International Standard 5196/1

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Magnesium alloys — Determination of thorium — Part 1 : Gravimetric method

Alliages de magnésium — Dosage du thorium — Partie 1 : Méthode gravimétrique

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Italy	Spain
China	Japan	Sweden
Czechoslovakia	Norway	United Kingdom
France	Philippines	USSR
Germany, F. R.	Portugal	Yugoslavia
Hungary	Romania	

No member body expressed disapproval of the document.

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Magnesium alloys — Determination of thorium — Part 1 : Gravimetric method

1 Scope and field of application

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

The method is applicable to products having thorium contents between 0,2 and 5,0 % (*m/m*).

2 Principle

Dissolution of a test portion in hydrochloric acid.

Preliminary separation of the thorium in the form of its benzoate.

Dissolution of the precipitate and reprecipitation of the thorium in the form of its oxalate. Calcination and weighing of the thorium oxide.

3 Reagents

During the analysis, use only reagents of recognized analytical quality and only distilled water or water of equivalent purity.

3.1 Hydroxylammonium chloride ($NH_2OH.HCl$).

3.2 Ammonium chloride (NH_4Cl).

3.3 Hydrochloric acid (ϱ approximately 1,19 g/ml), 38 % (*m/m*) or approximately 12 mol/l solution.

3.4 Hydrochloric acid (ϱ approximately 1,05 g/ml) or approximately 3 mol/l solution.

Dilute 250 ml of the hydrochloric acid solution (3.3) with water, make up the volume to 1 000 ml and mix.

3.5 Ammonium hydroxide solution (ϱ approximately 0,97 g/ml).

Dilute 250 ml of ammonium hydroxide solution (ϱ approximately 0,91 g/ml) with water, make up the volume to 1 000 ml and mix.

3.6 Benzoic acid, 20 g/l solution.

Dissolve 20 g of benzoic acid (C_6H_5COOH) in hot water. Allow to cool, filter if necessary, make up the volume to 1 000 ml and mix.

3.7 Benzoic acid, 2,5 g/l solution.

Dissolve 2,5 g of benzoic acid (C_6H_5COOH) in hot water, make up the volume to 1 000 ml and mix.

3.8 Oxalic acid, saturated solution at ambient temperature.

Dissolve 150 g of oxalic acid [$(COOH)_2.2H_2O$] in 1 000 ml of hot water. Allow to cool and filter.

3.9 Oxalic acid washing solution.

Dilute 70 ml of the oxalic acid solution (3.8) to 500 ml with water.

3.10 Bromophenol blue, 4 g/l alkaline solution.

Place 0,4 g of bromophenol blue in a mortar, add 8,25 ml of 5 g/l sodium hydroxide solution and crush until completely dissolved. Transfer quantitatively into a 100 ml one-mark volumetric flask, make up to the mark with water and mix.

4 Apparatus

Ordinary laboratory apparatus.

5 Sampling

5.1 Laboratory sample[1)]

5.2 Test sample

Chips having a thickness no greater than 1 mm, obtained by milling or drilling the laboratory sample.

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

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, approximately 5 g of the test sample (5.2) for thorium contents between 0,2 and 1,5 % (m/m), or 3 g for thorium contents between 1,5 and 3,0 % (m/m), or 2 g for thorium contents between 3,0 and 5,0 % (m/m).

6.2 Determination

6.2.1 Preparation of the test solution

Place the test portion (6.1) in a 400 ml beaker fitted with a watch glass, add 50 ml of water and then, in small amounts, add 7,5 ml of the hydrochloric acid solution (3.3) for each gram of test portion.

Once the reaction has stopped, boil the solution for a few minutes. If there is a residue, filter through a close textured filter paper, and wash the beaker and the residue with hot water adding the washings to the test solution (discard the residue). Bring the volume to approximately 100 ml, either by dilution or by evaporation and then cool.

NOTE — For the analysis of alloys containing silver, line the filter, before filtering, with a little paper pulp.

6.2.2 First precipitation of thorium

If rare earth elements are present, add 1 g of the hydroxylammonium chloride (3.1).

To the solution, add three drops of the bromophenol blue solution (3.10) and neutralize, until the indicator turns violet, either with the ammonium hydroxide solution (3.5) or with the hydrochloric acid solution (3.4).

Add 10 g of the ammonium chloride (3.2) and bring the solution to the boil. While stirring, add 100 ml of boiling benzoic acid solution (3.6) and continue to heat for 10 min.

Leave for a few minutes (do not allow the solution to cool), filter through a medium textured filter paper and wash carefully with the benzoic acid solution (3.7).

6.2.3 Precipitation of thorium oxalate

Quantitatively transfer the precipitate into the original beaker with 50 ml of boiling water. Wash the filter with 10 ml of the hydrochloric acid solution (3.4) and 50 ml of boiling water. Bring to the boil. Remove the beaker from the heat, wash the sides and dilute the solution to approximately 125 ml. Slowly add, while stirring, 25 ml of the oxalic acid solution (3.8). Leave for 12 h (one night) at ambient temperature.

6.2.4 Filtration, washing and weighing

Filter the precipitate on a close textured filter paper and wash carefully with the washing solution (3.9).

Place the filter paper and precipitate in a porcelain crucible which has been calcined beforehand at 950 °C, incinerate the filter paper moderately at a temperature of approximately 500 °C until complete combustion of the filter paper, then calcine at 950 °C to constant mass. Weigh after cooling in a desiccator containing anhydrous magnesium perchlorate.

7 Expression of results

The thorium (Th) content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times 0{,}878\,8 \times 100}{m_0}$$

where

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

m_1 is the mass, in grams, of thorium oxide obtained;

0,878 8 is the conversion factor from thorium oxide to thorium.

8 Test report

The test report shall include the following information :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noticed during the determination;

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