

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

Table of Contents

1	INTRODUCTION	2
2	SCOPE	2
3	SPECIMENS	2
4	EQUIPMENT	2
5	STANDARDS AND CONTROLS	2
6	PROCEDURE	3
6.1	Prepare Specimens	3
6.2	Prepare Instrument.....	3
6.3	Perform Analysis	3
7	ACCEPTANCE CRITERIA.....	3
7.1	Instrument Performance	3
8	LIMITATIONS	4
8.1	Limits of Quantitation	4
8.2	Relative Standard Deviation	4
9	SAFETY	4
10	REFERENCES.....	4
11	REVISION HISTORY	5

Analysis of Carbon and Low Alloy Steel Samples 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 major and minor alloying content and the impurities present in steel objects.

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 carbon and low alloy steels. These elements include Al, As, B, C, Co, Cr, Cu, Mn, Mo, Nb, Ni, P, S, Sb, Si, Sn, Ta, Ti, V, and Zr. 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 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 13mm diameter analysis area to permit their examination by the method outlined here. Any specimen that completely covers the opening in the plasma source stand plate is of adequate size.

4 EQUIPMENT

- SpectroLab LAS01 spectrometer
- Spectro iCAL optic re-profiling standard
- Spectro standardization materials SUS5, RH12, RH18, RH31, RE 12, and RN 19
- 60 - 120 grit zirconia grinding paper dedicated for steel OES preparation
- Bench grinder or other metallurgical grinder
- High purity argon
- One or more steel certified reference material(s) (CRM)
- 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

5 STANDARDS AND CONTROLS

A re-profiling standard (iCAL) 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 accuracy of the standardization of the SDAR-OES instrument over the ranges applicable to the alloy being

METAL-401-10: SDAR-OES of Steel	Page 2 of 5	Issue Date: 08/15/2024
---------------------------------	-------------	------------------------

analyzed. Additional reference materials may be used to further demonstrate instrument performance on a specific element within a similar matrix.

6 PROCEDURE

6.1 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. Retaining prepared specimens in laboratory air is usually sufficient to avoid oxidation. Should visible oxides develop during storage, regrind the specimen surface prior to testing.

6.2 Prepare Instrument

Steel analysis uses the “Fe-10 method” that resides on the SpectroLab LAS01 spectrometer. Follow the procedures detailed in METAL-400 to clean, re-profile, and standardize the instrument using the appropriate stage and standardization materials.

6.3 Perform Analysis

Follow METAL-400 to analyze the evidence.

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

7 ACCEPTANCE CRITERIA

7.1 Instrument Performance

- A. Adequate function of the SpectroLab LAS01 will be demonstrated on carbon and/or low alloy steel CRMs and recorded in the case notes.
- B. Accuracy is acceptable for an element if the instrument output concentration is within the greater of:
 1. the certificate uncertainty, or
 2. the % relative bias listed in Table 1.

For example, a result between 0.095 and 0.105 wt% would be acceptable for a certificate value of 0.100 wt% even if the certificate gave a range of 0.100 ± 0.003 wt %.

- C. Precision is acceptable for an element if the RSD of five burns is equal to or lower than listed in Table 1.

Table 1: Instrument performance criteria

Elemental Concentration (wt %)	Precision Acceptable RSD	Accuracy % relative bias
> 0.1 - 100	5%	± 5%
0.01 – 0.1	15%	± 10%
less than 0.01	25%	± 15%

8 LIMITATIONS

Redacted

8.2 Relative Standard Deviation

The relative standard deviation (RSD) ranges listed in METAL-400 Table 2 are used to provide guidance during testing. An insoluble element such as aluminum or sulfur, may produce a higher than expected RSD due to its potentially inhomogeneous distribution in steel.

9 SAFETY

- Wear safety glasses when preparing specimens and cleaning and operating the SDAR-OES instrument to prevent eye injury.
- Wear a lab coat and gloves when cleaning the instrument and changing the air filter.

10 REFERENCES

- METAL-400, Compositional Analysis by Spark Discharge in Argon - Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision

11 REVISION HISTORY

Revision	Issued	Changes
09	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.
10	08/15/2024	Revised to accommodate replacement instrument. Removed content redundant with METAL-400. Removed Principle section, its content to be retained in training materials.