

**भारतीय मानक**  
**Indian Standard**

**IS 13498 : 2017**  
**(Reaffirmed 2022)**

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## नहाने की बट्टी — विशिष्टि

( दूसरा पुनरीक्षण )

## Bathing Bar — Specification

( Second Revision )

ICS 71.100.40

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भारतीय मानक ब्यूरो  
BUREAU OF INDIAN STANDARDS  
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली-110002  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI-110002  
[www.bis.org.in](http://www.bis.org.in) [www.standardsbis.in](http://www.standardsbis.in)

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Price Group 6

## Soaps and Other Surface Active Agents Sectional Committee, CHD 25

### FOREWORD

This Indian Standard (Second Revision) was first published in 1992 and based on the feedback received from the users; it was further revised in 1997. The concerned technical Committee felt that the present Indian Standard on Toilet soap IS 2888, which is mainly based on composition, does not necessarily correspond to performance of toilet soaps. The performance of soap depends more on the type of fatty matter present rather than the total fatty matter of the soap. For example, the solubility of soap depends on the characteristics of fatty acids, namely chain length or saturation and on the cation. Further, the use of acceptable non-soap Surface Active Agents would result in the substitution of oils and fats which are scarce resource. It had therefore been felt desirable to formulate a separate specification for a bathing bar which may contain soaps of fatty acid and non-soapy surfactants, but ensure the performance and safety of the product to the consumer on use.

The important in-use criteria for bathing bars are safety, cleaning and economy. With regard to performance, it is important to guard against the removal of the beneficial skin proteins by the bathing bar and over-cleaning resulting in defatting of skin. This is ensured in the standard by allowing only such ingredients which are safe for use in personal washing products. Bathing bars may be used as a personal cleansing bar.

Since the last revision of this standard in 1997, new technological advances have taken place in the field of soap formulation and processing, which would enable optimization of TFM and synthetic surface active agent content at even lower levels with assurance of safety and skin compatibility.

The safety requirement has also been strengthened by specifying that manufacturers should test the product and satisfy themselves about the safety of the product to human skin as prescribed in the standard and make a declaration to that effect on the label.

In the modern day context, it is essential that synthetic surface active agents, used for manufacturing the product are eco-friendly in respect that these are ultimately biodegradable in the sewer system. Hence a requirement has been included to ensure the same.

In this revision, besides amalgamation of all amendments, a parameter of "Fatty matter from dissolved actives" is introduced as an alternative to TFM. Any of the requirement among these two could be followed. The amount of actives dissolved in water is important for cleansing and lathering during bathing instead of the total TFM content in a soap bar. Test method for determination of Fatty Matter from Dissolved Actives has been prescribed and test method for determination of cleaning efficiency test has also been updated. The standard is also performance-based to ensure safety and skin compatibility. The cleansing efficacy has been strengthened through introduction of particulate soil in addition to oily soil from the previous version of standard. The safety of the bar would be assured through safety testing.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical value (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

## BATHING BAR — SPECIFICATION

### *( Second Revision )*

#### 1 SCOPE

This standard prescribes requirements, methods of sampling and test for bathing bar.

#### 2 REFERENCES

The standards given in Annex A contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate by dated or undated the possibility of applying the most recent editions of the standards.

#### 3 TERMINOLOGY

**3.1** For the purpose of this standard, the definitions given in IS 286 and IS 7597 shall apply, in addition to the following.

**3.1.1 Bathing Bar** — The bathing bar shall be a product containing soap of fatty acids and synthetic surface active agents as active ingredients and which could be used for bathing purposes in soft and hard water.

**3.1.2 Fatty Matter from Dissolved Actives** — Fatty matter from dissolved actives refers to the amount of actives (soluble soap and surfactant) dissolved in water which is important for cleansing and lathering during bathing.

#### 4 MATERIALS

##### 4.1 General

The bathing bar shall contain adequate quantity of one or more ingredients listed in 4.2, in addition to bar structuring and processing aids given in Annex B.

##### 4.2 Ingredients

**4.2.1** Bathing bar shall contain soap of fatty acids and optionally one or more of the following surface active agents conforming to the relevant Indian Standards or manufacturer's specification (in case no Indian Standard exists) subject to compliance of requirement prescribed in 4.2.1.1.

Fatty acid ester sulphonates, fatty alkanolamides, sarcosinates, taurides, fatty isothionates, alpha olefin sulphonates, alcohol sulphates, amphoterics such as

betaines, fatty alcohol ethoxy sulphate, linear alkyl benzene sulphonates (LAS), alkyl poly glycosides (APG), fatty alcohol sulphosuccinate and fatty alkanol amido sulphosuccinate.

**4.2.1.1** The synthetic surface active agents shall pass the biodegradability test as given in IS 13933.

**4.2.2** In addition to the surface active agents and perfume, the bathing bar may contain other ingredients such as bar structuring and processing aids, colouring matter, permitted antioxidants, preservatives [see IS 4707 (Part 1) and IS 4707 (Part 2)] super fatting agents, humectants and such additional substances are declared on the label. A list of bar structuring and processing aids is given in Annex B. The quality of the material shall be according to relevant Indian Standards (as mentioned) or manufacturer's specification (in case no Indian Standard exists). All these materials shall be non-injurious to skin in use with the bathing bar. It is necessary that the raw materials used are such that in the concentrations in which they would be present in the finished bathing bar, after interaction with the other raw materials used in the formulations, are free from any harmful effects. For determining the suitability of a new formulation or of a new raw materials used in an old formulation on the skin and in respect of eye irritation, reference may be made to IS 4011. It shall be the responsibility of the manufacturer to satisfy themselves of the dermatological safety of their formulation according to IS 13424.

However, formulations containing novel ingredients shall require submission of the safety evaluation data based on alternative non-animal test methods for skin sensitization test to the concerned State Licensing Authority for their consideration and approval.

#### 5 REQUIREMENTS

**5.1** The bathing bar shall be firm and smooth. It shall also comply with the requirements given in Table 1.

**5.2** The formulation when tested by the method given in IS 13424 shall pass the dermatological safety test.

#### 6 PACKAGING AND MARKING

##### 6.1 Packaging

The bathing bar shall be packed as agreed to between the purchaser and the supplier.

## IS 13498 : 2017

**Table I Requirements for Bathing Bar**

(Clauses 5.1, 7.3 and 8.1 )

Sl No.	Characteristics	Requirements	Methods of Test, Ref to	
			Annex	Cl of IS 286
(1)	(2)	(3)	(4)	(5)
i)	Lather in ml, <i>Min</i>	200	C	—
ii)	Mush (loss in mass due to mashing), g/50 cm <sup>2</sup> , <i>Max</i>	15	D	—
iii)	Free caustic alkali (as NaOH), percent by mass, <i>Max</i>	0.05	—	6
iv)	Free carbonated alkali, percent by mass, <i>Max</i>	1.0	—	28
v)	Freedom from grittiness	To pass the test	E	—
vi)	Freedom from cracking	To pass the test	F	—
vii)	Cleaning efficiency	To pass the test	G	—
viii)	Fatty matter [product to comply with either (a) or (b)]			—
	a) i) Total fatty matter, percent by mass, <i>Min</i>	40	H	
	ii) Synthetic surface active agent, percent by mass, <i>Min</i>	4.0	H	
	b) Fatty matter from dissolved actives, <i>Min</i>	8.0	I	

## 6.2 Marking

The packages shall be securely closed and marked with the following:

- Name of the product, namely 'bathing bar' in a conspicuous manner (under the brand name);
- Manufacturer's name;
- Brand name of the material and recognized trade-mark, if any;
- Net mass when packed (packing should be in accordance with packing rules);
- Batch number or lot number in code or otherwise; wherever possible;
- Year and month of manufacture;
- A statement that the material has been tested for dermatological safety and found safe to human skin; and
- Following ingredients mentioning the actual

compound in descending order up to a limit of 0.5 percent by mass:

- Total fatty matter,
- Surface active agent, and
- Matter insoluble in alcohol.

## 6.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

**6.2.1.1** The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder and revised from time to time. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 7 SAMPLING

**7.1** The general precautions in sampling, scale of sampling and preparation of test samples shall be as prescribed in **3.1**, **3.2**, and **3.3** respectively of IS 286.

## 7.2 Number of Tests

Tests for all the characteristics shall be conducted on each one of the individual samples separately.

## 7.3 Criteria for Conformity

For each of the characteristics, the mean ( $\bar{X}$ ) and the range ( $R$ ) of the test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{the sum of test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{the difference between the maximum and the minimum value of test results.}$$

The test shall be deemed as conforming to requirements of this standard if the expression ( $\bar{X} - 0.6R$ ) is greater than or equal to minimum value given in Table 1 and ( $\bar{X} + 0.6R$ ) is less than or equal to maximum value given in Table 1.

## 8 TESTS

**8.1** Tests to evaluate the characteristics prescribed in Table 1 shall be conducted as prescribed in Annexure C to I and also IS 286 and IS 4955.

## 8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

## ANNEX A

### (Clause 2)

#### LIST OF REFERRED INDIAN STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
253 : 2014	Specification for common salt	4654 : 1993	Paraffin wax ( <i>second revision</i> )
255 : 1982	Sodium sulphate anhydrous (technical grade)	4707	Classification for cosmetic raw materials and adjuncts:
286 : 1978	Methods of sampling and test for soaps ( <i>second revision</i> )	(Part 1) : 2001	Dyes, colours and pigments ( <i>first revision</i> )
321 : 1964	Absolute alcohol ( <i>revised</i> )	(Part 2) : 2009	List of raw materials generally not recognized as safe for use in cosmetics ( <i>first revision</i> )
323 : 2009	Rectified spirit for industrial use		
878 : 2008	Laboratory glassware — Graduated measuring cylinders ( <i>first revision</i> )	4955 : 2001	Household laundry detergent powders ( <i>third revision</i> )
918 : 1985	Calcium carbonate precipitated for cosmetic industry	5785	Methods of performance tests for surface active agents : Part 4
1070 : 1992	Reagent grade water ( <i>third revision</i> )	(Part 4) : 1976	Relative detergency ( <i>first revision</i> )
1462 : 1985	Talc for cosmetic industry	6367 : 1971	Dextrin for adhesive industry
1463 : 1983	Kaolin for cosmetic industry	7597 : 2001	Surface active agents — Glossary of terms
2888 : 2004	Toilet soap ( <i>second revision</i> )	9601 : 1980	Sodium silicate for cosmetic industry
3986 : 1988	Sodium lauryl sulphate for cosmetic industry ( <i>second revision</i> )	11023 : 1984	Borax for cosmetic industry
		13424 : 2001	Safety evaluation of bathing bar and toilet soap — Method of test
4011 : 1997	Methods of tests for safety evaluation of cosmetics ( <i>first revision</i> )	13933 : 1995	Method of test for biodegradability of surface active agents (modified sturm test)

## ANNEX B

### (Clauses 4.1 and 4.2.2)

#### LIST OF BAR STRUCTURING AND PROCESSING AIDS USED IN BATHING BARS

Following is the list of structuring and processing aids used generally in bathing bars:

Borax (*see* IS 11023)

Cellulose and derivatives

Dextrin (*see* IS 6367)

Diethylene glycol monostearate

Fatty alcohol

Fatty acid ethanol amide

Glycerol monostearates

Kaolin (*see* IS 1463)

Paraffin (*see* IS 4654)

Polycarboxylates

Polyoxyethylene glycol

Polyols

Polyphosphates, pyrophosphate, hexametaphosphates

Precipitated calcium carbonate (*see* IS 918)

Sodium chloride (*see* IS 253)

Sodium silicate (*see* IS 9601)

Sodium sulphate (*see* IS 255)

Starch and its derivatives

Talc (*see* IS 1462)

IS 13498 : 2017

## ANNEX C

[ Clause 5.1 and Table 1, Sl No. (i) ]

### DETERMINATION OF LATHER (KITCHEN FOOD BLENDER METHOD)

#### C-1 APPARATUS

##### C-1.1 Kitchen Food Blender

It consists of a three speed kitchen blender with a glass container of 1 000 ml capacity, one 1 000 ml and one 500 ml capacity graduated measuring cylinder (*see* IS 878). (The liquidizer blade type that is usually fixed to a Philips mixie is suitable.)

#### C-2 PREPARATION OF SAMPLE

##### C-2.1 Particle Size Reduction of Sample

The bathing bar sample shall be grated through a 1.70 mm sieve (*see* Note). It may be necessary to clean the sieve more often during grating in the case of bathing bar that are soft and sticky. If the bar is too dry and gets powdery during the grating operation, a second sieving of the gratings through a 500 micron sieve is needed and particles passing through this sieve shall be rejected. The sieved gratings shall be stored in air tight containers before use in lather measurements.

NOTE — Reduction of particle size of bathing bars needs a certain extent of skill. A set of sieves as mentioned in the method can be used in conjunction with a food grater. Food graters with openings of various sizes and shapes are available and a suitable type should be chosen to grate bathing bars having different stickiness, moisture and other characteristics. The amount of manual pressure applied by operator during the grating operation also decides the average particle size and particle size distribution. With some experience the operator should be able to get particles suitable for use in lather measurements.

#### C-3 PREPARATION OF STANDARD SOLUTION

**C-3.1** Prepare 1 percent solution of sodium lauryl sulphate (*see* IS 3986, Type 2) in 300 ppm water (Ca : Mg = 3 : 2) [ *see* IS 5785 (Part 4)].

#### C-4 PROCEDURE

##### C-4.1 Measurement of Lather Volume

100 ml of the solution of sodium lauryl sulphate shall be poured into the blender jar of a mixie fixed with 450 W motor covered and blended on low speed for exactly 60 s. The blender shall be operated at an input voltage of  $230 \pm 10$  V at the low setting (popular kitchen mixies

have nearly identical speeds of rotation). The blender jar then shall be held inverted over the 1 000 ml or 500 ml capacity measuring cylinder (*see* Note 1) and any lather adhering to the jar shall be scooped out and allowed to drain. The lather volume shall be measured after leveling off the top surface of the foam. All experiments shall be carried out at an ambient temperature of  $27 \pm 2^\circ\text{C}$  (*see* Note 2). All apparatus and chemicals involved should also be maintained at the above temperature. The lather volume obtained with 1 percent solution of sodium lauryl sulphate should be  $600 \pm 100$  ml.

#### NOTES

1 If lather volume generated is less than 500 ml, use 500 ml cylinder, higher lather volume needs 1 000 ml capacity cylinder.

2 Measurement of temperature of water drained out of the foam is essential in spite of maintaining a constant environmental temperature. Depending upon the atmospheric temperature prevailing in the laboratory at the time of measurement and the nature of the mixie, the starting temperature of water has to be a few degrees lower or higher than the temperature at which measurement is needed.

##### C-4.2 Measurement of Lather Volume of Sample

Take 100 ml of 300 ppm hard water in the blender jar and add 5 g of the grated bathing bar sample to the water in the blender jar, cover and blend on low speed for exactly 60 s. Pour the lather quickly into the cylinder and measure the lather volume immediately after levelling off the top surface of the foam as in C-4.1. Only the top height should be read.

**C-4.2.1** Repeat C-4.1 and C-4.2 for next two sample. Take three determinations for each sample and report the average value (*see* Note). The value for sodium lauryl sulphate determined during each measurement should be used to normalize the value of the bathing bar on the basis of standard at 600 ml.

NOTE — Measurements are made on 3 pieces of each sample of bathing bar and measurements shall be repeated three times with each sample. The average of the three measurements on each sample shall be reported as the value for that sample bar. The average of the readings of the three bars shall be reported as the reading for that sample of bars. The reported readings shall be normalized to a sodium lauryl sulphate value of 600 ml.

**Remarks** — Procedure for normalization of values obtained for lather for the test sample to be provided.

## ANNEX D

[Clause 5.1 and Table 1, Sl No. (ii)]

### DETERMINATION OF MUSH (TABLET-IMMERSION METHOD)

#### D-1 GENERAL

The mush immersion test (objective mush test) described here gives a numerical value for loss of mass due to mashing.

#### D-2 OUTLINE OF THE METHOD

The bathing bars shall be cut down to a rectangular block, which is immersed in demineralized water at  $27 \pm 2^\circ\text{C}$  for  $2 \times 2$  h. The mush formed is scraped off and loss in mass of bar is determined.

#### D-3 APPARATUS

**D-3.1 Beakers** — 250-ml, squat form.

#### D-3.2 Sample Holder

A support to suspend the bathing bar (*see* Fig. 1).

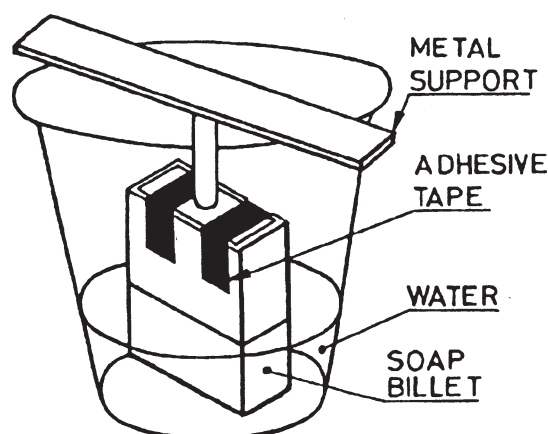


FIG. 1 SAMPLE HOLDER

#### D-3.3 Water Bath

Thermostatically controlled to  $27 \pm 0.5^\circ\text{C}$ .

#### D-3.4 Tablet Cutter

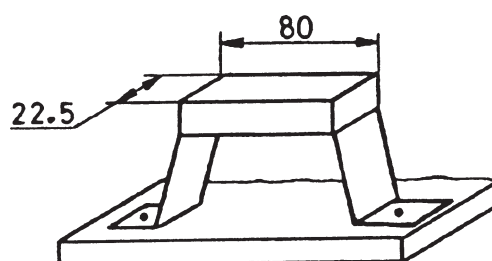
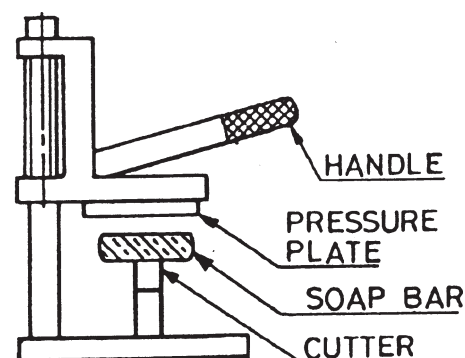
Plane knife, or cutting jig designed to cut sample to pre-determined size (*see* Fig. 2).

#### D-3.5 Scrapper

Spatula having a straight edge.

#### D-4 PROCEDURE

**D-4.1** Cut a rectangular billet from the bathing bar, to the required dimensions (*see* Note 1) using a knife or cutting jig. Measure the width and depth of the cut billet to the nearest 1 mm accurately. Weigh the billet ( $M_1$ ). Measure 50 mm from the bottom of the billet, and draw a line across the billet at this point. This is the immersion



DETAIL OF CUTTER

All dimensions in millimetres.

FIG. 2 TABLET CUTTER

depth (*see* Note 2). Attach the billet to the sample holder (*see* Fig. 1) and suspend the billet in an empty beaker. Add demineralized (or distilled) water at  $27 \pm 2^\circ\text{C}$  to the beaker until the level reaches the 50 mm mark on the billet (*see* Fig. 1 and Note 1).

Place the beaker in a water bath at  $27 \pm 2^\circ\text{C}$  and leave for exactly 2 h (*see* Note 3). Remove the sample holder and billet, empty the water from the beaker, and replace the sample holder and billet in the beaker and leave for about 1 min to drain off the excess water. Carefully scrape off all the mush from all 5 faces of the billet, and remove any remaining traces of mush by wiping gently with a tissue paper.

Repeat the above steps again. Weigh the billet within 5 min of second scraping ( $M_2$ ). Subject six replicates from each sample to the test and report the mean value.

#### NOTES

**1** If a plane is used to prepare the test billet, the plane should be fixed to a bench, and the bar moved over it, not *vice-versa*. To minimize the time taken, the bar should be planed to the largest rectangular shape (that is, remove all rounded edges, logos, etc), but if comparisons are to be made between different sizes of tablets it may be necessary to plane all samples down to the size of the smallest tablet.

## IS 13498 : 2017

2 If the shape and the size of the tablet are such that it cannot be immersed to a depth of 50 mm, a smaller depth of immersion is recommended. A pointed hook bent at right angle attached to the soap is helpful in immersing the tablet to the same level. An immersion depth of 50 mm is recommended, but with small soap tablets this is not always possible. The total area immersed should ideally be between 40 to 60 cm<sup>2</sup>. The volume of water should be about 100 ml. For tablets weighing 150 g and above, the suggested area may be higher (85 cm<sup>2</sup>).

3 If a lot of samples are to be tested simultaneously, it is useful to stagger the water addition to each sample (about 5 min intervals). This allows sufficient time to carry out all the steps mentioned in the procedure so that each sample has exactly 2 h immersion.

## D-5 CALCULATION

Loss in mass due to mush,  $M = M_1 - M_2$

$$\text{Mush} = \frac{M \times 50}{A}$$

where

$A$  = surface area in cm<sup>2</sup> calculated taking into consideration five faces of the tablet immersed.

## ANNEX E

[Clause 5.1 and Table 1, Sl No. (v)]

### DETERMINATION OF GRITTIENESS

#### E-1 PROCEDURE

Hold the bathing bar under running water at a temperature of 30°C and rub gently the two sides of the bar on the palm for 3 min. No gritty or rough feel shall be perceived while rubbing the bar surface. Examine the surface of the bar which shall not be rough

and shall feel smooth when the fingers are moved across the surface. Set the bar on and let it dry for 24 h at room temperature and examine the surface.

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

## ANNEX F

[Clause 5.1 and Table 1, Sl No. (vi)]

### DETERMINATION OF CRACKING

#### F-1 PROCEDURE

**F-1.1** Take minimum of 10 samples for the testing purpose. Rub each tablet in a pool of distilled water maintained at  $27 \pm 2^\circ\text{C}$  in a bucket by rotating the tablet 50 times between the palms and then allow the tablets to dry in a humidity chamber at 38°C and 70 percent relative humidity for 24 h.

#### F-2 EVALUATION

**F-2.1** Carefully observe the cracks on both the lateral

surfaces of the tablet. Locate the cracks which are 10 mm or longer and carefully measure their total length in mm using a scale.

Calculate the average cracking score as under:

$$\text{Average cracking score} = \frac{\text{Total length of cracks in mm}}{\text{Total number of tablets}}$$

If the average cracking score is less than 20 mm, the soap passes the test.

## ANNEX G

[Clause 5.1 and Table 1, Sl No. (vii)]

### DETERMINATION OF CLEANING EFFICIENCY

#### G-1 PROCEDURE

The panellist who participate in this test must give consent, shall be above 18 years of age, should not have any health concern, free of any skin diseases or irritation and do not have any known allergies to soap. Minimum 10 panellists should participate in testing of cleaning efficiency of the soap. The test should be supervised by an independent person who is not a member of the panel.

The test tablet is washed under water by giving it 40 rubs between the hands before the tablet is made available to panellists for actual testing.

The panellist should clean his/her hand with an ordinary soap and pat them dry with a dry towel. The standardized soil to be used in this test is a mixture of cosmetic black Iron Oxide (CI 77499) and refined Groundnut oil in a ratio 1:9 respectively. The amount of standard soil to be used is 50 il which is applied on clean inner sides of palm of panellist facing upwards.

The palms are rubbed 10 times against each other. The palms would look oily and blackish. The panellist is then required to wet his/her hands with water. The panellist is now ready for application of test tablet.

This wetted bar is then rubbed on palms only once (which constitutes one to and fro action from one end of the palm to another end). This is done on each palm with even pressure. After the soap application, the panellist is asked to rub palms 25 times against each other (one to and fro motion constitutes 1 rub). A small amount of water (10 ml) may be used for lubrication. At end of 25 rubs, panellists are required to rinse with water to remove all soap.

#### G-2 EVALUATION

The test palm is dried using a cloth towel and the hand (palm) cleanliness is evaluated. The sample is assessed to have passed the test if at least 7 out of 10 panellists rate the palm to clean and non-oily.

## ANNEX H

[Clause 5.1 and Table 1, Sl No. (viii) (a)]

### DETERMINATION OF TOTAL FATTY MATTER AND SYNTHETIC SURFACE ACTIVE AGENTS

#### H-1 OUTLINE OF THE METHOD

The test method of analysis for total fatty matter [TFM] given in 15 of IS 286 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.

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.

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

#### H-2 PRINCIPLE

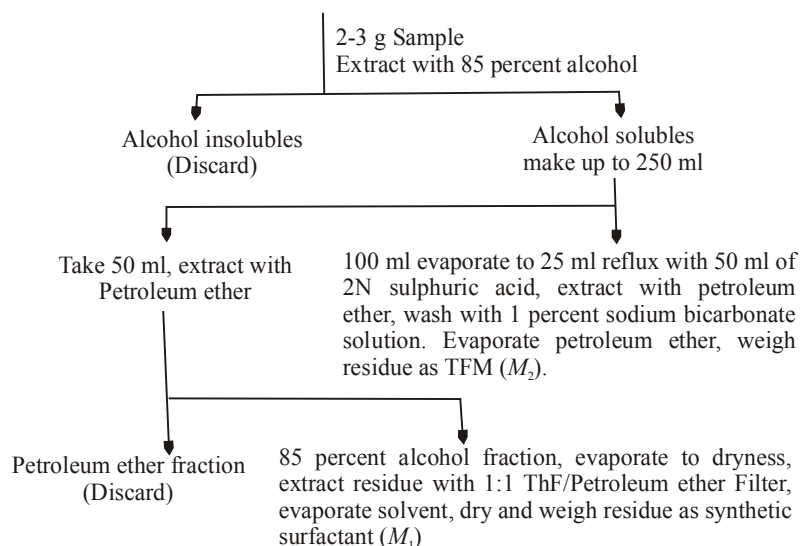
Since several synthetic surfactants are permitted to be

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 percent alcohol. An aliquot of 85 percent alcohol soluble matter is extracted with petroleum ether to remove hydrophobic constituents (perfume, unsaponifiable matter etc) and the 85 percent 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 percent 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.

IS 13498 : 2017

### H-3 ANALYTICAL STRATEGY



Total synthetic surfactant and total fatty matter are estimated independently.

### H-4 REAGENTS

**H-4.1 85 percent Alcohol (v/v)** — Ethyl alcohol (*see* IS 321) or rectified spirit (*see* IS 323).

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 percent v/v concentration).

**H-4.2 Sulphuric Acid 2 N (approximate)** — Add 6 ml of concentrated sulphuric acid (sp Gr 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.

**H-4.3 Methyl Orange Indicator Solution** — (0.1 percent aqueous solution)

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

**H-4.4 Phenolphthalein Indicator** — (1.0 percent w/v alcoholic solution)

Dissolve  $1.00 \pm 0.05$  g of the indicator in 100 ml of ethyl alcohol or rectified spirit and mix well.

**H-4.5 Sodium Hydroxide 0.1 N (approximate)** — 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.

**H-4.6 Petroleum Ether (40-60°C Boiling Range)** — Laboratory reagent grade.

**H-4.7 Aqueous Ethyl Alcohol** (or rectified spirit) — Approx 50 percent v/v.

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

**H-4.8 Sodium Bicarbonate Solution** — 1 percent w/v (approximate).

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

**H-4.9 Sodium Hydroxide Solution** — 1.0 percent (w/v) in 85 percent alcohol (approximate).

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

**H-4.10 Acetone** — AR grade.

**H-4.11 Tetrahydrofuran (THF)** — AR Grade.

**H-4.12 Solvent Mixture** — 1:1 mixture of tetrahydrofuran and petroleum ether (v/v).

**H 4.13 Rectified Spirit** (*see* IS 323)

**H 4.14 50 percent (w/w) Sulphuric Acid Solution** — Take 50ml 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.

### H-5 APPARATUS/GLASSWARE

**H-5.1 Beakers** — 250 ml, 500 ml and 1 000 ml capacity.

**H-5.2 Measuring Cylinder** — 10 ml, 25 ml and 50 ml capacity.

**H-5.3 Standard Volumetric Flask** — 10 ml, 250 ml, 500 ml and 1 000 ml capacity.

**H-5.4 Separating Funnel** — 500 ml capacity.

**H-5.5 Round Bottom Flask with Ground Joint** — 250 ml capacity.

**H-5.6 Air Condenser** — length 1 m.

**H-5.7 Conical Flask** — 500 ml capacity.

**H-5.8 Funnels**

**H-5.9 Glass Rods**

**H-5.10 Flash Rotary Evaporator/Distillation Set-Up**

**H-5.11 Whatman Filter Paper No. 541**

**H-5.12 Air Oven**

**H-5.13 Steam Bath**

## **H-6 METHOD**

### **H-6.1 Preparation of Sample**

#### **H-6.1.1 Cakes/Bars**

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

### **H-6.2 85 percent Alcohol Extraction**

**H-6.2.1** Weigh accurately about 2.0 to 3.0 g of sample into a 250-ml beaker ( $M$ ).

**H-6.2.2** Add 125 ml of 85 percent alcohol, heat to about 60°C with continuous stirring with a glass rod on a steam bath for 5 min (approximately) inside a fuming cupboard. Transfer into a 250-ml volumetric flask using a funnel by decantation.

**H-6.2.3** Add again 50 ml of 85 percent alcohol into the same beaker (**H-6.2.1**). Break the lumps, if any, with the glass rod and repeat the operation as in **H-6.2.2**.

**H-6.2.4** Repeat the extraction twice with 25 ml of 85 percent alcohol as in **H-6.2.2**.

**H-6.2.5** Collect all the 85 percent alcohol extracts in the 250-ml volumetric flask.

**H-6.2.6** Make up the volume to 250-ml with 85 percent alcohol and mixed well.

### **H-6.3 Estimation of Synthetic Surfactants**

**H-6.3.1** Transfer 50 ml of solution from **H-6.2.6** into a 250-ml beaker.

**H-6.3.2** Add 0.5 ml of phenolphthalein indicator and add 1.0 percent sodium hydroxide solution (drop by

drop) till the colour of the solution changes to pink. Add 1.0 ml excess beyond this point.

**H-6.3.3** Transfer quantitatively into a 500 ml separating funnel, extract 3 times with 50 ml aliquots of petroleum ether. Preserve the lower alcohol/water portion.

**H-6.3.4** Wash the combined petroleum ether extracts with 20 ml aliquots of 85 percent alcohol solution 2 times. Collect the alcohol washings and transfer quantitatively to the alcohol extract from **H-6.3.3** and discard the petroleum ether extract.

**H-6.3.5** Evaporate the alcoholic solution from **H-6.3.4** to dryness.

**H-6.3.6** Add 20 ml of acetone to the residue. Heat on water bath with continuous stirring to dryness. Ensure complete removal of water (Note 1).

#### **NOTES**

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

2 Use only Filter paper No. 541 (Porosity of 20-25  $\mu\text{m}$ ).

**H-6.3.7** Add 50 ml of solvent mixture (**H-4.12**) to the residue, heat to boiling (60-70°C) on a water bath with constant stirring for 2-3 min and filter through filter paper No. 541 (Whatman). Collect the filtrate into a tared 250-ml beaker/round bottom flask.

**H-6.3.8** Repeat the extraction two more times as described in **H-6.3.7**, 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.

**H-6.3.9** 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.

**H-6.3.10** Dry the beaker/round bottom flask containing the residue in an air oven at 105°C for 1 hour. Cool to room temperature inside a dessicator and weigh to constant weight ( $M_1$ ).

### **H-6.4 Estimation of TFM**

**H-6.4.1** Transfer quantitatively 100 ml of extract from **H-6.2.6** 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.

**H-6.4.2** Add 50 ml of 2 N sulphuric acid and reflux on steam bath for 2 h after fitting with an air condenser.

**H-6.4.3** Remove condenser. Add 8 g sodium chloride and continue refluxing for further 1 h and 30 min.

**H-6.4.4** Cool to room temperature. Add 20 ml of rectified spirit and transfer quantitatively to a 500-ml

## IS 13498 : 2017

separating funnel.

**H-6.4.5** 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.

**H-6.4.6** 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.

**H-6.4.7** Transfer the alcoholic layer to the second separating funnel and repeat the extraction with 75 ml of petroleum ether as given in **H-6.4.6**.

**H-6.4.8** Wash the petroleum ether extract with 30 ml aliquots of 1 percent (w/v) sodium bicarbonate solution till it is free from mineral acidity. Two washings are sufficient (test with methyl orange indicator).

**H-6.4.9** Transfer petroleum ether extract quantitatively to a tared 250-ml round bottom flask and evaporate petroleum ether on water bath by distillation inside a fuming cupboard.

**H-6.4.10** 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 (difference between weighings be less than 0.005 g) ( $M_2$ ).

## H-7 CALCULATION

$$\text{Total synthetic surfactants, percent} = \frac{M_1 \times 250 \times 100}{M \times 50}$$

where

$M_1$  = mass in g of residue (**H-6.3.10**), and

$M$  = mass in g of sample taken for test (**H-6.2.1**).

$$\text{Percent by mass, TFM} = \frac{M_2 \times 250}{M}$$

where

$M$  = mass in g of sample taken for test (**H-6.2.1**),  
and

$M_2$  = mass in g of residue (**H-6.4.10**).

## ANNEX I

[Clause 5.1 and Table 1, Sl No. (viii)]

### ESTIMATION OF FATTY MATTER FROM DISSOLVED ACTIVES

#### I-1 GENERAL

The method below describes quantitative estimation of fatty matter content from dissolved actives from bathing bar.

#### I-2 PROCEDURE

**I-2.1** 250 g of 1.0 percent (w/w) alkaline soap solution is prepared by stirring approx. 2.5 g ( $W$ ) of finely grated soap sample and 1.0 g of sodium hydroxide pellets (min 98 percent purity) with 247.5 g of distilled water. Stir using magnetic stirrer for 4 h at 27°C (care must be taken so that no frothing and overflow takes place).

**I-2.2** Filter the soap solution through fluted (flower) Whatman filter No.1 with a circle diameter of minimum 15 cm (preferably 24 cm).

*NOTE* — Ensure that the filtration funnel is selected in such a way that entire area of filter paper is supported and that there is no tear in the filter paper while filtration.

**I-2.3** Measure turbidity of the filtrate soap solution at 30°C by Digital nephelometric turbidity meter. 100 gm aliquot of clear soap filtrate is taken for analysis and Turbidity should be < 100 NTU.

*NOTE* — Since all the submicron soap particles would not be completely filtered, the filtrate would be slightly turbid.

**I-2.4** Take 100 g of the filtrate. Add 19.6 g of 50 percent (w/w) conc. Sulphuric acid (*see H-4.14*) to this filtrate with continuous stirring and reflux the contents for 2 h after fitting with an air condenser (18 inches height, *Min*).

**I-2.5** Remove condenser. Add 8 g sodium chloride and continue refluxing for further 1 h and 30 min.

**IS 13498 : 2017**

**I-2.6** Cool to room temperature. Add 20 ml of rectified spirit and transfer quantitatively to a 500-ml separating funnel.

**I-2.7** 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.

**I-2.8** 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.

**I-2.9** Transfer the alcoholic layer to the second separating funnel and repeat the extraction with 75 ml of petroleum ether as given in **I-2.8**.

**I-2.10** Wash the petroleum ether extract with 30 ml aliquots of 1 percent w/v sodium bicarbonate solution till it is free from mineral acidity. Two washings are sufficient (test with methyl orange indicator).

**I-2.11** 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.

**I-2.12** 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 (difference between weighings less than 0.005 g) *Z*.

**I-2.13** Calculate the Fatty matter from dissolved actives, percent using calculations given in **I-3**.

**I-3 CALCULATION**

Weight of soap in the total soap bar (in g) = *W*

Weight of TFM in 100 g of filtrate = *Z*

Therefore, weight of Fatty Matter from Dissolved Actives in filtrate =  $2.5 \times Z$

Estimation of Fatty Matter from Dissolved actives in filtrate as percent of soap bar weight

Fatty matter from dissolved actives, percent =  $\frac{2.5 \times Z \times 100}{W}$

IS 13498 : 2017

## ANNEX K

### (Foreword)

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