

TC 91

INTERNATIONAL STANDARD 2272

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

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

First edition – 1972-09-01

[ISO 2272:1972](https://standards.iteh.ai/catalog/standards/sist/5eee86d0-b2b5-4895-af44-dba6423baee0/iso-2272-1972)
<https://standards.iteh.ai/catalog/standards/sist/5eee86d0-b2b5-4895-af44-dba6423baee0/iso-2272-1972>

UDC 668.1 : 547.426

Ref. No. ISO 2272-1972 (E)

Descriptors : chemical analysis, determination of content, glycerol, soaps, spectrophotometry, surfactants.

Price based on 2 pages

ISO 2272-1972 (E)

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*.

ITeH STANDARD PREVIEW
(standards.iteh.ai)

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	

ISO 2272:1972

<https://standards.iteh.ai/catalog/standards/sist/5eee86d0-b2b5-4895-af44-dba642310000/iso-2272-1972>

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.

6.2 Burette, 5 ml, graduated in 0.01 ml, complying with Class A of ISO/R 385.

6.3 Water bath.

6.4 Separating funnels, 250 ml capacity.

6.5 Spectrophotometer.

7 PROCEDURE

7.1 Preparation of test portion

Weigh, to the nearest 0.001 g, 2 to 3 g of soap, into a glass-stoppered conical flask. Add 10 ml of the sulphuric acid (5.2) and heat on the water bath (6.3) until the fatty acids form a clear layer.

Transfer the mixture to a 250 ml separating funnel (6.4), rinsing the conical flask twice with 25 ml of the light petroleum (5.1) and then with 25 ml of water. Shake, allow to separate and draw off the aqueous layer into a conical flask. Extract the light petroleum layer twice, each time with 10 ml of water. Combine the wash waters with the first aqueous layer. Remove the light petroleum present in the aqueous layer by warming on the water bath (6.3). Transfer the solution to a 100 ml one-mark volumetric flask (6.1), dilute to the mark with water and mix well (solution S).

NOTE - If the solution is turbid (possibly due to the presence of titanium dioxide), transfer it to a 100 ml one-mark volumetric flask (6.1), passing it through a filter paper. Wash the filter paper repeatedly with water.

7.2 Determination

Using a pipette, transfer 2.00 ml of solution S to a 100 ml one-mark volumetric flask (6.1).

Add 1 ml of the periodate reagent (5.4) and allow to stand for 15 min.

NOTE - Depending on the free glycerol content, it may sometimes be preferable to transfer 1.00 ml of solution S to a 100 ml one-mark volumetric flask (6.1).

Add 1 ml of the tin (II) chloride solution (5.6) and 10 ml of the chromotropic acid reagent (5.5). Mix well and heat for 30 min on the water bath (6.3). Allow to cool to room temperature, dilute to the mark with 20 N sulphuric acid solution (5.3) and mix well (solution S₁).

Fill a 1 cm cell of the spectrophotometer (6.5) with this solution S₁. Measure the absorbance of the solution S₁ at a constant temperature between 15 and 25 °C, and at the wavelength corresponding to maximum absorption (approximately 571 nm, but the exact wavelength must be checked for each spectrophotometer), and deduct the absorbance of the blank test (7.3).

Read the mass, in micrograms, of free glycerol present in the solution S₁, from the calibration graph (7.4).

7.3 Blank test

Transfer 2 ml of water to a 100 ml one-mark volumetric flask and proceed as described in 7.2.

7.4 Calibration graph

Into a series of 100 ml one-mark volumetric flasks, transfer by means of the 5 ml burette (6.2) :

0.40 0.80 1.40 and 2.00 ml of the standard glycerol solution (5.7), corresponding to :
10 20 35 and 50 µg of glycerol.

Dilute to 2 ml with water and proceed as described in 7.2.

8 EXPRESSION OF RESULTS

The free glycerol content, as a percentage by mass, in the soap is equal to

$$\frac{m_1 \times 100 \times 100}{2 \times m_0 \times 1\,000 \times 1\,000} = 0.005 \frac{m_1}{m_0}$$

where

*m*₀ is the mass, in grams, of the test portion;

*m*₁ is the mass, in micrograms, of free glycerol present in solution S₁, read from the calibration graph.

NOTE - If 1.00 ml of the solution S is used instead of 2.00 ml, the free glycerol content, as a percentage by mass, is equal to

$$0.01 \frac{m_1}{m_0}$$

9 TEST REPORT

The test report shall include the following information :

- a) all information necessary for the complete identification of the sample;
- b) the method used;
- c) the results obtained;
- d) the test conditions;
- e) any operational details not specified in this International Standard, or optional, as well as all incidents likely to have influenced the results.

Plot a graph having, for example, the masses, in microgram of glycerol contained in 2 ml of the standard solutions as abscissae, and the corresponding values of absorbance as ordinates .