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# INTERNATIONAL STANDARD 713

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# Zinc — Determination of lead and cadmium contents — Polarographic method

*Zinc — Dosage du plomb et du cadmium — Méthode polarographique*

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**Descriptors :** zinc, chemical analysis, determination of content, lead, cadmium, polarographic analysis.



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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.

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 713 and found it technically suitable for transformation. International Standard ISO 713 therefore replaces ISO Recommendation R 713-1968 to which it is technically identical.

ISO Recommendation R 713 was approved by the Member Bodies of the following countries :

| | | |
|---|---|---|
| Belgium | India | Spain |
| Brazil | Ireland | Switzerland |
| Canada | Italy | Thailand |
| Chile | Japan | Turkey |
| Czechoslovakia | Korea, Rep. of | United Kingdom |
| Egypt, Arab Rep. of | Korea, D.P. Rep. of | U.S.A. |
| France | New Zealand | U.S.S.R. |
| Germany | Norway | Yugoslavia |
| Hungary | South Africa, Rep. of | |

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Australia

The Member Body of the following country disapproved the transformation of ISO/R 713 into an International Standard :

Spain



Printed in Switzerland

# Zinc — Determination of lead and cadmium contents — Polarographic method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarographic method for the simultaneous determination of the lead and cadmium contents of zinc.

The method is applicable to the following types of zinc : Zn 99,995, Zn 99,99 and Zn 99,95,[1] defined in ISO/R 752.

It is suitable for the determination of lead and cadmium contents between 0,001 and 0,05 %.

## 2 REFERENCES

ISO/R 752, *Zinc ingots.*

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

## 3 PRINCIPLE

Simultaneous polarographic determination of the lead and cadmium contents in a very slightly acid chloride medium.

## 4 REAGENTS

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

**4.1 Inert gas,** oxygen free.

**4.2 Hydrochloric acid,** $\rho$ 1,19 g/ml, free from lead and cadmium.

Check the purity by the following test :

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

**4.2.2** Attack the first and second test portions with 35 to 40 ml of hydrochloric acid (4.2), as indicated under 7.3, starting at 7.3.2.

**4.2.3** To the second test portion, add 1 ml of the standard solution (4.9).

**4.2.4** Attack the third test portion with 100 ml of hydrochloric acid (4.2).

**4.2.5** 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.

**4.3 Hydrogen peroxide,** 30 % (*m/m*) $H_2O_2$.

**4.4 Nitric acid,** $\rho$ 1,3 to 1,4 g/ml.

**4.5 Hydroxylammonium chloride.**

**4.6 Nickel chloride** solution containing 2 g of $NiCl_2 . 6H_2O$ per litre.

**4.7 Lead and cadmium,** standard solution No. 1.

In a 100 ml beaker, place 0,5 g of pure lead and 0,5 g of pure cadmium, weighed to the nearest 0,001 g. Cover with approximately 10 ml of water and dissolve with about 5 ml of nitric acid (4.4). Drive off the nitrous fumes. Cool. Transfer quantitatively into a 500 ml volumetric flask. Dilute to the mark and mix.

1 ml of this solution contains 1 mg of lead and 1 mg of cadmium.

**4.8 Lead and cadmium,** standard solution No. 2.

Dilute the standard lead and cadmium solution No. 1 (4.7) in the proportion of 1 part to 9 parts of water.

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

1) Although these types of zinc do not normally contain any thallium or indium, these two elements, if present, can interfere in this determination.

2) At present at the stage of draft.

**4.9 Lead and cadmium,** standard solution No. 3.

Dilute the standard lead and cadmium solution No. 2 (4.8) in the proportion of 1 part to 9 parts of water.

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

**4.10 Zinc chloride,** solution free from lead and cadmium.

Dissolve 100 g of Zn 99,995 zinc in 400 ml of hydrochloric acid (4.2). Evaporate to a syrupy consistency. Take up with 400 ml of water. Transfer to a 500 ml volumetric flask. Add 20 g of zinc dust. Agitate from time to time for at least 30 min. Dilute to the mark and mix. Allow to settle. Filter the solution without washing.

1 ml of this solution contains approximately 200 mg of zinc.

Verify that the step heights are less than those produced by adding 0,5 ml of the standard lead and cadmium solution No. 3 (4.9) to 25 ml of the solution of zinc chloride.

This test determines whether 25 ml of the solution of zinc chloride contains less than 0,005 mg of lead and less than 0,005 mg of cadmium.

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Polarograph.**

**5.2 Thermostatically controlled water bath.**

## 6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3751.

## 7 PROCEDURE

### 7.1 Test portion

Weigh 5 g of the test sample to the nearest 0,01 g.

### 7.2 Plotting of the calibration curve

Prepare a calibration curve such that it will include the expected content.

As an example, assuming that a calibration curve is to be established by 3 steps corresponding to lead and cadmium contents of 0,001 – 0,002 – 0,005 %, then

**7.2.1** Into a series of 100 ml beakers, transfer 25 ml of the solution of zinc chloride (4.10) corresponding to 5 g of metallic zinc. Add 5 ml, 10 ml and 25 ml respectively of the standard lead and cadmium solution No. 3 (4.9).

**7.2.2** Evaporate to a syrupy consistency and proceed as outlined in 7.3.3 to 7.3.5, then polarograph as outlined in 7.4.

**7.2.3** Construct a calibration curve from the step heights obtained.

### 7.3 Determination

**7.3.1** Transfer the test portion to a 100 ml beaker and attack with 35 to 40 ml of hydrochloric acid (4.2). Add 2 or 3 drops of hydrogen peroxide (4.3) to dissolve completely.

NOTE – If dissolution is very difficult, add 2 ml of nickel chloride solution (4.6) to expedite the attack.

**7.3.2** Evaporate to a syrupy consistency until the first appearance of a white solid film or a white foamy mass.

**7.3.3** Take up with water and if necessary add 1 or 2 drops of hydrochloric acid (4.2), taking care to obtain complete solution. If the presumed iron content is greater than 0,01 %, add one or two crystals of hydroxylammonium chloride (4.5) (approximately 5 mg); then warm gently to eliminate completely the possible influence of iron. Allow to cool.

**7.3.4** Transfer to a 25 ml volumetric flask and dilute to the mark.

**7.3.5** Transfer the appropriate quantity of this solution to the polarograph cell and place this in a thermostatically controlled water bath. De-aerate by passing inert gas (4.1) through the solution for at least 10 min.

### 7.4 Polarographic measurement

Polarograph between 0 and – 1 V.

The step for lead occurs at about – 0,4 V and the step for cadmium at about – 0,6 V relative to a mercury electrode or, respectively, at – 0,65 V and – 0,85 V relative to a saturated calomel electrode.

## 8 EXPRESSION OF RESULTS

Read from the calibration curve the values corresponding to the step heights obtained.

## 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