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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 852

SODIUM TRIPOLYPHOSPHATE AND SODIUM PYROPHOSPHATE FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2'-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

1st EDITION

October 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 852, *Sodium tripolyphosphate and sodium pyrophosphate 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. 1113) 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	India	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Switzerland
Bulgaria	Japan	Thailand
Chile	Korea, Rep. of	Turkey
Czechoslovakia	Morocco	U.A.R.
France	New Zealand	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	Romania	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 852 October 1968

SODIUM TRIPOLYPHOSPHATE AND SODIUM PYROPHOSPHATE FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2'-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a method for the spectrophotometric determination of the iron content of sodium tripolyphosphate and sodium pyrophosphate for industrial use, applicable to samples containing more than 0.001 % of iron expressed as Fe.

2. PRINCIPLE

Preliminary hydrolysis of polyphosphates by prolonged boiling in the presence of hydrochloric acid.

Reduction of trivalent iron by means of hydroxylammonium chloride.

Formation of a bivalent iron – 2,2'-bipyridyl complex in the 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.

3. REAGENTS

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

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

3.2 *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.

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

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

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

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

3.5 *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.

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

Transfer 10.0 ml of the iron standard solution (3.5) 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.

4. APPARATUS

Ordinary laboratory apparatus and

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

4.2 *Spectrophotometer*, or

4.3 *Photoelectric absorptiometer.*

5. PROCEDURE

5.1 Test portion

Weigh, to the nearest 1 mg, approximately 10 g of the test sample. If this quantity contains more than 1000 µg of Fe, reduce the quantity of test portion, so as to have a lower iron content, preferably between 400 and 600 µg.

5.2 Blank test

Place approximately 25 ml of water and 10 ml of the hydrochloric acid solution (3.1) in a conical flask of approximately 100 ml. Boil until the volume is reduced to about 5 ml.

Cool and transfer the solution to a beaker of suitable capacity (100 ml, for example), rinsing the flask with water so that the total volume of solution and washings is approximately 50 ml.

Add 1 ml of the hydroxylammonium chloride solution (3.4) and 5 ml of the 2,2'-bipyridyl solution (3.2). Allow to stand for 10 minutes, then add the same volume of the ammonium acetate solution (3.3) as that used in the determination (5.4.1).*

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

5.3 Preparation of calibration curve

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

5.3.1.1 PRELIMINARY CHECK OF THE pH. Place 10 ml of the hydrochloric acid solution (3.1) in a beaker of suitable capacity (100 ml, for example), dilute to approximately 50 ml, add 1 ml of the hydroxylammonium chloride solution (3.4) and 5 ml of the 2,2'-bipyridyl solution (3.2). Allow to stand for about 10 minutes and, using the pH-meter (4.1), adjust the pH of the solution to pH 3.1 by addition of the ammonium acetate solution (3.3). Note the quantity of ammonium acetate solution added for the pH adjustment and discard the solution.

5.3.1.2 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 (3.6) indicated in the following table :

Volume of standard iron solution (3.6)	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 10 ml of the hydrochloric acid solution (3.1) and dilute to approximately 50 ml.

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