Inorganic Application Note

Sulfur in Copper Base Materials

Instrument

CS-200, 300, 400, 444, and 600-Series Determinators (Differences in instrument setup for the 600-Series are noted in parenthesis.)

Calibration Standard

LECO 502-403 Copper Pin, NIST SRM 885 refined copper pin, or other suitable standards

Accessories

Preheated LECO 528-018 Ceramic Crucibles, 773-579 Metal Scoop, 502-403 Copper Pin or other similar-sized copper solid, 501-263 Copper Accelerator

Sample Weight

~1.0 gram

Sample Preparation

Surface contamination on the sample can cause significant errors in the analytical data and care must be taken to ensure a clean representative sample is analyzed.

Program Settings

Pre-Analyze Purge:	10 seconds
Pre-Analyze Delay:	25 seconds
Sulfur Minimum Time-Out:	60 seconds
Sulfur Comparator Level:	1.00%
Clean Interval:	Every 50 analyses
Power Level:	Refer to Instrument Set-up for details; typically the Power Level Knob is set between the 12 and 3 o'clock position. (*CS600-Series:* Typically the Furnace Power is set between 30 and 40.)

Notes

1. Solid samples burn less aggressively than chips or powders. Care should be given to setting up the instrument with the most dense sample. It is suggested that 502-403 Copper Pin Samples or a similar solid copper sample weighing ~1.0 g be used when setting up the instrument. Place the sample in the crucible on top of the accelerator.
2. A clean combustion tube and dust filter are essential before starting this procedure.

Instrument Setup

This method lowers the power level from the maximum set in a typical method in order to decrease the dust produced from combustion of the sample.

1. Turn the Power Level knob counterclockwise to a twelve o'clock position. The knob is located on the front panel. (*CS600-Series:* Set the Furnace Low and High Power to 30 in method parameters.)
2. Add ~1.0 g—one level 773-579 Metal Scoop—501-263 Copper Accelerator to a pre-heated 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
3. Place the LECO 502-403 Copper Pin or ~1.0 g copper solid into the crucible.
4. Enter a 1.0 g weight into the instrument.
5. Place crucible on furnace pedestal and analyze.
6. For a complete combustion, the following plate currents should be observed:
   Maximum: ~250 to 300 mA; 20 seconds into combustion cycle: ~180 to 220 mA
   (*CS600-Series:* Maximum: ~280 to 320 mA; 20 seconds into combustion cycle: ~220 to 260 mA)
7. The sulfur peak should start between 15 and 25 seconds after the combustion cycle begins. (*CS600-Series:* For solids, sulfur peak should start between 10 and 20 seconds after the combustion cycle begins. For powders and chips, the sulfur peak should start between 5 and 15 seconds.)
8. Immediately following the combustion cycle, open the furnace and remove the crucible using the tongs. Look at the sample while it is still red hot. It should visually be a flat smooth melt with no dark spots at the bottom of the crucible.
   Caution sample may be liquid and will be extremely hot!

CS-Series

Instrument Setup (continued)

9. If the plate current exceeds 320 mA (*CS600-Series:* 360 mA) for most of the analysis then dust has probably been generated which will more than likely cause sulfur recovery losses.
10. a. If steps 6 through 8 are satisfied, proceed to step 1 of Method.
    b. If plate current exceeds 320 mA (*CS600-Series:* 360 mA) for most of the analysis—step 9, turn power level control slightly counter-clockwise to reduce the power level. (*CS600-Series:* Reduce the Furnace Low and High Power settings in method parameters.) Manually brush dust filter and cleaner head to remove all dust that has been generated then repeat steps 3 through 8.
    c. If steps 6 through 8 are not satisfied, continue until desired plate current in achieved.

Method

1. Preheat ceramic crucibles in a muffle or tube furnace at 1250°C for not less than 15 minutes or at 1000°C for not less than 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes and placed in a desiccator for storage. If the crucibles are not used within four hours, they should be rebaked.
2. Determine the blank:
    a. Enter 1.000 gram weight into weight stack.
    b. Add ~1.0 g—one level 773-579 Metal Scoop—501-263 Copper Accelerator to a preheated 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
    c. Place crucible on furnace pedestal and analyze.
    d. Repeat steps 2a through 2c a minimum of five times.
    e. Enter blank following routine outlined in operator's instruction manual.
3. Calibrate:
    a. Add ~1.0 g—one level 773-579 Metal Scoop—501-263 Copper Accelerator to a preheated 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
    b. Weigh ~1.0 g calibration standard and place into the center of the crucible entering the weight into the weight stack.
    c. Place crucible on furnace pedestal and analyze.
    d. Repeat steps 3a through 3c a minimum of five times and calibrate the instrument following the auto calibration procedure as outlined in the operator's instruction manual.
    e. Verify the calibration by analyzing the calibration standard again. It should fall within the expected tolerance. If not repeat steps 3a through 3e.
4. Analyze samples:
    a. Add ~1.0 g—one level 773-579 Metal Scoop—501-263 Copper Accelerator to a preheated 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
    b. Weigh ~1.0 g sample and place into the center of the crucible entering the weight into the weight stack.
    c. Place crucible on furnace pedestal and analyze.

Typical Data *Obtained on a LECO CS-444*

Calibrated with BAM NR 227@0.122% S

Sample	*Weight (g)*	*Sulfur (%)*
BAM NR 227	0.9511	0.123
@ 0.122% S	0.9668	0.123
	0.9806	0.122
BCS 183/4	0.9768	0.110
Leaded Gunmetal	0.9549	0.110
Chips @0.11% S	0.9460	0.110
BCS 180/2	0.9287	0.0058
Copper-Nickel Chips	1.0416	0.0059
@0.006% S	0.9293	0.0056

Calibrated with NIST 885 @0.0018% S

Sample	*Weight (g)*	*Sulfur (%)*
NIST 885	0.8989	0.0018
Refined Copper	0.8803	0.0019
Pin @ 0.0018% S	0.8959	0.0018
LECO 502-403	0.9991	0.00093
Copper Pin	0.9930	0.00096
@0.00093% S	0.9957	0.00091

NOTE: If carbon analysis is required simultaneously; this same technique applies.

LECO Corporation
3000 Lakeview Ave. • St. Joseph, MI 49085-2396
Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • *ISO-9001 No. FM 24045*