Application Note

Instrument: O736 Series

EMPOWERING RESULTS

Oxygen Determination in Tin and Lead Based Solder Powders

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Introduction

One of the primary quality control procedures in the electronics industry is the determination of oxygen in tin and lead based solder powders. The amount of oxygen present is used as a purity metric, by relating the amount of oxygen to tin and lead oxides present in the solder. Oxides are detrimental to the soldering process for many reasons. They are considered dross in the industry, representing waste and a loss of efficiency. The more oxides present in the raw material, the faster oxides will continue to form in the molten solder, reducing the amount and value of metal in your solder. Oxides are abrasive, and cause damage to the pumps and nozzles of high speed/volume solder equipment. Excessive oxide content is also known to cause grainy or dull solder joints, with poor electrical and mechanical properties.

LECO 736 series oxygen determinators, which detect oxygen as CO_2, will quickly and accurately determine the amount of oxygen present in tin and lead-based solder powders. The following application note outlines the sample preparation, method parameters, analytical procedures, and expected performance of this test.

Sample Preparation

Samples should be uniform mesh size. When powder samples are to be analyzed, they cannot be placed directly in the loading head. Samples should be weighed into an open tin capsule. The entire operation must be accomplished using only clean tweezers.

Accessories

776-247 Graphite Crucibles, 502-040 Tin Capsules, 501-073 Graphite Powder, 611-351-182 Lower Electrode Tip for 776-247 Crucibles without automation, 611-351-181 Lower Electrode Tip for 776-247 Crucibles with automation, 766-053 Crucible Tweezers, 760-138 Sample Tweezers

Calibration Samples

LCRM®, LRM®, NIST, or other suitable reference materials. Due to the limited availability of tin/lead reference materials, copper reference materials can be used for calibration.

Analysis Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of three Blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Place a 502-040 Tin Capsule (leave capsule open) into the loading head.
   d. Press the Analyze button on the instrument screen again. The loading head slide-block will close and the lower electrode will open.
   e. Clean the upper and lower electrode either manually or, if applicable, remove the crucible and press the Analyze button again to clean with the automatic cleaner.
   f. Add ~0.05 g of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
   g. Firmly place the graphite crucible onto the lower electrode tip.
   h. Press the Analyze button on the instrument screen. The lower electrode will close and the analysis sequence will start and end automatically.
   i. Repeat steps 2b through 2h a minimum of three times.
   j. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Login a minimum of three Standard replicates.
   b. Weigh ~1.0 g of a calibration/drift reference material into a 502-040 Tin Capsule (leave capsule open).
   c. Enter the mass and sample identification into the appropriate replicate fields.
   d. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   e. Place the tin capsule containing the calibration/drift reference material into the open port at the top of the loading head.
   f. Press the Analyze button on the instrument screen again. The loading head slide-block will close and the lower electrode will open.
   g. Clean the upper and lower electrode either manually or, if applicable, remove the crucible and press the Analyze button again to clean with the automatic cleaner.
   h. Add ~0.05 g of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
   i. Firmly place the graphite crucible onto the lower electrode tip.
   j. Press the Analyze button on the instrument screen. The lower electrode will close and the analysis sequence will start and end automatically.
   k. Repeat steps 3b through 3j a minimum of three times for each calibration/drift reference material used.
   l. Calibrate/drift following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Login the appropriate number of Sample replicates.
   b. Weigh ~1.0 g of a sample into a 502-040 Tin Capsule (leave capsule open).
   c. Enter the mass and sample identification into the appropriate replicate fields.
   d. Repeat steps 3d through 3j for sample analysis.

Method Parameters

General Parameters	*Helium Carrier Gas Parameters*	*Argon Carrier Gas Parameters*
Sample Introduction	Automated Sample Drop	Automated Sample Drop
Analysis Delay	30 s	25 s
Auto Analyze on Mass Entry	No	No
Outgas Before Mass Entry	No	No
Wait for User to Load Sample	Yes	Yes
Vacuum On Time	18 s	18 s
Element Parameters	*Oxygen*	*Oxygen*
Integration Delay	0 s	0 s
Starting Baseline	2 s	2 s
Use Comparator	No	No
Integration Time	45 s	45 s
Use Endline	Yes	Yes
Ending Baseline	2 s	2 s
Furnace Parameters		
Furnace Control Mode	Power	Power
Outgas Furnace Settings		
Cycles	2	2
Power Mode	Constant	Constant
Power	5300* W	4500* W
Time	20 s	20 s
Cool Time	5 s	5 s
Analyze Furnace Settings		
Step 1 Power Mode	Constant	Constant
Low Power	4800* W	4200* W
Autocleaner Parameters		
Autocleaner State	Enabled**	Enabled**
Autocleaner Mode	During Analysis	During Analysis
Autocleaner State	8 s	8 s

**May vary, depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*
***(Enabled do not use in analysis) is used when autocleaner is disabled.*

Typical Results†

Using Helium Carrier Gas

Sample	*Mass (g)*	*% Oxygen*
Tin Alloy Powder	0.9937	0.0142
	1.0180	0.0142
	1.0237	0.0141
	1.0109	0.0141
	1.0149	0.0143
	Avg =	***0.0142***
	s =	***0.0001***
Sn63-Pb37 Alloy Powder	0.9833	0.0123
	1.0238	0.0123
	0.9995	0.0123
	0.9847	0.0125
	1.0085	0.0127
	Avg =	***0.0124***
	s =	***0.0001***

Using Argon Carrier Gas

Sample	*Mass (g)*	*% Oxygen*
Tin Alloy Powder	1.0130	0.0143
	1.0030	0.0144
	1.0214	0.0144
	1.0139	0.0144
	1.0183	0.0143
	Avg =	***0.0144***
	s =	***0.0001***
Sn63-Pb37 Alloy Powder	1.0199	0.0127
	1.0436	0.0125
	1.0102	0.0124
	1.0201	0.0124
	1.0047	0.0127
	Avg =	***0.0126***
	s =	***0.0001***

†Results based upon a linear, forced through origin calibration using LECO 501-149 Lot: 0577 Copper Pins LRM @ 0.0543% O.

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