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NITRIC ACID FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL PHOTOMETRIC METHOD

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BRIEF HISTORY

The ISO Recommendation R 1982, *Nitric acid for industrial use – Determination of iron content – 2,2'-bipyridyl photometric 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 led to the adoption of Draft ISO Recommendation No. 1982, which was circulated to all the ISO Member Bodies for enquiry in May 1970. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Iran	Romania
Austria	Ireland	South Africa, Rep. of
Belgium	Israel	Switzerland
Chile	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
France	New Zealand	U.A.R.
Germany	Peru	United Kingdom
Greece	Poland	U.S.A.
India	Portugal	U.S.S.R.

No Member Body opposed the approval of the Draft.

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

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ISO Recommendation | R 1982 | May 1971

NITRIC ACID FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL PHOTOMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a 2,2′-bipyridyl method for the photometric determination of the iron content of nitric acid for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of iron contents, expressed as Fe, greater than 0.000 1 % (m/m).

3. PRINCIPLE

Evaporation of a test portion, taking up by means of hydrochloric acid and reduction of iron (III) by means of hydroxylammonium chloride.

Formation of iron (II)-2,2′-bipyridyl complex in ammonium acetate medium.

Photometric 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*, ρ 1.19 g/ml approximately, 38 % (m/m) solution or approximately 12 N.

4.2 *Hydrochloric acid*, approximately N solution.

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

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

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

Dissolve 30 g of ammonium acetate (CH_3COONH_4) in water and dilute to 100 ml.

4.5 *2,2′-bipyridyl*, 10 g/l hydrochloric acid solution.

Dissolve 1 g of 2,2′-bipyridyl in 10 ml of the hydrochloric acid solution (4.2) and dilute to 100 ml.

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

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

1 ml of this standard solution contains 2.00 mg of Fe.

4.7 *Iron standard solution*, containing 0.200 g/l of Fe.

Transfer 50.0 ml of the iron standard solution (4.6) to a 500 ml one-mark volumetric flask and add 5 ml of 100 g/l sulphuric acid solution. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 0.20 mg of Fe.

The solution should be prepared just before use.

4.8 *Iron standard solution*, containing 0.010 g/l of Fe.

Transfer 50.0 ml of the iron standard solution (4.7) to a 1000 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 10 μg of Fe.

The solution should be prepared just before use.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Weighing pipette*, capacity 60 ml approximately, with ground glass stopper.

5.2 *Platinum or quartz dish*, approximately 100 ml capacity.

5.3 *Spectrophotometer*, or

5.4 *Photoelectric absorptiometer*.

6. PROCEDURE

6.1 Test portion

Fill the weighing pipette (5.1) with the test sample and weigh, by difference to the nearest 10 mg, a test portion of approximately 50 g. Transfer the test portion to the dish (5.2).

6.2 Blank test

At the same time as the analysis, carry out a blank test using the same procedure and the same quantities of all reagents employed in the test.

6.3 Preparation of calibration curve

6.3.1 *Preparation of the standard colorimetric solutions* for photometric measurement with 1 cm cell. Into each of a series of eleven 100 ml one-mark volumetric flasks, place the quantities of the standard iron solution (4.8) indicated in the following table :

Volume of the standard iron solution (4.8)	Corresponding mass of iron (Fe)
ml	μg
0*	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

* Compensation solution

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of the hydrochloric acid solution (4.2), 2 ml of the hydroxylammonium chloride solution (4.3) and, after 5 minutes, 5 ml of the ammonium acetate solution (4.4) and 1 ml of the 2,2′-bipyridyl solution (4.5). Dilute to the mark, mix thoroughly and wait 10 minutes.

6.3.2 *Photometric measurements.* Carry out the photometric measurements using either the spectrophotometer (5.3) at a wavelength of about 522 nm or the photoelectric absorptiometer (5.4) with suitable filters, adjusting the instrument to zero absorbance, using the compensation solution as reference.

6.3.3 *Preparation of calibration chart.* Prepare a calibration graph having, for example, the iron content in micrograms per 100 ml of the standard colorimetric solution as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of sample solution.* Place the dish containing the test portion (6.1) on a boiling water bath and evaporate to dryness. Cool, take up with 2 ml of the hydrochloric acid solution (4.1) and 5 ml of water. Evaporate a second time on a boiling water bath. Take up with 2 ml of hydrochloric acid solution (4.1) and 25 ml of water, warming to assist solution. Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and filter, if necessary, with a dry filter into a dry vessel.

6.4.2 *Colour development.* Transfer an aliquot of the sample solution (6.4.1) containing between 50 and 500 μg of iron, to a 100 ml one-mark volumetric flask. Dilute to approximately 50 ml if necessary, then add successively 2 ml of the hydrochloric acid solution (4.2), 2 ml of hydroxylammonium chloride solution (4.3) and, after 5 minutes, 5 ml of the ammonium acetate solution (4.4) and 1 ml of the 2,2′-bipyridyl solution (4.5). Dilute to the mark, mix and wait 10 minutes.

6.4.3 *Photometric measurements.* Carry out the photometric measurements according to the procedure given in clause 6.3.2, adjusting the instrument to zero absorbance using as reference the blank test solution (6.2).

7. EXPRESSION OF RESULTS

By reference to the calibration chart (see clause 6.3.3), read the iron content corresponding to the photometric measurement.

The iron content, expressed as Fe, is given, as a percentage by mass, by the formula

$$\frac{m_1 \times 100 \times 100}{V \times m_0}$$

where

m_0 is the mass, in grammes, of the test portion;

m_1 is the mass, in grammes, of iron determined in the aliquot of the sample solution;

V is the volume, in millilitres, of the sample solution taken for the colour reaction.

8. TEST REPORT

The test report should give the following particulars :

(*a*) the reference of the method used;

(*b*) the results, and the method of expression used;

(*c*) any unusual features noted during the determination;

(*d*) any operation not included in this ISO Recommendation or regarded as optional.

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