Application Note

LECO
EMPOWERING RESULTS

Instrument: CS744 Series

Sulfur Determination in Battery Paste (PbO/PbSO$_4$)

LECO Corporation; Saint Joseph, Michigan USA

Introduction

The determination of sulfur in battery paste is an important battery manufacturing quality control step as battery performance and life expectancy 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. LECO induction furnace sulfur determinators provide a reliable means of making this determination at a pace quick enough to meet the most demanding quality control plan.

Accessories

528-018HP Crucible (preheated*); 502-492 HP Copper Accelerator or 501-263 Copper Accelerator; 773-579 Metal Scoop; 502-937 Copper Pin LCRM, or other similar sized copper solid.

**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.

General Parameters

Purge Time:	10 s
Delay Time:	25 s
Sample Cool Time:	20 s
Furnace Power:	See Instrument Setup
Nominal Mass:	1.0000 g

Note: Solid samples burn less aggressively than chip or powder samples. Care should be given to setting up the instrument with the most dense sample. It is suggested that 502-937 Copper Pin LCRM samples or a similar solid copper sample weighing ~1.0 g be used when setting up the instrument.

Element Parameters

Regular Mode	*Sulfur*
Integration Delay	0 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	60 s
Use Endline	Yes
Ending Baseline	2 s

Instrument Setup

Prepare the instrument for operation as outlined in the 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. Set the Furnace Power to 50% in the method parameters.
2. Weigh ~1.0 g of Copper Accelerator into a preheated 528-018HP Crucible.
3. Place a 502-937 Copper Pin or ~1.0 g copper solid into the crucible.
4. Enter the sample mass and identification into the software.
5. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.
6. For complete combustion, the following Plate Currents should be observed during analysis:
   Maximum: 300 mA
   Nominal: 250 mA
7. The sulfur peak should start between 10 and 20 seconds after the combustion cycle begins.
8. Immediately following the combustion cycle, open the furnace and remove the crucible using 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 300 mA for most of the analysis, dust has most likely been generated which will cause sulfur recovery loss.
10. If steps 6 through 8 are satisfied, proceed to step 1 of the Procedure.
    a. If plate current exceeds 300 mA for most of analysis, reduce the Furnace Power settings in the method parameters. Manually brush the dust filter and cleaner head to remove all dust that has been generated, then repeat steps 2 through 8.
    b. If steps 6 through 8 are not satisfied, increase the Furnace Power settings in the method parameters. Repeat steps 2 through 8 until desired Plate Current is achieved.

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Login a minimum of three blank replicates.
   b. Weigh ~2.0 g of Copper Accelerator into a preheated 528-018HP Crucible.
   c. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.
   d. Repeat steps 2b through 2c a minimum of three times.
   e. Set the blank following the procedure outlined in the operator's instruction manual.

3. Calibrate/Drift Correct.
    a. Login a minimum of three standard replicates.
    b. Weigh ~0.05 to 0.15 g of a suitable reference material into a preheated 528-018HP Crucible.
    c. Enter the standard mass and identification into the software.
    d. Add ~2.0 g of Copper Accelerator into the crucible, covering the reference material.
    e. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.
    f. Repeat steps 3b through 3e a minimum of three times for each reference material utilized.
    g. Calibrate/Drift correct by following the procedure outlined in the operator's instruction manual.
4. Sample Analysis.
    a. Login a Sample with the appropriate number of replicates.
    b. Weigh ~0.05 to 0.10 g of sample into a preheated 528-018HP Crucible.
    c. Enter the sample mass and identification into the software.
    d. Add ~2.0 g of Copper Accelerator into the crucible, covering the sample.
    e. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.

*Typical Results**

Name	*Mass(g)*	*Sulfur (%)*
Lead (II) Oxide	0.1009	1.12
	0.1024	1.12
	0.1027	1.12
	0.1059	1.10
	0.1013	1.12
	Average=	***1.12***
	s=	***0.01***
Lead (IV) Oxide	0.1029	1.36
	0.1023	1.36
	0.1042	1.36
	0.1023	1.36
	0.1032	1.37
	Average=	***1.36***
	s=	***0.003***
Lead Paste - Desulfurized	0.1036	3.56
	0.1016	3.32
	0.1004	3.58
	0.1043	3.08
	0.1035	3.36
	Average=	***3.38***
	s=	***0.20***

**Note: Results based on a linear, forced through origin calibration utilizing Aldrich Lead (II) sulfide Lot: MKCD2716 (13.40% S).*

© 2018 LECO Corporation