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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 792

CHEMICAL ANALYSIS OF MAGNESIUM AND ITS ALLOYS

PHOTOMETRIC DETERMINATION OF IRON

(Orthophenanthroline method
applicable to iron content between 0.002 and 0.05 %)

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BRIEF HISTORY

The ISO Recommendation R 792, *Chemical analysis of magnesium and its alloys – Photometric determination of iron (Orthophenanthroline method applicable to iron content between 0.002 and 0.05 %)*, 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 1956 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In June 1966, this Draft ISO Recommendation (No. 965) 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	Korea, Rep of	Sweden
Austria	India	Switzerland
Belgium	Israel	Turkey
Brazil	Italy	U.A.R.
Bulgaria	Japan	United Kingdom
Canada	Netherlands	U.S.A.
Chile	Norway	U.S.S.R.
Czechoslovakia	Poland	Yugoslavia
France	South Africa,	
Germany	Rep. of	
Hungary	Spain	

One Member Body opposed the approval of the Draft :

Ireland

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

ISO Recommendation R 792 July 1968

CHEMICAL ANALYSIS OF MAGNESIUM AND ITS ALLOYS

PHOTOMETRIC DETERMINATION OF IRON

(Orthophenanthroline method
applicable to iron content between 0.002 and 0.05 %)

1. SCOPE

1.1 This ISO Recommendation describes a photometric method for the determination of iron in magnesium and its alloys.

1.2 The method is applicable to the determination of iron content between 0.002 and 0.05 %.

1.3 The method does not apply to the special case of magnesium alloys containing zirconium. In this ISO Recommendation this special case is not treated.

2. PRINCIPLE

2.1 Attack with hydrochloric acid.

2.2 Reduction of ferric iron to bivalent iron by hydroxylammonium chloride.

2.3 Formation in buffered solution between pH 3.5 and pH 4.5 of the orange-red coloured complex, bivalent iron-orthophenanthroline.

2.4 Photometric measurement at a wavelength of about 510 nm.*

3. REAGENTS

3.1 *Hydrochloric acid*, $d = 1.1$ (approximately 6 N).
Take 500 ml of hydrochloric acid, $d = 1.19$ (approximately 12 N), and make up the volume to 1000 ml with water.

3.2 *Hydroxylammonium chloride solution*, 10 g per litre.
Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and make up the volume to 1000 ml.

3.3 *Buffer solution.*
Dissolve 272 g of sodium acetate ($CH_3COONa.3H_2O$) in about 500 ml of water, filter, add 240 ml of glacial acetic acid (CH_3COOH), $d = 1.05$ (approximately 17.4 N), then make up the volume to 1000 ml with water.

* Copper, which may interfere if present in appreciable amounts, is largely eliminated in the attack. Of the elements normally present in aluminium and its alloys, some do not interfere, while others form colourless soluble complexes with orthophenanthroline, which do not absorb at the wavelength at which the photometric measurement is performed.

3.4 *Orthophenanthroline solution*, 10 g per litre.

3.4.1 *Aqueous solution of orthophenanthroline hydrochloride monohydrate*
Dissolve 10 g of orthophenanthroline hydrochloride monohydrate ($C_{12}H_8N_2$. HC1. H_2O) in water, warm slightly in order to complete the solution, cool and make up the volume to 1000 ml.

Alternatively

3.4.2 *Alcoholic solution of orthophenanthroline monohydrate*
Dissolve 10 g of orthophenanthroline monohydrate ($C_{12}H_8N_2$. H_2O) in ethanol (95 %) and make up the volume to 1000 ml with ethanol from the same source.

3.5 *Standard iron solution*, 0.2 g per litre (1 ml contains 0.2 mg of iron).

3.5.1 Dissolve 1.4045 g of ferrous ammonium sulphate $[(NH_4)_2\ Fe(SO_4)_2.6H_2O]$ in a little water and add 20 ml of hydrochloric acid (3.1). Transfer the solution to a 1000 ml volumetric flask and make up the volume to 1000 ml with water.*

Alternatively

3.5.2 Dissolve, by heating in a 100 ml beaker covered with a watch-glass, 0.2860 g of pure ferric oxide (Fe_2O_3), previously calcined at 600 °C, in 30 ml of hydrochloric acid (3.1). After cooling, transfer the solution to a 1000 ml volumetric flask and make up the volume to 1000 ml with water.

3.6 *Standard iron solution*, 0.01 g per litre (1 ml contains 0.01 mg of iron).
Transfer 50.0 ml of the standard iron solution (3.5) to a 1000 ml volumetric flask, then make up the volume to 1000 ml with water. Prepare the solution just before use.

4. APPARATUS

4.1 *Ordinary laboratory equipment*

All volumetric apparatus should comply with national standards. Glassware should be carefully washed with warm hydrochloric acid, thoroughly rinsed with water and finally rinsed with distilled water.

4.2 *Electrophotometer or spectrophotometer* (wavelength about 510 nm).

5. SAMPLING

5.1 **Laboratory sample**

See the appropriate national standard on sampling.

5.2 **Test sample**

Obtain chips not more than 1 mm thick from the laboratory sample by means of a beryllium bronze drill or a tungsten carbide-tipped drill. Pierce a preliminary hole to a depth of 2 to 3 mm and discard the first chips. Continue to drill, withdrawing chips carefully and keeping them in glass bottles or in plastic bags.

* If the effective strength of the ferrous ammonium sulphate is not known, measure it by titration with potassium dichromate and correct accordingly the mass to be taken for the standard iron solution (3.5).