Carbon and Sulfur Determination in Soda-Lime Glass

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS744 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 LECO CS744 series of carbon and sulfur determinators utilize an induction furnace for the rapid analysis of soda-lime glass. The CS744 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

528-018 or 528-018HP Ceramic Crucibles*, 761-929 Crucible Tongs, 773-579 Metal Scoop, 502-173 LECOCEL II accelerator, 502-231 HP Iron Chip accelerator.

**For best precision, ceramic crucibles should be baked off in a muffle or tube furnace (LECO TF4) at a minimum of 1250 °C for a minimum of 15 minutes, or at 1000 °C for 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and transferred to a desiccator for storage. If the ceramic crucibles are not used within four hours, they should be re-baked. After baking, handle crucibles with clean tongs only; do not use fingers.*

Reference Materials

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

Method Parameters

Analysis Parameters

Purge Time	15 s
Analysis Time	20 s
Sample Cool Time	10 s
Furnace Power	100%
Nominal Mass	1.0000 g

**Refer to CS744 Operator's Instruction Manual for Method Parameter definitions.*

Element Parameters

	Carbon	*Sulfur*
Integration Delay	0 s	0 s
Starting Baseline	2 s	2 s
Use Comparator	No	No
Integration Time	50 s	55 s
Use Endline	Yes	Yes
Ending Baseline	2 s	2 s

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 samples of a similar matrix.
3. Determine instrument blank.
   a. Login a minimum of three Blank replicates.
   b. Add one 773-579 Metal Scoop (~1.0 g) of LECOCEL II accelerator to the crucible.
   c. Add one metal scoop (~0.8 g) of Iron Chip accelerator to the crucible.
   d. Place the crucible on the pedestal or appropriate autoloader position.
   e. Initiate the analysis by pressing the Analyze button.
   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/Drift replicates 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 pre-baked ceramic crucible and enter the mass and reference material identification into the standard/drift login.

Inorganic Application Note

c. Add one metal scoop (~1.0 g) of LECOCEL II accelerator on top of the reference material.
d. Add one metal scoop (~0.8 g) of Iron Chip accelerator on top of the reference material.
e. Place the ceramic crucible on the furnace pedestal (or appropriate autoloader position if applicable).
f. Initiate the analysis by pressing the Analyze button.
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 a desired number of reps.
   b. Weigh ~0.25 g of sample into the pre-baked ceramic crucible and enter the mass and sample identification into the sample login.
   c. Add one metal scoop (~1.0 g) of LECOCEL II accelerator on top of the reference material.
   d. Add one metal scoop (~0.8 g) of Iron Chip accelerator on top of the reference material.
   e. Place the ceramic crucible on the furnace pedestal (or appropriate autoloader position if applicable).
   f. Initiate the analysis by pressing the Analyze button.
   g. Repeat steps 5a through 5f as necessary.

*Typical Results**

Name	*Description*	*Mass (g)*	*Carbon (%)*	*Sulfur (%)*
NIST SRM® 80a	0.087 % S	0.2546	0.005	0.084
Soda-Lime Glass	(0.068-0.111 % S range)	0.2582	0.005	0.085
(Beads)		0.2546	0.006	0.084
		0.2542	0.006	0.083
		0.2591	0.006	0.084
		0.2626	0.006	0.083
		0.2646	0.006	0.084
		0.2532	0.006	0.083
		0.2570	0.006	0.083
		0.2533	0.005	0.084
		Avg =	***0.006***	***0.084***
		s =	***0.0005***	***0.0006***
NIST SRM® 92	0.0164 % S	0.2575	0.132	0.013
Soda-Lime Glass,	(0.0122-0.0222 % S range)	0.2549	0.134	0.016
Low Boron (Powder)		0.2539	0.132	0.015
		0.2597	0.133	0.016
		0.2534	0.133	0.014
		0.2527	0.132	0.014
		0.2584	0.134	0.014
		0.2507	0.132	0.015
		0.2524	0.131	0.015
		0.2504	0.134	0.012
		Avg =	***0.133***	***0.015***
		s =	***0.001***	***0.0013***

**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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