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Paraformaldehyde for industrial use — Methods of test — Part III : Determination of iron content — 2,2′-Bipyridyl photometric method

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

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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 1391-1970 and found it technically suitable for transformation. The technical committee, however, divided the recommendation into four parts (ISO 1391, parts I to IV), which therefore replace ISO Recommendation R 1391-1970, to which they are technically identical.

ISO Recommendation R 1391 had been approved by the member bodies of the following countries :

Austria	Ireland	Romania
Belgium	Italy	South Africa, Rep. of
Brazil	Japan	Spain
Czechoslovakia	Korea, Rep. of	Sweden
France	Netherlands	Switzerland
Germany	New Zealand	Thailand
Hungary	Poland	Turkey
Iran	Portugal	United Kingdom

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

India

The member bodies of the following countries disapproved the transformation of the Recommendation into an International Standard :

France
Netherlands

Printed in Switzerland

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Paraformaldehyde for industrial use — Methods of test — Part III : Determination of iron content — 2,2′-Bipyridyl photometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1391 specifies a 2,2′-bipyridyl photometric method for the determination of the iron content of paraformaldehyde for industrial use.

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

2 PRINCIPLE

Conversion of any iron present in a test portion into the sulphate by hot sulphuric acid.

Oxidation of any iron present in the solution by hydrogen peroxide, followed by reduction, by hydroxylammonium chloride, of the trivalent iron. 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, approximately 5 N solution.

3.2 Hydrogen peroxide, 150 g/l solution.

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

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

3.5 2,2′-Bipyridyl, 5 g/l hydrochloric solution.

Dissolve 0,5 g of 2,2′-bipyridyl in 100 ml of 1 N hydrochloric acid solution.

3.6 Iron, standard solution corresponding to 0,100 g of Fe per litre.

Weigh, to the nearest 0,000 1 g, 0,702 2 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2SO_4.FeSO_4.6H_2O$] and dissolve in 50 ml of the sulphuric acid solution (3.1). Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of Fe.

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

Transfer 100 ml of the standard iron solution (3.6) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

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

Prepare this solution immediately before use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer, fitted with filters allowing maximum transmission at about 510 nm.

5 PROCEDURE

5.1 Preparation of calibration graph

5.1.1 *Preparation of standard matching solutions* relating to measurements carried out with cells of 4 or 5 cm optical path length

Into a series of seven 400 ml beakers, place the volumes of the standard iron solution (3.7) shown in the following table :

Standard iron solution (3.7)	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.

Add, to each beaker, in successive small portions, 10 ml of the hydrogen peroxide solution (3.2) and 10 ml of the sulphuric acid solution (3.1) and then heat on a sand bath in a fume cupboard until acid fumes are evolved.

5.1.2 *Colour development*

Treat the contents of each beaker as follows :

Allow to cool to ambient temperature and transfer the solutions quantitatively to 100 ml one-mark volumetric flasks. Add, to each, 2 ml of the hydroxylammonium chloride solution (3.3). Mix and allow to stand for 2 min. Then add 30 ml of the ammonium acetate solution (3.4) and 5 ml of the 2,2′-bipyridyl solution (3.5). Dilute to the mark and mix.

5.1.3 *Photometric measurements*

Using the spectrophotometer (4.1), at a wavelength of about 510 nm, or the photoelectric absorptiometer (4.2), fitted with suitable filters, carry out the photometric measurement of each standard matching solution, after having adjusted the instrument to zero absorbance against the solution for the blank test of the reagents for the calibration graph.

5.1.4 *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 (5.1.1) and, as ordinates, the corresponding measured values of the absorbance.

5.2 Determination

5.2.1 *Test portion*

Weigh accurately 3 to 4 g of the test sample into a 400 ml beaker.

5.2.2 *Preparation of test solution*

To the beaker containing the test portion (5.2.1), add 20 ml of water followed by 10 ml of the sulphuric acid solution (3.1). Evaporate the mixture on a sand bath in a fume cupboard until acid fumes are evolved.

Allow to cool to ambient temperature. When cold add, in successive small portions, 10 ml of the hydrogen peroxide solution (3.2) and heat on a sand bath in a fume cupboard until acid fumes are evolved.

5.2.3 *Colour development*

Carry out the colour development of the test solution as specified in 5.1.2.

5.2.4 *Photometric measurement*

Carry out the photometric measurement of the test solution, as specified in 5.1.3, after having adjusted the instrument to zero absorbance against water.

NOTE – As an alternative to the measurement of absorbance, the test solution, prepared in accordance with 5.2.2 and 5.2.3, may be compared visually with a series of standard matching solutions prepared under similar conditions, and its iron content deduced from this comparison.

6 EXPRESSION OF RESULTS

By reference to the calibration graph (5.1.4), 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

$$\frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (5.2.1);

m_1 is the mass, in micrograms, of iron found in the test solution (5.2.2).

ANNEX

ISO PUBLICATIONS RELATING TO PARAFORMALDEHYDE FOR INDUSTRIAL USE

ISO 1391/I – General.

ISO 1391/II – Determination of ash.

ISO 1391/III – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 1391/IV – Determination of water-insoluble matter.