Oxygen in Copper and Copper Alloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: O836*

*O/ON/OH/ONH836 also applicable

Introduction

Copper is the most important material in the electrical conductor wire industry due to its high benefit-cost ratio. Copper provides admirable surface quality, mechanical properties and high conductivity at a cost much less than silver or gold. Aluminum is the only metal that is routinely substituted for copper, but only in applications where weight is a major factor. One of the most important factors when grading copper is the level of residual impurities, primarily the oxygen level. Oxygen levels in electrical conductor wires typically range from 5-10 ppm (OF – oxygen free, OFE – oxygen-free electronic grades) to around 650 ppm (ETP – electrolytic tough pitch grade). Oxygen levels of up to 200 ppm can actually improve conductivity as it acts as a scavenger element that forms metal oxides with metal contaminants. Too much oxygen has been associated with an increase in hydrogen embrittlement problems.

Oxygen determination by the inert gas fusion infrared detection method is the most widely used and reliable method for broad range and high precision oxygen determination in copper and copper alloys. LECO Corporation offers several configurations of fusion determination for this analysis. The following application note outlines the process from sample generation to data.

Sample Preparation

The sample preparation of copper for oxygen analysis is important due to the oxidation properties of copper. There are two methods of sample preparation that are frequently used for the analysis of oxygen in copper. The standard method according to ASTM E2575 uses chemical etching to remove impurities from the surface of the copper. Subsequently the prepared sample is washed in a suitable solvent such as methanol and dried with warm air. Another method of preparation that is not included in the standard method is physical abrading. The sample can be cut and abraded with a file to remove the surface impurities. Care must be taken to not overheat the sample and change the chemistry of the copper. The prepared sample may be washed in a suitable solvent such as methanol. Both methods require immediate analysis of the prepared sample to minimize the oxidation of the prepared copper.

Accessories

776-247 Graphite Crucibles; 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.

Note: The 611-351-181 Lower Electrode Tip is only required if the instrument is equipped with automation.

Calibration Samples

LECO 501-147, 501-148, 501-149, 501-990 One-Gram Nickel Plated Copper Pins with O content determined; NIST, BCR or other suitable reference materials.

Note: Most LECO copper calibration materials are plated with nickel for stability and oxidation resistance. Sample preparation is not required and the samples are suitable for use straight from the bottle. Refer to the certificate of analysis for details.

Sample Preparation

1. Prepare the samples according to the standard method in ASTM E2575 for the analysis of Oxygen in Copper.
2. If conformance to ASTM E2575 is not required, the samples may be abraded with a file before analysis. *Note: Take care when abrading samples to not heat the samples. Analyze the samples within four (4) hours of abrading.*

Analysis Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of 3 Blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the instrument screen again; the loading head slide-block will close and the lower electrode will open.
   d. Remove the crucible and clean the upper and lower electrode either manually or, if applicable, press the Analyze button again to clean with the automatic cleaner.
   e. Add approximately 0.05 g of 501-073 Graphite Powder to the 776-247 crucible.
   f. Firmly place the graphite crucible on the lower electrode tip.
   g. Press the Analyze button on the instrument screen; the lower electrode will close and the analysis sequence will start and end automatically.

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Delivering the Right Results

Method Parameters**

General Parameters	
Sample Introduction	Automated Sample Drop
Analysis Delay	25 s
Auto Analyze on Mass Entry	No
Outgas Before Mass Entry	No
Wait for User to Load Sample	Yes
Vacuum On Time	18 s
Element Parameters	*Oxygen*
Integration Delay	5 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	30 s
Use Endline	Yes
Ending Baseline	2 s
Range Select	Auto
Range Lower Limit	800
Range Upper Limit	950
Furnace Control Mode	Power
Outgas Furnace Settings	
Cycles	2
Power Mode	Constant
Power	5800* W
Time	20 s
Cool Time	5 s
Surface Oxide Removal	
Remove Surface Oxide	No
Analyze Furnace Settings	
Step 1 Power Mode	Constant
Power	4825* W
Approximate Cycle Time	3.5 Minutes

**May vary, depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*
***The method parameters listed in the table above are optimized for the use of helium as a carrier gas. The use of argon as a carrier gas will require lengthened integration times, as well as reduced outgas and analysis power levels. Please contact the LECO Technical Services Laboratory for additional details.*

Automation Parameters (if equipped)

General Parameters	
Auto Cleaner State	Enabled
Auto Cleaner Mode	During Analysis
Clean Time	8 s

Procedure (continued)

h. Repeat steps 2b through 2g a minimum of three times.
i. Set the blank following the procedure outlined in the operator's instruction manual.

3. Instrument calibration/drift correction.
   a. Login a minimum of 3 Standard replicates.
   b. Weigh ~1.000 gram of a calibration/drift sample, enter the mass and sample identification into appropriate replicate fields.

   Note: LECO Nickel Plated Copper Pin Reference Materials do not require preparation. See the preparation statement on the Reference Material Certificate.

   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the calibration/drift sample into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen; the loading head slide-block will close and the lower electrode will open.
   f. 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.
   g. Add approximately 0.05 g of 501-073 Graphite Powder to the 776-247 crucible.
   h. Firmly place the graphite crucible on the lower electrode tip.
   i. Press the Analyze button on the instrument screen; the lower electrode will close and the analysis sequence will start and end automatically.
   j. Repeat steps 3b through 3i a minimum of three times for each calibration/drift sample used.
   k. 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 ~1.000 gram of sample, enter the mass and identification into appropriate replicate fields.
   c. Repeat steps 3c through 3i for sample analysis.

Typical Results

Note: LECO Nickel Plated Copper Pin Reference Materials do not require preparation. See the preparation statement on the Reference Material Certificate.

Sample	*Mass (g)*	*O%*
CRM	1.0 g	0.0583
CMO Cu600/I		0.0579
Copper Rod		0.0581
0.0581% O		0.0582
±0.0012%		0.0579
Etched		0.0578
		0.0584
		0.0581
		0.0582
		0.0580
	***X*=**	***0.0581***
	***S*=**	***0.0002***

Sample	*Mass (g)*	*O%*
CRM	1.0 g	0.0581
CMO Cu600/I		0.0581
Copper Rod		0.0580
0.0581% O		0.0581
±0.0012%		0.0580
Abraded		0.0583
		0.0583
		0.0583
		0.0579
		0.0580
	***X*=**	***0.0581***
	***S*=**	***0.0001***

CRM	1.0 g	0.0321
NIST 885		0.0318
Refined		0.0317
Copper		0.0325
0.031% O		0.0323
±0.002%		0.0317
Etched		0.0317
		0.0319
		0.0319
		0.0319
	***X*=**	***0.0319***
	***S*=**	***0.0003***

CRM	1.0 g	0.0321
NIST 885		0.0319
Refined		0.0323
Copper		0.0321
0.031% O		0.0321
±0.002%		0.0320
Abraded		0.0318
		0.0322
		0.0319
		0.0320
	***X*=**	***0.0320***
	***S*=**	***0.0001***

OFHC	1.0 g	0.00010
Copper Wire		0.00010
Uncertified		0.00009
Etched		0.00010
		0.00009
		0.00009
		0.00010
		0.00014
		0.00010
		0.00015
	***X*=**	***0.00011***
	***S*=**	***0.00002***

LECO	1.0 g	0.0238
501-147		0.0241
Nickel Plated		0.0240
Copper		0.0238
0.0239% O		0.0238
±0.0003%		0.0237
As Received		0.0240
		0.0237
		0.0239
		0.0237
	***X*=**	***0.0238***
	***S*=**	***0.0001***

Calibrated with CRM CMO Cu600/I utilizing a single standard calibration forced through the origin.

Inorganic Application Note

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