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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1707

FORMIC 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 1707, *Formic 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. 1707, which was circulated to all the ISO Member Bodies for enquiry in March 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Romania
Austria	Iran	Spain
Belgium	Israel	Sweden
Brazil	Japan	Switzerland
Canada	Netherlands	Turkey
Czechoslovakia	New Zealand	U.A.R.
France	Peru	United Kingdom
Germany	Poland	U.S.S.R.
Greece	Portugal	Yugoslavia
Hungary	South Africa, Rep. of	

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.

ISO Recommendation R 1707 August 1970

FORMIC ACID FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL PHOTOMETRIC METHOD

INTRODUCTION

This ISO Recommendation supplements ISO Recommendation R 731, *Formic acid for industrial use – Methods of test,* which describes the following methods of test :

- Determination of total acidity.
- Determination of acids other than formic acid.
- Limit test for inorganic chlorides.
- Limit test for inorganic sulphates.

A sample of the material not less than 200 g is necessary to carry out all the tests described in the two documents.

1. SCOPE

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

2. SAMPLE*

Take a volume of sample that is sufficient for the analysis to be carried out, so that it is representative of the bulk.

Place the sample in a clean, dry and airtight glass-stoppered bottle of such a size that it is nearly filled by the sample.

If it has been necessary to seal the container, care should be taken to avoid contaminating the contents in any way.

3. PRINCIPLE

Conversion of any iron present into the sulphate by evaporation to dryness in the presence of sulphuric acid, and reduction of trivalent iron by means of hydroxylammonium chloride.

Formation of a bivalent iron 2,2′-bipyridyl complex. Photometric measurement of the coloured complex at about 520 nm.

NOTE. – Although this method specifies the use of a spectrophotometer or photoelectric absorptiometer, it is permissible to employ, as an alternative procedure, a visual method (see Note to clause 6.4.3).

* Sampling of chemical products will form the subject of a further ISO Recommendation.

4. REAGENTS

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

4.1 *Sulphuric acid,* approximately ρ 1.84 (g/ml), 96 % (m/m) solution, diluted 1 + 6 by volume.

4.2 *Nitric acid,* approximately ρ 1.4 (g/ml), 68 % (m/m) solution, diluted 1 + 3 by volume.

4.3 *Urea,* solution.

Dissolve 100 g of urea in 100 ml of water.

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 *Ammonium acetate,* 500 g/l solution.

Dissolve 50 g of ammonium acetate (CH_3COONH_4) in water and dilute to 100 ml.

4.6 *2,2'-bipyridyl,* 5 g/l hydrochloric acid solution.

Dissolve 0.5 g of 2,2'-bipyridyl in 10 ml of approximately N hydrochloric acid solution and dilute to 100 ml.

4.7 *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 25 ml of the sulphuric acid solution (4.1) 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.

4.8 *Iron standard solution,* containing 0.20 g/l of Fe.

Transfer 50.0 ml of the iron standard solution (4.7) to a 500 ml one-mark volumetric flask, add 2.5 ml of the sulphuric acid solution (4.1). 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.

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

Transfer 50.0 ml of the iron standard solution (4.8) 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 *Spectrophotometer* or, alternatively,

5.2 *Photoelectric absorptiometer* or, alternatively,

5.3 *Two matched Nessler cylinders* of 100 ml capacity.

6. PROCEDURE

6.1 Test portion

Weigh 100 g of the test sample in a platinum basin of capacity about 150 ml.

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 the 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 seven 100 ml one-mark volumetric flasks, place respectively the quantities of standard iron solution (4.9) indicated in the following table :

Volume of standard iron solution (4.9)	Corresponding mass of iron
ml	μg
0*	0
2.0	20
4.0	40
7.0	70
10.0	100
15.0	150
20.0	200

* Compensation solution

Add to each volumetric flask 10 ml of the sulphuric acid solution (4.1), 20 ml of the nitric acid solution (4.2) and 2 ml of the urea solution (4.3), mix and add 2 ml of hydroxylammonium chloride solution (4.4), mix and allow to stand for 2 minutes. Then add 30 ml of the amonium acetate solution (4.5) and 5 ml of the 2,2′-bipyridyl solution (4.6). Dilute to the mark, mix thoroughly and allow to stand for 10 minutes.

6.3.2 *Photometric measurements.* Carry out the photometric measurements using either the spectrophotometer (5.1) at a wavelength of approximately 520 nm, or the photoelectric absorptiometer (5.2) with a suitable filter, adjusting the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of calibration chart.* Prepare a calibration chart having, for example, the iron content in microgrammes 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.* Add to the platinum basin containing the test portion (6.1) 10 ml of the sulphuric acid solution (4.1). Evaporate, first on a boiling water bath and finally on a sand bath until white fumes are just evolved. Allow to cool, add a few drops of the nitric acid solution (4.2) and evaporate again until white fumes just cease to be evolved. If tarry products remain, add a few more drops of nitric acid solution (4.2) and again evaporate on the sand bath. Take up the residue with 20 ml of the nitric acid solution (4.2), warming to assist solution of salts.

Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, rinsing the platinum basin, with about 5 ml of the nitric acid solution (4.2).