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R 1982

NITRIC ACID FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL PHOTOMETRIC METHOD

1st EDITION

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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.