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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 791

CHEMICAL ANALYSIS OF MAGNESIUM AND ITS ALLOYS

GRAVIMETRIC DETERMINATION OF ALUMINIUM
IN MAGNESIUM ALLOYS

(Aluminium content between 1.5 and 12.0 %)

1st EDITION

July 1968

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BRIEF HISTORY

The ISO Recommendation R 791, *Chemical analysis of magnesium and its alloys – Gravimetric determination of aluminium in magnesium alloys (Aluminium content between 1.5 and 12.0 %)*, was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1956 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In June 1966, this Draft ISO Recommendation (No. 964) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1968, to accept it as an ISO RECOMMENDATION.

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(Aluminium content between 1.5 and 12.0 %)

1. SCOPE

- 1.1 This ISO Recommendation describes a gravimetric method for the determination of aluminium in magnesium alloys other than those containing zirconium, thorium or rare earths.
- 1.2 The method is applicable to the determination of aluminium content between 1.5 and 12.0 %.

2. PRINCIPLE

- 2.1 Attack with hydrochloric acid.
- 2.2 Precipitation of the aluminium by ammonium benzoate in a reducing acetic medium.
- 2.3 Dissolution of the precipitate and reprecipitation of the aluminium as oxyquinolate in a buffered acetate medium, or in the presence of potassium cyanide.
- 2.4 Filtration, washing, drying and weighing of the precipitate.

3. REAGENTS

- 3.1 *Sodium sulphite* ($\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$).
- 3.2 *Hydrochloric acid*, $d = 1.19$ (approximately 12 N).
- 3.3 *Hydrochloric acid*, $d = 1.05$ (approximately 3 N).
Take 25 ml of hydrochloric acid, $d = 1.19$ (approximately 12 N), and make up the volume to 100 ml with water.
- 3.4 *Nitric acid*, $d = 1.33$ (approximately 11 N).
Take 75 ml of nitric acid, $d = 1.40$ (approximately 15 N), and make up the volume to 100 ml with water.
- 3.5 *Ammonia solution*, $d = 0.90$ (approximately 14.4 N).
- 3.6 *Ammonia solution*, $d = 0.97$ (approximately 3.6 N).
Take 25 ml of ammonia solution, $d = 0.90$ (approximately 14.4 N), and make up the volume to 100 ml with water.
- 3.7 *Complexing solution*.
Dissolve in a little water 50 g of hydroxylammonium chloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$), 50 g of ammonium chloride (NH_4Cl), 50 ml of glacial acetic acid, $d = 1.05$ (approximately 17.4 N), and make up the volume to 1000 ml with water.

- 3.8 *Ammonium benzoate solution*, 100 g per litre.
Dissolve 100 g of ammonium benzoate ($\text{C}_6\text{H}_5\text{COONH}_4$) in warm water, add 0.001 g of thymol and, after cooling, make up the volume to 1000 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, $d = 1.05$ (approximately 17.4 N).
- 3.10 *Tartaric acid solution*, 500 g per litre.
Dissolve 500 g of tartaric acid $[\text{HOOC}(\text{CHOH})_2\text{COOH}]$ in water and make up the volume to 1000 ml.
- 3.11 *Acetic acid*, $d = 1.01$ (approximately 1.7 N).
Take 100 ml of glacial acetic acid, $d = 1.05$ (approximately 17.4 N), and make up the volume to 1000 ml with water.
- 3.12 *Acetic acid solution of 8-hydroxyquinoline*, 20 g per litre.
Dissolve 20 g of 8-hydroxyquinoline ($\text{HO.C}_6\text{H}_3\text{N} : \text{CH.CH} : \text{CH}$) in 80 ml of glacial acetic acid, $d = 1.05$ (approximately 17.4 N), and make up the volume to 1000 ml with water.
(Store in a dark glass bottle).
- 3.13 *Ammonium acetate solution*, 600 g per litre.
Dissolve 600 g of ammonium acetate ($\text{CH}_3\text{COONH}_4$) in water and make up the volume to 1000 ml.
- 3.14 *Potassium cyanide solution*, 200 g per litre.
Dissolve 20 g of potassium cyanide (KCN) in water and make up the volume to 100 ml.
- 3.15 *Bromophenol blue alcoholic solution*.
Dissolve 0.20 g of bromophenol blue in 100 ml of ethanol (95 %).
- 3.16 *Neutral red alcoholic solution*.
Dissolve 0.05 g of neutral red in 100 ml of ethanol (95 %).

4. APPARATUS

Ordinary laboratory equipment.

All volumetric apparatus should comply with national standards.

5. SAMPLING

5.1 Laboratory sample

See the appropriate national standard on sampling.

5.2 Test sample

Chips not more than 1 mm thick should be obtained from the laboratory sample 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 %.