INTERNATIONAL STANDARD 1570

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

Zinc and zinc alloys — Determination of tin content — Spectrophotometric method

Zinc et alliages de zinc — Dosage de l'étain — Méthode spectrophotométrique

First edition — 1975-06-15

UDC 669.5 : 543.42 : 546.811

Ref. No. ISO 1570-1975 (E)

Descriptors : zinc, zinc alloys, chemical analysis, determination of content, tin, spectrophotometric analysis.

Price based on 3 pages

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, Technical Committee ISO/TC 18 has reviewed ISO Recommendation R 1570 and found it technically suitable for transformation. International Standard ISO 1570 therefore replaces ISO Recommendation R 1570-1970 to which it is technically identical.

ISO Recommendation R 1570 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1570 into an International Standard.

Printed in Switzerland

Zinc and zinc alloys — Determination of tin content — Spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination of the tin content of zinc and zinc alloys.

The method is applicable to the types of zinc defined in ISO/R 752, to the zinc alloys defined in ISO/R 301, and to die castings made from these alloys.

It is suitable for the determination of tin contents between 0,000 5 and 0,005 %.[1)]

2 REFERENCES

ISO/R 301, *Zinc alloy ingots.*

ISO/R 752, *Zinc ingots.*

ISO 3751, *Zinc ingots — Selection and preparation of samples for chemical analysis.*[2)]

ISO 3752, *Zinc alloy ingots — Selection and preparation of samples for chemical analysis.*[2)]

3 PRINCIPLE

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

4 REAGENTS

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

4.1 Zinc, at least 99,99 % pure.

4.2 Hydrochloric acid, ρ 1,19 g/ml.

4.3 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 part of acid to 9 parts of water.

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

4.5 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.

4.6 Ascorbic acid solution.

Dissolve 2 g of ascorbic acid in 100 ml of water.

This solution must be freshly prepared before use.

4.7 Quercetin acidified ethanolic solution.[3)]

Dissolve 500 mg of quercetin in 300 ml of ethanol (heat gently to dissolve). Cool. Add exactly 25 ml of hydrochloric acid (4.2). Make up the volume to 1 l with ethanol. Mix. Filter off any residue.

4.8 Methyl isobutyl ketone.

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

4.10 Nickel chloride solution.

Dissolve 0,5 g of pure nickel in the minimum amount of hydrochloric acid (4.2). Dilute to 1 l.

4.11 Tin standard solution.

Dissolve 0,500 g of pure tin in 100 ml of hydrochloric acid (4.2) in a 250 ml beaker covered with a watch-glass, heating gently. When dissolution is complete, transfer quantitatively to a 1 l volumetric flask. Cool. Dilute to the mark and mix. Transfer 10 ml of this solution to a 1 l volumetric flask. Add 100 ml of hydrochloric acid (4.2). Dilute to the mark and mix.

1 ml of this solution contains 0,005 mg of tin.

1) 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.

2) At present at the stage of draft.

3) The quality of the quercetin is satisfactory so long as the blank test gives an extinction less than 0,1.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, wavelength 440 nm, and **1 cm cells.**[1)]

6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3751 or ISO 3752, as appropriate.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 2 g of the test sample.

7.2 Blank test

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

7.2.1 Introduce 2 g of pure zinc (4.1) into a 100 ml beaker and attack with 15 ml of hydrochloric acid (4.2).

7.2.2 Add a few drops of hydrogen peroxide (4.4). Evaporate to a syrupy consistency to eliminate possible traces of tin.

7.2.3 Take up with 15 ml of hydrochloric acid (4.2). Cool.

7.2.4 Transfer quantitatively to a 50 ml volumetric flask. Dilute to the mark and mix.

7.2.5 Continue from 7.4.1.3.

7.3 Plotting of the calibration curve

7.3.1 Into each of five 100 ml beakers, place 2 g of pure zinc (4.1) and dissolve, without heating, with 18 ml of hydrochloric acid (4.2).

7.3.2 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 tin standard solution (4.11) and 20 — 16 — 8 — 4 and 0 ml of the diluted hydrochloric acid (4.3).[2)]

7.3.3 Continue as specified in 7.4.1.2 to 7.4.2 inclusive.

7.3.4 Measure the absorbance of these solutions against the solution to which no tin has been added, in the spectrophotometer at a wavelength of 440 nm.

7.4 Determination[3)]

7.4.1 *Attack*

7.4.1.1 Place the test portion (7.1) in a 100 ml beaker and attack without heating with 20 ml of hydrochloric acid (4.2).[4)]

7.4.1.2 Oxidize and complete the solution by adding a few drops of hydrogen peroxide (4.4).

Dilute. Cool. Transfer quantitatively to a 50 ml volumetric flask. Dilute to the mark and mix.

7.4.1.3 Introduce successively into a 125 ml separating funnel :

— 20 ml of thiourea solution (4.5);

— 5 ml of ascorbic acid solution (4.6);

— 20 ml of quercetin solution (4.7).

Mix.

Add 25 ml of the test solution (7.4.1.2).

Mix.

7.4.1.4 Allow to stand for 10 to 15 min.

7.4.1.5 Introduce exactly 15 ml of methyl isobutyl ketone (4.8). Shake for 1 min.

---

1) The amount of methyl isobutyl ketone indicated in 7.4.1.5 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.

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

3) If heterogeneity of the test sample necessitates a larger test portion, weigh 10 ± 0,01 g.

Sub-clause 7.4.1 then begins as follows :

7.4.1.1 Place the test portion in a 250 ml beaker and attack with 100 ml of hydrochloric acid (4.2). Oxidize and complete the solution by adding 1 ml of hydrogen peroxide (4.4). Cool.

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

Continue from 7.4.1.3.

4) In the case of a difficult dissolution, 2 ml of nickel chloride solution (4.10) can be added in order to activate the attack.

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

7.4.1.7 Introduce 25 ml of sulphuric acid solution (4.9) and shake for 30 s.

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

7.4.2 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.[1)]

7.5 Spectrophotometric measurement

Measure the absorbance of the solution against the blank solution in the spectrophotometer (5.1) at a wavelength of 440 nm.

8 EXPRESSION OF RESULTS

Determine the tin content by means of the calibration curve (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.

1) Filtration may be replaced by centrifuging.

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

This page intentionally left blank

This page intentionally left blank

This page intentionally left blank