

INTERNATIONAL STANDARD 1843 / III

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

Higher alcohols for industrial use — Methods of test — Part III : Determination of carbonyl compounds content — Potentiometric method

*Alcools supérieurs à usage industriel — Méthodes d'essai —
Partie III : Dosage des composés carbonylés — Méthode potentiométrique*

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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 1847-1970 and found it technically suitable for transformation. Number 1847, however, has been changed to 1843/III. International Standard ISO 1843/III therefore replaces ISO Recommendation R 1847-1970, to which it is technically identical.

ISO Recommendation R 1847 had been approved by the member bodies of the following countries :

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

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

Higher alcohols for industrial use – Methods of test – Part III : Determination of carbonyl compounds content – Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1843 specifies a potentiometric method for the determination of the carbonyl compounds content of C₆ to C₁₃ alcohols for industrial use.

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

2 PRINCIPLE

Reaction of carbonyl compounds present in a test portion with hydroxylammonium chloride to form an oxime, and potentiometric titration of the hydrochloric acid liberated with standard volumetric ethanolic potassium hydroxide solution.

3 REAGENTS

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

3.1 Ethanol, anhydrous.

3.2 Hydroxylammonium chloride, 10 g/l ethanolic solution.

Dissolve 50 g of hydroxylammonium chloride (NH₂OH.HCl) in 90 ml of water and dilute to 1 000 ml with 95 % (V/V) ethanol. Further dilute 100 ml of this solution to 500 ml with 95 % (V/V) ethanol.

3.3 Potassium hydroxide, 0,1 N standard volumetric solution in 95 % (V/V) ethanol, or

3.4 Potassium hydroxide, 0,01 N standard volumetric solution in 95 % (V/V) ethanol.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Two conical flasks, of borosilicate glass, of capacity 250 ml, fitted with ground glass stoppers.

4.2 Two water-cooled reflux condensers, with ground glass joints to fit the flasks (4.1).

4.3 pH meter, fitted with a glass measuring electrode and a calomel reference electrode.

5 PROCEDURE

5.1 Test portion

Into one of the conical flasks (4.1) already containing 10 ml of the hydroxylammonium chloride solution (3.2), weigh, to the nearest 0,001 g, 25 to 30 g of the laboratory sample.

5.2 Blank test

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

5.3 Determination

Add 10 ml of the ethanol (3.1) to the conical flask (4.1) containing the test portion (5.1). Attach one of the condensers (4.2) to the flask and reflux for 30 min on a boiling water bath. Remove the flask, still carrying its condenser, from the boiling water bath and allow to cool to ambient temperature. When cold, wash down the inside of the condenser with 10 ml of the ethanol. Transfer the contents of the flask quantitatively to a 250 ml beaker, washing with 125 ml of the ethanol.

Titrate with the potassium hydroxide solution (3.3) or, if greater precision is required, with the potassium hydroxide solution (3.4), using the pH meter (4.3). The volume/potential graph may be plotted directly, in which case the point of inflection corresponds to the end-point of the titration (pH value about 3). Alternatively, the first derived curve may be plotted, in which case the end-point of the titration corresponds to the turning point of the curve.

6 EXPRESSION OF RESULTS

The carbonyl compounds content, expressed in milliequivalents of carbonyl (CO) per gram, is given by the formula

$$\frac{V_1 - V_0}{10 \times m}$$

where

V_0 is the volume in millilitres, of the standard volumetric potassium hydroxide solution (3.3) used in the blank test (5.2);

V_1 is the volume, in millilitres, of the standard volumetric potassium hydroxide solution (3.3) used for the determination (5.3);

m is the mass, in grams, of the test portion (5.1).

If the standard volumetric potassium hydroxide solution (3.4) is used instead of the solution (3.3), the formula for calculation becomes

$$\frac{V_1 - V_0}{100 \times m}$$

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.