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INTERNATIONAL STANDARD 791

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Magnesium alloys — Determination of aluminium — 8-hydroxyquinoline gravimetric method

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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 791 replaces ISO Recommendation R 791-1968 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys.*

The Member Bodies of the following countries approved the Recommendation :

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

No Member Body expressed disapproval of the Recommendation.

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Magnesium alloys — Determination of aluminium — 8-hydroxyquinoline gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of aluminium in mangesium alloys other than those containing zirconium, thorium or rare earths.

The method is applicable to the determination of aluminium content between 1,5 and 12,0 %.

2 PRINCIPLE

Attack with hydrochloric acid. Precipitation of the aluminium by ammonium benzoate in a reducing acetic medium.

Dissolution of the precipitate and reprecipitation of the aluminium as oxyquinolate in a buffered acetate medium, or in the presence of potassium cyanide.

Filtration, washing, drying and weighing of the precipitate.

3 REAGENTS

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

3.1 Sodium sulphite ($Na_2SO_3.7H_2O$).

3.2 Hydrochloric acid, ρ 1,19 g/ml, approximately 12 N solution.

3.3 Hydrochloric acid, ρ 1,05 g/ml, approximately 3 N solution.

Take 25 ml of the hydrochloric acid (3.2) and make up the volume to 100 ml with water.

3.4 Nitric acid, ρ 1,33 g/ml, approximately 11 N solution.

Take 75 ml of nitric acid (ρ 1,40 g/ml), approximately 15 N and make up the volume to 100 ml with water.

3.5 Ammonia, ρ 0,90 g/ml, approximately 14,4 N solution.

3.6 Ammonia, ρ 0,97 g/ml, approximately 3,6 N solution.

Take 25 ml of the ammonia solution (3.5) and make up the volume to 100 ml with water.

3.7 Complexing solution

Dissolve in a little water 50 g of hydroxylammonium chloride ($NH_2OH.HCl$), 50 g of ammonium chloride (NH_4Cl), 50 ml of glacial acetic acid (ρ 1,05 g/ml), approximately 17,4 N, and make up the volume to 1 000 ml with water.

3.8 Ammonium benzoate, 100 g/l solution.

Dissolve 100 g of ammonium benzoate ($C_6H_5COONH_4$) in warm water, add 0,001 g of thymol and, after cooling, make up the volume to 1 000 ml with water.

3.9 Ammonium benzoate, wash solution

Dilute 100 ml of ammonium benzoate solution (3.8) with 900 ml of water and add 20 ml of glacial acetic acid (ρ 1,05 g/ml), approximately 17,4 N.

3.10 Tartaric acid, 500 g/l solution.

Dissolve 500 g of tartaric acid [$HOOC(CHOH)_2$ COOH] in water and make up the volume to 1 000 ml.

3.11 Acetic acid, ρ 1,01 g/ml, approximately 1,7 N solution.

Take 100 ml of glacial acetic acid (ρ 1,05 g/ml), approximately 17,4 N, and make up the volume to 1 000 ml with water.

3.12 8-hydroxyquinoline, 20 g/l acetic acid solution.

Dissolve 20 g of 8-hydroxyquinoline ($HO.C_6H_3N$: CH.CH : CH) in 80 ml of glacial acetic acid, ρ 1,05 g/ml, approximately 17,4 N and make up the volume to 1 000 ml with water.

Store in a dark glass bottle.

3.13 Ammonium acetate, 600 g/l solution.

Dissolve 600 g of ammonium acetate (CH_3COONH_4) in water and make up the volume to 1 000 ml.

3.14 Potassium cyanide, 200 g/l solution.

Dissolve 20 g of potassium cyanide (KCN) in water and make up the volume to 100 ml.

3.15 Bromephenol blue, ethanolic solution

Dissolve 0,20 g of bromophenol blue in 100 ml of ethanol 95 % (*V/V*).

3.16 Neutral red, ethanolic solution

Dissolve 0,05 g of neutral red in 100 ml of ethanol 95 % (*V/V*).

4 APPARATUS

Ordinary laboratory equipment.

5 SAMPLING

5.1 Laboratory sample[1)]

5.2 Test sample

Chips not more than 1 mm thick obtained by drilling or milling.

6 PROCEDURE

6.1 Test portion

Mass of test portion :

0,5 ± 0,001 g for aluminium contents between 1,5 and 5 %

1 ± 0,001 g for aluminium contents between 5 and 12 %

6.2 Determination

6.2.1 *Attack of the test portion and preparation of the main solution*

Place the test portion in a beaker of suitable capacity (for example 250 ml), provided with a watch-glass; add 25 ml of water and then, in small portions, the hydrochloric acid (3.2) (5 ml for a test portion of 0,5 g, 10 ml for a test portion of 1 g) followed by 2 ml of the nitric acid (3.4). Heat to complete the attack. If a residue remains, filter through a medium texture filter, wash the beaker and the residue 5 or 6 times with hot water and add the washings to the filtrate (discard the residue). Boil the solution for 1 to 2 min. Cool, wash the watch-glass and the sides of the beaker with a little water, then make up the volume to approximately 50 ml.

For a test portion of 1 g, transfer the solution to a 250 ml volumetric flask, make up the volume to 250 ml with water, mix and transfer 50,0 ml of this solution to a beaker of suitable capacity (for example 250 ml).

For a test portion of 0,5 g carry out the analysis on the whole of the solution.

6.2.2 *First precipitation of aluminium*

Add to the solution 40 ml of water, 2 or 3 drops of the bromophenol blue ethanolic solution (3.15) and neutralize with the ammonia solution (3.6) until the indicator becomes violet. Then add 20 ml of the complexing solution (3.7) and 20 ml of the ammonium benzoate solution (3.8). Heat the solution to boiling while stirring and keep boiling gently for 5 min, then filter through a medium texture filter. Wash the beaker and the precipitate 8 to 10 times with boiling ammonium benzoate wash solution (3.9). (Discard the filtrate.)

6.2.3 *Precipitation of aluminium oxyquinolate*

Dissolve the precipitate from the filter with small portions of a boiling solution, prepared by mixing 50 ml of the hydrochloric acid (3.3) and 10 ml of the tartaric acid solution (3.10). Wash the filter with warm water and collect the solution and the washings in the first beaker. Transfer the solution to a beaker of suitable capacity (for example 400 ml). Add 1 g of the sodium sulphite (3.1), a few drops of the neutral red ethanolic solution (3.16) and, carefully, the ammonia solution (3.5), until the indicator becomes yellow.

Then proceed as indicated in paragraph a) or in paragraph b) below.

a) Precipitation in a buffered acetate medium

Dilute the solution to about 200 ml then heat to about 70 °C. Add the acetic acid (3.11) until the indicator becomes red then, while stirring, 40 ml of the 8-hydroxyquinoline solution (3.12) and 50 ml of the ammonium acetate solution (3.13). Allow the precipitate to settle at about 70 °C for 30 min.

b) Precipitation in the presence of potassium cyanide

Dilute the solution to about 250 ml, then add the acetic acid (3.11) until the indicator becomes red. Add 10 ml of the potassium cyanide solution (3.14) and heat in a fume cupboard to about 70 °C. Add slowly, while stirring the solution, 40 ml of the 8-hydroxyquinoline solution (3.12). Allow the precipitate to settle at about 70 °C for 30 min.

6.2.4 *Filtration, washing, drying and weighing of the aluminium oxyquinolate*

Filter the precipitate on a tared sintered glass crucible of porosity between 3 and 15 µm, by applying weak suction, then wash 6 to 8 times with hot water. Dry to constant mass in an oven at 130 °C. Cool in a desiccator and weigh.

1) The sampling of magnesium alloys will form the subject of a future International Standard.

7 EXPRESSION OF RESULTS

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

$$\text{Al \% } (m/m) = \frac{m_1 \times 0{,}058\,7 \times 100 \times R}{m_0}$$

where

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

m_1 is the mass, in grams, of the aluminium oxyquinolate corresponding to the aliquot taken;

R is the ratio of the volume of the main solution to the volume of the aliquot taken;

0,058 7 is the conversion factor of aluminium oxyquinolate to aluminium.

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.