



## DRAFT EAST AFRICAN STANDARD

---

Laundry soap and washing bars — Specification.

FOR PUBLIC COMMENT ONLY



iii)

### Copyright notice

This EAC document is copyright-protected by EAC. While the reproduction of this document by participants in the EAC standards development process is permitted without prior permission from EAC, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from EAC.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to EAC's member body in the country of the requester:

© East African Community 2026 — All rights reserved  
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)

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement. Violators may be persecuted

## Contents

Page

Foreword .....	iv
<b>1 Scope .....</b>	<b>1</b>
<b>2 Normative references .....</b>	<b>1</b>
<b>3 Terms and definitions .....</b>	<b>Error! Bookmark not defined.</b>
<b>4 Grades of laundry soap .....</b>	<b>3</b>
<b>5 Requirements .....</b>	<b>3</b>
<b>5.1 General .....</b>	<b>3</b>
<b>5.1.1 Appearance .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.1.2 Texture .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.1.3 Odour .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.1.4 Colouring matter .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.1.5 Stability .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.1.6 Toxicity .....</b>	<b>Error! Bookmark not defined.</b>
<b>5.2 Specific requirements .....</b>	<b>4</b>
<b>6 Sampling .....</b>	<b>6</b>
<b>7 Methods of test .....</b>	<b>Error! Bookmark not defined.</b>
<b>8 Compliance with the standard .....</b>	<b>Error! Bookmark not defined.</b>
<b>9 Packing and marking .....</b>	<b>7</b>
<b>9.1 Packing .....</b>	<b>Error! Bookmark not defined.</b>
<b>9.2 Marking .....</b>	<b>7</b>
<b>9.2.1 Packaging .....</b>	<b>Error! Bookmark not defined.</b>
<b>9.2.2 Wrapper .....</b>	<b>7</b>
<b>9.2.3 Bars or cakes .....</b>	<b>Error! Bookmark not defined.</b>
<b>10 Sampling and inspection .....</b>	<b>Error! Bookmark not defined.</b>
<b>10.1 Sampling .....</b>	<b>Error! Bookmark not defined.</b>
<b>10.2 Inspection .....</b>	<b>Error! Bookmark not defined.</b>
<b>Annex A (normative) Determination of staining test of laundry bar soap .....</b>	<b>9</b>
<b>A.1 Method 1: Undissolved powder (5.0 % product concentration) .....</b>	<b>9</b>
<b>A.1.1 Principle .....</b>	<b>9</b>
<b>A.1.2 Materials .....</b>	<b>9</b>
<b>A.1.3 Procedure .....</b>	<b>9</b>
<b>A.2 Method 2: Pre-dissolved soap (2.5 % product concentration) .....</b>	<b>9</b>
<b>A.2.1 Principle .....</b>	<b>9</b>
<b>A.2.2 Materials .....</b>	<b>9</b>
<b>A.2.3 Procedure .....</b>	<b>9</b>

iii)

## 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.

In order to achieve this objective, the Community established an East African Standards Committee mandated to develop and issue East African Standards.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. 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 procedures of the Community.

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.

EAS 31 was prepared by Technical Committee EAS/TC 074, *Surface active agents*.

This fifth edition cancels and replaces the fourth edition (EAS 31:2021), which has been technically revised.

# Laundry soap and washing bars— Specification

## 1 Scope

This working draft East African Standard specifies requirements, sampling and test methods for laundry soaps and washing bars.

## 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.

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

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

ISO 672, *Analysis of soap — Determination of moisture and volatile matter content*

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

ISO 684, *Analysis of soap — Determination of total free alkali*

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

ISO 862, *Surface active agents — Vocabulary*

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

ISO 6839, *Anionic surface active agents — Determination of solubility in water<sup>3</sup> Terms and definitionsmatter*

ISO 2870, *Surface active agents — Detergents — Determination of anionic-active matter hydrolysable and non-hydrolysable under acid conditions*

ISO 2871-1, *Surface active agents — Detergents — Determination of cationic-active matter content Part 1: High-molecular-mass cationic-active matter*

ISO 2871-2, *Surface active agents — Detergents — Determination of cationic-active matter content Part 2: Cationic-active matter of low molecular mass (between 200 and 500)*

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

EN 1276 *Chemical disinfectants and antiseptics - Quantitative suspension test for the evaluation of bactericidal activity of chemical disinfectants and antiseptics used in food, industrial, domestic and institutional areas - Test method and requirements*

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

ISO 4313 Washing powders — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

### 3. Definitions

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

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

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

#### 3.1

##### **batch**

soap from one vat or pan. In the continuous production process, the soap from one day's production shall constitute a batch.

#### 3.2

##### **builder**

complementary component of soap, usually inorganic, which with reference to the washing action, adds its characteristic properties to those of the essential constituents

**NOTE** Builders are added to a soap to improve its effectiveness under the conditions of use. The action of builders is mostly physico-chemical and comprises a series of effects, which results in more economic usage and better cleansing action of soap especially in hard water areas. Substances commonly used as builders are soda ash, sodium silicates, sodium phosphates, borax and cellulose derivatives.

#### 3.3

##### **built laundry soap**

grade II laundry soap containing moderate quantities of builders (see Table 1)

#### 3.4

##### **colouring matter**

any dyestuff that may be used to colour laundry soap

#### 3.5

##### **fillers**

materials added to soaps to increase the mass of the product but which in themselves do not improve effectiveness of the soap under the conditions of use

**NOTE** Fillers are generally inert and of an almost or completely non-detergent character.

#### 3.6

##### **free caustic alkali**

uncombined caustic alkali present in a soap

#### 3.7

##### **laundry soap**

a cleaning agent specifically formulated for fabrics, primarily composed of natural fats and oils that have undergone saponification.

**Note1 to entry: Laundry soap may contain fillers, builders, colouring matter, perfume, optical brighteners, preservatives, glycerine or opacifiers.**

#### 3.8

##### **lot**

identified quantity of laundry soap produced under essentially the same conditions

### 3.9

#### **pure laundry soap**

well saponified soaps which, in addition to moisture, may contain amounts of other substances, such as colouring matter, perfume, preservative, opacifiers and optical brightening agents but do not contain fillers.

**Note to entry: the soap may contain antibacterial agents**

### 3.10

#### **total fatty matter**

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

### 3.11

#### **total free alkali**

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

### 3.12

#### **saponification**

chemical reaction in which a fat is converted into a soap by the action of alkaline

### 3.13

#### **washing bar**

bar processed for household washing purposes, in which the cleaning action is derived primarily from fatty acid soaps and synthetic surfactants (soapy washing bar) or synthetic surfactants only (non-soapy washing bar). Washing bars may contain structurants, builders, preservatives, moisture, suitable quantities of colouring matter, perfume, opacifiers and optical brightening agents.

'Note1 to entry: the bar may contain antibacterial agents'

## **4 Requirements**

### **4.1 General requirements**

#### **4.1.1 Grades of laundry soap**

Laundry soap shall be of the following types:

- a) pure laundry soap; and
- b) built/filled laundry soap.

#### **4.1.2 Grades of non-soapy washing bar**

Washing bar shall be of the following four grades depending on their active detergent content

- a) Grade 1,
- b) Grade 2,
- c) Grade 3, and
- d) Grade 4

4.1.3 laundry soap shall be in the form of cakes, tablets or bars produced from vegetable or animal oils or fats or a blend of all or part to these materials.

4.1.4 Washing bars shall be in the form of bars or tablets produced from synthetic surfactants and vegetable or animal oils or fat or a blend of all or part to these materials.

4.1.5 Laundry soap and washing bars shall be free from visible dirt and other foreign matter.

4.1.6 Laundry soap and washing bars shall be of firm texture and possess good lathering and cleaning properties.

4.1.7 Laundry soap and washing bars shall be free from objectionable odour. It shall not leave objectionable odour on clothes after washing and thoroughly rinsing with water.

4.1.8 When immersed in distilled water for one hour at 25 °C – 30 °C, laundry soap and washing bars shall not disintegrate, and when dried at room temperature for 25 h thereafter, it shall not crumble, crack or break.

4.1.9 Laundry soap and washing bars shall not be injurious to health, cause irritation to the skin during use or handling, damage the fabrics and shall be environmentally friendly/safe.

4.1.10 Washing bars shall be made of fatty acid soaps and synthetic surfactants, or synthetic surfactant only including but not limited to fatty acids, fatty acid ester sulphonates, fatty alkanolamides, fatty alcohol ethoxylates, fatty isethionates, alpha olefin sulphonates, alcohol sulphonates and other proven surface active agents

4.1.11 In addition to suitable surfactants, washing bars shall contain other ingredients such as electrolytes, bar structuring processing aids, permitted antioxidants and humectants. For guidance, a list of bar structuring aids is given in Annex J.

4.1.12 They may contain suitable quantities of colouring matter, perfume, opacifiers and optical brightening agents.

4.1.13 All ingredients used shall comply with the requirements of EAS 377 (all parts)

4.1.14 The colour of the product shall be uniform, except for multi-coloured bars or tablets.

## 4.2 Specific requirements

Laundry soap and washing bar shall comply with the specific requirements given in Table 1 and Table 2 respectively, when tested in accordance with the test methods specified therein

**Table 1 — Specific requirements for laundry soap**

S/No.	Characteristic	Requirement,		Test method
		Pure laundry soap Grade I	Built/filled laundry soap Grade II	
i)	Total fatty matter, %, m/m min	62	45	ISO 685
ii)	Matter insoluble in water, % m/m, max.	0.5	5	ISO 6839
iii)	Matter insoluble in ethanol, % m/m, max.	5	20	ISO 673
iv)	Free caustic alkali, as NaOH, m/m, max.	0.2	0.4	ISO 456 or Annex B
v)	Total free alkali, as NaOH, % m/m, max.	0.2	0.5	ISO 684 or Annex C

vi)	Total free fat (unsaponified and unsaponifiable fatty matter), %, m/m, max.	0.2	2	ISO 1067
vii)	Sodium Oxide (Na <sub>2</sub> O), % m/m, max.	0.25	0.4	ISO 456
viii)	Moisture and volatile matter content at 105 °C, % m/m, max.	30	30	ISO 672
ix)	Chloride content, as NaCl, % by mass, max	1.5	1.5	ISO 457
x)	Staining	Shall pass the test		Annex A
xi)	Antibacterial activity <sup>a</sup>	Shall pass test		EAS 794/ ASTM E1174/ EN 1276

<sup>a</sup>Applicable to products with antibacterial claims

NOTE 1 As laundry soap is liable to lose moisture on keeping, the results of analysis in respect to free caustic alkali, unsaponified fatty matter, unsaponifiable matter and matter insoluble in alcohol should be recalculated on the basis of the minimum specified total fatty matter by means of the equation

$$\text{Recalculated results} = \frac{\text{Actual results} \times \text{Minimum specified total fatty matter}}{\text{Actual total fatty matter}}$$

NOTE 2 As weight of laundry soap is liable to diminish owing to loss of moisture in the soap, the weight of the soap bars or cakes should be recalculated on the basis of the minimum specified total fatty matter.

EXAMPLE

If a soap bar during analysis is found to weigh 496 g, the actual total fatty matter of that bar is found to be 70.0 %, the value of unsaponified matter is found to be 0.56 % and the minimum content of total fatty matter is specified as 62.0 %, then recalculated value for unsaponified matter is:

$$\frac{0.56 \times 62.0}{70.0} = 0.469 \%$$

and the recalculated weight of the soap bar drying is:

$$\frac{496 \times 70.0}{62.0} = 560 \text{ g}$$

**Table 2 — Specific requirements for soapy washing bars**

S/N	Parameter	Requirement	Test method
i.	Wear, g/m, max.	1.5	Annex D
ii.	Mush g/30 cm <sup>2</sup> , max.	10	Annex E
iii.	Grittiness	To pass test	Annex F
iv.	Cleaning index, max.	2.0	Annex G
v.	Lather volume mL, min.	150	Annex H
vi.			
vii.	Total fatty matter, %, m/m min	35	ISO 685
viii.	Matter insoluble in water, % m/m, max.	5	ISO 6839
ix.	Total free alkali, as NaOH, % m/m, max.	0.5	ISO 684
x.	Total free fat (unsaponified and unsaponifiable fatty matter), %, m/m, max.	2	ISO 1067
xi.	Sodium Oxide (Na <sub>2</sub> O), % m/m, max	0.4	ISO 456
xii.	Moisture and volatile matter content at 105 °C, % m/m, max.	30	ISO 672
xiii.	Chloride content, as NaCl, % m/m, max	1.5	ISO 457
xiv.	LSD/NSD, % m/m, min	4	Annex P
xv.	Staining	To pass test	Annex A
NOTE: LSD = Lime soap dispensing agents URT NSD= Non-soapy detergent			

**Table 3 — Specific requirements for non-soapy washing bars (adopted from IS 8180:2020)**

S/N	Characteristic	Requirement				Test method
		Grade 1	Grade 2	Grade 3	Grade 4	
1	Active ingredient, % m/m, min	16	14	12	10	ISO 2271
2	Total phosphate (as P <sub>2</sub> O <sub>5</sub> ), %m/m max	5	8	5	5	ISO 4313
3	Active alkalinity (ml of 0.1N HCl to titrate 50 mL of 1% product solution to phenolphthalein end point), max	10	12	15	20	Annex L
4	Percent detergency, min	65	55	45	35	Annex M
5	Ash built up, percent, max	1	5	10	12	Annex N
<p>Note:</p> <p>1) Use of Zeolite is recommended as an alternative and substitute for phosphate based builder.</p>						

## 5 Packaging

The product shall be package in clean, sound and dry containers made of a material, which does not affect the product and which protects the product from excessive loss of moisture and shall be free from contamination during storage and transportation.

## 6 Labelling

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

- a) name of the product as “Pure laundry soap” or, “built/filled laundry soap or washing bar
- b) manufacturer’s name and physical address
- c) number of bars or cakes contained in the package;
- d) country of origin;
- e) batch/lot number;
- f) date of manufacture and best before date
- g) indication of antibacterial activity where applicable
- h) List of ingredients

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

### 6.2 Wrapper

On the wrapper in which each soap or washing bar is wrapped shall be labelled with the particulars contained in 6.1 (a) to (b).

### **6.3 Unwrapped laundry soaps and washing bar**

In case of soaps or washing which are not wrapped in individual wrapper, each unwrapped bars or cakes shall be legibly or indelibly marked with the following

- a) name or trademark of the manufacturer
- b) name of the product as “pure laundry Soap” or “built/filled Laundry Soap”; or ‘Washing Bar;
- c) nominal weight of the bar.

### **7 Sampling**

Sampling shall be done in accordance to Annex K

FOR PUBLIC COMMENT ONLY

## **Annex A** **(Normative)**

### **Determination of Staining test**

#### **A.1 Method 1: Undissolved powder (5.0 % product concentration)**

##### **A.1.1 Principle**

Test pieces of cloth of defined area are rubbed with soap or washing bar and then dipped in water overnight then scrubbed and rinsed in running water.

##### **A.1.2 Materials**

Pieces of white cotton, nylon and Crimplene C cloth

##### **A.1.3 Procedure**

**A.1.3.1** Rub evenly about 10 g of soap or washing bar over a 15 cm x 7.5 cm test swatch placed on a china plate.

**A.1.3.2** Pour gently 50-mL of hot water (approximately 55 °C) into the plate so that the test swatch is covered and left overnight (16 h).

**A.1.3.3** Hand rub the swatch 10 times and then rinse each of the three test swatches are rinsed twice in about two litres of water and then dried in the drier.

**NOTE** The staining test is conducted in triplicate for all cloth types.

#### **A.2 Method 2: Pre-dissolved soap (2.5 % product concentration)**

##### **A.2.1 Principle**

The method involves subjecting fabrics to prolonged soaking in a highly concentrated soap or washing bar solution.

##### **A.2.2 Materials**

Pieces of white cotton, nylon and Crimplene C cloth of dimension 15 cm x 7.5 cm

##### **A.2.3 Procedure**

**A.2.3.1** Weight 10 g of soap or washing bar in a honey jar and then add 200 mL of hot water at a temperature of approximately 60 °C, shake until when the soap is thoroughly dissolved.

**A.2.3.2** Place a test swatch A 15 cm x 7.5 cm in the soap or washing bar solution (A.2.3.1) and allow to stand overnight.

**A.2.3.3** Transfer the test swatch in a bowl containing one litre of water and then agitate vigorously by hand for 10 s.

**A.2.3.4** Rinse the test swatches in 5 L of water by hand. The times should be fixed for all washes, and then dry swatches. The time of the washing need to be specified for better results

**NOTE** The staining test should be conducted in triplicate for all cloth types.

A.2.3.5 The soap shall be taken to have passed the test if it does not leave any visible stains on the fabrics after washing

FOR PUBLIC COMMENT ONLY

## Annex B

(normative)

### Determination of free caustic alkali

#### B.1 Principle

The method involves dissolving the soap in alcohol. The insoluble carbonate is precipitated with  $\text{BaCl}_2$  to prevent it from interfering with titration reaction. The solution (containing free  $\text{OH}^-$ ) is the titrated with sulphuric acid, as the case may be. The acid only neutralizes the free caustic alkali, which is the only strong base remaining in the solution. The soap itself (a salt of a weak acid) is neutral to the Phenolphthalein endpoint. This method is based on ISO 456

#### B.2 Reagents

**B.2.1** Phenolphthalein indicator, dissolve 1 g in 100 mL of 95 % ethyl alcohol, rectified spirit

**B.2.2** Standard sulphuric acid or standard hydrochloric acid, approximately 0.1 N.

**B.2.3** Standard sodium hydroxide solution, approximately 0.1 N.

**B.2.4** Barium chloride solution, 10 % (m/v).

#### B.3 Procedure for Free Caustic Alkali

**B.3.1** For unbuilt laundry soaps, weigh accurately about 10 g of the sample into a 250-mL flask, add about 100mL of ethyl alcohol, insert a cork provided with a long tube to act as a reflux condenser and immerse into a boiling water-bath, shaking frequently until the soap is dissolved. Add about 5 mL of barium chloride solution to eliminate traces of carbonates which are usually present. Add a few drops of phenolphthalein indicator and titrate with standard sulphuric acid or hydrochloric acid.

**B.3.2** For soaps containing builders and fillers, take the filtrate heat it to boil. Add about 0.5 ml of phenolphthalein indicator and titrate with standard sulphuric or hydrochloric acid.

#### B.4

**B.5** Calculation Free caustic alkali (as NaOH) expressed as percent by mass, shall be calculated as follows:

$$= 4 V N/M$$

where

V is the volume, in millilitres, of the standard sulphuric acid or hydrochloric acid used;

N is the normality of standard sulphuric acid or hydrochloric acid; and

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

## **Annex C** (normative)

### **Determination of total free alkali in laundry soap**

#### **C.1 General**

Fatty acids in the sample of soap are separated by treatment with dilute sulphuric acid and are quantitatively extracted by ether. The ether extract is titrated in alcoholic medium with standard sodium hydroxide solution for determining the combined alkali. The total anhydrous soap is determined by evaporating and drying the titrated solution.

#### **C.2 Reagents**

**C.2.1 Dilute sulphuric acid, 1:1 (v/v)**

**C.2.2 Methyl orange indicator**, dissolve 0.1 g in 100 mL of water.

**C.2.3 Sodium chloride solution**, saturated

**C.2.4 Ethyl ether**

**C.2.5 Ethyl alcohol, 95% (v/v)**

**C.2.6 Standard sodium hydroxide solution**, approximately 0.5 N and carbonate-free

**C.2.7 Standard hydrochloric acid**, approximately 0.5 N

**C.2.8 Phenolphthalein indicator**, dissolve 1 g in 100 mL of 95 % rectified spirit.

#### **C.2 Procedure**

**C.2.1** Accurately weigh 5 g to 10 g of the sample, depending upon the anhydrous soap content, and dissolve in a 250-mL conical flask by warming in 100 mL of water. When dissolution is complete, add dilute sulphuric acid in slight excess (as judged by methyl orange indicator), insert a small funnel into the neck of the flask, and heat the flask to a temperature not exceeding 60 °C until the fatty acids separate as a clear layer. Add 50 mL of sodium chloride solution and cool. Transfer quantitatively to a separating funnel, draw off the aqueous acid layer into a second separating funnel and shake it with three 50-mL portions of ethyl ether. Dissolve the fatty acids in the ether used for washing the aqueous liquid and extract with 10-mL portions of water until the extracts are no longer acidic to methyl orange indicator. Mix the water portions used for washing and shake with 20 mL of ether. Wash this ether until the wash water is neutral to methyl orange indicator.

**C.2.2** Mix the ether solutions (if necessary, filter previously washing the paper with ether) in a suitable, weighed vessel, evaporate on a boiling water-bath to a volume not less than 50 mL, add 100 mL of ethyl alcohol and about 0.5 mL phenolphthalein indicator and titrate to exact neutrality with standard sodium hydroxide solution.

#### **C.3 Calculation**

**C.3.1** Ascertain the mass of soda soap and calculate as percentage on the mass of the material taken for the test. This mass of the soda soap includes that of any mineral oil, unsaponifiables and neutral fat which, if determined separately, should be deducted from the results to obtain the true soap. If desired, calculate the combined sodium oxide ( $\text{Na}_2\text{O}$ ) and deduct from the mass of the soda soap to give the acid anhydrides.

**C.3.2** If the original soap is a potash soap, combined alkali may be calculated as potassium oxide (K<sub>2</sub>O).

**C.3.3** If the soap shows an excess of free acid, proper corrections should be made in calculating the combined alkali in the original soap. A blank test should be made on the sodium hydroxide or potassium hydroxide solution for neutral salts and correction made, if necessary, by exactly neutralizing 20 mL of the standard sodium hydroxide solution with standard hydrochloric acid, using phenolphthalein indicator. Evaporate the neutral solution on a water-bath and dry to constant mass at 105 °C ± 2 °C. From the mass of the residue subtract the mass of sodium chloride (NaCl) calculated from the amount of hydrochloric acid used up. This difference will be due to neutral salts which should be calculated per mL of the caustic solution.

**C.3.4** The combined alkali, expressed as percent by mass, shall be calculated as follows:

$$\frac{V N E}{M}$$

where

*V* is the volume, in millilitres, of the standard sodium hydroxide solution required for the material;

*N* is the normality of standard sodium hydroxide solution;

*E* = 4.00, 3.10 or 4.71, depending upon whether the result is to be expressed as sodium hydroxide (NaOH), sodium oxide (Na<sub>2</sub>O) or potassium oxide (K<sub>2</sub>O) respectively; and *M* is the mass, in grams, of the material taken for the test.

**C.4** For soaps containing a large amount of soluble silicates and other builders, and soap products containing a high percentage of finely divided material insoluble in water, the procedure under **C.2** cannot be applied as given. In such cases, use the filtrate prepared as follows:

Weigh accurately 2 g to 10 g of the soap sample and digest with 200 mL of freshly boiled ethyl alcohol in a covered vessel on a steam bath until the soap is dissolved then filter into a filter flask through filter paper till neutral to phenolphthalein, or through a sintered glass crucible with suction, protect the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. Evaporate the alcohol on a steam-bath, and proceed according to the instructions given under **C.3.1** and **C.3.2**.

## Annex D (normative)

### Determination of the potential wear

#### D.1 Principle

A bar of fixed dimensions cut from the product is first preconditioned by allowing it to stand on a damp cloth. The bar is held in a weighted holder, and is moved across a wetted fabric in a prescribed manner. The weight loss from the bar during rubbing is measured and expressed as grams lost per 10 metres of rubbed fabric.

For each bar type to be tested, three (3) consecutive wear measurements are made on three (3) bars of the type. If the bar size is too small to allow three consecutive wear measurements on each sample, then nine (9) individual samples of each product shall be tested.

#### D.2 Apparatus and Chemicals

##### D.2.1 For bar preparation

- (a) Coarse kitchen cheese grater;
- (b) Sharp thin bladed knife;
- (c) A weighted bar holder.

The holder and the weight shall be fixed together or shall separate. The total weight of the holder and weight shall be standardized to  $110 \pm 25$  grams.

##### D.2.2 For the test procedure

- (a) De-sized cotton cloth with a weave of close to 32 by 32 threads per cm. Cut to pieces 61 cm x 39 cm.
- (b) A tray of non corrodible metal or rigid plastic sized to accommodate the 61 cm x 39 cm test cloths.
- (c) A number of small flat bottomed trays of non corrodible metal or plastic (bar dishes) equal to the number of samples to be tested.
- (d) De-sized cotton cloth pieces to fit as a double layer into the base of the bar dishes.

##### D.2.3 For weighing

Balance to weigh 1 300 grams to 0.1 g if the bar and weighted holder shall be weighed together.

If the weight can be easily separated from the holder, then it is only the holder (typically not more than 100 grams) plus bar which need be weighed and the balance can have a smaller capacity.

### D.3 Bar preparation

**D.3.1** A test piece is cut from each bar. The test piece shall have a working face (to be applied to the fabric) of 3.75 cm x 7.5 cm.

The working face shall be a fresh surface from the interior of the bar sample.

The 7.5 cm length of the test piece shall be from a direction parallel to the longest axis of the original bar sample.

The height of the test piece is whatever is needed to fit securely into the test piece holder and to leave sufficient product below the holder to allow for wear during the test. Typically, the depth shall be 2 cm to 3.5 cm.

**D.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.

### D.4 Test procedure

**D.4.1 Preconditioning the test bar** — In this stage it is not necessary to weigh the bar.

**D.4.1.1** A small tray (bar dish) plus double thickness of cloth is filled with demineralized 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. drained to dripping.

**D.4.1.2** The cut bar piece is placed on the wetted cloth for 1 hour at room temperature, approximately 25 °C. This procedure allows the bar surface to absorb water (from mush) in a way characteristic of the product during use. The mushed test piece is fitted into a bar holder with the mushed face to the outside.

**D.4.1.3** The bar is dried lightly with tissue. The bar is now preconditioned to a state typically met during normal use. The main wear test can now be performed.

#### D.4.2 The wear test

**D.4.2.1** A fresh piece of test fabric is immersed in normal supply water and is then held vertically by the corners of a shorter edge. The cloth shall be held so that one of the corners is at the lowest point. Water shall drain from the cloth, initially in a continuous stream and eventually as drops. As soon as the continuous stream ceases and drops are formed, the cloth is placed flat on the test tray and smoothed to remove wrinkles, creases or bubbles. A shorter edge of the cloth shall be nearest the operator.

**D.4.2.2** The test bar and weighted holder, or the holder plus bar only, are weighed to the nearest 0.1 g.

**D.4.2.3** The test face of the preconditioned bar in the holder is placed towards one edge of the fabric and is then moved away from the operator towards the other shorter edge.

**NOTE** The effective cloth length shall be the distance moved by the leading edge of the bar, not the length of the cloth.

**D.4.2.4** The holder/bar are repositioned at the edge closest to the operator in a new position adjacent to the previous position and such that it is on fabric which was not rubbed in the previous step.

**D.4.2.5** Steps **D.4.2.3** and **D.4.2.4** are repeated such that, in total the bar is moved four (4) times along the length of the fabric.

**D.4.2.6** The bar is dried lightly with tissue and the weighed holder plus bar, or holder plus bar only, are re-weighed to give the weight loss during the fabric application.

**D.4.2.7** Provided the sample size is adequate, the wear test can be repeated with two more measures of wear on the same sample. The complete wear test is then repeated with two more examples of the product giving nine (9) measures of wear. If the sample size is not adequate to allow replicate tests on a given sample then nine individual samples shall be used.

**D.4.2.8** The average weight loss for movement over 4 times the effective cloth length is calculated for the nine samples of a given product, and then expressed as weight loss per metre of application to the fabric

## **Annex E** (normative)

### **Determination of the potential mush**

#### **E.1 Principle**

A test bar 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. The bar is left for six hours on a piece of fabric which has been wetted and drained of excess water. During the six hours, the bar/cloth is 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 sq.cm of original surface area in contact with the cloth.

#### **E.2 Equipment**

##### **E.2.1 For sample preparation**

a) Coarse kitchen cheese grater;

Sharp thin blade knife;

Callipers or ruler to ensure the sample dimensions

##### **E.2.2 Other equipment/materials**

a) plastic or non corrodible trays which are suitably sized for the test piece; and

plastic bar dishes 7 cm x 11 cm x 2 cm

Cotton cloth pieces are cut to fit as a double layer inside the trays. The cotton cloth used for wear measurements as specified in annex G shall be suitable.

#### **E.3 Bar preparation**

**E.3.1** Nine (9) individual bars shall be tested. A test piece is cut from each bar. The test piece shall have a working face (to be applied to the fabric) of 4.0 cm x 7.5 cm.

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

The 7.5 cm length of the test piece is from a direction parallel to the longest axis of the original bar sample.

**E 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.

### E.3 Test procedure

For each test piece:

**E.4.1** The tray plus double thickness of cloth is filled with de-mineralized 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.

**E.4.2** The area of the working face of the test piece is measured.

**E.4.3** The bar is preconditioned by giving 18 twists of 180 degrees under running supply water at 25 °C in a bowl of supply water at that temperature.

**E.4.4** The working face of the bar is placed onto the damp fabric and then the tray plus bar are covered, e.g. with a sealed plastic bag, to prevent water loss.

**E.4.5** The covered test piece and holder are maintained at 25 °C for 6 hours.

**E.4.6** The mushed bar test piece is removed from the tray and is weighed.

**E.4.7** Mush is removed from the working face of the bar test piece by scraping with a blunt sided spatula or plastic ruler.

**E.4.8** The test piece is reweighed and the amount of mush removed is calculated. The mush is expressed as grams per 30 sq. cm of original test piece surface area.

**NOTE** The procedure for weighing the bar and removing the mush shall take some minutes. During that time the remaining bars will continue to form mush. While this time is not critical for a set of nine (9) test pieces from a given product, if more than one product is under test, it is advisable to stagger the start of the test for the second product. This shall give adequate time to complete work on the first set before the 6 hour storage time of the subsequent set is completed.

## **Annex F** (normative)

### **Test for freedom from grit**

#### **F.1 Outline of the method**

Soap is rubbed with water between the hands for a specified time and examined for its gritty or rough feel, if any.

#### **F.2 Procedure**

**F.2.1** Hold the laundry bar under running tap water (at a temperature between 20 °C – 30 °C) and rub gently the two sides of the bar on the palm for one minute each. The bar shall show no rough surface and shall feel smooth to the touch. Allow the used bar to dry in the open for 4 hours and examine the surface.

**F.2.2** The soap shall be taken to have passed the test if there is no gritty or rough feel on the surface.

## Annex G (normative)

### Determination of cleaning index

#### G.1 Principle and instrumentation

The cleaning index expresses the potential of the washing bar to lift off pre-defined stain from a known fabric under defined conditions.

The 'Cleaning Index' is a ratio of the surface tension of clean water to the difference between the surface tension of a standard washing bar solution in which a defined stained cloth is soaked for a defined time; and the surface tension of the standard concentration of the washing bar (detergent) in water.

Nu Nuoy Tensiometer (Cambridge Instrument Company Ltd.) shall be used to determine the surface tension (or any such suitable surface tension measuring equipment).

In this set up, a platinum iridium ring on the surface of the liquid is supported by a stir-up attached to the beam of the torsion balance. The ring is pricked upwards from the liquid by turning the torsion wire, thus applying a force which is known from calibration of the instrument for an idealized system. The force necessary to separate the ring from the liquid is equal to  $4\pi R\gamma$ , where  $R$  is the mean radius of the ring as shown in figure 1

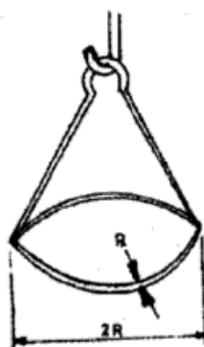


Figure G.1 — Platinum iridium ring

Doubling of the perimeter arises from the fact that there are two boundary lines between liquid and wire, one on the outside and one on the inside of the ring. The shape of the liquid held up influences the force necessary for breaking away

The shape is a function of  $R^3/V$  and  $R/r$  where  $v$  is the volume of the liquid held up and  $r$  is the radius of the wire. The surface tension is thus given by the equation:

$$\gamma = \frac{f}{4\pi R} F$$

where,

$f$  = maximum force registered on a torsion balance scale;

$F$  = correction factor due to shape of liquid held up and the ring dimensions.

Over extreme variations of  $R^3/V$  and  $R/r$ ,  $F$  varies between about 0.75 and 1.02. In ordinary cases it is close to 1.

## G.2 Sample solutions

**G.2.1 Preparation of stained cloth** — The staining solution consists of ethyl acetate analytical grade engine oil in the volume ratio of 4:1.

NOTE This method was developed using Caltex motor oil SAE 40.

Pieces of cotton cloth measuring 4 cm x 4 cm are then stained being soaked in the staining solution for 60 minutes (ensure uniform staining by continuously swirling the beaker). The cloth pieces are then dried in an oven at 80 °C for 48 hours.

**G.2.2 Detergent solutions** — These are prepared by dissolving 1.0 g of each bar sample in 300 mL of hot water (ordinary tap water) and left to stand for 21 hours.

Surface tension measurements of these solutions are then recorded to give  $y_{Det}$ .

Stained cloth pieces prepared in (G.2.1) are then soaked in 50 mL of the washing bar solution and let to stay for 21 hours.

The surface tension of the soiled solution is then determined to give  $y_{Det/Stain}$ .

The clearing index (C.I) is then computed from the expression:

$$C.I = \frac{y_{H_{20}}}{y_{Det/Stain} - y_{Det}} \times 10^{-1}$$

or

$$C.I = \frac{y_{H_{20}}}{\Delta y} \times 10^{-1}$$

where,

$y_{H_{20}}$  = the surface tension pure water; and

$$\Delta y = y_{Det/Stain} - y_{Det}$$

## Annex H (normative)

### Determination of Lather volume

#### H.1 Principle

A domestic kitchen blender is adjusted to deliver  $600 \pm 100$  mL of lather in 60 seconds from a 1 per cent solution of sodium lauryl sulphate in 300 ppm calcium hard water at 25 °C.

The bar is transformed into uniform 2 mm diameter noddles by hand extrusion through a perforated plate. 100 mL of 300 ppm calcium hard water at 25 °C and 5 grams of bar noodles are agitated in the adjusted kitchen blender for 60 seconds and the lather volume is measured. The bar lather is normalized to a volume of 600 mL from the SLS solution tested in the same blender.

#### H.2 Apparatus

**H.2.1 Kitchen food blender** — Hamilton beach commercial bar mixer with a low speed of 14 000 rpm, high speed of 18 000 RPM. It shall be operated at a speed which shall deliver a lather volume of  $600 \pm 100$  mL from a standard solution on sodium lauryl sulphate defined in Clause H.3. The test procedure is described in H.5.

**H.2.2 Bar extruder** — The bar extruder consists of a block of metal 10 cm x 10 cm x 50 cm drilled centrally through the longer axis with a 25 mm diameter hole. The hole is threaded and a matching threaded bolt is provided. A plate with well separated 2 mm diameter holes is bolted to one end of the metal block.

Any device which shall extrude bar through a plate with 2 mm diameter holes is quite suitable. The equipment consists of a block of metal 10 cm x 10 cm x 50 cm drilled centrally through the longer axis with a 25 mm diameter hole. The hole is threaded and a matching threaded bolt is provided. Plate with well separated 2 mm diameter holes is bolted to one end of the metal block.

Materials of construction, screw thread and dimensions other than the end plate holes are not critical. To ease cleaning of the equipment and to make extrusion easier, the end plate shall not be too thick. A 3 mm thickness plate is sufficiently robust.

For use, the device is conveniently clamped vertically in a bench vice and the bolt is turned by hand, or for higher time or aged products with a spanner or bar. The lower end is encased in a plastic bag held in place by elastic or tape to collect the bar noodles extruding from the end plate.

#### H.2.3 Other equipments and chemicals

Other equipments and chemicals are as follows:

- b) coarse cheese grater;
- c) measuring cylinder 1 000 mL;
- d) volume equipment/balance for hard water preparation; and
- e) sodium lauryl sulphate, that shall have minimum 99 per cent purity,  $C_{12}$  compounds by GLC shall be a minimum of 98 per cent.

The impurity level shall be as given in Table H.1 below.

**Table H.1 — Impurity level**

S/N	Maximum limit of impurities	Percent
i.	Loss on drying at 110 °C	1
ii.	Acidity or alkalinity	0.5 mL N %
iii.	Chloride	0.03
iv.	Phosphate as PO <sub>4</sub>	0.001
v.	Copper as Cu	0.0005
vi.	Iron as Fe	0.0001
vii.	Lead as Pb	0.0005

### H.3 Sodium lauryl sulphate solution

Prepare a 1% solution of sodium lauryl sulphate (SLS) in 300 ppm calcium hardness water. Solution more than 1 week old shall not be used.

### H.4 Sample preparation

**H.4.1** The requirement is for a lather measurement on three samples from each of three bars taken during the sampling procedure 6.

**H.4.2** Approximately 100 g of each bar is coarsely grated with a kitchen cheese grater. The gratings are quickly mixed by hand and then transferred to a labelled container which is closed to prevent moisture loss.

**H.4.3** Grated bar from the sample is introduced into the threaded barrel of the extruder described above. Sufficient bar shall be used to ensure production of at least 20 grams of coherent noodles by the procedure described in Clause H.2. The noodles from each bar shall be divided into 3 equal parts for the subsequent lather tests.

### H.5 Lather measurement

**H.5.1** All test shall be performed at 25 °C ± 2 °C.

**H.5.2 Lather from the standard SLS solution** — 100 mL of the standard SLS solution is poured into the jar of the kitchen food blender, the jar is covered and the blender is run for exactly 60 seconds. Immediately the blender jar is inverted over a 1 litre measuring cylinder and the liquid and foam are allowed to drain. A large plastic spatula or ruler shall be used to scoop residual foam from the jar into the measuring cylinder. The top of the foam is levelled off and the total volume of material in the cylinder is taken as the lather volume value, i.e. with no allowance for any liquid volume which may separate from the foam.

If the foam volume is not 600 ± 100 mL, adjust the mixer speed and repeat the test until a foam volume in this range is achieved.

Once the blender speed setting is confirmed, it shall be used for all subsequent tests on SLS solution and bar samples.

**H.5.3 Lather from bar sample** — Pour 100 mL of 300 ppm calcium hardness water into the blender jar. Add 5 grams of extruded bar noodles prepared as in B.4. Cover the jar and operate the blender for exactly 60 seconds at the speed identified in G.5.2 as giving 600 ± 100 mL of foam with the standard SLS solution. Quickly pour the foam and any residual liquid into a 500 mL or 1 litre measuring cylinder.

## H.6 Notes for guidance

### H.6.1 Temperature

Although an ambient temperature and equipment shall be maintained at  $25 \pm 2$  °C, this is not always easy to achieve. It is essential to measure the temperature of the SLS or bar foam after the volume measurement. If the temperature recorded is outside the range  $25 \pm 2$  °C, the test shall be repeated with the starting SLS solution or water temperature adjusted so that the final temperature of the foam is within the specified range

### H.6.2 Number of lather volume measurements

For each bar type to be tested, three (3) measurements are made on the noodles produced from three (3) bars of the type.

### H.6.3 Sequence of lather tests

Once the kitchen food blender is established as delivering  $600 \pm 100$  mL, the SLS solution and noodles prepared from each of 3 bars of the type the sequence of testing shall be as follows:

Lather volume SLS;  
Lather volume on noodles from bar 1;  
Normalize the bar lather to an SLS volume of 600 mL =  $V1/1$ ;

Lather volume SLS;  
Lather volume on bar noodles from bar 1;  
Normalize the bar lather to an SLS volume of 600 mL =  $V2/1$ ;

Lather volume SLS,  
Lather volume on bar noodles from bar 1;  
Normalize the bar lather to an SLS volume of 600 mL =  $V3/1$ .

The average of  $V1 - 3/1$  volumes is the sample value for bar 1.

This procedure is continued for bars 2 and 3 taken from the same sample lot. The average of the three sample values shall be reported as the lot value.

### H.6.4 Normalizing lather volumes

$$\text{Normalized bar volume} = \text{Actual bar volume} \times \frac{600}{\text{Actual SLS volume}}$$

## ANNEX J (Informative)

### Permitted structuring and processing aids

Following is a list of structuring and processing aids (excluding colouring matter, perfume, preservatives, opacifiers, and optical brightening agents) that shall generally be used in washing bars.

- a) starch and derivatives,
- b) cellulose and derivatives,
- c) mannitol,
- d) dextrin,
- e) kaolin,
- f) talc,
- g) bentonite,
- h) calcite/  $\text{CaCO}_3$ ,
- i) borax,
- j) soda ash,
- k) vegetable/animal fatty acid,
- l) silicates,
- m) sodium chloride,
- n) sodium sulphate,
- o) dolomite,
- p) fatty alcohol,
- q) fatty acid ethanolamide,
- r) diethylene glycol monostearate,
- s) paraffins,
- t) polyoxyethylene glycol; and
- u) glycerol monostearates

## Annex K (normative)

### Sampling

#### 1 Procedure

K.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 K.1.

Table K.1 — Scale of sampling

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

K.1.2 The packages shall be selected at random, using tables of random numbers. If these are not available, the following procedure shall be applied:

Starting from any package, count all the packages in one order as 1, 2, 3, ..., *N*, selecting every *k* th package,

Where

*k* is the integral part of  $N / n$ .

K.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.

K.1.4 Preparation of test samples

K.1.5 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.

K.1.6 Samples for testing

Immediately after preparation of composite sample (K.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.

FOR PUBLIC COMMENT ONLY

## Annex L

(normative)

### DETERMINATION OF ACTIVE (RESERVE) ALKALINITY

#### L.1 APPARATUS

L.1.1 pH Meter

L.1.2 Beaker, 100 ml capacity L.1.3 Magnetic Stirrer

L.1.4 Burette

#### L.2 REAGENTS

L.2.1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be used in tests.

NOTE — "Pure chemicals" shall mean chemicals that do not contain impurities which affect the results of analysis.

L.2.2 Hydrochloric Acid — 0.1 N.

#### L.3 Procedure

Weigh 10.0 g sample and dissolve it in 1 000 ml of distilled water. Pipette out 50 ml aliquot in 100 ml beaker. Place the beaker containing 1 percent solution of detergent product in aqueous vehicle on a magnetic stirrer and mix the contents thoroughly. Note down the pH of the solution using a pH meter. With the electrode of the pH meter dipping in the solution and keeping the pH meter 'ON', add drop by drop 0.1 N hydrochloric acid from a burette till the pH of the solution drops to 8. While adding hydrochloric acid, stir the solution continuously. Note the amount of 0.1 N hydrochloric acid required to bring down the pH of the solution to 8 which is a measure of the active alkalinity of the test sample. Mean of 2 replicate measurements will give active (reserve) alkalinity expressed as amount in ml of 0.1 N hydrochloric acid.

**Annex M**  
**(normative)**

**PROCEDURE FOR DETERGENCY TEST (POWDERS)**

**M.1 Outline of the method**

The method prescribed here is based on the use of Terg-O-tometer. Cloth is artificially soiled and the soil is removed by washing the soiled swatches of cloth with a solution of the detergent powder to be evaluated under standard conditions. A control detergent powder (see below for details) is also run simultaneously. The reflectance of the unsoiled, soiled and washed swatches is measured instrumentally using a standard photoelectric reflection meter. The detergency is expressed as percentage of soil removal.

**M.2 Apparatus**

**M.2.1 Photoelectric Reflection Meter**

With built-in galvanometer and tungsten lamp as an illuminant.

**M.2.2 Terg-O-tometer**

With a battery of four or six agitator washers in 2 | stainless steel beakers. The angle through which the agitators oscillated is 350°. The speed of rotation is adjustable and set at 100 strokes per minute, each back and forth movement representing one stroke. The beakers are fully immersed in an electrically controlled water-bath. The agitators and beakers are removable.

**M.2.3 Cloth Soiling Machine**

Electrically operated mangle with variable pressure arrangements which can be recorded and variable speed drive with attached air-drying chamber fitted with exhaust.

**M.3 Preparation of standard soiled cloth test specimens**

Prepare soiled cloth swatches as per 6 of IS 5785 (Part 4).

**M.4 Control powder formulation**

AD	19
STTP	05
Zeolite	10
Co-builder-Enzyme	0.4
Soda	10
Alk silicate	10
SCMC	1
Na <sub>2</sub> SO <sub>4</sub>	40
Moisture	4.6
	-100

## M.5 Washing procedure

**M.5.1** The ratio of cloth (in g) to the volume of solution (in ml) shall be 1:100.

**M.5.2** Prepare 1L solution of 0.5 percent in 300 ppm water considering the basis of product concentration used in washing. Introduce 5 soiled swatches into beakers and agitate. Wash the specimens for exactly 10 min at  $27 \pm 2^\circ\text{C}$ .

**M.5.3** Remove the beaker from the water-bath and decant the solution and rinse 3 times with 300 ppm water.

**M.5.4** Repeat with 5 more wash loads of 5 swatches each, for each formulation and control powder. Total of six replicate washings is given so that at the end, all the formulations are washed in all beakers.

**M.5.5** Air dry washed swatches and measure their reflectance.

## M.6 Evaluation of soil removal

Fold the desired unsoiled cloth, soiled swatches and washed swatches into four folds so as to minimize the effect of colour of background and take reflectance measurements. Operate the reflectometer in accordance with the instruction supplied with the instrument.

## M.7 Calculation and reporting

The detergency value expressed as percentage soil removed is calculated from the following equation.

$$\text{Percentage of soil removal} = \frac{W_o}{S_o} \times 100$$

Where,

$$W_o = \frac{(100-R_w)^2}{200R_w} - \frac{(100-R_3)^2}{200R_3};$$
$$S_o = \frac{(100-R_c)^2}{200R_c} - \frac{(100-R_3)^2}{200R_3}; \text{ and}$$

$R_c$ ,  $R_w$  and  $R_3$  are reflectances of clean, washed and soiled fabric pieces respectively.

Analysis of variance to be used for the handling of data and obtaining significant differences.

## M.8 Treatment of data and reporting

Assign a value of 70 percent detergency to control powder. Normalise the detergency values of products by applying a correction factor, that is 70 percent detergency value for control powders and report the normalised detergency value.

**Annex N**  
**(normative)**

**DETERMINATION OF ASH BUILT UP ON FABRICS**

**N.1 General**

This test determines the built-up of ash on a fabric under the condition of test.

**N.2 Reagents**

**N.2.1 Sodium Hexametaphosphate**

**N.2.2 Hard Water**, of 300 ppm (as CaCO<sub>3</sub>), made by adding 2.96 g of MgSO<sub>7</sub>HO and 2.64 g of CaCl<sub>2</sub>.2H<sub>2</sub>O to 10 l of water.

**N.2.3 Bleached cotton Long Cloth** (125 ± 25 g/m<sup>2</sup>)

**N.3 Equipment**

**N.3.1 Washing Appliance**, like standard Terg-O-tometer.

**N.3.2 Air Oven**, with arrangements for drying of washed swatches by hanging.

**N.4 Procedure**

**N.4.1** Cut the cotton cloth into swatches of 10 cm x 10 cm size. 10 swatches are required for each set.

**N.4.2** Number the raw swatches and wash them 2 times in a Terg-O-tometer with sodium hexametaphosphate solution (3 g/l) to remove the original salts present in the swatches. Rinse the swatches thoroughly with distilled water. Dry the swatches in an air oven at 105°C.

**N.4.3** Stir 10 prewashed swatches (see N.4.2) in 1 litre solution of 0.2 percent concentration of the test detergent product prepared in 300 ppm hard water at 27 ± 2°C for 20 min in a Terg-O-tometer. Rinse the swatches in 1 litre hard water of hardness 300 ppm by stirring in the Terg-O-tometer for 10 min at 27 ± 2°C.

**N.4.4** Dry the swatches at 50°C in an air oven. Repeat the washing, rinsing and drying operations 25 times. After 25 washes, dry the swatches in an air oven at 105°C. Determine the ash content of both prewashed (see N.4.2) and after washed fabric with detergent sample as explained above at 800°C

## Annex P (normative)

### Determination of total fatty matter and synthetic surface-active agents

#### P.1 Outline of the method

**P.1.1** The test method of analysis for total fatty matter (TFM) given in ISO 685 pertains to the analysis of fatty matter for soaps. In the context of a wider definition of TFM relevant to bathing bar, which may contain synthetic actives in addition, it becomes necessary to quantify the fatty chain(s) linked with synthetic surface-active agents and include their contribution to the TFM.

**P.1.2** Fatty chain anionic surfactants or soaps undergo hydrolysis when refluxed with mineral acids to give corresponding fatty matter which can be extracted by petroleum ether. Non-fatty anionics like LAS and AOS do not undergo hydrolysis and therefore do not interfere in the analysis.

**P.1.3** In this method, total synthetic surface-active agents and TFM are estimated independently by gravimetry.

#### P.2 Principle

**P.2.1** Since several synthetic surfactants are permitted to be used in bathing bar formulations, there is wide variation in the molecular weight range of these surfactants. Estimation of individual surfactants is not necessary from the analytical point. The analytical strategy involves the extraction of all surfactants (soaps, non-ionics, anionics and amphoteric) in 85 % alcohol. An aliquot of 85 % alcohol soluble matter is extracted with petroleum ether to remove hydrophobic constituents (perfume, unsaponifiable matter etc.) and the 85 % alcohol fraction is evaporated to dryness. The residue is extracted with 1:1 tetrahydro furan: petroleum ether mixture and filtered. The filtrate is evaporated and the residue weighed as synthetic surfactant. Another aliquot of the 85 % alcohol soluble portion is refluxed with 2 N sulphuric acid and extracted with petroleum ether. The residue obtained after evaporation of petroleum ether is the TFM comprising of free fatty acids, fatty matter from soap, as well as fatty matter associated with other surfactants.

**P.2.2** The analytical strategy involves the extraction of all surfactants (soaps, non-ionics, anionics and amphoteric) in 85 % alcohol. An aliquot of 85 % alcohol soluble matter is extracted with petroleum ether to remove hydrophobic constituents (perfume, unsaponifiable matter etc.) and the 85 % alcohol fraction is evaporated to dryness. The residue is extracted with 1:1 tetrahydro furan: petroleum ether mixture and filtered. The filtrate is evaporated and the residue weighed as synthetic surfactant. Another aliquot of the 85 % alcohol soluble portion is refluxed with 2 N sulphuric acid and extracted with petroleum ether. The residue obtained after evaporation of petroleum ether is the TFM comprising of free fatty acids, fatty matter from soap, as well as fatty matter associated with other surfactants.

**P.2.3** Figure C.1 illustrates the analytical strategy of total fatty matter and synthetic surface active agents.

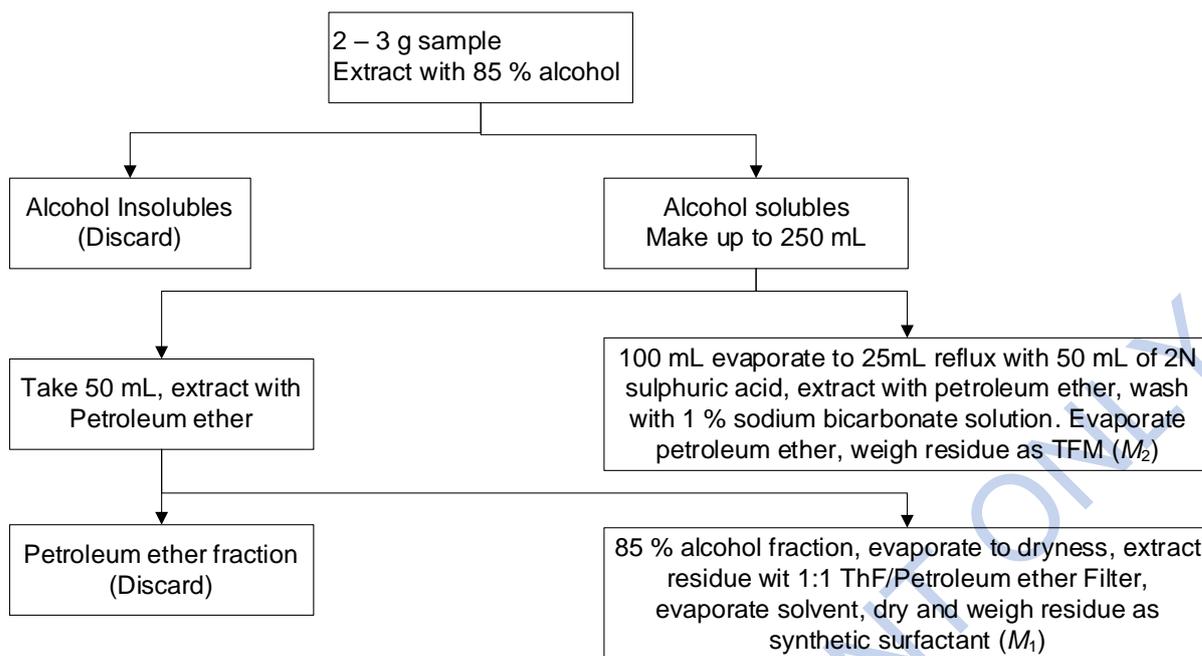


Figure C.1 — Analytical strategy for total synthetic surfactant and total fatty matter

### P.3 Reagents

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

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

#### P.3.2 Sulphuric acid, approximately 2 N

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

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

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

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

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

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

#### P.3.5 Sodium hydroxide, approximately 0.1 N

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

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

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

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

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

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

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

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

**P.3.10 Acetone**, AR Grade

**P.3.11 Tetrahydrofuran (THF)**, AR Grade

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

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

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

## **P.4 Apparatus/glassware**

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

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

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

**P.4.4 Separating funnel**, 500-mL capacity

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

**P.4.6 Air condenser**, length 1 m

**P.4.7 Conical flask**, 500-mL capacity

**P.4.8 Funnels**

**P.4.9 Glass rods**

**P.4.10 Flash rotary evaporator/distillation set-up**

**P.4.11 Whatman Filter Paper No. 541**

**P.4.12 Air oven**

**P.4.13 Steam bath**

## **P.5 Procedure**

### **P.5.1 Preparation of sample**

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

## P.5.2 85 % alcohol extraction

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

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

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

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

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

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

## P.5.3 Estimation of synthetic surfactants

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

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

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

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

**P.5.3.5** Evaporate the alcoholic solution from P.5.3.4 to dryness.

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

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

**NOTE 2** Use only Whatman filter paper No. 541 (porosity of 20  $\mu\text{m}$  to 25  $\mu\text{m}$ ).

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

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

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

**P.5.3.10** Dry the beaker/round bottom flask containing the residue in an air oven at 105 °C for 1 h. Cool to room temperature inside a desiccator and weigh to constant weight ( $M_1$ ).

## P.5.4 Estimation of total fatty matter (TFM)

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

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

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

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

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

**CAUTION** — Use safety glasses during extraction.

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

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

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

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

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

## P.6 Calculation

### P.6.1 Total synthetic surface actives

The total synthetic surface actives, expressed as percent, shall be calculated as follows:

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

where

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

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

### P.6.2 Total fatty matter

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

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

where

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

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

FOR PUBLIC COMMENT ONLY

## **Bibliography**

IS: 286 – 1978 (Reaffirmed 2008), Methods of sampling and test for soaps

EAS 31: 2021 Laundry soap

RS 88: 2021 Washing bars — Specification

IS 8180 : 2020: Household Laundry Detergent Bars

FOR PUBLIC COMMENT ONLY