

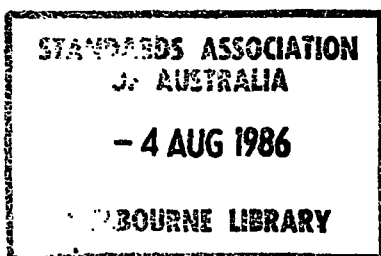
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METHODS FOR THE ANALYSIS OF ZINC AND ZINC ALLOYS— Part 1—DETERMINATION OF ALUMINIUM CONTENT (TITRIMETRIC METHOD)



AS 1329
Methods for the analysis of zinc
and zinc alloys

AS 1329.1—1986
Determination of aluminium
content—Titrimetric method
5pp C

Specifies a titrimetric method
for the determination of
aluminium in zinc and zinc
alloys. It is applicable in the
range of 2 percent to 13 percent
aluminium. The method is not
applicable to zinc alloys con-
taining titanium as this inter-
feres with the determination.

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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
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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 to supersede AS 1329.1—1972—Determination of Aluminium in Zinc Alloys (Volumetric Method).

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF ZINC AND ZINC ALLOYS—

PART 1—DETERMINATION OF ALUMINIUM CONTENT (TITRIMETRIC METHOD)

1 SCOPE. This standard sets out a titrimetric method for the determination of aluminium in zinc and zinc alloys.

2 APPLICATION. This method is applicable to the determination of aluminium in zinc and zinc alloys in the range 3 percent to 13 percent. This method is not applicable to zinc alloys containing titanium as titanium will report as aluminium.

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 2347 Methods for the Sampling of Zinc Metal and Zinc Alloys for Chemical Analysis
BS 4237 Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry.

4 PRINCIPLE. The sample is dissolved in hydrochloric acid and complexed with the disodium salt of diaminoethanetetra-acetic acid (EDTA). Excess EDTA is titrated with standard zinc solution. Aluminium is then complexed with sodium fluoride and the liberated EDTA titrated with standard zinc solution.

5 REAGENTS.

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

5.2 Solutions.

5.2.1 Hydrochloric acid solution (1 + 1). Add 500 mL of hydrochloric acid (ρ_{20} 1.16 g/mL) to 500 mL of water and mix.

5.2.2 Ammonia solution. (ρ_{20} 0.88 g/mL.)

5.2.3 Diaminoethanetetra-acetic acid disodium salt solution EDTA. (Approx 0.2 mol/L.) Dissolve 150 g of EDTA in 1500 mL of hot water, cool and dilute to 2 litres in a volumetric flask. Store in a polyethylene bottle.

5.2.4 Sodium acetate solution (300 g/L). Dissolve 150 g of sodium acetate trihydrate ($\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$) in water, dilute to 500 mL and mix.

5.2.5 Sodium fluoride solution (saturated). Dissolve 60 g of sodium fluoride (NaF) in 1 litre of boiling water, cool and filter. Store in a polyethylene bottle.

5.2.6 Hydrogen peroxide. (100 vols.)

5.2.7 Ethanol. (95% V/V.)

5.3 Indicators.

5.3.1 Bromo-cresol green (0.5 g/L). Dissolve 0.05 g of bromo-cresol green in 50 mL of ethanol (5.2.7) and dilute to 100 mL with water.

5.3.2 Methyl red (0.2 g/L). Dissolve 0.02 g of methyl red in ethanol (5.2.7) and dilute to 100 mL with ethanol.

5.3.3 PAN indicator (0.5 g/L). Dissolve 0.05 g of 1-(2-pyridyl-azo)-2-naphthol (PAN) in ethanol (5.2.7) and dilute to 100 mL with ethanol.

5.4 Standard zinc solution (0.05 mol/L). Weigh 3.269 ± 0.0001 g of high purity zinc (> 99.99% Zn) into a beaker, and dissolve in hydrochloric acid (5.2.1). Add two drops of methyl red indicator (5.3.2), neutralize with ammonia solution (5.2.2) and make just acid with hydrochloric acid (5.2.1). Boil for 5 min, cool and transfer to a 1 L volumetric flask. Dilute to volume with water and mix.

6 APPARATUS. The following apparatus is required:

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 Glass beads.

7 SAMPLING AND SAMPLES. Samples shall be taken in accordance with AS 2347.

8 PROCEDURE.

8.1 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 the reagents but omitting the sample. The blank value must not exceed 0.1 mL.

8.2 Check test. In each run, one analysis of a certified reference material or reference material of the same type of metal should be carried out in parallel with the analysis of the test sample and under the same conditions. However, in cases of dispute, an analysis of a certified reference material shall be carried out in parallel with the analysis of the test sample.

When the analysis is carried out on several samples of the same type of metal at the same time, the analytical value of one certified reference material may be used.

NOTES:

1. Certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in either case no significant changes in the analytical procedure would become necessary.
2. The certified reference material is used to validate the performance of the analytical procedure and expressly not to adjust analytical results.