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Surface active agents — Soaps — Determination of low contents of free glycerol by molecular absorption spectrometry

*Agents de surface — Savons — Dosage du glycérol libre en faibles teneurs par
spectrométrie d'absorption moléculaire*

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Reference number
ISO 2272 : 1989 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2272 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 2272: 1972), of which it constitutes a minor revision.

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Surface active agents — Soaps — Determination of low contents of free glycerol by molecular absorption spectrometry

1 Scope

This International Standard specifies a spectrometric method for the determination of low contents of free glycerol in soaps.

The method is applicable to soaps having a free-glycerol content of less than 0,5 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 6353-3 : 1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

ISO 8212 : 1986, *Soaps and detergents — Techniques of sampling during manufacture*.

3 Principle

Decomposition of the soap by sulfuric acid and extraction of the fatty acids with light petroleum. Oxidation of the free glycerol remaining in the aqueous phase by periodic acid to formic acid and formaldehyde.

On reaction with chromotropic acid, the aldehyde formed gives an absorbing compound whose absorbance is proportional to the free glycerol content. Spectrometric measurement of the absorbance at a wavelength of about 571 nm.

4 Reagents

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

4.1 Light petroleum, boiling range between 40 °C and 60 °C.

4.2 Sulfuric acid, 225 g/l solution, i.e. 20 % (*m/m*) (ρ_{20} 1,14 g/ml).

4.3 Sulfuric acid, 980 g/l solution, i.e. 64 % (*m/m*) (ρ_{20} 1,54 g/ml).

4.4 Sodium periodate, approximately 0,03 mol/l solution.

Weigh 1,6 g of sodium periodate (NaIO_4) (minimum purity 99,8 %) into a 250 ml one-mark volumetric flask and dissolve in about 100 ml of 25 g/l sulfuric acid solution. Dilute to the mark with 25 g/l sulfuric acid solution.

4.5 Chromotropic acid, solution.

Weigh either 0,25 g of di-sodium-1,8-dihydroxynaphthalene-3,6-disulfonate dihydrate or the corresponding mass, 0,23 g, of the anhydrous salt (minimum purity 99 % in each case) into a 250 ml one-mark volumetric flask and dissolve in 10 ml of water. Dilute to the mark with 1 500 g/l [83,6 % (*m/m*)] sulfuric acid solution.

If necessary, pass the solution through a sintered glass filter. Store the solution in the dark. It may be used until the percentage transmittance at 571 nm in a cell of optical path length 1 cm is less than 75 %.

4.6 Tin(II) chloride, solution.

Weigh 3,0 g of tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) into a 100 ml one-mark volumetric flask and dissolve in 3 ml of hydrochloric acid (ρ_{20} 1,18 g/ml). Dilute to the mark with water.

The reagent shall be freshly prepared.

4.7 Glycerol, standard solution, containing 25 mg of glycerol per litre.

Weigh, to the nearest 0,01 mg, 500,0 mg of glycerol (R 64) (ISO 6353-3) and transfer to a 1 000 ml one-mark volumetric flask; dissolve in water and dilute to the mark.