INTERNATIONAL STANDARD 685

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Analysis of soaps — Determination of total alkali content and total fatty matter content

Analyse des savons — Détermination des teneurs en alcali total et en matière grasse totale

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FOREWORD

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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 685 is the result of the combination of draft International Standards ISO/DIS 455 and ISO/DIS 685 drawn up by Technical Committee ISO/TC 91, *Surface active agents.*

Drafts ISO/DIS 455 and ISO/DIS 685 were circulated to the Member Bodies in November 1973 and December 1973 respectively.

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No Member Body expressed disapproval of the document.

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This International Standard cancels and replaces ISO Recommendations R 455-1965 and R 685-1968, of which it constitutes a technical revision.

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Analysis of soaps — Determination of total alkali content and total fatty matter content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the simultaneous determination of the total alkali1) content and the total fatty matter content of soaps, excluding compounded products.

This method for the determination of total alkali is not applicable to coloured soaps if the colour interferes with the methyl orange end-point.

2 REFERENCES

ISO 684, *Analysis of soaps — Determination of total free alkali.*

ISO . . ., *Soaps — Sampling.*2)

3 DEFINITIONS

For the purposes of this International Standard, the following definitions apply :

total alkali : The sum of the alkali bases combined as soap with fatty and rosin acids, as well as those corresponding to free alkali metal hydroxides or carbonates and to any silicates present which will be titrated under the test conditions.

The results are expressed as a percentage by mass of either sodium hydroxide (NaOH) or of potassium hydroxide (KOH), according to whether sodium or potassium soaps are concerned.

total fatty matter : The water-insoluble fatty material obtained by decomposing the soap with a mineral acid under the conditions specified. This term includes unsaponifiable matter, glycerides and any rosin acids contained in the soap, in addition to the fatty acids.

4 PRINCIPLE

Decomposition of the soap by a known volume of standard volumetric mineral acid solution, extraction and separation of the liberated fatty matter with light petroleum and determination of the total alkali content by titration of the excess of acid contained in the aqueous phase with a standard volumetric sodium hydroxide solution. After evaporation of the light petroleum from the extract, dissolution of the residue in ethanol and neutralization of the fatty acids with a standard volumetric potassium hydroxide solution. Evaporation of the ethanol and weighing of the soap formed to determine the total fatty matter content.

5 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

5.1 Acetone.

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

5.3 Ethanol, 95 % (*V*/*V*) solution, neutralized to the phenolphthalein solution (5.8).

5.4 Sulphuric acid or **hydrochloric acid,** approximately 1 N standard volumetric solution

5.5 Sodium hydroxide, approximately 1 N standard volumetric solution, standardized using the methyl orange solution (5.7) as indicator.

5.6 Potassium hydroxide, approximately 1 N standard volumetric solution in ethanol (5.3).

5.7 Methyl orange, 2 g/l solution.

5.8 Phenolphthalein, 10 g/l solution in ethanol (5.3).

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Beaker, capacity 250 ml, squat form, complying with ISO 3819.

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1) See also ISO 684.

2) In preparation.

6.2 Separating funnels, capacity 500 ml, or

6.3 Extraction cylinder, capacity 250 ml, diameter 39 mm and height 355 mm, fitted with a ground glass stopper.

6.4 Water bath.

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

7 SAMPLING

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

8 PROCEDURE

Carry out two determinations on the same sample.

8.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample into the beaker (6.1).

8.2 Determination

Dissolve the test portion (8.1) in about 100 ml of hot water.

Pour the solution into one of the separating funnels (6.2) or into the extraction cylinder (6.3) and wash the beaker with small quantities of water, adding the washings to the separating funnel or to the extraction cylinder.

Add a few drops of the methyl orange solution (5.7) and then, from a burette, add, while vigorously shaking the separating funnel or the extraction cylinder, an accurately measured known volume of the sulphuric acid or hydrochloric acid solution (5.4) until there is an excess of about 5 ml. Cool the contents of the separating funnel or of the extraction cylinder to about 25 °C and add 100 ml of the light petroleum (5.2). Insert the stopper and gently invert the separating funnel or the extraction cylinder, whilst maintaining a hold on the stopper. Open the stopcock of the separating funnel or the stopper of the extraction cylinder gradually to release any pressure, then close, gently shake and again release the pressure. Repeat the shaking until the aqueous layer has become clear, and then allow to stand.

a) *In the case of use of separating funnels*

Run off the aqueous layer into a second separating funnel (6.2) and extract with 50 ml of the light petroleum (5.2). Repeat the process, collect the aqueous layer in a conical flask and combine the three light petroleum extracts in the first separating funnel.

b) *In the case of use of an extraction cylinder*

Using a siphon, draw off the light petroleum layer as completely as possible into a separating funnel (6.2). Repeat the extraction twice with 50 ml of the light petroleum (5.2), combine the three light petroleum extracts in the separating funnel, transfer the aqueous layer as completely as possible to a conical flask and wash the extraction cylinder with small quantities of water, adding the washings to the conical flask.

Wash the light petroleum extract by shaking with water (about 25 ml) until the washings are neutral to the methyl orange solution (5.7). Usually three washings are sufficient.

NOTE — Allow each wash to stand for at least 5 min or such a time as is required to give a clear line of demarcation between the layers, before running off the aqueous layer.

After the final washing has been run off, impart a swirling motion to the contents of the separating funnel by rotating it sharply, but without inverting it, to remove any water droplets adhering to the sides.

Allow to stand for at least 5 min and run off any separated water.

Collect the washings of the light petroleum extract quantitatively in the conical flask already containing the aqueous layer.

8.2.1 *Determination of total alkali content*

Titrate the mixed acid aqueous layer and washings with the sodium hydroxide solution (5.5) using the methyl orange solution (5.7) as indicator.

8.2.2 *Determination of total fatty matter content*

Carefully transfer the washed light petroleum solution to a weighed, flat-bottomed flask, filtering if necessary through a dry filter paper. Wash the separating funnel with two or three small quantities of the light petroleum and filter the washings into the flask, taking precautions to prevent evaporation of the light petroleum during the filtration. Thoroughly wash the filter with the light petroleum, collecting the washings in the flask.

Evaporate off nearly all the light petroleum on the water bath (6.4), taking all necessary precautions, and under a slow stream of cold dry nitrogen or air.

Dissolve the residue in 20 ml of the ethanol (5.3), add a few drops of the phenolphthalein solution (5.8) and titrate with the ethanolic potassium hydroxide solution (5.6) to a faint permanent pink colour. Note the volume used.

Evaporate the ethanolic solution on the water bath (6.4). When the evaporation is near completion, rotate the flask in order to distribute the potassium soap in a thin layer on the sides and bottom of the vessel.

Carry out a preliminary drying of the potassium soap by adding acetone (5.1) and evaporating off the acetone on the water bath under a slow stream of cold dry nitrogen or air. Then heat to constant mass in the oven (6.5), controlled at 103 ± 2 °C, i.e. until the difference in mass after heating for an additional 15 min does not exceed 3 mg. Cool in a desiccator and weigh.