

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION R 807

CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM ALLOYS

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**POLAROGRAPHIC DETERMINATION OF ZINC**

(Zinc content between 0.1 and 4 %)

[ISO/R 807:1968](#)

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## BRIEF HISTORY

The ISO Recommendation R 807, *Chemical analysis of magnesium and magnesium alloys – Polarographic determination of zinc (Zinc content between 0.1 and 4 %)*, 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 1957 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1128) 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	India	South Africa,
Austria	Ireland	Rep. of
Belgium	Israel	Spain
Brazil	Italy	Switzerland
Canada	Japan	Sweden
Chile	Korea, Rep. of	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	U.S.A.
Germany	Norway	U.S.S.R.
Hungary	Poland	Yugoslavia

One Member Body opposed the approval of the Draft :

United Kingdom

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1958, to accept it as an ISO RECOMMENDATION.

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## CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM ALLOYS

## POLAROGRAPHIC DETERMINATION OF ZINC

(Zinc content between 0.1 and 4 %)

## 1. SCOPE

This ISO Recommendation describes a polarographic method for the determination of zinc in magnesium and its alloys which do not contain zirconium.

The method is applicable to the determination of zinc content between 0.1 and 4 %.

## 2. PRINCIPLE

Polarographic determination in an ammonium citrate and ammonium chloride medium at approximately pH 9. Zinc is reduced at about  $-1.2$  volt.

## 3. REAGENTS

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- 3.1 *Ammonium chloride* ( $\text{NH}_4\text{Cl}$ ).
- 3.2 *Pure magnesium* (99.99 %).
- 3.3 *Hydrochloric acid*,  $d = 1.19$  (approximately 12 N).
- 3.4 *Citric acid*, 400 g per litre.  
Dissolve 40 g of citric acid crystals ( $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 \cdot \text{H}_2\text{O}$ ) in water and make up the volume to 100 ml.
- 3.5 *Ammonia solution*,  $d = 0.90$  (approximately 14.4 N).
- 3.6 "Suppressor"
- 3.6.1 *Solution of gum arabic*, 10 g per litre.  
To 1 g of gum arabic add, in small portions and while stirring, 100 ml of water. Prepare just before use.
- 3.6.2 *Solution of tylose*, 20 g per litre.  
To 2 g of tylose (methyl - ethyl cellulose; viscosity 100 centipoises) add, in small portions and while stirring, 100 ml of water. Do not heat.
- 3.6.3 *Solution of Triton X-100*, 1 g per litre.  
Dissolve 0.1 g of Triton X-100 ( $p\text{-C}_8\text{H}_{17}\text{C}_6\text{H}_4(\text{OCH}_2\text{CH}_2)_n\text{OH}$ ) in water and make up the volume to 100 ml.

3.7 *Sodium sulphite solution*, 200 g per litre.

Dissolve 20 g of anhydrous sodium sulphite ( $\text{Na}_2\text{SO}_3$ ) in water and make up the volume to 100 ml. Prepare just before use.

3.8 *Standard zinc solution*, 2 g per litre (1 ml contains 2 mg of zinc).

Dissolve 2 g of very pure zinc in 30 ml of hydrochloric acid (3.3) and add 30 ml of water. When the dissolution is complete transfer to a 1000 ml volumetric flask and make up to volume with water.

3.9 *Standard zinc solution*, 0.5 g per litre (1 ml contains 0.5 mg of zinc).

Transfer 50 ml of standard zinc solution (3.8) to a 200 ml volumetric flask and make up to volume with water.

#### 4. APPARATUS

4.1 *Ordinary laboratory apparatus*

All volumetric apparatus should comply with national standards.

4.2 *Polarograph*

4.3 *Thermostat*

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#### 5. SAMPLING

5.1 **Laboratory sample**

See the appropriate national standard on sampling.  
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5.2 **Test sample**

Chips not more than 1 mm thick should be obtained from the laboratory sample by milling or drilling.

#### 6. PROCEDURE

6.1 **Plotting of the calibration graph**

Introduce into a series of seven tall-form beakers of suitable capacity 1 g of pure magnesium (3.2). Add to each beaker 10 ml of water and, in small portions, 8 ml of hydrochloric acid (3.3). When the attack is completed, boil for a few moments and transfer the solutions to 100 ml volumetric flasks.

Then introduce respectively 0 (blank test), 5, 10, 15, and 20 ml of standard zinc solution (3.9) and, into the two remaining flasks, 7.5 and 10 ml of standard zinc solution (3.8), corresponding to 0, 2.5, 5, 7.5, 10, 15, and 20 mg of zinc.

Add, mixing thoroughly after each addition,

- 20 ml of citric acid solution (3.4)
- 5 g of ammonium chloride crystals (3.1),
- 13 ml of ammonia solution (3.5),
- 2 ml of suppressor solution (3.6.1 or 3.6.2 or 3.6.3),
- 4 ml of sodium sulphite solution (3.7).

Make up the volume to 100 ml with water.

Introduce an aliquot of these solutions into the polarograph cells, and place them in a thermostat at 25 °C. Carry out the polarographic measurement.

Record on a graph the readings corresponding to different concentrations of zinc, adjusted for any zinc content of the reagents (blank test).

## 6.2 Test portion

- (a) For a presumed zinc content less than, or equal to, 2 % weigh 1.0 g of the test sample (see clause 5.2) with an accuracy of  $\pm 0.001$  g.
- (b) For a presumed zinc content greater than 2 %, weigh 0.5 g of the test sample (see clause 5.2) with an accuracy of  $\pm 0.001$  g.

## 6.3 Determination

6.3.1 *Attack of the test portion.* Place the test portion in a tall-form beaker of suitable capacity (e.g. 250 ml). In the case of a test portion weighing 0.5 g, add 0.5 g of very pure magnesium (3.2).

Add 10 ml of water and, in small portions, 8 ml of hydrochloric acid (3.3). Once the reaction is complete, boil for a few minutes then transfer to a 100 ml volumetric flask. If a residue remains, filter through a medium texture filter, washing with water and collecting the filtrate and washings in a 100 ml volumetric flask.

Add, mixing thoroughly after each addition,

- 20 ml of citric acid solution (3.4),
- 5 g of ammonium chloride crystals (3.1),
- 13 ml of ammonia solution (3.5),
- 2 ml of suppressor solution (3.6.1 or 3.6.2 or 3.6.3),

– 4 ml of sodium sulphite solution (3.7).

Make up the volume to 100 ml with water.

Introduce an aliquot of the solution into a polarographic cell, place it in a thermostat at 25 °C and carry out the polarographic measurement.

6.3.2 *Blank test.* Attack in the same manner 1 g of pure magnesium (3.2) and follow the procedure described in clause 6.3.1.

## 7. EXPRESSION OF RESULTS

By means of the calibration graph, determine the quantity of zinc, expressed in milligrammes, contained in 100 ml of solution.

The percentage, by mass, of zinc is calculated from the following formula :

$$\text{Zn } \% \text{ (m/m)} = \frac{A - B}{10 E}$$

where

- A* is the mass, in milligrammes, of zinc found in the solution of the test portion,
- B* is the mass, in milligrammes, of zinc found in the solution used in the blank test,
- E* is the mass, in grammes, of the test portion.

## 8. TEST REPORT

Report the following information :

- (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 laid down in this ISO Recommendation or regarded as optional.

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