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BHUTAN STANDARD

Natural Soap

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BHUTAN STANDARDS BUREAU

The National Standards Body of Bhutan

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FOREWORD

This Bhutan Standard for Natural Soap was developed by Bhutan Standards Bureau after the draft was finalised by the Sub-committee on Soaps (TC05/SC04 and Pharmaceuticals and Traditional Medicines Technical Committee (TC 05) and approved by the Bhutan Standards Bureau Board (BSB Board) on 2023.

This standard is subject to systematic review after five years to keep pace with the market trends, and industrial and technological developments. Any suggestions and further information may be directed to the concerned Technical Committee.

FD B T S

INTRODUCTION

Natural soap is made using ingredients that come from plants. It does not include harsh or synthetic ingredients that are not necessary for a thorough cleaning. For centuries, people have used natural soap for cleaning and personal hygiene needs. However, there is no clear and consistent definition of what constitutes natural soap and how it differs from other types of soap. This lack of clarity can lead to confusion among consumers, manufacturers, and regulators and can affect the quality, safety, and environmental impact of natural soap products.

As a result, a national standard that outlines the definition of natural soap, as well as its specifications and qualities, is necessary. This standard intends to specify requirements for natural soap products, including the ingredients, labelling, testing, and certification. Furthermore, it aims to protect the rights and interests of customers while promoting the safety and quality of natural soap products. The standard is based on the most up-to-date scientific and technical knowledge as well as input from pertinent stakeholders, including manufacturers, suppliers, consumers, regulators, and experts.

The standard is intended to be voluntary and flexible, allowing for innovation and diversity in the natural soap industry. The standard is also compatible with international standards and requirements for natural soap products. It is the responsibility and discretion of each individual or company to adopt or comply with this standard. The standard organisation or the technical committee shall not be liable for any untoward events, either health-related or material-related losses.

འབྲུག་ རང་བཞིན་འདུལ་ཐུང་(སྤང་ལེབ) གྱི་གནས་ཚད།

BHUTAN STANDARD

Natural Soap

1 Scope

This standard specifies essential characteristics or specifications for natural soap from Bhutan to facilitate the assessment of its quality. The purpose of this standard is to define the criteria and requirements for natural soap products, such as ingredients, labelling, testing and certification.

2 Normative References

There are no normative references for these documents.

3 Terms and Definition

For this standard the following definitions shall apply.

3.1 Colouring matter

Any dye that may be used to colour the natural soap.

3.2 Free caustic alkali

It is the free (uncombined) caustic alkali present in the soap.

3.3 Impurities

Insoluble extraneous matter found in the product. It may consist of but is not limited to dirt and miscellaneous debris, grit, mineral matter, and nitrogenous materials of animal or plant origin.

3.4 Mush

Mush is a soft, wet, pulpy mass formed on a bar soap due to mushing on a wet surface. The loss of mass of soap due to mushing is expressed as g/30 cm².

3.5 Natural Soap

Natural soap is a soap that is made from natural ingredients derived from plants that do not contain synthetic or harsh ingredients.

3.6 Soap

The product formed by the saponification or neutralisation of fats, oils, waxes, rosins or their acids with organic or inorganic bases.

3.7 Saponification

A chemical reaction permitting the separation of an ester into its constituent parts, acids and alcohol, or possibly phenol, by the action of a base, with the formation of salt from the acid. Saponification of fats produces soap.

3.8 Total fatty matter

Includes water-insoluble fatty matter obtained by decomposing the soap with a mineral acid under the specified conditions of the test.

3.9 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.

4 Acronyms and Symbols

AR	: Analytical grade
cm ²	: centimetre square
g	: grams
g/l	: gram/ litre
h	: hour
L	: Litre
M	: mass
Mg	: milligram
min	: minutes
ml	: millilitre
mm	: millimetre
m/v	: mass by volume
v/v	: volume by volume
v/w	: volume by weight
w/w	: weight by weight

5 Requirements

5.1 Description

Natural soap must be of the highest quality, completely saponified, milled, homogenised, or both, perfumed, or unscented, and compressed into smooth, firm cakes/bars. It must have effective lathering and cleaning abilities and shall be free from rancidity and rosins.

Natural soap shall not cause skin irritation and all ingredients used in the product shall be non-injurious to health.

5.2 Colour and odour

Natural soap shall be firm and of uniform texture and colour and shall be free from objectionable (disagreeable) odour. Perfumes and colouring matter may be added.

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5.3 Lather volume

The natural soap when lathered using standard hard water shall produce a lather volume of a minimum of 200 ml when determined using the method described in Annex A.

5.4 Texture and stability - Mushing properties of bar soap

The natural soap when immersed in 1 L of distilled water for 1 hour at 25°C to 30°C shall not show signs of disintegration, and when dried thereafter at room temperature for 24 hours shall not crumble and mush not more than 10 g/30 cm² when tested according to the method prescribed in Annex B.

5.5 Impurities

The soap shall be free of grittiness, sandiness or hard lumps before and after abrading when tested according to the method prescribed in Annex C.

5.6 Tests

5.6.1 pH value

The pH of natural soap should be 8 to 10 when tested using an appropriate pH meter or litmus paper.

5.6.2 Moisture and volatile matter content

The moisture and volatile matter content in the natural soap should be maximum of 20 percent by mass (at 105°C) when determined using the method described in Annex D.

5.6.3 Total alkali content

The total alkali content or alkalinity as NaOH should be maximum of 1 percent by mass when determined as per the method described in Annex E.

5.6.4 Total fatty matter content

The total fatty matter content in the soap should be a minimum of 40 percent by mass when determined using the method described in Annex E.

5.6.5 Free alkali content

The free alkali content as NaOH should be maximum of 0.1 percent by mass when determined as per the method in Annex F.

6 Packaging and Marking

6.1 Packing

The soap cakes or bars should be suitably wrapped to protect it from damage.

In general, the packaging materials must be inert toward the packed product to avoid damages to the product.

6.2 Labelling or Marking

The labelling materials shall be durable to withstand the transport conditions and avoid tampering.

Each wrapped soap bar or cake shall be marked or labelled with the following information, however not limited to:

- a) name of the product, and the trade name or brand name, if any;
- b) net weight or mass;
- c) batch identification;
- d) manufacturing date;
- e) full address of the manufacturer,
- f) Total fatty matter content (TFM); and
- g) country of origin.

7 Sampling and Inspection

7.1 Sampling

In a single consignment, all packages (cartons) containing natural soap cakes drawn from the same batch of production shall constitute a lot. For ascertaining the conformity of the lot to the requirements of this standard, tests shall be carried out on each lot separately. The number of packages to be selected for drawing the sample shall be in accordance with Table 1.

Table 1 — Scale of sampling

(Clause 7.1)

Number of packages (cartons) in the lot N	Number of packages (cartons) to be selected n
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

7.1.1 The packages shall be selected at random, using tables of random numbers. If these are not available, the following procedure shall be applied:

Starting from any package, count all the packages in one order as 1, 2, 3.... N, selecting every k^{th} package, where k is an integral part of $N \div n$.

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7.1.2 Every k^{th} package thus selected shall be withdrawn to obtain an adequate sample for the purpose of test.

7.2 Inspection

Inspect the soap bars or cakes selected for compliance with the requirements.

7.3 Preparation of test samples

7.3.1 Composite sample

Weigh each soap bar separately (including any material that may have adhered to the wrapper), and calculate the average mass. Keep one bar for the test under 5.4. Cut each of the remaining bars into eight parts by means of three cuts at right angles to each other through the middle. Grate finely the whole of two diagonally opposite eighths of each specimen. Mix the gratings and place in a clean, dry, airtight glass container.

7.3.2 Samples for testing

Immediately take at one time all test samples required for the tests in 5.6. Weigh out the test sample required for determination of free alkali or acid content in the end, and use it immediately.

7.4 Compliance with the standard

The lot shall be deemed to comply with the requirements of this standard if, after inspection and testing, the requirements of clause 5 are satisfied.

Annex A
(Normative)**Test for Lather Volume****A.1 General Principle**

A graduated cylinder containing the material suspended in standard hard water is inverted 12 times under the specified conditions. The inversion technique should be performed precisely as instructed. The foam volume is measured after letting the cylinder stand for 5 minutes.

A.2 Apparatus

- a) Graduated cylinder — Glass stoppered with graduation from 0 to 250 ml, with 2 ml divisions. Overall height about 35 cm and the height of the graduated portion about 20 cm.
- b) 100 ml glass beaker
- c) Thermometer of range 0 - 110°C

A.3 Reagents

- a) Calcium chloride $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, AR
- b) Magnesium sulphate $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, AR
- c) Distilled water

A.4 Preparation of standard hard water

Dissolve 0.220 g of calcium chloride dihydrate and 0.246 g of magnesium sulphate heptahydrate in distilled water. Dilute to 5 L with distilled water.

NOTE: This standard hard water has a hardness of approximately 50 ppm calculated as calcium carbonate.

A.5 Sample preparation

Cut away the outer edges of the soap bar using a knife. Using a grater, grate up to 10 g – 15 g of the soap bar into small chips.

A.6 Procedure

- a) Weigh 1 g of the grated chips of the soap bar accurately in a 100 ml glass beaker. Add 10 ml of the standard hard water. Cover the beaker with a watch glass and allow it to stand for 30 min.
- b) Stir the contents of the beaker with a glass rod and transfer the slurry to a 250 ml graduated cylinder ensuring that not more than 2 ml foam is produced.
- c) Repeat the transfer of the residue left in the beaker with further portions of 20 ml of standard hard water ensuring that all the matter in the beaker is transferred to the cylinder.
- d) Adjust the contents in the cylinder to 100 ml by adding sufficient standard hard water. Bring the contents of the cylinder to 30°C.
- e) Stir the contents of the cylinder with a glass rod or thermometer to ensure a uniform suspension.
- f) As soon as the temperature of the contents of the cylinder reaches 30°C, stopper the cylinder and give it 12 complete inversions, each inversion comprising of movements in a vertical plane, upside down and vice versa.
- g) After the 12 inversions, let the cylinder stand for 5 minutes. Take the following readings as shown in Figure A.1:
 - i) foam plus water (V_1 ml)
 - ii) water only (V_2 ml)

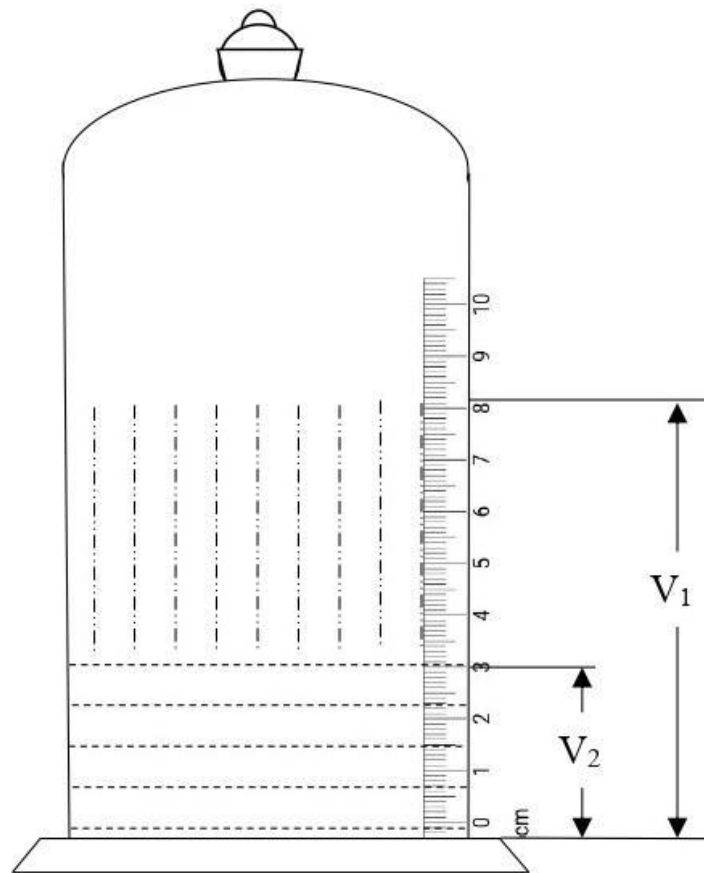


Fig A.1 - Measurement of foam

A.7 Calculation

Lather volume = $V_1 - V_2$

where

V_1 = Volume, in ml of foam + water;

V_2 = Volume, in ml of water only.

Annex B (Normative)

Evaluation of Stability and the Mushing properties

B.1 General Principle

A sample bar is cut to a specific size, excluding the hard outer layers. The sample is preconditioned by twisting 180 degrees for 18 times underwater at 25°C to prepare it for testing. The sample is placed on a wet cloth and covered for six hours to prevent drying. After six hours, the mush on the sample surface that touched the cloth is removed. The weight loss of the sample is calculated as mush per 30 cm² of original surface area that contacted the cloth.

B.2 Apparatus

- a) Coarse kitchen cheese grater
- b) Sharp thin blade knife
- c) Callipers or ruler
- d) Plastic or non-corrodible trays which are suitably sized for the test piece.
- e) Plastic soap dishes 7 x 11 x 2 cm.
- f) Cotton cloth pieces cut and folded to fit as a triple layer inside the trays.

B.3 Bar preparation

- a) Three (3) individual pieces of equal size are cut from each type of bar to be tested. The test piece should have a working face (to be applied to the fabric) of 6 ± 1 cm x 4 ± 1 cm. If the smallest range of bars to be tested is too small to allow a working face within these limits, then all bars should be cut to the maximum size possible from the smallest bar. The longest side of each test piece (6 ± 1 cm) should be parallel to the longest side of the original bar. Make a fresh surface on one face of each test piece. This is the working face. Mark the opposite face with a small hole. This helps to identify the working face after the preconditioning step.
- b) To cut the bar, it is convenient to first trim it to the approximate size using a coarse kitchen cheese grater and then to make the final adjustments to a smooth surface with a sharp thin-blade knife.

B.4 Test procedure

- a) Fill the tray lined with three layers of cloth with demineralised water. Hold the tray vertically to drain the water from the cloth until water ceases to run in a continuous stream.
- b) The area of the working face of the test piece is measured (A). The working face of the bar is placed onto the damp fabric and the tray is covered e.g. with a sealed plastic bag, to prevent water loss.
- c) The covered test piece and holder are maintained at 25°C for 6 hours.
- d) The mushed soap test piece is removed from the tray and weighed (W1).
- e) Mush is removed from the working face of the soap test piece by scraping with the edge of a blunt side spatula or plastic ruler.
- f) The test piece is reweighed (W2) and the amount of mush removed is calculated as in B.5. The mush is expressed as grams per 30 cm² of original test piece surface area.

NOTE: To weigh the bar and remove the mush, you will need some time. Meanwhile, the other soaps will keep forming mush. This is not a problem if you are testing only one product with three test pieces. But if you are testing more than one product, you should start the test for the second product later. This way, you will have enough time to finish the first set before the second set reaches 6 hours of storage time.

B.5 Calculation

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Weight of mush (grams) = $W = W_1 - W_2$

Surface area of bar (cm²) = $A = (\text{width} \times \text{breadth})$

Mush = $\frac{\square \times 30}{\square}$ grams per 30 cm²

B.6 Criteria for conformity

The test is done with three separate samples of each product type, and the mean value from three samples is recorded as (X). The range of values (R) is calculated as the difference between the highest and lowest values obtained for a given product type. The sample lot of products shall be declared as conforming to the requirements for this standard if $X + 0.6R$ is less than the maximum value.

Annex C
(Normative)

Determination of Impurities - Grittiness in a soap bar

C1. General Principle

The soap bar should be placed under a gentle stream of running water at a temperature of 30°C, and one side should be gently rubbed over the palm of the other hand for a minute each. After air drying the soap bar for hours, examine the surface for gritty particles.

C2. Apparatus

- a) Bowl, 5 litres capacity

C3. Test Procedure

- a) Hold the soap bar under a smooth stream of running water at a temperature of 30°C and gently rub the two sides of the bar on the palm of one hand for one minute each side, or immerse the soap in a bowl containing 5 litres of water at 30°C and gently rub two opposite bar faces with the palm of one hand for 30 seconds (15 seconds per bar face).
- b) Remove the bar from the water and continue to gently rub the two opposite bar faces for further 30 seconds (15 seconds per face).
- c) Allow the used bar to dry in the open for 4 hours and examine the surface.
- d) A set of 3 samples will be tested for each product.

NOTE: Hands will become hydrated and insensitive with prolonged immersion in water. Testers should wait 15 minutes between testing every 3 sets of products (9 grit tests). If using a bowl rather than running water, use fresh water after testing every set of 3 samples.

C4. Criteria for conformity

The sample lot of products shall be declared as conforming to the requirements if:

- a) Soap bar do not have a visibly rough surface and feels smooth to the touch during washing test;
- b) Has no gritty particles on the surface of the dried bar 4 hours after the washing test.

Annex D

(Normative)

Determination of moisture and volatile matter content - Oven method

D.1 General Principle

The oven method, which heats the natural soap ($103 \pm 2^\circ\text{C}$) and removes the water and other matter, can be used to detect the presence of water and volatile matter content in natural soap.

D.2 Apparatus

Usual laboratory apparatus and, in particular, the following are required:

- a) Evaporating dish or crystallising dish (diameter 6 to 8 cm and depth 2 to 4 cm)
- b) Glass stirring rod
- c) Sand, washed and calcined, or granulated pumice
- d) Oven (capable of being controlled at $103 \pm 2^\circ\text{C}$)
- e) Desiccator [containing an efficient desiccant, e.g., phosphorus (V) oxide (P_2O_5)]

D.3 Procedure

D.3.1 Test portion

Weigh, to the nearest 0.01 g, about 10 g of the laboratory sample soap bars, cut it into small pieces.

D.3.2 Determination

- a) Place the stirring rod in the dish and, only if the analysis is to be carried out on soft soap or on soap liquefiable at $103 \pm 2^\circ\text{C}$, also place in the dish about 10 g of the sand or pumice. Dry the dish and stirring rod, with or without added sand or pumice as appropriate, in the oven, controlled at $103 \pm 2^\circ\text{C}$. Allow to cool in the desiccator and weigh.
- a. Add the test portion to the dish and, if sand or pumice is used, mix this in by means of the stirring rod. Place the dish in the oven, controlled at $103 \pm 2^\circ\text{C}$.
- b. After 1 hour, remove from the oven and when cool, reduce the material to a fine powder by means of the stirring rod.
- c. Replace in the oven and after 1 hour, remove the dish. Place in the desiccator and leave just long enough for it to cool completely to ambient temperature and then weigh. Repeat the operations of heating for periods of 1 hour, cooling and weighing until the difference in mass between two successive weighings is less than 0.01g.
- d. Note the result of the final weighing.

D.4 Expression of result

D.4.1 Method of calculation

The formula provides the amount of moisture and volatile matter as a percentage of mass:

$$\frac{m_1 - m_2}{m_1 - m_0} \times 100$$

Where,

- \square_0 is the mass (grams) of the dish, rod and the sand or pumice (if used);
- \square_1 is the mass (grams), of the dish, rod and sand or pumice (if used) and the test portion before heating;
- \square_2 is the mass (grams), of the dish, rod and sand or pumice (if used) and the test portion after heating;

D.4.2 Reproducibility

Results from the same sample obtained in two different laboratories should not differ by more than 0.25%.

D.5 Test report

The following specifics shall be included in the test report:

- a) all details required for the sample's full identification;
- b) the method used as reference;
- c) the results and the method of expression;
- d) the test conditions;
- e) any operational details that are optional or not specified, as well as any events that may have had an impact on the results.

Annex E
(Normative)

Determination of total alkali content and total fatty matter content

E.1 Principle

The soap is decomposed using a known volume of a standard volumetric mineral acid solution, the liberated fatty matter is extracted and separated using light petroleum, and the total alkali content is determined by titrating the excess acid in the aqueous phase with a standard volumetric sodium hydroxide solution. After the light petroleum has been removed from the extract, the residue has been dissolved in ethanol, and the fatty acids have been neutralized with a typical volumetric potassium hydroxide solution. To calculate the total fatty matter content, the ethanol was evaporated, and the soap was weighed.

E.2 Apparatus

Usual laboratory apparatus and, in particular, the following are required:

- a) Beaker, capacity 250 ml
- b) Separating funnels, capacity 500 ml, or
- c) Extraction cylinder, capacity 250 ml, diameter 39 mm and height 355 mm, fitted with a ground glass stopper
- d) Water bath
- e) Oven, capable of being controlled at $103 \pm 2^\circ\text{C}$
- f) Cheese grater or a similar grinder.

E.3 Reagents

Use only the reagents of recognized analytical grade and distilled water.

- a) Acetone
- b) Light petroleum (boiling range: $30^\circ\text{C} - 60^\circ\text{C}$)
- c) Ethanol, 95 % (volume fraction) solution, neutralised to the phenolphthalein solution
- d) Sulphuric acid, approximately 0.5 mol/l (H_2SO_4) standard volumetric solution
- e) Sodium hydroxide, approximately 1 mol/l standard volumetric solution, standardised using the methyl orange solution as indicator
- f) Potassium hydroxide, approximately 1 mol/l standard volumetric solution in ethanol
- g) Methyl orange, 2 g/l solution
- h) Phenolphthalein, 10 g/l solution in ethanol

E.4 Procedure

Perform two determinations on the same sample.

E.4.1 Test portion

Weigh, to the nearest 0.001 g, about 5 g of the laboratory sample into the beaker.

E.4.2 Determination

E.4.2.1 General

In about 100 ml of hot water, dissolve the test portion. Pour the solution into the extraction cylinder or the separating funnels, then wash the beaker with small amounts of water. Add the washes to the extraction cylinder or the separating funnel.

Add a few drops of the methyl orange solution, and then, while vigorously shaking the separating funnel or the extraction cylinder, add an accurately measured known volume of the sulphuric acid solution until there is an excess of about 10 ml. Add 100 ml of the light petroleum after cooling the contents of the separating funnel or the extraction cylinder to about 25°C. Insert the stopper and gently invert the separating funnel or the extraction cylinder while maintaining a hold on the stopper. To release any pressure, gradually open the separating funnel stopcock or the extraction cylinder stopper. Then, close them, gently shake them, and release the pressure once more. Continue shaking until the water phase and organic phase can be clearly distinguished from one another (If the two-phase layer is not apparent, the emulsification can be broken by adding ethanol in an amount that does not exceed the volume of the water phase), and then let the mixture stand.

a) In the case of use of separating funnels

Drain the aqueous layer into a second separating funnel, and extract with 50 ml of the light petroleum. Repeat the procedure, gather 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

Draw as much of the light petroleum layer as possible into a separating funnel using a siphon. Repeat the extraction twice with 50 ml of the light petroleum, mix the three light petroleum extracts in the separating funnel, transfer the aqueous layer to the conical flask as completely as possible, and then wash the extraction cylinder with small amounts of water, adding the washings to the conical flask.

Wash the light petroleum extract with about 25 ml of water until it is neutral to the methyl orange solution (typically three washings are sufficient).

Before removing the aqueous layer from each wash, let it stand for at least 5 minutes or however long is necessary to clearly distinguish between the layers.

After the final washing has been run off, give the contents of the separating funnel a swirling motion by sharply rotating it without inverting it to get rid of any water droplets that have stuck to the sides.

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

Quantitatively collect the light petroleum extract washings in the conical flask that already has an aqueous layer in it.

E.4.2.2 Determination of total alkali content

Utilising the methyl orange solution as an indicator, titrate the mixture of the acid aqueous layer and washings with the sodium hydroxide solution. If the soap colour interferes with the methyl orange end-point, the end-point can be indicated during titration by using a pH metre (pH value 3.1 to 4.4, maintain 10 s).

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E.4.2.3 Determination of total fatty matter content

Transfer the washed light petroleum solution with care to a weighed flat-bottomed flask and, if necessary, filter it through a dry filter paper. Two or three small amounts of the light petroleum should be used to wash the separating funnel. The washings should then be filtered into the flask while taking care to prevent the light petroleum from evaporating during the filtration process. Thoroughly wash the filter with the light petroleum, collecting the washings in the flask.

Taking all necessary safety precautions and under a slow stream of cold dry nitrogen or air, evaporate nearly all of the light petroleum on the water bath.

The residue should be dissolved in 20 ml of ethanol, add a few drops of the phenolphthalein solution, and then titrate with an ethanolic potassium hydroxide solution until it acquires a faint, permanent pink colour. Note the volume used.

Evaporate the ethanolic solution on the water bath. Rotate the flask as the evaporation nears completion to evenly distribute the potassium soap in a thin layer on the sides and bottom of the container.

Preliminary dry off the potassium soap by adding acetone 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, controlled at 103 ± 2 °C, until the difference in mass after heating for an additional 15 min does not exceed 3 mg. Cool in a desiccator and weigh.

E.5 Expression of result

E.5.1 Total alkali content

E.5.1.1 Method of calculation and formulae

Calculate the total alkali content, expressed as a percentage mass fraction of sodium hydroxide (NaOH) for sodium soaps, w_{NaOH} , using the formula below:

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

Calculate the total alkali content, expressed as a percentage mass fraction of potassium hydroxide (KOH) for potassium soaps, w_{KOH} , using formula below:

$$\square_{\square\square\square} = 0.056 \times (V_0 T_0 - V_1 T_1) \times \frac{100}{m}$$

where

- m is the mass (grams) of the test portion;
- V_0 is the volume, in millilitres, of the standard volumetric acid solution used;
- V_1 is the volume in millilitres, of the standard volumetric sodium hydroxide solution used;
- T_0 is the exact normality of the standard volumetric acid solution;
- T_1 is the exact normality of the standard volumetric sodium hydroxide solution.

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

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

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

E.5.1.2 Reproducibility

The percentage mass fraction of total alkali found, expressed as sodium hydroxide or potassium hydroxide, should not differ between the results from the same sample in two different laboratories by more than 0.2%.

E.5.2 Total fatty matter content

E.5.2.1 Method of calculation and formula

Utilising the formula below, determine the total fatty matter content, expressed as a percentage mass fraction, w:

$$w = [m_1 - (V \times T \times 0.038)] \times \frac{100}{m_0}$$

where

- m_0 is the mass in grams of the test portion;
- m_1 is the mass in grams of the dried potassium soap;
- V is the volume in millilitres, of standard volumetric ethanolic potassium hydroxide solution used for the neutralization;
- T is the exact normality of the standard volumetric ethanolic potassium hydroxide solution.

Calculate the arithmetic mean of the duplicate determinations, and then round the result to the nearest 0.1 %.

E.5.2.2 Reproducibility

The percentage mass fraction of total fatty matter found should not vary between tests performed on the same sample in two different laboratories by more than 0.2 %.

E.6 Test report

The following specifics shall be included in the test report:

- a) all details required for the sample's complete identification;
- b) the method used as reference;
- c) the results and the method of expression;
- d) the test conditions;
- e) any operational details that are optional or not specified, as well as any events that may have had an impact on the results.

Annex F (Normative)

Determination of Total free alkali

F.1 General Principle

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The method involves dissolving soap in an ethanolic solution, neutralizing the free alkali with a known excess of sulphuric acid solution, and back titrating excess acid with an ethanolic potassium hydroxide solution.

If the soap contains additives (alkali silicates, etc.) that can be decomposed by sulphuric acid, the method is not applicable. This method is also not applicable if the colour from coloured soaps interferes with the phenolphthalein endpoint.

F.2 Apparatus

Ordinary laboratory apparatus such as the following are to be used:

- Conical flask of 250 ml capacity, with a conical ground glass joint.
- Reflux condenser, water-cooled, with a conical ground glass joint at the bottom.

F.3 Reagents

Only the reagents of recognized analytical grade and distilled water to be used.

- Ethanol, 95 % (V/V) solution, free from carbon dioxide.
(Reflux this solution for 5 min, cool to ambient temperature and neutralize with the ethanolic potassium hydroxide solution in the presence of 4 drops of the phenolphthalein solution per 200 ml of ethanol).
- Sulphuric acid
- Potassium hydroxide, approximately 0.1 N ethanolic standard volumetric solution.
- Phenolphthalein, solution of 1 g per 100 ml in 95 % (v/v) ethanol.

F.4 Procedure

F.4.1 Test portion

Weigh, to the nearest 0.001 g, about 5 g of the laboratory sample into the conical flask.

F.4.2 Determination

- Add 100 ml of the ethanol solution to the test portion.
- Fit the conical flask to the reflux condenser. Gently heat until the soap is completely dissolved, then add exactly 3 ml of the sulphuric acid solution and boil gently for at least 10 min. Allow to cool to ambient temperature.
- Titrate with the ethanolic potassium hydroxide solution in the presence of the indicator.
- Carry out two determinations on the same sample.

F.5 Expression of Results

F.5.1 Method of calculation and formulae

The total free alkali content, expressed as a percentage by mass of sodium hydroxide (NaOH) in the case of sodium soaps, is given by the formula:

$$0.040 \times \frac{(\square_0 \square_0 - \square_1 \square_1)}{\square} \times 100$$

The total free alkali content, expressed as a percentage by mass of potassium hydroxide (KOH) in the case of potassium soaps, is given by the formula:

$$0.056 \times \frac{(\square_0 \square_0 - \square_1 \square_1)}{\square} \times 100$$

where

- m is the mass, in grams, of the test portion;
- V_0 is the volume, in millilitres, of the sulphuric acid solution used in the determination;
- V_1 is the volume, in millilitres, of the potassium hydroxide solution used in the titration;
- T_0 is the exact normality of the sulphuric acid solution;
- T_1 is the exact normality of the potassium hydroxide solution.

The total free alkali may also be expressed in milliequivalents per gram by the formula:

$$\frac{(\square_0 \square_0 - \square_1 \square_1)}{\square}$$

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

F.5.2 Reproducibility

The results obtained on the same sample in two different laboratories, expressed either as sodium hydroxide (NaOH) or as potassium hydroxide (KOH), shall not differ by more than 0.05 %.

F.6 Test Report

The following specifics shall be included in the test report:

- a) all details required for the sample's complete identification;
- b) the method used as reference;
- c) the results and the method of expression;
- d) the test conditions;
- e) any operational details that are optional or not specified, as well as any events that may have had an impact on the results.

8 Bibliography

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