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Chemical analysis of magnesium and its alloys — Determination of rare earths — Gravimetric method

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FOREWORD

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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 2355 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys.*

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

Austria	Ireland	South Africa, Rep. of
Belgium	Italy	Sweden
Canada	Japan	Switzerland
Egypt, Arab Rep. of	Korea, Rep. of	Turkey
Finland	Netherlands	United Kingdom
France	New Zealand	U.S.A.
Germany	Norway	U.S.S.R.
Hungary	Poland	
India	Romania	

No Member Body expressed disapproval of the document.

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Chemical analysis of magnesium and its alloys – Determination of rare earths – Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of rare earths in magnesium alloys.

The method is applicable to the determination of contents of rare earths between 0.2 and 10 %, in the absence of thorium.

2 PRINCIPLE

2.1 Dissolution with hydrochloric acid.

2.2 Precipitation of zirconium by ammonia and filtration.

2.3 Preliminary separation of rare earths in the filtrate in the form of sebacates, in ammoniacal medium.

2.4 Dissolution of the two precipitates and re-precipitation of rare earths in the form of oxalates.

2.5 Ignition and weighing of rare earth oxides.

3 REAGENTS

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

3.1 Ammonium chloride (NH_4Cl).

3.2 Hydrochloric acid (ρ 1.19 g/ml) approximately 12 N solution.

3.3 Ammonia (ρ 0.95 g/ml) approximately 7 N solution.

Dilute 50 ml of ammonia solution (ρ 0.90 g/ml) approximately 14 N, with water, make up the volume to 100 ml and mix.

3.4 Ammonia (ρ 0.97 g/ml) approximately 3.5 N solution.

Dilute 25 ml of ammonia solution (ρ 0.90 g/ml) approximately 14 N, with water, make up the volume to 100 ml and mix.

3.5 Ammonia, 0.3 N solution approximately.

Dilute 2 ml of ammonia solution (ρ 0.90 g/ml) approximately 14 N, with water, make up the volume to 100 ml and mix.

3.6 Hydrogen peroxide (ρ 1.12 g/ml approximately) 33 % (m/m) solution approximately.

3.7 Nitric acid/hydrogen peroxide solution

Dilute 30 ml of hydrogen peroxide (3.6) with 150 ml of water and add 30 ml of nitric acid, (ρ 1.40 g/ml) approximately 15 N solution.

3.8 Sebacic acid, ammoniacal solution 50 g/l.

Dissolve 50 g of sebacic acid [$HOOC(CH_2)_8COOH$] in 400 ml of ammonia solution (ρ 0.90 g/ml) 14 N approximately, and 300 ml of water. Filter, dilute to 1 000 ml with water.

3.9 Oxalic acid, solution saturated at room temperature.

Dissolve 150 g of oxalic acid in 1 000 ml of hot water. Allow to cool and filter.

3.10 Oxalic acid wash solution

Dilute 70 ml of oxalic acid (3.9) to 500 ml with water.

3.11 Bromophenol blue alkaline solution 4 g/l.

Place 0.4 g of bromophenol blue in a mortar, add 8.25 ml of sodium hydroxide solution 5 g/l and crush to complete dissolution. Transfer quantitatively to a 100 ml volumetric flask, make up to volume with water and mix.

4 APPARATUS

4.1 Ordinary laboratory equipment.

All volumetric vessels should be checked in accordance with the official standards.

4.2 pH-meter.

5 SAMPLING

5.1 Laboratory sample [1)]

5.2 Test sample

Chips of thickness less than or equal to 1 mm obtained by drilling or milling.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, about 3 g of the test sample (5.2) for contents of rare earths between 0.2 and 2 %, 2 g for contents of rare earths between 2 and 5 % or 1 g for contents of rare earths between 5 and 10 %.

6.2 Determination

6.2.1 *Attack and preparation of test solution*

Place the test portion in a beaker of suitable capacity (for example 400 ml), fitted with a watch glass, add 75 ml of water then, in small portions, 8.5 ml of hydrochloric acid (3.2) for each gram of test portion.

When the reaction is completed, boil the solution for a few minutes. If a residue remains, filter through a medium texture filter, wash the beaker and the residue four to five times with hot water, adding the washings to the test solution (discard the residue). Make up the volume to about 100 ml, either by dilution or by evaporation, and then cool.

NOTE — In the case of analysis of alloys containing silver, line the filter with a little cellulose pulp before filtering.

6.2.2 *Precipitation of zirconium hydroxide*

Add to the solution 3 drops of bromophenol blue solution (3.11) and neutralize first with ammonia solution (3.3) until the indicator just changes to purple, and then with ammonia solution (3.4). Heat to boiling, remove the beaker and allow to settle for 5 min stirring from time to time.

Filter through a rapid filter and thoroughly wash the precipitate with boiling water. Reserve the filtrate (containing most of the rare earths) and the washings ensuring that the total volume does not exceed 250 ml (Filtrate A).

Dissolve the precipitate on the filter with 10 ml of boiling nitric acid/hydrogen peroxide solution (3.7), added in small portions, collecting the filtrate and washings in the beaker already used for the precipitation. After each addition of acid solution (3.7), wash with boiling water. Finally, complete the washing of the filter with hot water (five or six washings). Evaporate the solution to about 25 ml and reserve (Filtrate B).

6.2.3 *First precipitation of rare earths*

Add to filtrate A (see 6.2.2) 10 g of ammonium chloride (3.1). Checking by means of the pH-meter (4.2), adjust the pH of the solution to 8.5, by adding first ammonia solution (3.3), then ammonia solution (3.4). Finally add 10 ml of ammonia solution (3.4) in excess.

Heat on a hot plate at about 90 °C, remove the beaker and while stirring add 20 ml of sebacic acid solution (3.8). Allow to settle for 15 min, stirring from time to time.

Filter through a medium texture filter and thoroughly wash with ammonia solution (3.5). If zinc is present, wash the precipitate once more with 20 ml of ammonia solution (3.3).

Place the filter with the precipitate in a porcelain crucible, previously ignited at 950 °C, cooled in a desiccator and weighed, in an electric oven controlled at 750 to 800 °C, for about 30 min. Remove the crucible from the oven and allow to cool.

6.2.4 *Precipitation of rare earth oxalates*

Transfer quantitatively the contents of the crucible to the beaker containing filtrate B (see 6.2.2). Heat the solution and then add 2 or 3 drops of hydrogen peroxide (3.6) to complete the dissolution of rare earth oxides.

Remove the beaker from the source of heat, wash the walls and dilute to about 125 ml. Add slowly, while stirring, 25 ml of oxalic acid solution (3.9). Place the beaker on a boiling water bath for 30 min. Then allow to stand for 12 h (one night) at ambient temperature.

6.2.5 *Filtering, washing and weighing*

Filter the precipitate on a close texture filter and thoroughly wash with the wash solution (3.10).

Place the filter and the precipitate in the porcelain crucible, already used (see 6.2.3), gently heat the filter at a temperature of approximately 500 °C until complete combustion of the filter, then ignite at 950 °C to constant mass. Weigh, after cooling in a desiccator containing anhydrous magnesium perchlorate.

7 EXPRESSION OF RESULTS

The content of rare earths is given, as a percentage by mass, by the following formula :

$$\frac{m_1 \times F \times 100}{m_0}$$

1) An International Standard relating to sampling from supply batches will be studied as soon as Technical Committee ISO/TC 69, *Application of statistical methods*, has fixed the general principles to be adopted.

where

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

m_1 is the mass, in grams, of rare earth oxides weighed;

F is the *conventional* conversion factor for mischmetal = 0.832.

NOTE — When the rare earths are introduced in a composition different from that of mischmetal the following factors should be used.

La	0.852 7
Ce	0.814 1
Pr	0.827 7
Nd	0.857 4
Didymium	0.853

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results, and the form in which they are expressed;

c) any special details noted during the test;

d) all operations not specified in this International Standard, or any optional operations, which may have affected the results.

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