UDC 661.185 : 543.855/.86

Ref. No. : ISO/R 1067-1969 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1067

ANALYSIS OF SOAPS

DETERMINATION OF UNSAPONIFIABLE AND UNSAPONIFIED MATTER

1st EDITION
April 1969

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1067, *Analysis of soaps – Determination of unsaponifiable and unsaponified matter,* was drawn up by Technical Committee ISO/TC 91, *Surface active agents,* the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question led to the adoption of a Draft ISO Recommendation.

In December 1967, this Draft ISO Recommendation (No. 1471) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1067 April 1969

ANALYSIS OF SOAPS

DETERMINATION OF UNSAPONIFIABLE AND UNSAPONIFIED MATTER

1. SCOPE

This ISO Recommendation describes a method for the determination of the content of unsaponifiable and unsaponified matter in commercial soaps, excluding compound products.

2. FIELD OF APPLICATION

This method may be used for the determination of products other than free fatty acids, soluble in hexane (unsaponifiable + unsaponified matter), and those 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 by means of potassium hydroxide of the free fatty acids removed.

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

4. REAGENTS

4.1 *Ethanol* free from carbon dioxide, neutralized hot by means of the ethanolic solution of potassium hydroxide (4.4) with phenolphthalein as indicator.

4.2 *Sodium hydrogen carbonate,* solution of 1 g of sodium hydrogen carbonate per 100 ml of distilled water.

4.3 *n-Hexane, technical grade* or, failing this, *light petroleum* distilling at a temperature between 40 and 60 °C, with 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 reagent solution in ethanol.

4.6 *Phenolphthalein* solution of 1 g in 100 ml of 95 % (V/V) ethanol.

5. APPARATUS

Usual laboratory apparatus, and in particular

5.1 *Beaker,* 250 ml.

5.2 *Separating funnels,* 50 and 250 ml.

5.3 *Round-bottomed flasks,* 100 ml and 250 ml.

5.4 *Reflux condenser.*

5.5 *Microburette,* 2 ml.

5.6 *Pipette,* 10 ml.

5.7 *Analytical balance.*

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, about 5 g of finely grated soap into the 250 ml beaker.

6.2 Determination

Add to the test portion 50 ml of neutralized ethanol (4.1) and 50 ml of sodium hydrogen carbonate solution (4.2). Dissolve the soap by heating, without exceeding 70 °C.

After the soap is completely dissolved, allow the solution to cool; transfer the whole of the solution quantitatively into the 250 ml separating funnel, rinsing the beaker several times with a mixture of equal volumes of neutralized ethanol (4.1) and sodium hydrogen carbonate solution (4.2), and extract three times stirring carefully, each time using 50 ml of hexane (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 neutralized ethanol (4.1) and water. Normally three washings are sufficient. Transfer the solution quantitatively to the 250 ml flask, which has been tared. Distil the major part of the hexane (4.3), then dry the residue rapidly at 103 ± 2 °C until the difference between two successive weighings, separated by a period in the oven of 5 minutes, is less than 2 mg. Let this mass be M_1.

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

Add 10 ml of the ethanolic solution of potassium hydroxide (4.5). Bring the solution to boiling point and boil it under the reflux condenser for 30 minutes. Then add a volume of water equal to the volume of the solution and transfer the solution quantitatively into the 50 ml separating funnel, using a few millilitres of a mixture of equal volumes of neutralized ethanol (4.1) and water to rinse the flask. Extract three times, each time using 10 ml of hexane (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 neutralized ethanol (4.1) and water. Normally three washings are sufficient.

Transfer the solution quantitatively into the 100 ml flask. Distil the major part of the hexane and dry the residue rapidly at 103 ± 2 °C, until the difference between two consecutive weighings, separated by a period in the oven of 5 minutes, is less than 2 mg. Let this mass be M_2.