International Standard 1388/9

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

Ethanol for industrial use — Methods of test — Part 9 : Determination of esters content — Titrimetric method after saponification

Éthanol à usage industriel — Méthodes d'essai — Partie 9 : Dosage des esters — Méthode titrimétrique après saponification

First edition — 1981-12-01

UDC 661.722 : 543.851.1 **Ref. No. ISO 1388/9-1981 (E)**

Descriptors : industrial products, ethanols, tests, determination of content, esters, titration, saponification.

ISO 1388/9-1981 (E)

Price based on 3 pages

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 1388/9 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.

Printed in Switzerland

1388 PT 9-81 4851903 0002610 6

Ethanol for industrial use — Methods of test — Part 9 : Determination of esters content — Titrimetric method after saponification

1 Scope and field of application

This part of ISO 1388 specifies a titrimetric method, after saponification, for the determination of the esters content of ethanol for industrial use.

The method is applicable to products having esters contents, expressed as ethyl acetate, in the range 0,005 to 0,15 % (m/m).

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

2 Principle

Saponification of the esters present in a test portion by boiling with excess standard volumetric sodium hydroxide solution; determination of the residual sodium hydroxide, and hence the amount used for saponification, by titration with standard volumetric hydrochloric acid solution in the presence of phenolphthalein as indicator.

3 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade, and distilled water or water of equivalent purity, carbon dioxide-free, recently prepared.

3.1 Water, carbon dioxide-free.

Boil distilled water and allow it to cool in a flask fitted with a stopper carrying a soda-lime guard-tube.

3.2 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,1 mol/l.

3.3 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,1 mol/l.

3.4 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (V/V) ethanol and add the sodium hydroxide solution (3.2) until a pale pink coloration is obtained.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Conical flask, of borosilicate glass, of capacity 500 ml, and having a ground neck.

4.2 Reflux condenser, water-cooled having a ground glass joint to fit the flask (4.1).

NOTE — Clean the apparatus as follows.

Place a mixture of ethanol and sodium hydroxide solution (the proportions are not critical) in the flask (4.1), attach the reflux condenser (4.2) and heat the mixture gently under reflux for several minutes.

Discard the mixture and clean the flask and the condenser, first by washing with tap water and then by rinsing several times with distilled water.

5 Procedure

5.1 Test portion

Take 50,0 ml of the laboratory sample. If the esters content, expressed as ethyl acetate, is expected to be less than or equal to 0,01 % (m/m), take 100 ± 0,1 ml.

5.2 Determination

Place the test portion (5.1) in the flask (4.1), and add 20 ml of water and 0,5 ml of the phenolphthalein solution (3.4). Neutralize the solution [usually with the sodium hydroxide solution (3.2) until a pink coloration, persisting for about 15 s, appears].

Add 10,0 ml of the sodium hydroxide solution (3.2). Fit the condenser (4.2) and heat for 1 h on a boiling water bath. Fit a soda-lime guard-tube to the top of the condenser and cool the flask in water. Remove the soda-lime guard-tube and wash the inside of the condenser with two 10 ml portions of water, collecting the washings in the flask. Disconnect the flask and wash the ground neck with 10 ml of water, again collecting the washings in the flask.

Carefully titrate the solution with the standard volumetric hydrochloric acid solution (3.3) until the pink colour just disappears.

5.3 Blank test

Carry out a blank test on the neutral solution from the determination (5.2), using the procedure specified in 5.2, paragraphs 2 and 3.

6 Expression of results

The esters content, expressed as a percentage by mass of ethyl acetate ($CH_3COOC_2H_5$), is given by the formula

$$\frac{0{,}008\,8 \times (V_2 - V_1) \times 100}{V_0\,\varrho}$$

$$= \frac{0{,}88 \times (V_2 - V_1)}{V_0\,\varrho}$$

where

V_0 is the volume, in millilitres, of the test portion (5.1);

V_1 is the volume, in millilitres, of the hydrochloric acid solution (3.3) used for the determination;

V_2 is the volume, in millilitres, of the hydrochloric acid solution (3.3) used for the blank test;

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

0,008 8 is the mass, in grams, of esters, expressed as ethyl acetate, corresponding to 1 ml of sodium hydroxide solution, c(NaOH) = 0,100 mol/l.

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.