AS 3641.2

REFERENCE COP nder Revision see DR99005 STANDARDS AUSTRALIA

AS 3641.2-1990

SUPERSEDED BY: AS 3641.2-1999

Australian Standard®

Recommended practice for atomic emission spectrometric analysis

Part 2: Inductively coupled plasma excitation



This Australian Standard was prepared by Committee CH/16, Spectroscopy. It was approved on behalf of the Council of Standards Australia on 26 June 1990 and published on 12 November 1990.

The following interests are represented on Committee CH/16:

Bureau of Steel Manufacturers

Confederation of Australian Industry

CSIRO, Division of Fuel Technology

CSIRO, Division of Mineral Chemistry

Department of Defence

Government Chemical Laboratories, Qld

National Association of Testing Authorities, Australia

Additional interests participating in preparation of Standard:

Aluminium smelters

Rural Water Commission

State Electricity Commission, Vic.

Steel producers

Review of Australian Standards. To keep abreast of progress in industry, Australian Standards are subject to periodic review and are kept up-to-date by the issue of amendments or new editions as necessary. It is important therefore that Standards users ensure that they are in possession of the latest edition, and any amendments thereto.

Full details of all Australian Standards and related publications will be found in the Standards Australia Catalogue of Publications; this information is supplemented each month by the magazine 'The Australian Standard', which subscribing members receive, and which gives details of new publications, new editions and amendments, and of withdrawn Standards.

Suggestions for improvements to Australian Standards, addressed to the head office of Standards Australia, are welcomed. Notification of any inaccuracy or ambiguity found in an Australian Standard should be made without delay in order that the matter may be investigated and appropriate action taken.

This Standard was issued in draft form for comment as DR 89192.

AS 3641.2-1990

Australian Standard®

Recommended practice for atomic emission spectrometric analysis

Part 2: Inductively coupled plasma excitation

First published as AS 3641.2-1990.

PUBLISHED BY STANDARDS AUSTRALIA (STANDARDS ASSOCIATION OF AUSTRALIA) STANDARDS HOUSE, 80 ARTHUR ST, NORTH SYDNEY NSW

ISBN 0 7262 6460-1

PREFACE

This Standard was prepared by the Standards Australia Committee on Spectroscopy under the direction of the Chemical Standards Board.

This Standard is Part 2 of the AS 3641 series on recommended practice for atomic emission spectrometry and describes recommended procedures for the setting up and operation of inductively coupled plasma (ICP) instruments for use in chemical methods of analysis. The recommendations in this Standard are intended to apply to Australian Standard methods for ICP analysis. In the preparation of this Standard cognizance was taken of the following documents:

International Organization for Standardization, ISO/TC 147/SC 2/WG 32 N2, Miles, Douglas L. and Sharp, Barry L., *Inductively coupled plasma spectrometry*.

American Society for Testing and Materials, ASTM E 743, *Standard guide for spectrochemical laboratory quality assurance*, Philadelphia, USA, 1985.

Report by the Analytical Methods Committee. Evaluation of analytical instrumentation, Part III Polychromators for use in emission spectrometry with ICP sources, *Royal Society of Chemistry, Analytical Proceedings.* April, 1986, Vol. 23.

SARLIT, M.L. Verification of sequential ICP-AES instrument function. International Organization for Legal Metrology, September 1987.

© Copyright — STANDARDS AUSTRALIA

Users of Standards are reminded that copyright subsists in all Standards Australia publications and software. Except where the Copyright Act allows and except where provided for below no publications or software produced by Standards Australia may be reproduced, stored in a retrieval system in any form or transmitted by any means without prior permission in writing from Standards Australia. Permission may be conditional on an appropriate royalty payment. Requests for permission and information on commercial software royalties should be directed to the Head Office of Standards Australia.

Standards Australia will permit up to 10 percent of the technical content pages of a Standard to be copied for use exclusively in-house by purchasers of the Standard without payment of a royalty or advice to Standards Australia.

Standards Australia will also permit the inclusion of its copyright material in computer software programs for no royalty payment provided such programs are used exclusively in-house by the creators of the programs.

Care should be taken to ensure that material used is from the current edition of the Standard and that it is updated whenever the Standard is amended or revised. The number and date of the Standard should therefore be clearly identified.

The use of material in print form or in computer software programs to be used commercially, with or without payment, or in commercial contracts is subject to the payment of a royalty. This policy may be varied by Standards Australia at any time.

÷

.

.

.

CONTENTS

	•		,					·			Page
SECT	TION 1 SCOPE AND GENERAL										
1.1	SCOPE										4
1.2				••••			••••				4
1.3	DEFINITIONS				••••						4
1.4											4
1.5											4
SECT	TION 2 INSTRUMENTATION										
_										•	•
2.1					••••	••••	••••	••••	••••	••••	6
2.2								•••••	••••	••••	6
2.3								••••	••••	••••	6
2.4								••••	••••	••••	6
2.5							••••	••••	••••	••••	8
2.6		•						••••	••••	••••	8
2.7								••••	••••	••••	8
2.8									••••	••••	8
2.9	ADDITIONAL EQUIPMENT	••••	••••	••••	••••	••••	••••	••••	••••	••••	9
SECT	ION 3 INSTRUMENT OPTIMIZ	ATIO	N								
3.1	GENERAL										10
3.2										••••	10
3.3											10
5.5	SEQUENTIAL SPECTROMETE								10K		10
3.4	-					••••			••••		11
3.5						••••	••••			••••	11
3.6					••••	••••		••••		••••	12
					••••	••••	••••	••••	••••	••••	12
	ION 4 INSTRUMENT PERFORM										
4.1	GENERAL								••••	••••	13
4.2	FREQUENCY OF TESTING								••••	••••	13
4.3	REPEATABILITY OF THE INS							••••	••••	••••	13
4.4							••••	••••	••••	••••	14
4.5	TESTS ON MEASURING CIRC							••••	••••	••••	14
4.6	BACKGROUND EQUIVALENT							••••	••••	••••	14
4.7	LIMIT OF DETECTION	••••	••••	••••	••••	••••	••••	••••	••••	••••	15
SECT	ION 5 INSTRUMENT OPERATI	ON	•								
5.1	INTRODUCTION										16
							••••			••••	16
5.2				••••			••••	••••	••••		
5.4	CALIBRATION OF SPECTROM STANDARDIZATION			····		••••	••••	••••	••••	••••	17 17
						••••	••••	••••	••••	••••	
5.5							••••				19
	ACCEPTANCE OF RESULTS				••••	····	••••	••••	••••	••••	19
	ION 6 ANALYTICAL QUALITY			NCE							
6.1	GENERAL				••••						21
6.2	INSTRUMENT CALIBRATION	AND	STA	NDA	RDIZ	ATIC)N	••••	••••	••••	21
6.3	VALIDATION OF ANALYTICAI	. PRC	OCEI	OURE	••••	••••				••••	21
6.4	TREATMENT OF DATA FROM		VITC	RINC	G PRC	CED	URE			••••	21
6.5	TRACEABILITY OF RESULTS			••••							21
6.6	CORRECTIVE ACTION	••••									22
	NDICES										
											••
											23
B	FLOWSHEET ON THE PROCEDU	JRE E	TOR	THE	ACCE	PIA	NCE	OF A	NAL	Y 11-	24
	CAL VALUES FOR TEST SAMPL	. د.a			•• •••						24

STANDARDS AUSTRALIA

Australian Standard

Recommended practice for atomic emission spectrometric analysis

Part 2: Inductively coupled plasma excitation

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE This Standard sets out recommendations for instrumentation and operating procedures in the application of the inductively coupled plasma excitation to chemical analysis by atomic emission spectrometry.

NOTE: This Standard is intended to be read in conjunction with the instrument manufacturer's recommendations.

1.2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

2243 Safety in laboratories

2243.2 Part 2: Chemical aspects

2243.5 Part 5: Non-ionizing radiations

2772 Radiofrequency radiation

2772.2 Principles and methods of measurement—300 kHz to 100 GHz

2883 Analysis of metals—Procedures for the setting up, calibration and standardization of atomic emission spectrometers using arc/spark discharge

2929 Test methods—Guide to the format, style and content

3641 Recommended practice for atomic emission spectrometric analysis

3641.1 Part 1: Principles and techniques

ASTM

E 135 Terminology relating to emission spectroscopy

BS

5703 Guide to data analysis and quality control using cusum techniques

1.3 DEFINITIONS For the purpose of this Standard, the definitions in AS 2883 and ASTM E 135 apply.

1.4 PRINCIPLE OF THE TECHNIQUE When atoms or ions of elements are excited in an inductively coupled plasma (see Figure 1), they emit electromagnetic radiation in the form of spectra. The characteristic spectrum of each element, which is determined primarily by the electronic configuration of the atom, consists of a number of wavelengths which correspond to the emission of electromagnetic radiation arising from the transition of electrons in various excited states to lower energy states. The intensity of the electromagnetic radiation which is emitted at a characteristic wavelength is proportional to the concentration of the element in the sample. The intensity is measured using an appropriate spectrometer and photo-detecting system.

1.5 SAFETY PRECAUTIONS

1.5.1 General The general safety precautions listed in AS 2243 should be followed to minimize safety hazards.

1.5.2 Ultraviolet radiation Most ICP instruments contain a safety interlocking system which prevents ignition of the source when the ultraviolet-absorbing shield is not in position. It is essential that the interlock is not overridden.

1.5.3 Fume hoods It is strongly recommended that an efficient mechanical ventilation system be installed to exhaust toxic fumes and ozone issuing from the torch. It should be heat and corrosion resistant. The intake of the exhaust system should be placed directly above the torch. The position of the intake and the exhaust capacity of the system should be in accordance with the manufacturer's specification, as an incorrect position can cause torch instability. It is also recommended that the exhaust from the RF generator be vented away from the instrument, i.e. through the fume hood.