AS 1050.23—1990
Reconfirmed 2016

Australian Standard®

Methods for the analysis of iron and steel

Part 23: Determination of molybdenum content—Flame atomic absorption spectrometric 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 12 July 1990 and published on 12 November 1990.

The following interests are represented on Committee CH/10:

Aluminium Development Council
Australian Lead Development Association
Copper Technical Data Centre
National Association of Testing Authorities, Australia
Royal Australian Chemical Institute

Additional interests participating in preparation of Standard:

Department of Defence, Materials Research Laboratory
Steel manufacturers

This Standard was issued in draft form for comment as DR 90015.

STANDARDS AUSTRALIA

RECONFIRMATION

OF
AS 1050.23—1990
Methods for the analysis of iron and steel
Part 23: Determination of molybdenum content—Flame atomic absorption spectrometric 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.

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 31 July 2016.

The following are represented on Technical Committee CH-010:

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 the Standards Australia Committee on the Analysis of Metals, under the direction of the Chemical Standards Board, as a proposed further part of AS 1050. On publication, it will supersede AS K1, *Methods for analysis of iron and steel*, Part 23—1967: *Determination of molybdenum in iron and steel (photometric method)*.

The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. Laboratories from the following organizations participated in the test program to provide the data given in Table 2:

BHP Steel, Sheet and Coil Products Division
BHP Steel, Rod and Bar Products Division
BHP Steel, Slab and Plate Products Division
BHP Steel, Long Products Division

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STANDARDS AUSTRALIA

Australian Standard

Methods for the analysis of iron and steel

Part 23: Determination of molybdenum content—Flame atomic absorption spectrometric method

1 SCOPE This Standard sets out a flame atomic absorption spectrometric method for the determination of molybdenum in iron and steel. The method is applicable to iron and steel with molybdenum contents in the range of 0.005% to 2.5%.

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

AS
1213 Iron and steel — Methods of sampling

2134 Recommended practice for chemical analysis by atomic absorption spectrometry
2134.1 Part 1: Flame atomic absorption spectrometry

2164 One-mark volumetric flasks

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

3 PRINCIPLE The test sample is dissolved in hydrochloric, phosphoric and nitric acids. Hydrofluoric acid is added to remove silica and dissolve tungsten, if present. The test sample is then fumed with perchloric acid and the molybdenum content is determined by flame atomic absorption spectrometry.

4 REAGENTS

4.1 General requirements During the analysis only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity, shall be used.

4.2 High purity iron Iron of purity greater than 99.99% containing less than 2 µg/g molybdenum.

4.3 Solutions

4.3.1 *Hydrochloric acid (ϱ_{20} 1.16 g/mL to 1.18 g/mL).*

4.3.2 *Phosphoric acid (1 + 9)* Add 50 mL of phosphoric acid (ϱ_{20} 1.75 g/mL) to 450 mL of water and mix.

4.3.3 *Nitric acid (ϱ_{20} 1.42 g/mL).*

4.3.4 *Perchloric acid (ϱ_{20} 1.67 g/mL).*

WARNING: PERCHLORIC ACID REACTS EXPLOSIVELY WITH ORGANIC MATTER. IT IS CORROSIVE TO SKIN, AND MUCOUS MEMBRANES.

4.3.5 *Hydrofluoric acid (approx. ϱ_{20} 1.13 g/mL)* Nominal 40% solution.

WARNING: EVEN WHEN DILUTED, HYDROFLUORIC ACID IS EXTREMELY DANGEROUS AND HARMFUL TO THE EYES AND SKIN; RUBBER GLOVES AND GOGGLES SHOULD BE WORN WHEN USING THIS ACID. HYDROFLUORIC ACID ATTACKS GLASSWARE. CARE SHOULD BE TAKEN TO MINIMIZE THE TIME OF ACID CONTACT WITH GLASSWARE. USE ONLY IN A MECHANICALLY VENTILATED FUME CUPBOARD.

4.4 Standard solutions

4.4.1 *Aluminium solution (1 mL ≡ 0.01 g Al)* Dissolve 5.00 g of high purity aluminium (>99.99% Al) in a mixture of 60 mL of hydrochloric acid (4.3.1) and 60 mL of water, and dilute to about 250 mL with water. Filter into a 500 mL volumetric flask and dilute to volume with water.

4.4.2 *Iron solution (1 mL ≡ 0.01 g Fe)* Weigh 5.00 g of high purity iron (4.2) into a 250 mL beaker and add 30 mL of hydrochloric acid (4.3.1). Warm to dissolve, add 5 mL of nitric acid (4.3.3) to oxidize the iron, and then heat gently to remove excess nitrogen dioxide (brown fumes). Cool and dilute to 500 mL in a volumetric flask.

WARNING: DISSOLUTION OF THE IRON SHOULD BE CARRIED OUT IN A FUME CUPBOARD.

4.4.3 *Stock molybdenum solution (1 mL ≡ 0.01 g Mo)* Weigh 12.61 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) into a 600 mL beaker. Dissolve in 200 mL of water, transfer to a 500 mL volumetric flask and dilute to volume.

4.4.4 *Standard molybdenum solution (1 mL ≡ 1 mg Mo)* Pipette 100.0 mL of stock molybdenum solution (4.4.3) into a 1 L volumetric flask and dilute to volume with water.