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Magnesium and magnesium alloys — Determination of silicon — Spectrophotometric method with the reduced silicomolybdic complex

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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 1975 replaces ISO Recommendation R 1975-1971 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*.

The Member Bodies of the following countries approved the Recommendation :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Iran	Sweden
Canada	Israel	Switzerland
Chile	Italy	Thailand
Egypt, Arab Rep. of	Japan	United Kingdom
France	Netherlands	U.S.A.
Germany	New Zealand	
Greece	Norway	

No Member Body expressed disapproval of the Recommendation.

Magnesium and magnesium alloys — Determination of silicon — Spectrophotometric method with the reduced silicomolybdic complex

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method using the reduced silicomolybdic complex for the determination of silicon in magnesium and magnesium alloys.

This method is applicable to the determination of silicon content between 0,01 and 0,6 %.

The method does not apply to the special case of magnesium alloys containing rare earths or thorium (this case is being studied).

2 PRINCIPLE

Attack by sulphuric acid in the presence of bromine water. Complexing of the silicon by potassium fluoride and decomplexing by boric acid.

Formation of the oxidized silicomolybdic complex (yellow) under clearly defined conditions of acidity.

Selective reduction of the complex in a sulphuric acid medium of high acidity and in the presence of tartaric acid.

Spectrophotometric measurement of the blue-coloured complex at a wavelength of about 810 nm.

3 REAGENTS

For the analysis, use only doubly distilled water.

Keep all the reagent solutions, with the exception of the bromine water (3.1), in plastics bottles.

3.1 Bromine water saturated at ambient temperature.

3.2 Sulphuric acid, ρ 1,29 g/ml, approximately 10 N solution.

Carefully add 280 ml of sulphuric acid (ρ 1,84 g/ml), approximately 35,6 N, to approximately 700 ml of water. After cooling, make up the volume to 1 000 ml and mix.

3.3 Sulphuric acid, ρ 1,03 g/ml, approximately 1 N solution.

Dilute 100 ml of the sulphuric acid (3.2) with water and make up the volume to 1 000 ml.

3.4 Magnesium, 10 g/l solution.

Weigh, to within 0,01 g, 10 g of pure magnesium (purity greater than 99,9 %) and place in a tall-form beaker of suitable capacity (for example 600 ml). Add approximately 200 ml of water and, in small portions, 120 ml of the sulphuric acid (3.2). When the reaction is completed, boil the solution for 5 min. Cool and transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume and mix.

3.5 Potassium fluoride, 50 g/l solution.

Dissolve 50 g of potassium fluoride (KF) in water, filter if necessary, and make up the volume to 1 000 ml.

3.6 Boric acid, solution saturated at 20 °C (approximately 40 g/l).

Dissolve 40 g of boric acid (H_3BO_3) in hot water, dilute to approximately 900 ml, cool and make up the volume to 1 000 ml.

3.7 Molybdic solution

Use either of the solutions indicated below :

3.7.1 Sodium molybdate, 195 g/l solution.

Dissolve 19,5 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in hot water, and after cooling make up the volume to 100 ml.

The pH of this solution should be about 7.

3.7.2 Ammonium molybdate, 140 g/l solution.

Dissolve 14,0 g of ammonium molybdate [$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$] in lukewarm water, and make up the volume to 100 ml.

Prepare these solutions immediately before use.

3.8 Tartaric acid, 200 g/l solution.

Dissolve 200 g of tartaric acid ($\text{C}_4\text{H}_6\text{O}_6$) in water, and make up the volume to 1 000 ml.

3.9 Ascorbic acid, 20 g/l solution.

Dissolve 2 g of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in water, and make up the volume to 100 ml.

Use a freshly prepared solution.