SLOVENSKI STANDARD
SIST ISO 685:2020

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Analiza mila - Določevanje celotnih alkalij in celotnih maščobnih snovi

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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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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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 685:1975), which has been technically revised.

The main changes compared to the previous edition are as follows:

— “liquid soaps” has been added in the Scope;

— the sentence “This method for the determination of total alkali is not applicable to coloured soaps if the colour interferes with the methyl orange end-point.” has been deleted from the Scope;

— in 8.3.1, “until there is an excess of about 5 ml” has been changed to “until there is an excess of about 10 ml”;

— in 8.3.1, “Repeat the shaking until the aqueous layer has become clear” has been changed to “Repeat the shaking until the water phase is clearly separated from the organic phase (if the two-phase layer is not obvious, the emulsification can be broken by adding ethanol that does not exceed the volume of the water phase)”;

— in 8.3.2, the sentence “If the soap colour interferes with the methyl orange end-point, a pH meter can be used to indicate the end point (pH value 3,1 to 4.4, maintain 10 s) during titration.” has been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies a method for the simultaneous determination of the total alkali content and the total fatty matter content of soaps (including liquid soaps), excluding compounded products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3819, *Laboratory glassware — Beakers*

ISO 8212, *Soaps and detergents — Techniques of sampling during manufacture*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
total alkali
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

Note 1 to entry: The results are expressed as a percentage mass fraction of either sodium hydroxide (NaOH) or of potassium hydroxide (KOH), according to whether sodium or potassium soaps are concerned.

3.2
total fatty matter
water-insoluble fatty material obtained by decomposing the soap with a mineral acid under the conditions specified

Note 1 to entry: 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 grade and only distilled water or water of equivalent purity.

5.1 Acetone.

5.2 Light petroleum, boiling range between 30 °C and 60 °C.

5.3 Ethanol, 95 % (volume fraction) solution, neutralized to the phenolphthalein solution (5.8).

5.4 Sulphuric acid, approximately 0,5 mol/l (H_2SO_4) standard volumetric solution.

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

5.6 Potassium hydroxide, approximately 1 mol/l 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

Use ordinary laboratory apparatus and the following.

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

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.

6.6 Cheese grater or **a similar grinder**.

7 Sampling

The sampling shall be done in accordance with ISO 8212. The soap bar should be grated with a cheese grater (6.6). At least half of the bar should be grated to ensure a complete representation of the bar. The grated soap sample should be kept in an air-tight container to avoid moisture loss.

8 Procedure

8.1 General

Carry out two determinations on the same sample.

8.2 Test portion

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

8.3 Determination

8.3.1 General

Dissolve the test portion (8.2) 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 solution (5.4) until there is an excess of about 10 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, while 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 water phase is clearly separated from the organic phase (if the two-phase layer is not obvious, the emulsification can be broken by adding ethanol that does not exceed the volume of the water phase), 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 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.

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.

8.3.2 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. If the soap colour interferes with the methyl orange end-point, a pH meter can be used to indicate the end-point (pH value 3,1 to 4,4, maintain 10 s) during titration.