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Zinc

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STANDARDS AUSTRALIA~~

WITHDRAWN:

19980805

SUPERSEDED BY AS 2446-1981
UDC 669.5:543.05

AS 2347-1998

under revision see DR 99369.

Australian Standard 2446—1981

ZINC METAL AND ALLOYS— SAMPLING AND THE PREPARATION OF SOLID SAMPLES FOR OPTICAL EMISSION SPECTROMETRY



STANDARDS ASSOCIATION OF AUSTRALIA
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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 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

This standard, prepared by Committee CH/10, Analysis of Metals, was approved by the Council of the Standards Association of Australia on 9 February 1981, and was published on 1 June 1981.

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First published 1981

This standard was issued in draft form for public review as DR 79161.✓

ISBN 0 7262 2217 8

PREFACE

This standard was prepared by the Association's subcommittee for the analysis of non-ferrous metals under the direction of the Chemical Standards Board. The method of taking the sample and preparing it for analysis is an important preliminary to the analytical procedure.

The standard requires reference to ASTM E401—Recommended Practice for Bonding Thin Spectrochemical Samples and Standards to a Greater Mass of Material.

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard
for

**ZINC METAL AND ALLOYS—SAMPLING AND THE PREPARATION OF SOLID
SAMPLES FOR OPTICAL EMISSION SPECTROMETRY**

1 SCOPE. This standard sets out methods for the sampling of zinc metal and zinc alloys and for the preparation of the test samples required for the determination of composition by optical emission spectrometry. Samples may be taken from cast forms or from the molten metal.

NOTE: Samples taken from the cast forms and remelted are not suitable for the determination of arsenic, sodium, or antimony because of drossing.

2 DEFINITIONS. For the purpose of this standard, the following definitions apply:

2.1 Batch—a consignment of one grade designation of one cast form, such as slab, ingot or block.

2.2 Primary sample—the selected ingots or slabs from which the sample is prepared.

2.3 Laboratory sample—the final form of the material submitted for analysis, i.e. a cast disc or pin.

2.4 Test sample—a suitable part of the laboratory sample containing the same components in the same proportions as they occur in the relatively large mass of the original batch or batches of material.

3 SELECTION OF PRIMARY SAMPLE.

3.1 Selection of Primary Sample from Cast Forms. The minimum number of slabs, ingots, or blocks, which constitute the primary sample shall be selected at random from the consignment in accordance with Table 1.

TABLE 1
SELECTION OF PRIMARY SAMPLES

Number in consignment	Number to be sampled
less than 5	all
from 5 to 249	5
250 or more	1 from each 50

3.2 Selection of Primary Sample from Molten Metal. A primary sample shall be taken from the molten metal or alloy and prepared in accordance with Clause 6.

4 PREPARATION OF LABORATORY SAMPLES.

4.1 From Slabs or Ingots. Samples shall be obtained from slabs or ingots by drilling in accordance with the following procedure:

- (a) Clean the selected slabs or ingots to remove scale, dirt, grease, oxide film or other surface impurities, from their surfaces.
- (b) Arrange the selected slabs or ingots flat, side by side, and upside down with reference to the position occupied in the mould, in groups of a

maximum of five slabs or ingots as shown in Fig. 1. Ensure that the casting marks are arranged in the same way for each of the slabs or ingots.

- (c) In each group, draw a diagonal across the rectangle thus formed.
- (d) Mark three drilling points as shown in Fig. 1 on each slab or ingot.

NOTES:

1. Where the exact position of the point to be drilled coincides with a notch in the ingot, choose another point as close as possible.

2. If the sample consists of only one slab or ingot, select drilling points in a similar way along the diagonal of the single slab or ingot.

- (e) Carry out the drilling using a clean, high speed, tungsten carbide drill of about 15 mm diameter and without the use of a lubricant. The speed of drilling shall be so regulated that excessive heating and consequent oxidation of the sample are avoided. The holes shall be drilled through the total thickness of each slab or ingot and, if necessary, drilled from both directions, top and bottom. The drillings should, preferably, be uniformly small in size.

NOTE: To facilitate the preparation of drillings of satisfactory size, V-shaped chip breaking grooves may be formed in the cutting edge of the drill by grinding, or other convenient means, at approximately right angles to the cutting edge.

4.2 From Primary Samples Unsuitable for Drilling. The sampling of solid jumbo blocks (blocks of 1 t or more) can present problems and a sample should be taken from the molten metal in accordance with Clause 6, or as otherwise agreed between the interested parties.

4.3 Removal of Iron. Use a strong magnet to remove any particles of iron which may have been introduced during sampling.

4.4 Removal of Grease. Remove any coatings of grease with an appropriate solvent.

5 REMELTING THE LABORATORY SAMPLE. The laboratory sample shall be pressed into a briquette before it is melted. The briquette shall then be remelted in a graphite or other inert crucible under an inert atmosphere. The melt shall be maintained at a temperature well above the point at which any solid phase can be present, but shall not exceed 500° C.

6 SAMPLING MOLTEN METAL. Molten metal, which shall be maintained at a temperature well above the point at which any solid phase can be present, shall be sampled by the following procedure:

- (a) Use a clean graphite or ceramic ladle to push any dross away from the sampling area.
- (b) Dip the ladle sideways into the clear area, well below the surface, and stir momentarily.