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CHEMICAL ANALYSIS OF ZINC AND ZINC ALLOYS

SPECTROPHOTOMETRIC DETERMINATION OF TIN

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BRIEF HISTORY

The ISO Recommendation R 1570, *Chemical analysis of zinc and zinc alloys – Spectrophotometric determination of tin,* was drawn up by Technical Committee ISO/TC 18, *Zinc and zinc alloys,* the Secretariat of which is held by the Institut Belge de Normalisation (IBN).

Work on this question led to the adoption of Draft ISO Recommendation No. 1570, which was circulated to all the ISO Member Bodies for enquiry in April 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Spain
Belgium	Iran	Sweden
Brazil	Ireland	Thailand
Canada	Israel	Turkey
Czechoslovakia	Italy	U.A.R.
France	Norway	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	South Africa, Rep. of	Yugoslavia

No Member Body opposed the approval of the Draft.

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

ISO Recommendation | R 1570 | June 1970

CHEMICAL ANALYSIS OF ZINC AND ZINC ALLOYS

SPECTROPHOTOMETRIC DETERMINATION OF TIN

1. SCOPE

This ISO Recommendation describes a spectrophotometric method for the determination of tin in zinc and zinc alloys.

The method applies to the types of zinc defined in ISO Recommendation R 752, *Zinc ingots*, to the zinc alloys defined in ISO Recommendation R 301, *Zinc alloy ingots*, and to die castings made from these alloys.

It is suitable for the determination of tin contents between 0.0005 and 0.005 %.*

2. PRINCIPLE OF THE METHOD

Spectrophotometric determination of the tin-quercetin complex after extraction by methyl isobutyl ketone.

3. REAGENTS

All the reagents should be of the analytical reagent grade.

Tin-free distilled or demineralized water should be used for preparing solutions and during the actual determinations.

3.1 *Hydrochloric acid*, ρ 1.19 (g/ml).

3.2 *Hydrogen peroxide*, 30 % H_2O_2 (m/m), free of tin-containing stabilizer.

3.3 *Thiourea solution.*

Dissolve 12.5 g of thiourea in 100 ml of warm water (about 60 °C). Dilute to about 200 ml. Cool. Make up the volume to 250 ml.

3.4 *Ascorbic acid solution.*

Dissolve 2 g of ascorbic acid in 100 ml of water. Use a freshly prepared solution.

3.5 *Acidified quercetin solution.***

Dissolve 500 mg of quercetin in 300 ml of ethanol (heat gently to dissolve). Cool. Add exactly 25 ml of hydrochloric acid (3.1). Make up the volume to 1 litre with ethanol. Mix. Filter off any residue.

3.6 *Methyl isobutyl ketone.*

3.7 *Sulphuric acid solution*, 5 % (V/V).

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* The method may be extended to higher contents, using smaller samples and observing the condition that the calibration curve should be prepared in the presence of corresponding zinc contents.

** The quality of the quercetin is satisfactory so long as the blank test gives an extinction less than 0.1.

3.8 *Standard tin solution.*

Dissolve 0.500 g of pure tin in 100 ml of hydrochloric acid (3.1) in a 250 ml beaker covered with a watch-glass, heating gently. When dissolution is complete, transfer quantitatively to a 1 litre volumetric flask. Cool. Make up to volume with water. Mix. Transfer 10 ml of this solution to a 1 litre volumetric flask. Add 100 ml of hydrochloric acid (3.1). Make up to volume with water. Mix.

1 ml of this solution contains 0.005 mg of tin.

3.9 *Zinc* at least 99.99 % pure.

3.10 *Hydrochloric acid,ρ* 1.19 (g/ml) diluted one part of acid to nine parts of water.

3.11 *Nickel chloride solution.*

Dissolve 0.5 g of pure nickel in the minimum amount of hydrochloric acid (3.1). Dilute to 1 litre.

4. APPARATUS

4.1 *Ordinary laboratory equipment.*

4.2 *Spectrophotometer,* wavelength 440 nm, and 1 cm cells.*

5. SAMPLING

The requirements of ISO Recommendation R . . .,** *Selection and preparation of samples for analysis,* should apply.

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, 2 g of the test sample.

6.2 Blank test

Simultaneously with the actual determination, carry out a blank test, operating as follows :

6.2.1 Introduce 2 g of pure zinc (3.9) into a 100 ml beaker and attack with 15 ml of hydrochloric acid (3.1).

6.2.2 Add a few drops of hydrogen peroxide (3.2). Evaporate to a syrupy consistency to eliminate possible traces of tin.

6.2.3 Take up with 15 ml of hydrochloric acid (3.1). Cool.

6.2.4 Transfer quantitatively to a 50 ml volumetric flask. Make up to volume with water. Mix.

6.2.5 Continue from clause 6.4.1.3 of the "Determination".

6.3 Plotting of the calibration curve

6.3.1 In five 100 ml beakers, place 2 g of pure zinc (3.9) and dissolve, without heating, with 18 ml of hydrochloric acid (3.1).

6.3.2 Assuming that a calibration curve is to be made defined by five terms, corresponding to the tin contents of 0 – 0.001 – 0.003 – 0.004 and 0.005 %, add respectively 0 – 4 – 12 – 16 and 20 ml of the standard tin solution (3.8) and 20 – 16 – 8 – 4 and 0 ml of the diluted hydrochloric acid (3.10).***

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* The amount of methyl isobutyl ketone indicated in clause 6.4.1.5 is only valid when operating with 1 cm cells. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

** To be prepared later.

*** In this way, the quantity of concentrated hydrochloric acid is 20 ml, the same for all the terms.

6.3.3 Continue as described in clauses 6.4.1.2 to 6.4.2 inclusive.

6.3.4 Measure the optical density of these solutions against the solution to which no tin has been added, at a wavelength of 440 nm (4.2).

6.4 Determination*

6.4.1 *Attack*

6.4.1.1 Place the test portion in a 100 ml beaker and attack without heating with 20 ml of hydrochloric acid (3.1).**

6.4.1.2 Oxidize and complete the solution by adding a few drops of hydrogen peroxide (3.2).

Dilute. Cool. Transfer quantitatively to a 50 ml volumetric flask. Make up to volume with water. Mix.

6.4.1.3 Introduce successively into a 125 ml separating funnel :

- 20 ml of thiourea solution (3.3);
- 5 ml of ascorbic acid solution (3.4);
- 20 ml of quercetin solution (3.5).

Mix.

Add 25 ml of sample solution (6.4.1.2).

Mix.

6.4.1.4 Allow to stand for 10 to 15 minutes.

6.4.1.5 Introduce exactly 15 ml of methyl isobutyl ketone. Shake for 1 minute.

6.4.1.6 Allow to settle. After clear separation of the two layers, discard the aqueous phase.

6.4.1.7 Introduce 25 ml of sulphuric acid solution (3.7) and shake for 30 seconds.

6.4.1.8 Allow to settle for 5 minutes. After clear separation of the two layers, discard the aqueous phase.

6.4.2 Transfer a suitable portion of the organic layer into an absorption cell filtering through a small dry rapid filter paper (about 70 mm diameter) to remove droplets from the aqueous phase, discarding the first portions of the filtrate.***

6.5 Spectrophotometric measurement

Measure the optical density of the solution against the blank solution at a wavelength of 440 nm (4.2).

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* If heterogeneity of the test sample necessitates a larger test portion, weigh 10 ± 0.01 g.

Clause 6.4.1 then begins as follows :

6.4.1.1 Place the test portion in a 250 ml beaker and attack with 100 ml of hydrochloric acid (3.1). Oxidize and complete the solution by adding 1 ml of hydrogen peroxide (3.2). Cool.

6.4.1.2 Transfer quantitatively to a 250 ml volumetric flask. Make up to volume with water. Mix.

Continue from clause 6.4.1.3 of the "Determination".

** In the case of a difficult dissolution, 2 ml of nickel chloride solution (3.11) can be added in order to activate the attack.

*** Filtration may be replaced by centrifuging.

It is recommended to avoid exposure of the organic layer to direct sunlight.