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

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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.

International Standard ISO 2886 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in July 1972.

It has been approved by the Member Bodies of the following countries :

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

This international Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

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

2 PRINCIPLE

Evaporation to dryness of a test portion and dissolution of the residue in hydrochloric acid. 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 comparing the test solution with a series of reference solutions (see Note to 6.4.3).

3 REAGENTS

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

3.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

3.2 Sulphuric acid. Dilute the concentrated acid (ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution) 1 + 6 by volume.

3.3 Hydroxylammonium chloride, 100 g/l solution.

3.4 Ammonium acetate, 500 g/l solution.

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

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

3.6 Standard iron solution, corresponding to 0,01 g of Fe per litre.

Dissolve 0,702 g of ammonium iron(II) sulphate hexahydrate $[(NH_4)_2SO_4.FeSO_4.6H_2O]$ in 25 ml of the sulphuric acid solution (3.2), dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

Transfer 100,0 ml of the solution thus obtained 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.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum basin, about 150 ml capacity.

4.2 Electric oven, capable of being controlled at 110 ± 2 °C.

4.3 Spectrophotometer or alternatively,

4.4 Photoelectric absorptiometer.

4.5 Stirrer, of platinum wire.

5 SAMPLING

CAUTION : Acetaldehyde has an irritant vapour and is highly flammable.

Follow the principles described in ISO . . ., *Chemical products for industrial use — Sampling.*[1)]

Liquid acetaldehyde (boiling point 20,2 °C) exerts a vapour pressure of about 1,5 bar[2)] at 30 °C and laboratory samples must be taken with care and placed in clean stainless steel flasks purged with nitrogen, fitted with a screw cap and designed to withstand the internal pressure generated at foreseeable storage temperatures. Test portions shall only be taken from containers at temperatures below 20 °C unless equipment designed for transferring liquids under pressure is employed and the container is fitted with a valve for connection to the receiver.

1) In preparation.

2) 1 bar = 100 kPa.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 1 g, about 100 ml of the laboratory sample into the clean platinum basin (4.1), previously dried in the oven (4.2) at 110 ± 2 °C, cooled in a desiccator and weighed to the nearest 0,1 g.

6.2 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 reagents employed in the test.

6.3 Preparation of calibration curve

6.3.1 *Preparation of standard colorimetric solutions* for photometric measurements with 1 cm cells.

Into a series of seven 100 ml one-mark volumetric flasks, place the volumes of the standard iron solution (3.6) shown in the following table :

Standard iron solution (3.6)	Corresponding mass of iron (Fe)
ml	µg
0*	0
2,0	20
4,0	40
7,0	70
10,0	100
15,0	150
20,0	200

* Compensation solution.

To each flask add 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, mix and allow to stand for 10 min.

6.3.2 *Photometric measurements*

Measure the absorbances of the standard colorimetric solutions (6.3.1) using either the spectrophotometer (4.3) at a wavelength of about 522 nm or the photoelectric absorptiometer (4.4) with suitable filters after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of calibration chart*

Draw a graph plotting absorbances as a function of quantities of iron (expressed in micrograms) in 100 ml of the standard colorimetric solutions.

6.4 Determination

6.4.1 *Preparation of the test solution*

Evaporate the test portion (6.1) gently to dryness in a fume cupboard and then dry the residue in the oven (4.2) maintained at 110 ± 2 °C. Cool and add 5 ml of the hydrochloric acid solution (3.1). Heat the basin on a boiling water bath, agitating the contents of the basin with the stirrer of platinum wire (4.5) until all the residue has dissolved. Allow to cool and transfer the solution quantitatively to a 100 ml one-mark volumetric flask.

6.4.2 *Colour development*

Add 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), 5 ml of the 2,2'-bipyridyl solution (3.5), dilute to the mark, mix and allow to stand for 10 min.

6.4.3 *Photometric measurements*

Measure the absorbances of the test solution and of the blank by means of the spectrophotometer (4.3) or photoelectric absorptiometer (4.4), according to the procedure described in 6.3.2, after having adjusted the instrument to zero absorbance against water.

NOTE — As an alternative to measurement of absorbance using a spectrophotometer or photoelectric absorptiometer, the colour of the solution prepared as described in 6.4.2 may be compared visually with the standard colorimetric solutions (6.3.1).

7 EXPRESSION OF RESULTS

By reference to the calibration chart (see 6.3.3), determine the masses of iron corresponding to the absorbances of the test solution and that of the blank.

The iron content, expressed as Fe, is given, in milligrams per kilogram, by the formula

$$\frac{m_1 - m_2}{m_0}$$

where

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

m_1 is the mass, in micrograms, of iron found in the test solution;

m_2 is the mass, in micrograms, of iron found in the blank test.

8 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.

ANNEX

This document is one of a series of International Standards specifying methods of test for acetaldehyde for industrial use. A list of methods established or in course of preparation is as follows :

ISO 2513 – Determination of density at 15 °C.

ISO 2514 – Determination of water content by the Karl Fischer method.

ISO 2885 – Determination of total content of carbonyl compounds – Volumetric method.

ISO 2886 – Determination of iron content, 2,2'-bipyridyl photometric method.

ISO . . . – Determination of total chlorine content.[1)]

ISO . . . – Determination of acidity to phenolphthalein.[1)]

1) In preparation.

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