baked in a muffle furnace at 1000 °C for a minimum of 40 minutes. Once the ceramic combustion boats have cooled, they should be transferred to a desiccator for storage. If the ceramic combustion boats are not used within twenty-four hours, they should be re-baked. After baking, handle ceramic combustion boats with clean tongs only; do not use fingers.*

Reference Materials

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

*Analysis Parameters**

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

*Element Parameters**

	Sulfur (S832DR)	*Sulfur (S832)*
Wait for Baseline Stability	Yes	Yes
Starting Baseline	2 s	2 s
Use Comparator	Yes	Yes
Comparator	0.30%	0.30%
Minimum Integration Time	120 s	120 s
Maximum Integration Time	360 s	360 s
Range Lower Limit	800	—
Range Upper Limit	950	—

*Automatically Started Analysis Parameters**

	Sulfur (S832DR)	*Sulfur (S832)*
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 Analysis Parameters**

	Sulfur (S832DR)	*Sulfur (S832)*
Integration Delay	0 s	0 s

**Refer to S832 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. Select three or more replicates in the Login screen.
   b. Weigh ~0.25 to ~0.50 g of a material that is of a similar matrix to the samples being analyzed into a pre-baked 528-203 Ceramic Combustion Boat.
   c. Enter sample mass and identification into the Login screen.
   d. Place the ceramic 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 ceramic combustion boat containing the sample into the furnace and press the Analyze button.
   f. Repeat steps 2b through 2d (or 2b through 2e for manual loading systems) a minimum of three times.
   g. For autoloading systems, initiate the analysis sequence.
3. Determine Blank.
   a. Select three or more Blank replicates in the Login screen.
   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 pre-baked ceramic combustion boats (minimum of three) into the appropriate positions in the autoloader.
   c. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat into the furnace and press the Analyze button.
   d. For manual loading systems, repeat steps 3b through 3c a minimum of three times.
   e. For autoloading 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). Additional blanks beyond the recommended three may be required in order to achieve the recommended precision.
4. Calibrate/Drift Correct.
   a. Select the desired number of calibration/drift replicates in the Login screen (minimum of five).
   b. Weigh ~0.25 g to 0.05 g of a suitable reference material into a pre-baked 528-203 Ceramic Combustion Boat.
   c. Enter sample mass and identification into the Login screen.
   d. Place the ceramic 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 ceramic combustion boat containing the sample into the furnace and press the Analyze button.
   f. Repeat 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 autoloading systems, initiate the analysis sequence.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
5. Analyze samples.
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh ~0.25 g to 0.5 g of the unknown sample into a pre-baked 528-203 Ceramic Combustion Boat, and spread the sample evenly within the combustion boat.
   c. Enter sample mass and identification into the Login screen.
   d. Place the ceramic 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 ceramic combustion boat containing the sample into the furnace and press the Analyze button.
   f. Repeat steps 5b through 4d (or 5b through 5e for manual loading systems) for each sample to be analyzed.
   g. For autoloading systems, initiate the analysis sequence.

TYPICAL RESULTS

Data was generated utilizing a single standard (linear, forced through origin) calibration using ~0.5 g of 502-671 (Lot 17291) Coal LCRM (1.08 % S).

	S832DR		*S832*	
	Mass (g)	*% S*	*Mass (g)*	*% S*
Carbon Black	0.2515	1.437	0.2547	1.439
Sample (A)	0.2511	1.426	0.2536	1.439
	0.2538	1.433	0.2520	1.436
	0.2539	1.437	0.2524	1.441
	0.2528	1.432	0.2529	1.436
	Avg =	***1.433***	***Avg =***	***1.438***
	s =	***0.004***	***s =***	***0.002***
Carbon Black	0.2515	1.186	0.2525	1.203
Sample (B)	0.2530	1.192	0.2516	1.203
	0.2520	1.191	0.2534	1.203
	0.2546	1.190	0.2534	1.205
	0.2524	1.192	0.2521	1.198
	Avg =	***1.190***	***Avg =***	***1.202***
	s =	***0.002***	***s =***	***0.003***
Carbon Black	0.5005	0.0112	0.5022	0.0122
Sample (C)	0.5015	0.0115	0.5056	0.0123
	0.5035	0.0116	0.5061	0.0126
	0.5008	0.0115	0.5028	0.0119
	0.5017	0.0112	0.5032	0.0114
	Avg =	***0.0114***	***Avg =***	***0.0121***
	s =	***0.0002***	***s =***	***0.0004***

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