

Sulfur in Battery Paste (PbO/PbSO₄)

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS-Series

Introduction

The determination of sulfur in battery paste is an important battery manufacturing quality control step as battery performance and life are determined by the properties of the paste. Qualities such as manufacturability, electrochemical efficiency, and durability under charge/discharge cycling are all based upon the nature of the lead sulfate in the paste. The LECO CS line of elemental determinators provides a reliable means of making this determination at a pace quick enough to meet the most demanding quality control plan.

Instrument

CS-200, 230, 300, 400, 444, and 600-Series Determinators (Differences in instrument setup for the 600-Series are noted in parenthesis)

Calibration Standard

502-319 LECO Pulp, or other suitable standards

Accessories

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

Sample Weight ~0.05 to 0.15 grams

Sample Preparation None

Program Settings

Power Level:	See Instrument Setup
Pre-Analyze Purge:	10 seconds
Pre-Analyze Delay:	25 seconds
Sulfur Minimum Time-Out:	60 seconds (45 seconds)
Sulfur Comparator Level:	1.00%
Clean Interval:	Every 50 analyses

Notes: 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.

Instrument Setup

Prepare instrument for operation as outlined in the appropriate operator's instruction manual. 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. Typically the power level knob is set between the 12 and 3 o'clock position.

(CS600-Series: Set the Furnace Low and High Power to 45 in method parameters.)

2. Add ~1 g—one level 773-579 Metal Scoop—of 501-263 Copper Accelerator to a 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
3. Place the LECO 502-403 Copper Pin or a ~1.0 g copper solid into the crucible.
4. Enter 1.0000 g mass and sample identification into Sample Login (F3).
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!**
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 is achieved.

Method

1. Determine blank.
 - a. Enter 1.0000 g mass into Sample Login (F3); using Blank as the sample name.
 - b. Add two level 773-579 Metal Scoop of 501-263 Copper Accelerator to a 528-018 Crucible so that it is evenly distributed on the bottom of the crucible.
 - c. Place crucible on furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
 - d. Repeat steps 2a through 2c a minimum of three times.
 - e. Set blank following procedure outlined in operator's instruction manual.
2. Calibrate/Drift Correct.
 - a. Weigh ~0.05 to 0.15 g calibration/drift sample into the center of a 528-018 Crucible; enter mass and sample identification into Sample Login (F3).
 - b. Add two level 779-579 Metal Scoops of 501-263 Copper Accelerator to the crucible covering the sample.
 - c. Place crucible on furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
 - d. Repeat steps 2a through 2c a minimum of three times for each calibration/drift sample used. Calibrate/drift the instrument using the procedure outlined in the operator's instruction manual.
3. Analyze samples.
 - a. Weigh ~0.05 to 0.15 g sample and place into the center of a 528-018 crucible, enter mass and sample identification into Sample Login (F3).
 - b. Add two level 779-579 Metal Scoops of 501-263 Copper Accelerator to the crucible covering the sample.
 - c. Place crucible on furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).

Typical Results

Obtained on a LECO CS-200; Low Temperature Method

Sample	A	B	C	D	E	F
Sulfur %	0.446	2.081	2.170	1.485	0.971	1.562
	0.451	2.080	2.168	1.504	0.971	1.553
	0.465	2.081	2.146	1.487	0.972	1.563
	0.444	2.085	2.173	1.493	0.976	1.552
	0.440	2.056	2.151	1.495	0.973	1.565
Average S %	0.449	2.077	2.162	1.493	0.973	1.559
Std Dev.	0.010	0.012	0.012	0.007	0.002	0.006

Note: If carbon analysis is required simultaneously, this same technique applies.