STANDARDS AUSTRALIA

RECONFIRMATION

OF
AS 1050.12—1983
Methods for the analysis of iron and steel
Part 12: Determination of nitrogen in steel (steam distillation titrimetric 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 1 November 2016.

The following are represented on Technical Committee CH-010:

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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Australian Standard®

METHODS FOR THE ANALYSIS OF IRON AND STEEL

Part 12—DETERMINATION OF NITROGEN IN STEEL (STEAM DISTILLATION TITRIMETRIC METHOD)

This Australian standard was prepared by Committee CH/10, Analysis of Metals. It was approved on behalf of the Council of the Standards Association of Australia on 30 September 1982 and published on 10 January 1983.

The following interests are represented on the committee responsible for the preparation of this standard:

Aluminium Development Council
Australasian Institute of Mining and Metallurgy
Australian Lead Development Association
Australian Mineral Development Laboratories
Australian Tin Information Centre
Australian Zinc Development Association
Bureau of Steel Manufacturers of Australia
Confederation of Australian Industry
Copper Producers Association of Australia
Department of Defence
Electricity Supply Association of Australia
Metal Trades Industry Association of Australia
National Association of Testing Authorities
Railways of Australia Committee
Royal Australian Chemical Institute

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PREFACE

This standard has been prepared by the Association's Committee for the Analysis of Metals under the direction of the Chemical Standards Board. The method was previously given in AS K1, Part 12—1959, which was an endorsement of BS 1121: Part 39, but which was withdrawn when BS 1121 was replaced by BS Handbook No 19.

The main difference from AS K1, Part 12 is the determination of total nitrogen in the one step instead of determining the 'acid soluble' and 'insoluble' nitrogen contents separately and combining the results.

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 1:

Australian Iron and Steel Pty Ltd, Port Kembla

Commonwealth Steel Co. Ltd

John Lysaght (Australia) Limited

Materials Testing Laboratories, Department of Defence

The Broken Hill Proprietary Co. Ltd, Newcastle

STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF IRON AND STEEL

PART 12—DETERMINATION OF NITROGEN IN STEEL (STEAM DISTILLATION TITRIMETRIC METHOD)

1 SCOPE. This standard sets out a method for the determination of nitrogen in all types of steel.

NOTE: Complete recovery of nitrogen cannot be obtained from steel containing silicon nitride. This nitride has been found only in samples of silicon steels manufactured without aluminium additions and then only in sheet material.

2 APPLICATION. The method is applicable to the determination of nitrogen contents between 0.001 percent and 0.5 percent.

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 1213 Methods for the Sampling of Iron and Steel

AS 2243 Code of Practice for Safety in Laboratories
Part 2—Chemical

BS 4237 Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry.

4 REPRODUCIBILITY. A planned trial of the method was carried out in accordance with BS 4237.

The reproducibility index (2*s*) is obtained from the following formula:

$$2s = 2\sqrt{(s_b^2 + s_w^2)}$$

where

s_b = between-operator standard deviation

s_w = within-operator standard deviation

95 percent of the results obtained by any one analyst should be reproducible to within two standard deviations of the overall mean value derived from all laboratories (i.e. $\bar{x} \pm 2s$).

For further information, see BS 4237.

The planned trial was carried out by five analysts, each from a different laboratory. Five tests were carried out by each analyst on each of ten samples.

From the results obtained the 95 percent confidence limits (2s, Table I) have been calculated.

5 PRINCIPLE. The nitrogen is converted to ammonium ion by digestion in, and then by fuming with, sulphuric acid. The ammonium ion is separated as ammonia by distillation from a solution made alkaline with sodium hydroxide, collected in a receiver containing boric acid solution and titrated with hydrochloric acid.

6 REAGENTS.

6.1 General. Only reagents known to give a minimum nitrogen blank shall be used (see Clause 9.2).

6.2 Water and Ammonia-free Water. All references to water relate to distilled water except where ammonia-free water is specifically stated. Ammonia-free water shall be prepared by passing water through the cation exchange resin column described in Clause 7.

It is essential that the resin column should be acid washed before use to ensure its conversion to the hydrogen form. This is most conveniently done by passing 2 L of hydrochloric acid (6.4.4) through the column, then washing the column with water until it is free from acid.

It is essential that ammonia-free water be used instead of distilled water for the preparation of solutions. If such solutions require filtering, the filter paper shall be washed thoroughly with ammonia free water before use.

6.3 Solid

6.3.1. *Sodium sulphate, anhydrous* (Na_2SO_4).

6.4 Solutions.

6.4.1. *Sulphuric acid* (ρ_{20} 1840 kg/m^3). Sulphuric acid supplies shall be tested individually and selected for a low content of combined nitrogen in any form (less than 0.5 μg/g). Nitrogen as ammonia will usually be the major source of contamination and this may be tested by the normal methods, but nitrates may also be present and may be detected by the following method:

Add 6 mL of sulphuric acid to 2 mL of water and cool to 60°C. Add 1 drop of hydrochloric acid (ρ_{20} 1160 to 1180 kg/m^3) and the equivalent of 1 mg of sodium diphenylamine reagent. No blue colour should develop. Less than 0.1 μg/g can be detected by this test.