

भारतीय मानक
Indian Standard

IS 17117 : 2019

शिशुओं के लिए केश शैम्पू — विशिष्टि

Hair Shampoo for Babies — Specification

ICS 71.100.70

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Price Group 5

Cosmetics Sectional Committee, PCD 19

FOREWORD

This Indian Standard 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.

BIS has published an Indian standard IS 7884 : 2004 'Shampoo — Specification (*third revision*)' which is applicable for all types of shampoos and does not categorically specify different requirements for baby hair shampoo. Baby's hair are more fragile, weaker and prone to damage than adult hair. Generally baby's hair have low density of melanin and are composed of larger fractions of non-pigmented cuticle. Compared to adult hair, baby's hair are less exposed to environmental stress conditions like dirt and dust. Hence, the requirements for baby hair shampoo are different from that for adults. While majority of requirements for hair shampoo for babies are similar to that given in IS 7884 remains the same, requirements for total non-volatiles, active surfactant content and foam height are lowered down.

Hair shampoo for babies containing 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 hair shampoo for babies. However, it is necessary that the raw materials/ingredients used are such that in the concentrations in which they would be present in hair shampoo for babies, after interaction with other raw materials/ingredients used in the formulation, are free from any harmful effects. It shall be the responsibility of the manufacturers, to satisfy themselves of the safety of their formulation before releasing the product for sale.

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 values (*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

HAIR SHAMPOO FOR BABIES — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for surfactant based hair shampoo for babies below three years age.

2 REFERENCES

The second clause in the document is to be the references in the standard. The references can be either listed in this clause or referred in Annex. The standards listed in Annex A/ listed below contain provisions which, through reference in this text, constitute provisions 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 the possibility of applying the most recent editions of the standards listed in Annex A/ listed below. In case the standards are to be referred in this clause they are to be listed as follows:

<i>IS No.</i>	<i>Title</i>
265 : 1993	Hydrochloric acid — Specification (<i>fourth revision</i>)
321 : 1964	Specification for absolute alcohol (<i>revised</i>)
571 : 2000	Monosodium phosphate — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
1113 : 1965	Specification for ammonium chloride, technical and pure (<i>revised</i>)
3958 : 1984	Methods of sampling cosmetics (<i>first revision</i>)
4011 : 1997	Methods of test for safety evaluation of cosmetics (<i>second revision</i>)
4566 : 1979	Specification for methylene chloride (dichloromethene), technical (<i>first revision</i>)
4707	Classification of cosmetic raw materials and adjuncts
(Part 1) : 2017	Colourants (<i>third revision</i>)
(Part 2) : 2017	List of raw materials generally not recognized as safe for use in cosmetics (<i>fourth revision</i>)

IS No.

Title

4955 : 2001	Household laundry detergent powders — Specification (<i>fourth revision</i>)
5296 : 1995	Chloroform, pure and technical — Specification (<i>second revision</i>)
14648 : 2011	Microbiological examination of cosmetics and cosmetic raw materials — Methods of test (<i>second revision</i>)

3 REQUIREMENTS

3.1 General Requirements

3.1.1 The hair shampoo for babies shall be in the form of a liquid, emulsion or paste. It may be coloured and/ or perfumed.

3.1.2 The liquid hair shampoo for babies, 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. If in the form of a paste, it shall be free from any agglomeration.

3.2 Ingredients

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

3.2.2 The colourants used, if any, shall be as specified in IS 4707 (Part 1) and schedule Q of the Drugs and Cosmetics Rules.

3.2.3 Ingredients other than colourants used, if any, shall comply with the provisions of IS 4707 (Part 2).

3.2.4 Formaldehyde or formaldehyde releasing agents shall not be used as ingredient in manufacture of hair shampoo for babies.

3.3 For safety evaluation of novel ingredients used in formulation of hair shampoo for babies, the hair shampoo shall comply to IS 4011.

3.4 The hair shampoo for babies shall also comply with the requirements given in Table 1 when tested as prescribed in col 4 of Table 1.

IS 17117 : 2019

Table 1 Requirement for Hair Shampoo for Babies
(Clause 3.4 and E-5.1)

Sl No.	Characteristics	Requirement	Method of Test, ref to Annex/IS
(1)	(2)	(3)	(4)
i)	Active surfactant content, as SLES, percent by mass, <i>Min</i>	2	
	a) Active detergent content or		A
	b) Active non-ionic and/or anionic detergent content		B
ii)	Non-volatile alcohol soluble matter, percent by mass, <i>Min</i>	4	C
iii)	pH at 27 ± 2°C	4.0 – 7.5	D
iv)	Foam height of two percent solution, mm, <i>Min</i>	100	E
v)	Heavy metals (as Pb), parts per million, <i>Max</i>	20	F
vi)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i>	2	G
vii)	Microbial content/limit:		
	a) Total microbial count, CFU/ml, <i>Max</i>	100	IS 14648
	b) Yeast and mould count, CFU/ml, <i>Max</i>	100	IS 14648
	c) Escherichia coli, per gram or per ml	Absent	IS 14648
	d) Pseudomonas aeruginosa, per gram or per ml	Absent	IS 14648
	e) Staphylococcus aureus, per gram or per ml	Absent	IS 14648
	f) Candida albicans, per g or per ml	Absent	IS 14648

4 PACKING AND MARKING

4.1 Packing

Hair shampoo for babies shall be packed in suitable containers.

4.2 Marking

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

- Name of the product;
- Manufacturer's name, address and registered trade-mark, if any;
- Net content in volume for liquids and emulsions or in mass for pastes;
- Month and year of manufacture;
- Batch or Lot number, in code or otherwise;
- List the ingredients (at the time of manufacture) under the title 'Ingredients' as follows:
 - For ingredients more than 1 percent (by mass or volume) — List the ingredients in decreasing order of percentage.
 - For ingredients less than 1 percent (by mass or volume) — List the ingredients in any order.

NOTE — This is exempted in case of pack sizes less than 30 g of solid/semi-solid and 60 ml of liquid.

- 'Use before ' (Month and year to be declared by the manufacturer); and

- Any other information required by statutory authorities.

4.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.2.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

5 SAMPLING

5.1 Representative samples of the hair shampoo for babies 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 hair shampoo for babies 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 the tests.

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

ANNEX A

[Table 1, Sl No. i (a)]

DETERMINATION OF ACTIVE SURFACTANT CONTENT

A-1 GENERAL

Cetyl trimethyl ammonium bromide is generally used as a routine method for analysis of hair shampoo for babies. However, in case of any dispute, benzethonium chloride solution (also known as Hyamine 1622) shall be used for determination of active detergent content as per the method prescribed in IS 4955. In this annex, Cetyl trimethyl ammonium bromide method is prescribed.

A-2 OUTLINE OF THE METHOD

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

A-3 REAGENTS

A-3.1 Cationic Solution (Solution A)

Weigh 1.5 ± 0.001 g of cetyl trimethyl ammonium bromide into a 250 ml beaker. Add 100 ml of distilled water and stir until dissolved. Transfer it quantitatively to a 1 litre volumetric flask and make to volume. Mix thoroughly and standardize against solution B (A-3.2).

A-3.2 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 to 200 g of warm water. Transfer it quantitatively to a 1 litre volumetric flask and make to volume with water at room temperature.

Solution B is 0.004 N. This is the primary standard against which solution A is standardized.

A-3.3 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 dihydrogen orthophosphate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$) and shake until solution is complete. Dilute to the mark.

A-3.4 Chloroform

Analytical reagent grade (*see* IS 5296).

A-4 PROCEDURE

A-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 to 800 ml of warm water to transfer it 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 and 12 ml but never outside 5 and 15 ml.

A-4.2 Pipette 10.0 ml of the sample solution into a 100 ml glass stoppered cylinder (25×300 mm). Add 25.0 ± 0.5 ml of methylene blue solution and 10.0 ± 0.5 ml of chloroform (*see* Note). Titrate with solution A (A-3.1) to the correct end point, shaking the cylinder carefully after each addition (to avoid emulsions) and maintaining temperature within range of 20 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 even 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 that the same volumes are used in standardizing solutions A (A-3.1) and B (A-3.2).

A-5 CALCULATION

$$\text{A-5.1 Percent combined SO}_3 = \frac{V \times N \times 800}{M}$$

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.

IS 17117 : 2019

A-5.2 Present active detergent content =

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

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.

ANNEX B

[Table 1, Sl No. i (b)]

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

B-1 PRINCIPLE

B-1.1 For Non-ionic Surfactants

Ethylene oxide adducts produce a blue colour complex with cobalthiocyanate. 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 of the non-ionic surfactants.

B-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 hair shampoo for babies by UV spectrophotometry using photometric mode at wavelength 620 nm, *Max*.

B-1.3 For Anionic Surfactants

When equivalent amounts of cationic and anionic surfactants are present in a two-phase mixture of distilled 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 surfactant can be titrated with a standard solution of cetyl trimethyl ammonium bromide.

B-2 APPARATUS

B-2.1 UV Spectrophotometer

B-2.2 Separating Funnel of 125 ml Capacity.

B-2.3 Volumetric Flasks — Of capacity 1 litre, 250 ml, 200 ml, 100 ml, 50 ml, 25 ml and 10 ml; volumetric graduated pipettes; Erlenmeyer flask of capacity 250 ml; stopper cylinder of capacity 250 ml and 100 ml; graduated beakers.

B-2.4 Volumetric flasks with capacity of 1 litre, 250, 100, 50, 25, 20 and 10 ml pipettes; and 250 and 100 ml stopper cylinder.

B-2.5 Membrane Filters (0.45 µ) Whatman filter paper No. 41 or equivalent.

B-2.6 Weighing Balance Least count...0.1 mg.

B-2.7 Desiccator

B-2.8 Water Bath

B-2.9 Stop Watch Least count...1 s.

B-3 REAGENTS

B-3.1 Dichloromethane Analytical reagent grade (*see* IS 4566).

B-3.2 Acid Phosphate Buffer

Dissolve 100 g sodium dihydrogen orthophosphate, analytical reagent grade (*see* IS 571) in distilled water and dilute to 1 litre.

B-3.3 Cobalthiocyanate 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.

B-3.4 Methylene Blue Indicator (A-3.3)

B-3.5 Chloroform Analytical reagent grade (*see* IS 5296).

B-3.6 Sulfuric Acid Technical grade.

B-3.7 Hydrochloric Acid Technical grade.

B-3.8 Formic Acid Assay ≥ 98 percent.

B-3.9 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.

B-4 PROCEDURE FOR DETERMINATION OF NON-IONIC SURFACTANTS

B-4.1 Preparation of Standard Solution

B-4.1.1 Weigh 0.5 g of non-ionic surfactants (Tween 20) to the nearest 0.1 mg accuracy into a single 250 ml volumetric flask.

B-4.1.2 Dilute it to the volume with distilled water and mix thoroughly which is called standard stock solution.

B-4.1.3 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.

B-4.1.4 For Plotting Calibration Curve of Standard

B-4.1.4.1 In individual 125 ml separating funnels, add 20 ml of dichloromethane, 20 ml of cobalt thiocyanate reagent and add 2, 4, 6, 8 and 10 ml of the diluted standard solution.

B-4.1.4.2 Stopper the funnels and shake for 1 min.

B-4.1.4.3 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.

B-4.1.4.4 Now measure the absorbance of standards against dichloromethane as a blank at 640 nm wavelength.

B-4.1.4.5 Plot the graph of absorbance (Y-axis) versus volume of standard taken for extraction (X-axis).

B-4.2 Sample Preparation

B-4.2.1 Weigh 5 g of sample to the nearest 0.1 mg accuracy into a single 250 ml volumetric flask.

B-4.2.2 Dilute to volume with distilled water and mix thoroughly which is called sample stock solution.

B-4.2.3 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.

B-4.2.4 In an individual 125 ml separating funnel, add 20 ml of dichloromethane, 20 ml of cobalto thiocyanate reagent and add 20 ml of the diluted sample solution.

B-4.2.5 Stopper the funnels and shake for one minute.

B-4.2.6 Allow the two 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.

B-4.2.7 Now measure the absorbance of sample against dichloromethane as a blank at 640 nm wavelength.

B-4.2.8 From the graph of standard, measure the volume of standard corresponding to the absorbance of the sample.

B-5 PROCEDURE FOR DETERMINATION OF COCO-GLUCOSIDE NON-IONIC SURFACTANTS

B-5.1 General UV Spectrophotometer Settings

Measurement type	Photometry
Data mode	Absorbance
Wavelength, <i>Max</i>	620 nm
Slit width	1.0 mm
Path length	10 mm

B-5.2 Preparation of Standard Solution

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

B-5.3 Preparation of Sample Solution

Weigh accurately 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.

B-5.4 Measurement Procedure

B-5.4.1 Pipette 1.0 ml of standard solution (**B-5.2**) and sample solution (**B-5.3**) into separate Erlenmeyer flasks of capacity 250 ml. Pipette 1.0 ml hydrochloric acid (**B-3.7**) and 0.1 ml formic acid (**B-3.8**) into each Erlenmeyer flask 250 ml. Pipette 8.0 ml anthrone reagent (**B-3.9**) into each Erlenmeyer flask 250 ml. Place the Erlenmeyer flask on boiling water bath for 12 min (using stop watch) and allow cooling at room temperature.

NOTE — Precaution should be taken while adding anthrone reagent as it causes bubble formation.

B-5.4.2 Prepare a blank by proceeding as at **5.4.1** with 1.0 ml of distilled water.

B-5.4.3 Measure absorbance of standard and sample against blank solution at 620 nm and 1.0 cm path length on UV Spectrophotometer.

IS 17117 : 2019

B-6 PROCEDURE FOR DETERMINATION OF ANIONIC SURFACTANTS

B-6.1 Preparation of Standard Solution

B-6.1.1 Weight 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 1). Use 700 to 800 ml of warm distilled water, to transfer quantitatively to a 1 litre volume. Warm on steam bath and shake gently till sample is dissolved and then cool the solution to room temperature, dilute to the mark and mix thoroughly.

B-6.1.2 Pipette 10 ml of the sample solution into a 100 ml glass stoppered cylinder (25 mm \times 300 mm). Add 25 ± 0.5 ml of methylene blue solution and 10 ± 0.5 ml chloroform (*see* Note 2). Titrate with solution A (**A-3.1**) to the correct end point while shaking the cylinder carefully after each addition (to avoid emulsions) and maintaining temperature within prescribed limits of 20 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 shall 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 0.1 ml will cause a distinct change in colour distribution at or near the equivalence point.

NOTES

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

2 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.

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

B-7 CALCULATIONS

B-7.1 For Non-ionic Surfactants Content

From diluted standard solution (*see* **B-4.1.3**), 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 surfactant 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 v/w of non-ionic surfactants present in sample

$$(G) = \frac{S \times 100}{\text{Mass of sample} \times 1000}$$

B-7.2 For Coco-glucoside Non-ionic Surfactants Content

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

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

Where,

A_1 = absorbance of sample solution;

W_0 = mass in g, of standard; and

P = Purity in percent, of standard;

A_0 = absorbance of standard solution;

W_1 = mass in g, of sample.

B-7.3 For Anionic Surfactants Content

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

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 surfactants content (I) =

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

B-7.4 Percent w/w of total non-ionic and anionic surfactants present in the shampoo = $G + H + I$

ANNEX C

[Table 1, SI No (ii)]

DETERMINATION OF NON-VOLATILE ALCOHOL SOLUBLE MATTER

C-1 GENERAL

This method determines the amount of non-volatile alcohol soluble matter in surfactant based hair shampoo for babies.

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 150 ml 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. 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) =

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

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:

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

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, SI No. (iii)]

DETERMINATION OF pH

D-1 APPARATUS

D-1.1 pH meter equipped with glass electrode.

D-1.2 Suitable buffer solutions.

D-2 PROCEDURE

Determine the pH at a temperature of (27± 2)°C. In case of liquid hair shampoo for babies, determine the pH directly from the sample. In case, hair shampoo is in form of emulsion or paste, mix 1 g of the sample with 9 ml of water and determine the pH of the resulting solution.

IS 17117 : 2019

ANNEX E

[Table 1, Sl No. (iv)]

DETERMINATION OF FOAM HEIGHT

E-1 OUTLINE OF THE METHOD

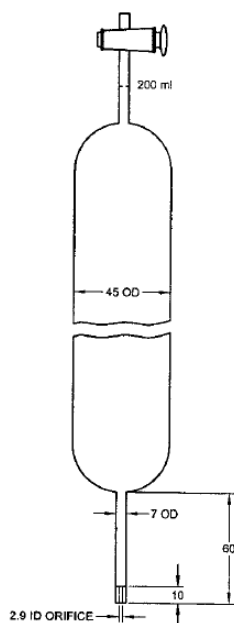
In order to check the ability of a hair shampoo to produce lather, the volume of foam obtained under specific experimental conditions is determined.

E-2 APPARATUS

E-2.1 Pipette

The pipette as shown in Fig.1 shall be constructed from standard wall chemically resistant glass tubing having the following dimensions:

- For the bulb, 45.0 ± 1.5 mm outside diameter; and
- For the lower stem, 7.0 ± 0.5 mm outside diameter.



All dimensions in millimetres.

FIG. 1 FOAM PIPETTE

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 seaks of the bulb to the stems shall be hemispherical in shape. The lower stem shall be 60 ± 2 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 ± 0.02 mm and a length of 10.00 ± 0.05 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

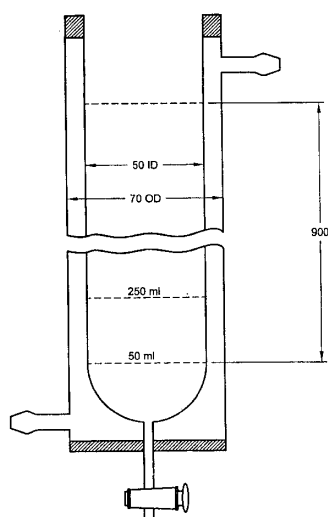
heated with a sharp pointed flame in the blow torch. The pipette shall be calibrated to contain 200.0 ± 0.2 ml at 20°C . The calibration mark shall be on the upper stem at least 15 mm below the barrel of the stopcock and shall completely encircle the stem.

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 ± 0.8 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, 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 ± 0.5 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 $48.0 \pm 0.5^\circ\text{C}$ 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 center the pipette in the receiver and bring the lower tip of the pipette lever 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.

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 $30 \pm 2^\circ\text{C}$. Add 500 ml of water to 10 g of hair shampoo for babies 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 $30 \pm 2^\circ\text{C}$ for a total period of 30 min counting the time when the hair shampoo is first added to the water.



All dimensions in millimetres.

FIG. 2 FOAM RECEIVER

E-4 PROCEDURE

While the hair shampoo solution is aging, circulate water at $30 \pm 2^\circ\text{C}$ 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 stop-watch, 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

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

E-5.1 The foam height of two percent hair shampoo solution shall not be less than the specified limit in Table 1.

ANNEX F

[Table 1, SI No. (v)]

TEST FOR HEAVY METALS

F-1 OUTLINE OF THE METHOD

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

F-2 APPARATUS

F-2.1 Nessler Cylinders — 50-ml capacity.

F-3 REAGENTS

F-3.1 Dilute Hydrochloric Acid — Approximately 5 N.

F-3.2 Dilute Acetic Acid — Approximately 1 N.

F-3.3 Dilute Ammonium Hydroxide — Approximately 5 N.

F-3.4 Hydrogen Sulphide Solution — Standard.

F-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 milliliter of this solution contain 0.01 mg of lead (as Pb).

F-4 PROCEDURE

F-4.1 Weigh about 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. 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.

F-4.2 Add 10 ml of hydrogen sulphide solution to each Nessler cylinder and make up the volume with water to

IS 17117 : 2019

50 ml. Mix and allow to stand for 10 min. Compare the colour produced in the two Nessler's cylinders. Blank determination without samples are recommended to avoid errors arising out of reagents.

F-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.

ANNEX G

[Table 1, Sl No. (vi)]

DETERMINATION OF ARSENIC

G-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.

G-2 REAGENTS

G-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.

G-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 liter.

G-2.3 Concentrated Hydrochloric Acid (*see* IS 265)

G-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.

G-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 F-4.1.

For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

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