

Australian Standard™

**Recommended practice for chemical
analysis by atomic absorption
spectrometry**

**Part 1: Flame atomic absorption
spectrometry**

This Australian Standard was prepared by Committee CH/16, Spectroscopy. It was approved on behalf of the Council of Standards Australia on 29 October 1999 and published on 5 December 1999.

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Australian Chamber of Commerce and Industry
CSIRO Energy Technology
CSIRO Manufacturing Science and Technology
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STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 2134.1—1999

Recommended practice for chemical analysis by atomic absorption spectrometry

Part 1: Flame atomic absorption spectrometry

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NOTES

Australian Standard™

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PREFACE

This Standard was prepared by the Standards Australia Committee CH/16, Spectroscopy, to supersede AS 2134.1—1988.

This Standard will be Part 1 of a series comprising:

AS

2134 Recommended practice for chemical analysis by atomic absorption spectrometry

2134.1 Part 1: Flame atomic absorption spectrometry

2134.2 Part 2: Graphite furnace atomic absorption spectrometry

2134.3 Part 3: Vapour generation atomic absorption spectrometry

The objective of this Standard is to set out recommended practices for the operation of flame atomic absorption spectrometers used in chemical analysis. The Standard includes a description of instrumentation, optimization procedures, calibration, test procedures and information on factors affecting atomic absorption. A section on analytical quality assurance has been added to the Standard.

CONTENTS

	<i>Page</i>
SECTION 1 SCOPE AND GENERAL	
1.1 SCOPE	4
1.2 PRINCIPLE	4
1.3 REFERENCED DOCUMENTS	4
1.4 DEFINITIONS	5
SECTION 2 INSTRUMENTATION	
2.1 GENERAL	6
2.2 SOURCES OF RADIATION	6
2.3 NEBULIZER-SPRAY CHAMBER SYSTEM	7
2.4 BURNERS	8
2.5 SAFETY PRECAUTIONS	8
2.6 FUEL-OXIDANT CONTROL SYSTEM	9
2.7 MONOCHROMATOR	10
2.8 MEASUREMENT SYSTEM	10
SECTION 3 INSTRUMENT OPTIMIZATION	
3.1 GENERAL	12
3.2 FACTORS AFFECTING ATOMIC ABSORPTION	12
3.3 MATRIX AND IONIZATION EFFECTS	12
3.4 OPTIMUM INSTRUMENT SETTINGS TO MINIMIZE INTERFERENCE .	12
3.5 SPECTRAL INTERFERENCE	12
3.6 SETTING UP PROCEDURE	13
SECTION 4 CALIBRATION AND ANALYSIS	
4.1 CONTAMINATION AND REAGENT PURITY	15
4.2 CERTIFIED REFERENCE MATERIALS	15
4.3 CALIBRATION AND TEST SOLUTIONS AND CALIBRATION GRAPHS	15
4.4 CALIBRATION AND TEST PROCEDURE	16
4.5 ACCEPTANCE OF RESULTS	21
SECTION 5 ANALYTICAL QUALITY ASSURANCE	
5.1 GENERAL	23
5.2 INSTRUMENT CALIBRATION AND STANDARDIZATION	23
5.3 VALIDATION OF ANALYTICAL PROCEDURE	23
5.4 TREATMENT OF DATA FROM MONITORING PROCEDURE	24
5.5 TRACEABILITY OF RESULTS	24
5.6 CORRECTIVE ACTION	24
APPENDICES	
A FLOWSHEET ON THE PROCEDURE FOR THE ACCEPTANCE OF ANALYTICAL VALUES FOR TEST SAMPLES	25
B GLOSSARY OF TERMS USED IN FLAME ATOMIC ABSORPTION SPECTROSCOPY	26

STANDARDS AUSTRALIA

Australian Standard

Recommended practice for chemical analysis by atomic absorption spectrometry**Part 1: Flame atomic absorption spectrometry**

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE This Standard sets out recommendations for instrumentation and operating techniques suitable for chemical analysis by flame atomic absorption spectrometry (FAAS) and includes a summary of testing procedures and recommendations for safe operation.

NOTES:

- 1 Graphite furnace atomization and vapour generation techniques are dealt with in Parts 2 and 3 of the AS 2134 series of Standards.
- 2 This Standard should be read in conjunction with the instrument manufacturer's recommendations.
- 3 A flowsheet on the procedure for the acceptance of analytical values for test samples is given in Appendix A.

1.2 PRINCIPLE Flame atomic absorption spectrometry (FAAS) relies upon—

- (a) heating a sample sufficiently to produce free atoms;
- (b) free atoms of an element being able to absorb energy only at certain discrete wavelengths (usually resonance wavelengths: refer Appendix B); and
- (c) the energy absorbed being a function of the concentration of the absorbing atoms.

The technique described in this Standard involves nebulizing a solution of the sample into a flame and measuring the absorption of energy at a specified wavelength. The apparatus is so designed that the absorption measurement is independent of any radiation emitted by the flame.

1.3 REFERENCED DOCUMENTS The documents below are referred to in this Standard:

AS

1674	Safety in welding and allied processes
1674.1	Part 1: Fire precautions
1940	The storage and handling of flammable and combustible liquids
2134	Recommended practice for chemical analysis by atomic absorption spectrometry
2134.2	Part 2: Graphite furnace spectrometry
2134.3	Part 3: Vapour generation atomic absorption spectrometry
2162	Verification and use of volumetric apparatus
2162.1	Part 1: General—Volumetric glassware

AS	
2508	Safe storage and handling information card
2508.2.001	Part 2.001: Acetylene (Dissolved) (Ethyne)
2850	Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
2929	Test methods—Guide to the format, style and content
BS	
5703	Guide to data analysis and quality control using cusum techniques (series)

1.4 DEFINITIONS For the purpose of this Standard the definitions in Appendix B apply.

SECTION 2 INSTRUMENTATION

2.1 GENERAL A flame atomic absorption spectrometer consists essentially of the following components:

- (a) A source of radiation which emits the required atomic spectrum of the analyte element (Clause 2.2).
- (b) A nebulizer-spray chamber system (Clause 2.3).
- (c) A burner (Clause 2.4)
- (d) A control system for the fuel-oxidant mixture (Clause 2.6)
- (e) A device for isolating essentially monochromatic radiant energy (Clause 2.7).
- (f) A suitable optical system (Clause 2.7).
- (g) A measurement system which usually consists of a detector, amplifier and a suitable display (Clause 2.8).

2.2 SOURCES OF RADIATION

2.2.1 Line sources—Hollow-cathode lamp

2.2.1.1 Principle of operation A hollow-cathode lamp has a cathode constructed of, or containing, the analyte element. It is generally operated to give a pulsed signal by electronic modulation of the lamp or mechanical chopping of the radiant beam. The amplifier used for measurement is designed to respond only to the frequency of the modulated signal, so that radiation emitted by the flame does not significantly alter the signal measured at any time during the analysis (including the period of nebulization of the sample).

2.2.1.2 Operating current In general, the hollow-cathode lamp should be operated according to manufacturer's recommendations. Lower currents may improve sensitivity but may also result in a decrease in signal-to-noise ratio. Higher currents increase the possibility of self absorption in the lamp which contributes to non-linearity of the calibration graph. In practice, the operating current should be chosen as that which provides the optimum signal-to-noise ratio.

2.2.1.3 Stability and testing The hollow-cathode lamp should be stable and should be periodically tested to ensure that the signal intensity and stability are maintained.

2.2.1.4 Multi-element type Combination (or multi-element) lamps are available so that a single lamp may serve as a convenient light source for two or more elements. The single-element lamps are to be preferred as the intensity of radiation of the line for a particular element will be the greater at a given operating current.

Combinations of elements which produce relatively intense spectral lines close to the wavelengths of the normally utilized resonance lines should be avoided. Such nearby lines may necessitate the selection of unusually narrow spectral bandwidths on the monochromator thus causing a deterioration of the signal-to-noise ratio and hence, precision.

2.2.2 Line sources—other lamps Other types of lamp may be used as alternatives provided that they meet or exceed the same criteria of stability, signal intensity and line width as hollow-cathode lamps. Electrodeless discharge lamps and boosted discharge hollow-cathode lamps, which are commercially produced, provide suitable light sources for some elements.

NOTE: Some modern hollow-cathode lamps have electronic coding to enable the instrument to recognize the elements.

2.2.3 Continuum sources Continuum light sources are used for background correction but should conform to adequate standards of intensity and stability. For correction in the ultraviolet region of the spectrum, deuterium lamps are commonly employed, while for the visible region of the spectrum, tungsten lamps or quartz-halogen lamps should be used (see also Clause 3.5).

2.3 NEBULIZER-SPRAY CHAMBER SYSTEM

2.3.1 General The sample is presented to the radiant energy as an atomic vapour after nebulization of a solution of the sample into the fuel-oxidant mixture and atomization in the flame.

The system consists of three essential parts, a nebulizer, spray chamber and drain system.

2.3.2 Nebulizer The most common type of nebulizer uses oxidant (air or nitrous oxide) under pressure to aspirate the liquid from a container through a capillary by the venturi effect. Many nebulizers are adjustable to allow variation of the rate of liquid aspiration, which may be useful when organic solvents are used. The nebulizer uptake rate may be adjusted to provide maximum sensitivity, maximum signal-to-noise ratio or minimal chemical interference effects depending on the requirements of the test method.

NOTE: Nebulizers are susceptible to wear and corrosion and their performance should be checked periodically.

2.3.3 Spray chamber To increase the number of fine droplets, the spray produced by a nebulizer may impinge on an impact bead, the position of which may be adjustable. The aerosol then enters a spray chamber where larger drops are rejected. The spray chamber may contain baffles or paddles to assist in this drop-removal process and to ensure homogeneous mixing of the aerosol with fuel gas, which is introduced at this point. The mixture of fine droplets, oxidant and fuel is carried into the flame.

2.3.4 Drain system It is essential that the drain system operates smoothly and without risk of blockage or formation of gas bubbles.

Where organic solvents are used, a non-flammable, solvent resistant container should be used to store waste liquid from the drain system. In most instruments, the drain from the spray chamber ends in a liquid trap. This trap provides for an effective head of liquid (approximately 50 mm) for safe and efficient operation of the system, and should be so designed that it is resistant to organic solvents and is free from blockages caused by salts or airlocks.

WARNINGS:

- 1 DANGER OF EXPLOSION. ALWAYS FILL THE LIQUID TRAP WITH THE SOLVENT BEFORE THE FLAME IS IGNITED.**
- 2 FOR SAFETY REASONS, IT IS ESSENTIAL THAT LARGE VOLUMES OF ORGANIC SOLVENT ARE NOT ALLOWED TO ACCUMULATE IN THE DRAIN SYSTEM (SEE ALSO AS 1940).**

2.3.5 System design The system is required to—

- produce a steady rate of spraying, substantially independent of the height of sample liquid relative to that of the nebulizer;
- produce an aerosol containing an adequate population of appropriate droplet size;
- maintain a constant flow of aerosol to the burner;
- have a minimal tendency to block;
- be easy to clean;
- resist corrosion by reagents and conditions typically used in normal operation;

- (g) provide minimal contamination from one solution to the next, i.e. it does not retain significant quantities of residual solution;
- (h) achieve maximum nebulization efficiency with minimal gas consumption consistent with safety from flashback (explosion of the combustible mixture of gases in the spray chamber); and
- (i) have a pressure release mechanism, situated to the rear or side of the spray chamber, in case of flashback.

2.4 BURNERS

2.4.1 General Various flame temperatures and conditions are necessary to atomize different elements. In general, the lowest temperature necessary to cause atomization consistent with freedom from interferences within the flame is preferred. Different flame types and temperatures are achieved by using different gas mixtures, such as air/acetylene and nitrous oxide/acetylene, with appropriate burner heads. The proportions of fuel and oxidant may be varied to achieve oxidizing, stoichiometric, or reducing flames.

2.4.2 Inscription Each burner head should be permanently inscribed to indicate the gas mixtures for which it may be used. The manufacturer should be consulted before attempting to use a burner for other than intended gas mixtures.

2.4.3 Construction Burners should be constructed to provide for—

- (a) a steady and reproducible flame (the aerosol should be free from turbulence where it enters the flame);
- (b) corrosion resistance;
- (c) minimum carbon build-up and salt encrustation; and
- (d) safety interlocks on each type of burner to prevent misuse with the wrong gas mixture.

In addition, the burner mounting should be constructed for precise horizontal, vertical and rotational adjustment of the position of the burner in relation to the path of the light beam.

2.5 SAFETY PRECAUTIONS

2.5.1 Deposition of salts Deposition of salts in the burner slot will cause an uneven flame unsuitable for accurate analysis, and may cause an explosion due to flashback. The flame profile and burner slot should be carefully inspected and the burner slot carefully cleaned at regular intervals to ensure that salts do not build up. Do not attempt to remove salt deposition while the burner is operating.

2.5.2 Perchloric acid and perchlorates Particular care should be taken when aspirating solutions containing perchloric acid or metal perchlorates into a nitrous oxide/acetylene flame. Some metal perchlorates are unstable and may lead to detonation and flashback. In general, the perchloric acid concentration should be kept to a minimum and the solution should be aspirated for the shortest time consistent with a satisfactory measurement being made. Water should be aspirated between solutions and at the completion of analysis to minimize salt build-up.

2.5.3 Silver salts Particular care should be taken when aspirating high concentrations of silver salts into an acetylene-fuelled flame. Silver acetylides may be deposited and water cause an explosion. Similar problems may occur with high concentrations of copper and mercury salts. Water should be aspirated between solutions and at the completion of analysis to minimize salt build-up.