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Surface active agents — Analysis of soaps — Determination of low contents of free glycerol — Spectrophotometric method

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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.

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

It was approved in August 1971 by the Member Bodies of the following countries :

Austria	New Zealand	Switzerland
Belgium	Poland	Thailand
Egypt, Arab Rep. of	Portugal	Turkey
France	Romania	United Kingdom
Germany	South Africa, Rep. of	U.S.A.
Hungary	Spain	U.S.S.R.
Japan	Sweden	

No Member Body expressed disapproval of the document.

Surface active agents — Analysis of soaps — Determination of low contents of free glycerol — Spectrophotometric method

1 SCOPE

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

2 FIELD OF APPLICATION

This method is applicable to soaps having a free glycerol content of less than 0.5 % (m/m).

3 REFERENCES

ISO/R 385, *Burettes*.

ISO/R 1042, *One-mark volumetric flasks*.

ISO/R 1066, *Analysis of soaps — Determination of glycerol*.

4 PRINCIPLE

Decomposition of the soap by sulphuric 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.

After reaction with chromotropic acid, the aldehyde formed gives a coloration of intensity proportional to the free glycerol content.

5 REAGENTS

The water used shall be distilled water or water of at least equivalent purity.

The reagents used shall have the following properties :

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

5.2 Sulphuric acid, 4.6 N solution, i.e. 20 % (m/m) ($\rho_{20} = 1.14$ g/ml).

5.3 Sulphuric acid, 20 N solution, i.e. 64 % (m/m) ($\rho_{20} = 1.54$ g/ml).

5.4 Sodium periodate reagent (solution approximately 0.03 M), prepared as follows :

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 0.5 N sulphuric acid. Dilute to the mark with 0.5 N sulphuric acid.

5.5 Chromotropic acid reagent, solution prepared as follows :

Weigh either 0.25 g of *di*-sodium 1,8 dihydroxynaphthalene-3,6-disulphonate dihydrate or the corresponding mass, 0.23 g, of the anhydrous salt (minimum purities 99 %) into a 250 ml one-mark volumetric flask and dissolve in 10 ml of water. Dilute to the mark with 30 N sulphuric acid (83.6 % (m/m) of H_2SO_4).

If necessary, pass the solution through a sintered glass filter. The reagent must be stored in the dark. It may be used until the transmittance at 571 nm in a 1 cm cell is less than 75 %.

5.6 Tin (II) chloride reagent, solution prepared as follows :

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 ($\rho_{20} = 1.18$ g/ml).

Dilute to the mark with water.

The reagent shall be freshly prepared.

5.7 Standard glycerol solution

Weigh an amount of glycerol corresponding to 500.0 mg of 100 % material (as determined by the method described in ISO/R 1066) and transfer to a 1 000 ml one-mark volumetric flask; dissolve in water and dilute to the mark.

Transfer 50 ml of the well-mixed solution into another 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well. 1 ml of this solution contains 25 µg of glycerol.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 One-mark volumetric flasks, 100 ml capacity, complying with Class A of ISO/R 1042.