



DRAFT EAST AFRICAN STANDARD

Bathing soap — Specification — Part 1: Solid

EAST AFRICAN COMMUNITY

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 074, *Surface active agents*.

This second edition cancels and replaces the first edition (EAS 186-1:2021), which has been technically revised.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

EAS 186-1 consists of the following parts, under the general title *Bathing soap — Specification*:

- *Part 1: Solid*
- *Part 2: Liquid*

Bathing soap — Specification — Part 1: Solid

1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for solid bathing soap.

This standard does not apply to carboric soap or specialty soaps such as, transparent soap, floating soap, liquid soap, beauty soap or sea-water soap.

The standard does not apply to products, for which therapeutic claims are made.

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.

EAS 377 (all parts), *Cosmetics and cosmetics products*

EAS 794, *Determination of the microbial inhibition of cosmetic soap bars and liquid hand and body washes — Test method*

EAS 814 *Determination of biodegradability of surfactants — Test method*

ISO 457, *Analysis of soap — Determination of chloride content — Titrimetric method*

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

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

ISO 673, *Analysis of soap — Determination of ethanol insoluble matter*

ISO 862, *Surface active agents — Vocabulary*

ISO 1067, *Analysis of soap — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 862 and the following apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1 soap
product formed by the saponification or neutralization of fats, oils, waxes, rosins or their acids with organic or inorganic bases

3.2 bathing soap
soap which is intended for use in bathing

3.3 toilet soap
bathing soap containing fatty acids and does not contain synthetic surface-active agents

Note 1 to entry: it may contain antibacterial agents.

3.4 bathing bar
bathing soap containing fatty acids with or without synthetic surface-active agents

Note 1 to entry: It may contain antibacterial agents.

3.5 colouring matter
any safe dye that may be used to colour toilet soap

3.6 free caustic alkali
uncombined caustic alkali present in a soap

3.7 total fatty matter
water-insoluble or ether soluble fatty matter under the specified conditions of test

3.8 antibacterial activity
ability of a bathing soap to inhibit the growth or destroy bacteria and other harmful microorganisms (germs). This activity is commonly found in products designed to prevent infections and promote hygiene, including antibacterial agents, antiseptic agents, and anti-germ formulations.

4 Requirements

4.1 General requirements

4.1.1 Bathing soap shall be classified as either

- a) toilet soap; or
- b) bathing bar.

4.1.2 Bathing soap shall not cause skin irritation and shall have good lathering and cleansing properties.

4.1.3 Perfumes and colouring matter may be added.

4.1.4 Bathing soap shall be firm and of uniform texture and colour and shall be free from objectionable (disagreeable) odour.

4.1.5 Bathing soap shall remain hard, smooth and not crumble when tested in accordance with Annex A.

- 4.1.6 All the substances used in bathing soap shall comply with the relevant parts of EAS 377.
- 4.1.7 Antibacterial bathing soap shall contain permitted antibacterial agents.
- 4.1.8 Active ingredients used shall be biodegradable when tested in accordance with EAS 814.

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4.2 Specific requirements

4.2.1 Toilet soap shall comply with the specific requirements given in Table 1 when tested in accordance with the test methods specified therein.

Table 1 — Specific requirements for toilet soap

S/No.	Characteristic	Requirement	Test method
i.	Total fatty matter content, % m/m, min.	76.0	ISO 685
ii.	Content of matter insoluble in ethanol ^a , % m/m, max.	2.5	ISO 673
iii.	Free caustic alkali content as NaOH ^a , % m/m, max.	0.1	ISO 456
iv.	Free fatty acids content as oleic acid, % m/m, max.	0.3	Annex B
v.	Chloride content as NaCl, % m/m, max.	0.8	ISO 457
vi.	Unsaponified fatty matter content, % m/m, max.	0.5	ISO 1067
vii.	Antibacterial activity ^{b, c}	To pass the test	EAS 794

^a Solid toilet soap is liable to lose moisture on storage. The results of analysis in respect to free caustic alkali, free carbonated alkali and matter insoluble in alcohol should be recalculated in relation to the minimum specified total fatty matter by means of the following equation:

$$\text{Corrected result} = \frac{\text{actual result} \times \text{minimum specified total fatty matter}}{\text{actual total fatty matter}}$$

The corrected results should be used to determine whether the product is up to standard.

^b ASTM E1174 and EN 1276 factoring log reduction may be used as alternative test methods to determine antibacterial activity. When these test methods are used, the log reduction shall not be less than 99%.

^c This test only applies to products with antibacterial claims.

4.2.2 Bathing bars shall comply with the specific requirements given in Table 2 when tested in accordance with the test methods specified therein.

Table 2 — Specific requirements for bathing bars

S/No.	Characteristic	Requirement	Test method
i.	Fatty matter (product shall comply with either of the following): i) Product without synthetic surfactant, total fatty matter, % m/m, min. ii) Product with synthetic surfactant, % m/m, min. <ul style="list-style-type: none">• Total fatty matter• Synthetic surface active agent present iii) Total fatty matter from dissolved actives, %, min.	50 40 4 8	ISO 685 Annex C Annex D
ii.	Lather, mL, min.	200	Annex E
iii.	Mush (loss in mass due to mushing on a wet surface), g/30 cm ² , max.	10	Annex F
iv.	Freedom from grittiness	To pass the test	Annex G
v.	Total alkalinity (as NaOH) % m/m, max.	1.0	ISO 685
vi.	Rosins, as % of total fatty matter, max.	2	Annex H
vii.	Antibacterial activity ^{a, b}	To pass the test	EAS 794
<p>a This test only applies to products with antibacterial claims.</p> <p>b ASTM E1174 and EN 1276 factoring log reduction may be used as alternative test methods to determine antibacterial activity. When these test methods are used, the log reduction shall not be less than 99%.</p>			

5 Packaging

Bathing soap shall be packaged in such a manner as to protect it from damage and excessive loss or gain of moisture.

6 Labelling

Each package shall be legibly and indelibly labelled in English and/or any other official language (French, Kiswahili, etc.) used in the importing East African Partner State with the following information:

- a) name of the product as “Toilet soap” or “Bathing bar”;
- b) indication of antibacterial activity (where applicable);

- c) manufacturer's name and physical address. The name, physical address of the distributor/supplier and trade mark may be added;
- d) batch number or lot number;
- e) net content;
- f) country of origin;
- g) antibacterial agent(s) used (for antibacterial soap/bar);
- h) list of ingredients in descending order of quantity; and
- i) date of manufacture and best before date.

7 Sampling

Sampling shall be done in accordance with Annex J.

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Annex A
(normative)

Texture and stability test

When immersed in 1 L of distilled water for 1 h at 25 °C – 30 °C, toilet soap shall not show signs of disintegration, and when dried at room temperature for 25 h thereafter, it shall not crumble, crack or break.

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Annex B (normative)

Determination of free fatty acids content as oleic acid

B.1 Barium chloride method

B.1.1 Apparatus

- B.1.1.1 Conical flask, 500 mL
- B.1.1.2 Reflux condenser, to fit the flask

B.1.2 Reagents

- B.1.2.1 Distilled water or water, of at least equivalent purity, free from carbon dioxide
- B.1.2.2 Ethanol, 95 % (v/v), free from carbon dioxide and distilled over potassium hydroxide
- B.1.2.3 Ethanol, aqueous solution, 60 % (v/v), neutralized

Mix 125 mL ethanol (B.1.2.2), 75 mL distilled water (B.1.2.1) and 1 mL of indicator (B.1.2.7). Neutralize to a violet colour with an aqueous solution of potassium or sodium hydroxide (B.1.2.4). Heat under reflux for 10 min. Allow to cool to room temperature. Add 1 mL of indicator (B.1.2.7). Neutralize with the hydrochloric acid solution (B.1.2.6) until the violet colour disappears.

- B.1.2.4 Potassium or sodium hydroxide, 0.1 N aqueous solution
- B.1.2.5 Barium chloride, aqueous solution

Dissolve 10 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in 90 mL of distilled water (B.1.2.1). Neutralize with potassium or sodium hydroxide (B.1.2.4) in the presence of indicator (B.1.2.7) until a violet colour appears.

- B.1.2.6 Hydrochloric acid, 0.1 N aqueous solution, accurately standardized
- B.1.2.7 Indicator mixture, phenolphthalein-thymol blue, ethanolic solution

Dissolve 1 g of phenolphthalein and 0.5 g of thymol blue in 100 mL of hot ethanol (B.1.2.2). Filter.

B.2 Procedure

B.2.1 Weigh, to the nearest 0.01 g, about 5 g of toilet soap into a conical flask (B.1.1.1). Add 200 mL of ethanol (B.1.2.3). Connect the reflux condenser (B.1.1.2). Bring to the boil for 10 min. Add an excess of 0.1 N ethanolic potassium hydroxide solution of exactly known normality.

B.2.2 Add to this boiling solution 20 mL of barium chloride solution (B.1.2.5) in small portions shaking thoroughly. Cool with running water to room temperature.

B.2.3 Add 1 mL of the indicator mixture (B.1.2.7). Titrate immediately with the hydrochloric acid solution (B.1.2.6) until the violet colour disappears.

B.3 Expression of results

The free fatty acids as oleic acid, expressed as a percentage by mass, of potassium hydroxide, shall be calculated as follows:

$$\frac{5.6 \times V \times T}{m}$$

where

V is the volume, in millilitres, of hydrochloric acid solution (B.1.2.6) used;

T is the exact normality of the hydrochloric acid solution (B.1.2.6) used;

m is the mass, in grams, of the test portion.

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Annex C (normative)

Determination of total fatty matter and synthetic surface active agents

C.1 Outline of the method

The test method of analysis for total fatty matter (TFM) given in ISO 685 pertains to the analysis of fatty matter for soaps. In the context of a wider definition of TFM relevant to bathing bar, which may contain synthetic actives in addition, it becomes necessary to quantify the fatty chain(s) linked with synthetic surface active agents and include their contribution to the TFM.

Fatty chain anionic surfactants or soaps undergo hydrolysis when refluxed with mineral acids to give corresponding fatty matter which can be extracted by petroleum ether. Non-fatty anionics like LAS and AOS do not undergo hydrolysis and therefore do not interfere in the analysis.

In this method, total synthetic surface active agents and TFM are estimated independently by gravimetry.

C.2 Principle

Since several synthetic surfactants are permitted to be used in bathing bar formulations, there is wide variation in the molecular weight range of these surfactants. Estimation of individual surfactants is not necessary from the analytical point. The analytical strategy involves the extraction of all surfactants (soaps, non-ionics, anionics and amphoteric) in 85 % alcohol. An aliquot of 85 % alcohol soluble matter is extracted with petroleum ether to remove hydrophobic constituents (perfume, unsaponifiable matter etc.) and the 85 % alcohol fraction is evaporated to dryness. The residue is extracted with 1:1 tetrahydro furan : petroleum ether mixture and filtered. The filtrate is evaporated and the residue weighed as synthetic surfactant. Another aliquot of the 85 % alcohol soluble portion is refluxed with 2 N sulphuric acid and extracted with petroleum ether. The residue obtained after evaporation of petroleum ether is the TFM comprising of free fatty acids, fatty matter from soap, as well as fatty matter associated with other surfactants.

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Figure C.1 illustrates the analytical strategy of total fatty matter and synthetic surface active agents.

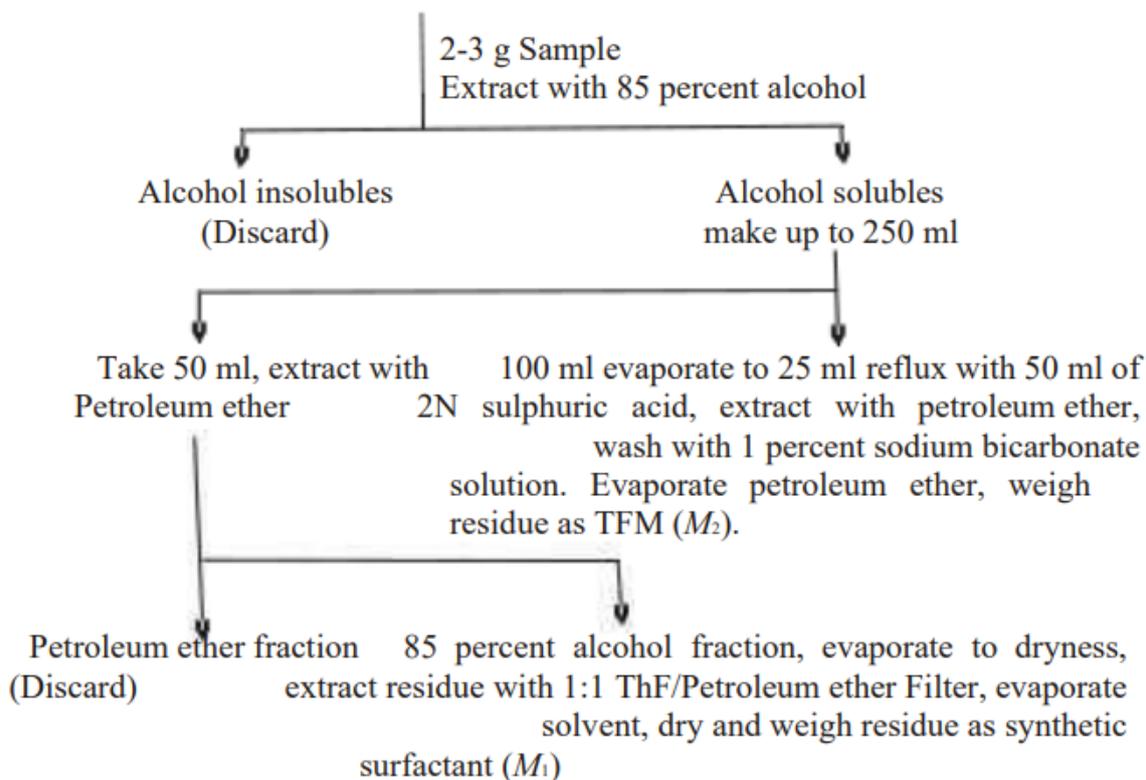


Figure C.1 Analytical strategy for total synthetic surfactant and total fatty matter

C.3 Reagents

C.3.1 85 % alcohol (v/v), ethyl alcohol or rectified spirit

Mix 85 parts by volume of ethyl alcohol with 15 parts by volume of distilled water, mix well and neutralise with 0.1 N NaOH to phenolphthalein end point (if rectified spirit is used, dilution should be accordingly made so as to obtain 85 % v/v concentration).

C.3.2. Sulphuric acid, approximately 2 N

Add 6 mL of concentrated sulphuric acid (sp Gr 1.84) to 50 mL of cold distilled water (10 °C approximate) with stirring and dilute with distilled water to 100 mL in a standard volumetric flask and mix well.

CAUTION — Wear safety glasses and rubber gloves while handling concentrated sulphuric acid.

C.3.3 Methyl orange indicator solution, 0.1 % w/v, aqueous solution

Dissolve 0.1 g of the indicator in 100 mL distilled water.

C.3.4 Phenolphthalein indicator, 1.0 % w/v, alcoholic solution

Dissolve 1.00 g ± 0.05 g of the indicator in 100 mL of ethyl alcohol or rectified spirit and mix well.

C.3.5 Sodium hydroxide, approximately 0.1 N

Dissolve 2 g (AR grade) sodium hydroxide in distilled water and make up to 500 mL with distilled water in a standard volumetric flask and mix well.

C.3.6 Petroleum ether, 40 °C – 60 °C boiling range (laboratory reagent grade)

C.3.7 Aqueous ethyl alcohol, (or rectified spirit), approximately 50 % v/v

Mix equal volumes of ethyl alcohol or rectified spirit and distilled water.

C.3.8 Sodium bicarbonate solution, approximately 1 % w/v

Dissolve 2.5 g (AR grade) sodium bicarbonate in 125 mL of distilled water and make up to 250 mL in a volumetric flask.

C.3.9 Sodium hydroxide solution, approximately 1 % (w/v) in 85 % alcohol

Dissolve 2.5 g of sodium hydroxide in 85 % alcohol and dilute to 250 mL in a volumetric flask.

C.3.10 Acetone, AR Grade

C.3.11 Tetrahydrofuran (THF), AR Grade

C.3.12 Solvent mixture, 1:1 mixture of tetrahydrofuran and petroleum ether (v/v)

C.3.13 Sulphuric acid solution, 50 % (w/w)

Take 50 mL of ice chilled distilled water in a 250-mL conical flask. Slowly add 50 g concentrated sulphuric acid with continuous stirring. Care must be taken to avoid the spurting.

C.4 Apparatus/glassware

C.4.1 Beakers, 250-mL, 500-mL and 1 000-mL capacity

C.4.2 Measuring cylinder, 10-mL, 25-mL and 50-mL capacity

C.4.3 Standard volumetric flask, 10-mL, 250-mL, 500-mL and 1 000-mL capacity

C.4.4 Separating funnel, 500-mL capacity

C.4.5 Round bottom flask, with ground joint, 250-mL capacity

C.4.6 Air condenser, length 1 m

C.4.7 Conical flask, 500-mL capacity

C.4.8 Funnels

C.4.9 Glass rods

C.4.10 Flash rotary evaporator/distillation set-up

C.4.11 Whatman Filter Paper No. 541

C.4.12 Air oven

C.4.13 Steam bath

C.5 Procedure

C.5.1 Preparation of sample

Cut the cake/bar into two halves. Grate approximately 2 g to 10 g from the centre of each half of the cake/bar. Homogenize the gratings from the two halves and use this for further analysis.

C.5.2 85 % alcohol extraction

C.5.2.1 Weigh accurately about 2.0 g to 3.0 g of sample into a 250-mL beaker (*M*).

C.5.2.2 Add 125 mL of 85 % alcohol, heat to about 60 °C with continuous stirring with a glass rod on a steam bath for approximately 5 min inside a fuming cupboard. Transfer into a 250-mL volumetric flask using a funnel by decantation.

C.5.2.3 Add again 50 mL of 85 % alcohol into the same beaker (C.5.2.1). Break the lumps, if any, with the glass rod and repeat the operation as in C.5.2.2.

C.5.2.4 Repeat the extraction twice with 25 mL of 85 % alcohol as in C.5.2.2.

C.5.2.5 Collect all the 85 % alcohol extracts in the 250-mL volumetric flask.

C.5.2.6 Make up the volume to 250-mL with 85 % alcohol and mix well.

C.5.3 Estimation of synthetic surfactants

C.5.3.1 Transfer 50 mL of solution from C.5.2.6 into a 250-mL beaker.

C.5.3.2 Add 0.5 mL of phenolphthalein indicator and add 1.0 % sodium hydroxide solution (drop by drop) till the colour of the solution changes to pink. Add 1.0 mL excess beyond this point.

C.5.3.3 Transfer quantitatively into a 500-mL separating funnel, extract 3 times with 50 mL aliquots of petroleum ether. Preserve the lower alcohol/water portion.

C.5.3.4 Wash the combined petroleum ether extracts with 20 mL aliquots of 85 % alcohol solution 2 times. Collect the alcohol washings and transfer quantitatively to the alcohol extract from C.5.3.3 and discard the petroleum ether extract.

C.5.3.5 Evaporate the alcoholic solution from C.5.3.4 to dryness.

C.5.3.6 Add 20 mL of acetone to the residue. Heat on water bath with continuous stirring to dryness. Ensure complete removal of water (see Note 1).

NOTE 1 This is a very important step, any moisture present will form a stable gel with the solvent mixture, thus making extraction impossible.

NOTE 2 Use only Whatman filter paper No. 541 (Porosity of 20 µm - 25 µm).

C.5.3.7 Add 50 mL of solvent mixture (C.3.12) to the residue, heat to boiling (60 °C – 70 °C) on a water bath with constant stirring for 2 min to 3 min and filter through Whatman filter paper No. 541. Collect the filtrate into a tared 250-mL beaker/round bottom flask.

C.5.3.8 Repeat the extraction two more times as described in C.5.3.7, collecting the filtrate into the same beaker. Wash the filter paper 3 times with 20 mL aliquot of the solvent mixture, collect the washings into the same beaker/round bottom flask.

C.5.3.9 Evaporate the solvent on a water bath (inside a fume hood) if washings collected into the same beaker or alternately distil off solvent using a rotary evaporator if washings are collected into the round bottom flask.

C.5.3.10 Dry the beaker/round bottom flask containing the residue in an air oven at 105 °C for 1 h. Cool to room temperature inside a desiccator and weigh to constant weight (*M*₁).

C.5.4 Estimation of total fatty matter (TFM)

C.5.4.1 Transfer quantitatively 100 mL of extract from C.5.2.6 into a 250-mL beaker, evaporate on steam bath to about 25 mL and transfer quantitatively into a 250-mL ground jointed round bottom flask.

C.5.4.2 Add 50 mL of 2 N sulphuric acid and reflux on steam bath for 2 h after fitting with an air condenser.

C.5.4.3 Remove condenser. Add 8 g sodium chloride and continue refluxing for further 1 h and 30 min.

C.5.4.4 Cool to room temperature. Add 20 mL of rectified spirit and transfer quantitatively to a 500-mL separating funnel.

C.5.4.5 Add 75 mL of petroleum ether, place stopper and shake vigorously for 1 min. Release pressure slowly, remove stopper and allow the two immiscible phases to separate.

CAUTION — Use safety glasses during extraction.

C.5.4.6 Draw off the lower aqueous/alcoholic layer into another separating funnel. Add 75 mL petroleum ether, replace stopper and shake vigorously for 1 min. Release pressure slowly, remove stopper and allow the two immiscible phases to separate. Draw off the lower alcoholic layer into the 250-mL beaker and transfer the petroleum ether extract to the first separating funnel.

C.5.4.7 Transfer the alcoholic layer to the second separating funnel and repeat the extraction with 75 mL of petroleum ether as given in C.5.4.5.

C.5.4.8 Wash the petroleum ether extract with 30 mL aliquots of 1 % (w/v) sodium bicarbonate solution, 5 to 6 times, till it is free from mineral acidity (test with methyl orange indicator).

C.5.4.9 Transfer petroleum ether extract quantitatively to a tared 250-mL round bottom flask and evaporate petroleum ether on water bath by distillation, inside a fume cupboard.

C.5.4.10 Dry the contents of the flask in an air oven at a temperature of 90 °C for 10 min. Remove it from oven and blow with air for 15 s. Allow the flask to cool and weigh. Return the flask to the oven at 90 °C for another 10 min. Cool and reweigh. Repeat the procedure until constant weight, M_2 (difference between weighings is less than 0.005 g).

C.6 Calculation

C.6.1 Total actives

Total actives, expressed as percent, shall be calculated as follows:

$$\% \text{Total actives}, T_1 = \frac{M_1 \times 250 \times 100}{M \times 50}$$

where

M_1 is the mass, in grams, of residue (C.5.3.10);

M is the mass, in grams, of sample taken for test (C.5.2.1).

C.6.2 Total fatty matter

Total fatty matter, expressed as percent by mass, shall be calculated as follows:

$$\%TFM, T_2 = \frac{M_2 \times 250}{M}$$

where

M_1 is the mass, in grams, of sample taken for test (C.5.2.1);

M_2 is the mass, in grams, of residue (C.5.4.10).

C.6.3 Percentage synthetics

Synthetic surfactants, expressed as percent by mass, shall be calculated as follows:

$$\% \text{ Synthetic Surfactants} = T_1 - T_2$$

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Annex D

(normative)

Determination of fatty matter from dissolved actives

D.1 General

The method below describes quantitative estimation of fatty matter content from dissolved actives from bathing bar.

D.2 Procedure

D.2.1 250 g of 1.0 % (w/w) alkaline soap prepared by stirring approx. 2.5 g (W) of finely grated soap sample and 1.0 g of sodium hydroxide pellets (minimum 98 % purity) with 247.5 g of distilled water. Stir using magnetic stirrer for 4 h at 27 °C (care must be taken so that no frothing and overflow takes place).

D.2.2 Filter the soap solution through fluted (flower) Whatman filter No.1 with a circle diameter of minimum 15 cm (preferably 24 cm).

NOTE — Ensure that the filtration funnel is selected in such a way that entire area of filter paper is supported and that there is no tear in the filter paper while filtrating.

D.2.3 Measure turbidity of the filtrate soap solution at 30 °C by digital nephelometric turbidity meter. 100 gm aliquot of clear soap filtrate is taken for analysis and turbidity should be < 100 NTU.

NOTE Since all the submicron soap particles would not be completely filtered, the filtrate would be slightly turbid.

D.2.4 Take 100 g of the filtrate. Add 19.6 g of 50 % (w/w) conc. Sulphuric acid (see C.3.13) to this filtrate with continuous stirring and reflux the contents for 2 h after fitting with an air condenser (45.7 cm height, min.).

D.2.5 Remove the condenser. Add 8 g sodium chloride and continue refluxing for further 1 h and 30 min.

D.2.6 Cool to room temperature. Add 20 mL of rectified spirit and transfer quantitatively to a 500-mL separating funnel.

D.2.7 Add 75 mL of petroleum ether, place stopper and shake vigorously for 1 min. Release pressure slowly, remove stopper and allow the two immiscible phases to separate.

CAUTION — Use safety glasses during extraction.

D.2.8 Draw off the lower aqueous/alcoholic layer into another separating funnel. Add 75 mL petroleum ether, replace stopper and shake vigorously for 1 min. Release pressure slowly, remove stopper and allow the two immiscible phases to separate. Draw off the lower alcoholic layer into the 250-mL beaker and transfer the petroleum ether extract to the first separating funnel.

D.2.9 Transfer the alcoholic layer to the second separating funnel and repeat the extraction with 75 mL of petroleum ether as given in D.2.8.

D.2.10 Wash the petroleum ether extract with 30 mL aliquots of 1 % w/v sodium bicarbonate solution till it is free from mineral acidity. Two washings are sufficient (test with methyl orange indicator).

D.2.11 Transfer petroleum ether extract quantitatively to a tared 250-mL round bottom flask and evaporate petroleum ether on water bath by distillation inside a fume cupboard.

D.2.12 Dry the contents of the flask in an air oven at a temperature of 90 °C for 10 min. Remove it from oven and blow with air for 15 s. Allow the flask to cool and weigh. Return the flask to the oven at 90 °C for another

10 min. Cool and reweigh. Repeat the procedure until constant weight (difference between weighings less than 0.005 g) Z.

D.2.13 Calculate the fatty matter from dissolved actives, using the calculation given in D.3.

D.3 Calculation

Fatty matter from dissolved actives in filtrate, expressed as percent of soap bar weight, shall be calculated as follows:

$$\text{Fatty matter from dissolved actives, percent} = \frac{2.5 \times Z \times 100}{W}$$

W is the weight, in grams, of soap in the total soap bar;

Z is the weight, in grams, of TFM in 100 g of filtrate.

Therefore, weight of fatty matter from dissolved actives in filtrate = $2.5 \times Z$

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Annex E (normative)

Test for lather volume of bathing bar

E.1 General

Strict attention shall be paid to all details of the procedure in order to ensure concordant results. Particular care should be taken to invert the cylinder exactly as described.

E.2 Outline of the method

A suspension of the material in standard hard water is taken in a graduated cylinder and given 12 inversions under prescribed conditions. The volume of the foam formed is observed after keeping the cylinder for 5 min.

E.3 Reagents

E.3.1 Calcium chloride ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$), AR

E.3.2 Magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), AR

E.3.3 Distilled water

E.4 Apparatus

E.4.2 Glass beaker, 100-mL

E.4.3 Thermometer, of range $0\text{ }^\circ\text{C}$ – $110\text{ }^\circ\text{C}$

E.5 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 The standard hard water has a hardness of approximately 50 mg/kg calculated as calcium carbonate.

E.6 Sample preparation

Cut away the outer edges of bathing bar using a knife.

Using a stand-up type of grater, grate up to 10 g – 15 g of the bathing bar into small chips.

E.7 Procedure

E.7.1 Weigh 1 g of the grated chips bathing 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 to stand for 30 min. The operation is carried out to disperse the bathing bar.

E.7.2 Stir the contents of the beaker with a glass rod and transfer the slurry to a 500-mL graduated cylinder ensuring that not more than 2 mL foam is produced. 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.

E.7.3 Adjust the contents in the cylinder to 100 mL by adding sufficient standard hard water. Bring the contents of the cylinder to 30 °C. Stir the contents of the cylinder with a glass rod or thermometer to ensure a uniform suspension.

E.7.4 As soon as the temperature of the contents of the cylinder reach 30 °C, stopper the cylinder and give it 12 complete inversions, each inversion comprising movements in a vertical plane, upside down and vice versa. After the 12 inversions, let the cylinder stand for 5 min. Take the following readings as shown in Figure E.1:

- a) foam plus water (V_1 mL).
- b) water only (V_2 mL).

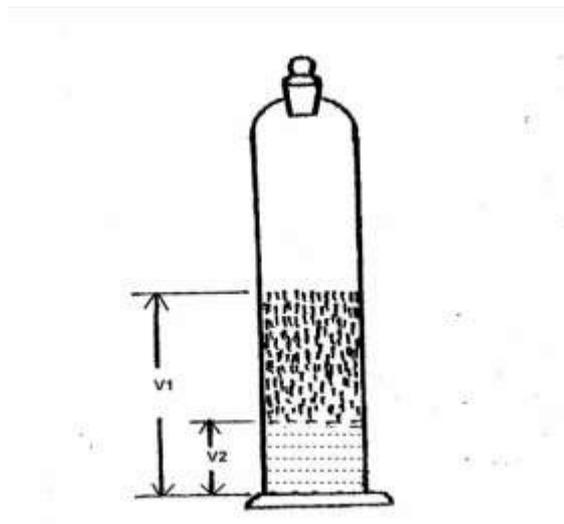


Figure E.1 — Measurement of foam

E.8 Calculation

Lather volume, expressed in millilitres, shall be calculated as follows:

$$\text{Lather volume} = V_1 - V_2$$

where

V_1 is the volume, in millilitres, of foam + water;

V_2 is the volume, in millilitres of water only.

Annex F (normative)

Evaluation of the mashing properties of a bathing bar

F.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 cm² of original surface area in contact with the cloth.

F.2 Apparatus

F.2.1 For sample preparation

- F.2.1.1 **Coarse kitchen cheese grater**
- F.2.1.2 **Sharp thin blade knife or carpenter's plane**
- F.2.1.3 **Callipers or ruler**, to ensure the sample dimensions

F.2.2 Other equipment/materials for the test

F.2.2.1 Plastic or non-corrodible trays, suitably sized for the test piece. Plastic soap dishes 7 cm x 11 cm x 2 cm are quite suitable.

F.2.2.2 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.

F.3 Bar preparation

F.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 cm ± 1 cm x 4 cm ± 1 cm.

F.3.2 All bars in a set shall 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.

F.3.3 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.

F.3.4 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.

F.3.5 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 carpenter's plane. If a plane is used, it is better to move the bar over the plane blade.

F.4 Test procedure

F.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.

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

F.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.

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

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

F.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.

F.4.7 The test piece is reweighed (W2) and the amount of mush removed is calculated as in F.5. The mush is expressed as grams per 30 cm² 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.

F.4.8 Repeat F.4.1 to F.4.7 for each test piece.

F.5 Calculation

The weight of mush, expressed in grams, shall be calculated as follows:

$$W = W1 - W2$$

Surface area of bar (cm²) A = (width x breadth)

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

F.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 (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 $X + 0.6R$ is less than the maximum value given in Table 2.

Annex G (normative)

Determination of grittiness in bathing bar

G.1 Procedure

Either

Hold the bathing 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 L of water at 30 °C and gently rub two opposite bar faces with the palm of one hand for 30 s (15 s per bar face). Remove the bar from the water and continue to gently rub the two opposite bar faces for a further 30 s (15 s per face).

Allow the used bar to dry in the open for 4 h 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 for 15 min 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.

G.2 Criteria for conformity

During manipulation under running water the bathing 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 h after the washing test.

Annex H (normative)

Determination of rosins

H.1 General

H.1.1 Only Colophonium (commercial rosins) shall be considered as rosin for the purpose of this standard. The mean equivalent weight of the rosin acid is taken as 346.

H.1.2 The method described in this test gives results approximately one percent higher than the actual amount of rosin present. As a result, the percentage of actual rosin acids present is one less than the percentage of rosin acids found experimentally and hence minus one in the formula.

H.2 Reagents

H.2.1 Dilute sulphuric acid, 30 % (w/v) obtained by cautiously adding 16 mL of sulphuric acid, specific gravity 1.84 mL to 70 mL of water

H.2.2 Beta-naphthalene sulphuric acid solution (C₁₀H₇SO₃H), obtained by dissolving 40 g of the chemical in 1 L of chemically pure, absolute methyl alcohol.

H.2.3 Standard alcoholic potassium hydroxide solution, approximately 0.2 N in 95 % (v/v) ethyl alcohol or in rectified spirit, accurately standardized. Since alcohol is volatile, frequent restandardization is necessary.

H.2.4 Phenolphthalein indicator, obtained by dissolving 1 g in 100 ml of 95 % (v/v) ethyl alcohol

H.3 Procedure

H.3.1 Dissolve 10 g to 50 g of the sample in about 500 mL of hot water. Add 10 mL to 50 mL of the dilute sulphuric acid to split the bar, keep in Steam-bath until the fatty matter separates as a clear layer and siphon off the lower aqueous acid layer. Add 300 mL of hot water, boil gently for a few minutes and siphon off the aqueous layer. Repeat the washing with hot water several times until the wash liquor is free of mineral acids. Complete the acidification and washing in as a short period as possible, keeping the beaker covered to prevent oxidation of the acids. Remove the last traces of water from the fatty acids through one or two thickness of filter paper and dry at 105 °C ± 2 °C for 45 min to 50 min.

H.3.2 Weigh accurately 2 g of the mixture of fatty and rosin acids into an esterification flask and add 25 mL of beta-naphthalene sulphonic acid solution. Boil gently under a reflux condenser for 30 min, adding a few glass beads to ensure smooth boiling. Cool the contents of the flask and titrate immediately with standard alcoholic potassium hydroxide solution, using 0.5 mL of phenolphthalein indicator. The end point is reached when the pink colour persists for 30 s.

H.3.3 Conduct simultaneously a blank determination with 25 mL of the etherifying agent alone.

H.4 Calculation

H.4.1 Uncorrected rosin

Rosin acids in fatty matter (uncorrected), expressed as percent by mass, shall be calculated as follows:

$$\frac{34.6(S - B)N}{M}$$

where

S is the volume, in millilitres, of standard alcoholic potassium hydroxide solution required for the material;

B is the volume, in millilitres, of standard alcoholic potassium hydroxide solution required for the blank;

N is the normality of alcoholic potassium hydroxide;

M is the mass, in grams, of the material taken for the test.

H.4.2 Corrected rosin

The method described above gives results approximately one percent higher than the actual amount of rosin present. As a result, the actual percentage of rosin acids present is one less than the percentage of rosin acids found experimentally.

Rosin in fatty acids, percent by mass, corrected = (Rosin in fatty acids, percent by mass, uncorrected – 1.0)

NOTE: When the quantity of rosin, expressed as percent by mass, is less than 5 in the bars, the results by this method are not so accurate as with bars containing higher rosin content. This method is also liable to give erroneous results with certain types of carbolic soaps containing high boiling tar acids and with other germicidal soaps, for example, soaps containing hexachlorophene.

H.4.3 Liebermann-Storch test

H.4.3.1 General

In all cases where the rosin content is found to be less than 5 %, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test.

H.4.3.2 Reagents

H.4.3.2.1 Acetic anhydride, pure

H.4.3.2.2 Dilute sulphuric acid, relative density 1.53

H.4.3.3 Procedure

Transfer 1 mL to 2 mL of the sample of fatty acids to a test-tube, treat with 5 mL to 10 mL of acetic anhydride and warm on a steam-bath. After cooling, pour 1 mL to 2 mL into a white porcelain dish and allow a drop or two of sulphuric acid to run down the side of the vessel. If rosin is present, a fugitive violet colouration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. Check the test with a sample of fatty acids to which a small amount of rosin has been added.

Annex J (normative)

Sampling

J.1 Procedure

J.1.1 In a single consignment, all packages (cartons) containing bathing 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 J.1.

Table J.1 — Scale of sampling

Number of packages (cartons) in the lot <i>N</i>	Number of packages (cartons) to be selected <i>n</i>	Number of samples
4 to 15	3	3
16 to 40	4	4
41 to 65	5	2
66 to 110	7	2
111 and above	10	1

J.1.2 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 the integral part of N/n .

J.1.3 From each package thus selected, draw at random an equal number of cakes so as to obtain a total mass of at least 2 kg.

J.2 Preparation of test samples

J.2.1 Composite sample

Weigh each cake separately (including any material that may have adhered to the wrapper), and calculate the average mass. Cut each of the remaining cakes 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.

J.2.2 Samples for testing

Immediately after preparation of composite sample (J.2.1), take at one time all test samples required for the tests in 4.2. Weigh out the test sample required for determination of free alkali or acid content, and use it immediately.

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- [1] EAS 186-1:2021: *Bathing soap — Specification — Part 1: Solid*
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