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Magnesium alloys — Determination of insoluble zirconium — Alizarin sulphonate photometric method

Alliages de magnésium — Dosage du zirconium insoluble — Méthode photométrique à l'alizarine sulfonate

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

ISO 2354 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*. This second edition contains the note, added at the end of clause 1, which was circulated in November 1975, in the form of an Addendum, directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This second edition cancels and replaces the first edition (i.e. ISO 2354-1972), which had been approved by the Member Bodies of the following countries :

Austria	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Canada	Italy	Sweden
Egypt, Arab Rep. of	Japan	Switzerland
Finland	Netherlands	Thailand
France	New Zealand	United Kingdom
Germany	Norway	U.S.A.
India	Poland	U.S.S.R.

No Member Body had expressed disapproval of the document .

Magnesium alloys – Determination of insoluble zirconium – Alizarin sulphonate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an alizarin sulphonate photometric method for the determination of insoluble zirconium in magnesium alloys containing zirconium as an alloying element.

Rare earths, thorium and silver do not interfere.

The method is applicable to alloys having insoluble zirconium contents between 0,02 and 0,3 % (*m/m*).

NOTE – Hafnium which is normally present in conjunction with zirconium (up to 1 % zirconium) will be determined with zirconium.

2 PRINCIPLE

Hydrochloric acid attack (the normality of the hydrochloric acid and the length of the attack have been conventionally fixed).

Filtration, dissolution of the insoluble residue in perchloric acid in the presence of hydrofluoric acid, and taking of an aliquot.

Formation, when hot, of the zirconium-alizarin sulphonate complex, in 1,5 N hydrochloric medium.

Photometric measurement of the coloured complex at a wavelength of about 525 nm.

3 REAGENTS

During the analysis use only reagents of recognized analytical quality and only distilled water or water of equivalent purity.

3.1 Hydrochloric acid, ρ approximately 1,18 g/ml, about 37 % (*m/m*) solution, or approximately 12 N.

3.2 Hydrofluoric acid, approximately 40 % (*m/m*) solution.

3.3 Perchloric acid, ρ approximately 1,54 g/ml, about 60 % (*m/m*) solution.

3.4 Sodium alizarin sulphonate, 1,5 g/l solution.

Dissolve 1,5 g of sodium alizarin sulphonate in about 300 ml of warm water, filter, cool, make up the volume to 1 000 ml and mix.

3.5 Magnesium chloride, 420 g/l solution.

Dissolve 42 g of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in water, make up the volume to 100 ml and mix.

This solution contains 50 g of magnesium per litre.

3.6 Zirconium, 5 g/l standard solution.

Prepare this solution according to one of the following procedures :

3.6.1 Weigh, to the nearest 0,001 g, 0,500 g of pure zirconium (purity $\geq 99,9$ %) and transfer to a dry beaker. Add 30 ml of methanol and, while cooling, 5 ml of bromine. When the reaction has ceased, heat gently to complete the attack. Add 20 ml of the hydrochloric acid (3.1), heat to boiling and continue boiling until a colourless solution is obtained, maintaining the volume of the solution at approximately 50 ml by adding water.

Cool, transfer quantitatively to a 100 ml volumetric flask, make up to volume and mix.

3.6.2 Dissolve 1,77 g of zirconium oxychloride octahydrate ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$) in water, add 10 ml of the hydrochloric acid (3.1), filter, make up the volume to 100 ml and mix.

NOTE – The zirconium oxychloride used shall not be moist. However, it is not possible to dry the product in an oven, as part of it could be transformed into a form which, although soluble and able to be determined by a gravimetric method (see "Standardization of the solution"), reacts very slowly with alizarin S.

Standardization of the solution 3.6.2. Use one of the following procedures :

a) GRAVIMETRIC DETERMINATION WITH MANDELIC ACID

Transfer 10,0 ml of the standard zirconium solution (3.6.2) to a beaker of suitable capacity (for example 250 ml). Dilute to about 40 ml and add 30 ml of the hydrochloric acid (3.1). Boil and add 50 ml of 150 g/l mandelic acid solution.

Allow to stand at 80 °C for about 20 min. Allow to cool, then filter through a medium texture filter paper. Wash with a solution containing 20 ml of the