Sulfur in Copper Base Materials

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS744

Introduction

Copper is an extremely useful metal due to its high thermal and electrical conductivity, abundance, and relatively low cost. Copper is used in numerous applications, but is most commonly associated with electronics, construction materials, and industrial machinery. One of the more important quality control parameters that is monitored during copper production is the concentration of sulfur in the copper or copper alloy. High sulfur levels have been associated with embrittlement, a pronounced decrease in ductility. Producers that are able to certify the amount of sulfur present can insure that users of their product will be able to form, bend, and spool without concern of cracking.

Accessories

528-018 or 528-018HP (preheated*) ceramic crucibles; copper accelerator (501-263) or HP copper accelerator (502-492); metal scoop (773-579); tongs (761-929).

**Note: Ceramic crucibles are baked in a muffle or tube furnace (LECO TF-10) at 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 then transferred to a desiccator for storage. After baking, the crucibles should only be handled using clean tongs. If the crucibles are not used within four hours, they should be re-baked.*

Calibration

NIST, JK, JSS, and BCS are certified bodies that have a variety of certified copper base reference materials (SRM/CRM) available. Single or multipoint calibration curves can be utilized. Refer to the operator's instruction manual for details.

Method Parameters

General Parameters

Purge Time:	10 seconds
Analysis Delay:	20 seconds
Sample Cool Time:	10 seconds
Furnace Power†:	50 ±10%

Element Parameters	*Sulfur*
Integration Delay:	0 seconds
Starting Baseline:	2 seconds
Use Comparator:	No
Integration Time:	60 seconds
Use Endline:	Yes
Ending Baseline:	2 seconds

†Note: Furnace power may need to be adjusted based on facility incoming line-power under load. The furnace power should be lowered if oxides are formed during analysis.

Analysis Notes

1. Solid samples burn less aggressively than chips or powders. Care should be given when setting up the instrument with the most dense sample. It is suggested that LECO 502-403 Copper Pin Samples or a similar solid copper sample weighing ~1.0 g be used when setting up the instrument.
2. A clean combustion tube and dust filter are essential before starting this procedure.

Instrument Setup

This procedure lowers the power level from the maximum set in a typical method in order to decrease the dust produced from the combustion of the sample.

1. The power settings are listed in the Method Parameters section.
2. Add ~1.0 g (one level 773-579 Metal Scoop) Copper Accelerator to a pre-heated 528-018 or 528-018HP Crucible so that it is evenly distributed on the bottom of the crucible.
3. Place the LECO 502-403 Copper Pin or ~1.0 g copper solid into the crucible.
4. Enter a 1.0 g weight into the Sample login.
5. Place the crucible on the furnace pedestal and press Analyze.
6. For complete combustion, the following plate current should be observed: Maximum ~280 to 320 mA; Stabilizing ~220 to 260 mA.
7. Immediately following the combustion cycle, open the furnace and remove the crucible using tongs. Observe the sample while still hot. The sample should appear as a flat, smooth melt with no dark spots at the bottom of the crucible. **Caution—sample may be molten and will be extremely hot!**
8. If the plate current exceeds 260 mA for most of the analysis, excess oxide dust has likely been generated, which will cause low sulfur recovery. See Photo 1, Photo 2, and Photo 3 for examples of sample burns.
9. If dust has been generated, clean the combustion tube and dust filter, reduce the Furnace Power and repeat from Step 2. If steps 6 and 7 are satisfied, proceed to Method step 1.

Photo 1

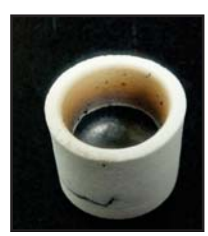

Photo 2

Photo 3

Photo 1 is an example of a good burn. Photo 2 is an example of the power parameters that are slightly high. Photo 3 is an example of the power parameters significantly high.

Inorganic Application Note

Delivering the Right Results

Method

1. Prepare the instrument and crucibles as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of three Blank reps.
   b. Add one scoop (~1.0 g) of copper accelerator to the preheated crucible.
   c. Place the crucible on the pedestal (or the appropriate autoloader position if applicable) and press Analyze.
   d. Repeat steps 2b to 2c a minimum of three times.
   e. Set the blank according to the procedure set forth in the operator's instruction manual.
3. Set the instrument calibration/drift correction
   a. Login a minimum of three Standard reps.
   b. Weigh ~1 g of calibration/drift standard into the crucible and enter the mass and identification of the standard.
   c. Add one scoop (~1.0 g) of copper accelerator to the preheated crucible.
   d. Place the crucible on the pedestal (or the appropriate autoloader position if applicable) and press Analyze.
   e. Repeat steps 3b to 3d a minimum of three times.
   f. Calibrate/Drift according to the procedure set forth in the operator's instruction manual.
4. Analyze the Sample.
   a. Weigh ~1.0 g sample into the crucible and enter the weight into the instrument's sample log in.
   b. Add one scoop (~1.0 g) of copper accelerator to the crucible.
   c. Place the crucible on the pedestal (or the appropriate autoloader position if applicable) and press Analyze.
   d. Repeat as necessary.

Typical Results

	Mass	*Sulfur %*
IARM 90a	~1.0 g	0.0371
0.037% S		0.0371
±0.0017% S		0.0370
		0.0368
		0.0372
		0.0370
		0.0371
		0.0368
		0.0369
		0.0372
Average:		***0.0370***
Std Dev:		***0.0002***

	Mass	*Sulfur %*
IARM 86a	~1.0 g	0.0275
0.029% S		0.0278
±0.0030% S		0.0277
		0.0277
		0.0276
		0.0276
		0.0275
		0.0275
		0.0277
		0.0274
Average:		***0.0276***
Std Dev:		***0.0001***

	Mass	*Sulfur %*
BCS 180/2	~1.0 g	0.0073
0.006% S		0.0068
		0.0072
		0.0073
		0.0066
		0.0067
		0.0063
		0.0067
		0.0054
		0.0072
Average:		***0.0067***
Std Dev:		***0.0006***

NIST 885	~0.9 g	0.00173
0.0018% S		0.00178
±0.0003% S		0.00164
		0.00181
		0.00178
		0.00177
		0.00176
		0.00182
		0.00171
		0.00181
Average:		***0.00176***
Std Dev:		***0.00006***

LECO 502-403	~1.0 g	0.00095
0.00091% S		0.00094
±0.000073% S		0.00096
		0.00093
		0.00095
		0.00095
		0.00095
		0.00094
		0.00092
		0.00092
Average:		***0.00094***
Std Dev:		***0.00001***

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