AS 1671.4—1988
Reconfirmed 2016

Australian Standard®

LEAD AND LEAD ALLOYS

Part 4—DETERMINATION OF TIN IN ANTIMONIAL LEAD—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 the Standards Association of Australia on 21 January 1988 and published on 5 April 1988.

The following interests are represented on Committee CH/10:

Aluminium Development Council

Australasian Institute of Mining and Metallurgy

Australian Lead Development Association

Australian Mineral Development Laboratories

Australian Zinc Development Association

Bureau of Steel Manufacturers of Australia

Confederation of Australian Industry

Copper Technical Data Centre

Department of Defence

Metal Trades Industry Association of Australia

National Association of Testing Authorities

Railways of Australia Committee

Royal Australian Chemical Institute

This Standard was issued in draft form for comment as DR 87008.

STANDARDS AUSTRALIA

RECONFIRMATION

OF
AS 1671.4—1988
Lead and lead alloys
Part 4: Determination of tin in antimonial lead—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 Association's Committee on the Analysis of Metals under the direction of the Chemical Standards Board.

It was prepared as part of the AS 1671 series of Standards covering the analysis of lead and lead alloys. The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. The following laboratories participated in the test program to provide the data given in Table 2:

BHP Steel International, Coated Products Division, Port Kembla
Broken Hill Associated Smelters Pty Ltd
Electrolytic Refining and Smelting Co.
Metal Manufactures Pty Ltd
Sulphide Corporation Pty Ltd

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

LEAD AND LEAD ALLOYS

Part 4: DETERMINATION OF TIN IN ANTIMONIAL LEAD—FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

1 SCOPE. This Standard sets out a flame atomic absorption spectrometric method for the determination of tin in antimonial lead.

2 APPLICATION. This method is applicable to the determination of tin in antimonial lead in the range of 0.01 percent (*m/m*) to 0.5 percent (*m/m*) in the presence of up to 1.0 percent antimony.

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

AS

2134 Recommended practice for chemical analysis of materials by atomic absorption spectrometry
Part 1: Flame atomic absorption spectrometry (AS 2134.1)

2162 Code of practice for the use of volumetric glassware

2164 One-mark volumetric flasks

2165 Burettes and bulb burettes

2166 One-mark pipettes

2534 Lead and lead alloys—Sampling and preparation of samples for chemical analysis

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

4 PRINCIPLE. The sample is dissolved in ammonium fluoride solution and nitric acid and the tin content is determined by flame atomic absorption spectrometry.

5 REAGENTS.

5.1 General requirements. Except where otherwise specified, only reagents of recognized analytical grade and distilled water or water of an equivalent purity shall be used.

5.2 Solids.

5.2.1 *High purity tin*—purity not less than 99.99 percent.

5.2.2 *High purity lead*—purity not less than 99.99 percent.

5.2.3 *Ammonium fluoride.*

5.3 Solutions.

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

5.3.2 *Ammonium fluoride solution (20 g/L).* Dissolve 20.0 g of ammonium fluoride (5.2.3) in water, dilute to 1 L and store in a plastics bottle.

5.4 Standard solution.

5.4.1 *Standard tin solution (1 mL ≡ 1 mg Sn).* Dissolve 0.5000 g of pure tin (5.2.1) and 5 g of ammonium fluoride (5.2.3) in 100 mL of water and add 20 mL of nitric acid (5.3.1). Cool, transfer to a 500 mL volumetric flask and dilute to volume with water.

6 APPARATUS.

6.1 Glassware. Grade A volumetric glassware shall be used throughout. Volumetric flasks shall comply with AS 2164, burettes shall comply with AS 2165, and pipettes shall comply with AS 2166. Use of volumetric glassware shall comply with AS 2162.

6.2 Atomic absorption spectrometer. The spectrometer shall be set up in accordance with AS 2134.1 and be capable of isolating the resonance wavelength of 235.4 nm. A nitrous oxide/acetylene flame shall be used. Background correction may be required.

7 SAMPLING. Samples for analysis shall be obtained in accordance with AS 2534.

8 PROCEDURE.

8.1 Number of determinations. This method is written for a single determination only and may be used in that form. Where replicate determinations are required, procedures for acceptance of the results so obtained are given in Clause 11.

8.2 Blank test. A blank test shall be carried out in parallel with the analysis using the same procedure as for the analysis and the same quantities of all reagents used in the determination and containing 2 g of high purity lead (or 5 g if the larger sample is used in the determination) in accordance with the basic composition of the test sample.

8.3 Check test. It is recommended that a check test using a certified reference material or another validated reference material, be carried out with the determination. The procedure for the evaluation of the check test results is given in Clause 11.

NOTE: The composition of the reference material should match as closely as possible that of the sample.

8.4 Dissolution of test sample. The procedure shall be as follows:

(a) Transfer 2 ±0.001 g (for the expected range of 0.2 to 0.5 percent) or 5 ±0.001 g, (for the expected range 0.01 to 0.2 percent) of the test sample to a 250 mL beaker.

(b) Add 50 mL of water, 20 mL of ammonium fluoride solution (5.3.2) and 15 mL of nitric acid (5.3.1). Boil to expel oxides of nitrogen and cool the solution to ambient temperature.

(c) Transfer the solution to a 100 mL volumetric flask and dilute to volume with water.