

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 29 October 1999 and published on 5 December 1999.

The following interests are represented on Committee CH/16:

Australian Chamber of Commerce and Industry
CSIRO Energy Technology
CSIRO Manufacturing Science and Technology
Department of Defence (Australia)
Department of Natural Resources, Qld
National Association of Testing Authorities, Australia
Queensland Health Scientific Services

Keeping Standards up-to-date

Standards are living documents which reflect progress in science, technology and systems. To maintain their currency, all Standards are periodically reviewed, and new editions are published. Between editions, amendments may be issued. Standards may also be withdrawn. It is important that readers assure themselves they are using a current Standard, which should include any amendments which may have been published since the Standard was purchased.

Detailed information about Standards can be found by visiting the Standards Australia web site at www.standards.com.au and looking up the relevant Standard in the on-line catalogue.

Alternatively, the printed Catalogue provides information current at 1 January each year, and the monthly magazine, *The Australian Standard*, has a full listing of revisions and amendments published each month.

We also welcome suggestions for the improvement in our Standards, and especially encourage readers to notify us immediately of any apparent inaccuracies or ambiguities. Contact us via email at mail@standards.com.au, or write to the Chief Executive, Standards Australia International Ltd, PO Box 1055, Strathfield, NSW 2135.

This Standard was issued in draft form for comment as DR 99005.

STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 3641.2—1999

**Recommended practice for atomic emission spectrometric analysis
Part 2: Inductively coupled plasma excitation**

RECONFIRMATION NOTICE

Technical Committee CH-016 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

Certain documents referenced in the publication may have been amended since the original date of publication. Users are advised to ensure that they are using the latest versions of such documents as appropriate, unless advised otherwise in this Reconfirmation Notice.

Approved for reconfirmation in accordance with Standards Australia procedures for reconfirmation on 5 October 2016.

The following are represented on Technical Committee CH-016:

Australian Institute of Physics
Environmental Laboratory Industry Group
National Association of Testing Authorities Australia
National Measurement Institute

NOTES

Australian Standard™

Recommended practice for atomic emission spectrometric analysis

Part 2: Inductively coupled plasma excitation

Originated as AS 3641.2—1990.
Second edition 1999.

COPYRIGHT

© Standards Australia International

All rights are reserved. No part of this work may be reproduced or copied in any form or by any means, electronic or mechanical, including photocopying, without the written permission of the publisher.

Published by Standards Australia International Ltd
PO Box 1055, Strathfield, NSW 2135, Australia
ISBN 0 7337 3070 1

PREFACE

This Standard was prepared by the Standards Australia Committee CH/16, Spectroscopy to supersede AS 3641.2—1990.

The objective of this Standard is to describe recommended procedures for the setting up and operation of inductively coupled plasma (ICP) instruments. The recommendations in this Standard are intended to apply to Australian Standard methods for ICP analysis.

This Standard is part of a series as follows:

AS

3641 Recommended practice for atomic emission spectrometric analysis

3641.1 Part 1: Principles and techniques

3641.2 Part 2: Inductively coupled plasma excitation

CONTENTS

	<i>Page</i>
SECTION 1 SCOPE AND GENERAL	
1.1 SCOPE	5
1.2 REFERENCED DOCUMENTS	5
1.3 DEFINITIONS	5
1.4 PRINCIPLE OF THE TECHNIQUE	6
1.5 SAFETY PRECAUTIONS	6
SECTION 2 INSTRUMENTATION	
2.1 PRINCIPLE OF OPERATION	8
2.2 GENERAL	8
2.3 TORCH AND POWER SUPPLY	8
2.4 SAMPLE INTRODUCTION SYSTEM	9
2.5 GAS CONTROL SYSTEM	10
2.6 OPTICAL SYSTEM	11
2.7 DETECTION AND MEASUREMENT SYSTEM	11
2.8 DATA PROCESSING AND COMPUTER CONTROL	11
2.9 ADDITIONAL EQUIPMENT	12
SECTION 3 INSTRUMENT OPTIMIZATION	
3.1 GENERAL	13
3.2 PROFILING OF A SIMULTANEOUS SPECTROMETER	13
3.3 WAVELENGTH CALIBRATION OF A SEQUENTIAL SPECTROMETER ..	13
3.4 OPTICAL ALIGNMENT OF PLASMA	14
3.5 PLASMA OPTIMIZATION	14
3.6 INTERFERENCES	15
SECTION 4 INSTRUMENT PERFORMANCE TESTS	
4.1 GENERAL	17
4.2 FREQUENCY OF TESTING	17
4.3 REPEATABILITY OF THE INSTRUMENT	17
4.4 REPEATABILITY OF THE MEASURING SYSTEM	18
4.5 TESTS ON MEASURING CIRCUITS	19
4.6 BACKGROUND EQUIVALENT CONCENTRATION	19
4.7 LIMIT OF DETECTION	20
SECTION 5 INSTRUMENT OPERATION	
5.1 INTRODUCTION	21
5.2 METHOD DEVELOPMENT	21
5.3 CALIBRATION OF SPECTROMETER	23
5.4 STANDARDIZATION	23
5.5 ANALYSIS PROCEDURE	24
5.6 ACCEPTANCE OF RESULTS	26

SECTION 6 ANALYTICAL QUALITY ASSURANCE

6.1	GENERAL	28
6.2	INSTRUMENT CALIBRATION AND STANDARDIZATION	28
6.3	VALIDATION OF ANALYTICAL PROCEDURE	28
6.4	TREATMENT OF DATA FROM MONITORING PROCEDURE	29
6.5	TRACEABILITY OF RESULTS	29
6.6	CORRECTIVE ACTION	29

APPENDICES

A	TROUBLESHOOTING PROCEDURES	30
B	FLOWSHEET ON THE PROCEDURE FOR THE ACCEPTANCE OF ANALYTICAL VALUES FOR TEST SAMPLES	32

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 (ICP) excitation to chemical analysis by atomic emission spectrometry.

NOTE: This Standard is intended to be read in conjunction with AS 3641.1 and the instrument manufacturer's recommendations.

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

AS

2162 Verification and use of volumetric apparatus

2162.1 Part 1: General—Volumetric glassware

2243 Safety in laboratories

2243.5 Part 5: Non-ionizing radiations

2772 Radiofrequency radiation

2772.2 Part 2: Principles and methods of measurement—300 kHz to 100 GHz

2850 Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct

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

AS/NZS

2243 Safety in laboratories

2243.2 Part 2: Chemical aspects

ASTM

E 135 Terminology relating to analytical chemistry for metals, ores and related materials

BS

5703 Guide to data analysis and quality control using cusum techniques (series)

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/NZS 2243.2 and AS/NZS 2243.5 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.

1.5.4 Radiofrequency leakage For safety reasons, it is recommended that the ICP torch compartment be checked for radiofrequency (RF) radiation leakage by a recognized testing authority. Radiofrequency leakage tests should be performed when considered necessary; however they should always be carried out on commissioning the instrument and when a modification to the torch compartment has been carried out. Recommended exposure limits to non-ionizing radiation are given in AS 2243.5 and methods of measurement are given in AS 2772.2.

WARNING: RADIOFREQUENCY RADIATION HAS THE CAPABILITY OF HEATING BIOLOGICAL TISSUE. THERMAL DAMAGE MAY OCCUR IF THE INTENSITY OF THE RADIATION IS SUFFICIENTLY HIGH.

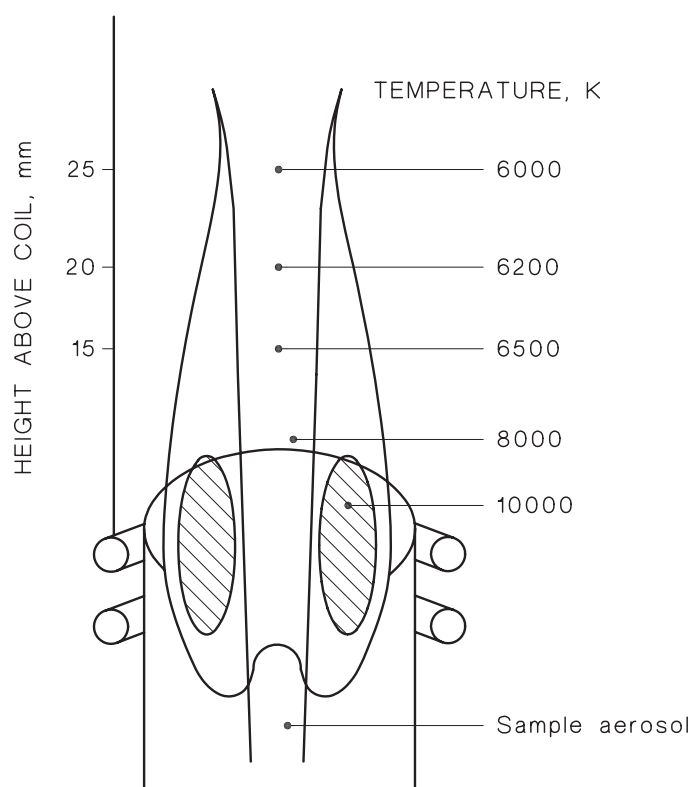


FIGURE 1 SCHEMATIC REPRESENTATION OF A TYPICAL ICP

SECTION 2 INSTRUMENTATION

2.1 PRINCIPLE OF OPERATION A plasma is a gas in which there is a significant population of ions and free electrons. An inductively coupled plasma is formed by the interaction of the ions with a time-dependent magnetic field which is produced by a high-frequency generator connected to an induction coil.

When a gas, typically argon, flows through a quartz tube located centrally within the induction coil, the high frequency currents flowing in the induction coil generate oscillating magnetic fields whose lines of force are axially oriented inside the quartz tube. These lines of force follow elliptical closed paths outside the coil and axial magnetic fields induce seed electrons and ions to flow in closed annular paths inside the quartz tube space.

Since the induced magnetic fields are time-dependent in their direction and strength, the electrons are accelerated on each half cycle where they meet resistance to their flow. This causes heating of the gas, resulting in additional ionization. The consequence of this is the almost instantaneous formation of a steady-state plasma having a gas temperature of between 9 000 K and 10 000 K in the region of maximum eddy current flow. Equilibrium is reached when the energy loss is equal to the power input. The plasma is thermally isolated from the quartz tube by the introduction of argon in a tangential stream and the incorporation of an inner concentric tube. With this arrangement, the plasma is anchored near the exit end of the concentric tube. A central injection tube allows the sample aerosol, produced by an appropriate nebulization system, to be directed into the central core of the plasma (see Figure 1).

Sample aerosol droplets entering the plasma are desolvated, evaporated and the constituents excited. The specific radiant energies emitted by the sample atoms and ions are detected and measured using similar optical dispersion devices to those used in other forms of emission spectrometry. Because of the high temperature existing in the plasma, most elements are highly ionized and hence the most sensitive characteristic emission lines are usually those originating from ionized species.

2.2 GENERAL Instrumentation for inductively coupled plasma atomic emission spectrometry consists of the following components:

- (a) A torch and associated radiofrequency generator.
- (b) A sample introduction system.
- (c) A suitable optical system which may allow either axial or radial viewing or both.
- (d) A measurement system incorporating a detector, amplifier and suitable display.

2.3 TORCH AND POWER SUPPLY The torch consists, generally, of three concentric quartz tubes (see Figure 2). The outer tube into which a tangential flow of argon is introduced, thermally isolates the plasma. This gas flow is called the coolant gas. The second innermost tube carries the auxiliary gas. This has the effect of regulating the position of the plasma at the exit relative to the injection tube which directs the aerosol into the central core of the plasma. For use with samples containing hydrofluoric acid, the injection tube may be replaced by one made of a more corrosion-resistant material.

Gas flow rates vary depending on the size and configuration of the torch, but are generally as follows:

- (a) Plasma (Coolant) gas—8 L/min to 15 L/min.
- (b) Auxiliary gas—0.5 L/min to 1.0 L/min.
- (c) Carrier gas—0.5 L/min to 2.0 L/min.

The radiofrequency generator may be either crystal-controlled or free-running. ICP spectrometers operate at discrete frequencies which can range from 27.12 MHz up to 50 MHz. The power output can be up to 3 kW. Power is coupled to the torch via the induction coil, which is usually water-cooled. With a crystal controlled generator, an impedance-matching network, commonly called the matchbox, is used to match the load (i.e. the plasma) to the power input, using a tuning capacitor to minimize the reflected power. This tuning is facilitated either by manual adjustment or automatically by a sensing circuit. With a free-running generator, the induction coil is directly coupled and is part of the tuner circuit. Changes in plasma impedance are compensated by variations in the frequency.

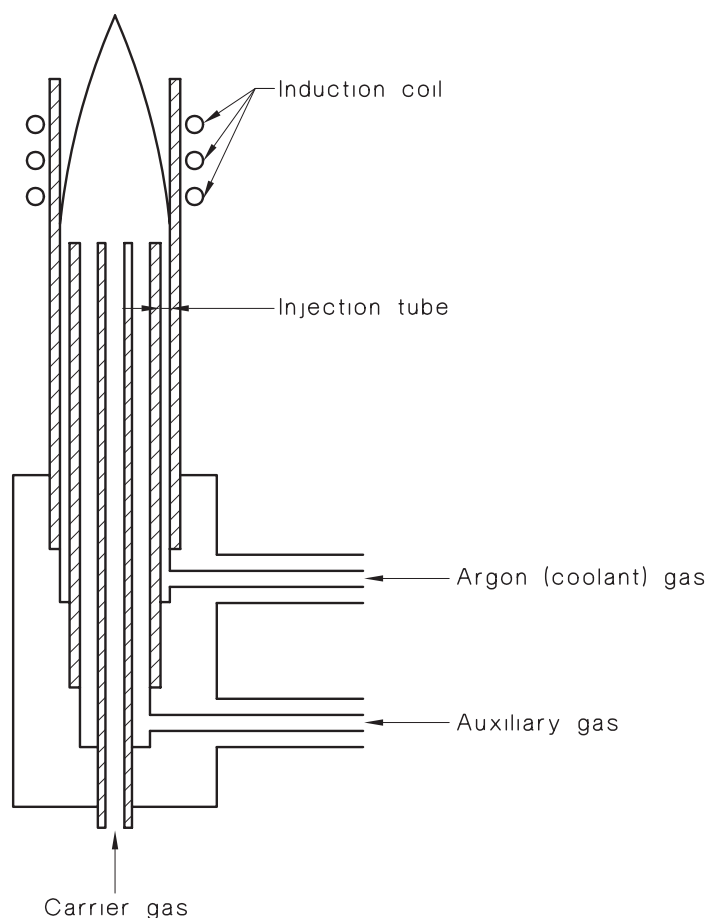


FIGURE 2 THE INDUCTIVELY COUPLED ARGON PLASMA TORCH ASSEMBLY

2.4 SAMPLE INTRODUCTION SYSTEM

2.4.1 Nebulization system The aerosol is produced by a nebulization system consisting of a nebulizer and spray chamber. The nebulizer produces droplets having a wide range of diameters which pass into a spray chamber where the larger droplets are removed by impingement on the spray chamber walls. The spray chamber therefore acts as a filtering device, producing drops of narrow size distribution and of small median diameter. The efficiency of the nebulization system ranges from about 0.2% to 1%.