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CHEMICAL ANALYSIS OF ZINC

POLAROGRAPHIC DETERMINATION OF LEAD AND CADMIUM

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BRIEF HISTORY

The ISO Recommendation R 713, *Chemical analysis of zinc – Polarographic determination of lead and cadmium,* 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 by the Technical Committee began in 1963 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In September 1966, this Draft ISO Recommendation (No. 992) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

Australia

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in May 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 713 May 1968

CHEMICAL ANALYSIS OF ZINC

POLAROGRAPHIC DETERMINATION OF LEAD AND CADMIUM

1. SCOPE

This ISO Recommendation describes a polarographic method for the simultaneous determination of lead and cadmium in zinc.

The method applies to the following types of zinc : Zn 99.995, Zn 99.99 and Zn 99.95,* defined in ISO Recommendation R 752, *Zinc ingots.*

It is suitable for the determination of lead and cadmium contents between 0.001 and 0.05 %.

2. PRINCIPLE OF THE METHOD

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

3. REAGENTS

All reagents should be of analytical reagent grade.

Distilled or demineralized water should be used for preparing solutions and during the actual determination.

3.1 *Inert gas*, oxygen-free.

3.2 *Hydrochloric acid* ($d = 1.19$), free from lead and cadmium.

Check the purity by the following test :

3.2.1 Take three 5 g test portions of Zn 99.995 zinc or Zn 99.99 zinc for die castings.

3.2.2 Attack the first and second test portion with 35 to 40 ml of hydrochloric acid (3.2), as indicated under clause 6.3 starting at clause 6.3.2.

3.2.3 To the second test portion, add 1 ml of the standard solution (3.9).

3.2.4 Attack the third test portion with 100 ml of hydrochloric acid (3.2).

3.2.5 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.

3.3 *Hydrogen peroxide*, 30 % H_2O_2 (m/m)

3.4 *Nitric acid* ($d = 1.3$ to 1.4).

3.5 *Hydroxylammonium chloride*

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

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

3.7 *Standard solution of lead and cadmium No. 1*

In a 100 ml beaker, place 0.5 g of pure lead and 0.5 g of pure cadmium, weighed with an accuracy of ± 0.001 g. Cover with approximately 10 ml of water and dissolve with about 5 ml of nitric acid (3.4). Drive off the nitrous fumes. Cool. Transfer quantitatively into a 500 ml volumetric flask. Make up the volume to 500 ml with water. Mix. 1 ml of the solution contains 1 mg of lead and 1 mg of cadmium.

3.8 *Standard solution of lead and cadmium No. 2*

Dilute the standard solution of lead and cadmium No. 1 (3.7) in the proportion of one part to nine parts of water. 1 ml of the solution contains 0.1 mg of lead and 0.1 mg of cadmium.

3.9 *Standard solution of lead and cadmium No. 3*

Dilute the standard solution of lead and cadmium No. 2 (3.8) in the proportion of one part to nine parts of water. 1 ml contains 0.01 mg of lead and 0.01 mg of cadmium.

3.10 *Solution of zinc chloride, free from lead and cadmium*

Dissolve 100 g of Zn 99.995 zinc in 400 ml of hydrochloric acid (3.2). 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 minutes. Make up to volume with water. Mix. Allow to settle. Filter the solution without washing. This solution contains approximately 200 mg of zinc per millilitre.

Verify that the step heights are less than those produced by adding 0.5 ml of the standard solution of lead and cadmium No. 3 (3.9) 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.

4. APPARATUS

4.1 *Ordinary laboratory equipment*

4.2 *Polarograph*

4.3 *Thermostatically controlled water bath*

5. SAMPLING

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

6. PROCEDURE

6.1 **Test portion**

Weigh 5 g of the test sample with an accuracy of ± 0.01 g.

6.2 **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 and 0.005 %, then

6.2.1 Into a series of 100 ml beakers, transfer 25 ml of the solution of zinc chloride (3.10) corresponding to 5 g of metallic zinc. Add 5 ml, 10 ml and 25 ml respectively of the standard solution of lead and cadmium No. 3 (3.9).

6.2.2 Evaporate to a syrupy consistency and proceed as outlined in clauses 6.3.3 to 6.3.5, then polarograph as outlined in clause 6.4.

* To be prepared later.

6.2.3 Construct a calibration curve from the step heights obtained.

6.3 Determination

6.3.1 Transfer the test portion to a 100 ml beaker and attack with 35 to 40 ml of hydrochloric acid (3.2). Add two or three drops of hydrogen peroxide (3.3) to dissolve completely.

NOTE. – If dissolution is very difficult, add 2 ml of nickel chloride solution (3.6) to expedite the attack.

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

6.3.3 Take up with water and if necessary add one or two drops of hydrochloric acid (3.2), taking care to obtain complete solution. If the presumed iron content is greater than 0.01 %, add one or two crystals of hydroxylammonium chloride (3.5) (approximately 5 mg); then warm gently to eliminate completely the possible influence of iron. Allow to cool.

6.3.4 Transfer to a 25 ml volumetric flask and make up to volume with water.

6.3.5 Transfer the appropriate quantity of this solution to the polarograph cell and place this in a thermostatically controlled water bath. De-aerate by passing an inert gas (3.1) through the solution for at least 10 minutes.

6.4 Polarographic measurement

Polarograph between 0 and - 1 volt.

The step for lead occurs at about - 0.4 volt and the step for cadmium at about - 0.6 volt relative to a mercury electrode or, respectively, at - 0.65 volt and - 0.85 volt relative to a saturated calomel electrode.

7. EXPRESSION OF RESULTS

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

8. TEST REPORT

The test report should mention the method used and the results obtained. It should also mention all operative details not provided for in this ISO Recommendation or any optional details, as well as any circumstances which could have any influence on the results.

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