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Chemical analysis of zinc alloys — Polarographic determination of lead and cadmium in zinc alloys containing copper

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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.

International Standard ISO 2576 was drawn up by Technical Committee ISO/TC 18, *Zinc and zinc alloys*.

It was approved in February 1972 by the Member Bodies of the following countries :

Austria	Ireland	Spain
Belgium	Italy	Sweden
Czechoslovakia	New Zealand	Thailand
Egypt, Arab Rep. of	Norway	United Kingdom
France	Poland	U.S.A.
Germany	Romania	U.S.S.R.
India	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Chemical analysis of zinc alloys — Polarographic determination of lead and cadmium in zinc alloys containing copper

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarographic method for the simultaneous determination of lead and cadmium in zinc alloys containing copper.

The method applies to the zinc alloys defined in ISO/R 301¹⁾.

It is suitable for the determination of lead and cadmium contents exceeding 0,000 5 %,

2 REFERENCES

ISO/R 301, *Zinc alloy ingots.*

ISO/R 713, *Chemical analysis of zinc. Polarographic determination of lead and cadmium.*

ISO ..., *Sampling and preparation of samples for analysis.* (In preparation.)

3 PRINCIPLE

Simultaneous polarographic determination of lead and cadmium in a very slightly acid medium, after elimination of copper by precipitation with salicylaldoxime, followed by the removal of the excess of reagent by washing with chloroform. The last traces of organic matter are destroyed by chlorate.

4 REAGENTS

All the reagents shall be of the analytical reagent grade.

Double-distilled or demineralized water shall be used for preparing the solutions and during the actual determination.

4.1 Hydrochloric acid, $d = 1,19$, free from lead and cadmium.

Check this purity by the following test :

4.1.1 Take three 5 g test portions of zinc Zn 99,995 or zinc Zn 99,99 for die castings.

4.1.2 Attack the first and second test portions with 35 to 40 ml of hydrochloric acid (4.1), as indicated under 7.3 starting at 7.3.2.

4.1.3 To the second test portion, add 1 ml of the standard solution No. 2 (4.9).

4.1.4 Attack the third test portion with 100 ml of hydrochloric acid (4.1).

4.1.5 The acid is suitable for use if the differences in step heights between the first and third test portions are less than those between the first and second test portions. If these conditions are satisfied the volume of acid used for the dissolution contains less than 0,005 mg of lead and less than 0,005 mg of cadmium.

4.2 Hydrogen peroxide, H₂O₂ 30 % (m/m).

4.3 Chloroform, pure.

4.4 Salicylaldoxime solution.

Dissolve 4 g of pure salicylaldoxime in 40 ml of 94 % (V/V) ethanol. Add, while stirring, 50 ml of water at 80 °C.

4.5 Wash solution.

Dilute a portion of the solution (4.4) with water about 200 times.

4.6 Potassium chlorate solution, saturated at ambient temperature.

4.7 Nitric acid, $d = 1,4$.

4.8 Lead and cadmium standard solution No. 1.

In a 100 ml beaker, place 0,5 g of lead and 0,5 g of cadmium, weighed to the nearest 0,001 g. Cover with approximately 10 ml of water and dissolve with about 5 ml of nitric acid (4.7). After dissolution, boil to drive off the nitrous fumes. Cool. Transfer quantitatively to a 500 ml volumetric flask. Make up to volume with water. Mix. Take exactly 25 ml of this solution. Transfer to a 1 l volumetric flask. Make up to volume with water. Mix.

1) Although copper-free zinc alloys can be analysed by this method, Zn Al 4 samples containing less than 0,002 % of copper may be analysed by the quicker method for the determination of lead and cadmium in zinc, described in ISO/R 713.

1 ml of this solution contains 0,025 mg of lead and 0,025 mg of cadmium.

4.9 Lead and cadmium standard solution No. 2.

Transfer 10 ml of the standard solution No. 1 (4.8) to a 25 ml volumetric flask. Make up to volume with water. Mix.

1 ml of this solution contains 0,01 mg of lead and 0,01 mg of cadmium.

4.10 Pure cylinder nitrogen or pure carbonic acid gas.

4.11 Zinc chloride solution free from lead and cadmium.

In a 1 l tall-form beaker dissolve 125 g of zinc Zn 99,99 or Zn 99,995 in about 500 ml of hydrochloric acid (4.1). If necessary, complete the dissolution by the addition of an additional quantity of hydrochloric acid (4.1). Evaporate to a syrupy consistency. Take up with 200 ml of water and transfer quantitatively to a 500 ml volumetric flask. Add 20 g of zinc dust and allow to react for 1 to 2 h. Make up to volume with water. Mix. Allow to settle. Filter the solution without washing. Check the absence of lead and cadmium in this solution with the polarograph.

4.12 Aluminium solution.

Dissolve 2,5 g of aluminium of purity at least 99,9 % with 50 ml of hydrochloric acid (4.1) in a 250 ml beaker covered with a watchglass. Remove and rinse the watchglass and transfer the solution to a 250 ml volumetric flask. Make up to volume. Mix. Check the absence of lead and cadmium in this solution with the polarograph, proceeding as follows :

Transfer 20 ml of the zinc chloride solution (4.11) to a 100 ml beaker and continue the operations as outlined in 7.2.3 to 7.2.6.

No wave due to the presence of lead or cadmium shall be obtained.

5 APPARATUS

Ordinary laboratory apparatus, and

5.1 Polarograph.

5.2 Water bath, thermostatically controlled.

6 SAMPLING

The samples shall be taken according to the requirements of ISO

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, a 5 g test portion.

7.2 Plotting of the calibration curve

7.2.1 Into each of a series of six 100 ml beakers transfer 20 ml of the zinc chloride solution (4.11).

7.2.2 Add respectively 1 – 2 – 5 – 10 – 15 – 20 ml of the lead and cadmium standard solution No. 1 (4.8), corresponding respectively to concentrations of 0,000 5 – 0,001 – 0,002 5 – 0,005 – 0,007 5 – 0,01 % of lead and cadmium.

7.2.3 Introduce 20 ml of the aluminium solution (4.12) (i.e. 5 ml for each 1 % of aluminium contained in the test portion).

7.2.4 Evaporate to a syrupy consistency. Add 10 ml of hydrochloric acid (4.1). Evaporate to a syrupy consistency. Add again 10 ml of hydrochloric acid (4.1). Finally evaporate to a syrupy consistency, without going to complete dryness.

7.2.5 Take up with about 5 ml of water. Transfer quantitatively to 25 ml volumetric flasks. Make up to volume.

The pH should be between 2 and 3.

7.2.6 Carry out the measurements with the polarograph under the same conditions as for the actual determinations, selecting the appropriate instrument sensitivity.

7.3 Determination

7.3.1 Place the test portion in a 250 ml beaker and attack with 40 ml of hydrochloric acid (4.1). Add a few drops of hydrogen peroxide (4.2) to complete dissolution.

7.3.2 Evaporate to a syrupy consistency.

7.3.3 Take up with 125 ml of water and warm gently to ensure that all the salts are dissolved.

7.3.4 Add 10 ml of the salicylaldoxime solution (4.4) (i.e. sufficient for alloys containing about 1 % of copper). Allow to stand for 30 min and stir from time to time. Filter. Wash with water containing salicylaldoxime (4.5) and evaporate the filtrate to a volume of about 30 ml.

7.3.5 Transfer quantitatively to a 125 ml separating funnel and rinse the beaker with approximately 20 ml of water. Wash the aqueous phase twice with 25 ml of pure chloroform (4.3).

7.3.6 Collect the aqueous phase quantitatively in a 100 ml beaker and rinse the separating funnel with a few millilitres of water. Evaporate to a syrupy consistency.

7.3.7 Add approximately 2,5 ml of the saturated potassium chlorate solution (4.6). Cover the beaker with a watchglass. Evaporate to a syrupy consistency by heating strongly without going to complete dryness.

7.3.8 Again add 2,5 ml of the potassium chlorate solution (4.6). Evaporate again to a syrupy consistency under the same conditions as above. Remove and rinse the watchglass.

7.3.9 Add 5 ml of hydrochloric acid (4.1). Evaporate to a syrupy consistency.

7.3.10 Again add 5 ml of hydrochloric acid (4.1). Evaporate to a syrupy consistency, without going to complete dryness.

7.3.11 Take up with 5 ml of water, shaking until dissolution is complete.

7.3.12 Transfer quantitatively to a 25 ml volumetric flask. Cool, make up to volume and mix.

7.3.13 Transfer the appropriate quantity of this solution to the polarograph cell. De-aerate by passing an inert gas (4.10) through the solution for at least 10 min.

7.4 Polarographic measurement

Introduce only at this moment the mercury serving as the anode. Carry out the measurements with the polarograph between 0 and -1 V relative to the mercury electrode, choosing the appropriate sensitivity. The half-wave potential of lead is approximately $-0,4$ V, and of cadmium is $-0,6$ V, relative to a mercury electrode.

8 TEST REPORT

The test report shall include the following particulars :

- a) the method used and the results obtained;
- b) all operational details not provided for in this International Standard or any optional details;
- c) any circumstances which could have influenced the results;
- d) all details required for complete identification of the sample.

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