

Instrument: S832

Sulfur Determination in Rubber

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Sulfur content in rubber compounds may be used to estimate the performance properties of the materials related to the vulcanization curing process. The vulcanization process for rubber materials leads to the cross linking of the rubber macromolecules and the sulfur curing agent. The amount of sulfur present is correlated to the degree of cross linking in the material, which determines the physical properties such as plasticity and elasticity of the rubber materials. The sulfur concentration in a rubber material may also be used to provide insight into the type of curing system used for the material (conventional, efficient, or peroxide for example).

Sample Preparation

A representative, uniform sample is required.

Accessories

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

Calibration Samples

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

General Method Parameters

Furnace Temperature	1350 °C
Lance On Delay	20 s
Nominal Blank Mass	1.0000 g

Element Parameters

Wait for Baseline Stability	Yes
Starting Baseline	2 s
Use Comparator	Yes
Comparator	0.30%
Minimum Integration Time	90 s
Maximum Integration Time	360 s

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing at least two replicates of a rubber sample.
3. Determine instrument blank.
 - a. Login a minimum of three blanks.
 - b. Add ~1.0 g of 502-321 Com-Cat to a crucible.
 - c. Place the ceramic boat in front of the furnace entrance or appropriate autoloader position if applicable.
 - 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 3b through 3e a minimum of three times.
 - g. 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 ~0.10 to 0.15 g of a reference material into a ceramic boat and enter the mass and reference material identification into the standard login.
 - c. Add ~1.0 g of 502-321 Com-Cat on top of the sample.
 - d. Place the ceramic boat in front of the furnace entrance or appropriate autoloader position if applicable.
 - e. Initiate the analysis by pressing the Analyze button.
 - f. For manual systems, load the sample into the furnace when prompted by the software.
 - g. Repeat steps 4b through 4f a minimum of three times.
 - h. Calibrate/drift correct by following the procedure in the operator's instruction manual.
5. Sample Analysis.
 - a. Login a Sample with the desired amount of replicates.
 - b. Weigh ~0.10 to 0.25 g of a rubber sample into the ceramic boat and enter the mass and sample identification into the sample login.
 - c. Add ~1.0 g of 502-321 Com-Cat on top of the sample.
 - d. Place the ceramic boat in front of the furnace entrance or appropriate autoloader position if applicable.
 - e. Initiate the analysis by pressing the Analyze button.
 - f. For manual systems, load the sample into the furnace when prompted by the software.
 - g. Repeat steps 5b through 5f as necessary.

Typical Results*

Name	Mass (g)	% Sulfur
Natural Rubber	0.2247	0.026
	0.2337	0.025
	0.2637	0.025
	0.2719	0.026
	0.2437	0.022
	0.2302	0.023
	Avg =	0.024
	s =	0.002

Compound	0.1379	1.87
Rubber 1	0.1522	1.96
	0.1513	1.94
	0.1417	1.97
	0.1543	1.98
	Avg =	1.94
	s =	0.05

Compound	0.1432	1.46
Rubber 2	0.1703	1.47
	0.1519	1.49
	0.1498	1.44
	0.1466	1.48
	Avg =	1.47
	s =	0.02

*Data was generated utilizing a Linear, force through origin calibration with LECO 502-897 Lot: 1000 BBOT LCRM at 7.35% Sulfur and verified with a LECO 502-852 Lot: 1000 Mineral Oil at 0.105% Sulfur.



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