INTERNATIONAL STANDARD ISO 731/VI

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION·МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ·ORGANISATION INTERNATIONALE DE NORMALISATION

Formic acid for industrial use — Methods of test — Part VI : Determination of iron content — 2,2′-Bipyridyl photometric method

Acide formique à usage industriel — Méthodes d'essai — Partie VI : Dosage du fer — Méthode photométrique au bipyridyle-2,2′

First edition — 1977-03-15

UDC 661.732.1 : 546.72 : 543.422

Ref. No. ISO 731/VI-1977 (E)

Descriptors : formic acid, tests, chemical analysis, determination of content, acidity, chlorides, sulphates.

Price based on 3 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1707-1970 and found it technically suitable for transformation. Number 1707, however, has been changed to ISO 731/VI. International Standard ISO 731/VI therefore replaces ISO Recommendation R 1707-1970, to which it is technically identical.

ISO Recommendation R 1707 had been approved by the member bodies of the following countries :

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

No member body had expressed disapproval of the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

Formic acid for industrial use — Methods of test — Part VI : Determination of iron content — 2,2′-Bipyridyl photometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 731 specifies a 2,2′-bipyridyl photometric method for the determination of the iron content of formic acid for industrial use.

The method is applicable to products having Fe contents in the range 0,1 to 2 mg/kg.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Conversion of the iron in a test portion to iron(III) sulphate by evaporation to dryness in the presence of sulphuric acid.

Dissolution of the residue in nitric acid. Reduction, by hydroxylammonium chloride, of the trivalent iron contained in the solution thus obtained. Formation of the coloured complex iron(II)-2,2′-bipyridyl in a buffered medium. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

3.1 Sulphuric acid, ρ approximately 5 N solution.

3.2 Nitric acid, approximately 4 N solution.

3.3 Urea (NH_2–CO–NH_2), solution.

Dissolve 100 g of urea in 100 ml of water.

3.4 Hydroxylammonium chloride ($NH_2OH.HCl$), 100 g/l solution.

3.5 Ammonium acetate (CH_3COONH_4), 500 g/l solution.

3.6 2,2′-Bipyridyl, 5 g/l hydrochloric solution.

Dissolve 0,5 g of 2,2′-bipyridyl in 10 ml of approximately 1 N hydrochloric acid solution and dilute to 100 ml.

3.7 Iron, standard solution corresponding to 2,00 g of Fe per litre.

Weigh, to the nearest 0,001 g, 7,022 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2SO_4.FeSO_4.6H_2O$], and dissolve in 25 ml of the sulphuric acid solution (3.1). Transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 2,00 mg of Fe.

3.8 Iron, standard solution corresponding to 0,20 g of Fe per litre.

Place, in a 500 ml one-mark volumetric flask, 50,0 ml of the standard iron solution (3.7), add 2,5 ml of the sulphuric acid solution (3.1), dilute to the mark and mix.

1 ml of this standard solution contains 0,20 mg of Fe.

Prepare this solution just before use.

3.9 Iron, standard solution corresponding to 0,010 g of Fe per litre.

Place, in a 1 000 ml one-mark volumetric flask, 50,0 ml of the standard iron solution (3.8), dilute to the mark and mix.

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

Prepare this solution just before use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum dish, of capacity about 150 ml.

4.2 Spectrophotometer, or

4.3 Photoelectric absorptiometer, fitted with filters allowing maximum transmission between 500 and 520 nm.

5 PROCEDURE

5.1 Test portion

Weigh 100 ± 1 g of the laboratory sample into the platinum dish (4.1).

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used for the determination, but omitting the test portion.

5.3 Preparation of calibration graph

5.3.1 *Preparation of standard colorimetric solutions* for photometric measurements with cells of 1 cm optical path length

Into a series of seven 100 ml one-mark volumetric flasks, place the volumes of the standard iron solution (3.9) shown in the following table :

Standard iron solution (3.9)	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

* Blank test of reagents for calibration graph.

5.3.2 *Colour development*

Treat the contents of each flask as follows :

Add 10 ml of the sulphuric acid solution (3.1), 20 ml of the nitric acid solution (3.2), 2 ml of the urea solution (3.3) and 2 ml of the hydroxylammonium chloride solution (3.4). Mix and allow to stand for 2 min. Then add 30 ml of the ammonium acetate solution (3.5) and 5 ml of the 2,2′-bipyridyl solution (3.6). Dilute to the mark, mix and allow to stand for 10 min.

5.3.3 *Photometric measurements*

Using the spectrophotometer (4.2), at a wavelength of about 510 nm, or the photoelectric absorptiometer (4.3) fitted with suitable filters, carry out the photometric measurement of each standard colorimetric solution, after having adjusted the instrument to zero absorbance against the solution for the blank test of the reagents for the calibration graph.

5.3.4 *Plotting of the graph*

Plot a graph having, for example, as abscissae, the values, expressed in micrograms, of the quantities of iron (Fe) contained in 100 ml of standard matching solution (5.3.1) and, as ordinates, the corresponding measured values of the absorbance.

5.4 Determination

5.4.1 *Preparation of the test solution*

Add 10 ml of the sulphuric acid solution (3.1) to the platinum dish containing the test portion (5.1). Evaporate in a fume cupboard, 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 (3.2) and evaporate again until white fumes just cease to be evolved. If tarry products remain, add a few more drops of the nitric acid solution and again evaporate on a sand bath. Add to the residue 20 ml of the nitric acid solution, warming to facilitate dissolution of the salts.

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

5.4.2 *Colour development*

Carry out the colour development of the test solution as specified in 5.3.2, but omitting the addition of 20 ml of the nitric acid solution (3.2).

5.4.3 *Photometric measurement*

Carry out the photometric measurement of the test solution as specified in 5.3.3, after having, however, adjusted the instrument to zero absorbance against the blank test solution (5.2).

NOTE — As an alternative to the measurement of absorbance, the test solution, prepared in accordance with 5.4.1 and 5.4.2, may be compared visually with a series of standard colorimetric solutions prepared under similar conditions, and its iron content deduced from this comparison.

6 EXPRESSION OF RESULTS

By reference to the calibration graph (5.3.4), determine the mass of iron corresponding to the absorbance of the test solution.

The iron content, expressed in milligrams of iron (Fe) per kilogram, is given by the formula

$$\frac{m}{100}$$

where m is the mass, in micrograms, of iron found in the test solution (5.4.1).

ANNEX

ISO PUBLICATIONS RELATING TO FORMIC ACID FOR INDUSTRIAL USE

ISO 731/I – General.

ISO 731/II – Determination of total acidity – Titrimetric method.

ISO 731/III – Determination of content of other acids – Potentiometric method.

ISO 731/IV – Visual limit test for inorganic chlorides.

ISO 731/V – Visual limit test for inorganic sulphates.

ISO 731/VI – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 731/VII – Determination of low contents of other volatile acids – Titrimetric method after distillation.

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