G-41-01
G-41-03

INTERNATIONAL STANDARD ISO 792

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Magnesium and magnesium alloys — Determination of iron — Orthophenanthroline photometric method

First edition — 1973-11-15

UDC 669.721 : 546.72 : 543.42

Ref. No. ISO 792-1973 (E)

Descriptors : magnesium, magnesium alloys, chemical analysis, determination of content, iron, photometry.

Price based on 3 pages

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 792 replaces ISO Recommendation R 792-1968 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys.*

The Member Bodies of the following countries approved the Recommendation :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Ireland

Printed in Switzerland

Magnesium and magnesium alloys — Determination of iron — Orthophenanthroline photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of iron in magnesium and magnesium alloys.

The method is applicable to the determination of iron content between 0,002 and 0,05 %.

The method does not apply to the special case of magnesium alloys containing zirconium. In this International Standard this special case is not treated.

2 PRINCIPLE

Attack with hydrochloric acid. Reduction of iron(III) to iron(II) by hydroxylammonium chloride.

Formation in buffered solution between pH 3,5 and pH 4,5 of the orange-red coloured complex, bivalent iron-orthophenanthroline.

Photometric measurement at a wavelength of about 510 nm.[1]

3 REAGENTS

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

3.1 Hydrochloric acid, ρ 1,1 g/ml, approximately 6 N solution.

Take 500 ml of hydrochloric acid (ρ 1,19 g/ml), approximately 12 N, and make up the volume to 1 000 ml with water.

3.2 Hydroxylammonium chloride, 10 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH \cdot HCl$) in water and make up the volume to 1 000 ml.

3.3 Buffer solution

Dissolve 272 g of sodium acetate ($CH_3COONa \cdot 3H_2O$) in about 500 ml of water, filter, add 240 ml of glacial acetic acid (CH_3COOH). (ρ 1,05 g/ml), approximately 17,4 N, then make up the volume to 1 000 ml with water.

3.4 Orthophenanthroline, 10 g/l solution.

3.4.1 Orthophenanthroline hydrochloride monohydrate, aqueous solution.

Dissolve 10 g of orthophenanthroline hydrochloride monohydrate ($C_{12}H_8N_2 \cdot HCl \cdot H_2O$) in water, warm slightly in order to complete the solution, cool and make up the volume to 1 000 ml.

Alternatively

3.4.2 Orthophenanthroline monohydrate, ethanolic solution.

Dissolve 10 g of orthophenanthroline monohydrate ($C_{12}H_8N_2 \cdot H_2O$) in ethanol 95 % (*V/V*) and make up the volume to 1 000 ml with ethanol from the same source.

3.5 Iron, 0,02 g/l standard solution (1 ml contains 0,2 mg of iron).

3.5.1 Dissolve 1,404 5 g of ferrous ammonium sulphate [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$] in a little water and add 20 ml of hydrochloric acid (3.1). Transfer the solution to a 1 000 ml volumetric flask and make up the volume to 1 000 ml with water.[2]

Alternatively

3.5.2 Dissolve, by heating, in a 100 ml beaker covered with a watch-glass, 0,286 0 g of pure ferric oxide (Fe_2O_3), previously calcined at 600 °C, in 30 ml of the hydrochloric acid (3.1). After cooling, transfer the solution to a 1 000 ml volumetric flask and make up the volume to 1 000 ml with water.

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[1] 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.

[2] 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 iron standard solution (3.5).

3.6 Iron, 0,01 g/l standard solution (1 ml contains 0,01 mg of iron).

Transfer 50,0 ml of the iron standard solution (3.5) to a 1 000 ml volumetric flask, then make up the volume to 1 000 ml with water. Prepare the solution just before use.

4 APPARATUS

4.1 Ordinary laboratory equipment

Glassware shall be carefully washed with warm hydrochloric acid, thoroughly rinsed with water and finally rinsed with distilled water.

4.2 Spectrophotometer, or

4.3 Photoelectric absorptiometer.

5 SAMPLING

5.1 Laboratory sample[1]

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.

6 PROCEDURE

6.1 Test portion

Mass of test portion :

0,5 ± 0,001 g for iron contents between 0,01 and 0,05 %

1 ± 0,001 g for iron contents between 0,002 and 0,01 %

6.2 Preparation of the calibration curve

Introduce into a series of fifteen 100 ml volumetric flasks 0 (compensating solution) – 2,0 – 4,0 – 6,0 – 8,0 – 10,0 – 12,0 – 14,0 – 16,0 – 18,0 – 20,0 – 22,0 – 24,0 – 26,0 and 28,0 ml respectively of the iron standard solution (3.6) containing 0,01 mg of iron per millilitre.

Dilute each solution to about 50 ml, add 4 ml of the hydroxylammonium chloride solution (3.2), 15 ml of the buffer solution (3.3) and 2 ml of the orthophenanthroline solution (3.4), make up the volume to 100 ml and mix. After 30 min, carry out the photometric measurements at the maximum of the absorption curve (wavelength about 510 nm), after having adjusted the instrument to zero absorbance against the compensation solution.

Plot a graph having, for example, the amount of iron contained in 100 ml as abscissae, against the corresponding values of absorbance as ordinates.

6.3 Blank test

Introduce into a 100 ml tall-form beaker the amount of the hydrochloric acid (3.1) used for the attack of the test portion (15 ml for a test portion of 1 g and 7,5 ml for a test portion of 0,5 g). Evaporate almost to dryness (about 0,2 ml) on a water bath. Take up with a little water and transfer the solution and the washings into a 100 ml volumetric flask. Dilute to 50 ml with water and proceed according to the directions given in 6.4.2.

6.4 Determination

6.4.1 *Attack of the test portion*

Place the test portion in a beaker of suitable capacity (for example 150 ml) provided with a watch-glass; add 10 ml of water, then, in small portions, the hydrochloric acid (3.1) (15 ml for a test portion of 1 g and 7,5 ml for a test portion of 0,5 g). Heat to complete the attack. If a residue remains, filter through a medium texture filter and wash the beaker and the residue 5 or 6 times with warm water. On a water bath evaporate the solution to a syrupy consistency (i.e a volume of about 4 ml for a test portion of 0,5 g and about 8 ml for a test portion of 1 g). Add 10 ml of water and transfer the solution into a 100 ml volumetric flask. Wash the beaker with small portions of water, collecting the washings in the same volumetric flask, and dilute to about 50 ml with water. Allow to cool.

6.4.2 *Colour reaction*

Proceed as follows, according to whether the analysis is of pure magnesium or of a magnesium alloy :

a) Pure magnesium

Into the volumetric flask containing the solution of the test portion, add 4 ml of the hydroxylammonium chloride solution (3.2), 15 ml of the buffer solution (3.3) and 2 ml of the orthophenanthroline solution (3.4). Make up the volume to 100 ml with water and mix.

b) Magnesium alloys

Into the volumetric flask containing the solution of the test portion, add 4 ml of the hydroxylammonium chloride solution (3.2), 15 ml of the buffer solution

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[1] The sampling of magnesium and magnesium alloys will form the subject of a future International Standard.

(3.3), and 6 ml of the orthophenanthroline solution (3.4) in the case of magnesium alloys with zinc contents lower than 2 %, or 12 ml of the orthophenanthroline solution (3.4) in the case of magnesium alloys with zinc contents between 2 and 4 %. Make up the volume to 100 ml with water and mix.

6.4.3 *Photometric measurements*

After 30 min, carry out the photometric measurements under the same conditions as were used for plotting the calibration curve, after having adjusted the instrument to zero absorbance against the blank test solution.

7 EXPRESSION OF RESULTS

By means of the calibration graph, determine the mass of iron (in milligrams) corresponding to the value of the photometric measurement of the solution of the test portion.

Calculate the iron content, as a percentage by mass, by the formula

$$\text{Fe}\ \%\ (m/m) = \frac{m_1}{10\, m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass of iron, in milligrams, found in the solution of the test portion.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.