INTERNATIONAL STANDARD 714

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

Zinc — Determination of iron content — Photometric method

Zinc — Dosage du fer — Méthode photométrique

First edition — 1975-07-15

UDC 669.5 : 543.4 : 669.12

Ref. No. ISO 714-1975 (E)

Descriptors : zinc, chemical analysis, determination of content, iron, photometric 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 714 and found it technically suitable for transformation. International Standard ISO 714 therefore replaces ISO Recommendation R 714-1968 to which it is technically identical.

ISO Recommendation R 714 was approved by the Member Bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Ireland	Switzerland
Canada	Israel	Thailand
Chile	Italy	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

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

Japan

No Member Body disapproved the transformation of ISO/R 714 into an International Standard.

Printed in Switzerland

Zinc — Determination of iron content — Photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of the iron content of zinc.

The method is applicable to the types of zinc defined in ISO/R 752, provided that the copper content does not exceed 0,01 %.

It is suitable for the determination of iron contents between 0,001 and 0,1 %.

2 REFERENCES

ISO/R 752, *Zinc ingots.*

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

3 PRINCIPLE

Photometric determination of the yellow colour of the sulphosalicylic acid ferric complex formed in an ammoniacal solution.

4 REAGENTS

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

4.1 Ammonia solution, ρ 0,91 g/ml.

4.2 Hydrochloric acid, ρ 1,19 g/ml.

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

4.4 Sulphosalicylic acid, 400 g/l solution.

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

4.6 Iron, standard solution.

Weigh 0,250 g of pure iron to the nearest 0,001 g and attack with a few millilitres of hydrochloric acid (4.2). Oxidize with a few drops of hydrogen peroxide (4.3). Decompose the excess hydrogen peroxide by boiling. Cool. Transfer quantitatively to a 1 l volumetric flask. Dilute to the mark and mix. Transfer 100 ml of this solution to a 500 ml volumetric flask. Dilute to the mark and mix.

1 ml of this solution contains 0,050 mg of iron.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Photometer, wavelength 425 nm, and **1 cm cells.** [2]

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.

7.2 Blank test

Simultaneously with the actual determination, carry out a blank test using the same quantities of each reagent and following the same procedure.

7.3 Plotting of the calibration curve [3]

7.3.1 Introduce into a series of 100 ml volumetric flasks, 0, 2, 5, 10 and 20 ml respectively of the standard iron solution (4.6).

1) At present at the stage of draft.

2) The dilutions and aliquot parts defined in this International Standard only apply if 1 cm cells are used. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

3) Valid for 1 cm cells and a range of contents of 0 — 0,1 — 0,25 — 0,5 and 1 mg of iron corresponding to 0 — 0,01 — 0,025 — 0,05 and 0,1 % in the case of the procedure specified in 7.4.2, and to 0 — 0,001 — 0,002 5 — 0,005 and 0,01 % in the case of the procedure specified in 7.4.3. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

7.3.2 Add successively

– 5 ml of sulphosalicylic acid solution (4.4),

– ammonia solution (4.1) until the solution has a yellow colour, then 20 ml in excess.

7.3.3 Cool. Dilute to the mark and mix.

7.3.4 Measure the absorbance of these solutions against the solution to which no iron has been added, with the photometer (5.1) at a wavelength of 425 nm.

7.4 Determination

7.4.1 Transfer the test portion to a 500 ml conical flask and attack with 50 ml of hydrochloric acid (4.2). Oxidize and complete the solution by adding a few drops of hydrogen peroxide (4.3). Decompose the excess hydrogen peroxide by boiling.

NOTE – If dissolution is very difficult, 2 ml of nickel chloride solution (4.5) may be added to expedite the attack.

7.4.2 For iron contents equal to or greater than 0,01 %, proceed as follows :

7.4.2.1 Allow to cool. Transfer quantitatively to a 250 ml volumetric flask. Dilute to the mark and mix.

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

7.4.2.3 Add successively

– 25 ml of water,

– 5 ml of sulphosalicylic acid solution (4.4),

– ammonia solution (4.1) until the solution has a yellow colour, then 20 ml in excess.

7.4.2.4 Cool. Dilute to the mark and mix.

7.4.3 For iron contents less than 0,01 %, proceed as follows :

7.4.3.1 Evaporate just to a syrupy consistency.

7.4.3.2 Cool.

7.4.3.3 Take up with a minimum of water and transfer quantitatively to a 100 ml volumetric flask so as not to exceed 30 ml.

7.4.3.4 Add successively

– 5 ml of sulphosalicylic acid solution (4.4),

– ammonia solution (4.1) until the solution has a yellow colour, then 50 ml in excess.

7.4.3.5 Cool. Dilute to the mark and mix.

7.5 Photometric measurement

Measure the absorbance of the solution against the blank solution at a wavelength of 425 nm.

8 EXPRESSION OF RESULTS

Determine the iron content by means of the appropriate calibration curve (7.3).

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.