DEAS 1119-4: 2023

ICS 71.100.70

DRAFT EAST AFRICAN STANDARD

Skin applied mosquito repellent — Specification — Part 4: Bathing soap

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 078, *Healthcare and medical devices.*

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.

DEAS 1119 consists of the following parts, under the general title *Skin applied mosquito repellents — Specification:*

— *Part 1: Lotions, creams, gels and ointments*

— *Part 2: Sprays and roll-ons*

— *Part 3: Wipes*

— *Part 4: Bathing soaps*

— *Part 5: Bracelets, wristbands and patches*

— *Part 6: Jelly*

Skin applied mosquito repellent — Specification — Part 4: Bathing soap

1 Scope

This Draft East African Standard specifies the requirements, sampling and test methods for skin applied mosquito repellent bathing soap.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

DEAS 1120-1*, Mosquito repellents — Performance test guidelines — Part 1: Skin applied repellents*

EAS 377 (all parts), Cosmetics and cosmetic products

EAS 814, Determination of biodegradability of surfactants — Test method

EAS 846

ISO 2271

ISO 673

EAS 127

EAS 794

ISO 457

ISO 1067

ISO 4315

EAS 346

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

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

ISO 862, Surface active agents — Vocabulary

US ISO 24153, Random sampling and randomization procedures

US 2373-1, Mosquito repellents — Performance test guidelines — Part 1: Skin applied repellents

US EAS 186-1:2021, Bathing soap -Specification -Part 1: Solid

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EAS 846 and the following apply.
ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp.

3.1
mosquito
blood-sucking dipterous insect of the family Culicidae. Aedes, Anopheles, Culex, Mansonia, and Stegomyia are genera containing most species involved in the transmission of protozoan and other disease-causing parasites

3.2
mosquito repellent
substance applied to deter mosquito from approaching or settling

3.3
natural repellents
repellents that contain, plant-based compounds

3.4
synthetic repellents
conventional repellents containing chemical compounds manufactured to imitate the natural compounds.

3.5
bathing soap
soap which is intended for use in bathing

3.6
toilet soap
bathing soap containing fatty acids and does not contain synthetic surface-active agents

3.7
antibacterial toilet soap
toilet soap containing antibacterial agent(s)

3.8
bathing bar
bathing soap containing fatty acids and/or synthetic surface-active agents

3.9
antibacterial bathing soap
bathing bar containing antibacterial agent(s)

4 Symbols and/or abbreviated terms

DEET N, N-Diethyl-meta-toluamide or diethyltoluamide

IR3535 ethyl butylacetylaminopropionate

Picaridin/ icaridin 1-(1-methylpropoxycarbonyl)-2-(2-hydroxyethyl) piperidine or 2-(2-hydroxyethyl)-1-piperidinecarboxylic acid 1-methylpropyl ester

5 Active ingredients

5.1 Natural repellents

5.1.1 Active ingredients used in natural repellents shall be plant-based compounds which are able to deter mosquitoes from approaching or settling. Such shall be essential oils or any other plant extract approved by relevant authority as mosquito repellents.

5.1.2 The manufacturer shall provide adequate data on the repellence of such ingredients. Adequate data shall include laboratory studies showing estimation of effective dose (technical material) and estimation of complete protection time; and the outcomes of field trials showing efficacy and persistence of technical material; efficacy and persistence of formulated product.

5.1.3 The manufacturer shall have adequate data justifying the safety and proportion of ingredient(s) used in the product, for which claims are made.

5.1.4 The essential oils used and other plant extracts in natural repellents shall be, but not limited to:

a) cedarwood;

b) tea tree;

c) geranium;

d) rosemary;

e) lemongrass;

f) citronella;

g) eucalyptus;

h) cinnamon;

i) neem;

j) peppermint

k) garlic

l) lavender

m) cloves

n) basils

o) jasmine; and

p) pyrethrum

5.1.5 The proportion of single or blended active ingredient (s) in natural repellent shall be set by the manufacturer in accordance with the relevant standard and records shall be availed.

5.1.6 Pyrethrum extracts such as pyrethrins shall be considered in natural repellents.

5.2 Synthetic repellents

5.2.1 Synthetic repellents shall contain synthetic chemical compounds which are able to deter mosquitoes from approaching or settling on the surface.

5.2.2 If a synthetic active ingredient is blended with other active ingredient (s), either natural or synthetic, the proportion shall be set by the manufacturer based on scientific research and records shall be availed.

5.2.3 Synthetic repellents and their active ingredients shall be approved and registered by relevant authority before being released to the market.

6 Requirements

6.1 General requirements

6.1.1 The product shall constitute a mosquito repellent that is formulated as bathing soap and shall be essentially a product which has active ingredient(s) added to a certain level.

6.1.2 Bathing soap shall include the following:

a) Toilet soap
b) Bathing bar
c) liquid

6.1.3 The product shall be in the form of cake or liquid or bar.

6.1.4 When applied to the skin, the product shall have the benefit of repelling mosquitoes and shall not have a harmful effect to the skin

6.1.5 Bathing soap in the form of cake or bar shall be firm and of uniform texture and colour, and shall be free from objectionable (disagreeable) odour and shall remain hard, smooth and not crumble when tested in accordance with Annex E.
6.1.6 Liquid bathing soap shall be a homogeneous, clear, translucent or opaque liquid with good lathering and cleaning properties. It may contain permissible synthetic detergents.
6.1.7 All ingredients shall comply with the requirements of EAS 377.

6.2 Specific requirements

6.2.1 Active ingredients and their content in mosquito repellent bathing soap shall meet the requirements prescribed in Table 1, when tested in accordance with the test methods specified therein

Table 1 — Active ingredients content for mosquito repellent bathing soap

S/N	Characteristic	Requirement	Test method
i	DEET [a], % w/w.	4 – 30	Annex A
ii	IR3535 [a], % w/w.	7.5 – 20.07	Annex B
ii i	Picaridin [a], % w/w.	5 – 20	Annex C
iv	Pyrethrins [b], % w/w	0.3 - 0.5	Annex D
[a] Applicable to synthetic mosquito repellent bathing soap [b] Applicable to mosquito repellent containing extracts from pyrtherum.			

6.2.2 Skin applied mosquito repellent liquid bathing soap shall comply with the specific requirements given in Table 2 when tested in accordance to the methods described therein.

Table 2 — Specific requirements for skin applied mosquito repellent of liquid bathing soap

S.No.	Characteristics	Requirement		Test method
		Liquid bathing soap	Antibacterial liquid bathing soap	
i.	Total fatty matter, % by mass, min	15.0	15.0	ISO 685
ii.	Free caustic alkali, (K2O), % by mass, max.	0.03	0.03	ISO 456
iii.	Synthetic detergents, % by mass, max.	2.0	2.0	ISO 2271
iv.	Matter insoluble in ethanol, % by mass, max	5.0	5.0	ISO 673
v.	Antibacterial agent TricIosan(TCN) and Trichlorocarbanilide (TCC), % by mass, max	N/A	1.0 either singly or in combination	Annex G
vi.	Chloroaniline content, ppm, max	N/A	10.0	Annex H
vii.	Phosphate	Absent	Absent	EAS 127
viii.	Antibacterial activity	N/A	To pass test	EAS 794
ix.	pH at 27°C ± 2°C	7.5 – 9.5	7.5 – 9.5	Annex D
Note: Trichlorocarbanilide (TCC) is not heat stable and decomposes into chloroanilines 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.				

6.2.3 Skin applied mosquito repellent toilet soap shall comply with the specific requirements given in Table 3 when tested in accordance to the methods described therein.

Table 3 — Specific requirements for skin applied mosquito repellent toilet soap

S.No.	Characteristics	Requirement		Test method
		Toilet soap	Antibacterial toilet soap	
i.	Total fatty matter content, % by mass, min	70.0	70.0	ISO 685
ii.	Content of matter insoluble in ethanol, % by mass, max	2.5	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 F
v.	Chlorides content as NaCI, % 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 G
viii.	Chloroaniline content, ppm, max	N/A	10.0	Annex H

ix.	Antibacterial activity	N/A	To pass thetest	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:

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

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..

6.2.4 Mosquito repellent bathing bar shall comply with the specific requirements Table 4 when tested in accordance to the methods described therein.

Table 4 — Specific requirements for skin applied mosquito repellent bathing bar

S.No.	Characteristics	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 I
iii.	Mush (loss in mass due mushing on a wetsurface), g/30 cm^2, max	10.0	10.0	Annex J
iv.	Freedom from grittiness	To pass a test	To pass atest	Annex K
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 L
vii.	Antibacterial agent TricIosan (TCN) and Trichlorocarbanilide (TCC), % by mass, max.	n/a	1.0 either singly or in combination	Annex G
viii.	Chloroaniline content, ppm, max	n/a	10.0	Annex H
ix.	Antibacterial activity	n/a	To pass thetest	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:

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

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..

6.3 Biological efficacy

When tested in accordance with DEAS 1120-1, the product shall repel 100% of the mosquitoes from approaching or climbing on that surface, within protection time indicated by the manufacturer.

7 Packaging

The product shall be packaged in suitable well-sealed containers that shall protect the contents and shall not cause any contamination, deterioration or reaction with the product.

8 Labelling

In addition to the labelling requirements given in EAS 346, the 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 “Mosquito repellent bathing soap”

b) form of product as “toilet soap”, “bathing bar” or “liquid ”;

c) instructions for use;

d) active ingredient (s) content;

e) expiry date;

f) date of manufacture;

g) batch number; and

h) precaution/warning

9 Sampling

Sampling shall be done in accordance with ISO 24153.

Annex A
(normative)

Determination of DEET content

A.1 General

The sample is dissolved in carbon disulfide and the difference in absorbance at 14.18 µm and at 14.48 µm is determined. The quantity of meta-isomer is obtained from this value by means of a calibration curve prepared by the use of a reference standard.

A.2 Apparatus

A.2.1 Double-beam infrared spectrophotometer

A.2.2 Two equivalent infrared absorption cells, with sodium chloride windows and a path length of approximately 0.4 mm.

A.3 Preparation of calibration curve

A.3.1 Weigh (to the nearest 0.1 mg) into four volumetric flasks sufficient amounts of the reference DEET standard of known purity to give concentrations of approximately 20, 40, 60 and 80 g/L when dissolved in carbon disulfide.

A.3.2 Fill the reference cell with carbon disulfide and the sample cell with each of the standard solutions in turn, and record the spectra. The spectrum may be scanned rapidly, except for the region 12 – 15 µm, where a normal speed should be used. Carry out a blank measurement with carbon disulfide to correct for any inequality in the paired cells and to determine whether a cell correction is required.

A.3.3 Measure the absorbance at 14.18 µm and at 14.48 µm and calculate the difference between these values, ΔA, for each of the solutions. Plot the values of ΔA against the concentration (mg/L) of the meta-isomer.

A.3.4 If a cell correction is required, the value of ΔA is determined from the formula:

ΔA = [A14.18 – A14.48] ref. - [A14.48] blank

Where ref. = determination with reference standard

blank = determination on CS2 blank

A.4 Procedure

Weigh (to the nearest 0.1 mg) about 0.5 g of the sample, transfer quantitatively to a 10 mL volumetric flask, and make up to the mark with carbon disulfide. Measure the infrared absorption at 14.18 µm and 14.48 µm using the same conditions as described in clause A.3. Determine the concentration of meta-isomer by comparing this value with the calibration curve. A standard sample should be run each day to check the calibration of the instrument.

A.5 Calculation

DEET content (g/kg)

$= \frac{(C_1 \times P)}{C_2}$ where,

C_1 concentration (g/L) of standard DEET found from calibration curve

C_2 concentration (g/L) of sample taken

P purity (g/kg) of the reference standard

Annex B
(normative)

Determination of ethyl butylacetamidopropionate (IR3535).

B.1 Outline of method

B.1.1 Ethyl butylacetamidopropionate is determined by capillary gas chromatography using flame ionisation detection and internal standardisation.

B.1.2 The retention time of the ethyl butylacetamidopropionate peak of the sample solution should not deviate by more than 2 % from that of the calibration solution.

B.2 Reagents

B.2.1 Acetonitrile

B.2.2 Ethyl butylacetamidopropionate, standard of known purity

B.2.3 Methyl tridecanoate, internal standard

B.2.4 Calibration solution.

B.2.4.1 Weigh (to the nearest 0.1 mg in duplicate) into volumetric flasks (10 ml) about 100 mg ethyl butylacetamidopropionate standard (s mg) and about 100 mg methyl tridecanoate (r mg).

B.2.4.2 Dissolve in acetonitrile and fill to the mark with acetonitrile (solutions C1 and C2). The solutions are stable for one week at room temperature.

B.3 Apparatus

B.3.1 Gas chromatograph equipped with a split/splitless injection and a flame ionisation detector

B.3.2 Capillary column fused silica, 25 m × 0.32 (i.d.) mm, coated with CP-Sil 5 CB, film thickness: 1.2 µm

B.3.3 Electric integrator or data system

B.4 Procedure

B.4.1 Chromatographic conditions (typical)

B.4.1.1 Column fused silica, 25 m × 0.32mm (i.d.), film thickness: 1.2 µm, coated with CP-Sil 5 CB

B.4.1.2 Injection system

B.4.1.2.1 Injector: split injection

B.4.1.2.2 Split ratio:1:50

B.4.1.2 Detector: Flame ionisation

B.4.1.3 Temperatures

B.4.1.3.1 Injection port:300 °C

B.4.1.3.2 Detector:310 °C

B.4.1.3.3 Oven program

B.4.1.3.3.1 initial: 120 °C

B.4.1.3.3.2 Program rate: 10 °C/min

B.4.1.3.3.3 Final: 260 °C

B.4.1.4 Injection volume: 5 µl

B.4.1.5 Gas flow rates

B.4.1.5.1 Helium 1.1 ml/min

B.4.1.5.2 Helium (make up) 45 ml/min

B.4.1.5.3 Hydrogen 40 ml/min

B.4.1.6 Retention times

B.4.1.6.1 ethyl butylacetamidopropionate: about: 10.4 min

B.4.1.6.2 methyl tridecanoate: about: 10.9 min

B.5 Preparation of sample.

B.5.1 Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (10 ml) sufficient sample to contain about 100 mg ethyl butylacetamidopropionate (w mg) and about 100 mg methyl tridecanoate (q mg).

B.5.2 Dissolve in acetonitrile and fill to the mark with acetonitrile (solutions S1 and S2). The solutions are stable for one week at room temperature.

B.6 System equilibration

Inject 0.1 µl portions of the calibration solution and repeat the injections until retention times and calibration factors vary by less than 0.1 % of the mean of three successive injections.

B.7 Determination.

B.7.1 Inject 0.1 µl portions of the calibration solution and sample solutions in the following sequence: C_1, S_1, C_2, S_2,…etc.

B.7.2 Determine the peak areas and calculate the mean response factor (*f*) of the calibration solution injections bracketing the injections of the sample solutions.

B.8 Calculation

B.8.1 Individual response factor f_i is calculated as follows:

$$f_i = \frac{I_r \times S \times P}{H_s \times r}$$

B.8.2 Ethyl butylacetamidopropionate shall be calculated as follows:

$$\frac{H_w \times f \times q}{I_s \times w}$$

where:

f_i individual response factor

f mean response factor

H_s peak area of ethyl butylacetamidopropionate in the calibration solution

H_w peak area of ethyl butylacetamidopropionate in the sample solution

I_r peak area of the internal standard in the calibration solution

I_q peak area of the internal standard in the sample solution

s mass of ethyl butylacetamidopropionate standard in the calibrationsolution (mg)

r mass of internal standard in the calibration solution (mg)

q mass of internal standard in the sample solution (mg)

w mass of sample taken (mg)

P purity of ethyl butylacetamidopropionate standard (g/kg)

Repeatability r 18 g/kg at 1002 g/kg active ingredient content

Reproducibility R 18 g/kg at 1002 g/kg active ingredient content

Annex C
(normative)

Determination of icaridin

C.1 outline of method

Icaridin is determined by capillary gas chromatograph using internal standardisation and flame ionisation detection.

C.2 Reagents

C.2.1 Icaridin reference standard with known content

C.2.2 Dimethyl Phthalate internal standard

C.2.3 Propan-2-ol

C.2.4 Calibration solution.

Weigh (to the nearest 0.1 mg) about 100mg icaridin reference substance (*s* mg) and 100 mg dimethyl phthalate (*r* mg) into a volumetric flask (20 ml). Fill to the mark with propan-2-ol and homogenise.

C.3 Apparatus

C.3.1 Gas chromatograph capable of operating in the range 150 °C to 330 °C, fitted with a flame ionisation detector, a split injector and an autosampler

C.3.2 Column quartz,30 m ×0.25 mm (i.d), coated with dimethyl polysiloxane/ diphenyl polysiloxane 95/5% (e.g. DB5), film thickness 0.25 µm

C.3.3 Electronic integrator or data system

C.4 procedure

C.4.1 Chromatographic conditions (typical)

C.4.1.1 Column: Quartz,30 m × 0.25mm (i.d.), coated with dimethyl polysiloxane/ diphenyl polysiloxane 95/5% (e.g. DB5), film thickness 0.25 µm

C.4.1.2 Injection system

C.4.1.2.1 Injector: split injection

C.4.1.2.2 Split flow:40ml/min

C.4.1.2 Detector: Flame ionisation

C.4.1.3 Temperatures

C.4.1.3.1 Injector:240 °C

C.4.1.3.2 Detector:330 °C

C.4.1.3.3 Oven program: 150°C hold for 2 min, gradient: 10°C/ min to 330 °C, hold for 3 min.

C.4.1.4 Injection volume: 1 µl

C.4.1.5 Gas flow rates

C.4.1.5.1 Helium (carrier) 1.5 ml/min (100kpa)

C.4.1.5.2 Hydrogen: about 30ml/min

C.4.1.5.3 Air: about 300ml/min

C.4.1.5.4 Nitrogen (makeup): about 25ml/min

C.4.1.6 Run time: about 25 min

C.4.1.7 Retention times

C.4.1.6.1 Dimethyl Phthalate: about 3 min

C.4.1.6.2 Icaridin: about 4.5 min

C.5 Preparation of sample

Weigh (to the nearest 0.1 mg) into a volumetric flask (20 ml) sufficient sample to contain about 100 mg icaridin (*w* mg) and about 100 mg dimethyl phthalate (*q* mg). Fill to the mark with propan-2-ol and homogenise.

C.6 Equilibration of the system

Inject 1 µl portion of calibration solution and repeat the injections until retention times and the icaridin to the internal standard peak area ration vary by less than 0.5% of the mean for successive injections.

C.7 Determination

C.7.1 Inject in duplicate 1 µ portions of the calibration solution (C_1 and C_2) and of the sample solution (S_1, S_2, etc.) in the following sequence:C_1, S_1, S_2, ...C_2).

C.7.2 Determine the peak areas and calculate the response factor (*f*) from the calibration solutions bracketing the injections of the sample solutions. Calculate the content of the sample solutions.

C.8 Calculation

C.8.1 Response factor *f* is calculated as follows:

$$f = \frac{I_r \times S \times P}{H_s \times r}$$

C.8.2 Icaridin content shall be calculated in g/kg as follows:

$$\frac{H_w \times f \times q}{I_s \times w}$$

where:

f mean response factor

H_s peak area of icaridin in the calibration solution

H_w peak area of icaridin in the sample solution

I_r peak area of dimethyl phthalate in the calibration solution

I_q peak area of dimethyl phthalate in the sample solution

s mass of icaridin in the calibration solution (mg)

r mass of dimethyl phthalate in the calibration solution (mg)

q mass of dimethyl phthalate in the sample solution (mg)

w mass of sample taken (mg)

P purity of icaridin standard (g/kg)

Repeatability r 12 g/kg to 14 g/kg at 986 g/kg active ingredient content

Reproducibility R 14 g/kg at 986 g/kg active ingredient content

Annex D
(normative)

Determination of total pyrethrins

D.1 General

The active ingredients in pyrethrum extract may be determined using a HPLC system first by injecting a solution of the analyte into the chromatograph, followed by the separation and comparison of peaks areas of the analytes in the sample with that of an external standard containing a known amount of the analytes. The peaks are eluted in the following order: Cinerin II, Pyrethrin II, Jasmolin II (total Pyrethrins II) and Cinerin I, Pyrethrin I, Jasmolin I (total Pyrethrins I).

D.2 Reagents

D.2.1 World pyrethrum standard, 50 %

D.2.2 Acetonitrile, HPLC grade

D.2.3 Water, HPLC grade

D.3 Apparatus

A liquid chromatography System equipped with an auto-sampler, a Variable Wavelength Detector (or equivalent) and a Column {Phenomenex, 250 x 4.6 mm Luna Phenyl-Hexyl 5µ Reverse Phase (or equivalent).

D.4 Operating conditions

D.4.1 Flow rate: 1.5 ml/min

D.4.2 Composition: 40:60 (%, v/v water/acetonitrile)

D.4.3 Elution: Isocratic

D.4.4 Oven temperature: 40 ºC

D.4.5 Wavelength: 240 nm

D.4.6 Injection Volume: 15 µl

D.4.7 Stop time: 22 min

D.4.8 Post time: 1 min

D.5 Preparation of the standard

Weigh 20 mg of the pyrethrum standard to the nearest 0.0001 g in a 100 mL volumetric flask and dilute to volume with acetonitrile and label it. Transfer a small portion to a sample vial and label it accordingly.

D.6 Sample preparation

In a 100 ml volumetric flask, weigh 20 mg to the nearest 0.0001 g of the sample to be analyzed and dilute to volume with Acetonitrile. Sample this solution using a vial and label it accordingly.

D.7 Procedure

After the chromatograph is stable, make a minimum of three injections for the standard solution as well as for the analyte and average the area counts. The relative standard deviation between injections should be within 2 %.

D.8 Calculation of the % total pyrethrins (active ingredient)

The total pyrethrins, expressed as percent shall be calculated as follows;

$$= \frac{(\text{Average sample area X weight of standard X Purity of the standard (in \%)})}{(\text{Average standard area X Weight of sample})}$$

Annex E
(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 F
(normative)

Determination of free fatty acids content as oleic acid

F.1 Barium chloride method

F.1.1 Apparatus

F.1.1.1 500 mL conical flask.

F.1.1.2 Reflux condenser to fit the flask.

F.1.2 Reagents

F.1.2.1 Distilled water or water**,** of at least equivalent purity, free from carbon dioxide.

F.1.2.2 Ethanol, 95 per cent (V/V), free from carbon dioxide and distilled over potassium hydroxide.

F.1.2.3 Ethanol**,** aqueous solution 60 per cent (V/V), neutralized.

Mix 125 mL ethanol (F.1.2.2), 75 mL distilled water (F.1.2.1) and 1 mL of indicator (F.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 (F.1.2.7). Neutralize with the hydrochloric acid solution (3.5.6) until the violet colour disappears.

F.1.2.4 Potassium or sodium hydroxide, 0.1 N aqueous solution.

F.1.2.5 Barium chloride, aqueous solution.

Dissolve 10 g of barium chloride dihydrate (BaCl2. 2H20) in 90 ml of distilled Neutralize with potassium or sodium hydroxide in the presence of indicator (F.1.2.7) until a violet colour appears.

F.1.2.6 Hydrochloric acid, 0.1 N aqueous solution, accurately standardized.

F.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 (F.1.2.2). Filter.

F.2 Procedure

Weigh, to the nearest 0.01 g, about 5 g of soft soap into a conical flask (F.1.1.1). Add 200 ml of ethanol (F.1.2.3). Connect the reflux condenser (F.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 (F.1.2.5) in small portions shaking thoroughly. Cool with running water to room temperature.

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

F.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 (F.1.2.6) used;

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

m *is the mass, in g, of the test portion.*

Annex G
(normative)

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

G.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.

G.2 Normal phase HPLC

G.2.1 Reagents

G.2.1.1 Iso-octane, HPLC grade

G.2.1.2 Iso-propanol (2-propanol), HPLC grade

G.2.1.3 Hexane, HPLC grade

G.2.1.4 Standard TCC, 99 % pure

G.2.1.5 Standard TCN, 99 % pure

G.2.2 Apparatus

G.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

G.2.2.2 Standard volumetric flasks

G.2.2.3 Pipette

G.2.2.4 Magnetic stirrer

G.2.2.5 Millipore filter apparatus with 0.5 micron filter

G.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.

G.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

F.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.

G.2.3 Procedure

G.2.3.1 Standard preparation

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).

G.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.

G.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.

G.2.4 Calculation

TCN shall be expressed as follows:

$$\frac{\text{Area of sample for TCN} \times \text{Concentrat ion of standard TCN}}{\text{Area of standard TCN} \times \text{Concentrat ion of sample}} \times 100$$

TCN, percent by mass=

$$\frac{\text{Area of sample for TCC} \times \text{Concentrat ion of standard TCC}}{\text{Area of standard TCC} \times \text{Concentrat ion of sample}} \times 100$$

G.3 Reverse phase

G.3.1 Reagents

G.3.1.1 Methanol, HPLC grade

G.3.1.2 Sodium Dihydrogen Phosphate Monohydrate, chemical grade

G.3.1.3 Standard TCC

G.3.1.4 Standard TCN (TCS)

G.3.2 Apparatus

G.3.2.1 Column

G.3.2.1.1 Octyldimethylsilyl (C-DB)

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

G.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.

G.3.3 Procedure

C.3.3.1 Standard preparation (see Note under G.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 G.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, expressed as percent by mass, shall be calculated as follows:

$$\text{TCN} = \frac{M_s \times A_r \times F}{A_s \times M_t \times 100}$$

$$\text{TCC} = \frac{M_s \times A_r \times F}{A_s \times M_t \times 100}$$

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 H
(normative)

Determination of chloroaniline

H.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.

H.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.

H.3 Reagents

H.3.1 Dimethyl Sulphoxide (DMSO), AR grade

H.3.2 Hydrochloric acid, concentrated (specific gravity, 1.18)

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

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

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

H.3.6 *n*-Butanol, AR grade

H.3.7 Sand, acid purified 40 - 100 micron mesh

H.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.

H.3.9 4-Chloroaniline and 3, 4-Dichloroaniline, AR grade

H.4 Apparatus

H.4.1 Spectrophotometer, suitable for use at 554 nm

H.4.2 Cuvettes, glass (matched pair) 10 mm

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

H.4.4 Stopwatch

H.4.5 Standard laboratory glassware

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

H.5 Procedure

H.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)].

H.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.

H.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 into each flask and return them to the water bath for exactly 10 min (measure with a stop watch).

H.5.4 Then add 2 mL of reagent (see H.3.4) into each flask and return them to the water bath for exactly 10 min. Swirl the flask occasionally.

H.5.5 Then add 2 mL of reagent (see H.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 H.5.6.

H.5.6 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 into the flask and return it to the water bath for exactly 10 min. Then add 2 mL of reagent 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 H.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.

H.5.7 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

H.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 H.3.3) into the flask and return it to the water bath for exactly 10 min (measure with a stopwatch). Then add 2 mL of reagent (see H.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 H.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).

H.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 H.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 H.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.

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

H.7 Calculations

The amount of mixed chloroanilines in the aliquot of test solution from the calibration graph, expressed as milligrams per kilogram, shall be calculated as follows:

$$\frac{250\ (M+\ M_1)M_3}{20M_2M}$$

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:

$$\frac{Mass\ of\ the\ sample}{\text{Average absorbance of 1 g mixed chloroanil ines (AA)}}$$

Where

$$\frac{Sum\ of\ the\ OD\ of\ the\ standards}{Sum\ of\ concentrat\ ion\ of\ standard\ chloroanil\ ines\ in\ g}$$

Weight of soap used, in grams = $\frac{M_2\ M}{M+M_1}$

Annex I
(normative)

Test for lather volume of Bathing bar

I.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.

I.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.

I.3 Reagents

I.3.1 Calcium chloride CaCl2.2H2O, AR

I.3.2 Magnesium sulphate MgSO4.7H2O, AR

I.3.3 Distilled water

I.4 Apparatus

I.4.2 100-mL glass beaker

I.4.3 Thermometer of range 0◦C - 110◦C

I.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.

I.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.

I.7 Procedure

I.7.1 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.

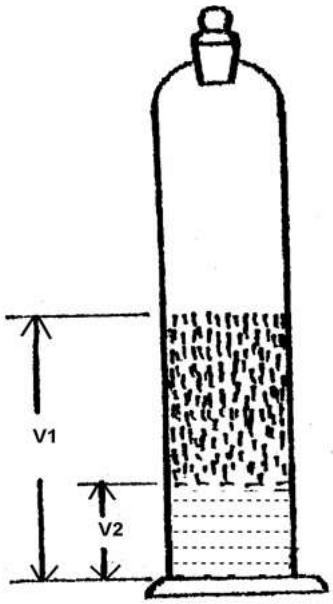
I.7.2 Stir the contents of the beaker with a glass rod and transfer the slurry to a 250 500-mL graduated cylinder ensuring that not more than 2 mL foam is produced. Repeat the transfer of the residue left in the beaker with further portions of 20 mL of standard hard water ensuring that all the matter in the beaker is transferred to the cylinder.

I.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.

I.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 A.1:

a) foam plus water (V_1 mL).

b) water only (V_2mL).



I.8 Calculation

Lather volume = $V_1 - V_2$

where

V_1 is the Volume, in mL of foam + water; and

V_2 is the Volume, in mL of water only.

Annex J
(normative)

Evaluation of the mushing properties of a bathing bar

J.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.

J.2 Equipment

J.2.1 For sample preparation

J.2.1.1 Coarse kitchen cheese grater

J.2.1.2 Sharp thin blade knife or carpenters plane

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

J.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.

J.3 Bar preparation

J.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.

J.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.

J.4 Test procedure

For each test piece

J.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.

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

J.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.

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

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

J.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.

J.4.7 The test piece is reweighed (W_2) and the amount of mush removed is calculated as in J.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.

J.5 Calculation

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

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

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

J.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 4.

Annex k
(normative)

Determination of grittiness in bathing bar

K.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.

K.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 L
(normative)

Determination of rosins

L.1 General

L.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.

L.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.

L.2 Reagents

L.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.

L.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.

L.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.

L.2.4 Phenolphthalein Indicator — Obtained by dissolving 1 g in 100 ml of 95 % (v/v) ethyl alcohol.

L.3 Procedure

L.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.

L.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.

L.3.3 Conduct simultaneously a blank determination with 25 ml of the etherifying agent alone.

L.4 Calculation

L.4.1 Uncorrected rosin

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

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

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.

L.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 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.

L.4.3 Liebermann-Storch test

L.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,

L.4.3.1 Reagents

L.4.3.1.1 Acetic anhydride — pure.

L.4.3.1.2 Dilute sulphuric acid — relative density 1.53.

L.4.3.2 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.

Bibliography

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

[2] EAS 186-2: 2021 Bathing soap — Specification — Part 2: Liquid

[3] RS 392-4: 2018, Skin applied mosquito repellents — Specification — Part 4: Bathing soaps

[4] RS 392-1: 2018, *Skin applied mosquito repellents — Specification — Part 1: Lotions, creams, gels and ointments*

[5] US 2296-4:2022, Skin applied mosquito repellents - Specification - Part 4: Bathing soaps

