AS 3719.1—1989
Reconfirmed 2016

Australian Standard®

Aluminium and aluminium alloys

Part 1: Determination of iron—Spectrophotometric method

This Australian Standard was prepared by committee CH/10, Analysis of Metals. It was approved on behalf of the Council of Standards Australia on 28 April 1989 and published on 13 October 1989.

The following interests are represented on Committee CH/10:

- Aluminium Development Council
- Australian Lead Development Association
- Bureau of Steel Manufacturers of Australia
- Confederation of Australia Industry
- Copper Technical Data Centre
- National Association of Testing Authorities
- Railways of Australia Committee
- Royal Australia Chemical Institute

This Standard was issued in draft form for comment as DR 88125.

STANDARDS AUSTRALIA

RECONFIRMATION

OF
AS 3719.1—1989
Aluminium and aluminium alloys
Part 1: Determination of iron—Spectrophotometric method

RECONFIRMATION NOTICE

Technical Committee CH-010 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.

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Australian Aluminium Council
Bureau of Steel Manufacturers of Australia
International Copper Association Australia
International Precious Metals Institute
National Association of Testing Authorities Australia

NOTES

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PREFACE

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

This Standard is technically equivalent to ISO 793—1973, *Aluminium and aluminium alloys—Determination of iron—Orthopenanthroline photometric method.*

It was prepared as part of a series of Standards covering the analysis of aluminium and aluminium alloys. An inter-laboratory test program to obtain information on the repeatability and reproducibility of the method was organized. The following laboratories participated in the test program to provide the data given in Table 2:

Alcoa of Australia Ltd

Boyne Smelters Pty Ltd

Comalco (Bell Bay) Pty Ltd

Department of Defence—Materials Testing Laboratories

Sims Metal Ltd

Tomago Aluminium Co Pty Ltd

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STANDARDS AUSTRALIA

Australian Standard
Aluminium and aluminium alloys

Part 1: Determination of iron content—Spectrophotometric method

1 SCOPE. This Standard sets out a spectrophotometric method for the determination of iron in aluminium and aluminium alloys. This method is applicable to the determination of iron contents between 0.05% and 2.50% (*m/m*) in aluminium and aluminium alloys. It does not apply completely to the following special cases, for which it should be modified as described in Appendix A or Appendix B:

(a) Unalloyed aluminium, aluminium-silicon alloys and any other aluminium alloy that is not easily attacked with hydrochloric acid (see Appendix A).

(b) Alloys containing a copper content greater than 5%, zinc content greater than 4%, nickel content greater than 2% or alloys with a proportional combination of these elements greater than 5% total (see Appendix B).

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

AS	
2162	Code of practice for the use of volumetric glassware
2164	One-mark volumetric flasks
2166	One—mark pipettes
2612	Aluminium and aluminium alloys—Sampling and preparation of solid samples for optical emission spectrometry
2850	Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
CK 19	Code of recommended practice for the chemical analysis of materials by ultraviolet/visible spectrophotometry

3 PRINCIPLE. The sample is dissolved in hydrochloric acid and the iron(III) present is reduced to iron(II) with hydroxylammonium chloride. The solution is buffered to pH 3.5 to 4.5 and the tris(1,10-phenanthroline) iron(II) complex is formed on the addition of 1, 10-phenanthroline. This orange-red coloured complex is determined spectrophotometrically at a wavelength of about 510 nm.

4 REAGENTS.

4.1 General requirements. During the analysis, only reagents of recognized analytical grade and distilled water or water of an equivalent purity shall be used. All solutions shall be freshly prepared and, where necessary, filtered.

4.2 Solutions.

4.2.1 *Hydrochloric acid* (ρ_{20}*1.16 g/mL)*

4.2.2 *Hydrochloric acid (1+1).* Add 500 mL of hydrochloric acid (4.2.1) to 500 mL of water.

4.2.3 *Sodium hydroxide solution (200 g/L).* Dissolve 200 g of sodium hydroxide in 400 mL of water. After cooling, transfer to a 1 L volumetric flask and dilute to volume with water. Store the solution in a plastics container.

4.2.4 *Mixed reagent.* Prepare the reagent by mixing the following solutions in the ratio of 1:1:3 by volume:

(a) *Solution A—hydroxylammonium chloride.* Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and dilute to 1 L.

(b) *Solution B—1,10-phenanthroline.* Dissolve 2.5 g of 1,10—phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$) or 3 g of 1,10-phenanthroline hydrochloride ($C_{12}H_8.N_2.HCl.H_2O$) in water. Warm slightly to complete dissolution, cool and dilute to 1 L.

(c) *Solution C—buffer.* Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in about 500 mL of water, filter, add 240 mL of glacial acetic acid (ρ_{20} 1.048 g/mL) and dilute to 1 L with water.

The mixed reagent should be stored in a dark coloured glass container; it should not be used after storage of more than 4 weeks.

4.2.5 *Hydrogen peroxide (100 vol).*

4.2.6 *Thiourea solution (1 mol/L).* Dissolve 3.8 g of thiourea (NH_2CSNH_2) in 50 mL of water.

4.3 Standard solutions.

4.3.1 *Stock iron solution (1 mL ≡ 0.2 mg Fe).* The following procedures for preparing the stock iron solution are listed in order of preference:

(a) Dissolve 0.2 ± 0.0001 g of cleaned iron (purity greater than 99.9% in hydrochloric acid (4.2.1). Cool, transfer to a 1 L volumetric flask and dilute to volume.

(b) Dissolve 0.2860 g of pure iron(III) oxide (Fe_2O_3), previously calcined at 600°C, in 30 mL of hydrochloric acid (4.2.2) by heating in a 100 mL beaker covered with a watch glass. Cool, transfer to a 1 L volumetric flask and dilute to volume.

(c) Dissolve 1.4045 g of iron(II) ammonium sulfate hexahydrate ($(NH_4)_2Fe(SO_4)_2.6H_2O$) in water and add 20 mL of hydrochloric acid (4.2.2). Transfer the solution to a 1 L volumetric flask and dilute to volume with water.

NOTE: If the actual iron(II) content of the iron(II) ammonium sulfate is not known, it should be measured by titration with standard potassium dichromate solution after reducing any iron(III) to iron(II) with tin(II) chloride solution, and the mass taken for the iron standard solution corrected accordingly.

4.3.2 *Standard iron solution (1 mL ≡ 0.01 mg Fe).* Pipette 50.0 mL of stock iron solution (4.3.1) into a 1 L volumetric flask and dilute to volume with water. This solution shall be freshly prepared before use.