



SLOVENSKI STANDARD

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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

ICS: 71.100.40

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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:

- Added liquid soaps in scope;
- Deleted “This method for the determination of total alkali is not applicable to coloured soaps if the colour interferes with the methyl orange end-point.” in scope;
- In [8.2](#) determination, “until there is an excess of about 5 ml” was changed to “until there is an excess of about 10 ml” ;
- In [8.2](#) determination, “Repeat the shaking until the aqueous layer has become clear” was changed to “Repeated 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 [8.2.1](#) Determination of total alkali content, added the sentence “If the soap colour interferes with the methyl orange end-point, the pH meter can be used to indicate the end point (pH value 3.1~4.4, maintain 10s) during titration”.

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 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

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.

3.2 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.

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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 % (V/V) solution, neutralized to the phenolphthalein solution ([5.8](#)).

5.4 Sulphuric acid, approximately 1 mol/l ($1/2\text{H}_2\text{SO}_4$) standard volumetric solution.

5.5 Sodium hydroxide, approximately 1 mol/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

Ordinary laboratory apparatus, and

6.1 Beaker, capacity 250 ml, squat form, complying 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 $(103 \pm 2)^\circ\text{C}$.

6.6 Cheese grater or other 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

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 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, 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. Repeated 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.

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. If the soap colour interferes with the methyl orange end-point, the pH meter can be used to indicate the end point (pH value 3.1~4.4, maintain 10s) during titration.

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.

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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 $(103 \pm 2)^\circ\text{C}$, 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.

9 Expression of results

9.1 Total alkali content

9.1.1 Method of calculation and formulae

Calculate the total alkali content, expressed as a percentage by mass of sodium hydroxide (NaOH) for sodium soaps, w_{NaOH} , using the formula (a), and expressed as a percentage by mass of potassium hydroxide (KOH) for potassium soaps, w_{KOH} , using the formula (b).

$$\text{a) } w_{\text{NaOH}} = 0.040 \times (V_0 T_0 - V_1 T_1) \times \frac{100}{m}$$

$$\text{b) } w_{\text{KOH}} = 0.056 \times (V_0 T_0 - V_1 T_1) \times \frac{100}{m}$$

Where

m is the mass, in grams, of the test portion (8.1);

V_0 is the volume, in millilitres, of the standard volumetric acid solution (5.4) used;

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (5.5) used;

T_0 is the exact normality of the standard volumetric acid solution (5.4);

T_1 is the exact normality of the standard volumetric sodium hydroxide solution (5.5)

The total alkali content may also be expressed in milliequivalents per gram, X , using the formula:

$$X = \frac{V_0 T_0 - V_1 T_1}{m}$$

Take as the result the arithmetic mean of the duplicate determinations.

9.1.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not differ by more than 0,2 % from the value of the percentage by mass of total alkali found, expressed either as sodium hydroxide or as potassium hydroxide.