International Standard 1388/3

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Ethanol for industrial use — Methods of test — Part 3 : Estimation of content of carbonyl compounds present in small amounts — Photometric method

Éthanol à usage industriel — Méthodes d'essai — Partie 3 : Évaluation de la teneur en composés carbonylés présents en faible quantité — Méthode photométrique

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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.

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

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

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 3 : Estimation of content of carbonyl compounds present in small amounts — Photometric method

1 Scope and field of application

This part of ISO 1388 specifies a photometric method for estimation of the content of carbonyl compounds present in small amounts in ethanol for industrial use.

The method is applicable to products having carbonyl compounds contents, expressed as acetaldehyde, between 0,000 25 and 0,01 % (*m*/*m*).

NOTE — This method, which is used commercially, allows determination of only those carbonyl compounds which react under the specified conditions.

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

2 Principle

Reaction in acid medium of the carbonyl compounds in a test portion with 2,4-dinitrophenylhydrazine. Formation of the corresponding 2,4-dinitrophenylhydrazones, which, after making the solution alkaline, take on a red coloration.

Photometric measurement of this red coloration at a wavelength of about 445 nm.

3 Reagents

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

3.1 Ethanol, free from carbonyl compounds, purified as follows.

Boil under reflux 500 ml of ethanol with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of the hydrochloric acid solution (3.3), for 2 to 3 h. Distil the ethanol slowly using a Widmer distillation column, about 300 mm long and about 25 mm in diameter, or any other suitable column. Reject the first 50 ml of distillate and collect the next 400 ml, rejecting the remainder. If the distillate is coloured, redistil it.

3.2 2,4-Dinitrophenylhydrazine, saturated solution in the ethanol (3.1) at ambient temperature.

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

3.4 Potassium hydroxide, 100 g/l solution in a 70 % (*V*/*V*) solution of the ethanol (3.1).

3.5 Carbonyl compounds, standard solution corresponding to 0,440 g of carbonyl compounds, expressed as acetaldehyde, per litre.

Weigh, to the nearest 0,000 1 g, 1,200 g of acetophenone, and dissolve it in a little of the ethanol (3.1). Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark with ethanol of the same quality and mix. Take 10,0 ml of this solution, transfer it to a 100 ml one-mark volumetric flask, dilute to the mark with the ethanol (3.1) and mix.

1 ml of this standard solution contains 440 µg of carbonyl compounds, expressed as acetaldehyde.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Water bath, capable of being controlled at 50 ± 2 °C.

4.2 Test tubes, fitted with ground glass stoppers.

4.3 Spectrophotometer, or

4.4 Photoelectric absorptiometer, fitted with filters giving maximum transmission at a wavelength of about 445 nm.

5 Procedure

5.1 Test portion

Take 1,0 ml of the laboratory sample and place it in one of the test tubes (4.2).

5.2 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 the reagents used for the determination, but replacing the test portion by 1,0 ml of the ethanol (3.1).

5.3 Preparation of the calibration graph

5.3.1 Preparation of dilute standard solutions, with a view to preparation of standard colorimetric solutions

Into a series of seven 25 ml one-mark volumetric flasks, introduce the volumes of the standard carbonyl compounds solution (3.5) indicated in the following table and dilute to the mark with the ethanol (3.1).

Standard carbonyl compounds solution (3.5)	Corresponding mass of carbonyl compounds, expressed as CH_3CHO	Mass of carbonyl compounds in 1 ml of dilute standard solution
ml	µg	µg
0*	0	0
0,15	66,0	2,6
0,25	110,0	4,4
0,50	220,0	8,8
0,75	330,0	13,2
1,00	440,0	17,6
1,25	550,0	22,0

* Compensation solution.

5.3.2 Preparation of standard colorimetric solutions, for photometric measurements carried out in cells of optical path length 1 cm

Into a series of seven of the test tubes (4.2), place 1,0 ml of each of the dilute standard solutions (5.3.1).

5.3.3 Colour development

Add 1,0 ml of the 2,4-dinitrophenylhydrazine solution (3.2) and one drop of the hydrochloric acid solution (3.3). Stopper the tubes and heat for 30 min on the water bath (4.1), controlled at 50 ± 2 °C. Allow to cool, add 5,0 ml of the potassium hydroxide solution (3.4), mix, and allow to stand for 5 min.

5.3.4 Photometric measurements

Immediately carry out the photometric measurements on each of the standard colorimetric solutions, using either the spectrophotometer (4.3), set at a wavelength of about 445 nm, or the photoelectric absorptiometer (4.4) fitted with appropriate filters, after having first adjusted the instrument to zero absorbance against the ethanol (3.1).

5.3.5 Plotting the graph

Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions (5.3.2). Plot a graph having, for example, the masses, in micrograms, of carbonyl compounds in 1 ml of each dilute standard solution (5.3.1) as abscissae, and the corresponding corrected values of absorbance as ordinates.

5.4 Determination

5.4.1 Colour development

Treat the test portion (5.1) in the test tube, following the procedure specified in 5.3.3.

5.4.2 Photometric measurements

Immediately carry out the photometric measurements on the test solution and the blank test solution following the procedures specified in 5.3.4, after having adjusted the instrument to zero absorbance against the ethanol (3.1).

NOTE — If the absorbance exceeds the maximum of the calibration graph, repeat the determination (5.4) using as the test portion 1,0 ml of a test solution prepared by diluting 1,0 ml of the laboratory sample with an appropriate volume (not more than 4,0 ml) of the ethanol (3.1).

6 Expression of results

By means of the calibration graph (5.3.5), determine the masses of carbonyl compounds corresponding to the values of the photometric measurements.

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

$$\frac{(m_1 - m_0) \times 100}{1,0 \times \varrho \times 10^6} \times r_D$$

$$= \frac{m_1 - m_0}{\varrho \times 10^4} \times r_D$$

where

m_0 is the mass, in micrograms, of carbonyl compounds found in the blank test solution;

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

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see ISO 1388/1, clause 4);

r_D is the ratio of the volume of the diluted test solution (see the note to 5.4.2) to the volume of the aliquot portion taken for the determination (if the test portion was not diluted, r_D is equal to 1);

1,0 is the volume, in millilitres, of the test portion (5.1).

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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