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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 849

PHOSPHORIC ACID FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

1st EDITION

October 1968

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BRIEF HISTORY

The ISO Recommendation R 849, *Phosphoric acid for industrial use – Determination of iron content – 2,2′-bipyridyl spectrophotometric method,* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1960 and led, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1110) 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 :

Austria
Belgium
Brazil
Bulgaria
Chile
Czechoslovakia
France
Germany
Hungary
India
Israel
Italy
Japan
Korea, Rep. of
New Zealand
Poland
Romania
South Africa, Rep. of
Spain
Switzerland
Thailand
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

One Member Body opposed the approval of the Draft :

Netherlands

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

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ISO Recommendation R 849 October 1968

PHOSPHORIC ACID FOR INDUSTRIAL USE

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2,2′-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

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1. SCOPE

This ISO Recommendation describes a method for the spectrophotometric determination of the iron content of phosphoric acid for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of iron content, expressed as Fe, greater than 0.001 %.

2.1 Special case

Phosphoric acid for industrial use containing polyphosphoric acids (see section 8).

NOTE. – In case there is no certainty of absence of polyphosphoric acids, carry out the determination according to the procedure of the special case directly.

3. PRINCIPLE

Preliminary reduction of trivalent iron by means of hydroxylammonium chloride.

Formation of a bivalent iron - 2,2′-bipyridyl complex in presence of ammonium acetate at pH 3.1, at a temperature of 75 °C (under the test conditions, phosphate ions do not interfere).

Spectrophotometric measurement of the coloured complex at a wavelength of about 522 nm.

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Hydrochloric acid,* approximately $d = 1.19$, 37 % (m/m) or 12 N solution.

4.2 *2,2′-bipyridyl,* 5 g/l hydrochloric acid solution.

Dissolve 0.50 g of 2,2′-bipyridyl in 10 ml of approximately $d = 1.19$ hydrochloric acid solution and dilute to 100 ml.

4.3 *Ammonium acetate,* 300 g/l solution.

Dissolve 300 g of ammonium acetate in water and dilute to 1000 ml.

4.4 *Hydroxylammonium chloride,* 100 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and dilute to 100 ml.

4.5 *Iron standard solution,* containing 2.00 g/l of Fe.

Weigh, to the nearest 1 mg, 7.022 g of ammonium ferrous sulphate hexahydrate and place in a beaker of suitable capacity. Add 50 ml of 100 g/l sulphuric acid solution, transfer quantitatively to a 500 ml one-mark volumetric flask and dilute to the mark.

1 ml of this standard solution contains 2.0 mg of Fe.

4.6 *Iron standard solution,* containing 0.020 g/l of Fe.

Transfer 10.0 ml of the standard solution (4.5) to a 1000 ml one-mark volumetric flask and dilute to the mark.

1 ml of this standard solution contains 20 μg of Fe.

The solution should be prepared just before use.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *pH-meter,* fitted with glass electrode, sensitivity at least 0.1 pH unit.

5.2 *Spectrophotometer,* or

5.3 *Photoelectric absorptiometer.*

6. PROCEDURE

6.1 Test portion

Weigh by difference, to the nearest 1 mg, approximately 5 g of the test sample.

If this quantity contains more than 500 μg of Fe, reduce the quantity of test portion, so as to have a lower iron content preferably between 200 and 300 μg.

6.2 Blank test

At the same time as the analysis, carry out a blank test using the procedure described in clause 6.4 and the same quantities of all reagents. However, for the preparation of the coloured solution, use the same volume of ammonium acetate solution (4.3) as that used in the determination.*

6.3 Preparation of calibration curve

6.3.1 *Preparation of standard matching solutions* for spectrophotometric measurement in a 1 cm cell.

6.3.1.1 PRELIMINARY CHECK OF THE pH. Place 5 ml of hydrochloric acid solution (4.1) in a beaker of suitable capacity (100 ml, for example), dilute to approximately 50 ml, add 1 ml of the hydroxylammonium chloride solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.2). Allow to stand for about 10 minutes and, using the pH-meter (5.1), adjust the pH of the solution to pH 3.1 by addition of the ammonium acetate solution (4.3). Note the quantity of ammonium acetate solution added for the pH adjustment and discard the solution

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* The difference between the pH of the sample solution and blank solution is not significant.

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6.3.1.2 PREPARATION OF STANDARD MATCHING SOLUTIONS. Into each of a series of five beakers of suitable capacity (100 ml, for example), place respectively the quantities of standard iron solution (4.6) indicated in the following table :

Volume of standard iron solution (4.6)	Corresponding mass of Fe
ml	μg
0*	0
5.0	100
10.0	200
15.0	300
25.0	500

* Compensation solution.

To each solution add 5 ml of the hydrochloric acid solution (4.1) and dilute to approximately 50 ml.

6.3.1.3 COLOUR DEVELOPMENT. To each of the above-mentioned solutions, add 1 ml of the hydroxylammonium chloride solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.2). Allow to stand for about 10 minutes, then add the quantity of ammonium acetate solution (4.3) determined following the procedure of clause 6.3.1.1.

Heat the solutions on a water bath at a temperature of approximately 75 °C for about 15 minutes and cool to room temperature. Transfer to 100 ml one-mark volumetric flasks, dilute to the mark and mix thoroughly.

6.3.2 *Spectrophotometric measurement.* Carry out the measurements using either the spectrophotometer (5.2) at a wavelength of about 522 nm or the photoelectric absorptiometer (5.3) with a suitable filter, adjusting the instrument to zero optical density using as reference the compensation solution.

6.3.3 *Preparation of calibration chart.* Prepare a calibration chart having, for example, the iron (Fe) content in microgrammes per 100 ml of the standard matching solution as abscissae and the corresponding values of optical density as ordinates.

6.4 Determination

6.4.1 *Preparation of sample solution and colour development.* Place the test portion (6.1) into a beaker of suitable capacity (100 ml, for example) and dilute to approximately 50 ml. Add 1 ml of the hydroxylammonium chloride solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.2).

Allow to stand for about 10 minutes then, using the pH-meter (5.1), adjust the pH of the solution to pH 3.1 by addition of the ammonium acetate solution (4.3).

Heat the solution on a water bath at a temperature of approximately 75 °C for about 15 minutes and cool to room temperature.

Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

6.4.2 *Spectrophotometric measurement.* Measure the optical density of the solution (6.4.1), adjusting the instrument to zero optical density using as reference the blank test solution (6.2) and following the procedure described in clause 6.3.2.

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7. EXPRESSION OF RESULTS

By reference to the calibration chart (see clause 6.3.3), read the iron content corresponding to the spectrophotometric measurement (6.4.2). The iron content, expressed as Fe, is given as a percentage, by mass, by the following formula :

$$\frac{A \times 100}{E}$$

where

A is the mass, in grammes, of iron determined in the sample solution;

E is the mass, in grammes, of the test portion.

8. SPECIAL CASE

Phosphoric acid for industrial use containing polyphosphoric acids.

8.1 Principle

Preliminary hydrolysis of polyphosphoric acids by prolonged boiling in the presence of hydrochloric acid. Determination by the general method.

8.2 Reagents

See section 4.

8.3 Apparatus

See section 5.

8.4 Procedure

8.4.1 *Test portion.* See clause 6.1.

8.4.2 *Blank test.* In a 100 ml conical flask, place approximately 25 ml of water and 10 ml of the hydrochloric acid solution (4.1). Boil for about 20 minutes.

Cool the solution and transfer to a beaker of suitable capacity (200 ml, for example), dilute with water to 40 ml and then add 10 ml of the hydrochloric acid solution (4.1).

Add 1 ml of the hydroxylammonium chloride solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.2). Then proceed as described in the second paragraph of clause 6.4.1.

8.4.3 *Preparation of calibration curve.* Follow the procedure described in clause 6.3.

However, in clauses 6.3.1.1 (preliminary check of the pH) and 6.3.1.2 (preparation of standard solutions), use 10 ml of hydrochloric acid solution (4.1) instead of 5 ml.

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