UDC 669.5 : 545.33 : 669.4

Ref. No. : ISO/R 715-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 715

CHEMICAL ANALYSIS OF ZINC

POLAROGRAPHIC DETERMINATION OF LEAD

1st EDITION
May 1968

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 715, *Chemical analysis of zinc – Polarographic determination of lead,* 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. 994) 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	Israel	Spain
Australia	Ireland	Switzerland
Belgium	Italy	Thailand
Canada	Korea, Rep. of	Turkey
Chile	Korea, D.P. Rep. of	U.A.R.
Czechoslovakia	New Zealand	United Kingdom
France	Norway	U.S.A.
Hungary	South Africa, Rep. of	U.S.S.R.
India		Yugoslavia

One Member Body opposed the approval of the Draft :

Germany

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 715 May 1968

CHEMICAL ANALYSIS OF ZINC

POLAROGRAPHIC DETERMINATION OF LEAD

1. SCOPE

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

The method applies to the following types of zinc : Zn 99.5, Zn 98.5 and Zn 98, defined in ISO Recommendation R 752, *Zinc ingots.*

It is suitable for the determination of lead content between 0.1 and 3 %.

2. PRINCIPLE OF THE METHOD

Polarographic determination of lead in an ammoniacal tartrate cyanide solution.

3. REAGENTS

All the reagents should be of analytical reagent grade.

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

3.1 *Zinc* 99.99 % pure.

3.2 *Nitric acid* (d = 1.3 to 1.4).

3.3 *Hydrochloric acid* (d = 1.19).

3.4 *Ammonia solution* (d = 0.91).

3.5 *Tartaric acid solution,* 300 g per litre.

3.6 *Potassium cyanide solution,* 100 g per litre.

3.7 *Pure gelatine (sulphate free) solution,* 5 g per litre. To preserve the solution, add salicylic acid in the proportion of 1 g per litre.

3.8 *Standard lead solution No. 1*

Dissolve 1 g of pure lead in 10 ml of nitric acid (3.2) and 50 ml of water. Allow to cool. Transfer quantitatively to a 500 ml volumetric flask. Make up to volume with water.

1 ml of this solution contains 2 mg of lead.

3.9 *Standard lead solution No. 2*

Transfer 10 ml of standard lead solution No. 1 (3.8) to a 100 ml volumetric flask. Make up to volume with water.

1 ml of this solution contains 0.2 mg of lead.

4. APPARATUS

4.1 *Ordinary laboratory equipment.*

4.2 *Polarograph.*

4.3 *Thermostatically controlled water bath.*

5. SAMPLING

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

6. PROCEDURE

6.1 Test portion

Weigh 10 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 corresponding to the seven lead contents of 0, 0.1, 0.2, 0.5, 1, 2 and 3 %, then

6.2.1 Weigh 10 g of pure zinc (3.1) with an accuracy of ± 0.01 g and continue as outlined in clause 6.3.1 to clause 6.3.3.

6.2.2 Transfer 25 ml aliquots to seven 100 ml volumetric flasks.

6.2.3 Add respectively 0, 5, 10 and 25 ml of standard lead solution No. 2 (3.9) and 5, 10 and 15 ml of standard lead solution No. 1 (3.8).

6.2.4 For each flask, proceed as outlined in clause 6.3.5 to clause 6.3.7, then polarograph as outlined in clause 6.4.

6.2.5 Construct a calibration curve from the step heights obtained.

6.3 Determination

6.3.1 Transfer the test portion to a 250 ml beaker and attack with 50 ml of aqua-regia [one volume of nitric acid (3.2) and three volumes of hydrochloric acid (3.3)], added in small portions.

6.3.2 After complete solution add 50 ml of water and boil for a short time. Allow to cool.

6.3.3 Transfer quantitatively to a 250 ml volumetric flask and make up to volume with water. Mix.

6.3.4 Transfer a 25 ml aliquot to a 100 ml volumetric flask.

6.3.5 Add successively

- 10 ml of tartaric acid solution (3.5)
- 25 ml of ammonia solution (3.4).

Cool.

6.3.6 Add

- 10 ml of potassium cyanide solution (3.6)
- 3 ml of gelatine solution (3.7).

6.3.7 Make up the volume to 100 ml with water. Mix. Allow to stand for 10 minutes to ensure that oxygen is eliminated. Transfer the appropriate quantity of this solution to the polarograph cell and place this in a thermostatically controlled water bath.

6.4 Polarographic measurement

Polarograph. The half-wave potential of lead is approximately – 0.25 volt relative to a mercury electrode, or – 0.6 volt relative to a saturated calomel electrode.

* To be prepared later.

** If the lead content is greater than that at the monoeutectic point (0.9 %) or if the sample is heterogeneous, it is recommended that a larger test portion mass should be taken and an appropriate aliquot used.

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 influence on the results.

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