INTERNATIONAL STANDARD 2226

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

Formaldehyde solutions for industrial use — Determination of iron content — 2,2′-bipyridyl photometric method

First edition — 1972-12-01

UDC 661.727.1 : 542.61 : 546.72 : 543.42

Ref. No. ISO 2226-1972 (E)

Descriptors : formaldehyde, chemical analysis, determination of content, photometry, iron.

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.

International Standard ISO 2226 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in July 1971 by the Member Bodies of the following countries :

Austria	Ireland	Sweden
Belgium	Israel	Switzerland
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	U.S.A.
Germany	Romania	U.S.S.R.
Hungary	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Printed in Switzerland

Formaldehyde solutions for industrial use — Determination of iron content — 2,2′-bipyridyl photometric method

WARNING

Formaldehyde is toxic. It is therefore necessary to avoid inhaling its vapour during sampling and testing.

1 SCOPE

This International Standard specifies a 2,2′-bipyridyl photometric method for the determination of iron content of formaldehyde solutions for industrial use.

2 FIELD OF APPLICATION

The method as described is applicable to the determination of iron contents less than 0,000 4 %.

NOTE — The field of application may be extended to iron contents greater than 0,000 4 % by reducing the mass of the test portion in a suitable manner.

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 a wavelength of about 522 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 7.4.3).

4 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Sulphuric acid, ρ 1,84 g/ml, approximately 96 % (*m/m*) solution, diluted 1 + 6 by volume.

4.2 Hydrogen peroxide, 150 g/l 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, 500 g/l solution.

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

4.5 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.

4.6 Iron (II) standard solution, containing 2,00 g of Fe per litre.

Weigh, to the nearest 0,001 g, 7,022 g of ammonium iron(II) 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.

4.7 Iron (II) standard solution, containing 0,200 g of Fe per litre.

Transfer 50,0 ml of the iron standard solution (4.6) 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 shall be prepared just before use.

4.8 Iron (II) standard solution, containing 0,010 g of Fe per litre.

Transfer 50,0 ml of the iron standard solution (4.7) to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 0,01 mg of Fe.

The solution shall 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.

6 SAMPLING

Follow the principles given in ISO ...[1].

Attention is drawn to the following recommendation. Place the laboratory sample, representative of the material taken from the bulk, in a clean, dry, and air-tight glass bottle, fitted with a ground glass stopper, of such a size that it is nearly filled by the sample.

If it is necessary to seal this bottle care shall be taken to avoid the risk of contamination.

Owing to polymerization, paraformaldehyde will tend to be deposited on standing and this will occur more rapidly if the temperature is allowed to fall below 25 °C. Accordingly the material shall be sampled as soon as possible after receipt.

7 PROCEDURE

7.1 Test portion

Weigh 50 ± 0,5 g of the laboratory sample into a 400 ml beaker.

7.2 Blank test

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

7.3 Preparation of calibration curve

7.3.1 *Preparation of the standard matching solutions* for photometric measurements with 1 cm cells.

Into each of a series of seven 400 ml beakers, place respectively the quantities of the iron standard solution (4.8) indicated in the following table :

Volume of the iron standard solution (4.8)	Corresponding mass of iron (Fe)
ml	mg
0*	0
2,0	0,020
4,0	0,040
7,0	0,070
10,0	0,100
15,0	0,150
20,0	0,200

* Compensation solution

Add to each beaker, in successive small portions, 10 ml of the hydrogen peroxide solution (4.2) and 10 ml of the sulphuric acid solution (4.1), then heat on a sand bath until acid fumes are evolved.

Allow to cool to room temperature and transfer the solutions quantitatively to 100 ml one-mark volumetric flasks. Add to each flask 2 ml of the hydroxylammonium chloride solution (4.3). Mix and allow to stand for 2 min. Then add 30 ml of the ammonium acetate solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.5). Dilute to the mark, mix thoroughly and allow to stand for 10 min.

7.3.2 *Photometric measurements*

Carry out the photometric measurements using either the spectrophotometer (5.1) at a wavelength of approximately 522 nm, or the photoelectric absorptiometer (5.2) with suitable filters, adjusting the instrument to zero absorbance against the compensation solution.

7.3.3 *Preparation of calibration chart*

Prepare a calibration chart having, for example, the iron contents in milligrams per 100 ml of the standard matching solutions as abscissae and the corresponding values of absorbance as ordinates.

7.4 Determination

7.4.1 *Preparation of test solution*

Add to the beaker containing the test portion (7.1) 10 ml of the sulphuric acid solution (4.1) and evaporate on a sand bath until white fumes are just evolved.

Allow to cool to ambient temperature. Add to the cold solution, in successive small portions, 10 ml of the hydrogen peroxide solution (4.2) and heat on the sand bath until white fumes are just evolved.

Allow to cool to room temperature, and transfer the solution quantitatively to a 100 ml one-mark volumetric flask.

7.4.2 *Colour development*

Add 2 ml of the hydroxylammonium chloride solution (4.3) to the test solution (7.4.1) in the volumetric flask, mix and allow to stand for 2 min. Then add 30 ml of the ammonium acetate solution (4.4) and 5 ml of the 2,2′-bipyridyl solution (4.5), dilute to the mark, mix thoroughly and allow to stand for 10 min.

7.4.3 *Photometric measurement*

Carry out the photometric measurement according to the procedure of 7.3.2, adjusting the instrument to zero absorbance against the blank test solution (7.2).

1) Sampling from the consignment of the product will form the subject of a future International Standard.

NOTE — As an alternative to measurement of absorbance using a spectrophotometer or a photoelectric absorptiometer, the test solution prepared as in 7.4.1 may be compared visually in the matched Nessler cylinders (5.3), either with standard matching solutions (7.3.1) of colour intensity similar to that of the test solution, or with standard colorimetric discs, and its iron content (mg Fe/100 ml) deduced.

8 EXPRESSION OF RESULTS

By reference to the calibration chart (see 7.3.3), 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

$$\frac{m_1}{10\, m_0}$$

where

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

m_1 is the mass, in milligrams, of iron found in the test solution.

9 TEST REPORT

The test report shall include 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 International Standard, or regarded as optional.

This page intentionally left blank

This page intentionally left blank

This page intentionally left blank