INTERNATIONAL STANDARD

ISO 2272

Second edition
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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

Reference number
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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 ($SnCl_2 \cdot 2H_2O$) 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.

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 standard solution contains 25 µg of glycerol.

5 Apparatus

Ordinary laboratory apparatus and

5.1 One-mark volumetric flasks, 100 ml capacity, complying with the requirements of class A of ISO 1042.

5.2 Burette, 5 ml capacity, graduated at 0,01 ml intervals, complying with the requirements of class A of ISO 385-1.

5.3 Water bath.

5.4 Separating funnels, 250 ml nominal capacity.

5.5 Spectrometer, with selector for continuous variation of wavelength between 350 nm and 760 nm.

6 Sampling

The laboratory sample shall be prepared and stored in accordance with the instructions given in ISO 8212.

7 Procedure

7.1 Preparation of test solution

Weigh, to nearest 1 mg, 2 g to 3 g of the laboratory sample (see clause 6) into a conical flask with a ground glass stopper. Add 10 ml of the sulfuric acid solution (4.2) and heat on the water bath (5.3) until the fatty acids form a clear layer.

Transfer the mixture to a 250 ml separating funnel (5.4), rinsing the conical flask twice with 25 ml of the light petroleum (4.1), and then with 25 ml of water. Shake, allow to separate and draw off the aqueous phase into a conical flask. Extract the light petroleum twice more, each time with 10 ml of water, combining these two aqueous extracts with the initial 25 ml aqueous extract. Remove the light petroleum present in the combined aqueous extract by warming on the water bath. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask (5.1), dilute to the mark with water and mix well. [If the solution is turbid (due to the possible presence of titanium dioxide), transfer it to the 100 ml one-mark volumetric flask passing it through a filter paper. Dilute to the mark, washing the filter paper repeatedly with water.]

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all reagents as used for the determination, diluting to the same volume but replacing the test solution by 2,00 ml of water.

7.3 Calibration

7.3.1 Preparation of calibration solutions for spectrometric measurements carried out using cells of 1 cm optical path length

Into a series of 100 ml one-mark volumetric flasks (5.1), introduce, by means of the 5 ml burette (5.2):

0,40 ml, 0,80 ml, 1,40 ml and 2,00 ml of the standard glycerol solution (4.7), corresponding to:

10 µg, 20 µg, 35 µg and 50 µg of glycerol.

Dilute to 2 ml with water and proceed as described in 7.4.1.

7.3.2 Spectrometric measurements

Measure the absorbance of the calibration solutions (7.3.1) and that of the blank test solution (see 7.2), after having adjusted the instrument (5.5) to zero absorbance against water, following the procedure specified in 7.4.2.

7.3.3 Plotting the calibration graph

Deduct the absorbance of the blank test solution from that of each calibration solution and plot a graph, having, for example, the glycerol contents, in micrograms per 100 ml of calibration solution, as abscissae and the corresponding values of absorbance as ordinates.

7.4 Determination

7.4.1 Formation of the absorbing compound

Using a pipette, transfer 2,00 ml of the test solution (see 7.1) to a 100 ml one-mark volumetric flask (5.1).

Add 1 ml of the sodium periodate solution (4.4) 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 the test solution to the 100 ml one-mark volumetric flask (5.1).

Add 1 ml of the tin(II) chloride solution (4.6) and 10 ml of the chromotropic acid solution (4.5). Mix well and heat for 30 min on the water bath (5.3). Allow to cool to room temperature, dilute to the mark with sulfuric acid solution (4.3) and mix well.

7.4.2 Spectrometric measurements

Fill a 1 cm optical path length cell of the spectrometer (5.5) with the solution prepared in 7.4.1. Measure the absorbance of the solution at a constant temperature between 15 °C and 25 °C, and at the wavelength corresponding to maximum absorption (approximately 571 nm, but the exact wavelength shall be checked for each spectrometer), after having adjusted the instrument to zero absorbance against water.