Analysis of Copper by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

Table of Contents

Analysis of Copper by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

1 INTRODUCTION

This procedure applies METAL-400, *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES),* to characterize the impurities present in copper specimens.

2 SCOPE

This document applies to case working personnel using the associated instrument in support of metallurgy examinations. This procedure determines the concentrations of several elements, if present above the limits of quantitation (LOQ), in relatively pure copper. These elements include Zn, Sn, Mn, Pb, P, Ni, Mg, Cr, Co, Fe, Ag, Te, As, Sb, Cd, Bi, Al, S, Ti, Se, Si, and O. Extension of the procedure to other elements is possible if additional validation is completed. Operation of the SDAR-OES instrument will follow procedures defined in METAL-400 and the specific parameters described below.

3 PRINCIPLE

Compositional analysis of copper by SDAR-OES requires using the predetermined spark sequence to generate characteristic light emissions from the elements present in the specimen. Quantitative determination of individual elemental concentrations is achieved by comparison of measured emission intensities to the manufacturer determined calibration curves resident on the instrument's computer system. The analysis is verified by demonstrating adequate performance on reference materials of the same metal type as the evidentiary items.

4 SPECIMENS

This procedure can be used for the analysis of copper sheets, plates, bars, pipes, and any other object having sufficiently large physical dimensions. Generally, specimens must be large enough to cover the 13mm diameter analysis area to permit their examination by the method outlined here. Any specimen that completely covers the opening in the insert of the small specimen spark stand plate is of adequate size.

5 EQUIPMENT

- SpectroLab LAV M10 spectrometer
- Spectro RH 18/30 optic re-profiling standard
- Spectro standardization materials: RC 11/4, RC 12/12, and RC 14/20
- Lathe
- Carbide or diamond-tipped cutting tool for lathe dedicated for use on copper
- High purity argon
- One or more copper certified reference material(s), CRM(s)
- Additional reference materials, as needed
- Spark stand insert (waveguide), tungsten electrode and wire brush dedicated to copper alloys
- Vacuum cleaner with precision nozzle attachment

- Lint-free wipes

6 STANDARDS AND CONTROLS

A re-profiling standard is provided by the instrument manufacturer. The standardization materials used in this procedure are specific to the copper alloy class and are specified in the Equipment section. Appropriate CRM(s) are selected by the operator to verify the standardization of the SDAR-OES instrument over the ranges applicable to the alloy being analyzed. Additional reference materials may be used to further demonstrate instrument performance on a specific element within a similar matrix.

7 PROCEDURE

7.1 Prepare Specimens

Specimens measured by SDAR-OES must be flat and free of debris and scale over the entire analysis region. Prepare the specimen by mounting it in a lathe and machining the surface to be analyzed to produce a bright finish. To prevent re-oxidation of the surface prior to analysis, specimens should be tested within a short time (several hours) after machining. If significant time elapses, the specimen should be re-machined. This is especially important for oxygen measurement, in which case the specimen should be tested immediately (within one hour) after machining.

7.2 Prepare Instrument

Copper analysis uses the “Cu-10 method” that resides on the SpectroLab LAV M10 spectrometer. Follow the procedures detailed in METAL-400 to clean, re-profile, and standardize the instrument using the appropriate stage, spark source components, and standardization materials prior to analyzing any copper specimens.

7.3 Perform Analysis

Follow METAL-400 to analyze the evidence.

A. Verify by non-destructive means (e.g., X-ray fluorescence spectrometry) that the evidence is relatively pure copper without a significant amount (< ~1%) of total alloying additions.

B. Make several test burns on the evidence to estimate elemental composition for CRM selection.

C. Select CRM(s) and, if needed, additional reference materials, that contain concentrations of elements of interest that appropriately bound the concentrations present in evidentiary materials.

8 ACCEPTANCE CRITERIA

8.1 Instrument Performance

Adequate function of the SpectroLab LAV M10 will be demonstrated on copper CRMs and the results recorded in the case notes.

8.2 Quantitative Analysis

The measured emission intensity of each element of interest is compared to the emission intensity of a selected matrix line and the system calibration curves. This data is used to determine the weight percentage of each of the elements detected. Quantitation of the data is performed automatically by the system program and is not directly controlled by the user.

8.3 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student's t-test statistic of the sample means is typically used for the comparison, as described in METAL-400.

9 LIMITATIONS

Redacted

9.2 Relative Standard Deviation

The relative standard deviation (rsd) ranges listed in METAL-400 Table 2 are used to provide guidance during testing. (Refer to Perform Analysis). An insoluble element, such as oxygen or phosphorous, may produce a higher than expected rsd due to its potentially inhomogeneous distribution in copper.

10 SAFETY

A. Wear safety glasses when preparing specimens and cleaning and operating the SDAR-OES instrument to prevent eye injury.

B. Wear a lab coat and gloves when cleaning the instrument and changing the air filter.

11 REFERENCES

- METAL-400, Chemistry Unit, latest revision

12 REVISION HISTORY

Revision	Issued	Changes
06	09/30/2022	Revised to comply with new formatting requirements. Combined descriptive information from previous Decision Criteria and Calculations sections into Acceptance Criteria. Removed redundancies with METAL-400. Removed informational references.