International Standard 1388/8

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Ethanol for industrial use — Methods of test — Part 8 : Determination of methanol content [methanol contents between 0,10 and 1,50 % (*V*/*V*)] — Visual colorimetric method

Éthanol à usage industriel — Méthodes d'essai — Partie 8 : Dosage du méthanol [teneurs de 0,10 *à* 1,50 % *(V/V)] — Méthode colorimétrique visuelle*

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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 1388/8 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.

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

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Ethanol for industrial use — Methods of test — Part 8 : Determination of methanol content [methanol contents between 0,10 and 1,50 % (*V*/*V*)] — Visual colorimetric method

1 Scope and field of application

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

The method is applicable to products having methanol contents between 0,10 and 1,50 % (*V*/*V*).

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

2 Principle

Conversion of the methanol present in a test portion to formaldehyde by oxidation with a solution of potassium permanganate in phosphoric acid. Reaction of the formaldehyde formed with Schiff reagent. Visual comparison of the colour obtained with the colours of standard colorimetric solutions containing known quantities of formaldehyde.

3 Reagents

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

3.1 Potassium permanganate, 30 g/l solution in phosphoric acid.

Dissolve 3 g of potassium permanganate in a little water, add 15,5 ml of orthophosphoric acid solution, ϱ 1,69 g/ml, dilute to 100 ml with water and mix.

3.2 Oxalic acid, 50 g/l solution in sulphuric acid.

WARNING — Harmful in contact with skin and if swallowed. Avoid contact with skin and eyes.

Dissolve 5 g of oxalic acid in 100 ml of 50 % (*V*/*V*) sulphuric acid solution, prepared by diluting sulphuric acid, ϱ approximately 1,84 g/ml, about 98 % (*m*/*m*) solution, 1 + 1 (*V*/*V*) with water.

3.3 Schiff reagent.

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

3.3.1 Preparation

Place 1 500 ml of water in a 3 000 ml conical flask, add 4,500 ± 0,005 g of *p*-rosaniline hydrochloride (basic fuchsin) and swirl to dissolve. Add 9,6 ± 0,05 g of *di*sodium disulphite [sodium metabisulphite ($Na_2S_2O_5$)], 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.

3.3.2 Determination and adjustment of free sulphur dioxide content

Transfer 10 ml of the colourless reagent (3.3.1) 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, $c(1/2\ I_2)$ = 0,1 mol/l, until the characteristic dark blue colour is just obtained.

NOTE - 1 ml of iodine solution, $c(1/2\ I_2)$ = 0,1 mol/l, corresponds to 0,003 2 g of 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 as appropriate, increasing the level by adding a calculated quantity of *di*sodium disulphite or decreasing it by bubbling air through the reagent solution.

3.4 Methanol, standard solution corresponding to 0,2 % (*V*/*V*) of methanol.

Place 2,00 ml of absolute methanol in a 1 000 ml one-mark volumetric flask, add a quantity of methanol-free ethanol corresponding to 98 ml of anhydrous ethanol, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,002 ml of absolute methanol.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Colorimetric tubes, of capacity approximately 20 ml, fitted with ground glass stoppers.

4.2 Water bath, capable of being controlled at 20 ± 1 °C.

5 Procedure

5.1 Test portion and preparation of the test solution

Take as the test portion a volume (V_1) of the laboratory sample, corresponding to 10,0 ml of anhydrous ethanol, and place it in a 100 ml one-mark volumetric flask. Prepare the test portion by diluting to the mark with water and mixing. Transfer 5,0 ml of this solution to one of the colorimetric tubes (4.1).

5.2 Preparation of standard solutions, used for the preparation of standard colorimetric solutions

Into a series of five 100 ml one-mark volumetric flasks, place the volumes of the standard methanol solution (3.4) indicated in the following table, dilute to the mark with 10 % (V/V) solution of methanol-free ethanol in water and mix.

Standard methanol solution (3.4)	Corresponding volume of methanol
ml	ml
5,00	0,010
10,0	0,020
25,0	0,050
50,0	0,100
75,0	0,150

5.3 Preparation of standard colorimetric solutions

Into a series of five of the colorimetric tubes (4.1), place 5,0 ml of each of the standard methanol solutions (5.2).

Treat the contents of each of the tubes, including the tube containing 5,0 ml of the test solution (5.1), as follows.

Add 2,0 ml of the potassium permanganate solution (3.1), mix and allow to stand for 10 min in the water bath (4.2), controlled at 20 ± 1 °C. Then add 2,0 ml of the oxalic acid solution (3.2) and mix. At this stage, the solutions should be colourless and free from precipitated manganese. Add 5 ml of the Schiff reagent (3.3), mix and allow to stand for 1 h.

5.4 Determination

Examine the tubes vertically and note the standard colorimetric solution having a colour matching most closely that developed in the test solution.

NOTE — In cases of doubt, choose the standard colorimetric solution having the lower concentration.

6 Expression of results

The methanol content, expressed as methanol as a percentage by volume, is given by the formula

$$\frac{V_0 \times 100}{V_1}$$

where

V_0 is the volume, in millilitres, of methanol in the dilute standard solution (see 5.2) from which was prepared the standard colorimetric solution having a colour matching most closely that developed in the test solution;

V_1 is the volume, in millilitres, of the test portion.

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.