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**INTERNATIONAL STANDARD**



**1169**

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## **Zinc alloys — Determination of aluminium content — Volumetric method**

*Alliages de zinc — Dosage de l'aluminium — Méthode titrimétrique*

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**Descriptors** : zinc alloys, chemical analysis, determination of content, aluminium, volumetric analysis.

# Zinc alloys – Determination of aluminium content – Volumetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the aluminium content of zinc alloys.

The method is applicable to zinc alloys defined in ISO/R 301, and to die castings made from these alloys.

## 2 REFERENCES

ISO/R 301, *Zinc alloy ingots*.

ISO 3752, *Zinc alloy ingots – Selection and preparation of samples for chemical analysis*.<sup>1)</sup>

## 3 PRINCIPLE

Addition of an excess of EDTA to the hydrochloric solution of a test portion. Quantitative complexing of the excess with a standard zinc solution. Decomposition of the aluminium-EDTA complex with sodium fluoride and titration of the liberated EDTA with a standard zinc solution.

## 4 REAGENTS

During the analysis, use only reagents of analytical reagent grade and distilled or demineralized water.

**4.1 Hydrochloric acid**, approximately 6 N.

**4.2 Hydrogen peroxide**, 30 % (m/m) H<sub>2</sub>O<sub>2</sub>.

**4.3 Hydroxylammonium chloride**, 200 g/l solution.

**4.4 Ethylenediamine tetra-acetic acid disodium salt (EDTA) solution**.

Dissolve 65 g of EDTA in approximately 750 ml of warm water. Cool. Make up the volume to 1 l.

**4.5 Ammonia solution**,  $\rho$  0,91 g/ml.

**4.6 Sodium fluoride**, saturated solution

Dissolve 60 g of sodium fluoride in 1 l of boiling water. Cool. Filter.

**4.7 Buffer solution** at pH 5 to 5,5.

Dissolve 135 g of sodium acetate (CH<sub>3</sub>COONa.3H<sub>2</sub>O) in about 300 ml of water. Introduce 13 ml of glacial acetic acid (17 N). Check that the pH is within the range 5 to 5,5. Make up the volume to 500 ml with water.

**4.8 Zinc standard solution**, 0,05 M.

Dissolve 3,269 g of high purity zinc in 20 ml of hydrochloric acid (4.1) in a 250 ml beaker covered with a watch-glass. Dilute with 100 ml of water. Add 2 drops of methyl red solution (4.9). Neutralize with ammonia solution (4.5). Add hydrochloric acid (4.1) drop by drop until the colour changes to red. Transfer quantitatively to a 1 l volumetric flask. Dilute to the mark and mix.

1 ml of this solution corresponds to 1,349 mg of aluminium.

**4.9 Methyl red ethanolic solution**

Dissolve 0,02 g of methyl red in 100 ml of ethanol.

**4.10 Xylenol orange solution**

Dissolve 1 g of xylenol orange sodium salt in 100 ml of water.

## 5 APPARATUS

Ordinary laboratory apparatus.

## 6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3752.

## 7 PROCEDURE

### 7.1 Test portion

Weigh, to the nearest 0,001 g, 5 g of the test sample.

### 7.2 Determination

**7.2.1** Place the test portion in a 500 ml beaker and attack carefully with 50 ml of hydrochloric acid (4.1).

1) At present at the stage of draft.

**7.2.2** Oxidize and complete the dissolution by adding a few drops of hydrogen peroxide (4.2). Add 5 ml of hydroxylammonium chloride solution (4.3) to decompose the excess of hydrogen peroxide.

Dilute. Cool. Transfer quantitatively to a 250 ml volumetric flask. Dilute to the mark and mix.

**7.2.3** Transfer a 25 ml aliquot to a 500 ml conical flask.

**7.2.4** Add :

- approximately 100 ml of water;
- 50 ml of EDTA solution (4.4);
- 5 drops of methyl red solution (4.9).

**7.2.5** Neutralize exactly with ammonia solution (4.5) until the colour changes to yellow. Add 25 ml of buffer solution (4.7).

**7.2.6** Boil for 2 to 3 min. Cool.

**7.2.7** Add 2 to 3 drops of xylenol orange solution (4.10).<sup>1)</sup>

**7.2.8** Titrate the excess of EDTA with standard zinc solution (4.8) until the colour changes to purple.

**7.2.9** Add 25 ml of sodium fluoride solution (4.6). Boil for 2 to 3 min. Cool.

### 7.3 Volumetric measurement

Titrate the liberated EDTA with the standard zinc solution (4.8) until the colour changes to purple.

## 8 EXPRESSION OF RESULTS

The aluminium content is given, as a percentage by mass, by the formula

$$V \times 0,269 8$$

where  $V$  is the volume, in millilitres, of standard zinc solution used to titrate the liberated EDTA (see 7.3).

## 9 TEST REPORT

The test report shall mention the method used and the results obtained. It shall also mention all operational details not provided for in this International Standard, or any optional details, as well as any circumstances which could have influenced the results.

The test report shall include all details required for complete identification of the sample.

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1) For some operators the end point can be made even more readily detectable by the addition of not more than 1 ml of a 1 g/l aqueous solution of xylene cyanol.

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