UDC 661.725.6 : 547.26 : 543.24

Ref. No. : ISO/R 1850-1970 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1850

HIGHER ALCOHOLS FOR INDUSTRIAL USE

DETERMINATION OF TOTAL ALCOHOLS CONTENT

(VOLUMETRIC METHOD)

1st EDITION

November 1970

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1850, *Higher alcohols for industrial use – Determination of total alcohols content (Volumetric method),* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1850, which was circulated to all the ISO Member Bodies for enquiry in April 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member bodies :

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

The following Member Body opposed the approval of the Draft :

New Zealand

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1850 | November 1970

HIGHER ALCOHOLS FOR INDUSTRIAL USE

DETERMINATION OF TOTAL ALCOHOLS CONTENT

(VOLUMETRIC METHOD)

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of total alcohols content of C_6 to C_{13} alcohols for industrial use.

2. PRINCIPLE

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

3. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

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

3.2 *Acetic anhydride/pyridine mixture.*

Mix together 60 g of acetic anhydride and 440 g of pyridine and store the mixture in an airtight container of dark coloured glass.

3.3 *Potassium hydroxide,* 0.2 N standard volumetric solution in ethanol.

Wash some solid potassium hydroxide rapidly with 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 using a 0.1 N standard volumetric solution of sulphuric acid with phenolphthalein solution (3.4) as indicator.

3.4 *Phenolphthalein,* 5 g/l 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,* 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,* capacity 20 ml, graduated in 0.02 ml divisions.

NOTE. – *Washing the apparatus.* The apparatus should be washed after each use, by the method described below; rubber gloves should be worn during the washing operations :

1. *Flasks.* Wipe the joints with cellulose paper; rinse with water; wash with petroleum spirit in a bath; rinse with hot water, then with methanol (3.1) and dry in a heated cabinet.
2. *Glass tubes.* Wipe the joints with cellulose paper; rinse with methanol (3.1), allow to drain and dry in a current of air dried over silica gel.

5. SAMPLING

Follow the principles given in ISO Recommendation R . . .*.

Place the sample in a clean, dry, glass-stoppered bottle of such a size that it is nearly filled up. If it is necessary to seal this bottle, care should be taken to avoid the risk of contamination.

6. PROCEDURE

6.1 Test portion

Take a quantity of the laboratory sample, weighed to the nearest 0.1 mg, as shown in the table below :

Sample	R	x Equivalents of alcohol/kg (theoretical)	Mass of test portion 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_{7-9} 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

6.2 Blank test

At the same time as the determination, carry out a blank test using the procedure described in clause 6.3 and the same quantities of all reagents as used in the determination.

6.3 Determination

Place in a flask (4.1), 4 ml of the acetic anhydride/pyridine mixture (3.2), measured from the micro-burette (4.3).

Add to the flask the test portion (6.1). Fit a glass tube (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 hours. At the end of this time add 2 ml of water. Shake, allow to stand for 10 minutes, then cool the flask in running water. Rinse the glass tube (4.2) with methanol (3.1) and titrate with the ethanolic potassium hydroxide solution (3.3), using a few drops of the phenolphthalein solution (3.4) as indicator.

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* Sampling from the consignment of a chemical product will be the subject of a future ISO Recommendation.

7. EXPRESSION OF RESULTS

Total alcohols content, expressed as a percentage by mass, is given by the following formula :

$$\frac{0.2\,(V_1 - V_2)}{m \times x} \times 100$$

where

V_1 is the volume, in millilitres, of the 0.2 N potassium hydroxide solution (3.3) used in titrating the excess acetic acid in the blank test;

V_2 is the volume, in millilitres, of the 0.2 N potassium hydroxide solution (3.3) used in titrating the excess acetic acid in the determination;

m is the mass, in grammes, of the test portion;

x is the theoretical equivalents of alcohol/kg (see table under clause 6.1).

For mixed alcohols of unknown composition, total alcohols content may be represented in equivalents per kilogramme by the following formula :

$$\frac{0.2\,(V_1 - V_2)}{m}$$

8. TEST REPORT

The test report should give the following particulars :

(*a*) the reference of the method used;

(*b*) the results and the method of expression used;

(*c*) any unusual features noted during the determination;

(*d*) any operation not included in this ISO Recommendation or regarded as optional.

ANNEX

This document forms one of a series of ISO Recommendations on methods of test for C_6 to C_{13} alcohols; the complete list of ISO Recommendations under the general title, *Higher alcohols for industrial use,* is as follows :

ISO/R 1843 *Measurement of colour in Hazen units.*

ISO/R 1844 *Determination of density at 20 °C.*

ISO/R 1845 *Determination of distillation yield.*

ISO/R 1846 *Determination of acidity to phenolphthalein.*

ISO/R 1847 *Determination of carbonyl compounds (Hydroxylammonium chloride potentiometric method).*

ISO/R 1848 *Determination of bromine index in the presence of mercury (II) chloride.*

ISO/R 1849 *Determination of water content by the Karl Fischer method.*

ISO/R 1850 *Determination of total alcohols content (Volumetric method).*

ISO/R 1851 *Determination of ash (Gravimetric method).*

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

NOTE. – A sample of the material not less than 750 ml is necessary to carry out the whole series of tests described in these documents.

Date of the first printing :
December 1970

Price based on 4 pages