

Analysis of Small Carbon and Low Alloy Steel Specimens by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

Table of Contents

1	INTRODUCTION	2
2	SCOPE	2
3	PRINCIPLE	2
4	SPECIMENS	2
5	EQUIPMENT	2
6	STANDARDS AND CONTROLS	3
7	PROCEDURE	3
7.1	Prepare Specimens.....	3
7.2	Prepare Instrument	3
7.3	Perform Analysis	3
8	ACCEPTANCE CRITERIA	4
8.1	Instrument Performance	4
8.2	Quantitative Analysis.....	4
8.3	Comparative Analysis	4
9	LIMITATIONS	4
9.1	Limits of Quantitation	4
9.2	Relative Standard Deviation	5
10	SAFETY	5
11	REFERENCES	5
12	REVISION HISTORY	5

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1 INTRODUCTION

This procedure applies METAL-400, *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*, to characterize the major and minor alloying content and the impurities present in small steel specimens. Steel objects of sufficient size (possessing an analysis surface 13mm or larger) should be analyzed using METAL-401 to optimize limits of quantitation and relative standard deviations.

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 small specimens of carbon and low alloy steel. These elements include C, Si, Mn, S, P, Cr, Mo, Al, Ni, Co, Cu, Nb, Ti, V, Sn, As, Zr, Ta, and B. 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 steel alloys by SDAR-OES requires using the pre-determined 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 alloy type as the evidentiary items.

4 SPECIMENS

This procedure can be used for the analysis of plain carbon and low alloy steel sheets, plates, bars, pipes, and any other object having sufficiently large physical dimensions. Generally, specimens must be large enough to cover the 6mm 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
- Small specimen spark stand plate with insert, 6 mm diameter opening, dedicated for steel use
- Spectro RH 18/30 optic re-profiling standard
- Spectro standardization materials RE 12/80, RN 19/24, and BS XCCS
- 60 - 120 grit zirconia grinding paper dedicated for steel OES preparation
- Bench grinder or other metallurgical grinder
- High purity argon

METAL-402-06: SDAR-OES of Small Steel Specimens	Page 2 of 5	Issue Date: 09/15/2022
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- One or more steel certified reference material(s), CRM(s)
- Additional reference materials, as needed
- Spark stand insert (waveguide), tungsten electrode and wire brush dedicated to steel 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 steel 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 surfaces for analysis by dry grinding on 60 – 120 grit zirconium oxide abrasive paper dedicated for steel use.

7.2 Prepare Instrument

Steel analysis uses the “Fe-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. Reprofile and standardize using the spark stand plate with the standard sized opening.

7.3 Perform Analysis

Follow METAL-400 to analyze the evidence.

- Verify by non-destructive means (e.g., X-ray fluorescence spectrometry) that the evidence is iron-based.
- Insert the small specimen spark stand plate and ensure the ceramic insert has no cracks or chips.
- Make several test burns on the evidence to estimate elemental composition for CRM selection.
- 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 carbon and/or low alloy steel CRMs using the small specimen spark stand plate, 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 aluminum or sulfur, may produce a higher than expected rsd due to its potentially inhomogeneous distribution in steel.

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
- METAL-401, Chemistry Unit, latest revision

12 REVISION HISTORY

Revision	Issued	Changes
06	09/15/2022	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.