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# International Standard



# 1388/5

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## **Ethanol for industrial use — Methods of test — Part 5 : Determination of aldehydes content — Visual colorimetric method**

*Éthanol à usage industriel — Méthodes d'essai — Partie 5 : Dosage des aldéhydes — Méthode colorimétrique visuelle*

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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 1388/5 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in February 1980.

It has been approved by the member bodies of the following countries :

|                |                |                       |
|----------------|----------------|-----------------------|
| Australia      | Germany, F.R.  | Romania               |
| Austria        | Hungary        | South Africa, Rep. of |
| Belgium        | India          | Switzerland           |
| Brazil         | Italy          | Thailand              |
| Bulgaria       | Korea, Rep. of | United Kingdom        |
| China          | Netherlands    | USSR                  |
| Czechoslovakia | Philippines    |                       |
| France         | Poland         |                       |

No member body expressed disapproval of the document.

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

International Standards ISO 1388/1 to ISO 1388/12 cancel and replace ISO Recommendation R 1388-1970, of which they constitute a technical revision.

# Ethanol for industrial use — Methods of test —

## Part 5 : Determination of aldehydes content — Visual colorimetric method

### 1 Scope and field of application

This part of ISO 1388 specifies a visual colorimetric method for the determination of the aldehydes content of ethanol for industrial use.

The method is applicable to products having aldehyde contents, expressed as acetaldehyde, in the range 0,000 25 to 0,001 25 % (*m/m*).

This document should be read in conjunction with ISO 1388/1 (see the annex).

### 2 Principle

Reaction of the aldehydes present in a test portion with Schiff reagent. Visual comparison of the colour obtained with the colours of standard colorimetric solutions containing known quantities of acetaldehyde.

### 3 Reagents

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

**3.1 Ethanol**, 95 % (*V/V*), aldehydes-free, purified as follows.

Boil 1 500 ml of absolute ethanol, under reflux, for 2 h, with 15 g of *m*-phenylenediamine. Distil the mixture, rejecting the first and last 50 ml fractions of the distillate. Adjust the concentration to 95 % (*V/V*) by adding an appropriate volume of water and mix.

Use the procedure specified in clause 5 to verify that the purified ethanol is free from aldehydes.

#### 3.2 Schiff reagent.

**WARNING** — Basic fuchsin is carcinogenic. Avoid skin contact with basic fuchsin and its solutions and inhalation of its dust.

##### 3.2.1 Preparation

Place 1 500 ml of water in a 3 000 ml conical flask, add  $4,500 \pm 0,005$  g of *p*-rosaniline hydrochloride (basic fuchsin), and swirl to dissolve. Add  $9,60 \pm 0,05$  g of disodium disulphite

[sodium metabisulphite ( $\text{Na}_2\text{S}_2\text{O}_5$ )], mix and allow to stand for 5 to 10 min. Add 40 ml of approximately 295 g/l sulphuric acid solution, mix thoroughly, stopper the flask and allow to stand for about 12 h. Decolorize the solution if necessary, by treatment with activated carbon.

#### 3.2.2 Determination and adjustment of free sulphur dioxide content

Transfer 10 ml of the colourless reagent (3.2.1) to a 250 ml conical flask. Add 20 ml of water and 5 ml of freshly prepared starch solution, and titrate the solution with standard volumetric iodine solution,  $c(1/2 \text{ I}_2) = 0,1$  mol/l, until the characteristic dark blue colour is just obtained.

**NOTE** — 1 ml of iodine solution,  $c(1/2 \text{ I}_2) = 0,1$  mol/l, corresponds to 0,003 2 g of  $\text{SO}_2$ .

If the free sulphur dioxide content does not fall within the optimum range (0,18 to 0,31 g per 100 ml of reagent), adjust it as appropriate, increasing the level by adding a calculated quantity of disodium disulphite or decreasing it by bubbling air through the reagent solution.

**3.3 Acetaldehyde**, standard solution corresponding to 1 g of acetaldehyde per litre.

Weigh, to the nearest 0,000 1 g, 0,693 0 g of acetaldehyde ammonia [ $\text{CH}_3\text{CH}(\text{NH}_2)\text{OH}$ ] and dissolve it in the ethanol (3.1). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark with ethanol of the same quality and mix.

1 ml of this standard solution contains 0,001 g of acetaldehyde.

**NOTE** — If acetaldehyde ammonia of analytical grade is not available, purify the commercial product as follows.

Dissolve about 5 g of acetaldehyde ammonia in a small quantity of absolute ethanol and precipitate it by adding twice the volume of dry diethyl ether ( $\text{C}_2\text{H}_5\text{OC}_2\text{H}_5$ ). Using a Buchner funnel, filter the precipitate and, after washing it with more of the diethyl ether, transfer it immediately to a vacuum desiccator containing sulphuric acid,  $\rho$  approximately 1,84 g/ml, 98 % (*m/m*) solution, as desiccant, and allow to dry for 3 to 4 h. Repeat the purification if necessary, until the product is colourless.

**WARNING** — Diethyl ether is highly flammable and its vapour is harmful. Avoid breathing vapour.

**3.4 Acetaldehyde**, standard solution corresponding to 0,1 g of acetaldehyde per litre.

Transfer 25,0 ml of the standard acetaldehyde solution (3.3) to a 250 ml one-mark volumetric flask, dilute to the mark with the ethanol (3.1) and mix.

1 ml of this standard solution contains 0,000 1 g of acetaldehyde.

## 4 Apparatus

Ordinary laboratory apparatus, and

**4.1 Colorimetric tubes**, fitted with ground glass stoppers, of capacity about 20 ml, and graduated at 10 and 14 ml.

**4.2 Graduated pipettes**, of capacity 5 ml, graduated in 0,02 ml divisions.

## 5 Procedure

### 5.1 Test portion

Using one of the graduated pipettes (4.2), measure 3,0 ml of the laboratory sample into one of the colorimetric tubes (4.1).

### 5.2 Preparation of the test solution and standard colorimetric solutions

Into a series of six 100 ml one-mark volumetric flasks, place the volumes of the standard acetaldehyde solution (3.4) indicated in the following table, dilute to the mark with the ethanol (3.1) and mix.

| Standard acetaldehyde solution (3.4) | Corresponding mass of acetaldehyde |
|--------------------------------------|------------------------------------|
| ml                                   | g                                  |
| 2,0                                  | 0,000 2                            |
| 3,0                                  | 0,000 3                            |
| 5,0                                  | 0,000 5                            |
| 7,0                                  | 0,000 7                            |
| 9,0                                  | 0,000 9                            |
| 10,0                                 | 0,001 0                            |

Using the graduated pipettes (4.2), measure, into a series of six of the colorimetric tubes (4.1), 3,0 ml of each of these diluted standard acetaldehyde solutions.

Treat the contents of each tube, including those of the tube containing the test portion (5.1), as follows.

Dilute to 10 ml with water and add sufficient of the Schiff reagent (3.2) to bring the volume to 14 ml. Stopper the tubes, mix the solutions (preferably simultaneously) and allow to stand in a rack for 25 min.

### 5.3 Determination

Compare the colour of the test solution with the colours of the standard colorimetric solutions, in dispersed daylight.

NOTE — If the colour of the test solution is deeper than that of the most concentrated standard colorimetric solution, repeat the test using more of the laboratory sample suitably diluted with the ethanol (3.1), and take this into account in the calculation of results.

## 6 Expression of results

The aldehydes content, expressed as acetaldehyde ( $\text{CH}_3\text{CHO}$ ) as a percentage by mass, is given by the formula

$$\frac{m}{\rho}$$

where

$m$  is the mass, in grams (see the table), of acetaldehyde used to prepare the diluted standard solution giving a colour matching most closely that developed in the test solution.

$\rho$  is the density, in grams per millilitre, of the sample at 20 °C (see ISO 1388/1, clause 4).

## Annex

### ISO Publications relating to ethanol for industrial use

ISO 1388/1 — General.

ISO 1388/2 — Detection of alkalinity or determination of acidity to phenolphthalein.

ISO 1388/3 — Estimation of content of carbonyl compounds present in small amounts — Photometric method.

ISO 1388/4 — Estimation of content of carbonyl compounds present in moderate amounts — Titrimetric method.

ISO 1388/5 — Determination of aldehydes content — Visual colorimetric method.

ISO 1388/6 — Test for miscibility with water.

ISO 1388/7 — Determination of methanol content [methanol contents between 0,01 and 0,20 % (V/V)] — Photometric method.

ISO 1388/8 — Determination of methanol content [methanol contents between 0,10 and 1,50 % (V/V)] — Visual colorimetric method.

ISO 1388/9 — Determination of esters content — Titrimetric method after saponification.

ISO 1388/10 — Estimation of hydrocarbons content — Distillation method.

ISO 1388/11 — Test for detection of furfural.

ISO 1388/12 — Determination of permanganate time.

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