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Acetaldehyde for industrial use — Determination of total content of carbonyl compounds — Volumetric method

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FOREWORD

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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 2885 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 :

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	
Ireland	South Africa, Rep. of	

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 total content of carbonyl compounds — Volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the total content of carbonyl compounds in acetaldehyde for industrial use.

2 PRINCIPLE

Reaction of the carbonyl compounds present with hydroxylammonium chloride, and titration with standard volumetric sodium hydroxide solution in the presence of bromophenol blue as indicator.

3 REAGENTS

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

3.1 Hydroxylammonium chloride, 72,5 g/l solution.

3.2 Sodium hydroxide, 1,00 M standard volumetric solution.

3.3 Bromophenol blue, 0,4 g/l solution in alkaline methanol.

Dissolve 0,04 g of bromophenol blue in a mixture of 20 ml of methanol and 80 ml of water, and add 0,1 N sodium carbonate solution until the colour is wine-red.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Glass ampoules, with two capillary arms, of 2 to 3 ml capacity.

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.

6 PROCEDURE

6.1 Test portion

Introduce about 1,5 g of the test sample into each of two of the ampoules (4.1), weighed to the nearest 0,1 mg, by immersing the ampoules in a test tube containing the test sample.

Seal the upper capillaries of the ampoules.

Take out the ampoules and place them on a capsule filled with solid carbon dioxide, making sure that the capillaries which are not sealed are slightly raised. Allow to cool for 1 to 2 min.

Seal the second capillaries.

Rinse the ampoules with a jet of acetone, then with a jet of ether, and wait until they have attained ambient temperature. Weigh the ampoules and contents to the nearest 0,1 mg.

6.2 Blank test

Carry out a blank test by titrating 50 ml of the hydroxylammonium chloride solution (3.1) and 25 ml of water with the sodium hydroxide solution (3.2), using the same quantity of the bromophenol blue solution (3.3) as in the determination.

6.3 Determination

Place in each of two 250 ml conical flasks fitted with conical ground glass necks, four or five lengths of glass (length 20 mm, diameter 6 mm) cut from a rod (these lengths of glass serve to break up the ampoule completely) and 50 ml of the hydroxylammonium chloride solution (3.1).

1) In preparation.

2) 1 bar = 100 kPa.

Introduce the ampoules containing the test portions (6.1), stopper the flasks and cool them in ice.

Break the ampoules by shaking and make sure that both the capillaries are broken.

Continue to shake gently for 5 min.

Rinse the stoppers and the necks of the flasks with a few millilitres of water and titrate with the standard volumetric sodium hydroxide solution (3.2), using a few drops of the bromophenol blue solution (3.3) as indicator, until a colour is obtained equivalent to that in the blank test.

7 EXPRESSION OF RESULTS

Total content of carbonyl compounds, expressed as acetaldehyde (CH_3CHO), is given, as a percentage by mass, by the formula

$$\frac{0{,}044\ (V_1 - V_2)}{m} \times 100 = \frac{4{,}4\ (V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.2) used in the determination;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.2) used in the blank test;

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

0,044 is the number of grams of acetaldehyde (CH_3CHO) equivalent to 1 ml of 1,00 M sodium hydroxide solution.

Express the result as the arithmetic mean of the duplicate determinations.

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.