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Bathing bars — Specification

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Introduction

This standard has been developed to reflect the trends in the latest technological developments while ensuring the performance and safety of the product to the consumer. This will in particular facilitate use of acceptable non-soapy surfactants and structuring aids, some of which are locally available. These will often substitute oils and fats, most of which are imported and expensive, and hence their use can help to boost the country's economy.

The important in-use criteria for bathing bars are safety, cleaning and economy. With regard to safety it is important to guard against the removal of the beneficial skin lipids by the product and over-cleaning resulting in defatting of the skin. This is ensured in the present standard by allowing only surfactants that have a history of safe use in personal washing products. A list of the proposed surfactants is contained in Annex A. Other surface-active agents may be added to the list at a future revision if there is adequate evidence of their safety.

The lather requirement is intended to ensure the presence of adequate level of surfactants in the bathing bar for cleaning. The test is designed to take into account dissolution of the bar and subsequent lather generation. The mush test covers the aspect of economy. Limits are prescribed for caustic alkali and alkalinity of alcohol insolubles to restrict the use of alkaline ingredients.

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Bathing bars — Specification

1 Scope

This African Standard prescribes the requirements and methods of test for bathing bars. The standard applies to bathing bars supplied in the form of cakes and produced from vegetable or animal oils or fats, fatty acids, or from a blend of all or part of these materials, with or without the addition of rosins or non-soapy/synthetic surfactants.

2 Normative references

The following referenced documents are indispensable for the application 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.

ARS 1462, *Powder detergents — Biodegradability*

ISO 4315, *Surface active agents — Determination of alkalinity — Titrimetric method*

ISO 456, *Surface active agents — Analysis of soaps — Determination of free caustic alkali*

ISO 457, *Soaps — Determination of chloride content — Titrimetric method*

ISO 672, *Soaps — Determination of moisture and volatile matter content — Oven method*

ISO 673, *Soaps — Determination of content of ethanol-insoluble matter*

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

ISO 685, *Analysis of soaps — Determination of total alkali content and total fatty matter content*

ISO 862, *Surface active agents — Vocabulary*

ISO 4314, *Surface active agents — Determination of free alkalinity or free acidity — Titrimetric method*

ISO 4315, *Surface active agents — Determination of alkalinity — Titrimetric method*

ISO 4316, *Surface active agents — Determination of pH of aqueous solutions — Potentiometric method*

ISO 6839, *Anionic surface-active agents — Determination of solubility in water*

Regulation (EC) No 1223/2009 of the European Parliament and of the Council of 30 November 2009 on Cosmetic Products

3 Definitions

For the purpose of this standard the definitions given in ISO 862 and the following shall apply.

3.1
bathing bars
the bathing bar is a product containing soap of fatty acids with or without synthetic surface-active agents as active ingredients and which could be used for bathing purposes in soft and hard water

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4 Requirements

4.1 General requirements

4.1.1 Bathing bars shall be firm. The of the product colour shall generally be uniform, except for multi-coloured products.

4.1.2 The product shall not be injurious to health if used in a manner and purpose meant for their use or under reasonably foreseen conditions.

NOTE Bathing bar soaps are for personal washing purposes.

4.2 Specific requirements

Bathing bars shall also comply with the requirements given in Table 1 when tested against the methods described.

Table 1 — Specific requirements

SL. No	Characteristic	Requirement	Test method
1.	Lather, mL, minimum.	200	Annex B
2.	Mush (loss in mass due to mushing on a wet surface) (g/30cm^2, maximum).	10	Annex C
3.	Freedom from grittiness	To pass the test	Annex D
4.	Total alkalinity (as NaOH) % by mass, maximum.	1.0	ISO 685
5.	Free caustic alkali (as NaOH), % by mass, maximum.	0.3	ISO 456
6*	Synthetic surface-active agents (if used)	Pass the biodegradability test	ARS 1462

4.3 Ingredients

4.3.1 The bathing bar shall have one or more of the following surfactants;

a) There is no restriction on the use of soap of fatty acids, fatty acid ester sulphonates, fatty alkalonamide, fatty alcohol ethoxylates, sarcosinates, taurides, fatty isothionates, alpha olefin sulphonates, alcohol sulphates and amphoterics such as betaines and fatty alcohol ethoxy sulphate, linear alkyl benzene sulphonates (LAS) and akyl poly glycosides (APG) and fatty alcohol sulphosuccinate and fatty alkanol amido sulphosuccinate or any other safe surfactant

b) Rosins, as % of total fatty matter, shall not exceed 2 % when tested against annex F

4.3.2 In addition to the surfactants and perfume, the bathing bar may contain other ingredients such as electrolytes, bar structuring and processing aids, colouring matter, permitted antioxidants, preservatives, permissible germicides, super fatting agents, humectants and such additional substances as are declared on the label.

4.3.3 All other ingredients except water shall be declared on the label.

For guidance, a list of bar structuring and processing aids is given in Annex A. All these materials shall be non-injurious to skin in use with the bathing bar.

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4.3.4 The bathing bar shall not contain any materials prohibited by regulations such as Regulation (EC) No 1223.

5 Packaging and labelling

5.1 Packaging

Each bar may be unwrapped or may be wrapped. Unwrapped or wrapped bars may be packed in suitable boxes, packages or cartons to avoid contamination or damage during transportation.

5.2 Labelling

5.2.1 Wrapped bars

Each bathing bar shall be marked legibly and indelibly with the following particulars:

a) the words 'Bathing Bar';

b) Name of the product

c) manufacturer's name and physical address and trade mark if any;

NOTE The name, physical address of the distributor/supplier may be added as required.

d) nominal weight of each bar or cake at the time of packaging;

e) number of bars or cakes contained in the package;

f) batch number or code number;

g) all ingredients;

h) date of manufacture and best before date;

i) country of origin, if different from manufacturer address

5.2.2 Unwrapped bathing bars

Each unwrapped bathing bar shall be marked legibly and indelibly with the following particulars:

a) name of product

b) Nominal weight of the bar

5.2.3 Cartons/boxes/ other packages

a) the words 'Bathing Bar';

b) manufacturer's name and physical address and trade mark if any;

c) batch number or code number;

d) number of bars or cakes contained in the package;

e) date of manufacture and best before date;

f) country of origin, if different from manufacturer address

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6 Sampling

6.1 Scale of sampling

The consignment of bathing bar cakes to be sampled shall be divided into lots, each lot containing about 20 cakes from the same source and of the same declared weight and brand.

From each lot, a sample of 9 cakes shall be drawn at random. The cakes of soap thus chosen shall be kept, until tested, in a clean closed container, marked so as to identify the lot from which they have been chosen.

The cakes shall be tested separately for each of the tests mentioned in Table 1.

This scale of sampling will ensure that there are a further 9 samples remaining with which to repeat any of the tests that give results not conforming to the criteria for conformity, or in the event of a dispute over the results.

7 Test methods

7.1 Preparation for test samples

The cakes of a given type (brand) of bathing bar shall be packed into a clean airtight container until tested.

The requirements given in Table1 shall be tested according to Annexes B to D. Testing should commence as soon as possible after sampling.

7.2 Criteria for conformity

Criteria for conformity to tests are included in the Annexes B to D describing the individual test methodologies.

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Annex A
(normative)

Permitted structuring and processing aids

Following is the list of structuring and processing aids used generally in bathing bars.

A.1 Starch and derivatives

A.2 Cellulose and derivatives

A.3 Propylene glycol

A.4 Sorbitol

A.5 Glycerol

A.6 Dextrin

A.7 Kaolin

A.8 Talc

A.9 Bentonite

A.10 Calcite

A.11 Sodium lactate

A.12 Soda ash

A.13 Vegetable/animal oil fatty acids and salts

A.14 Phosphates

A.15 Sodium chloride

A.16 Sodium sulphate

A.17 Dolomite

A.18 Fatty alcohol

A.19 Rosin and rosin salts

A.20 Fatty acid ethanolamide

A.21 Diethylene glycol monostearate

A.22 Paraffin

A.23 Polyoxyethylene glycol

A.24 Glycerol monostearates

A.25 Silicates

A.26 Any other internationally accepted builder cleared by the relevant authorities

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Annex B
(normative)

Test for lather volume

B.1 Principle

The speed of a domestic kitchen blender is adjusted so that when operated for 60 seconds it will deliver 600 ± 100 mL of lather (foam) from a 1 % solution of sodium lauryl sulphate in 300 ppm calcium hardness water at 25 ºC.

Soap samples are grated to a uniform noodle of about 2-3 mm diameter (domestic cheese grater). Five grams of the noodles are mixed in the domestic blender for 60 seconds with 100 mL of 300ppm calcium hardness water at 25 ºC.

The volume of the lather generated from the soap is normalised to a volume of 600 mL delivered from the sodium lauryl sulphate solution tested in the same blender at the same speed.

B.2 Equipment/reagents

A variable speed kitchen blender with a container/ jar of at least 1000 mL capacity.

B.2.1 Kitchen cheese grater

B.2.2 1000 mL measuring cylinder

B.2.3 100 mL measuring cylinder

B.2.4 Thermometer 0 – 100 ºC

B.2.5 Water containing 300ppm calcium hardness

To make 2 litres of a 300 ppm hard water dissolve 1.66 g of analytical grade anhydrous calcium chloride in demineralised water and make to 2 litres.

1 % SLS solution in water containing 300 ppm calcium water hardness.

Dissolve 20g of analytical grade sodium lauryl sulphate (SLS) in water and make to 2 litres with the 300 ppm calcium hardness water.

Note especially that this SLS solution should be prepared fresh every day.

B.3 Sample preparation

B.3.1 The requirement is for duplicate lather measurements from three bars of each product, and for six replicate lather measurements from the freshly made solution of sodium lauryl sulphate.

B.3.2 A sample of the bathing bar is grated to give at least 20 grams of 2 – 3 mm diameter noodles.

Cut-off the end third of the bar before grating the freshly exposed face of the remaining two thirds. This will limit the proportion of over dried surface soap that is converted into noodles.

Immediately place the grated noodles onto a coarse sieve e.g. aperture 850 microns or similar and shake gently to allow any fine and over dried soap to pass through.

Small diameter noodles can lose water very quickly and the gratings should be transferred very quickly from the sieve into a labelled container that can be closed to prevent such water loss.

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Duplicate lather determinations on an individual sample of a bathing bar will only require 2 x 5 g samples of the noodles. However, it is always wise to take at least 20 grams to allow for any repeat tests.

B.4 Procedure for lather determination

NOTE All tests are to be performed with solutions and equipment at 25 ± 2 ^{0}C.

B.4.1 Lather volume from standard sodium lauryl sulphate (SLS) solution

B.4.1.1 Pour 100 mL of 1 % SLS solution into the blender, cover the blender jar.

B.4.1.2 Run the blender for exactly 60 seconds.

B.4.1.3 Invert the blender jar over the 1000 mL measuring cylinder and allow to drain. If necessary a long plastic spatula or ruler can be used to scoop the residual foam from the jar into the cylinder.

B.4.1.4 If necessary level the top of the foam with the long plastic spatula or ruler and measure the lather (foam) volume (S).

NOTE The total volume of material in the cylinder is taken as the lather volume. There is no allowance for any liquid that may separate from the actual foam.

B.4.1.5 Measure the temperature of the foam in the measuring cylinder.

B.4.1.6 If the lather volume is not 600 ± 100 mL adjust the blender speed and repeat the test until a volume in this range is achieved.

B.4.2 Lather volume from a bathing bar sample

B.4.2.1 Prepare 20g of 2 – 3mm diameter noodles from each sample to be tested, as described in Section B.3.

B.4.2.2 Quickly weigh 5 ± 0.05 g of the grated sample.

B.4.2.3 Add the sample to the blender jar which should contain 100 mL of water with 300 ppm Ca water hardness.

B.4.2.4 Run the blender for exactly 60 seconds.

B.4.2.5 Invert the blender over the 1000 mL measuring cylinder and allow to drain. If necessary a long plastic spatula or ruler can be used to scoop the residual foam from the jar into the cylinder.

B.4.2.6 If necessary level the top of the foam with the long plastic spatula or ruler and measure the lather (foam) volume (*X)

NOTE The total volume of material in the cylinder is taken as the lather volume. There is no allowance for any liquid that may separate from the actual foam.

B.4.2.7 Measure the temperature of the foam in the measuring cylinder. The target temperature is 25 ± 2 °C. See 4.4.

B.4.3 Calculation

The normalized value (X) of lather for the soap sample will be calculated as follows:

$$X = {}^{*}X \times \frac{600ml}{S}$$

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B.4.4 Criteria for conformity

The test for lather will give six test results for a given product, obtained from duplicate determinations of the parameter from three samples of the product.

The mean and the range of the corresponding test result shall be calculated as:

$$\text{Mean } (\bar{x}) = \frac{\text{The sum of test results}}{\text{The number of results}}$$

Range (R) = The difference between the highest and the lowest value obtained for the test results.

The sampled lot of products shall be declared as conforming to requirements for this standard if the expression $\bar{x}$ - 0.6R is greater than or equal to the minimum value in Table 1.

B.4.5 Notes for guidance

B.4.5.1 The kitchen blender

The requirement is for a kitchen food blender operated at a speed, which will deliver a lather volume of 600 ± 100 mL from a standard solution of sodium lauryl sulphate. Commercial blenders may have adequate integral speed controls. Any blender may be operated through an external electronic speed controller or a variable output transformer. Normally the lowest speed will be required from blenders having integral speed control.

B.4.5.2 Preparing the noodles

The hole size in the grater should be 2 – 3 mm diameter, but is not critical. The size of holes in a grater can be checked quickly by using a magnifier with a built-in measurement scale, or very simply by inserting the shafts of twist drills into the holes.

A cheese grater with a rotary drum action may be used.

Older soaps may create some dust/ broken noodles during the grating operation. This fine material will have been removed by sieving the noodles immediately after preparation.

Prepared noodles can be kept is a wide top, screw cap, jar or a sealed plastic bag. A sealed plastic bag is less efficient at retaining moisture but is adequate for 1 day. Ideally the samples should be kept in a refrigerator until it is re-equilibrated back to room temperature for about 1 hour before testing.

B.4.5.3 Temperature control

All tests should be carried out at 25 ±2 °C and all apparatus and solutions should be at this temperature.

The temperature of the foam is measured in the measuring cylinder, immediately after the foam volume measurement.

It is not always easy to achieve this level of temperature control. To achieve a foam temperature in the specified range it may be necessary to start with the 300 ppm hard water supply adjusted to a slightly higher or slightly lower temperature than ambient.

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Annex C
(normative)

Evaluation of the mushing properties of a bathing bar

C.1 Principle

A test piece of defined size is cut from the sample bar to remove harder outer layers. The test piece is preconditioned by giving 18 x 180 degree twists under running water at 25 °C or in a bowl of water at 25 °C. The bar is left for six hours on a piece of fabric that has been wetted and drained of excess water. During the six hours the soap/ cloth are covered to prevent drying. At the end of the test period mush is removed from the test piece face in contact with the cloth. Weight loss from the test piece is expressed as mush per 30 cm^2 of original surface area in contact with the cloth.

C.2 Equipment

C.2.1 For sample preparation

— Coarse kitchen cheese grater
— Sharp thin blade knife or carpenters plane
— Callipers or ruler to ensure the sample dimensions

C.2.1 Other equipment/ materials for the test

Plastic or non-corrodible trays which are suitable sized for the test piece. Plastic soap dishes 7 x 11 x 2 cm are quite suitable.

Cotton cloth pieces cut and folded to fit as a triple layer inside the trays. Normal, flat weave, cotton sheeting as used for bed sheets will be quite suitable.

C.3 Bar preparation

C.3.1 Three (3) individual bars of a type should be tested. A test piece is cut from each bar. The test piece should if possible have a working face (to be applied to the fabric) of 6 ±1 cm x 4 ±1 cm.

All bars in a set must be cut to have the same face size. If the smallest of the range of bars to be tested at a given time 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 axis of the test piece (6 ±1 cm) should be from a direction parallel to the longest axis of the original bar sample.

The working face should be a fresh surface from the interior of the bar sample. The face opposite the working face should be identified by making a small hole with a sharp object. This enables the working face to be identified after the preconditioning step.

C.3.2 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-bladed knife or carpenters plane. If a plane is used, it is better to move the bar over the plane blade.

C.4 Test procedure

For each test piece

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C.4.1 The tray plus triple thickness of cloth is filled with demineralised water. The tray is then held vertically to drain the water from the cloth. The vertical position is maintained until water ceases to run from the dish in a continuous stream i.e. starts to drip.

C.4.2 The area of the working face of the test piece is measured (A).

C.4.3 The working face of the bar is placed onto the damp fabric and then the tray plus soap are covered e.g. with a sealed plastic bag, to prevent water loss.

C.4.4 The covered test piece and holder are maintained at 25 °C for 6 hours.

C.4.5 The mushed soap test piece is removed from the tray and is weighed (W1).

C.4.6 Mush is removed from the working face of the soap test piece by scraping with the edge of a blunt sided spatula or plastic ruler.

C.4.7 The test piece is reweighed (W2) and the amount of mush removed is calculated as in C.5. The mush is expressed as grams per 30cm^2 of original test piece surface area.

NOTE The procedure for weighing the bar and removing the mush will take some minutes. During that time the remaining soaps will continue to form mush. While this time is not critical for a set of three test pieces from a given product, if more than one product is under test it is advised to stagger the start of the test for the second product. This will give adequate time to complete work on the first set before the 6-hour storage time of the subsequent set is completed.

C.5 Calculation

Weight of mush (grams) = W = W1 – W2

Surface area of bar (cm^2) = A = (width x breadth)

Mush = $\frac{W \times 30}{A}$ grams per 30 cm^2

C.6 Criteria for conformity

The test is done with three (3) separate samples of each product type, and the mean value from three samples is quoted $\bar{X}$. The range of values (R) is quoted 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 $(\bar{X} + 0.6R)$ is less than the maximum value given in Table 1.

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Annex D
(normative)

Determination of grittiness in a bathing bar

D.1 Procedure

Either

Hold the soap tablet 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). Remove the bar from the water and continue to gently rub the two opposite bar faces for a further 30 seconds (15 seconds per face).

Allow the used bar to dry in the open for 4 hours and examine the surface.

A set of 3 samples will be tested for each product.

NOTE 1 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).

NOTE 2 If using a bowl rather than running water use fresh water after testing every set of 3 samples.

D.2 Criteria for conformity

The performance criteria are:

During manipulation under running water the washing bar will not have a visibly rough surface and will feel smooth to the touch.

No gritty particles will be observed on the surface of the dried bar 4 hours after the washing test.

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Annex E
(normative)

Identification of rosin content of fatty matter in soaps

E.1 Scope

This Annex specifies the Halphen-Grimaldi and Liebermann-Storch method for the identification of rosin in fatty matter.

E.2 Reagents

NOTE Use only analytical grade reagents and distilled water.

E.2.1 Acetic anhydride.

E.2.2 Sulphuric acid, density 1.53 g/mL. Cautiously add 97 mL of sulphuric acid (density 1.84 g/mL) to 100 mL of water.

E.2.3 Solution A. Dissolve 10 g of phenol in 27 mL of chloroform.

E.2.4 Solution B. Dissolve 10 mL of bromine in 40 mL of chloroform.

E.3 Procedure

E.3.1 Halphen-Grimaldi method

E.3.1.1 Place one or two drops of the fatty matter, reserved from the determination of fatty matter (ISO 685), in a porcelain basin and add approximately 2 mL of the solution A to dissolve it. Wet the walls of the basin by tilting and turning it and let it stand for a few seconds so that the walls of the basin are covered with a very thin film of the solution.

E.3.1.2 Hold the neck of the flask containing the solution B in such a position that the bromine vapour diffuses into the porcelain dish and comes into contact with the walls. In the presence of rosin a blueish-grey to purple colour develops immediately.

E.3.1.3 Carry out a comparison test with a sample of fatty matter to which 2 % (*m/m*) of rosin has been added.

NOTE Should the colour reaction in E.3.1.2 be masked by other colours, conduct a second qualitative test, using the Liebermann-Storch method.

E.3.2 Liebermann-Storch method

E.3.2.1 Place 1 g - 2 g of the fatty matter in a test tube, add 5 mL - 10 mL of the acetic anhydride, and heat the mixture in a boiling water bath for approximately 3 min. Cool to room temperature and pour 1 mL - 2 mL of the solution into a white porcelain basin.

E.3.2.2 Allow one or two drops of the sulphuric acid to run down the side of the basin. If rosin is present, a violet colour immediately develops where the acid is in contact with the solution. This colour then turns brown on standing.

E.3.2.3 Carry out a comparison test with a sample of fatty matter to which 2 % (*m/m*) of rosin has been added.

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Annex F
(normative)

Determination of rosin content of fatty matter in soaps

F.1 Scope

This Annex specifies a method for the determination of the rosin content of fatty matter in soaps.

NOTE 1 Before proceeding with the quantitative determination of rosin, first establish its presence by a qualitative test (see Annex E).

NOTE 2 The following method is not accurate for rosin concentrations below a mass fraction of 5 %.

F.2 Reagents

NOTE Use only analytical grade reagents and distilled water.

F.2.1 Diethyl ether

Free from peroxides.

F.2.2 Sodium chloride

F.2.3 Sodium sulfate

Anhydrous.

F.2.4 Hydrochloric acid solution

Dilute one volume of concentrated hydrochloric acid (density 1.16 g/mL) with two volumes of distilled water.

F.2.5 Sodium hydroxide solution

10 % mass fraction aqueous solution.

F.2.6 Naphthalene-2-sulfonic acid solution

Dissolve 40 g of naphthalene-2-sulfonic acid in 1 L of absolute methanol.

F.2.7 Sodium chloride solution, saturated

Shake an excess of sodium chloride with water at ambient temperature until no more dissolves. Keep the solution over solid sodium chloride.

F.2.8 Sodium chloride solution, 10 %

Dissolve 10 g of sodium chloride in 100 mL of water.

F.2.9 Standard ethanolic potassium hydroxide solution, 0.2 N

F.2.9.1 Preparation

Purify 95 % (by volume fraction) ethanol by boiling 1.5 L of it over 20 g of potassium hydroxide for 1 h under reflux. Distil, discarding the first 50 mL of the distillate and stopping the distillation when approximately 1.3 L have been distilled. Dissolve 12 g of potassium hydroxide in 1 L of the purified ethanol, allow the solution to stand for approximately one week, and then decant the clear supernatant liquid from any potassium carbonate that has precipitated.

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F.2.9.2 Standardization

Accurately weigh out approximately 1 g of potassium hydrogen phthalate (previously dried at 110 °C ± 5 °C for 3 h) into a 250 mL Erlenmeyer flask. Add approximately 100 mL of carbon dioxide-free distilled water and three drops to five drops of the phenolphthalein indicator, and swirl gently until the solid has dissolved. Titrate the solution with the standard ethanolic potassium hydroxide solution, until a permanent pink colour is formed.

F.2.9.3 Calculation

Normality of the standard ethanolic potassium hydroxide solution (*N*):

$$N = \frac{A \times 4.897}{B}$$

where

A is the mass of the potassium hydrogen phthalate, in grams;

B is the volume of the standard ethanolic potassium hydroxide solution used for the titration, in millilitres.

F.2.10 Methyl orange indicator

Dissolve 0.2 g of methyl orange in 100 mL of carbon dioxide-free water.

F.2.11 Phenolphthalein indicator

Dissolve 0.5 g of phenolphthalein in 100 mL of freshly boiled, 95 % (by volume fraction) ethanol.

F.3 Procedure

F.3.1 Preparation of fatty matter

F.3.1.1 Carbolic soaps

F.3.1.1.1 Weigh out accurately into a 600 mL beaker such quantity of the test sample as contains approximately 40 g of fatty matter and dissolve it in approximately 400 mL of hot water, to which 40 mL of the sodium hydroxide solution has been added.

F.3.1.1.2 Salt out the soap by adding sufficient sodium chloride to the hot solution to saturate it (at ambient temperature) with sodium chloride. Filter the soap quantitatively, and allow it to drain.

F.3.1.1.3 Dissolve the drained soap in approximately 400 mL of hot water, and repeat the salting out and filtering procedure.

F.3.1.1.4 Wash the soap thoroughly with the saturated sodium chloride solution, and proceed in accordance with F.3.1.2.2 to F.3.1.2.7.

F.3.1.1.5 Reserve the combined filtrates and washings for the determination of carbolic acids.

F.3.1.2 Other soaps

F.3.1.2.1 Weigh accurately into a 600 mL beaker such quantity of the test sample as contains approximately 40 g of fatty matter.

F.3.1.2.2 Dissolve the soap in approximately 400 mL of hot distilled water, cool the solution, and slowly add an excess of the hydrochloric acid solution. Cover the beaker with a watch-glass and heat

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the contents until the fatty matter separates into a clear layer, but do not allow the temperature to exceed 60 °C.

F.3.1.2.3 In the cases of liquid and gel soaps, acidify the soap as it is.

F.3.1.2.4 Cool to approximately 25 °C and transfer the contents of the beaker to a separating funnel. Rinse the watch-glass and the beaker with portions of the diethyl ether totalling 100 mL and add them to the separating funnel. Shake the mixture in the separating funnel vigorously for 1 min, and let it stand until the two phases have separated.

F.3.1.2.5 Draw off and discard the aqueous layer and wash the ether extract with 50 mL portions of the sodium chloride solution until the last washing is neutral to the methyl orange indicator.

F.3.1.2.6 Filter the washed ether extract into a 250 mL beaker through a filter paper containing approximately 5 g of anhydrous sodium sulfate, and wash the separating funnel and the filter with small portions of the diethyl ether.

F.3.1.2.7 Evaporate the ether extract plus washings on a warm water bath. When the residue is just dry, heat it rapidly to 130 °C and immediately place the beaker in a desiccator to cool.

NOTE F.3.1.2.2 to F.3.1.2.7 should be completed in the shortest possible period of time, so as to prevent oxidation of the fatty acids.

F.3.2 Determination of rosin content

F.3.2.1 Weigh accurately into a 150 mL flask with a ground-glass joint approximately 2 g of the prepared fatty matter (F.3.1.2.7) and reserve the rest in the case of soap powders for the fatty matter titre determination.

F.3.2.2 Add exactly 25 mL of the naphthalene-2-sulfonic acid solution to the flask and two or three glass beads, and boil under reflux for 30 min.

F.3.2.3 Carry out a blank test at the same time, using exactly 25 mL of the naphthalene-2-sulfonic acid solution only.

F.3.2.4 Cool the contents of both flasks (sample and blank) to ambient temperature, add 0.5 mL of the phenolphthalein indicator to each flask, and immediately titrate to the end point with the standard ethanolic potassium hydroxide solution.

F.4 Calculation

Rosin content of fatty matter, as a mass fraction percentage (R):

$$R = \frac{(A - B) \times N \times 34.6}{C} - 1.0$$

where

A is the volume of the standard ethanolic potassium hydroxide solution used for the titration of the sample solution, in millilitres;

B is the volume of the standard ethanolic potassium hydroxide solution used for the titration of the blank solution, in millilitres;

N is the normality of the standard ethanolic potassium hydroxide solution;

C is the mass of the fatty matter taken, in grams.

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