



**DEAS 186-2: 2020**

ICS 71.100.40

## **DRAFT EAST AFRICAN STANDARD**

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**Bathing soap — Specification — Part 2: Liquid**

**EAST AFRICAN COMMUNITY**

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East African Community  
P. O. Box 1096  
Arusha  
Tanzania  
Tel: 255 27 2504253/8  
Fax: 255 27 2504481/2504255  
E-mail: [eac@eachq.org](mailto:eac@eachq.org)  
Web: [www.eac-quality.net](http://www.eac-quality.net)

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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) 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 second edition cancels and replaces the first edition (EAS 766-2:2013), which has been technically revised and numbered as EAS 186-2.

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

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

## Introduction

Human skin provides a favourable environment for the existence and multiplication of a variety of microbes. The conventional toilet soap washes away the germs but does not kill them. The function of an antibacterial or antiseptic toilet soap is not only to clean the skin, but also to reduce drastically the bacterial count on the skin. This prevents skin infections and perspiration odour caused by the decomposition of perspiration by bacteria.

Antibacterial toilet soap is a toilet soap that has antibacterial agents incorporated into it. It not only cleans the skin, but also reduces drastically the bacterial count on the skin. This prevents skin infections and perspiration odour caused by the decomposition of sweat by bacteria. The antibacterial toilet soap is especially effective against *staphylococcus* and similar bacteria which have the habit of residing in the under layers of the skin. The antibacterial soaps have to be used regularly to be effective.

In this revision, hexachlorophene has not been permitted to be used as antibacterial agent. The Trichlorocarbanilide (TCC) on heating decomposes to chloroanilines which are harmful to skin hence the limit and method for determination of chloroaniline is included. Use of other antibacterial agents not included in Annex A will be considered when need arises as long as their safety is assured.



## Bathing soap — Specification — Part 2: Liquid

### 1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for liquid bathing soap. It does not apply to hand wash liquid detergents, shampoo and products for specific purposes such as those for industrial and surgical uses.

### 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 127, *Synthetic laundry detergents for household use — Specification*

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*

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

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

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

ISO 862, *Surface active agents — Vocabulary*

ISO 2271 *Surface active agents — Detergents — Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure*

### 3 Terms and definitions

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



## 4 Requirements

### 4.1 General requirements

**4.1.1** The liquid bathing soap shall consist of essentially of an aqueous solution of potassium soaps, sodium soaps or both, made from oils, fatty acids or their mixture. It shall be a homogeneous, clear, translucent or opaque liquid with good lathering and cleaning properties. It may contain permissible synthetic detergents.

**4.1.2** The liquid bathing soap shall remain as homogeneous stable product and shall show no sign of separation or sedimentation when kept at 5 °C for 24 h.

**4.1.3** The liquid antibacterial bathing soap shall contain permitted antibacterial agent in accordance with Annex A.

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

**4.1.5** Liquid bathing soap shall pass the test for dermatological safety.

### 4.2 Specific requirements

Liquid bathing soap shall also comply with the specific quality requirements specified in Table 1.

**Table 1 — Specific requirements for liquid bathing soap**

| SI No. | Characteristic   | 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, (K <sub>2</sub> O), % 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 Triclosan (TCN) and Trichlorocarbanilide (TCC), % by mass, max | n/a                 | 1.0<br>either singly<br>or in<br>combination | Annex B     |
| vi.    | Chloroaniline content, ppm, max  | n/a                 | 10.0   | Annex C     |
| vii.   | Phosphate  | absent              | absent                                       | EAS 127     |
| viii.  | Antibacterial activity   | n/a                 | To pass test                                 | EAS 794     |
| ix.    | pH at 27 ± 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 Packaging and labelling**

### **6.1 Packaging**

The bathing soap shall be packed in protective containers that will not allow for damage of the product or its contamination.

### **6.2 labelling**

- a) Each container 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 name of the product as “liquid bathing soap or antibacterial liquid bathing 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) batch number or lot number;
- e) date of manufacture and best before date;
- f) country of origin;
- g) the antibacterial agent used (for antibacterial bathing soap); and
- h) list of ingredients in descending order of quantity

## **7 Sampling**

Sampling shall be done in accordance to Annex E

## **8 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)

**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 xlenols;
- e) Plant extracts;
- f) Any other internationally accepted antibacterial agent.

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## Annex B (normative)

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

#### B.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.

#### B.2 Normal phase HPLC

##### B.2.1 Reagents

- B.2.1.1 **Iso-octane**, HPLC grade
- B.2.1.2 **Iso-propanol (2-propanol)**, HPLC grade
- B.2.1.3 **Hexane**, HPLC grade
- B.2.1.4 **Standard TCC**, 99 % pure
- B.2.1.5 **Standard TCN**, 99 % pure

##### B.2.2 Apparatus

- B.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
- B.2.2.2 **Standard volumetric flasks**
- B.2.2.3 **Pipettes**
- B.2.2.4 **Magnetic stirrer**
- B.2.2.5 **Millipore filter apparatus** with 0.5 micron filter
- B.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.

##### B.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.

**B.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.

**B.2.3 Procedure**

**B.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).

**B.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.

**B.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.

**B.2.4 Calculation**

TCN shall be expressed as follows:

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

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

## B.3 Reverse phase

### B.3.1 Reagents

B.3.1.1 Methanol, HPLC grade

B.3.1.2 Sodium Dihydrogen Phosphate Monohydrate, chemical grade

B.3.1.3 Standard TCC

B.3.1.4 Standard TCN (TCS)

### B.3.2 Apparatus

B.3.2.1 Column

B.3.2.1.1 Octyldimethylsilyl (C-DB)

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

B.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.

### B.3.3 Procedure

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

B.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.

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

B.3.3.1.3 Accurately pipette 10 mL of the solution prepared in B.3.3.1.1 into the volumetric flask containing TCC (B.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.

B.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.

B.3.3.3 HPLC conditions

The HPLC conditions include the following:

- a) Detector wavelength: 280 nm;

- b) Column temperature: 35 °C;
- 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.

### B.3.4 Calculations

The TCN and TCC shall be expressed as follows:

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

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

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

$M$  is the mass, in grams, of the test sample.

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

## Annex C (normative)

### Determination of chloroaniline

#### C.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.

#### C.2 Safety precautions

Dimethyl sulfoxide (DMSO) is readily absorbed into the skin. Inhalation or skin penetration must be avoided. DMSO should never be pipetted 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.

#### C.3 Reagents

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

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

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

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

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

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

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

**C.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 HCl. Cool the mixture and add DMSO.

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



## C.4 Apparatus

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

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

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

**C.4.4 Stopwatch**

**C.4.5 Standard laboratory glassware**

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

## C.5 Procedure

**C.5.1** Dissolve 0.349 g of 3,4-dichloroaniline and 0.2753 g of 4-chloroaniline in solvent mixture (see C.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)].

**C.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.

**C.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 C.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 C.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 C.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 C.5.4.

**C.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 C.3.3) into the flask and return it to the water bath for exactly 10 min. Then add 2 mL of reagent (see C.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.

**C.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.

## C.6 Determination of chloroanilines

**C.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 C.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 C.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. Read the absorbance at 554 nm against blank (prepared as below).

**C.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 C.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 solution as a blank for reading sample only.

**C.6.3** Deduce the amount of chloroanilines ( $\mu\text{g}$ ) from the calibration graph curve.

NOTE The determination should be completed in one day.

## C.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 D** **(normative)**

### **Determination of pH**

#### **D.1 General**

pH determination should be made in an acid free atmosphere.

#### **D.2 Apparatus**

**D.2.1 Any standard pH meter**, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solutions (see D.3.2) before use.

**D.2.2 Volumetric flask**, 1000-mL capacity

**D.2.3 Beakers**, 1000-mL

#### **D.3 Reagents**

**D.3.1** Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 27 °C. The residue on evaporation when heated at 105 °C for one hour shall not exceed 0.5 mL per litre.

**D.3.2** Standard buffer solutions with the pH range of 9 to 11 at 27 °C for calibrating the pH meter.

#### **D.4 Procedure**

Weigh to the nearest milligram approximately 10 g of the material and transfer to a 1-L volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to 27 °C ± 2 °C and fill to the calibration mark with distilled water, stopper the flask mix thoroughly and allow the solution to stand at a temperature of 27 °C ± 2 °C for two hours prior to measuring the pH. Measure the pH of the solution at 27 °C ± 2 °C using a glass electrode.

## Annex E (normative)

### Sampling

#### E.1 Procedure

**D.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 E.1.

Table E.1 — Scale of sampling

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

**E.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  $k^{\text{th}}$  package, where  $k$  is the integral part of  $N \div n$ .

**E.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.

#### E.2 Preparation of test samples

##### E.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.

##### E.2.2 Samples for testing

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



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