INTERNATIONAL STANDARD 1067

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

Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter

Analyse des savons — Détermination de la teneur en matières insaponifiables, en matières insaponifiées et en matières saponifiables insaponifiées

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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 now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 91 has reviewed ISO Recommendation R 1067 and found it suitable for transformation. International Standard ISO 1067 therefore replaces ISO Recommendation R 1067-1969.

ISO Recommendation R 1067 was approved by the Member Bodies of the following countries :

Austria	India	Portugal
Belgium	Iran	Romania
Canada	Ireland	South Africa, Rep. of
Chile	Israel	Spain
Czechoslovakia	Japan	Sweden
Egypt, Arab Rep. of	Korea, Rep. of	Switzerland
France	Netherlands	Turkey
Germany	New Zealand	United Kingdom
Hungary	Poland	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 1067 into an International Standard :

United Kingdom

Printed in Switzerland

Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter

1 SCOPE

This International Standard specifies a method for the determination of the contents of unsaponifiable, unsaponified and unsaponified saponifiable matter in commercial soaps, excluding compound products.

2 FIELD OF APPLICATION

This method is applicable to the determination of the contents of products, other than free fatty acids, which are soluble in hexane or light petroleum (unsaponifiable + unsaponified matter), and which can be saponified (unsaponified saponifiable matter).

The method is not applicable to soaps enriched with sterols or long chain alcohols, nor to soaps containing perfume.

3 PRINCIPLE

Extraction of matter soluble in hexane, and titration of the free fatty acids removed, with potassium hydroxide solution.

Saponification of products soluble in hexane neutralized in this way and extraction of the unsaponifiable matter by hexane.

4 REAGENTS

The water used shall be distilled water or water of equivalent purity. The reagents shall have the following characteristics :

4.1 Ethanol, free from carbon dioxide, neutralized hot with the ethanolic potassium hydroxide solution (4.4) using the phenolphthalein solution (4.6) as indicator.

4.2 Sodium hydrogen carbonate, 10 g/l solution.

4.3 *n*-**Hexane,** technical grade or, failing this, **light petroleum** distilling at between 40 and 60 °C, having a bromine number less than 1, and free from residue.

4.4 Potassium hydroxide, 0,1 N standard volumetric solution in ethanol.

4.5 Potassium hydroxide, 2 N standard volumetric solution in ethanol.

4.6 Phenolphthalein, 10 g/l solution in 95 % (*V*/*V*) ethanol.

1) In preparation.

5 APPARATUS

Ordinary laboratory apparatus and, in particular :

5.1 Beaker, 250 ml.

5.2 Separating funnels, 50 ml and 250 ml.

5.3 Round-bottomed flasks, 100 ml and 250 ml, complying with ISO/R 1773.

5.4 Microburette, 2 ml.

5.5 Pipette, 10 ml, complying with ISO/R 648.

5.6 Oven, capable of being controlled at 103 ± 2 °C.

6 SAMPLING

The laboratory sample of soap shall be prepared and stored in accordance with the instructions given in ISO . . ., *Soaps — Sampling.* [1)]

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample, the soap being finely grated, into the 250 ml beaker (5.1).

7.2 Determination

Add 50 ml of the neutralized ethanol (4.1) and 50 ml of the sodium hydrogen carbonate solution (4.2) to the test portion (7.1). Dissolve the soap by heating to not above 70 °C.

After the soap is completely dissolved, allow the solution to cool. Transfer the solution quantitatively to the 250 ml separating funnel (5.2), rinsing the beaker several times with a mixture of equal volumes of the neutralized ethanol (4.1) and the sodium hydrogen carbonate solution (4.2), and extract three times, stirring carefully, each time with 50 ml of the hexane or the light petroleum (4.3). Combine the extracts, filter them if necessary, and wash them until

neutral to phenolphthalein, using for each wash 50 ml of a mixture of equal volumes of the neutralized ethanol (4.1) and water. Normally three washings are sufficient. Transfer the solution quantitatively to the 250 ml flask (5.3), previously dried in the oven (5.6) controlled at 103 ± 2 °C, allowed to cool in a desiccator and weighed to the nearest 0,2 mg.

Evaporate most of the solvent on a boiling water bath and remove the last traces with the aid of a gentle current of dry air directed into the flask while it is held obliquely, almost entirely immersed in the bath, and rotated.

Dry the flask and residue for 5 min in the oven (5.6) controlled at 103 ± 2 °C, allow it to cool in a desiccator and weigh to the nearest 0,2 mg. Repeat the operations of drying, cooling and weighing until the difference between two successive weighings does not exceed 2 mg. Let this mass be m_1.

Dissolve the residue in a few millilitres of the neutralized ethanol (4.1). Using the microburette (5.4), titrate the free acidity with the potassium hydroxide solution (4.4), using the phenolphthalein solution (4.6) as indicator, until the solution turns pink. Note the volume V of this solution used for the titration.

Add 10,0 ml of the potassium hydroxide solution (4.5), using the pipette (5.5). Bring the solution to boiling point and boil it under reflux for 30 min. Then add a volume of water equal to the volume of the solution and transfer the solution quantitatively to the 50 ml separating funnel (5.2), using a few millilitres of a mixture of equal volumes of the neutralized ethanol (4.1) and water to rinse the flask. Extract three times, each time with 10 ml of the hexane or the light petroleum (4.3). Combine the extracts and wash them until neutral to phenolphthalein, using for each wash 10 ml of a mixture of equal volumes of the neutralized ethanol (4.1) and water. Normally three washings are sufficient. Transfer the solution quantitatively into the 100 ml flask (5.3), previously dried in the oven (5.6) controlled at 103 ± 2 °C, allowed to cool in a desiccator and weighed to the nearest 0,2 mg.

Evaporate most of the solvent on a boiling water bath and remove the last traces with the aid of a gentle current of dry air directed into the flask while it is held obliquely, almost entirely immersed in the bath, and rotated.

Dry the flask and residue for 5 min in the oven (5.6) controlled at 103 ± 2 °C, allow it to cool in a desiccator and weigh to the nearest 0,2 mg. Repeat the operations of drying, cooling and weighing until the difference between two successive weighings does not exceed 2 mg. Let this mass be m_2.

8 EXPRESSION OF RESULTS

The percentage, by mass, of unsaponified matter + unsaponifiable matter in the soap is equal to

$$\left(m_1 - \frac{V \times M}{10\,000}\right) \times \frac{100}{m_0}$$

The percentage, by mass, of unsaponifiable matter in the soap is equal to

$$m_2 \times \frac{100}{m_0}$$

The percentage, by mass, of unsaponified saponifiable matter in the soap is equal to

$$\left(m_1 - \frac{V \times M}{10\,000} - m_2\right) \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the first extract;

m_2 is the mass, in grams, of the second extract;

M is the mean relative molar mass of the fatty acids of the soap (see note);

V is the volume, in millilitres, of the 0,1 N standard volumetric ethanolic potassium hydroxide solution (4.4) used to determine the acidity of the first extract.

NOTE – The mean relative molar mass M of the fatty acids of the soap can be determined by titration of the fatty acids isolated after the saponification of a sample of the original soap, the elimination of unsaponifiable matter and the acidification of the soap solution.

9 TEST REPORT

The test report shall indicate the method used and the results obtained. It shall also mention all test conditions and procedural details not specified in this International Standard, or which are optional, and any factors which may have affected the results.

The test report shall give all information necessary for the complete identification of the sample.