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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 810

CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM ALLOYS

PHOTOMETRIC DETERMINATION OF MANGANESE

PERIODATE METHOD

(Manganese content less than 0.01 %)

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 810, *Chemical analysis of magnesium and magnesium alloys – Photometric determination of manganese – Periodate method (Manganese content less than 0.01 %)*, was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys,* the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1957 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1131) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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 opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 810 August 1968

CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM ALLOYS

PHOTOMETRIC DETERMINATION OF MANGANESE

PERIODATE METHOD

(Manganese content less than 0.01 %)

1. SCOPE

This ISO Recommendation describes a photometric method for the determination of manganese in magnesium and magnesium alloys which do not contain zirconium, rare earths, or thorium.

The method is applicable to the determination of manganese content less than 0.01 %.

2. PRINCIPLE

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

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

2.3 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* (KIO_4).

3.2 *Ammonium persulphate* [$(NH_4)_2S_2O_8$].

3.3 *Sulphuric acid, d* = 1.26 (approximately *9 N).*

Carefully add 25 ml of sulphuric acid, *d* = 1.84 (approximately 35.6 N), to water, cool and make up the volume to 100 ml.

3.4 *Phosphoric acid, d* = 1.71 (approximately 45 N).

3.5 *Water free from reducing agents*

Bring to the boil water acidified with 10 ml per litre of sulphuric acid (3.3); add a few crystals of potassium periodate (3.1) and maintain at boiling point for approximately 10 minutes. (The term "water" without qualification indicates doubly distilled water).

3.6 *Sodium nitrite solution,* 20 g per litre.

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

3.7 *Standard manganese solution,* 1 g per litre (1 ml contains 1 mg of manganese).

Either :

3.7.1 In a tall-form beaker of suitable capacity (e.g. 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 (3.3). Reduce the permanganate solution by adding a few crystals of sodium sulphite or by adding hydrogen peroxide (100 to 110 volumes). Boil the solution to eliminate excess of sulphur dioxide or hydrogen peroxide, cool, transfer to a 1000 ml volumetric flask and make up to volume with water.

Or :

3.7.2 In a tall-form beaker of suitable capacity (e.g. 600 ml), dissolve 1 ± 0.001 g of electrolytic manganese (purity ⩾99.9 %) with 40 ml of sulphuric acid (3.3) and approximately 80 ml of water. Boil the solution for several minutes. Cool, transfer to a 1000 ml volumetric flask and make up to volume.

NOTE. – Free the electrolytic manganese of any surface oxidation present by placing several grammes of the metal in a glass beaker of approximately 250 to 300 ml capacity, containing 60 to 80 ml of sulphuric acid (3.3), and approximately 100 ml of water. Stir and after a few minutes decant the acid solution and introduce some doubly distilled water into the beaker. Decant and wash with doubly distilled water several times; finally, place the manganese in acetone and shake. Decant the acetone, dry the metal in a hot air oven at 100 °C for approximately 2 minutes, then allow to cool in a desiccator.

3.8 *Standard manganese solution,* 0.02 g per litre (1 ml contains 0.02 mg of manganese).

Take 20.0 ml of standard manganese solution (3.7), place in a 1000 ml volumetric flask and make up to volume with water.

4. APPARATUS

4.1 *Ordinary laboratory apparatus*

All volumetric apparatus should comply with national standards.

4.2 *Electrophotometer or spectrophotometer* (wavelength about 525 nm).

5. SAMPLING

5.1 **Laboratory sample**

See the appropriate national standard on sampling.

5.2 **Test sample**

Chips not more than 1 mm thick should be obtained from the laboratory sample by milling or drilling.