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# INTERNATIONAL STANDARD



# 810

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## **Magnesium and magnesium alloys — Determination of manganese — Periodate photometric method (Manganese content less than 0,01 %)**

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 810 replaces ISO Recommendation R 810-1968 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*.

The Member Bodies of the following countries approved the Recommendation :

Argentina	India	South Africa, Rep. of
Austria	Ireland	Spain
Belgium	Israel	Switzerland
Bulgaria	Italy	Sweden
Canada	Japan	Turkey
Chile	Korea, Rep. of	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
France	New Zealand	U.S.S.R.
Germany	Norway	Yugoslavia
Hungary	Poland	

No Member Body expressed disapproval of the Recommendation.

# Magnesium and magnesium alloys — Determination of manganese — Periodate photometric method (Manganese content less than 0,01 %)

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of manganese in magnesium and magnesium alloys which do not contain zirconium, rare earths, or thorium<sup>1)</sup>.

The method is applicable to the determination of manganese content less than 0,01 %.<sup>2)</sup>

## 2 PRINCIPLE

Attack of the sample with sulphuric acid, followed by oxidation with ammonium persulphate.

Oxidation of manganese(II) to manganese(VII) by means of potassium periodate (acidity approximately 2 N), in the presence of phosphoric acid.

Photometric measurement at a wavelength of about 525 nm.

## 3 REAGENTS

For the preparation of solutions and during the analysis, use doubly distilled water.

**3.1 Potassium periodate** ( $\text{KIO}_4$ ).

**3.2 Ammonium persulphate** [ $(\text{NH}_4)_2\text{S}_2\text{O}_8$ ].

**3.3 Sulphuric acid**,  $\rho$  1,26 g/ml, approximately 9 N solution.

Carefully add 25 ml of sulphuric acid ( $\rho$  1,84 g/ml), approximately 35,6 N, to 60 ml of water. After cooling make up the volume to 100 ml and mix.

**3.4 Phosphoric acid**,  $\rho$  1,71 g/ml, approximately 45 N solution.

**3.5 Water free from reducing agents**

Heat to boiling, water acidified with 10 ml per litre of the

sulphuric acid (3.3); add a few crystals of the 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 ( $\text{NaNO}_2$ ) 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 ( $\text{KMnO}_4$ ) in about 200 ml of water and add 40 ml of the sulphuric acid (3.3). Reduce the permanganate solution by adding a few crystals of sodium sulphite or hydrogen peroxide (36 % (m/m)). Boil the solution to remove excess  $\text{SO}_2$  or  $\text{H}_2\text{O}_2$ , 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  $1 \pm 0,001$  g of electrolytic manganese (purity above 99,9 %) with 40 ml of the sulphuric acid (3.3) 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 the sulphuric acid (3.3) and about 100 ml of water. Shake and, after a few minutes, decant the acid solution and introduce into the beaker doubly distilled water. Repeat the decantation and washing with doubly 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**, 0,02 g/l (1 ml contains 0,02 mg of Mn).

Transfer 20,0 ml of the manganese standard solution (3.7) to a 1 000 ml volumetric flask, make up to volume and mix.

1) These special cases are treated in ISO 2353, *Magnesium and its alloys — Determination of manganese in magnesium alloys containing zirconium, rare earths, thorium and silver — Periodate photometric method*.

2) For manganese contents greater than 0,01 %, see ISO 809, *Magnesium and magnesium alloys — Determination of manganese — Periodate photometric method (Manganese content between 0,01 and 0,8 %)*.