AS 3587.3—1991

Australian Standard®

Ferroalloys—Chemical analysis

Part 3: Determination of chromium content of ferrochromium and ferrosilicochromium This Australian Standard was prepared by Committee MN/6, Ferroalloys. It was approved on behalf of the Council of Standards Australia on 12 June 1991 and published on 9 August 1991.

The following interests are represented on Committee MN/6: Australian Foundry Institute Bureau of Steel Manufacturers of Australia Confederation of Australian Industry

Additional interests participating in preparation of Standard:

Australian steelmaking organizations

Department of Defence

Queensland Railways

State Rail Authority of New South Wales

This Standard was issued in draft form for comment as DR 88177.

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which subscribing members receive, and which gives details of new publications, new editions and amendments, and of withdrawn Standards.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.

Australian Standard®

Ferroalloys—Chemical analysis

Part 3: Determination of chromium content of ferrochromium and ferrosilicochromium

First published as AS 3587.3-1991.

PUBLISHED BY STANDARDS AUSTRALIA (STANDARDS ASSOCIATION OF AUSTRALIA) 1 THE CRESCENT, HOMEBUSH, NSW 2140

ISBN 0 7262 7009 1

PREFACE

This Standard was prepared by the Standards Australia Committee on Ferroalloys, under the direction of the Minerals Standards Board, as one of a series of Standards for the chemical analysis of ferroalloys. It is based on the International Standard ISO 4140:1979, *Ferrochromium and ferrosilicochromium*— *Determination of chromium content*—*Potentiometric method*.

CONTENTS

| | | Page |
|----|--------------------------------|------|
| 1 | SCOPE | . 3 |
| 2 | REFERENCED DOCUMENTS | . 3 |
| 3 | PRINCIPLE | . 3 |
| 4 | REACTIONS | . 3 |
| 5 | REAGENTS | . 3 |
| 6 | APPARATUS | . 4 |
| 7 | SAMPLING AND SAMPLES | . 4 |
| 8 | PROCEDURE | . 4 |
| 9 | CALCULATION OF RESULT | . 5 |
| 10 | ACCEPTANCE OF RESULTS | . 6 |
| 11 | PRECISION OF THE DETERMINATION | . 6 |
| 12 | TEST REPORT | . 6 |
| | | |

© 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.

STANDARDS AUSTRALIA

Australian Standard Ferroalloys—Chemical analysis

Part 3: Determination of chromium content of ferrochromium and ferrosilicochromium

1 SCOPE This Standard specifies a potentiometric method for the determination of the chromium content of ferrochromium and ferrosilicochromium of all grades.

NOTE: For information on laboratory safety, reference should be made to the relevant parts of AS 2243 and AS 2508.

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

- AS
- 2243 Safety in laboratories

2508 Safe handling and storage information cards for hazardous materials

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

3 PRINCIPLE A sample is fused with sodium peroxide, the melt is dissolved in water, and the aqueous solution is acidified with sulfuric acid. Alternatively, for some ferroalloys, the sample is dissolved by direct acid attack. Chromium is oxidized with ammonium peroxydisulfate in the presence of silver ions as catalyst. The sum of chromium and vanadium is determined by potentiometric titration with ammonium iron(II) sulfate. The vanadium is reoxidized with potassium permanganate; the excess of permanganate is destroyed by nitrite, and the excess of nitrite is destroyed by sulfamic acid. Vanadium is determined by potentiometric titration with ammonium iron(II) sulfate, and the chromium content is determined by difference.

4 **REACTIONS** The major reactions are as follows:

(a) $\operatorname{Cr}_{2}O_{7}^{2-}$ + 6Fe²⁺ + 14H⁺ \rightarrow 2Cr³⁺ + 6Fe⁺ + 7H₂O

(b)
$$VO_2^+ + Fe^{2+} + 2H^+ \rightarrow VO^{2+} + Fe^{3+} + H_2O$$

- (c) $5\text{VO}^{2+} + \text{MnO}_4^- + \text{H}_2\text{O} + \rightarrow 5\text{VO}_2^+ + \text{Mn}^{2+} + 2\text{H}^+$
- (d) $5NO_2^- + 2MnO_4^- + 6H^+ \rightarrow 5NO_3^- + 2Mn^{2+} + 3H_2O$

5 REAGENTS

5.1 General During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

- **5.2** Sodium peroxide (Na_2O_2) —fine powder.
- **5.3** Sodium carbonate (Na₂CO₃)—anhydrous fine powder.
- 5.4 Ammonium peroxydisulfate (ammonium persulphate) ((NH₄)₂S₂O₈)
- 5.5 Sulfamic acid (HSO₃NH₂)
- 5.6 Phosphoric acid, $\boldsymbol{\varrho}_{20}$ 1.7 kg/L

WARNING: HAZARDOUS WHEN HEATED TO FUMING.

- 5.7 Nitric acid, $\boldsymbol{\varrho}_{20}$ 1.4 kg/L
- 5.8 Hydrofluoric acid, **Q**₂₀ 1.14 kg/L

WARNING: CORROSIVE. EXTREMELY HARMFUL TO SKIN AND EYES.

5.9 Sulfuric acid (diluted 1 + 1) To 500 mL of water, add cautiously 500 mL of sulfuric acid (ϱ_{20} 1.84 kg/L), mix and cool.

5.10 Hydrochloric acid (diluted 1 + 1.5) To 60 mL of water, slowly add 40 mL of hydrochloric acid ($\boldsymbol{\varrho}_{20}$ 1.16 kg/L), and mix.

5.11 Silver nitrate solution (5 g/L) Dissolve 5 g of silver nitrate $(AgNO_3)$ in water and dilute to 1 L.

5.12 Potassium permanganate solution (2.5 g/100 mL) Dissolve 2.5 g of potassium permanganate (KMnO₄) in water and dilute to 100 mL.

5.13 Potassium nitrite solution (1 g/L) Dissolve 1 g of potassium nitrite (KNO_2) in water and dilute to 1000 mL.