Sulfur and Carbon Determination in Soda-Lime Glass

LECO Corporation; Saint Joseph, Michigan USA

Instrument: SC832HT Series

Introduction

Glass is an amorphous solid, with physical properties that make it one of the most useful materials known to man. Glass is chemically inert, resistant to heat, easily formed (when molten), relatively inexpensive to manufacture, and transparent. Of the many types of glasses, one of the most common is soda-lime glass, which is used for windows, bottles, jars, and glassware components that do not need to withstand high temperatures.

During processing, sulfur (in the form of sulfate or sulfide) is added to the batch to help remove small gas bubbles from the melt and to control the color of the final product. The amount of sulfate present will influence the sulfur reactions and redox state of the melt, which determines the efficiency of bubble removal. Sulfate levels are also known to affect the ductility of glass, which is an important quality when making glass fiber or rods. Sulfides will react with ferrous iron to form iron (III) sulfide, which absorbs light at approximately 410–420 nm, giving the glass that amber-brown color commonly seen with liquid containers. When oxygen is added to the melt atmosphere, the sulfides will be oxidized, thus reducing the amount retained, resulting in a green/dark green glass.

The SC832HT will determine the total amount of sulfur present in the glass, which is relative to the amount of sulfide or sulfate retained. This value can be used to relate the quality (amount of bubbles present) and color of the glass to the sulfur level, giving production and quality control personnel a direct metric of overall process effectiveness.

The following application note will outline the sample preparation, accessories, method parameters, and procedure to quickly and precisely determine the amount of sulfur present in soda-lime glass.

Sample Preparation

A representative, uniform sample is required.

Accessories

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

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

Reference Materials

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

Method Parameters

Furnace Temperature	1550 °C
Lance On Delay	20 s
Manual Analysis Model	Single Sample
Nominal Blank Mass	1.0000 g

Element Parameters

	Sulfur	*Carbon*
Wait for Baseline Stability	Yes	Yes
Starting Baseline	2 s	2 s
Use Comparator	Yes	Yes
Comparator	0.30	1.00
Minimum Integration Time	60 s	180 s
Maximum Integration Time	360 s	595 s

Automatically Started Analysis

	Sulfur	*Carbon*
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

	Sulfur	*Carbon*
Integration Delay	0 s	0 s

**Refer to SC832 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 minimum of three ~0.25 to 0.50 g samples of a similar matrix.
3. Determine instrument blank.
   a. Login a minimum of three Blank replicates.
   b. Place the pre-baked 528-203 Ceramic Boat in front of the furnace entrance or in 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.

Organic Application Note

Delivering the Right Results

4. Instrument calibration/drift correction.
   a. Login a minimum of three Standard/Drift replicates for each calibration/drift reference material to be used for calibration/drift.
   b. Weigh ~0.25 to 0.50 g of a calibration/drift reference material into the pre-baked ceramic boat, spread evenly within the boat, then enter the mass and reference material identification into the standard/drift login.
   c. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
   d. Initiate the analysis by pressing the Analyze button.
   e. For manual systems, load the sample into the furnace when prompted by the software and press the analyze button when prompted by the software.
   f. Repeat steps 4b through 4e a minimum of three times.
   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 0.50 g of sample into the pre baked ceramic boat, spread evenly within the boat, then enter the mass and sample identification into the sample login.
   c. Place the ceramic boat in front of the furnace entrance or in the appropriate autoloader position.
   d. Initiate the analysis by pressing the Analyze button.
   e. For manual systems, load the sample into the furnace when prompted by the software and press the analyze button when prompted by the software.
   f. Repeat steps 5b through 5e as necessary.

*Typical Results**

Name	*Description*	*Mass (g)*	*Sulfur (%)*	*Carbon (%)*
NIST SRM® 80a	0.087 % S	0.5009	0.080	0.006
Soda-Lime Glass (Beads)	(0.068-0.111 % S range)	0.5010	0.088	0.006
		0.5066	0.088	0.006
		0.5024	0.082	0.006
		0.5000	0.083	0.006
		0.5062	0.083	0.006
		0.5040	0.082	0.005
		0.5004	0.084	0.006
		0.5030	0.083	0.007
		0.5044	0.082	0.006
		Avg =	***0.084***	***0.006***
		s =	***0.0025***	***0.0006***
NIST SRM® 92	0.0164 % S	0.5052	0.014	0.132
Soda-Lime Glass, Low	(0.0122-0.0222 % S range)	0.5047	0.015	0.132
Boron (Powder)		0.5012	0.014	0.133
		0.5061	0.014	0.132
		0.5058	0.014	0.132
		0.5087	0.014	0.132
		0.5027	0.015	0.131
		0.5021	0.014	0.133
		0.5041	0.014	0.132
		0.5048	0.014	0.145
		Avg =	***0.014***	***0.133***
		s =	***0.0004***	***0.004***

**LECO 502-914 Lot: 1001, Synthetic Carbon and Sulfur Certified Reference Material (0.98 % Carbon, 0.97 % Sulfur) was used for the calibration. Results based on using a linear force through origin calibration.*

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