International Standard 1388/7

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Ethanol for industrial use — Methods of test — Part 7 : Determination of methanol content [methanol contents between 0,01 and 0,20 % (*V*/*V*)] — Photometric method

Éthanol à usage industriel — Méthodes d'essai — Partie 7 : Dosage du méthanol [teneurs de 0,01 *à* 0,20 % *(V/V)] — Méthode photométrique*

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Foreword

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

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

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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

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1 Scope and field of application

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

The method is applicable to products having methanol contents between 0,01 and 0,20 % (*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 chromotropic acid.

Photometric measurement of the violet coloration obtained at a wavelength of about 570 nm.

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 *Di*sodium disulphite [sodium metabisulphite ($Na_2S_2O_5$)], 100 g/l solution.

Dissolve 10 g of sodium metabisulphite in water and dilute to 100 ml.

3.3 4,5-Dihydroxynaphthalene-2,7-disulphonic acid (chromotropic acid), solution in sulphuric acid.

3.3.1 Preparation of the solution

Dissolve 0,1 g of chromotropic acid, or its *di*sodium salt, in 10 ml of water. Add, while cooling, 90 ml of sulphuric acid, ϱ approximately 1,81 g/ml, about 90 % (*m*/*m*) solution, and mix.

Prepare this solution at the time of use.

If the solution causes significant coloration during colour development of the compensation solution (5.3.1) or the blank test solution (5.2), purify the chromotropic acid, or its *di*sodium salt, in accordance with the procedure specified in 3.3.2.

3.3.2 Purification of the chromotropic acid

Dissolve about 10 g of chromotropic acid, or its *di*sodium salt, in 25 ml of water. If the *di*sodium salt is used, add, while cooling, 2 ml of sulphuric acid, ϱ approximately 1,84 g/ml, to convert it to the free acid. Add 50 ml of methanol, heat just to boiling and filter through a sintered glass filter funnel, porosity grade P 10*.

Add 100 ml of propan-2-ol to the solution to precipitate the chromotropic acid. Collect the precipitate on a sintered glass filter funnel, porosity grade P 10, and wash it with small portions of propan-2-ol. Allow to dry, initially in air, and finally in a desiccator over sulphuric acid, ϱ approximately 1,84 g/ml, about 98 % (*m*/*m*) solution, as desiccant.

If, after purification, the blank test solution is still coloured, reject the chromotropic acid.

3.4 Methanol, standard solution corresponding to 0,05 % (*V*/*V*) of methanol.

Place 1,00 ml of absolute methanol in a 250 ml one-mark volumetric flask, add a quantity of methanol-free ethanol corresponding to 99 ml of anhydrous ethanol, dilute to the mark with water and mix.

* See ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*