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CHEMICAL ANALYSIS OF ZINC

PHOTOMETRIC DETERMINATION OF IRON

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BRIEF HISTORY

The ISO Recommendation R 714, *Chemical analysis of zinc – Photometric determination of iron,* 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. 993) 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 :

One Member Body opposed the approval of the Draft :

Japan

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 714 May 1968

CHEMICAL ANALYSIS OF ZINC

PHOTOMETRIC DETERMINATION OF IRON

1. SCOPE

This ISO Recommendation describes a photometric method for the determination of iron in zinc.

The method applies to the types of zinc defined in ISO Recommendation R 752, *Zinc ingots*, provided that the copper content does not exceed 0.01 %.

It is suitable for the determination of iron content between 0.001 and 0.1 %.

2. PRINCIPLE OF THE METHOD

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

3. REAGENTS

All the reagents should be of analytical reagent grade.

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

3.1 *Ammonia solution* (d = 0.91).

3.2 *Hydrochloric acid* (d = 1.19).

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

3.4 *Sulphosalicylic acid solution* containing 400 g per litre.

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

3.6 *Standard iron solution*

Weigh 0.250 g of pure iron with an accuracy of ± 0.001 g and attack with a few millilitres of hydrochloric acid (3.2). Oxidize with a few drops of hydrogen peroxide (3.3). Decompose the excess hydrogen peroxide by boiling. Cool. Transfer quantitatively to a 1 litre volumetric flask. Make up the volume to 1 litre with water. Mix. Transfer 100 ml of this solution to a 500 ml volumetric flask. Make up the volume to 500 ml with water. Mix.

1 ml of this solution contains 0.050 mg of iron.

4. APPARATUS

4.1 *Ordinary laboratory equipment.*

4.2 *Photometer,* wavelength 425 nm, and 1 cm cells.*

5. SAMPLING

The requirements of ISO Recommendation R ...,** *Selection 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 Blank test

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

6.3 Plotting of the calibration curve***

6.3.1 Introduce into a series of 100 ml volumetric flasks, 0, 2, 5, 10 and 20 ml respectively of standard iron solution (3.6).

6.3.2 Add successively

- 5 ml of sulphosalicylic acid solution (3.4),
- ammonia solution (3.1) until the solution has a yellow colour then 20 ml in excess.

6.3.3 Cool. Make up the volume to 100 ml with water. Mix.

6.3.4 Measure the optical density of these solutions against the solution to which no iron has been added, with the photometer (4.2) at a wavelength of 425 nm.

6.4 Determination

6.4.1 Transfer the test portion to a 500 ml conical flask and attack with 50 ml of hydrochloric acid (3.2). Oxidize and complete the solution by adding a few drops of hydrogen peroxide (3.3). Decompose the excess hydrogen peroxide by boiling.

NOTE. – If dissolution is very difficult, 2 ml of nickel chloride solution (3.5) may be added to expedite the attack.

6.4.2 For iron content equal to or greater than 0.01 %.

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

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

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

** To be prepared later.

*** 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 described in clause 6.4.2, and to 0, 0.001, 0.0025, 0.005, and 0.01 % in the case of the procedure described in clause 6.4.3. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

6.4.2.3 Add successively

- 25 ml of water,
- 5 ml of sulphosalicylic acid solution (3.4),
- ammonia solution (3.1) until the solution has a yellow colour then 20 ml in excess.

6.4.2.4 Cool. Make up the volume to 100 ml with water. Mix.

6.4.3 For iron content lower than 0.01 %.

6.4.3.1 Evaporate just to a syrupy consistency.

6.4.3.2 Cool.

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

6.4.3.4 Add successively

- 5 ml of sulphosalicylic acid solution (3.4),
- ammonia solution (3.1) until the solution has a yellow colour then 50 ml in excess.

6.4.3.5 Cool. Make up the volume to 100 ml with water. Mix.

6.5 Photometric measurement

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

7. EXPRESSION OF RESULTS

Determine the iron content by means of the appropriate calibration curve (6.3).

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