INTERNATIONAL STANDARD 1843 / V

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Higher alcohols for industrial use — Methods of test — Part V : Determination of total alcohols content — Titrimetric method

Alcools supérieurs à usage industriel — Méthodes d'essai —
Partie V : Détermination de la teneur totale en alcools — Méthode titrimétrique

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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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry,* has reviewed ISO Recommendation R 1850-1970 and found it technically suitable for transformation. Number 1850, however, has been changed to 1843/V. International Standard ISO 1843/V therefore replaces ISO Recommendation R 1850-1970, to which it is technically identical.

ISO Recommendation R 1850 had been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Switzerland
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Peru	U.S.S.R.
Germany	Poland	
Greece	Portugal	

No member body had expressed disapproval of the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

Higher alcohols for industrial use — Methods of test — Part V : Determination of total alcohols content — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1843 specifies a titrimetric method for the determination of the total alcohols content of C_6 to C_{13} alcohols for industrial use.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Esterification of alcohols present in a test portion by means of acetic anhydride, and titration of the excess acetic anhydride with a standard volumetric ethanolic potassium hydroxide solution, in the presence of phenolphthalein as indicator.

3 REAGENTS

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

3.1 Methanol, 95 % (*V/V*).

3.2 Acetic anhydride/pyridine mixture.

WARNING — Because of the toxicity and unpleasant odour of pyridine, it is recommended that it should be handled with care and in a well-ventilated fume cupboard.

Mix 60 g of acetic anhydride [$(CH_3CO)_2O$] and 440 g of pyridine (C_5H_5N) and store the mixture in an airtight container of dark-coloured glass.

3.3 Potassium hydroxide, 0,2 N standard volumetric ethanolic solution.

Wash some solid potassium hydroxide rapidly with the methanol (3.1) to remove any potassium carbonate adhering to the surface and prepare an 11,2 g/l solution in 95 % (*V/V*) ethanol. Standardize this solution against 0,1 N standard volumetric sulphuric acid solution, using the phenolphthalein solution (3.4) as indicator.

3.4 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol, and make faintly pink by the addition of dilute sodium hydroxide solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Flat-bottomed flasks, of capacity 100 ml, with ground glass stoppers.

4.2 Glass tubes, at least 1,5 m long, fitted with ground glass joints for connecting to the flasks (4.1).

4.3 Microburette, of capacity 20 ml, graduated in 0,02 ml divisions.

5 PROCEDURE

5.1 Preparation of apparatus

Wash the apparatus after each use, by the method specified below; wear rubber gloves during the washing operations.

5.1.1 *Flasks*

Wipe the joints with cellulose paper; rinse with water; wash with petroleum spirit in a bath; rinse with hot water, then with the methanol (3.1) and dry in a heated cabinet.

5.1.2 *Glass tubes*

Wipe the joints with cellulose paper; rinse with the methanol (3.1), allow to drain and dry in a current of air dried over silica gel.

5.2 Test portion

Take a quantity of the laboratory sample, weighed to the nearest 0,000 1 g, as shown in the table.

Alcohol	Alkyl radical	Equivalents of alcohol per kilogram (theoretical)	Mass of test portion
	R	x	g
Hexyl alcohols	C_6H_{13}	9,8	0,200 to 0,300
Heptyl alcohols	C_7H_{15}	8,61	0,200 to 0,300
Octyl alcohols	C_8H_{17}	7,69	0,250 to 0,350
C_7-C_9 mixed alcohols		7,7	0,250 to 0,350
Nonyl alcohols	C_9H_{19}	6,94	0,250 to 0,350
Decyl alcohols	$C_{10}H_{21}$	6,32	0,400
Undecyl alcohols	$C_{11}H_{23}$	5,8	0,400 to 0,450
Dodecyl alcohols	$C_{12}H_{25}$	5,4	0,450
Tridecyl alcohols	$C_{13}H_{27}$	5	0,450 to 0,550

5.3 Blank test

Carry out a blank test at the same time as the determination, following the same procedure but omitting the test portion.

5.4 Determination

Place in one of the flasks (4.1) 4 ml of the acetic anhydride/pyridine mixture (3.2), measured form the microburette (4.3).

Add the test portion (5.2) to the flask. Fit one of the glass tubes (4.2) to the flask, using silicone grease to lubricate the joints. Transfer the flask to a boiling water bath and allow it to remain there for 2 h. At the end of this time, add 2 ml of water. Shake, allow to stand for 10 min, then cool the flask in running water. Rinse the glass tube with the methanol (3.1) and titrate with the potassium hydroxide solution (3.3), using a few drops of the phenolphthalein solution (3.4) as indicator.

6 EXPRESSION OF RESULTS

The total alcohols content, expressed as a percentage by mass, is given by the formula

$$\frac{0{,}2\,(V_0 - V_1)}{m \times x} \times 100$$

where

V_0 is the volume, in millilitres, of the potassium hydroxide solution (3.3) used in titrating the excess acetic anhydride in the blank test (5.3);

V_1 is the volume, in millilitres, of the potassium hydroxide solution (3.3) used in titrating the excess acetic anhydride in the determination (5.4);

m is the mass, in grams, of the test portion (5.2);

x is the theoretical number of equivalents of alcohol per kilogram (see the table in 5.2).

For mixed alcohols of unknown composition, the total alcohols content, expressed in equivalents per kilogram, is given by the formula

$$\frac{0{,}2\,(V_0 - V_1)}{m}$$

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

ANNEX

ISO PUBLICATIONS RELATING TO HIGHER ALCOHOLS FOR INDUSTRIAL USE

ISO 1843/I — General.

ISO 1843/II — Determination of acidity to phenolphthalein — Titrimetric method.

ISO 1843/III — Determination of carbonyl compounds content — Potentiometric method.

ISO 1843/IV — Determination of bromine number — Titrimetric method in the presence of mercury(II) chloride.

ISO 1843/V — Determination of total alcohols content.

ISO 1843/VI — Determination of ash.

ISO/R 1845 — Determination of distillation yield.

ISO/R 1852 — Test for colour with sulphuric acid.