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Second edition
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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

Reference number
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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.