DRAFT TANZANIA STANDARD

Mosquito repellent soap – Specification

TANZANIA BUREAU OF STANDARDS

Foreword

This Draft Tanzania Standard is being developed by the Soap and Detergents Technical Committee under supervision of the Chemicals Divisional Standards Committee and it is in accordance with the procedures of the Bureau.

This is the first edition of the draft Tanzania standard for mosquito repellent soap. In reporting the results of analysis of a test if the final value is to be rounded off, it shall be done in accordance with TZS 4 *Rounding off numerical values*

Mosquito repellent soap – Specification

1 Scope

1.1 This Draft Tanzania Standard specifies requirements, sampling and test methods for mosquito repellent soap for body cleaning and protection from mosquito bites.

1.2 This standard applies to mosquito repellent soaps containing N, N-diethyl-3-methylbenzamide (DEET) as the active ingredient supplied in the form of bars/cakes and produced from vegetable or animal oils or fats, fatty acids, or from a blend of all or part of these materials, with or without the addition of rosins or non-soapy surfactants.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

TZS 638 (All parts)/EAS 377*(all parts)*, *Cosmetics and cosmetics*

TZS 2102/EAS 794, *Determination of the microbial inhibition of cosmetic soap bars and liquid hand and body washes — Test method*

TZS 1780/EAS 814, *Determination of biodegradability of surfactants — Test method*

TZS 1396-4/ISO 456, *Surface active agents — Analysis of soaps— Determination of free caustic alkali*

TZS 1396-6/ISO 685, *Analysis of soap — Determination of alkali content and total fatty matter content*

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

ISO 862, *Surface active agents — Vocabulary*

3 Terms and definitions

For the purposes of this standard the following terms and definitions shall apply.

3.1 Mosquito repellent soap

product in the form of a bars or cakes containing mosquito repellent agent(s) and soap of fatty acids and/or synthetic surface active agents

3.2 All terms and definitions given in ISO 862

4 Requirements

4.1 General requirements

4.1.1 The product shall:

a) be in the form of cakes or bars;

b) not be injurious to health when used in a manner and purpose meant for their use or under reasonably foreseen conditions;

c) not have an unpleasant odour;

d) be firm and smooth in texture; and

e) not contain any ingredients in amounts that are harmful to the human body and environment.

4.2.2 The colour of the cake or bar shall generally be uniform. For the case of genuinely mottled products, the colour may not be uniform.

4.2.3 Synthetic surface active agents, may be used and when used shall not be more than 4% by mass when tested in accordance with Annex D.

4.2.4 All the ingredients used in the mosquito repellent soap shall comply with the requirements of all parts of TZS 638

4.3 Specific requirements

Mosquito repellent soap shall conform to the specific requirements given in Table 1 when tested in accordance with the methods prescribed therein.

Table 1 — specific requirements

SL. No	Characteristic	Requirement	Test method
i)	Total fatty matter, % by mass, min.	50	TZS 1396-6
ii)	Lather, mL, min.	200	Annex A
iii)	Mush (loss in mass due mushing on a wet surface), g/30 cm^2, max..	10	Annex B
iv)	Freedom from grittiness	To pass the test	Annex C
v)	Total alkalinity (as NaOH) % by mass, max.	1.0	TZS 1396-6 ISO 4315
vi)	Free caustic alkali (as NaOH), % by mass, max.	0.05	TZS 1396-4
vii)	Rosins, as % of total fatty matter, max	2	Annex F
viii)	Biodegradability test	To pass the test	EAS 814
ix)	Mosquito repellent agents (DEET- N, N-diethyl-3-methylbenzamide,)	10% – 30%	Annex H

5 Packaging and labelling

5.1 Packaging

Mosquito repellent soap shall be wrapped as to protect them from damage, excessive loss or gain of moisture and to conserve the mosquito repellent agent contained.

5.2 Labelling

Each package shall be legibly and indelibly labelled with the information from (a) to (i) either in English and/or Kiswahili or combination. Other languages may be added as agreed between the manufacturer and supplier.

a) name of the product as ‘mosquito repellent soap;

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) net content;

d) active ingredients/mosquito repellent agent contained with its level;

e) batch number or code number;

f) date of manufacture and best before date; and

g) country of origin.

h) Precaution: not to be used to children under 12 years

i) Storage condition

6 Sampling

Sampling shall be done in accordance with Annex G.

Annex A
(normative)

Test for lather volume

A.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.

A.2 Outline of the method

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

A.3 Reagents

A.3.1 *Calcium chloride* $CaCl_2.2H_2O$**,** AR

A.3.2 *Magnesium sulphate* $MgSO_4.7H_2O$**,** AR

A.3.3 *Distilled water*

A.4 Apparatus

A.4.1 *Graduated cylinder* — Glass stoppered with graduation from 0 mL- 250 mL, with 2-mL divisions. Overall height about 35 cm and the height of the graduated portion about 20 cm.

A.4.2 100mL glass beaker

A.4.3 Thermometer of range 0ºC– 110ºC

A.5 Preparation of standard hard water

Dissolve 0.220 g of calcium chloride dehydrate 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 mg/kg calculated as calcium carbonate.

A.6 Sample preparation

Cut away the outer edges of mosquito repellent soap using a knife.

Using a stand up type of grater, grate up to 10 g – 15 g of the mosquito repellent soap into small chips.

A.7 Procedure

A.7.1 Weigh 1 g of the grated chips accurately in a 100mL 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 mosquito repellent soap.

A.7.2 Stir the contents of the beaker with a glass rod and transfer the slurry to a 250-mL graduated cylinder ensuring that not more than 2 mL foam is produced. 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.

A.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.

A.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).

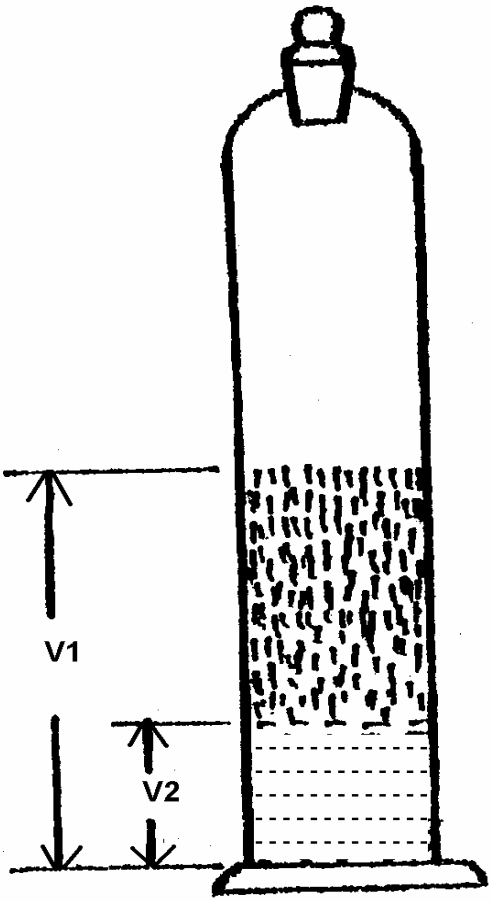

Figure A.1 — Measurement of foam

A.8 Calculation

Lather volume = V_1 - V_2

where

V_1 Volume, in millilitres of foam + water;

V_2 Volume, in millilitresl of water only.

Annex B

(normative)

Evaluation of the mushing properties of a mosquito repellent soap

B.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.

B.2 Equipment

B.2.1 For sample preparation

B.2.1.1 Coarse kitchen cheese grater

B.2.1.2 Sharp thin blade knife or carpenters' plane

B.2.1.3 Callipers or ruler to ensure the sample dimensions

B.2.2 Other equipment/materials for the test

B.2.2.1 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.

B.2.2.3 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.

B.3 Bar preparation

B.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).

B.3.2 All bars in a set must be cut to have the same face size. If the smallest of the range of bars to be tested at a given time is too small to allow a working face within these limits, then all bars should be cut to the maximum size possible from the smallest bar.

B.3.3 The longest axis of the test piece (6 cm ±1 cm) should be from a direction parallel to the longest axis of the original bar sample.

B.3.4 The working face should be a fresh surface from the interior of the bar sample. The face opposite the working face should be identified by making a small hole with a sharp object. This enables the working face to be identified after the preconditioning step.

B.3.5 To cut the bar it is convenient to first trim it to the approximate size using a coarse kitchen cheese grater and then to make the final adjustments to a smooth surface with a sharp thin-bladed knife or carpenters' plane. If a plane is used, it is better to move the bar over the plane blade.

B.4 Test procedure

B.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.

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

B.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.

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

B.4.5 The mushed bar test piece is removed from the tray and is weighed (W_1).

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

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

B.5 Calculation

The mush of mosquito repellent soap shall be expressed as follows:

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

Surface area of bar, A (square centimetres) = (width x breadth)

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

B.6 Criteria for conformity

B.6.1 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.

B.6.2 The sample lot of products shall be declared as conforming to the requirements for this standard if

$X + 0.6 \times R$ is less than the maximum value given in Table 1.

Annex C

(normative)

Determination of grittiness in a mosquito repellent soap

C.1 Procedure

C.1.1 Hold the mosquito repellent soap 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.

C.1.2 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).

C.1.3 Allow the used bar to dry in the open for 4 h and examine the surface. A set of three 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 three sets of products (nine grit tests).

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

C.2 Criteria for conformity

The performance criteria are:

a) during manipulation under running water, the mosquito repellent soap shall not have a visibly rough surface and shall feel smooth to the touch; and

b) no gritty particles shall be observed on the surface of the dried bar 4 h after the washing test.

Annex D
(normative)

Determination of active detergent content

D.1 Outline of the method

When equivalent amounts of cationic and anionic detergents are present in a two-phase mixture of water and chloroform, methylene blue will colour the two phases to the same degree. Sodium alkyl benzene sulphonate and sodium lauryl sulphate or any other detergent can be titrated with a standard solution of cetyltrimethyl ammonium bromide.

D.2 Reagents

Weigh 1.5 g ± 0.001 g of cetyltrimethylammonium ammonium bromide into a 250-mL beaker. Add 100 mL of distilled water and stir until dissolved. Transfer quantitatively to a one-litre volumetric flask and make to volume. Mix thoroughly and standardize against solution B (see D.2.1).

D.2.1 Anionic solution (Solution B)

Weigh accurately such amount of standard alkyl sulphate of known combined SO_3 or active content so as to give exactly 0.320 g of combined SO_3into a 250-mL beaker. Dissolve in 100 mL- 200 mL of warm water. Transfer quantitatively to one-litre volumetric flask and make to volume with water at room temperature. Mix thoroughly. This is the primary standard against which solution A, is standardized. Solution B is 0.004 N.

D.2.2 Methylene blue indicator

Dissolve 0.1 g of methylene blue in 100 mL of water. Transfer 30 mL of this solution to a one-litre flask. Add 500 mL of water, 6.8 mL of concentrated sulphuric acid, 50 g of ($NaH_2PO_4 \cdot H_2O$) sodium dihydrogen phosphate monohydrate and shake until dissolution is complete. Dilute to the mark.

D.2.3 Chloroform

Analytical reagent grade.

D.3 Procedure

D.3.1 Weigh accurately a sample of sufficient size to give approximately 0.320 g of combined SO_3 into a 250-mL beaker. Sample size is crucial (see Note). Use 700 mL- 800 mL of warm water to transfer quantitatively to a one-litre volumetric flask. Warm on steam bath and shake gently until the sample is dissolved and solution is clear. Cool, dilute to the mark and mix thoroughly.

NOTE The titration value V should be as near as to 10 mL as possible, say between 8 mL and 12 mL but never outside 5 mL and 15 mL.

D.3.2 Pipette 10.0 mL of the sample solution into a 100-mL glass stoppered cylinder (25 mmx 300 mm). Add 25.0 mL ± 0.5 mL of methylene blue solution and 10mL ± 0.5 mL of chloroform (see Note). Titrate with solution A to the correct end point, shaking the cylinder carefully after such addition to avoid emulsion and maintaining temperature within prescribed limits of 20 °C - 30 °C by immersion in water bath, if necessary. As the end point is approached, the rate of transfer of colour increases and solution A shall be added dropwise with vigorous shaking after each addition. If the approximate titration is known, 80 % of the required titrating solution should be added before shaking since this avoids emulsion formation. Application of vacuum to the titration cylinder may help to break some emulsions, if formed. The end point is reached when both layers have same colour intensity. The end point is very sharp and 0.05 mL will cause a distinct change in colour distribution at or near the equivalence point.

NOTE The titration value *V* should be as near to 10 mL as possible, say between 8 mL and 12 mL but never outside 5 mL and 15 mL.

D.3.3 Pipette 10.0 mL of the sample solution into a 100-mL glass stoppered cylinder (25 mm x 300 mm). Add 25.0 mL ± 0.5 mL of chloroform (see Note). Titrate with solution A to the correct end point, shaking the cylinder carefully after such addition to avoid emulsion and maintaining temperature within prescribed limits of 20ºC - 30ºC by immersion in water bath if necessary. As the end point is approached, the rate of transfer of colour increases and solution A shall be added dropwise with vigorous shaking after each addition. If the approximate titration is known, 80 % of the required titrating solution should be added before shaking since this avoids emulsion formation.

Application of vacuum to the titration cylinder may help to break some emulsions, if formed. The end point is reached when both layers have same colour intensity. The end point is very sharp and 0.05 mL will cause a distinct change in colour distribution at or near equivalence point.

NOTE The volume of methylene blue solution and chloroform may be changed if found advantageous provided the same volumes are used in standardizing solutions A and B.

D.3.4 Calculation

D.3.4.1 The percent combined SO_3 shall be expressed as follows:

$$\% \text{ combined } SO_3 = \frac{V \times N \times 8.0}{M}$$

where,

V volume, in millilitres, of solution A used in the titration;

N normality of solution A; and

M mass, in grams, of the sample in the aliquot.

D.3.4.2 The percent active detergent content shall be expressed as follows:

Percent active detergent content = percent combined SO_3 x Molecular weight of active detergent.

NOTE The molecular weight of active detergent should be supplied by the manufacturer on request.

D.4 Alternative method for determination of active detergent content

(To be used only if the first method (Clause D.1) fails to work on the product).

D.4.1 Field of application

This method is applicable to the analysis of alkylbenzene sulphonates, alkyl sulphonate, sulphates and hydroxy-sulphates, alkylphenol and fatty alcohol ethoxysulphates and dialkyl sulphosuccinates and to the determination of active materials containing one hydrophilic group per molecule.

D.4.2 Principle

Determination of anionic-active matter in a medium consisting of an aqueous and chloroform phase, by volumetric titration with a standard cationic-active solution (benzethonium chloride), in the presence of an indicator which consists of a mixture of a cationic dye (dimidium bromide) and an anionic dye (acid blue 1).

D.4.3 Reagents

D.4.3.1 The water used shall be of distilled quality.

D.4.3.2 **Chloroform**, (specific gravity = 1.48, distilling between 59.5ºC and 61.5ºC).

D.4.3.3 **Sulphuric acid**, 2.5 M solution.

D.4.3.4 **Sulphuric acid**, 0.5 M solution.

D.4.3.5 **Sodium hydroxide**, 1.0 M standard volumetric solution.

D.4.3.6 **Sodium lauryl sulphate (sodium dodecyl sulphate)** ($CH_3(CH_2)_{11}OSO_3Na$), 0.004 M standard volumetric solution. Check the purity of the sodium lauryl sulphate and simultaneously prepare the standard solution.

D.4.3.6.1 Determination of purity of sodium lauryl sulphate — Weigh, 5 g ± 0.2 g of the product into a 250-mL round bottom flask with ground glass neck. Add exactly 25 mL of the sulphuric acid solution (D.4.3.4) and reflux into a water condenser.

During the first 5 min - 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask.

In order to avoid excessive foaming, instead of refluxing the solution may be left on a boiling water bath for 60 min.

After a further 10 min the solution clarifies and foaming ceases. Reflux for further 90 min. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 mL of ethanol followed by water.

Add a few drops of the phenolphthalein solution (D.4.3.8) and titrate the solution with the sodium hydroxide solution (D.4.3.5).

Carry out a blank test by titrating 25 mL of the sulphuric acid solution (D.4.3.4) with the sodium hydroxide solution (D.4.3.5).

The purity of the sodium lauryl sulphate, expressed as a percentage,

$$= \frac{28.84(V_1 - V_O)\ M_O}{M_1}$$

where,

V_0 is the volume, in millilitres, of sodium hydroxide solution used for the blank test;

V_1 is the volume, in millilitres, of sodium hydroxide solution used for the sample;

M_1 is the mass, in grams, of the sodium lauryl sulphate to be checked; and

M_0 is the exact molarity of the sodium hydroxide solution.

D.4.3.6.2 Weigh 0.004 M sodium lauryl sulphate standard volumetric solutions. Weigh, to the nearest 1 mg between 1.14 g and 1.16 g of sodium lauryl sulphate and dissolve in 200 mL of water. Transfer to a ground glass stoppered 1-L one-mark volumetric flask and dilute to the mark with water.

Calculate the molarity, M_1, of the solution by means of the solution by means of the formula:

$$M_1 = \frac{m_2 \text{ x } purity(\%)}{288.4 \text{x} 100}$$

where,

m_2 = mass in grams of sodium lauryl sulphate.

D.4.3.7 Benzethonium chloride 0.004 M standard volumetric solution

Weigh, to the nearest 1 mg, between 1.75 g and 1.85 g benzethonium chloride and dissolve in water. Transfer to a ground glass-stoppered 1-L one-mark volumetric flask and dilute to the mark with water.

NOTE In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105°C, weigh 1.792 g, to the nearest 1 mg, dissolve in water and dilute to 1 L.

D.4.3.8 Phenolphthalein, ethanolic solution containing 10 g/L. Dissolve 1 g of phenolphthalein in 100 mL of 95 % (v/v) ethanol.

D.4.3.9 Mixed indicator

D.4.3.9.1 Stock solution

Weigh 0.5 g ± 0.005 g dimidium bromide into a 50-mL beaker, and 0.025 g ± 0.005 g of acid blue 1 into a second 50-mL beaker.

Add between 20 mL and 30 mL of hot 10 %. (v/v) ethanol to each beaker. Stir until dissolved and transfer the solutions to a 250-mL one mark volumetric flask. Rinse the beakers into the volumetric flask with ethanol and dilute to the mark with 10 % (v/v) ethanol.

D.4.3.9.2 Mixed acid indicator solution

Take 20 mL of the stock solution prepared above, put it in a 500-mL one-mark volumetric flask. Add 200 mL of water, and 20 mL of 2.5 M sulphuric acid (D.4.3.3) mix and dilute to the mark with water. Store away from direct sunlight.

D.4.4 Apparatus

Ordinary Laboratory apparatus, and

a) bottles, 200-mL, glass stoppered, or measuring cylinders, glass stoppered KNY cross check.

b) burettes, 25-mL and 50-mL.

c) one-mark volumetric flask, 1-L capacity glass stoppered.

d) one-mark pipette, 25-mL.

D.4.5 Procedure

D.4.5.1 Standardization of benzethonium chloride solution

By means of the pipette transfer 25 mL of the 0.004 M sodium lauryl sulphate solution to a bottle or measuring cylinder, add 10 mL of water, 15 mL of the chloroform and 10 mL of the mixed indicator solution.

Titrate with the 0.004 M benzethonium chloride solution. Stopper the bottle or measuring cylinder after each addition and shake well. The lower layer will be coloured pink. Continue the titration with repeated vigorous shaking. As the end point approaches, the emulsions formed during shaking tend to break easily continue the titration drop by drop. Shaking after each addition of titrant, until the end point is reached. This is at the moment when the pink colour is completely discharged from the chloroform layer, which becomes a faint greyish blue.

The molarity, M, of the benzethonium chloride solution is given by the formula:

$$M = \frac{M_1 \times 25}{V_2}$$

where,

M_1 is the molarity of the sodium lauryl sulphate solution; and

V_2 is the Volume, in, millilitres of benzethonium chloride added.

D.4.5.2 Determination

Weigh to the nearest 1 mg a sample of 30 g; dissolve the test portion in water. Add a few drops of the phenolphthalein solution and neutralize to a faint pink colour with the sodium hydroxide solution or sulphuric acid solution as required.

Transfer to one-litre volumetric flask and dilute to the mark with water. Mix thoroughly and, by means of the pipette transfer 25 mL of this solution to a bottle or measuring cylinder, add 10 mL of water, and 15 mL of chloroform. Titrate with the benzethonium chloride solution as described in D.4.5.1.

D.4.6 Expression of results

The content as a percentage by mass, of anionic-active matter

$$= \frac{V_3 \times M \times 1\,000 \times M_o \times 100}{25 \times 1\,000 \times M_o}$$

$$= 4V_3M$$

The amount of active matter, expressed in milli-equivalents per gram,

$$= \frac{40 \times V_3 \times M_1}{M_o}$$

where,

M_0 is the mass, in grams, of the test portion;

M is the relative molar mass of anionic-active matter;

M_1 is the molarity of the benzethonium chloride solution;

V_3 is the volume, in millilitres, of benzethonium chloride solution used for the titration of a 25-mL aliquot of anionic-active matter solution.

Annex F
(normative)

Determination of rosins

F.1 General

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

F.1.2 The method described in this test gives results approximately one per cent higher than the amount or rosin actually present. Consequently, the percentage of rosin acids actually present is one less than the percentage or rosin acids found experimentally and hence minus one in the formula.

F.2 Reagents

F.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.

F.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.

F.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.

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

F.3 Procedure

F.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 ºC ± 2 ºC for 45 min - 50 min.

F.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.

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

F.4 Calculation

F.4.1 Rosin acids in fatty matter shall be expressed as follows:

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

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.

F.4.2 In all cases where the rosin content is found to be less than 5 percent, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test,

F.4.2.1 Reagents

a) Acetic anhydride — pure.

b) Dilute sulphuric acid — relative density 1.53.

F.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 1mL - 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 G
(normative)

Sampling

G.1 Procedure

G.1.1 In a single consignment, all packages (cartons) containing mosquito repellent soaps 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 G.1.

Table G.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

G.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:

G.1.3 Starting from any package, count all the packages in one order as 1, 2, 3.... *N*, selecting every *k* thpackage, where *k* is the integral part of $N \div n$.

G.1.4 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.

G.2 Preparation of test samples

G.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.

G.2.2 Samples for testing

Immediately after preparation of composite sample (C.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 H

(normative)

Determination of DEET content

H.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.

H.2 Apparatus

H.2.1 Double-beam infrared spectrophotometer. Perkin-Elmer model 21 or equivalent.

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

H.3 Preparation of calibration curve

H.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.

H.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.

H.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 (g/L) of the meta-isomer.

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

$$\Delta A = [A_{14.18} - A_{14.48}]_{\text{ref.}} - [A_{14.48}]_{\text{blank}}$$

Where, ref. = determination with reference standard
blank = determination on CS_2 blank

H.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 disulphide. Measure the infrared absorption at 14.18 µm and 14.48 µm using the same conditions as described in clause H.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.

H.5 Calculation

$$\text{DEET content (g/kg)} = \frac{C_1 \times P}{C_2}$$

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