

International Standard**1388/4**

W-25-04

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**Ethanol for industrial use — Methods of test —
Part 4 : Estimation of content of carbonyl compounds
present in moderate amounts — Titrimetric method***Éthanol à usage industriel — Méthodes d'essai — Partie 4 : Évaluation de la teneur en composés carbonylés présents en quantité modérée — Méthode titrimétrique***First edition — 1981-12-01****UDC 661.722 : 543.854.6****Ref. No. ISO 1388/4-1981 (E)****Descriptors :** industrial products, ethanols, tests, determination, carbonyl compounds, titration.

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 1388/4 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.

© International Organization for Standardization, 1981 ●

Printed in Switzerland

Ethanol for industrial use — Methods of test — Part 4 : Estimation of content of carbonyl compounds present in moderate amounts — Titrimetric method

1 Scope and field of application

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

The method is applicable to products having carbonyl compounds contents, expressed as acetaldehyde, equal to or greater than 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 of hydroxylammonium chloride with the carbonyl compounds in a test portion and titration of the hydrochloric acid formed with standard volumetric sodium hydroxide solution, in the presence of bromophenol blue as indicator.

3 Reagents

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

3.1 Hydroxylammonium chloride reagent.

WARNING — Corrosive and irritating. Avoid contact with eyes and skin.

Dissolve 4 g of hydroxylammonium chloride in 20 ml of water and dilute to 200 ml with 95 % (*V/V*) ethanol. Heat under reflux for 30 min on a boiling water bath; cool to ambient temperature, add 5 ml of the bromophenol blue solution (3.4) and just sufficient of the sodium hydroxide solution (3.2) to produce a dichroic green coloration.

3.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

3.3 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,1 \text{ mol/l}$.

3.4 Bromophenol blue, 2 g/l ethanolic solution.

Dissolve 0,2 g of bromophenol blue in 1,5 ml of the sodium hydroxide solution (3.2) and dilute to 100 ml with 95 % (*V/V*) ethanol.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Conical flasks, of capacity 150 ml, fitted with ground glass stoppers.

5 Procedure

5.1 Test portion

Take $50 \pm 0,1$ ml of the laboratory sample and place it in one of the conical flasks (4.1).

5.2 Determination

Place 50 ml of the hydroxylammonium chloride reagent (3.1) in a second conical flask (4.1), to be used as the colour standard.

Add 1,25 ml of the bromophenol blue solution (3.4) to the flask containing the test portion (5.1) and add, drop by drop, either the sodium hydroxide solution (3.2) or the hydrochloric acid solution (3.3) until the colour matches that of the colour standard. Then add, to each of the flasks, 25 ml of the hydroxylammonium chloride reagent (3.1) and stopper the flask containing the colour standard.

Loosely stopper the flask containing the test solution and heat it for 10 min on a boiling water bath. Remove the flask from the water bath, cool to ambient temperature and titrate the solution with the sodium hydroxide solution (3.2) until the colour matches as closely as possible that of the colour standard.

6 Expression of results

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

$$\frac{0,004\,405 \times V \times 100}{50 \times \rho}$$
$$= \frac{0,008\,81 V}{\rho}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (3.2) used for the determination;

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

0,004 405 is the mass, in grams, of carbonyl compounds, expressed as acetaldehyde, corresponding to 1 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0,100 \text{ mol/l}$;

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

NOTE — If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, an appropriate correction should be made.

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.