Australian Standard®

Ferroalloys—Chemical analysis

Part 3: Determination of chromium content of ferrochromium and ferrosilicochromium

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Australian Foundry Institute

Bureau of Steel Manufacturers of Australia

Confederation of Australian Industry

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Australian steelmaking organizations

Department of Defence

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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.*

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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) $Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \rightarrow 2Cr^{3+} + 6Fe^{+} + 7H_2O$

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

(c) $5VO^{2+} + MnO_4^- + H_2O + \rightarrow 5VO_2^{+} + Mn^{2+} + 2H^+$

(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 (Na_2CO_3)—anhydrous fine powder.

5.4 Ammonium peroxydisulfate (ammonium persulphate) ($(NH_4)_2S_2O_8$)

5.5 Sulfamic acid (HSO_3NH_2)

5.6 Phosphoric acid, ϱ_{20} 1.7 kg/L

WARNING: HAZARDOUS WHEN HEATED TO FUMING.

5.7 Nitric acid, ϱ_{20} 1.4 kg/L

5.8 Hydrofluoric acid, ϱ_{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 (ϱ_{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 ($KMnO_4$) 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.