INTERNATIONAL STANDARD 715

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

Zinc — Determination of lead content — Polarographic method

Zinc — Dosage du plomb — Méthode polarographique

First edition — 1975-06-01

UDC 669.5 : 543.253 : 546.815

Ref. No. ISO 715-1975 (E)

Descriptors : zinc, chemical analysis, determination of content, lead (metal), polarographic analysis.

Price based on 2 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 715 and found it technically suitable for transformation. International Standard ISO 715 therefore replaces ISO Recommendation R 715-1968 to which it is technically identical.

ISO Recommendation R 715 was approved by the Member Bodies of the following countries :

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

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

Germany

The Member Body of the following country disapproved the transformation of ISO/R 715 into an International Standard :

Spain

Printed in Switzerland

Zinc — Determination of lead content — Polarographic method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarographic method for the determination of the lead content of zinc.

The method is applicable to the following types of zinc : Zn 99,5, Zn 98,5 and Zn 98, defined in ISO/R 752.

It is suitable for the determination of lead contents between 0,1 and 3 %.

2 REFERENCES

ISO/R 752, *Zinc ingots.*

ISO 3751, *Zinc ingots — Selection and preparation of samples for chemical analysis.* [1)]

3 PRINCIPLE

Polarographic determination of lead in an ammoniacal tartrate cyanide solution.

4 REAGENTS

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

4.1 Zinc, 99,99 % pure.

4.2 Nitric acid, ρ 1,3 to 1,4 g/ml.

4.3 Hydrochloric acid, ρ 1,19 g/ml.

4.4 Ammonia solution, ρ 0,91 g/ml.

4.5 Tartaric acid, 300 g/l solution.

4.6 Potassium cyanide, 100 g/l solution.

4.7 Pure gelatine (sulphate free), 5 g/l solution.

To preserve the solution, add salicylic acid in the proportion of 1 g per litre.

4.8 Lead, standard solution No. 1.

Dissolve 1 g of pure lead in 10 ml of nitric acid (4.2) and 50 ml of water. Allow to cool. Transfer quantitatively to a 500 ml volumetric flask. Dilute to the mark.

1 ml of this solution contains 2 mg of lead.

4.9 Lead, standard solution No. 2.

Transfer 10 ml of the lead standard solution No. 1 (4.8) to a 100 ml volumetric flask. Dilute to the mark.

1 ml of this solution contains 0,2 mg of lead.

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 10 g of the test sample to the nearest 0,01 g.[2)]

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 corresponding to the seven lead contents of 0 — 0,1 — 0,2 — 0,5 — 1 — 2 and 3 %, then

1) At present at the stage of draft.

2) 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.

7.2.1 Weigh 10 g of pure zinc (4.1) to the nearest 0,01 g and continue as outlined in 7.3.1 to 7.3.3.

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

7.2.3 Add respectively 0, 5, 10 and 25 ml of the lead standard solution No. 2 (4.9) and 5, 10 and 15 ml of the standard lead solution No. 1 (4.8).

7.2.4 For each flask, proceed as outlined in 7.3.5 to 7.3.7, then polarograph as outlined in 7.4.

7.2.5 Construct a calibration curve from the step heights obtained.

7.3 Determination

7.3.1 Transfer the test portion to a 250 ml beaker and attack with 50 ml of aqua-regia (1 volume of nitric acid (4.2) and three volumes of hydrochloric acid (4.3)), added in small portions.

7.3.2 After complete dissolution, add 50 ml of water and boil for a short time. Allow to cool.

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

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

7.3.5 Add successively

- 10 ml of tartaric acid solution (4.5)
- 25 ml of ammonia solution (4.4).

Cool.

7.3.6 Add

- 10 ml of potassium cyanide solution (4.6)
- 3 ml of gelatine solution (4.7).

7.3.7 Dilute to the mark and mix. Allow to stand for 10 min to ensure that oxygen is eliminated. Transfer the appropriate quantity of this solution to the polarograph cell (5.1) and place this in the thermostatically controlled water bath (5.2).

7.4 Polarographic measurement

Polarograph. The half-wave potential of lead is approximately − 0,25 V relative to a mercury electrode, or − 0,6 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.