Oxygen in Lead and Lead Alloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TC600 Series

Introduction

The determination of oxygen in lead is an important quality control procedure for lead and lead alloy producers. The amount of oxygen present is an indication of alloy purity, but more specifically, it represents the potential amount of oxide present in the material.

Sample Preparation

This method is written for solid samples; a clean representative sample is required in order to obtain optimum results. Surface contamination must be removed by abrading sample. The prepared sample should be analyzed immediately after preparation.

Accessories

776-247 Graphite Crucible, 611-351-182 Electrode Tip, 761-739 Tin Flux, 501-073 Graphite Powder.

Calibration

LECO 501-147, 501-148, 501-149 or other suitable reference materials.

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	25 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled
Element Parameters	*Oxygen*
Minimum Analysis Time	90 seconds
Significant Digits	5
Conversion Factor	1.000000
Integration Delay	5 seconds
Comparator Level	5.000000 %
Stop if below (%)	0.00000
Gas Dose Parameters	*Oxygen*
Minimum Gas Dose Time	30 seconds
Integration Delay	5 seconds
Comparator Level	1.00000 %
Gas Dose Cycles	1
Furnace Parameters	
Furnace Control Mode	Current
Purge Time	15 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Current	1050 amps*
Outgas High Current	1050 amps*
Outgas Ramp Rate	—
Analyze Low Current	400 amps*
Analyze High Current	850 amps*
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None
Furnace on Time	30 seconds
Temperature Sustain	Step

Step	Start Current	End Current Time
1 400 amps	400 amps	20 seconds
2 850 amps	850 amps	70 seconds

Peak Find Parameters	Oxygen
Look for Shoulders	No
Peak Threshold	0.000000
Calibration Parameters	Oxygen

y = + 1.0x + 0 (default setting)

**May vary depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   d. Place three 761-739 Tin Pellets and ~0.07 g 501-073 Graphite Powder into a 776-247 Graphite Crucible.
   e. Place crucible on electrode pedestal.
   f. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2a through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~1.0 g of a calibration sample; enter mass and sample identification into Sample Login (F3).
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place sample into open port at top of loading head.
   d. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.

Inorganic Application Note

e. Place three 761-739 Tin Pellets and ~0.07 g 501-073 Graphite Powder into a 776-247 Graphite Crucible.
f. Place crucible on the electrode pedestal.
g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
h. Repeat steps 3a through 3g a minimum of three times for each calibration/drift sample used.
I. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Weigh a freshly prepared sample of ~1.0 to 1.5 g; enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3g.

Typical Results

Sample	*Mass(g)*	*% Oxygen*
Lead Sample #1	0.8900	0.0003
	0.9738	0.0002
	0.9535	0.0004
	0.9886	0.0003
	1.1736	0.0004
	$\bar{\chi}$=	***0.0003***
	***s*=**	***0.00008***
Lead Sample #2	1.2098	0.0002
	1.1861	0.0003
	0.9336	0.0003
	1.4829	0.0003
	1.4074	0.0003
	$\bar{\chi}$=	***0.0003***
	***s*=**	***0.00008***

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