AS 1671.3—1987
Reconfirmed 2016

Australian Standard®

LEAD ALLOYS

Part 3—DETERMINATION OF HIGH CONCENTRATIONS OF ANTIMONY IN LEAD ALLOYS CONTAINING NOT MORE THAN 2.5 PERCENT ARSENIC AND 1.0 PERCENT COPPER—TITRIMETRIC METHOD

This Australian standard was prepared by Committee CH/10—Analysis of Metals under the direction of Chemical Standards Board. It was approved on behalf of the Council of the Standards Association of Australia on 24 February 1987 and published on 4 May 1987.

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 Tin Information Centre

Australian Zinc Development Association

Bureau of Steel Manufacturers of Australia

Confederation of Australian Industry

Copper Technical Data Centre

Department of Defence

Electricity Supply Association of Australia

Metal Trades Industry Association of Australia

National Association of Testing Authorities

Railways of Australia Committee

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STANDARDS AUSTRALIA

RECONFIRMATION

OF
AS 1671.3—1987
Lead alloys
Part 3: Determination of high concentrations of antimony in lead alloys containing not more than 2.5 percent arsenic and 1.0 percent copper—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.

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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

PREFACE

This edition of this standard was prepared by the Association's Committee on the Analysis of Metals under the direction of the Chemical Standards Board. It supersedes AS 1671.3—1975, Methods for the Analysis of Lead Alloys, Higher Concentration Antimony in Lead and White Metal Alloys Containing Not More Than 2.5 percent Arsenic and 1.0 percent Copper (Volumetric Method).

CONTENTS

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PART 3—DETERMINATION OF HIGH CONCENTRATIONS OF ANTIMONY IN LEAD ALLOYS CONTAINING NOT MORE THAN 2.5 PERCENT ARSENIC AND 1.0 PERCENT COPPER—TITRIMETRIC METHOD

1 SCOPE. This standard sets out a titrimetric method for the determination of antimony in lead alloys.

2 APPLICATION. The method is applicable to lead alloys with antimony contents in the range 2.0 to 27.0 percent and containing not more than 2.5 percent arsenic and 1.0 percent copper.

NOTE: For samples containing greater than 1.0 percent copper, an additional amount of copper (as copper-sulphate solution 5.3.3)) equivalent to the copper present should be added to calibration solutions after sulphuric acid addition.

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

AS 2162 Code of Practice for the Use of Volumetric Glassware

AS 2164 One-mark Volumetric Flasks

AS 2165 Burettes and Bulb Burettes

AS 2166 One-mark Pipettes

AS 2534 Lead and Lead Alloys—Sampling and Preparation of Samples for Chemical Analysis

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

4 PRINCIPLE. Antimony is reduced to the trivalent state with sodium sulphite in a sulphuric acid/hydrochloric acid medium and determined by potassium bromate titration.

5 REAGENTS.

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

5.2 Solids.

5.2.1 *Antimony.* Purity not less than 99.99 percent.

5.2.2 *Lead.* Purity not less than 99.99 percent and containing less than 5 µg Sb/g.

5.2.3 *Sodium sulphite (anhydrous).*

5.3 Solutions.

5.3.1 *Hydrochloric acid* (ϱ_{20} 1.16 g/mL).

5.3.2 *Sulphuric acid* (ϱ_{20} 1.84 g/mL).

5.3.3 *Copper sulphate solution (5 g/L).* Dissolve 0.5 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in water, dilute to 100 mL and mix.

5.3.4 *Methyl orange indicator.* Dissolve 0.05 g of methyl orange in about 50 mL of water. Filter, dilute to 100 mL and mix.

5.4 Standard solutions.

5.4.1 *Potassium bromate solution.* Weigh 1.3917 ± 0.0001 g of potassium bromate ($KBrO_3$) previously dried at 105° to 110°C to constant mass. Dissolve in water, transfer to a 1 L volumetric flask, dilute to volume and mix. This solution shall be standardized by the procedure in Appendix A prior to use.

5.4.2 *Potassium bromate solution.* Weigh 2.7834 ± 0.0001 g of potassium bromate ($KBrO_3$) previously dried at 105° to 110°C to constant mass. Dissolve in water, transfer to a 1 L volumetric flask, dilute to volume and mix. This solution shall be standardized by the procedure in Appendix A prior to use.

6 APPARATUS.

6.1 Glassware. Grade A volumetric glassware shall be used throughout. Pipettes shall comply with AS 2166 and burettes with AS 2165. Use of volumetric glassware shall be in accordance with AS 2162.

6.2 Analytical balance. Capable of weighing accurately to 0.0001 g.

7 SAMPLING. Sampling shall be in accordance with AS 2534.

NOTES:

1. The tendency of most lead and tin alloys to segregate should be stressed and care should be taken to avoid a biased analysis caused by sample segregation.
2. Adhering contamination should be cleaned from all surfaces of the sample pieces (see Appendix B).
3. Where hacksaw sampling produces a mixture of coarse and fine particles, they should be screened to separate the coarse and fine fractions. These should then be weighed to determine the proportion of the fractions. Suitable masses of these sized fractions should be combined for the assay samples.
4. Alternatively, for antimonial-lead alloys containing in excess of 10 percent antimony, it is recommended that a molten sample be chill cast in a suitable iron mould to produce two or four prong pin samples. The diameter of the pins should be 6 mm and the length of the pins should be approximately 50 mm below the lug of the metal.

To obtain the desired mass of sample for analysis, snip approximately 5 mm from the bottom of a pin. File a sample from the clean edge of the remaining pin, collecting the filings on a clean sheet of sample paper. When sufficient sample has been collected, pass a magnet over the sample before weighing. Transfer all filings from the sample sheet to the balance pan. This is essential as segregation can occur in filings of different particle size. A clean, sharp 200 mm flat bastard file is a suitable tool for this operation.