

# Inorganic Application Note

## Sulfur in Battery Paste (PbO/PbSO<sub>4</sub>)

### Instrument

CS-200, 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
Sulfur Comparator Level:	1.00%
Clean Interval:	Every 50 analyses

### 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.
2. This method may be utilized for simultaneous carbon analysis.

### 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. 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 30 in method parameters.)
2. Add ~ 1 g—one level 773-579 Metal Scoop—of 501-263 Copper Accelerator to a preheated 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 a 1 gram 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.)



## CS-Series

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. 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 two level 773-579 Metal Scoop of 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. Weigh ~0.15 g calibration standard into the center of a preheated 528-018 Crucible entering the weight into the weight stack.
  - b. Add two level 779-579 Metal Scoop of 501-263 Copper Accelerator to the crucible covering the sample.
  - 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. Weigh ~ 0.10 g sample and place into the center of the crucible entering the weight into the weight stack.
  - b. Add two level 779-579 Metal Scoop of 501-263 Copper Accelerator to the crucible covering the sample.
  - c. Place crucible on furnace pedestal and analyze.

## 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
<b>Average S %</b>	<b>0.449</b>	<b>2.077</b>	<b>2.162</b>	<b>1.493</b>	<b>0.973</b>	<b>1.559</b>
<b>Std Dev.</b>	<b>0.010</b>	<b>0.012</b>	<b>0.012</b>	<b>0.007</b>	<b>0.002</b>	<b>0.006</b>

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