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Magnesium and its alloys — Determination of manganese in magnesium alloys containing zirconium, rare earths, thorium and silver — Periodate photometric method

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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.

International Standard ISO 2353 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*.

It was approved in December 1971 by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the document.

Magnesium and its alloys – Determination of manganese in magnesium alloys containing zirconium, rare earths, thorium and silver – Periodate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of manganese in magnesium alloys containing zirconium, and/or rare earths, and/or thorium, and/or silver.

The method is applicable to the determination of manganese contents between 0.002 and 0.2 %.

2 PRINCIPLE

2.1 Dissolution of a sample by sulphuric acid, followed by oxidation with nitric acid, in the presence of fluoboric acid.

2.2 Oxidation of manganese (II) to manganese (VII) by means of potassium periodate in a medium of acidity approximately 5 N.

2.3 Photometric measurement at a wavelength of about 545 nm.

3 REAGENTS

During the analysis use only distilled water or water of equivalent purity.

3.1 Potassium periodate (KIO_4)

3.2 Sulphuric acid ($\rho = 1.26 \text{ g/ml}$), approximately 9 N solution.

Carefully add 25 ml of sulphuric acid ($\rho = 1.84 \text{ g/ml}$), 35.6 N approximately, to 60 ml of water. After cooling, make up the volume to 100 ml and mix.

3.3 Nitric acid ($\rho = 1.40 \text{ g/ml}$), approximately 15 N solution.

The acid shall be free from nitric fumes. To eliminate these fumes boil for a short while or pass through a current of carbon dioxide.

3.4 Fluoboric acid, 0.4 % (m/m) solution.

Dilute 10 ml of fluoboric acid 40 % (m/m) to 1 000 ml with water.

3.5 Water free from reducing agents

Heat to boiling, water acidified with 10 ml per litre of sulphuric acid (3.2); add a few crystals of potassium periodate (3.1) and continue boiling for about 10 min.

3.6 Sodium nitrite, 20 g/l solution.

Dissolve 2 g of sodium nitrite (NaNO_2) in a little water and make up the volume to 100 ml.

3.7 Manganese standard solution, 1 g/l (1 ml contains 1 mg of Mn),

prepared by one of the following methods.

3.7.1 In a tall form beaker of suitable capacity (for example 400 ml) dissolve 2.877 g of very pure potassium permanganate (KMnO_4) in about 200 ml of water and add 40 ml of sulphuric acid (3.2). Reduce the permanganate solution by adding a few crystals of sodium sulphite or hydrogen peroxide (36 % (m/m)). Boil the solution to remove excess SO_2 or H_2O_2 , cool, transfer quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

3.7.2 In a tall form beaker of suitable capacity (for example 600 ml) dissolve $1 \pm 0.001 \text{ g}$ of electrolytic manganese (purity above 99.9 %) with 40 ml of sulphuric acid (3.2) and about 80 ml of water. Boil the solution for a few minutes. Cool, transfer quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

NOTE — Free the electrolytic manganese from any surface oxide by placing a few grams of the metal in a glass beaker, of about 250 to 300 ml capacity, containing 60 to 80 ml of sulphuric acid (3.2) and about 100 ml of water. Shake and, after a few minutes, decant the acid solution and introduce into the beaker double distilled water. Repeat the decantation and washing with double distilled water several times; then place the metallic manganese in acetone and shake. Decant the acetone, dry the metal in a hot air oven at 100°C for about 2 min and allow it to cool in a desiccator.

3.8 Manganese standard solution, 0.1 g/l (1 ml contains 0.1 mg of Mn).