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ANALYSIS OF METALS—PROCEDURES FOR THE SETTING UP, CALIBRATION AND STANDARDIZATION OF ATOMIC EMISSION SPECTROMETERS USING ARC/SPARK DISCHARGE

This Australian standard was prepared by Committee CH/10, Analysis of Metals, under the direction of the Chemical Standards Board. It was approved on behalf of the Council of the Standards Association of Australia on 9 April 1986 and published on 7 July 1986.

The following interests are represented on Committee CH/10:

- Aluminium Development Council
- Australasian Institute of Mining and Metallurgy
- Australian Lead Development Association
- Australian Mineral Development Laboratories
- Australian Tin Information Centre
- Australian Zinc Development Association
- Bureau of Steel Manufacturers of Australia
- Confederation of Australian Industry
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This standard was issued in draft form for comment as DR 83192.

AS 2883—1986

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First published 1986

PUBLISHED BY STANDARDS AUSTRALIA
(STANDARDS ASSOCIATION OF AUSTRALIA)
1 THE CRESCENT, HOMEBUSH, NSW 2140
ISBN 0 7262 4215 2

PREFACE

This standard was prepared by the Association's Committee on the Analysis of Metals under the direction of the Chemical Standards Board.

It was prepared in order to standardize procedures and terms associated with the setting-up and use of atomic emission spectrometers for the analysis of metals. A glossary of terms specific to spectrometric analysis of metals is included in this standard as an Appendix and should be referred to in addition to AS 1503 — Glossary of Terms used in Emission Spectroscopy.

Additional standards will describe methods for the atomic emission spectrometric analysis of specific metals or groups of metals.

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for

ANALYSIS OF METALS PROCEDURES FOR THE SETTING UP, CALIBRATION AND STANDARDIZATION OF ATOMIC EMISSION SPECTROMETERS USING ARC/SPARK DISCHARGE

1 SCOPE. This standard describes practices which shall be observed in the setting up, calibration and standardization of equipment for the atomic emission spectrometric analysis of metals using arc/spark discharge. This standard details test procedures used to ensure optimum response from this equipment. A glossary of terms used in this standard is included for reference.

NOTES:

1. Photographic instrumentation is specifically excluded from this standard.
2. Instrument calibration using standard solutions is excluded from this standard.

2 ATOMIC EMISSION EQUIPMENT.

2.1 General. Atomic emission equipment shall contain the following:

(a) A source of energy to cause the atomic emission of electromagnetic radiation from excited elements in the sample.

(b) An optical system for the isolation of specific wavelengths of the emergent radiation.

(c) A system for the detection and measurement of the intensity of the radiation.

2.2 Source of energy. Arc or spark discharges or combinations of various electrical parameters as defined in AS 1503.

2.3 Optical system. An instrument with an entrance slit, a dispersion device (diffraction grating or prism) and one or more exit slits enabling the electronic measurement of emergent radiation to be made at selected wavelengths within the spectral range.

2.4 Detection and measurement system. In a spectrometer, the dispersed radiant energies of selected wavelengths pass through exit slits and are detected by photomultiplier tubes. Electrical outputs of the tubes corresponding to the selected wavelengths are measured, relative to those of the internal standard wavelengths if used. These intensity measurements are converted to concentrations by reference to analytical curves prepared from appropriate reference materials excited under the same conditions.

3 OPTICAL ALIGNMENT.

3.1 General. Misalignment of the optical system can lead to such problems as spectral interferences, high background equivalent concentrations (BEC) and poor repeatability of the equipment (see Clause 5.1). Tests can be carried out to verify the optical alignment, most of which should be performed by the manufacturer or under his instruction. However, profile checking of each programmed element may be carried out by the laboratory operator in accordance with Clause 3.2. The technique used differs for individual spectrometers and should be in accordance with the manufacturer's recommendations.

3.2 Profiling. Most atomic emission spectrometers are equipped with a profile control which enables lateral displacement of the entrance slit image to achieve precise alignment of spectral lines at their respective exit slits.

It is realized that developments in spectrometer design may not enable the procedure in Clause 3.2.1 to be adopted. Nevertheless, it must be ensured that any spectral line is on profile before analytical measurement is carried out.

3.2.1 *Profiling of each spectral line.* Profile checking of each spectral line shall be carried out using a sample of suitable matrix (e.g. copper, brass, steel, aluminium) containing an appropriate concentration of the element being checked. Commonly, 1 to 2 percent concentration of the particular element in its matrix is adequate. However, for some elements a much lower concentration is necessary to prevent alignment error caused by self absorption.

Assessment of the tabulated profile settings permits selection of a single setting (nominal profile position) which allows near-optimal response on all channels simultaneously. The limit within which the individual line may deviate from this nominated profile position will be included in standards for the atomic emission spectrometric analysis of specific metals. If these cannot be achieved, it may indicate the need for some optical re-alignment.

3.2.2 *Profile deviation.* The difference between the nominated profile position and profile setting is expressed in nanometres. This is calculated by multiplying the lateral displacement of the slit by the linear reciprocal dispersion of the spectrometer.

3.2.3 *Routine profiling of the spectrometer.* For routine spectrometer profiling, a particular spectral line shall be selected which, at the nominated profile position, produces maximum radiation at the exit slit. If the spectrometer is in a stable condition the profile positions of other lines relative to the reference line will remain fixed, and correction for drift in the profile position of this line will also correct for profile drift on the other lines. This reference line and an appropriate sample shall be used for all future routine profiling of the spectrometer. Frequency of profiling will depend on the long-term stability of equipment.

4 CALIBRATION AND STANDARDIZATION OF A SPECTROMETER.

4.1 Instrument settings. Preliminary curves shall be derived for each element in a spectrometer program by relating intensity measurement to the concentration of