DEAS 186-1: 2020

ICS 71.100.40

DRAFT EAST AFRICAN STANDARD

Bathing soap — Specification – Part 1: Solid

EAST AFRICAN COMMUNITY

Fourth Edition 2020

Copyright notice

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) and other deliverables. 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 and other deliverables 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*

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.

This fourth edition cancels and replaces the third edition (EAS 186:2013), which has been technically revised. It incorporated the following three East African Standards which upon its approval they will be withdrawn

EAS 766-1:2013 *Antibacterial toilet soap – Specification – Part 1: Solid;*

EAS 877:2017 *Bathing bar – Specification;* and

EAS 878:2018 *Antibacterial bathing bar - Specification*

EAS 186 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. It does not apply to carbolic soap or specialty soaps such as, transparent soap, floating soap, liquid soap or sea-water soap.

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 377-1, *Cosmetics and cosmetics products — Part 1: List of substances prohibited in cosmetic products*

EAS 377-2, *Cosmetics and cosmetics products — Part 2: List of substances which cosmetic products must not contain except subject to restrictions laid down*

EAS 377-3, *Cosmetics and cosmetics products — Part 3: List of colourants allowed in cosmetic products*

EAS 377-4, *Cosmetics and cosmetics products — Part 4: List of preservatives allowed in cosmetic products*

EAS 377-5, *Cosmetics and cosmetics products — Part 5: Use of UV filters in cosmetic 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 684, *Analysis of soap — Determination of total free 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 saponfiable matter*

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

3 Terms and definitions

For the purposes of this East African Standard the terms and definitions given in ISO 862 and the following apply.

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

3.4
Antibacterial toilet soap

toilet soap containing antibacterial agent(s)

3.5
Bathing bar

bathing soap containing fatty acids and/or synthetic surface-active agents.

3.6
Antibacterial bathing soap

bathing bar containing antibacterial agent(s)

3.7
saponification
chemical reaction in which a fat is converted into a soap by the action of a base

3.8
colouring matter
any safe dyestuff that may be used to colour toilet soap

3.9
free caustic alkali
uncombined caustic alkali present in a soap

3.10
total fatty matter
water-insoluble or ether soluble fatty matter under the specified conditions of test

3.11
total free alkali
sum of the free caustic alkali and the free carbonate alkali contents

4 Requirements

4.1 General requirements

4.1.1 Bathing soap shall include the following;

a) Toilet soap

b) Antibacterial toilet soap

c) Bathing bar

d) Antibacterial bathing bar

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

4.1.2 Perfumes and colouring matter may be added.

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

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

4.1.5 All the substances used in bathing soap shall comply with the requirements of all parts of EAS 377.

4.1.6 The antibacterial bathing soap shall pass the antibacterial activity test when determined by the method given in EAS 794.

4.1.7 The active ingredients used shall be biodegradable when tested according to EAS 814

4.2 Specific requirements

Bathing soap shall comply with the specific requirements specified in Table 1 for toilet soap and Table 2 for bathing bar

Table 1 — Specific requirements for toilet soap

S/No.	Characteristic	Requirements		Method of test
		Toilet soap	Antibacterial toilet soap	
i.	Total fatty matter content, % by mass, min	76.0	76.0	ISO 685
ii.	Content of matter insoluble in ethanol, % by mass, max	2.0	2.5	ISO 673
iii.	Free caustic alkali content as NaOH, % by mass, max	0.1	0.1	ISO 456
iv.	Free fatty acids content as oleic acid, % by mass, max	0.3	0.3	Annex B
v.	Chlorides content as NaCl, % by mass, max	0.8	0.8	ISO 457
vi.	Unsaponified fatty matter content, % by mass, max	0.5	0.5	ISO 1067
vii.	Antibacterial agent TricIosan (TCN) and Trichlorocarbanilide (TCC), % by mass, max.	n/a	1.0 either singly or in combination	Annex C
viii.	Chloroaniline content, ppm, max	n/a	10.0	Annex D
ix.	Antibacterial activity	n/a	To pass the test	EAS 794.

NOTE 1 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.

NOTE 2 Trichlorocarbanilide (TCC) is not heat stable and decomposes into chloro anilines on prolonged heating above 60 °C. If TCC is used in soap, the manufacturer should take care that such soap is not subjected to temperature above 60°C during the entire manufacturing process or during storage.

Table 2 – Specific requirements for bathing bar

Sl No.	Characteristic	Requirement		Test method
		bathing bar	Antibacterial bathing bar	
i.	Total fatty matter, % by mass, min	50.0	50.0	ISO 685
ii.	Lather, mL, min	200	200	Annex E
iii.	Mush (loss in mass due mushing on a wet surface), g/30 cm^2, max	10.0	10.0	Annex F
iv.	Freedom from grittiness	To pass a test	To pass a test	Annex G
v.	Total alkalinity (as NaOH) % by mass, max	1.0	1.0	ISO 4315
vi.	Rosins, as % of total fatty matter, max	2	2	Annex H
vii.	Antibacterial agent TricIosan (TCN) and Trichlorocarbanilide (TCC), % by mass, max.	n/a	1.0 either singly or in combination	Annex C
viii.	Chloroaniline content, ppm, max	n/a	10.0	Annex D
ix.	Antibacterial activity	n/a	To pass the test	EAS 794.

NOTE 1 Solid bathing bar 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.

NOTE 2 Trichlorocarbanilide (TCC) is not heat stable and decomposes into chloro anilines on prolonged heating above 60 °C. If TCC is used in soap, the manufacturer should take care that such soap is not subjected to temperature above 60°C during the entire manufacturing process or during storage.

5 Packaging and labelling

5.1 Packaging

Toilet soap shall be so wrapped as to protect them from damage and excessive loss or gain of moisture.

5.2 Labelling

Each package shall be legibly and indelibly labelled either in English, Kiswahili or French or combination or any other language as agreed between the manufacturer and supplier with the following information:

a) name of product as “Toilet soap, Antibacterial toilet soap, Bathing bar or Antibacterial bathing bar”;

b) manufacturer’s name and physical address;

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

c) batch number or lot number;

d) 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.

6 Sampling

Sampling shall be done in accordance to Annex I.

7 Criteria for conformity

The lot shall be deemed to comply with the requirements of this standard if, after inspection and testing, the requirements of Clause 4 and 5 are satisfied.

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.

Annex B
(normative)

Determination of free fatty acids content as oleic acid

B.1 Barium chloride method

B.1.1 Apparatus

B.1.1.1 500 mL conical flask.

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 per cent (V/V), free from carbon dioxide and distilled over potassium hydroxide.

B.1.2.3 Ethanol, aqueous solution 60 per cent (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 ref lux for 10 min. Allow to cool to room temperature. Add 1 mL of indicator (B.1.2.7). Neutralize with the hydrochloric acid solution (3.5.6) 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 ($BaCl_2$. $2H_20$) 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

Weigh, to the nearest 0.01 g, about 5 g of soft 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.

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.

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 (m/m) of potassium hydroxide, is given by the formula

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

Where

V is the number of ml 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 g, of the test portion.

Annex C
(normative)

Determination of Trichlorocarbanilide (TCC) and Triclosan (TCN) in soaps by HPLC

C.1 Principle

TCC and TCN are antibacterial agents, which are separated from other components in soap by normal phase or reverse phase liquid chromatography, detected spectrophotometrically and quantified by comparison with standard TCC and TCN. The method can estimate as low as 1 ppm of the above compounds:

Procedures for both normal and reverse HPLC has been described and provide the option to use either method whichever is available to the users. Both methods are comparable.

C.2 Normal phase HPLC

C.2.1 Reagents

C.2.1.1 **Iso-octane**, HPLC grade

C.2.1.2 **Iso-propanol (2-propanol)**, HPLC grade

C.2.1.3 **Hexane**, HPLC grade

C.2.1.4 **Standard TCC**, 99 % pure

C.2.1.5 **Standard TCN**, 99 % pure

C.2.2 Apparatus

C.2.2.1 **High Performance Liquid Chromatograph** consisting of a pump, a sample injector of fixed volume with UV detector having variable wavelengths and a recorder

C.2.2.2 **Standard volumetric flasks**

C.2.2.3 **Pipettes**

C.2.2.4 **Magnetic stirrer**

C.2.2.5 **Millipore filter apparatus** with 0.5 micron filter

C.2.2.6 **Column,** comprising:

a) **Silica column**, stainless steel 25 cm x 0.46 cm packed with Normal phase-silica 5 micron (Lichrosorb Si -60); or

b) **Cyano column**, stainless steel 25 cm x 0.40 cm packed with (Lichrospher 100) cyano 5 micron.

NOTE Either, of the above columns can be used depending on the availability.

C.2.2.7 **Mobile phase:**

a) for silica column, transfer 20 mL of iso-propanol into a 500-mL volumetric flask and make up to mark with iso-octane and mix well. Assemble millipore filter apparatus and filter the solvent system prior to use; and

b) for cyano column, transfer 50 mL of HPLC grade iso-propanol (2-propanol) into a 500-mL volumetric flask, fill up to the mark with hexane and mix well. Assemble millipore filter apparatus and filter the solvent system prior to use.

C.2.2.8 **HPLC conditions** which include the following:

a) Detector wavelength flow rate: 280 nm;

b) Flow rate: 0.5 mL/min;

c) Injection volume: 20 µL;

d) Retention time;

e) Silica column:

- TCN - 7.5 min; and
- TCC - 19.2 min;

f) Cyano column:

- TCN - 4.0 min; and
- TCC - 7.5 min.

C.2.3 Procedure

C.2.3.1 Standard preparation (see note under B.3.4)

Weigh accurately 25 mg of Triclosan (TCN) and 25 mg of TCC into a 100-mL volumetric flask and make up to volume with the mobile phase and mix well. Pipette 1.0 mL of this solution in a 50 mL volumetric flask and dilute with mobile phase. Final concentration of TCC and TCN is 250 µg/50 mL (5.0 ppm).

C.2.3.2 Sample preparation

Weigh accurately 1 g of homogenized sample into a 100-mL standard flask, and dilute to the mark with mobile phase. Pipette 10 mL of the supernatant liquid to a 50-mL volumetric flask, dilute with mobile phase, to the mark, and filter through 0.45 µm filter.

C.2.3.3 Chromatography

Equilibrate the column, maintained at a temperature of 30 °C, with the mobile phase with a flow rate of 0.5 mL /min for iso-octane - iso-propanol mobile phase and 1.0 mL/min for Hexane - iso-propanol mobile phase for 30 min. Set the wavelength at 280 nm. Inject 20 µL of standard solution and then sample solutions.

Measure area of the peaks of respective retention time for standard and sample.

C.2.4 Calculation

TCN shall be expressed as follows:

$$\text{TCN, percent by mass} = \frac{\text{Area of sample for TCN x Concentration of standard TCN}}{\text{Area of standard TCN x Concentration of sample}} \text{ x } 100$$

$$\text{TCC, percent by mass} = \frac{\text{Area of sample for TCC x Concentration of standard TCC}}{\text{Area of standard TCC x Concentration of sample}} \text{ x } 100$$

C.3 Reverse phase

C.3.1 Reagents

C.3.1.1 Methanol, HPLC grade

C.3.1.2 Sodium Dihydrogen Phosphate Monohydrate, chemical grade

C.3.1.3 Standard TCC

C.3.1.4 Standard TCN (TCS)

C.3.2 Apparatus

C.3.2.1 Column

C.3.2.1.1 Octyldimethylsilyl (C-DB)

C.3.2.1.2 Supercosil LC-8-DB, 15 cm x 4.6 mm. 5 micron

C.3.2.2 Mobile phase

MeOH/0.01 M Phosphate buffer 62:38 v/v

0.01 M Phosphate buffer: Dissolve 1.38 g sodium dihydrogen phosphate monohydrate in 1 000 mL of distilled water. Prepare to pH 3.0 by 10 % phosphate solutions.

C.3.3 Procedure

C.3.3.1 Standard preparation (see Note under C.3.4)

C.3.3.1.1 Weigh accurately about 90 mg of TCN. Dissolve in methanol and make up to 1 000 mL volumetric flask with methanol.

C.3.3.1.2 Weigh about 110 mg of TCC, dissolve well with methanol, and make up the volume to 1 000 mL.

C.3.3.1.3 Accurately pipette 10 mL of the solution prepared in C.3.3.1.1 into the volumetric flask containing TCC (C.3.3.1.2). And make up to the volume with methanol. Then accurately pipette 5 ml of the solution into a 50-mL volumetric flask. Make up to the volume with methanol. Filter this standard solution through 0.45 µm filter.

C.3.3.2 Sample preparation

Weigh accurately about 1.0 g of product, dissolve in methanol and make up to 100 mL in a volumetric flask with methanol. Filter this sample solution through 0.45 µm filter.

C.3.3.3 HPLC conditions

The HPLC conditions include the following:

a) Detector wavelength: 280 nm;

b) Column temperature: 35 ºC;

c) Flow rate: 1.0 mL/min; and

d) Injection volume: 10 µL.

Prepare the standard solution and the sample solution at the same time. Inject the standard solution three times and calculate the average of each ingredients peak count. Inject 10 µg the sample solution and determine each ingredients percentage by the calculation shown.

C.3.4 Calculations

The TCN and TCC shall be expressed as follows:

$$\text{TCN, percent by mass} = \frac{(M_s \times A_r \times F)}{(A_s \times M_t \times 100)}$$

$$\text{TCC, percent by mass} = \frac{(M_s \times A_r \times F)}{(A_s \times M_t \times 100)}$$

where

A_r is the peak area of the test sample;

A_s is the averaged peak area of the standard;

F is the purity, expressed as percent, of the standard;

M_s is the mass, in grams, of the standard; and

M_t is the mass, in grams, of the test sample.

NOTE Both TCC and TCN are photosensitive, hence standards should be freshly prepared.

Annex D
(normative)

Determination of chloroaniline

D.1 Principle

The chloroanilines are extracted from soap with dimethyl sulfoxide and diazotized with nitrous acid. The reaction products are then coupled with N-1-(naphthyl) ethylenediamine hydrochloride to produce coloured compounds which are estimated spectrophotometrically.

D.2 Safety precautions

Dimethyl sulfoxide (DMSO) is readily absorbed into the skin. Inhalation or skin penetration must be avoided. DMSO should never be pi petted using mouth. Always use pipette bulb. The standard chloroanilines and N-1-(naphthyl)-ethylenediamine hydrochloride shall not be allowed to come into contact with the skin. If they should, then wash the contaminated parts thoroughly with soap and water.

A supply of diluted sodium hypochlorite should be at hand at all times to deal with accidental spillages of chloroaniline solution. Spillage on laboratory surface should be treated immediately with the sodium hypochlorite solution, followed by water.

D.3 Reagents

D.3.1 **Dimethyl Sulphoxide (DMSO)**, AR grade

D.3.2 **Hydrochloric acid**, concentrated (specific gravity, 1.18)

D.3.3 **Sodium nitrite**, 0.4 % w/v analytical grade, freshly prepared (aqueous)

D.3.4 **Ammonium sulphamate**, 2 % w/v solution freshly prepared (aqueous)

D.3.5 **N-1-(naphthyl) ethylene**, 0.1 % w/v solution diamine hydrochloride freshly prepared (aqueous)

D.3.6 ***n*-Butanol**, AR grade

D.3.7 **Sand**, acid purified 40 - 100 micron mesh

D.3.8 **Solvent mixture** comprising:

- DMSO 5 volumes
- *n*-Butanol 2 volumes
- Distilled water 2 volumes
- Hydrochloric acid 1 volume

Mix *n*-butanol, water and HCI. Cool the mixture and add DMSO.

D.3.9 **4-Chloroaniline and 3, 4-Dichloroaniline,** AR grade

D.4 Apparatus

D.4.1 **Spectrophotometer,** suitable for use at 554 nm

D.4.2 **Cuvettes**, glass (matched pair) 10 mm

D.4.3 **Water bath,** thermostatically controlled at 25 °C

D.4.4 **Stopwatch**

D.4.5 **Standard laboratory glassware**

D.4.6 **Filter Paper,** Whatman No. 541

D.5 Procedure

D.5.1 Dissolve 0.349 8 g of 3,4-dichloroaniline and 0.2753 g of 4-chloroaniline in solvent mixture (see D.3.8) in a 250 mL amber volumetric flask.

Dilute to mark with solvent mixture [1 mL is 2.5 mg mixed chloroanilines (stock solution)].

D.5.2 Dilute this stock solution with solvent mixture as given below:

a) take 5 mL of stock solution and dilute it to 250 mL with solvent mixture (1 mL = 50 µg mixed chloroanilines); and

b) take 5 mL of the above solution [see (a)] and further dilute to 250 mL with solvent mixture. [1 mL = 1 µg mixed chloroanilines].

Use this solution for preparation of calibration curve.

Transfer using a burette 0, 1 mL, 2 mL, 5 mL, 10 mL, 20 mL, 40 mL into 50 mL amber volumetric flasks.

D.5.3 From a burette, add sufficient solvent mixture to make total volume to 40-mL in each flask. The flasks are incubated in a water bath at 25 °C for 20 min: After exactly 20 min, add 2-mL of reagent (see D.3.3) into each flask and return them to the water bath for exactly 10 min (measure with a stop watch).

Then add 2 mL of reagent (see D.3.4) into each flask and return them to the water bath for exactly 10 min. Swirl the flask occasionally.

Then add 2 mL of reagent (see D.3.5) into each flask and remove them from the water bath. Dilute to volume with distilled water, mix and allow to stand for 30 min. Measure absorbance at 554 nm against the blank solution as prepared in D.5.4.

D.5.4 In preparing the blank solution, take 40 mL of solvent mixture in a 50 mL amber volumetric flask. Incubate the flask in a water bath at 25 °C for 20 min. After exactly 20 min, add 2 mL of reagent (see D.3.3) into the flask and return it to the water bath for exactly 10 min. Then add 2 mL of reagent (see D.3.4) into the flask and return it to the water bath for exactly 10 min (swirl the flask occasionally). Then add 2 mL of reagent (see C.3.5) into the flask and remove it from the water bath. Dilute to volume with distilled water, mix and allow to stand for 30 min. Use this blank solution for preparation of calibration curve only.

D.5.5 Prepare a graph by plotting weight (µg) of chloroanilines contained in each 50 mL-flask against absorbance. The linear calibration will pass through the origin/or determine the average absorbance (*AA*) of 1 µg of mixed chloroanilines by dividing sum of absorbances of all different aliquots of the standard by sum of µg of chloroanilines in all different aliquots of standard.

D.6 Determination of chloroanilines

D.6.1 Weigh to the nearest mg 3.0 g - 15 g of finely grated soap and add 10.0 g - 15.0 g of acid purified sand. Transfer quantitatively the sample and the sand into a mortar and grind the mixture thoroughly with a pestle to give a homogenous mass. Transfer the mass to a previously weighed 250-mL flat bottom flask quantitatively and reweigh. Add DMSO (100 mL), stopper firmly and attach the flask to an automatic shaker. Shake for 1 h. Filter the DMSO extract through Whatman No. 541 into a 250 mL amber volumetric flask. Wash the flask and filter paper with small aliquots of DMSO. Allow the filtrate to drain completely, dilute to volume with DMSO and mix. Transfer 20 mL DMSO extract into a 50-mL amber volumetric flask. Add 20 mL of solvent mixture. The flask is incubated in a water bath at 25 °C for 20 min. After exactly 20 min, add 2 mL of reagent (see D.3.3) into the flask and return it to the water bath for exactly 10 min (measure with a stop watch). Then add 2 mL of reagent (see D.3.4) into the flask and return it to the water bath for exactly 10 min (swirl the flask occasionally). Then add 2 mL of reagent (see D.3.5) into the flask and remove it from the water bath. Dilute to volume with distilled water, mix and allow to stand for 30 min. Read the absorbance at 554 nm against blank (prepared as below).

D.6.2 Prepare the blank solution by mixing 20 mL of DMSO extract of sample and 20 mL of solvent mixture in a 50 mL amber volumetric flask. Incubate the flask in a water bath at 25 °C for 20 min.

After exactly 20 min, add 2 mL of distilled water into the flask and return it to the water bath for exactly 10 min. Then add 2 mL of reagent (see D.3.4) into the flask and return it to the water bath for exactly 10 min (swirl the flask occasionally). Then add 2 mL of reagent (see D.3.5) into the flask and remove it from the water bath. Dilute to volume with distilled water, mix and allow to stand for 30 min. Use this solution as a blank for reading sample only.

D.6.3 Deduce the amount of chloroanilines (µg) from the calibration graph curve.

NOTE The determination should be completed in one day.

D.7 Calculations

Determine the amount of mixed chloroanilines in the aliquot of test solution from the calibration graph.

$$\text{Chloroaniline content (in ppm)} = \frac{250(M + M_1)M_3}{20M_2M}$$

where

M is the mass, in grams, of soap;

M_1 is the mass, in grams, of sand;

M_2 is the mass, in grams, of soap and sand transferred to the flask; and

M_3 is the mass, in micrograms, of mixed chloroanilines found from calibration graph/or it can be calculated as given below:

$$M_3 = \frac{\text{Mass of the sample}}{\text{Average absorbance of 1 } \mu\text{g mixed chloroanilines (AA)}}$$

where

$$\text{AA} = \frac{\text{Sum of the OD of the standards}}{\text{Sum of concentration of standard chloroanilines in } \mu\text{g}}$$

$$\text{Weight of soap actually used, in g} = \frac{M_2M}{(M + M_1)}$$

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 minutes.

E.3 Reagents

E.3.1 Calcium chloride $CaCl_2.2H_2O$, AR
E.3.2 Magnesium sulphate $MgSO_4.7H_2O$, AR
E.3.3 Distilled water

E.4 Apparatus

E.4.2 100-mL glass beaker
E.4.3 Thermometer of range 0 - 110◦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 This standard hard water has a hardness of approximately 50 ppm 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
Weigh 1 g of the grated chips antibacterial 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 antibacterial bathing bar.

Stir the contents of the beaker with a glass rod and transfer the slurry to a ~~250~~ 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.

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.

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 A.1:
a) foam plus water (V_1 mL).
b) water only (V_2mL).

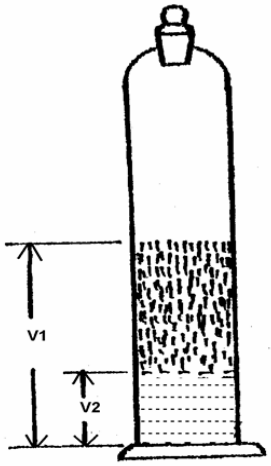

Figure E.1 — Measurement of foam

E.8 Calculation

Lather volume = V1 - V2

where

V1 = Volume, in mL of foam + water;
V2 = Volume, in mL of water only.

Annex F
(normative)

Evaluation of the mushing 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 cm^2 of original surface area in contact with the cloth.

F.2 Equipment

F.2.1 For sample preparation

F.2.1.1 Coarse kitchen cheese grater

F.2.1.2 Sharp thin blade knife or carpenters plane

F.2.1.3 Callipers or ruler, to ensure the sample dimensions

F.2.1.4 Other equipment/ materials for the test

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

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.

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.

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.

F.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.

F.4 Test procedure

For each test piece

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 ^{o}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 D.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.

F.5 Calculation

Weight of mush (grams) W = W1 – W2

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

Mush = $\frac{W \times 30\ g}{A}$ per 30 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 antibacterial 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 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 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

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 h after the washing test.

Annex H
(normative)

Determination of rosins

H.1 General

H.1.1 Colophonium (commercial rosins) only 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 to 70 ml of water.

H.2.2. Beta-naphthalene Sulphuric Acid Solution — $C_{10}H_7SO_3H$) — Obtained by dissolving 40 g of the chemical in one litre 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 - 50 g of the sample in about 500 ml of hot water. Add 10 ml - 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 - 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 Rosin acids in fatty matter shall be expressed as follows:

a) Rosin in fatty acids, percent by mass, uncorrected = $\frac{34.6(S-B)N}{M}$

where

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

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

N is the normality of alcoholic potassium hydroxide, and

M is the mass in g of the material taken for the test.

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.

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

NOTE 1 — The mean equivalent mass of the rosin acids is taken as 346.

NOTE 2 — 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.4.2 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.4.2.1 Reagents

a) **Acetic anhydride** — pure.

b) **Dilute sulphuric acid** — relative density 1.53.

H.4.2.2 Procedure

Transfer 1 ml - 2 ml of the sample of fatty acids to a test-tube, treat with 5 ml - 10 ml of acetic anhydride and warm on a steam-bath. After cooling, pour 1 ml - 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 I
(normative)

Sampling

I.1 Procedure

I.1.1 In a single consignment, all packages (cartons) containing toilet 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 I.1.

Table I.1 — Scale of sampling

Number of packages (cartons) in the lot ***N***	Number of packages (cartons) to be selected ***n***	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

I.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 $N \div n$.

I.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.

I.2 Preparation of test samples

I.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.

I.2.2 Samples for testing

Immediately after preparation of composite sample (I.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.

Annex J
(Informative)

Permitted antibacterial agents

The following is the list of antibacterial agents used generally in antibacterial soap:

a) Triclosan (TCN);

b) Trichlorocarbanilide (TCC);

c) Zinc oxide;

d) Chloro xylenols;

e) Plant extracts;

f) Any other internationally accepted antibacterial agent.