

भारतीय मानक
Indian Standard

IS 7884 : 2023

शैम्पू — विशिष्टि
(चौथा पुनरीक्षण)

Shampoo — Specification
(Fourth Revision)

ICS 71.100.70

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FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was published in 1975 and first revised in 1978. Second revision of the standard was carried out in 1992 when minimum limit for total active detergent content as total non-volatile alcohol soluble matter was prescribed and minimum requirement limit for anionic detergent content was also fixed. Besides, requirements for volatile matter and inorganic salts were deleted, as these were not serving any meaningful purpose. An additional requirement for lather was included from consumer's satisfaction point of view. Many of the surfactants which are frequently used in shampoos as cleansing agents develop abundance lacy foam in soft water but this lather drops drastically in presence of oily soil, therefore, ingredients commonly known as foam boosters are added in shampoos to improve quality volume and characteristics of lather. Minimum foam height was specified to quantify this requirement. In the second revision of the standard, the requirement of active detergent as SLES or its equivalent, was subsequently deleted through an amendment. This was done to allow the manufacturers to use cationic, anionic and amphoteric type of detergents alone or in combinations in place of limiting the concentration of one kind of detergents.

Third revision of this standard was carried out in 2004. In the third revision, a procedure for determination of non-volatile alcohol soluble matter was rectified/modified. Two new requirements namely 'best use before' and list of key ingredients were also included in marking clause to safeguard consumer interest. For determining the suitability of a new formulation or of a new raw material used in an old formulation, on the skin and in respect of eye irritation, reference was made to IS 4011 : 2018 'Methods of test for safety evaluation of cosmetics (*third revision*)' was mentioned. It was also mentioned that it shall be the responsibility of the manufacturers of shampoo, to satisfy themselves of the dermatological safety of their formulation according to that standard before releasing the product for sale.

Subsequently five amendments were issued to the standard which included the following changes. The title of the standard was changed from 'Shampoo, surfactant based' to 'Shampoo'. Requirement of Active detergent content as SLES or alternate Active detergent content as non-ionic and/or anionic as SLES was introduced along with a requirement for microbial content and its maximum permissible limits. Test methods were prescribed for determination of active detergent content both anionic and non-ionic including ethoxylated non-ionic surfactants and coco-glucoside non-ionic surfactants.

Additional test requirements for heavy metals as lead and arsenic as As_2O_3 and their maximum permissible limit in the product were introduced through another amendment.

In this revision, all the five amendments issued after the third revision of the standard have been incorporated.

Shampoos, which contain ingredients that have an effect on the physiological functions of the body, or for which therapeutic claims are made, are not included in this standard.

No stipulations have been made in this standard regarding the composition of the shampoo. However, it is necessary that the raw materials used are such that in the concentrations in which they would be present in the finished shampoo, after interaction with the other raw materials used in the formulation, are free from any harmful effects. Furthermore, information about various attributes of an acceptable shampoo which are not adequate for standardization are included in [Annex M](#) to the standard so that the formulators may try to attain as many qualities as possible in their products to have consumer acceptance.

(Continued on third cover)

Indian Standard
SHAMPOO — SPECIFICATION
(*Fourth Revision*)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for shampoo.

2 REFERENCES

The standards listed in [Annex A](#) contain provisions which, through reference in this standard, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

3 REQUIREMENTS

3.1 Description

The shampoo shall be in the form of a liquid, emulsion or paste. It may be coloured and perfumed. Any other format is out of scope of this standard.

3.2 Physical Characteristics

The clear/transparent liquid shampoo, when examined visually, shall be free from any sediment. If in the form of an emulsion, it shall be homogeneous and there shall be no visible signs of

the emulsion having broken. Shampoo in the form of a paste shall be free from any agglomerated particles.

3.3 Ingredients

Unless specified otherwise, all the raw materials used in the manufacture of shampoo shall conform to the requirements prescribed in the relevant Indian Standards where such standards exist.

3.3.1 Dyes

The dyes used, if any, shall comply with the provisions of [IS 4707 \(Part 1\)](#).

3.3.2 Other Ingredients

Other ingredients used, if any, shall comply with the provisions of [IS 4707 \(Part 2\)](#).

3.3.3 For safety evaluation of novel ingredients used in formulation of a shampoo, the shampoo shall comply to IS 4011.

3.3.4 A list of ingredients conventionally used in formulation of shampoos is given, for guidance, in [Annex B](#).

3.4 The shampoo shall comply with the requirements given in Table 1 when tested as prescribed in col (4) and (5) of [Table 1](#).

Table 1 Requirements for Shampoo

(Clauses [3.4](#) and [E-5](#))

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex	
(1)	(2)	(3)	(4)	(5)
i)	Non-volatile alcohol soluble matter; percent by mass, <i>Min</i>	8	C	-
ii)	pH (at 27 °C ± 2 °C)	4.0 to 9.0	D	-
iii)	Foam height for two percent solution, <i>Min</i>	150 mm	E	-
iv)	Active surfactant content, percent by mass, <i>Min</i> (either a and/or b to be complied with) Anionic Anionic and/or non-ionic, and/or amphoteric	5	a) F (this method to be used when the product contains only anionic surfactants in absence of any other type of	-

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Table 1 (Concluded)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex	
(1)	(2)	(3)	(4)	(5)
			surfactant) or G - Alternate method using potentiometer b) H (this method to be used when the product contains anionic and/or non-ionic, and/or amphoteric) or J - Alternate method using potentiometer	
v)	Microbial limit			
	a) Total microbial count, cfu/g, <i>Max</i>	< 1 000 cfu		IS 14648
	b) Yeast and mould count, cfu/g, <i>Max</i>	< 100 cfu		
	c) <i>Pseudomonas aeruginosa</i> , per gram	Absent		
	d) <i>Escherichia coli</i> , per gram	Absent		
	e) <i>Staphylococcus aureus</i> , per gram	Absent		
	f) <i>Candida albicans</i> , per gram	Absent		
vi)	Heavy metals as lead (Pb), parts per million, <i>Max</i> ¹⁾	20	K	or IS 16913
vii)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i> ²⁾	2	L	IS 16913 or IS 17495
viii)	Mercury (Hg), parts per million, <i>Max</i>	1	-	IS 16913 or IS 17495

3.5 Additional Requirements for ECO-Mark (Optional)

3.5.1 General Requirements

3.5.1.1 The product shall confirm to the requirements for quality, safety and performance given in **3.5.1.2** to **3.5.1.5**.

3.5.1.2 All the ingredients that go into formulation of cosmetics shall comply with the provisions of

[IS 4707 \(Part 1\)](#) and [IS 4707 \(Part 2\)](#). The product shall also meet specific requirements as given in the standard.

3.5.1.3 The product package shall display a list of key ingredients in descending order of quantity present.

3.5.1.4 The product shall not be manufactured from any carcinogenic ingredients.

¹⁾ In the event of any dispute, colorimetric method mentioned in [Annex K](#) would be treated as referee method.

²⁾ In the event of any dispute, colorimetric method mentioned in [Annex L](#) would be treated as referee method.

4 PACKING AND MARKING

4.1 Shampoo shall be packed in glass or plastic containers or any other suitable containers.

4.2 The containers shall be legibly marked with the following information:

- a) Name of the material;
- b) Manufacturer's name and/or his recognized trade-mark, if any;
- c) Net content in volume for liquids and emulsions and in mass for pastes;
- d) Month and year of manufacturing/packing;
- e) Batch or lot number, in code or otherwise;
- f) Use before or expiry date as per statutory requirements;
- g) List of ingredients as per statutory requirements; and
- h) Any other information required by statutory authorities.

4.2.1 BIS Certification Marking

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4.2.2 If the product is covered under ECO-Mark (optional), it shall be suitably marked with ECO-Mark logo besides Standard Mark. The label may clearly specify that ECO-Mark is applicable to the contents or the package or both, as case may be. If the product package is not separately covered under ECO-Mark scheme, it shall be clearly mentioned on the product that ECO-Mark label is applicable to contents only.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in [IS 3958](#).

5.2 Tests for all the requirements shall be carried out on a composite sample.

5.3 The shampoo shall be taken to have conformed to this standard if the composite sample passes all the tests.

6 QUALITY OF REAGENTS

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

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

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ANNEX A

([Clause 2](#))

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)	(Part 1) : 2020	Colourants (<i>fourth revision</i>)
IS 321 : 1964	Specification for absolute alcohol (<i>first revision</i>)	(Part 2) : 2017	List of raw materials generally not recognized as safe for use in cosmetics (<i>fourth revision</i>)
IS 571 : 2000	Monosodium phosphate — Specification (<i>second revision</i>)	IS 4955 : 2020	Household laundry detergent powders — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)	IS 5296 : 1995	Chloroform, pure and technical — Specification (<i>second revision</i>)
IS 1113 : 1965	Specification for ammonium chloride, technical and pure (<i>revised</i>)	IS 14648 : 2011	Microbiological examination of cosmetics and cosmetic raw materials — Methods of test (<i>second revision</i>)
IS 2088 : 2023	Methods for determination of arsenic (<i>third revision</i>)	IS 16913 : 2018	Methods of test for cosmetics — Determination of heavy metals (arsenic, cadmium, lead and mercury) by atomic absorption spectrometry (AAS)
IS 3958 : 2021	Methods of sampling cosmetics (<i>second revision</i>)	IS 17495 : 2021 /ISO/TR 17276 : 2014	Cosmetics — Analytical approach for screening and quantification methods for heavy metals in cosmetics
IS 4011 : 2018	Methods of test for safety evaluation of cosmetics (<i>third revision</i>)		
IS 4566 : 2020	Specification for methylene chloride (dichloromethane) — Technical (<i>second revision</i>)		
IS 4707	Classification for cosmetic raw materials and adjuncts:		

To access Indian Standards click on the link below:

https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knownyourstandards/Indian_standards/isdetails/

ANNEX B

(Clause 3.3.4)

ILLUSTRATIVE LIST OF RAW MATERIAL CONVENTIONALLY USED IN FORMULATION OF SHAMPOOS

B-1 DETERGENTS

- a) Sodium or potassium or ethanolamine salts of lauryl sulphonic acid;
- b) Lauryl ether sulphates;
- c) Sulphated monoglycerides;
- d) Sodium alkyl sulfo-acetate;
- e) Alkyl benzenepolyoxyethylsulphonates;
- f) Sodium n-lauryl sarcosinate;
- g) Sodium alpha olefin sulphonates; and
- h) Other synthetic detergents.

B-2 FOAM STABILIZERS

- a) Ethanolamides or isopropanolamides of fatty acids;
- b) Amine oxides;
- c) Cocobetaines; and
- d) Cocoamidopropylbetaines.

B-3 CHELATING AGENTS

- a) Sodium polyphosphates; and
- b) Sodium salts of ethylenediaminetetra-acetic acid.

B-4 SOLUBILIZING AGENTS

- a) Urea;
- b) Aliphatic alcohols;
- c) Sodium toluene sulphonate; and
- d) Sodium xylene sulphonate.

B-5 PRESERVATIVES

- a) Alcohols;
- b) Formaldehyde;
- c) Esters of p-hydroxyl benzoic acid;
- d) Sorbic acid; and
- e) Imidazolidinyl urea.

B-6 OPACIFYING AGENTS

- a) Higher fatty alcohols;
- b) Ethylene/propylene glycol stearates;
- c) Mono and di-stearates of glycerol;
- d) Zinc, calcium, and magnesium salts of fatty acids;
- e) PEG-distearate 6000; and
- f) Polyacrylates.

B-7 INORGANIC SALTS

- a) Sodium chloride;
- b) Sodium sulphate;
- c) Sodium phosphate;
- d) Ammonium sulphate;
- e) Ammonium phosphate; and
- f) Ammonium chloride.

B-8 EMOLLIENTS

Lanolin and its derivatives.

B-9 THICKENING AGENTS

- a) Sodium carboxymethyl cellulose;
- b) Methyl cellulose;
- c) Methyl isopropyl cellulose; and
- d) Guar gum.

B-10 OTHER GROUPS OF INGREDIENTS

- a) Perfumes;
- b) Dyes/colours/pigment;
- c) Conditioning agents;
- d) Quaternary compounds;
- e) Vitamins;
- f) Vegetable oils;
- g) Silicones;
- h) Proteins; and
- j) Sunscreens, etc.

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ANNEX C

[Table 1, [SI No. \(i\)](#)]

DETERMINATION OF NON-VOLATILE ALCOHOL SOLUBLE MATTER

C-1 GENERAL

This method determines the amount of non-volatile alcohol soluble matter in shampoos.

C-2 REAGENTS

C-2.1 Ethyl Alcohol — neutral, conforming to [IS 321](#)

C-2.2 Methyl Red Indicator Solution — dissolve 0.1 g of methyl red in 300 ml of ethyl alcohol and 200 ml of water

C-2.3 Potassium Chromate Solution — 10 percent solution

C-2.4 Nitric Acid Dilute — 1 : 4 (v/v)

C-2.5 Silver Nitrate Solution — 0.1 M

C-3 PROCEDURE

C-3.1 Weigh accurately about 10 g of the sample into a suitable beaker. Evaporate on a steam-bath to almost complete dryness. Digest with 50 ml of 96 percent ethyl alcohol by heating on a steam bath for about 2 min. Filter the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Wash the beaker and the residue in the sintered glass funnel 5 times with 30 ml portions of hot ethyl alcohol.

C-3.2 Transfer the filtrate in the Buchner flask to a weighed wide mouth flat-bottomed flask or suitable beaker. Evaporate nearly to dryness on a water bath and drive off the remaining alcohol by directing a gentle stream of dry air into the flask whilst continuously rotating the latter in the water bath.

Heat the flask in an air oven at a temperature of 105 °C until constant mass. Calculate mass percent of residue obtained.

Mass, percent of residue (Y) =

$$\frac{\text{Mass of residue obtained}}{\text{Mass, in g, of the material taken for test}} \times 100$$

C-3.3 Dissolve the residue in 50 ml of distilled water and add to it 2 drops of methyl red indicator solution. If the solution is yellow in colour, neutralize it by adding dilute nitric acid drop by drop to pink colour. Titrate the solution with silver nitrate solution using 2.5 ml of potassium chromate solution as indicator, till a brown colour is obtained. Carry out a blank determination using the same quantity of all reagents except the sample.

C-3.4 Calculate the chloride content in shampoo in terms of molecular mass of sodium chloride (X) in percent by the formula:

$$\text{Sodium chloride in percent (X)} = V \times \frac{0.5844}{M}$$

where

V = volume, in ml, of standard silver nitrate solution required for the material minus volume, in ml, of standard silver nitrate solution required for the blank; and

M = mass, in g, of the material taken for test.

C-3.5 Calculation

To calculate percent non-volatile alcohol soluble matter, subtract the mass percent of sodium chloride as determined in [C-3.4](#) as (X) from the mass percent of the residue (Y) obtained in [C-3.2](#).

Percent non-volatile alcohol soluble matter = Y – X

ANNEX D

[Table 1, [Sl No. \(ii\)](#)]

DETERMINATION OF pH

D-1 APPARATUS

pH meter equipped with glass electrode.

D-2 PROCEDURE

Determine the pH at a temperature of $27\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. In the case of liquid shampoo, read the pH directly in the sample in the pH meter. In the case of a shampoo in the form of a paste, mix 1 g of the sample with 9 ml of water and determine the pH of the resulting solution.

ANNEX E

[Table 1, [Sl No. \(iii\)](#)]

DETERMINATION OF FOAM HEIGHT

E-1 OUTLINE OF THE METHOD

In order to check the ability of a shampoo to produce lather, the volume of foam obtained under specific experimental conditions is determined

E-2 APPARATUS

E-2.1 Pipette

The pipette shown in [Fig. 1](#) shall be constructed from standard-wall chemically resistant glass tubing having the following dimensions:

- For the bulb, $45.0\text{ mm} \pm 1.5\text{ mm}$ outside diameter; and
- For the lower stem, $7.0\text{ mm} \pm 0.5\text{ mm}$ outside diameter.

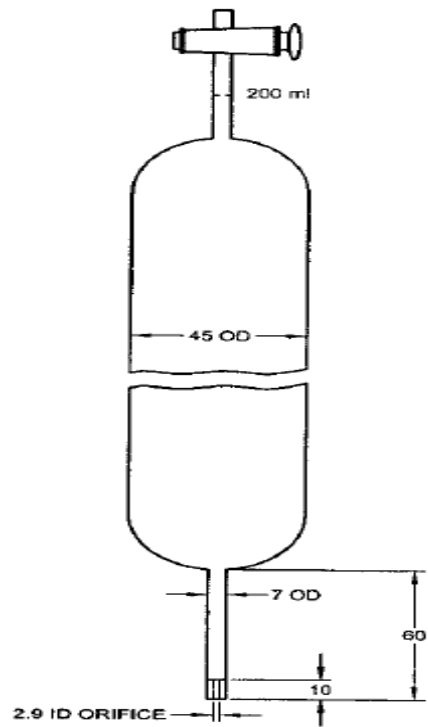
The upper stem shall be constructed to contain a solid-stopper, straight bore, No. 2, standard taper stopcock having a 2 mm bore and stems 8 mm in outside diameter. Both the upper and lower seals of the bulb to the stems shall be hemispherical in shape. The lower stem shall be $60\text{ mm} \pm 2\text{ mm}$ in length from the point of attachment to the bulb and shall contain an orifice sealed into the lower end. The orifice shall be constructed from precision bore tubing having an inside diameter of $2.9\text{ mm} \pm 0.02\text{ mm}$ and a length of $10.00\text{ mm} \pm 0.05\text{ mm}$, with both ends ground square. The orifice shall have an outside diameter so as to fit snugly into the lower stem and form a secure seal to the stem when heated with a sharp pointed flame in the blow torch. The pipette shall be calibrated to contain $200.0\text{ ml} \pm 0.2\text{ ml}$ at $20\text{ }^{\circ}\text{C}$. The calibration mark shall be on the upper stem at least 15 mm below the barrel of the stopcock and shall completely encircle the stem.

E-2.2 Receiver

The receiver shown in [Fig. 2](#) shall be constructed from standard-wall, chemically resistant glass tubing having an internal diameter of $50.0\text{ mm} \pm 0.8\text{ mm}$, with one end constricted and sealed to a straight-bore, solid-plug, standard taper No. 6 stopcock having a 6 mm bore and 12 mm stems. The receiver shall have three calibration marks which shall completely encircle the tube. The first mark shall be at the 50 ml point, it shall be measured with the stopcock closed, and shall not be on any curved portion of the constriction. The second mark shall be at the 250 ml point, and the third mark at a distance of $90.0\text{ cm} \pm 0.5\text{ cm}$ above the 50 ml mark. The receiver tube shall be mounted in a standard-wall tubular water jacket, having an external diameter of not less than 70 mm, fitted with inlet and outlet connections.

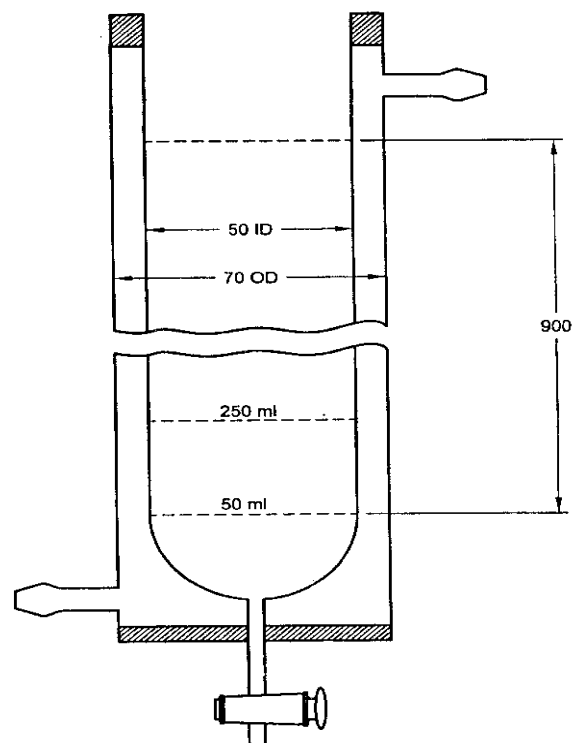
The jacket may be attached to the receiver with rubber stoppers as practicable. The assembled receiver and jacket shall be mounted securely in a plumb position and the jacket connected to a source of water thermostatically maintained at $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for circulating through the jacket. At the top of the receiver there shall be a platform, flush with the top of the assembly, having a metal plate in which is drilled three indexing holes circumferentially placed around the receiver and having an angular displacement of 120° from each other. A clamp which may be securely attached to the upper part of the pipette shall fit into the holes. The clamp shall have three levelling screws and lock nuts and when properly mounted shall exactly centre the pipette in the receiver and bring the lower tip of the pipette level with the upper calibration mark on the receiver. A meter stick shall be fastened to the side or behind the receiver with the zero point level with the 250 ml calibration point on the receiver.

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All dimensions in millimetres.

FIG. 1 FOAM PIPETTE



All dimensions in millimetres.

FIG. 2 FOAM RECEIVER

E-3 PREPARATION OF SAMPLE SOLUTION

Distilled water, or water hardness 100 expressed in parts per million of calcium carbonate shall be taken for test. Preheat the water to a temperature of $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Add 500 ml of water to 10 g of shampoo solution while stirring vigorously. Continue stirring in such a manner until miscibility of shampoo with water is complete. Age the solution at a temperature of $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for a total period of 30 min counting the time when the shampoo is first added to the water.

E-4 PROCEDURE

While the shampoo solution is aging, circulate water at $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ through the water jacket of the receiver so as to bring it to the proper temperature. Rinse down the walls of the receiver with distilled water and, as an indication of cleanliness, observe whether the water drains down the walls in an unbroken film. At the completion of the aging period close the stopcock at the bottom of the receiver. Rinse the walls of the receiver with 50 ml of the solution using a pipette, and after draining to

the bottom of the receiver, adjust the stopcocks so that the level of the solution in the receiver is exactly at the 50 ml mark. Fill the pipette with the solution to the 200 ml mark, using a slight suction for the purpose. Immediately place it in position at the top of the receiver and open the stopcock. When all of the solution has run out of the pipette, start a stopwatch, take a reading of the foam height and take a second reading at the end of 5 min. Take the reading by measuring the foam production at the top of the foam column at the highest average height to which the rim of the foam has reached. This height is proportional to the volume of air remaining in the foam.

E-5 REPORTING

- a) Concentration, in g/l;
- b) Temperature of the test;
- c) Degree of hardness of water;
- d) Initial foam height; and
- e) Foam height after 5 min.

The foam height of two percent shampoo solution shall not be less than the specified limit in [Table 1](#).

ANNEX F

[Table 1, [Sl No. \(iv\)](#)]

DETERMINATION OF ACTIVE DETERGENT CONTENT

F-1 PRINCIPLE FOR ACTIVE DETERGENT CONTENT

F-1.1 When equivalent amounts of cationic and anionic detergents are present in a two-phase mixture of water and chloroform, methylene blue will colour the two phases to the same degree. Sodium alkyl benzene sulphonate and sodium lauryl sulphate or any other detergent can be titrated with a standard solution of cetyltrimethyl ammonium bromide.

F-1.2 Cetyltrimethyl ammonium bromide may be used for analysis of shampoo as a routine method. However, in case of any dispute benzethonium chloride solution which is commercially known as hyamine 1622 should be used. [IS 4955](#) may be referred for determination of active detergent content using benzethonium chloride. Cetyltrimethyl ammonium bromide method is described in this annex.

F-2 REAGENTS

F-2.1 Cationic Solution (Solution A)

Weigh 1.5 ± 0.001 g of cetyltrimethyl ammonium bromide into a 250 ml beaker. Add 100 ml of distilled water and stir until dissolved. Transfer quantitatively to a 1 litre volumetric flask and make to volume. Mix thoroughly and standardize against Solution B.

F-2.2 Anionic Solution (Solution B)

Weigh accurately such amount of standard alkyl sulphate of known combined SO_3 or active content so as to give exactly 0.320 g combined SO_3 into a 250 ml beaker. Dissolve in 100 g to 200 g of warm water. Transfer quantitatively to a 1 litre volumetric flask and make to volume with water at room temperature. This is the primary standard against which Solution A is standardized. Solution B is 0.004 N.

NOTE — The 0.004 N hyamine solution is prepared by weighing 1.864 g hyamine 1622 (molecular weight = 466) into a beaker. Dissolve the hyamine 1622 in about 100 ml of deionized water. Quantitatively transfer the solution to a 1 000 ml volumetric flask. Dilute to volume and mix well. Standardize the solution against 0.007 N sodium lauryl sulfate (SLS).

F-2.3 Methylene Blue Indicator

Dissolve 0.1 g of methylene blue in 100 ml of water.

Transfer 30 ml of this solution to a 1 litre flask. Add 500 ml of water, 6.8 ml of concentrated sulphuric acid, 50 g of sodium dihydrogenorthophosphate ($\text{NaH}_2\text{PO}_4\text{H}_2\text{O}$) and shake until solution is complete. Dilute to the mark.

F-2.4 Distilled Water — see [IS 1070](#)

F-2.5 Chloroform — analytical reagent grade (see [IS 5296](#))

F-3 PROCEDURE

F-3.1 Weigh accurately a sample of sufficient size to give approximately 0.320 g of combined SO_3 into a 250 ml beaker. Sample size is crucial (see Note). Use 700 ml to 800 ml of warm water to transfer quantitatively to a 1 litre volumetric flask. Warm on steam bath and shake gently until the sample is dissolved and solution is clear. Cool, dilute to the mark and mix thoroughly.

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

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

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

F-3.3 Percent combined; $\text{SO}_3 = \frac{V \times N \times 800}{M}$

where

V = volume, in ml, of Solution A, used in the titration;

N = normality of Solution A; and

M = mass, in g, of the sample in the aliquot.

F-3.4 Percent w/w of active detergent content =

$\frac{\text{SO}_3 \times \text{Molecular mass of active detergent}}{80}$

80

NOTE — For SLES detergent, the molecular mass may be taken as 400. In case sodium lauryl sulphate or sodium alpha olefin sulphonate or any other anionic detergent is used in formulation whose molecular mass is less than 350, the actual molecular mass of detergent as declared by the manufacturer may be used for calculation.

ANNEX G

[Table 1, [Sl No. \(iv\)](#)]

ANIONIC SURFACTANT TITRATION IN A COSMETIC PRODUCT BY POTENTIOMETRY

G-1 PRINCIPLE

G-1.1 General Case

The anionic substance is dissolved in water and titrated by potentiometry in acid medium by hyamine 1622 (titrated cationic solution), using a specific electrode.

Anionic surfactants such as sulfates and sulfonates may be titrated throughout the whole pH range but experience has shown that the titration is optimal in acid medium. However, at very acidic pH, there is a risk of interferences of amphoteric compounds which are in protonic form and thus cationic at $\text{pH} < 2.5$. There is also a risk of hydrolysis of sulfates and consequently loss of their surfactant properties at $\text{pH} < 1$. The titration is therefore performed at pH 3.00 which optimizes the titration while eliminating the risks of interferences of amphoteric compounds.

The specific electrode with plastic membrane is composed of plasticizer, PVC and ionophores (or ion carriers). These ionophores, present at a concentration of about 0.1 percent to 1 percent, are the electro-active compounds responsible for the detection of surfactants and for the change in potential.

The specific electrode is sensitive to anionic and cationic surfactants but is optimized for anionic surfactants. It detects a low potential in the presence of anionic surfactants and a high potential in the presence of cationic surfactants.

During the titration, the anionic surfactants form ion pairs with hyamine 1622, which decreases the quantity of anionic surfactant available and consequently increases the potential detected by the electrode. The electrode tends to its equilibrium potential, which corresponds to the potential in a medium free from surfactants and reaches the

equilibrium potential at the equivalence. An excess of hyamine 1622 causes then an important potential increase, which explains the appearance of an ascending potential jump.

G-1.2 Case of Formulas Containing Fatty Compounds

The active cationic substance is dissolved in a methylisobutylketone (MIBK)/ ethanol mixture (1 : 1) under heating and titrated by potentiometry in acid medium using hyamine 1622 (titrated cationic solution) and a specific electrode.

Quaternary ammoniums and quaternary esters are cationic throughout the whole pH range so they may be titrated at any pH. However, the stability of quaternary esters, such as 71435, is very low in most pH range. The greatest stability is generally achieved at a pH value between 2 and 4. The titration is therefore performed at pH 3.00 so that the cationic surfactants are titrated in totality.

The specific electrode consists of a PEEK stem and renewable active surface. This active surface is a moldable and plastic consistency carbon graphite paste containing ionophores (or ioncarriers). These ionophores, present at a concentration of about 0.1 percent to 1 percent, are the electro-active compounds responsible for the detection of surfactants and for the change in potential. The specific electrode is sensitive to anionic and cationic surfactants. It detects a low potential in the presence of anionic surfactants and a high potential in the presence of cationic surfactants.

During the titration, the anionic surfactants form ion pairs with hyamine 1622 and these ion pairs are transferred to the organic phase. This phenomenon causes a decrease in the quantity of available anionic surfactants and so increases the potential detected by the electrode. The electrode tends to its equilibrium potential, which corresponds to the potential in a

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medium free from surfactants and reaches the equilibrium potential at the equivalence. An excess of hyamine 1622 causes then an important potential increase, which explains the appearance of an ascending potential jump.

G-2 EQUIPMENTS

G-2.1 Common Laboratory Apparatus

G-2.2 ISE Electrode for the General Case

G-2.3 Surfactrode Refill — for the fatty compounds containing formulas

G-2.4 Ag/AgCl Reference Electrode

G-2.5 20 ml Burette Compatible with the Memotitrator

G-2.6 Helical Stirrer Compatible with the Memotitrator

G-2.7 pH Electrode

NOTES

1 The ISE electrode is not resistant to most organic solvents due to its PVC membrane. It should be stored in a dry place and adherent deposits caused by clogging have to be removed using a softpaper towel which should be dry or impregnated with methanol if necessary.

2 Before the first use, the electrode must be conditioned by performing 3 anionic titrations on any formula. These 3 titrations should not be taken into account for the rest of the analyses.

3 The active compound in the sensor is removed layer by layer during the titration, so it must be filled daily before the first analysis of the day. To do this, fill the orifice of the surfactrode with the carbon graphite paste supplied with the electrode and press the paste with the tool provided in order to obtain a smooth surface. Remove any excess with a soft non-fluffy paper towel.

CAUTION — After refilling of the active material of the sensor, it can be necessary to eliminate the first value of assay if it is too different from the other results.

4 Use necessarily glass beaker for the assay in two-phase middle because the MIBK affects the plastic.

G-3 REAGENTS

G-3.1 Titrated Solution of 0.004 M Hyamine 1622

G-3.2 0.1 N Solution of Hydrochloric Acid

G-3.3 Demineralized Water

NOTE — Hyamine 1622 is adsorbed by the silica of the glass. Consequently, at the time of first use of the glass burette, the hyamine 1622 solution must be absolutely left in the burette for about 1 h in order to saturate the glass. Then purge the burette and fill it again with the titrated solution of hyamine 1622.

G-4 METHOD OF OPERATION

Two operating modes are available in this method: one for the general case and the other one for the formulae rich in fatty compounds. The choice of the operating mode to be applied will be clarified in the working document (officialization).

G-4.1 General Case

Load the anionic surfactant titration method (*see H-4*) in the memotitrator. Weigh in the beaker, a sample of the product to be analyzed corresponding to an equivalent volume of 12 ml of 0.004 M hyamine 1622:

$$\text{Sampling (g)} =$$

$$\frac{\text{Real titer of the hyamine1622 solution (mol/l)} \times 12}{\text{Expected titer (m}_{\text{eq}}/\text{g})}$$

Add 80 ml of demineralized water. Place the solution under stirring during a few minutes in order to dissolve the product and adjust the pH to 3.00 ± 0.05 using 0.1 N HCl solution. Immerse the ISE electrode, the Ag/AgCl reference electrode and the tip of the burette in the solution to be analyzed (rinse the electrodes first using demineralized water). Start the titration with 0.004 M hyamine 1622. At the end of the titration, rinse the electrodes abundantly with demineralized water and wipe them with a soft non-fluffy paper towel.

G-4.2 Case of Formulas Containing Fatty Compounds

Load the anionic surfactant titration method (*see H-4*) in the memotitrator. Weigh, in a beaker, a sample of the product to be analyzed corresponding to an equivalent volume of 6 mL of 0.004 M hyamine 1622:

$$\text{Sampling (g)} =$$

$$\frac{\text{Real titer of Sodium Lauryl Sulfate solution (mol/l)} \times 6}{\text{Expected titer (m}_{\text{eq}}/\text{g})}$$

Add 20 ml of the MIBK/ethanol mixture (1 : 1). Place under stirring and heat to $45 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ during 5 min in order to dissolve the product to be analyzed.

CAUTION — The flash point of the mixture MIBK/ethanol (1 : 1) measured according to the standard ASTM D6450, is about $14 \text{ }^{\circ}\text{C}$.

NOTE — some substances such as starch, PEG or some silicones are not soluble in organic medium and will therefore remain in suspension.

Add 60 ml of demineralized water. Adjust the pH to 3.00 ± 0.05 using 0.1 N HCl solution. Immerse the surfactrode. Refill, the Ag/AgCl reference electrode and the tip of the burette in the solution to be titrated (rinse the electrodes first using demineralized

water). Start the titration with 0.004 M hyamine 1622 solution. At the end of the titration, rinse the electrodes abundantly with demineralized water and wipe them using a soft non-fluffy paper towel.

G-5 RESULTS

G-5.1 Calculation

$$\frac{\text{Anionic titer (\%)} \times \text{Equivalent volume (ml)} \times \text{Real titer of the hyamine 1622 solution (mol/l)}}{\text{Sampling (g)}}$$

ANNEX H

[Table 1, [Sl No. \(iv\)](#)]

DETERMINATION OF NON-IONIC AND/OR ANIONIC ACTIVE DETERGENT CONTENT

H-1 Principle

H-1.1 For Ethoxylated Non-ionic Surfactants

Ethylene oxide adducts produce a blue colour complex with cobaltthiocyanate. The complex is soluble in dichloromethane (methylene chloride) and can be extracted rapidly from an aqueous solution. The depth of colour is related linearly to the concentration for the non-ionic surfactants.

H-1.2 For Coco-glucoside Non-ionic Surfactants

Anthrone reagent produces green colour on reaction with coco-glucoside non-ionic surfactant. This method involves determination of nonionic surfactant (coco-glucoside) in Shampoo by UV spectrophotometry using photometric mode at wavelength 620 nm, *Max*.

H-1.3 For Anionic Surfactants

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

Principle for alternate method — The anionic-active matter forms a salt with the cationic dye (dimidium bromide) which dissolves in the chloroform to give this layer a pink colour. In the course of the titration, the benzethonium chloride displaces the dimidium bromide from this salt and the pink colour disappears from the chloroform layer as the dye passes into the aqueous phase. Excess benzethonium chloride forms a salt with the anionic dye (disulphine blue or acid blue 1), which dissolves in the chloroform layer and colours it greyish blue

H-2 Apparatus

H-2.1 UV Spectrophotometer

H-2.2 Separating Funnel — 125 ml capacity

H-2.3 Volumetric Flasks — with capacity of 1 litre, 250 ml, 100 ml, 50 ml, 25 ml, 20 ml and 10 ml pipettes; and 250 ml and 100 ml stopper cylinder

H-2.4 Membrane Filters (0.45 µ) — Whatman filter paper No. 41 or equivalent

H-2.5 Weighing Balance — least count 0.1 mg

H-2.6 Sonicator

H-2.7 Water Bath

H-2.8 Stop Watch — least count 1 s

H-3 REAGENTS

H-3.1 Distilled water — see [IS 1070](#)

H-3.2 Dichloromethane — analytical reagent grade (see [IS 4566](#)).

H-3.3 Acid Phosphate Buffer

Dissolve 100 g sodium dihydrogenorthophosphate, analytical reagent grade (see [IS 571](#)) in distilled water and dilute to 1 litre.

H-3.4 Cobaltthiocyanate Reagent

Dissolve 30 g cobalt nitrate (analytical reagent grade), 143 g ammonium chloride, pure (see [IS 1113](#)) and 256 g potassium thiocyanate (analytical reagent grade) in distilled water and dilute to 1 litre.

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H-3.5 Sulphuric Acid — technical grade

H-3.6 Hydrochloric Acid — technical grade

H-3.7 Formic Acid — assay ≥ 98 percent

H-3.8 Anthrone Reagent — weigh about 0.08 g of anthrone (technical grade) and dissolve in 100 ml of 80 percent sulphuric acid. Warm the solution on water bath, if required, for complete dissolution.

H-3.9 Distilled Water — see [IS 1070](#)

H-3.10 Chloroform — analytical reagent grade (see [IS 5296](#))

H-3.11 Cationic Solution (Solution A)

Weigh 1.5 ± 0.001 g of cetyltrimethyl ammonium bromide into a 250 ml beaker. Add 100 ml of distilled water and stir until dissolved. Transfer quantitatively to a 1 litre volumetric flask and make to volume. Mix thoroughly and standardize against Solution B.

H-3.12 Anionic Solution (Solution B)

Weigh accurately such amount of standard alkyl sulphate of known combined SO_3 or active content so as to give exactly 0.320 g combined SO_3 into a 250 ml beaker. Dissolve in 100 g to 200 g of warm water. Transfer quantitatively to a 1 litre volumetric flask and make to volume with water at room temperature. This is the primary standard against which Solution A is standardized. Solution B is 0.004 N.

H-3.13 Methylene Blue Indicator

Dissolve 0.1 g of methylene blue in 100 ml of water. Transfer 30 ml of this solution to a 1 litre flask. Add 500 ml of water, 6.8 ml of concentrated sulphuric acid, 50 g of sodium dihydrogenorthophosphate ($\text{NaH}_2\text{PO}_4\text{H}_2\text{O}$) and shake until solution is complete. Dilute to the mark.

H-3.14 Distilled Water — see [IS 1070](#)

H-3.15 Chloroform — analytical reagent grade (see [IS 5296](#))

H-4 PROCEDURE FOR ANIONIC SURFACTANTS CONTENT

H-4.1 Weigh accurately a sample of sufficient size to give approximately 0.320 g of combined SO_3 into a 250 ml beaker. Sample size is crucial (see Note).

Use 700 ml to 800 ml of warm water to transfer quantitatively to a 1 litre volumetric flask. Warm on steam-bath and shake gently until the sample is dissolved and solution is clear. Cool, dilute to the mark and mix thoroughly.

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

H-4.2 Pipette 10.0 ml of the sample solution into a 100 ml glass stoppered cylinder (25 mm \times 300 mm). Add $25.0 \text{ ml} \pm 0.5 \text{ ml}$ of methylene blue solution and $10.0 \text{ ml} \pm 0.5 \text{ ml}$ chloroform (see Note). Titrate with Solution A to the correct end point, shaking the cylinder carefully after each addition (to avoid emulsions) and maintaining temperature within prescribed limits of 20°C to 30°C by immersion in water bath, if necessary. As the end point is approached, the rate of transfer of colour increases and Solution A shall be added drop wise with vigorous shaking after each addition. If the approximate titration volume of Solution A is known 80 percent of the required titrating solution should be added before shaking since this avoids emulsion formation. Application of vacuum to the titration cylinder may help to break some emulsions, if formed. The end point is reached when both layers have same colour intensity. The end point is very sharp and 0.05 ml will cause a distinct change in colour distribution at or near the equivalence point.

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

H-4.3 Calculations for Anionic Surfactants Content

$$\text{Percent combined } \text{SO}_3 = \frac{V \times N \times 800}{M}$$

where

V = volume, in ml, of Solution A, used in the titration;

N = normality of Solution A; and

M = mass, in g, of the sample in the aliquot.

Percent w/w of Anionic surfactant content (H) =

$$\frac{\text{SO}_3 \times \text{Molecular mass of active detergent}}{80}$$

NOTE — For SLES detergent, the molecular mass may be taken as 400. In case sodium lauryl sulphate or sodium alpha olefin sulphonate or any other anionic detergent is used in formulation whose molecular mass is less than 350, the actual molecular mass of detergent as declared by the manufacturer may be used for calculation.

H-4.4 Determination of Anionic Active Detergent Content

H-4.4.1 Principle

The anionic-active matter forms a salt with the cationic dye (dimidium bromide) which dissolves in the chloroform to give this layer a pink colour. In the course of the titration, the benzethonium chloride displaces the dimidium bromide from this salt and the pink colour disappears from the chloroform layer as the dye passes into the aqueous phase. Excess benzethonium chloride forms a salt with the anionic dye (disulphine blue or acid blue 1), which dissolves in the chloroform layer and colours it greyish blue.

H-4.5 Reagents

H-4.5.1 The Water Used Shall be of Distilled Quality

H-4.5.2 Chloroform

H-4.5.3 Sulphuric Acid — 2.5 M solution

H-4.5.4 Sulphuric Acid — 0.5 M solution

H-4.5.5 Sodium Hydroxide — 1.0 M standard volumetric solution

H-4.5.6 Sodium Dodecyl Sulphate (Sodium Lauryl Sulphate) ($\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$) — 99 percent pure. Check the purity of the sodium lauryl sulphate and simultaneously prepare the standard solution (0.004 M).

H-4.5.7 Determination of Purity of Sodium Dodecyl Sulphate

Weigh to the nearest 1 mg, 0.2 g of the product into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of the sulphuric acid solution ([H-4.5.4](#)) using a pipette and reflux into a water condenser. During the first 5 min to 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask. In order to avoid excessive foaming, instead of refluxing the solution may be left on a boiling water bath for 60 min. After a further 10 min the solution clarifies and foaming ceases. Reflux for further 90 min. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water.

Add a few drops of the phenolphthalein solution ([H-4.5.10](#)) and titrate the solution with the sodium hydroxide solution ([H-4.5.5](#)).

Carry out a blank test by titrating 25 ml of the sulphuric acid solution ([H-4.5.4](#)) with the sodium hydroxide solution ([H-4.5.5](#)).

The purity of the sodium lauryl sulphate, expressed as a percentage, = $\frac{28.84 (V_1 - V_0) M_0}{M_1}$

where

V_1 = Volume, in ml, of sodium hydroxide solution used for the sample;

V_0 = Volume, in ml, of sodium hydroxide solution used for the blank test;

M_0 = exact molarity of the sodium hydroxide solution; and

M_1 = mass, in g, of the sodium lauryl sulphate to be checked.

H-4.5.8 Preparation of 0.004 M Sodium Dodecyl Sulphate Standard Volumetric Solution

Weigh, to the nearest 1 mg between 1.14 g and 1.16 g of sodium dodecyl sulphate and dissolve in 200 ml of water. Transfer to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to the mark with water.

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

$$M_1 = \frac{M_2 \times \text{purity} (\%)}{288 \times 100}$$

where

M_2 = mass, in g, of sodium dodecyl sulphate.

NOTE — Report normality in 5 decimal places.

H-4.5.9 Benzethonium Chloride 0.004 M Standard Volumetric Solution

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

H-4.5.10 Phenolphthalein, ethanolic solution containing 10 g/l. Dissolve 1 g of phenolphthalein in 100 ml of 95 percent (v/v) ethanol.

H-4.5.11 Mixed Indicator

H-4.5.12 Stock Solution

Weigh to the nearest 1 mg, 0.5 ± 0.005 g dimidium bromide into a 50 ml beaker, and 0.025 ± 0.002 g of acid blue 1 (disulphine blue) into a second 50 ml beaker.

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Add between 20 ml and 30 ml of hot 10 percent (v/v) ethanol to each beaker. Stir until dissolved and transfer both the solutions to a 250 ml volumetric flask. Rinse the beakers with ethanol and transfer to 250 ml volumetric flask. Dilute to the mark with 10 percent (v/v) ethanol.

H-4.5.13 Mixed Indicator Solution

Take 20 ml of the stock solution prepared above, put it in a 500 ml volumetric flask. Dilute to the mark with water. Store out of direct sunlight.

H-4.6 Apparatus

H-4.6.1 Ordinary Laboratory Apparatus

H-4.6.2 Bottles — 200 ml, glass stoppered, or measuring cylinders, flask stoppered

H-4.6.3 Burettes — 25 ml and 50 ml

H-4.6.4 One-Mark Volumetric Flask — 1 litre capacity glass stoppered

H-4.6.5 One-Mark Pipette — 25 ml

H-4.7 Procedure

H-4.7.1 Standardization of Benzethonium Chloride Solution

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

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

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

$$M = \frac{M_1 \times 10}{V_2}$$

where

M_1 = molarity of the sodium lauryl sulphate solution; and

V_2 = volume, in ml, of benzethonium chloride added.

NOTE — Repeat the procedure twice and calculate average molarity of benzethonium chloride solution and report

normality in 5 decimal places.

H-4.7.2 Procedure for Anionic Surfactant Content

Weigh to the nearest 0.01 g, approximately 1.5 g to 2.0 g of product and dissolve in approximately 50 ml hot water and cool. Add a few drops of the phenolphthalein solution ([H-4.5.10](#)) and neutralize to a faint pink colour with the sodium hydroxide solution ([H-4.5.5](#)) or sulfuric acid solution ([H-4.5.4](#)) as required. Transfer quantitatively to a 100 ml graduated flask and dilute to volume with water and mix well.

Take 10 ml of this solution into a 100 ml stoppered measuring cylinder, add 15 ml chloroform, 10 ml distilled water, and 10 ml Mixed indicator solution and Titrate with the benzethonium chloride solution as described in [H-4.5.9](#). Let n (ml) be the volume of titrant used.

H-4.8 Expression of Results

The content as a percentage by mass, of anionic-active matter (%) = $\frac{n \times T \times M}{E}$

where

n = molume, in ml, of titrant used;

T = molarity, mol/l, of benzethonium chloride titrant solution;

M = molecular weight of anionic active (as specified by the manufacturer); and

E = mass, in g, of sample.

NOTE — This method is not suitable if soap is present in the formulation. For SLES detergent, the molecular mass may be taken as 400. In case sodium lauryl sulphate or sodium alpha olefin sulphonate or any other anionic detergent is used in formulation whose molecular mass is less than 350, the actual molecular mass of detergent as declared by the manufacturer may be used for calculation.

H-5 PROCEDURE FOR DETERMINATION OF ETHOXYLATED NON-IONIC SURFACTANTS

H-5.1 Preparation of Standard Solution

H-5.1.1 Weigh 0.5 g of non-ionic surfactants (for example, tween 20 or surfactant used in manufacturing of the finished product) to the nearest 0.1 mg accuracy into a single 250 ml volumetric flask.

H-5.1.2 Dilute to volume with distilled water and mix thoroughly which is called standard stock solution.

H-5.1.3 Pipette 50 ml of the stock standard into a 100 ml volumetric flask, add 20 ml of acid phosphate buffer and dilute to volume with distilled

water and mix thoroughly. The solution thus formed is called diluted standard solution.

H-5.2 For Plotting Calibration Curve of Standard

H-5.2.1 In individual 125 ml separating funnels, add 20 ml of dichloromethane, 20 ml of cobalt thiocyanate reagent and add 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the diluted standard solution.

H-5.2.2 Stopper the funnels and shake for 1 min.

H-5.2.3 Allow the two phases to get separated. Collect the dichloromethane layer in dry stoppered test tubes after filtering through Whatman No. 41 or any equivalent filter paper, if required.

H-5.2.4 Now measure the absorbance of standards against dichloromethane as a blank at 640 nm wavelength.

H-5.2.5 Plot the graph of absorbance (Y-axis) versus volume of standard taken for extraction (X-axis).

H-5.3 Sample Preparation

H-5.3.1 Weigh 5 gm of sample to the nearest 0.1 mg accuracy into a single 250 ml volumetric flask.

H-5.3.2 Dilute to volume with distilled water and mix thoroughly which is called sample stock solution.

H-5.3.3 Pipette 50 ml of the sample stock solution into a 100 ml volumetric flask, add 20 ml of acid phosphate buffer and dilute to volume with distilled water and mix thoroughly. The solution thus formed is called diluted sample solution.

H-5.3.4 In an individual 125 ml separating funnel, add 20 ml of dichloromethane, 20 ml of cobalt thiocyanate reagent and add 20 ml of the diluted sample solution.

H-5.3.5 Stopper the funnels and shake for one minute.

H-5.3.6 Allow the phases to get separated. Collect the dichloromethane layer in a dry stoppered test tube after filtering through Whatman No. 41 or any equivalent filter paper if required. In case haziness is found in sample, then transfer the dichloromethane layer in 25 ml volumetric flask and dilute to the mark with isopropanol.

H-5.3.7 Now measure the absorbance of sample against dichloromethane as a blank at 640 nm wavelength.

H-5.3.8 From the graph of standard, measure the volume of standard corresponding to the absorbance of the sample.

H-5.4 Calculations for Ethoxylated Non-ionic Surfactants Content

From diluted standard solution (see [H-4.5.8](#)) calculate amount of non-ionic surfactants present per ml that is, mg/ml (Z).

From graph of absorbance against volume of diluted non-ionic surfactants taken for extraction, find out the volume of non-ionic surfactants for the absorbance of the sample (Y). Amount of non-ionic surfactant present in y ml of sample solution (E) = $Y \times Z$

For 100 ml of diluted sample solution contains (D) = $5 \times E$

For 250 ml of stock sample solution contains (S) = $5 \times D$

Percent w/w of non-ionic surfactants present in sample (G) = $\frac{S \times 100}{\text{Mass of sample} \times 1000}$

H-6 PROCEDURE FOR DETERMINATION OF COCO GLUCOSIDE NON-IONIC SURFACTANT

H-6.1 General UV Spectrophotometer Settings

Sl No. (1)	Measurement Type (2)	Photometry (3)
i)	Data mode	Absorbance
ii)	Wavelength Max	620 nm
iii)	Slitwidth	1.0 mm
iv)	Path length	10 mm (1.0 cm)

H-6.2 Preparation of Standard Solution

Weigh about 0.06 g of standard coco-glucoside (approximately 50 percent active) into 200 ml volumetric flask. Dissolve and dilute to 200 ml volume with water and mix well.

H-6.3 Preparation of Sample Solution

Weigh about 0.8 g of sample into 200 ml volumetric flask. Dissolve and dilute to 200 ml volume with water and mix well. Sample mass can be adjusted depending upon surfactant concentration in shampoo formulation.

H-6.4 Measurement Procedure

H-6.4.1 Pipette 1.0 ml of standard solution (see [H-6.2](#)) and sample solution (see [H-6.3](#)) into

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separate Erlenmeyer flasks of capacity 250 ml. Pipette 1.0 ml hydrochloric acid (see [H-3.6](#)) and 0.1 ml formic acid (see [H-3.7](#)) into each Erlenmeyer flask 250 ml. Pipette 8.0 ml anthrone reagent (see [H-3.8](#)) into each Erlenmeyer flask 250 ml. Precaution should be taken while adding anthrone reagent as it causes bubble formation. Place the Erlenmeyer flask on boiling water bath for 12 min (using stop watch) and allow cooling at room temperature.

H-6.4.2 Prepare a blank by proceeding as at [H-6.4.1](#) with 1.0 ml of distilled water.

H-6.4.3 Measure absorbance of standard and sample against blank solution at 620 nm and 1.0 cm path length on UV Spectrophotometer.

H-6.5 Calculations for Coco-Glucoside Non-ionic Surfactants Content

Percent, w/w of coco-glucoside non-ionic surfactants in sample:

$$(I) = \frac{A_1 \times W_0 \times P}{A_0 \times W_1}$$

where

A_1 = absorbance of sample solution;

W_0 = mass, in g, of standard;

P = purity in percent of standard;

A_0 = absorbance of standard solution; and

W_1 = mass, in g, of sample.

H-7 Percent w/w of total non-ionic and anionic surfactants present in the shampoo = $[G + H + I]$

ANNEX J

[Table 1, [Sl No. \(iv\)](#)]

METHOD FOR NON-IONIC AMPHOTERIC SURFACTANTS

J-1 OBJECTIVE OF THE METHOD

Titration of non-ionic (polyoxyethylenated type) and amphoteric (cocoyl betaines, disodium cocoamphodiacetate, cocoamidopropyl betaine, disodium cocoamphodipropionate type) surfactants present in finished products such as shampoos.

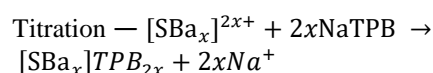
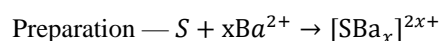
J-2 AREA(S) OF APPLICATION

This method can be applied to titration of non-ionic surfactants of polyoxyethylenated and amphoteric type, such as cocoyl betaines, disodium cocoamphodiacetate, cocoamidopropyl betaine, and disodium cocoamphodipropionate.

J-3 PRINCIPLE

Potentiometric titration of non-ionic surfactants of polyoxyethylenated type (NIO in the remainder of the document) and amphoteric surfactants (TAAM in the remainder of the document) of betaine, cocoyl betaine, disodium cocoamphodiacetate, cocoamidopropyl betaine, and disodium cocoamphodipropionate type by a solution of sodium tetraphenylborate (NaTPB).

Barium ions are added to the NIO and TAAM surfactants, with which they associate to form a pseudo-ionic compound. The pseudo-ionic compound thus formed can be titrated with sodium tetraphenylborate, which involves formation of low-solubility precipitates (Fig. 3 represents a model of the precipitation product). The following equations describe the process:



where

S = surfactants; and

NaTPB = sodium tetraphenylborate.

To the degree that mixtures of NIO and TAAM surfactants exist in the finished products, the NIO and TAAM surfactants are never homogeneous substances and precipitation with NaTPB does not follow strict stoichiometric rules.

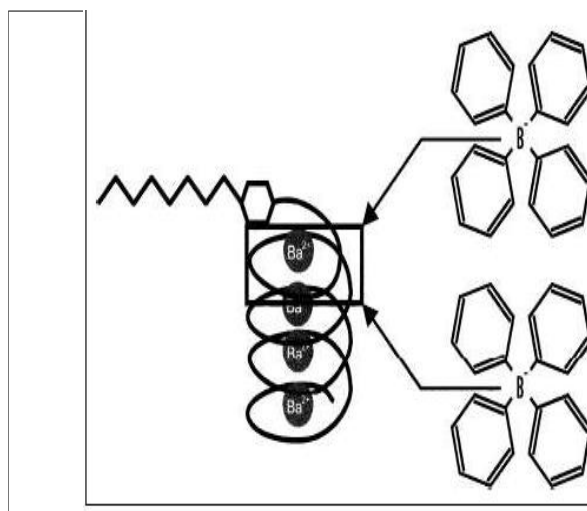


FIG. 3 MODEL OF THE PRECIPITATION PRODUCT

As for other analytic methods (for example, HPLC), a calibration factor (f) is first determined. To do this, the NIO surfactant triton X-100, defined as a reference standard against which the levels of NIO and TAAM surfactants will be measured for all the products, is used.

J-4 EQUIPMENTS

J-4.1 Mettler DL53 or Mettler DL55 Titrator or other Mettler or Metrohm Titrator

J-4.2 Propeller Stirrer

J-4.3 6.0507.010 NIO Electrode with 6.2104.020 Electrode Cable

J-4.4 6.0726.100 Ag/AgCl Reference Electrode with 6.2106.020 Electrode Cable

J-4.5 Inner Electrolyte $c(\text{KCl}) = 3 \text{ mol/l}$

J-4.6 Bridge Electrolyte $c(\text{NaCl}) = 1 \text{ mol/l}$

J-4.7 Mettler Scales

J-4.8 Precision Glassware

J-5 REAGENTS AND STANDARDS

J-5.1 Titration Reagent

J-5.1.1 Sodium Tetraphenylborate — for example, fluka No. 72020

J-5.1.2 Polyvinyl Alcohol — merck No.114266

J-5.1.3 $\text{pH} = 10.0$ Buffer Solution — for example, VWR No. 32040.298

J-5.2 Auxiliary Solution

J-5.2.1 Barium Chloride (BaCl_2) or ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) — for example, carlo erba No. 425026

J-5.2.2 Hydrochloric Acid at 37 Percent Concentration — for example, Carlo Erba No. 7647-01-0

J-5.3 Standard Reference Surfactants

Triton X-100 (alkyl phenyl polyethylene glycol) Fluka No. 93426, Merck No. 112298.

J-5.4 Standard Reagent for Measuring Titrant

Papaverine hydrochloride (for example, fluka No. 76223).

J-6 Preparation of the Solutions

J-6.1.1 Titration Reagent

J-6.1.1.1 Titration reagent — $c(\text{NaTPB}) = 0.01 \text{ mol/l}$

3.422 3 g sodium tetraphenylborate is weighed into a beaker and dissolved in 300 ml distilled water. In a second beaker, 10 g polyvinyl alcohol is also dissolved in 300 ml distilled water while heating. After cooling, both solutions are decanted, rinsing with distilled water, into a 1 l flask; 10 ml of $\text{pH} = 10.0$ buffer solution is added.

J-6.1.1.2 Auxiliary solution — $c(\text{BaCl}_2) = 0.1 \text{ mol/l}$

21 g of BaCl_2 or 25 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ is dissolved in distilled water, then mixed with 1 ml concentrated HCl, after which distilled water is added to bring the

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volumeto 1 litre.

J-6.2 Standard Titration Solution for the NaTPB Solution

It can be useful to check the actual titre of the NaTPB titration solution. The check can be carried out by titrating a standard solution of papaverine hydrochloride.

J-6.3 Preparation of Standard Surfactant Solutions

Triton X-100: weigh 0.3 g into a 100 ml volumetric flask; complete the volume with distilled water; stir until completely dissolved (stock solution).

J-6.4 Preparation, Maintenance and Storage of the NIO Electrode

The electrode is conditioned by performing two to three titrations, discarding the results. It is also recommended that a waiting time of 30 s be observed before each titration to allow the electrode to adjust to the sample matrix to be titrated.

After each series of 3 to 4 titrations, the electrode is rinsed with methanol or wiped with a tissue moistened with methanol. At the end of a series of analyses, the electrode is again wiped with a tissue moistened with methanol, then stored dry.

In the event of frequent NIO titrations, the electrode can be kept in a 1 percent solution of polyethylene glycol 1 000 (in distilled water). It is then ready for use immediately and can be used without the preparatory titrations noted above.

J-7 PROCEDURE

J-7.1 Measurement of the Actual Titre of the NaTPB Solution

0.04 g papaverine hydrochloride is weighed to within 0.1 mg in a glass beaker. It is dissolved in 100 ml water, then 3 to 4 drops concentrated HCl are added while stirring properly; titration is carried out with the sodium tetraphenylborate solution.

J-7.2 Determination of the Calibration Factor for the Standard Reference Surfactant

Collect 10 ml of the triton X-100 stock solution and pour into a 100 ml titration beaker; add 10 ml of the BaCl₂ solution and 50 ml distilled water. Then titrate with the sodium tetraphenylborate solution.

Titration of finished products (shampoos): Weigh 0.15 g of finished product into a 100 ml titration beaker, then add 10 ml of the BaCl₂ solution and 50

ml distilled water. Titrate with the sodium tetraphenylborate solution.

J-8 CALCULATIONS

J-8.1 Determination of the Actual Titre of the NaTPB Titrating Solution

Four measurements are performed each time. The resulting average value is calculated to four decimal places.

Calculation of the titration factor:

$$\text{Factor } (k) = \frac{C00 \times C01}{(EP1 \times C02 \times C03)}$$

C00 = mass, in g, of papaverine hydrochloride;

C01 = 1 000 (conversion factor);

EP1 = VEQ in ml;

C02 = 375.9 (molecular mass, g/mol, of papaverine hydrochloride in); and

C03 = 0.01 (nominal concentration, mol/l, of the titration reagent in).

J-8.2 Determination of the Calibrating Factor for the Standard Reference Surfactants

Calculation of the titre cannot be carried out directly as NIO and TAAM surfactants are never homogeneous substances and precipitation in NaTPB does not follow strictly stoichiometric rules. As for other analytic methods (for example, HPLC), a calibration factor (*f*) is first determined. To do this, the surfactant triton X-100, defined as a standard, is used. The triton X-100 surfactant is titrated as above; then the calibration factor *f* is calculated as follows:

$$f = \frac{E \times 1\,000}{V}$$

where

F = calibration factor (mg/ml);

E = test sample in g (calculated relative to 100 percent triton X-100 surfactant); and

V = volume, in ml, of NaTPB solution consumed.

J-8.3 Determination of the Surfactant Level Expressed in g%g of Triton X-100

The titre in NIO and TAAM surfactant of the samples is then calculated according to the formula:

$$\text{Surfactant titre in g\%g} = \frac{V \times f}{10 \times E}$$

where

V = volume, in ml, of NaTPB solution consumed;

F = calibration factor, in mg/ml, (previously calculated); and

E = test sample, in g.

J-7 COMMENTS/REMARKS

The dynamic control parameters of the titration should be defined so as to provide a sufficient number of measurement points for evaluation; the titration curve should be continuous and not show significant peaks (check with a derivative curve).

During titration, the solution should be optimally stirred. To achieve this, the propeller stirrer should be adjusted so that only a minimum stirring vortex is formed, and air bubbles are not introduced into the solution.

ANNEX K

[Table 1, [SI No. \(vi\)](#)]

DETERMINATION OF HEAVY METALS

K-1 OUTLINE OF THE METHOD

The colour produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.

K-2 APPARATUS

K-2.1 Nessler Cylinders — 50 ml capacity

K-3 REAGENT

K-3.1 Dilute Hydrochloric Acid — approximately 5 N

K-3.2 Dilute Acetic Acid — approximately 1 N

K-3.3 Dilute Ammonium Hydroxide — approximately 5 N

K-3.4 Hydrogen Sulphide Solution — standard

K-3.5 Standard Lead Solution — Dissolve 1.600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this

solution contains 0.01 g of lead (as Pb).

K-4 PROCEDURE

Weigh 2.000 g of material in a crucible and heat on a hot plate and then in a muffle furnace to ignite it at 600 °C to constant mass. Add 3 ml of dilute hydrochloric acid, warm (wait till no more dissolution occurs) and make up the volume to 100 ml with distilled water. Filter the solution. Transfer 25 ml of the filtrate into a Nessler's cylinder. In the second Nessler's cylinder, add 2 ml of dilute acetic acid, 1.0 ml of standard lead solution and make up the volume with water to 25 ml.

Add 10 ml of hydrogen sulphide solution to each Nessler cylinder and make up the volume with water to 50 ml. Mix and allow to stand for 10 min. Compare the colour produced in the two Nessler's cylinders. Blank determinations without samples are recommended to avoid errors arising out of reagents.

K-5 RESULTS

The sample may be taken to have passed the test, if the colour developed in the sample solution is less than that of standard solution.

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ANNEX L

[Table 1, [SI No. \(vii\)](#)]

DETERMINATION OF ARSENIC

L-1 OUTLINE OF THE METHOD

Arsenic present in a solution of the material is reduced to arsine, which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

L-2 REAGENTS

L-2.1 Mixed Acid — dilute one volume of concentrated sulphuric acid with four volumes of water. Add 10 g of sodium chloride for each 100 ml of the solution

L-2.2 Ferric Ammonium Sulphate Solution — dissolve 64 g of ferric ammonium sulphate in water containing 10 ml of mixed acid and make up to one litre.

L-2.3 Concentrated Hydrochloric Acid — *see* [IS 265](#)

L-2.4 Stannous Chloride Solution — dissolve 80 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 100 ml of water containing 5 ml of concentrated hydrochloric acid

L-3 PROCEDURE

Carry out the test as prescribed in [IS 2088](#), adding into the Gutzeit bottle, 2 ml of ferric ammonium sulphate solution, 0.5 ml of stannous chloride solution and 25 ml of sample solution as prepared in [L-2.4](#). For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

ANNEX M

(Foreword)

IDEAL PROPERTIES OF A SHAMPOO

M-1 EASE OF APPLICATION

The shampoo should be viscous enough to stay in the hand before application to the hair and scalp, yet during application the shampoo must spread easily and disperse quickly over the head and hair.

M-2 RINSING

The shampoo should rinse out easily and should not leave a residual tackiness or stickiness. It should not precipitate in hard water since insoluble calcium and magnesium salts forms a dulling film on the hair.

M-3 EASY WET COMBING

After rinsing, the hair should comb through easily without tangling.

M-4 MANAGEABILITY

The hair should be left in a manageable condition when combed dry. There should be no fly away or frizziness.

M-5 LUSTER

The hair should be left in a lustrous condition.

M-6 BODY

The hair should have body when dry, that is hair should not be limp or over conditioned.

M-7 FRAGRANCE

A fragrance should be used that not only covers any objectionable odour due to components used to formulate the shampoo but which develops a clean refreshing scent during shampooing and leaves a clean residual scent on the hair. This could be a major factor in consumer acceptance of the product.

M-8 LOW LEVEL OF IRRITATION

The formulator should try to accomplish all of the above qualities while keeping the irritation level as low as possible.

M-9 WELL PRESERVED

The product must be properly preserved against bacterial and fungal contamination.

M-10 GOOD STABILITY

The product should have good stability for at least two or three years at room temperature as well as when stored in daylight or in warehouse with low or high ambient temperatures.

M-11 ECONOMICAL

The product should not be over-formulated. The formulation should be as simple as possible using only those raw materials that are necessary to accomplish the desired goal.

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ANNEX N

(Foreword)

COMMITTEE COMPOSITION

Cosmetics Sectional Committee, PCD 19

<i>Organization</i>	<i>Representative (s)</i>
Central Drugs Standard Control Organization, New Delhi	DR RAJEEV SINGH RAGHUVANSHI (Chairperson)
All India Cosmetic Manufacturers Association, Gurugram	MS KAJAL ANAND SHRI VIRENDRA V. CHAVAN (<i>Alternate</i>)
Cavinkare Private Limited, Chennai	DR T. KUMAR DR GIREESH KUMAR (<i>Alternate</i>)
Central Drugs Standard Control Organization, New Delhi	SHRI ASEEM SAHU Ms SHRADDHA SRIVASTAVA (<i>Alternate</i>)
Central Drugs Testing Laboratory, Chennai	Ms C. VIJAYA LAKSHMI
Central Drugs Testing Laboratory, Mumbai	SHRIMATI S. U. WARDE SHRIMATI SUJATA S. KAISARE (<i>Alternate</i>)
Central Revenue Control Laboratory, New Delhi	SHRI V. SURESH SHRI SHIVRAJ SINGH (<i>Alternate I</i>) SHRI MRITUNJOY MAITY (<i>Alternate II</i>)
Chemstar Limited, Thane	SHRI SUNIL JOSHI
Colgate Palmolive India Limited, Mumbai	SHRI MANAS V. VYAS Ms SHRUTI HARDIKAR (<i>Alternate I</i>) SHRI PURUSHOTTAM JADHAV (<i>Alternate II</i>)
Consumer Guidance Society of India, Mumbai	DR SITARAM DIXIT DR M. S. KAMATH (<i>Alternate</i>)
Consumer Voice, New Delhi	SHRI H. WADHWA
CSIR - Indian Institute of Toxicology Research, Lucknow	DR R. S. RAY DR A. B. PANT (<i>Alternate</i>)
Dabur India Limited, Sahibabad	SHRI PRASUM BANDYOPADHYAY SHRI SONU PANWAR (<i>Alternate</i>)
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Directorate of Food and Drugs Administration, Bambolim	SHRIMATI JYOTI J. SARDESSAI
Drugs Control Department, New Delhi	SHRI A. K. NASA SHRI K. R. CHAWLA (<i>Alternate</i>)
Drugs Controller for the State of Karnataka, Bengaluru	SHRI P. RAMESH
EnvisBE Solutions Private Limited, Mumbai	SHRI BENEDICT M. MASCARENHAS

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Food and Drugs Administration, Panchkula	SHRI MANMOHAN TANEJA
Food and Drugs Control Administration, Ahmedabad	SHRI H. G. KOSHIA SHRI V. R. SHAH (<i>Alternate</i>)
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Godrej Consumer Products Limited, Mumbai	MS RUPINDER KAUR RAWAT DR MANOJ GAUR (<i>Alternate</i>)
Himalaya Wellness Company, Bengaluru	SHRI SUKUMARAN D. DR CHANDRIKA MAHENDRA (<i>Alternate</i>)
Hindustan Unilever Limited, Mumbai	MS VRINDA RAJWADE DR NIMISH SHAH (<i>Alternate</i>)
Hygienic Research Institute Private Limited, Mumbai	MS JAYASHREE ANAND SHRI MANOJ SARKAR (<i>Alternate</i>)
ITC Life Sciences and Technology Centre, Bengaluru	DR GURU PRASAD K. V. DR JOHN BOSCO STANISLAUS (<i>Alternate I</i>) DR JAMES BHASKAR (<i>Alternate II</i>)
Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Pharmacopoeia Commission, Ghaziabad	DR ANIL KUMAR TEOTIA DR MANOJ KUMAR PANDEY (<i>Alternate</i>)
Johnson and Johnson Private Limited, Mumbai	DR DILIP TRIPATHI SHRI RAJNEESH KUMAR (<i>Alternate</i>)
Kaya Limited, Mumbai	MS RUCHI SUSHEEL MITTAL MS MOHINI KUTE (<i>Alternate</i>)
Kelkar Education Trust's Scientific Research Centre, Mumbai	DR S. S. BARVE
Koel Colours Private Limited, Mumbai	SHRI DHRUBHAI DESAI SHRI RISHABH D. DESAI (<i>Alternate</i>)
Loreal India Private Limited, Mumbai	SHRI DHIMOY ROY DR GURUBASAVARAJA K. M. (<i>Alternate</i>)
MSME Testing Center, New Delhi	SHRI MANOJ KUMAR SHRI VIPUL GAIKWARD (<i>Alternate</i>)
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Procter and Gamble India, Mumbai	SHRI GIRISH PARHATE
Voluntary Organisation in Interest of Consumer Education (VOICE), New Delhi	SHRI M. A. U. KHAN
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Member Secretary
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(Continued from second cover)

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The composition of the Committee responsible for the formulation of this standard is given in [Annex N](#).

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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This Indian Standard has been developed from Doc No.: PCD 19 (19518).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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