

**PAKISTAN STANDARD SPECIFICATION**  
**FOR**  
**SYNTHETIC DETERGENT FOR GENERAL PURPOSES**



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**PAKISTAN STANDARD SPECIFICATIONS**  
**FOR**  
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**0. FOREWORD**

- 0.1** This Pakistan Standard was adopted by the Pakistan Standard and Quality Control Authority on **28-02-2017** after the draft finalized by oil, fats, soap and detergent Technical Committee had been approved by the National Standards Committee for Chemical .
- 0.2** Synthetic detergent or non-soapy detergent (NSD) as they are usually termed is products specially formulated to promote the development of detergency and comprise essential components (Surface active agent) and generally complementary components like builders etc. The NSD produced at present are mainly of the aryl type such as sodium salt of Dodecyl benzene sulphuric acid and thus they are different from soaps, the other class of detergent, which are mainly sodium salts of higher fatty acid. The limitation of soap for use in hard water areas have led to the development of synthetic detergents.
- 0.3** In the revised standard the requirements of Matter insoluble in water, percent by mass, has been revised due to harmonization with regional Standard. Assistance taken from Bangladesh Standard BDS 1445:2003 for Synthetic Detergent Powder. The option for testing method is open and relevant testing method of PS, ISO, ASTM, FAO, WHO and other International recognized standard method may be taken into account for analysis purpose . In the preparation of this standard specification the views of the representative from the manufacturers, research institutions and consumers were sought.
- 0.4** 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, shall be rounded off in accordance with PS: 103 – 1991. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**1. SCOPE:**

- 1.1 This standard prescribes requirements and methods of sampling and test for synthetic anionic detergents for general purposes.

**2. TERMINOLOGY**

- 2.1.1 For the purposes of this standard the definitions given in PS: 1399 – 1977 shall apply.

**3. TYPES**

- 3.1 The material shall be of three types based on its physical form, namely:

- a) Type 1 – Powder Detergent
- b) Type 2 - Paste, Detergent and
- c) Type 3 – Liquid Detergent

- 3.2** Powder Detergent type is further differentiate into three grade, namely:

- i) Super concentrated
- ii) Premium
- iii) Economy

**4. REQUIREMENTS:**

- 4.1 *Description* –The formulation may contain one or more of the builders of additives given in Appendix A or any other builders or additives keeping in view the end-use of the product.
- 4.2 The material shall be in the form of a free-flowing powder, paste or liquid, free from visible dirt and impurities and shall not give any unpleasant odour. It shall have good cleaning and lathering properties.
- 4.3 The material shall also comply with the requirements given in Table – 1.

**TABLE – 1**  
**REQUIREMENTS FOR SYNTHETIC DETERGENTS FOR GENERAL PURPOSES**  
**(Clause 4.3)**

Sl.#	CHARACTERISTIC	REQUIREMENTS					**Method of Test Rf. To Cl. No. In (Appendix )
		POWDER DETERGNET			Liquid Detergent	Paste Detergent	
		Super Concentrated	Premium	Economy			
(2)	(3)	(4)	(5)	(6)	(7)	(8)	
1	Moisture and volatile Matter content, percent by mass, Max	5	10	10	--	15	B
2	Active ingredient, percent by mass, Min.	30	20	10	15	20	C & D
3	Clear point, °C, Max.	--	--	--	10	--	E
4	pH of 1 percent solution ( m / v ) at 30°C	9-11	9-11	9-11	8-10	8-10	F
5	Matter insoluble in water, percent by mass, Max.	10.0	10.0	10.0	--	20	G

\*\* The relevant testing method of PS, ISO, ASTM, FAO, WHO and other International recognized standard method may be taken into account for analysis purpose.

#### 5. **PACKING AND MARKING:**

5.1 The material shall be supplied in suitable, well – closed packs / containers, as agreed to between the purchaser and the supplier.

5.2.1.1 The packs /containers shall be securely closed and legibly marked with the following information.

- a) Name of the manufacturer and address
- b) Name type and its recognized trademark, if any.
- c) Net mass when packed.
- d) Batch number or lot number in code or otherwise; and
- e) Month and year of manufacture.
- f) Best before Use

#### 6. **SAMPLING:**

6.1 Representative samples of the material shall be drawn as prescribed in Appendix H.

7. **TESTS:**

7.1 Test shall be carried out as prescribed in Appendices B to G. References to the relevant appendices is given in col. 6 of Table 1.

*QUALITY OF REAGENTS:* - Unless specified otherwise, pure chemicals and distilled water (PS: 593 – 1991) shall be employed in tests.

**APPENDIX - A**

(Clause 4.1)

1. Trisodium phosphate
2. Sodium carbonate
3. Sodium sulphate
4. Tetra sodium pyrophosphate
5. Sodium tri polyphosphate
6. Sodium hexa metaphosphate
7. Sodium carboxymethyle cellulose.
8. Sodium silicate
9. Optical brightener
10. Lather booster
11. Hydro tropes
12. Perfume
13. Preservatives
14. Chelating Agents (sequestering agents).
15. Colours

**APPENDIX - B**

[Table 1, Item (1)]

**DETERMINATION OF MOISTURE AND VOLATILE MATTER CONTENT****B.1 APPARATUS:**

- B-1.1 *Weighing dish / moisture dish* - (Porcelain or Silica Dish) 6 to 8 cm in diameter and 2 to 4 cm in depth
- B-1.2 *Desiccator* – Containing an efficient desiccant, such as phosphorus pent oxide.
- B-1.3 *Air-Oven* – preferably electrically heated, with temperature control device.

**B-2 PROCEDURE:**

- B-2.1 Weigh accurately about 5 g of the material into a dry tarred dish, and dry to constant mass in an air-oven at a temperature of  $105 \pm 1^{\circ}\text{C}$ . Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for three hours (min) or till the constant weight is staved.

**B-3 CALCULATION:**

$$\text{Moisture and volatile matter content Percent by mass} = \frac{(M - m) \times 100}{M}$$

where;

M = mass in g of the material taken for the test, and  
m = mass g of the material after drying.

**APPENDIX - C**

[Table 1, Item (ii)]

**DETERMINATION OF ACTIVE MATTER BY CATIONIC TITRATION****C.0. GENERAL:**

- C-0.1 In the method prescribed the molecular mass of active matter has been taken as 348. In practice, the molecular mass of sodium alkyl benzene sulphonate varies from 342 to 372 depending on the molecular mass of alkyl benzene used for sulphonation. This method as prescribed should, therefore be used for routine analysis. In case of any dispute or doubt, the molecular mass of sodium alkyl benzene sulphonate shall be determined as prescribed in Appendix D and then used in calculating the active matter content by this method.

**C-1 OUT LINE OF THE METHOD**

- C-1.1 A solution of the anionic detergent containing added methylene blue is shaken with chloroform, which dissolves the methylene blue salt of the detergent. The mixture is titrated with a cationic-active agent, which, after it has combined with all the free anionic detergent, begins to displace methylene blue from the salt. The end point is taken when sufficient methylene blue has been displaced into the aqueous layer to produce phases of equal colour intensity. As the reaction is not stoichiometric, it is essential to carry out standardization using a known anionic detergent similar in nature to the unknown.

**NOTE: -** *Hypochlorite and sulphate interfere with detection of the end point and should be destroyed by the addition of ferrous sulphate and hydrogen provide respectively.*

**C-2 APPARATUS:**

- C-2.1 Volumetric Flasks – 1000, 500 and 250 ml
- C-2.2 Stoppered Graduated Cylinder – 100 ml.
- C-2.3 Graduated Cylinder – 50 - ml.
- C-2.4 Burette – 25 ml
- C-2.5 Pipette -10 ml.
- C-2.6 Beaker – 250 ml

**C-3. REAGENTS:**

- C-3.1 Chloroform-chemically pure
- C-3.2 *Sulphuric Acid* – 5 N solution, carefully add 134 ml of sulphuric acid (relative density 1.84) to 300 ml of water and dilute to 1 liter.
- C-3.3 *Standard sulphuric Acid* – 1.0 N
- C-3.4 *Standard Sodium Hydroxide Solution* – 1.0 N
- C-3.5 *Standard Sodium Lauryl Sulphate Solution* – 0.004 M. Check the purity of the sodium Lauryl sulphate as given C-3.5.1 and simultaneously prepare the standard solution.
- C-3.5.1 **Determination of Purity of Sodium Lauryl Sulphate** - Weigh, to the nearest 1 mg,  $5 \pm 0.2$  g of the material into a 250-ml round bottom flask with ground-glass neck. Add exactly 25 ml of standard sulphuric acid solution (1.0 N) and reflux under a water condenser. During the first 5 to 10 minutes, 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 1 hour. After 10 minutes the solution clarifies and foaming ceases. Reflux for 1½ hour.
- 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 phenolphthalein solution and titrate with standard sodium hydroxide solution. Carry out a blank test by titrating 25 ml of sulphuric acid solution (1.0 N) with standard sodium hydroxide solution.

$$\text{Purity of the sodium lauryl sulphate, } \% = \frac{28.84 (V_1 - V_0) N_1}{M_1}$$

Where,

$V_1$  = volume in ml of standard sodium hydroxide solution used for the sample.

$V_0$  = volume in ml of standard sodium hydroxide solution used for the blank.

$N_1$  = normality of standard sodium hydroxide solution, and

$M_1$  = mass in a g of sodium lauryl sulphate under test.

- C-3.5.2 **Procedure** – Weigh to the nearest 1 mg, between 1.14 and 1.16 g of sodium lauryl 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,  $T_1$ , of the solution by means of the formula:

$$T_1 = \frac{M_2 \times \text{purity percent by mass}}{288.4 \times 100}$$

Where,

$M_2$  = mass in g of sodium lauryl sulphate taken.

- C-3.6 **Standard Benzethonium Chloride Solution** \* = 0.004 M. Weigh to the nearest 1 mg between 1.75 and 1.85 g of benzethonium chloride and dissolve in water. Transfer to a ground-glass Stoppard 1-litre one-mark volumetric flask and dilute to the mark with water.

**NOTE: 1** – In order to prepare to prepare a 0.004 M solution, dry the benzethonium chloride at  $105^\circ\text{C}$ , weigh 1.792 g to the nearest 1 mg, dissolve in water and dilute to 1 litre. While drying, take care not to raise the temperature beyond  $105^\circ\text{C}$ .

\* The full name of this chemical is benzyl di methyl 2- [ 2- $\beta$  (1,1,3,3 –tetra methyl butyl) phenoxy-ethoxy ] ethyl ammonium chloride, mono-hydrate  $[\text{CH}_3]_3 .\text{C} .\text{CH}_2 .\text{C} .(\text{CH}_3)_2 .\text{C}_6 \text{H}_4 .\text{CH}_2 .\text{OCH}_2 .\text{OCH}_2 .\text{CH}_2\text{N}(\text{CH}_3)_2 .\text{CH}_2 .\text{C}_6 \text{H}_5]^+ \text{Cl}^- .\text{H}_2\text{O}$  It is commercially sold under the name “Hyamine 1622”.

**NOTE: 2** – Other cationic reagents, such as cetyl trimethyl ammonium bromide and benzalkonium chloride, give results identical to those obtained using benzalkonium chloride. However, these tests have not been carried

out in sufficient number to make it possible to state that the results will be identical no matter what the product analysed; for that reason, if benzethonium chloride is not available it is permitted to use another reagent provided that this is stated in the test report. However, in case of doubt and always in case of a dispute, only benzethonium chloride should be used.

- C-3.7 Phenolphthalein Solution – Dissolve 1 g of phenolphthalein in 100 ml of 95 percent (v/v) ethanol.
- C-3.8 Methylene Blue Solution – 0.005 percent. Dissolve 0.05 g of methylene blue, 50 g of sodium sulphate and 6.8 ml of concentrated Sulphuric acid in water and make up the volume to 1 litre with water.
- C-3.9 Sample Solution – Weigh a suitable quantity of the sample containing 100 to 160 mg of anionic active matter per 100 ml of solution. About 1g of sodium alkyl benzene sulphonate or 3 g of the sample having around 20 percent anionic active matter content per 500 ml of the solution is suitable.

C-4 **PROCEDURE**

C-4.1 *Standardization of Benzethonium chloride Solution.*

C-4.1.1 Pipette 10 ml of standard sodium lauryl sulphate solution (see C-3.5) into a 100 ml graduated cylinder provided with a glass stopper. Add 15 ml of chloroform and 25 ml of methylene blue reagent to the cylinder. Shake well. The chloroform layer (lower) shall be coloured blue or greenish blue.

C-4.1.2 Add from the burette benzethonium chloride solution slowly initially in portions of 0.2 ml. After each addition, stopper the cylinder, shake well and allow the phases to separate. Initially the chloroform phase will be coloured blue or greenish blue. Towards the end the colour would start migrating to the aqueous layer. Note the reading at which the colour intensity in both the phases is the same when viewed under standard conditions of light, for example against a white porcelain tile under normal daylight.

C-4.1.3 Calculate the molarity of benzethonium chloride solution as follows:

$$\text{Molarity of benzethonium chloride solution, } T_2 = \frac{10 T_1}{V_1}$$

Where,

$T_1$  = molarity of sodium lauryl sulphate solution, and

$V_1$  = volume in ml of benzethonium chloride solution added.

C-4.2 *Determination of Anionic Active Matter*– Processed as described in C-4.1, taking 10 ml of the sample solution (C-3.9) instead of sodium lauryl sulphate solution.

C-4.3 *Calculation* – Calculate the anionic active under as sodium alkyl benzene sulphate as follows:

Where,

$$\text{Anionic active matter percent by mass} = \frac{348 V_2 T_2 \times 5}{M_2}$$

$V_2$  = volume in ml of benzethonium chloride solution added,

$T_2$  = molarity of benzethonium chloride solution (see C-4.1.13).

$M_2$  = mass in g of the sample taken, and

348 = molecular mass of sodium alkyl benzene sulphonate taken for calculation.

**A P P E N D I X - D**

(Caluse C-0.1)

**DETERMINATION OF MOLECULAR MASS OF SULPHONIC ACID OR SODIUM SALT OF SULPHONIC ACID****D-1 APPARATUS:**

- D-1.1 *Beakers* – 150 ml and 1000 ml capacity.
- D-1.2 *Buchner Flask* – 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4 ).
- D-1.3 *Evaporating Basin*
- D-1.4 *Separating Funnel* – 1000 ml capacity.
- D-1.5 *Steam-Bath*
- D-1.6 *Wide-mouthed Flat-Bottomed Flask* – 200 ml capacity.
- D-1.7 *Air-Oven* – preferably electrically heated with temperature control device.

**D-2 REAGENTS:**

- D-2.1 *Caustic Soda Solution* - 10 percent (m/v).
- D-2.2 *Ethyl Alcohol* – 30 percent, 96 percent, and absolute (v/v)
- D-2.3 *Diethyl Ether*
- D-2.4 *Acetone*
- D-2.5 *Phenolphthalein Indicator* – 1 percent solution in 95 percent (v/v) ethyl alcohol.
- D-2.6 *Methyl Orange Indicator* – 0.1 percent (m/v).
- D-2.7 *Ferric Ammonium Sulphate Indicator* – Saturated solution.
- D-2.8 *Standard Sulphuric Acid* – Approximately 0.1 N.
- D-2.9 *Standard Ammonium Thiocyanate Solution* – Approximately 0. 1N.
- D-2.10 *Standard Silver Nitrate Solution* – Approximately 0. 1 N.
- D-2.11 *Nitric Acid* – Concentrated, relative density 1.42.
- D-2.12 *Nitrobenzene*

**D-3 PROCEDURE:**



- D-3.1 Weigh about 2 g of the material into a 150-ml beaker. Dissolve in minimum quantity of water and neutralize with caustic soda solution. Evaporate on a steam-bath to almost complete dryness. Digest with 50 ml of 96 percent ethyl alcohol by heating on steam-bath for about 2 minutes. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which solution is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30-ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and finally, wash the residue several times with hot ethyl alcohol to remove all the alcohol soluble. Evaporate the combined filtrate to a small bulk in an evaporating dish and transfer it to a separating funnel. Rinse the evaporating dish once with 50 ml of 96 percent ethyl alcohol and then four times with 50-ml portions of water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether, swirl gently to ensure adequate mixing, and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker, and combine the three ether extracts.
- D-3.2 Take the combined ether extracts in a clean separating funnel. Wash three times with successive 50-ml portion of 30 percent ethyl alcohol and then with successive 50-ml portion of water until the ether phase is free from alcohol usually 7 to 10 water washes are necessary. Combine all the alcoholic and aqueous extracts, neutralize to phenolphthalein and evaporate on a steam-bath until the volume of absolute alcohol and evaporate to dryness. The solution shall remain just pink to phenolphthalein throughout evaporation. To ensure that the residue is completely anhydrous, add 30 ml of absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with six further consecutive 30-ml portions of hot 96 percent ethyl alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 ml of hot 96 percent ethyl alcohol from the jet of a wash bottle.
- D-3.3 Transfer the filtrate and washings in the Buchner flask to a wide mouth flat-bottomed flask, evaporate nearly to dryness on a water-bath, and drive off the remaining solvent by directing a gentle stream of dry air into the flask whilst continuously rotating the latter in the water-bath. A thin film of active matter, easy to dry, is thereby obtained. Add 10 ml of acetone, evaporate and remove the last traces of solvent as described above, cool in a desiccator and weigh. Heat the flask for not more than 5 minutes in an air-oven at a temperature of  $100 \pm 1^{\circ}\text{C}$ , gently blow out with as current of air, cool and re-weigh. Repeat this drying process until the difference between two successive weighing does not exceed 3 mg.
- D-3.4 The extract obtained contains the active matter, some sodium chloride and possibly traces of alkali carbonates, which may have passed through the filter in the presence of the detergent. Find out the percentage of sodium carbonate and sodium chloride in the extract by using a portion of the extract, as it will be prescribed in D-3.4.1. and D-3.4.2 respectively.

D-3.4.1 *Determination of Alkali Carbonates* – Weigh accurately about 1 g of the extract. Dissolve it in cold water, add a few drops of methyl orange indicator and titrate with standard sulphuric acid to methyl orange end point.

D-3.4.1.1 *Calculation:*

$$\text{Mass in g of sodium carbonate} = 0.053V_1 N_1 \times \frac{M_1}{M_2}$$

where;

$V_1$  = volume in ml of standard sulphuric acid solution used.

$N_1$  = normality of the standard sulphuric acid solution.

$M_1$  = mass in g of the total extract (see D-3.3), and

$M_2$  = mass in g of the extract take for analysis.

D-3.4.1.2 Reserve the solution for the estimation of chlorides.

D-3.4.2 *Determination of Chlorides* – To the solution remaining after the estimation of alkali carbonates (see D-3.4.1.2), and add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

D-3.4.2.1 **Calculation**

$$\text{Mass in g of sodium chloride} = 0.0585(20N_3 - V_2N_2) \times \frac{M_1}{M_2}$$

where;

$V_2$  = volume in ml of standard ammonium thiocyanate solution used

$N_2$  = normality of the standard ammonium thiocyanate solution.

$N_3$  = normality of standard silver nitrate solution.

$M_1$  = mass in g of the total extract (see D-3.3), and

$M_2$  = mass in g of the extract taken for analysis in D-3.4.1.

D-3.5 Weigh accurately about 1 g of the extract. Dissolve in water and make up to 500 ml. Follow the titration procedure given in C-4.1 taking 10 ml of the solution for titration.

D-4. *Calculation:*

$$\text{Molecular mass of sodium Salt of sulphonic acid} = \frac{(100 - M_1 - M_2) \times M}{5 \times V_1 \times T_1}$$

where

$M_1$  = percent by mass of sodium carbonate (see D-3.4.1).

$M_2$  = percent by mass of sodium chloride (see D-3.4.2).

$M$  = mass in g of the extract taken.

$V_1$  = volume in ml of benzethonium chloride solution added and

$T_1$  = molarity of benzethonium chloride solution.

**A P P E N D I X - E**

[Table 1, Item (iii)]

**DETERMINATION OF CLEAR POINT TEMPERATURE****E-0 GENERAL:**

E-01 This method is not applicable to materials showing reverse solubility temperature effect.

E-0.2 This clear point is defined as the temperature at which the clarification occurs under the conditions of the test.

**E – 1 APPARATUS:**

E-1.1 *Beaker* – 2 litre capacity to serve as a water-bath.

E-1.2 *Wide – Mouthed Bottle* - 450 ml capacity, height 190 mm and inside diameter of neck 38 mm.

E – 1.3 *Test Tube* – 100 mm in length and 25 mm in diameter.

E – 1.4 *Stirrer* – Made of stainless steel or glass with one end bent in the form of a loop of 19 mm outside diameter.

E – 1.5 *Thermometer* – Range – 80<sup>0</sup> to 100<sup>0</sup>C.

E – 1.6 *Cooling Mixture* – Isopropyl alcohol or ethanol to which solid carbon dioxide is added as required.

**E – 2 PROCEDURE**

E – 2.1 Place 25 ml of the liquid product itself in one of the tubes and cool it whilst carefully stirring to avoid incorporation of air bubbles, until solid material under test becomes solid and opaque. If no solid material separates after cooling up to 0<sup>0</sup>C, the test shall be discontinued and the sample shall be taken as passing the requirement of the test.

E – 2.2 After solid separation has taken place, heat the bath gradually whilst stirring, until the solution becomes clear. Note the temperature at which this occurs and record it as the clear point of the solution.

**APPENDIX - F**

[Table 1, Item (IV)]

**DETERMINATION OF pH**

F – 0.1 pH determination shall be made in an acid-free atmosphere.

F – 0. **GENERAL**

F – 1. **APPARATUS:**

F – 1.1 *pH Meter* – Any standard electrometric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solution (see F-2.2) before use.

F – 1.2 *Volumetric Flask* – 1000 ml capacity.

F – 1.3 *Beaker* – 100 ml capacity

F – 2. **REAGENTS:**

F – 2.1 Distilled water – Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide, and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 30 °C. The residue on evaporation when heated at 105 °C for one hour shall not exceed 0.5 mg per litre.

F – 2.2 *Standard Buffer Solutions* – Any two suitable buffer solutions within the pH range of 7 to 11 at 30 °C for calibrating the pH meter.

F -3 **PROCEDURES:**

F – 3.1 Weigh  $10 \pm 0.001$  g of the material and transfer to 1-litre volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to  $30 \pm 0.5$  °C, and fill to the calibration mark with distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 30 °C for two hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.

**APPENDIX - G**

[Table 1, Item (v)]

**DETERMINATION OF MATTER INSOLUBLE IN WATER****G.1. PROCEDURE:**

G-1.1 Weigh accurately about 5 g of the material into a beaker and digest with 50 ml of ethyl alcohol by heating on a steam-bath for about 2 minutes. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Bucher flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30-ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and finally wash the residue several times with hot ethyl alcohol to remove the alcohol soluble.

G-1.2 Change the receiver, extract the residue with successive portion of distilled water at about 60°C, and wash the residue several times to remove all the water soluble. Dry the sintered glass funnel with the residue in an air-oven at temperature of 105 ± 2°C until constant mass is obtained.

**G-1.3 CALCULATION**

$$\text{Matter insoluble in water Percent by mass} = 100 \frac{m}{M}$$

Where,

m = mass in g of matter insoluble in water, and

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

**APPENDIX - H**

(Clause 5.1)

**SAMPLING PROCEDURE FOR SYNTHETIC DETERGENTS****H-1 GENERAL REQUIREMENTS**

H-1.1 In drawing, preparing, storing and handling samples, the following precautions shall be observed.

H-1.1.1. Samples shall be taken in a protected place, not exposed to damp air, dust or soot.

H-1.1.2. The sampling instruments shall be clean and dry when used.

H-1.1.3. The samples, the materials being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

H-1.1.4. The samples shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

H-1.1.5 Each container shall be sealed airtight after filling and marked with full detail of sampling, date of sampling, batch or code number, name of manufacturer, and other important particulars of the consignment.

H-1.1.6 The sample shall be stored in such a manner that the temperature of material does not vary unduly from the normal temperature and that they are protected from light.

## H-2 SCALE OF SAMPLING

H-2.1 *Lot* – In a single consignment all the packages containing materials of same type, and drawn from the same batch of manufacture shall constitute a lot. If the consignment consists of packages containing detergents of different types, then the packages containing detergents of same type and batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

H – 2.2 For ascertaining the conformity of lot to the requirements describe in the standard, test shall be carried out on each lot separately. The number (n) of packages to be selected for drawing the sample shall depend upon the size (N) of the lot, and shall be accordance with Table – 2.

**TABLE 2 SCALE OF SAMPLING**

NO. OF PACKAGES IN THE LOT (N)	NO. OF PACKAGES TO BE SELECTION (N)
04 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

**NOTE:** When the size of the lot is 3 packages or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

H-2.3 The packages shall be selected at random. In order to ensure the randomness of selection a random number table shall be used. For guidance and use of random number tables, PS: ----- may be used.

In the absence of a random number table, the following procedure may be adopted.

‘Starting from any package in the lot, count them in one order as 1,2,3, up to ‘r’ and so on where ‘r’ is the integral part of  $N/n$  (N being the lot size and n the number of packages to be selected). Every rth package thus counted shall be withdrawn to give the required sample size.

## H-3 Preparation of gross samples, test samples and referee samples:

### H-3.1 Gross Samples

H-3.1.1 From each one of the packages selected as in H-2, draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for purposes of test as indicated in H-4.

H-3.1.1.1 The material from the containers selected as in H-3.1.1. shall be disintegrated, if necessary, and mixed thoroughly to give the gross sample for the package.

### H-3.2 Test Samples

H-3.2.1 Segregate carefully the gross samples. From each gross sample take a small but equal quantity of material and mix it thoroughly into a composite sample, which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under H-4. The composite samples representing each type of synthetic detergent shall be divided into three equal parts – one for the purchaser, another for the supplier and the third for the referee.

H-3.2.2 The remaining portion of the material in each one of the gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  selected packages shall be for the purchaser, another for the supplier and the third for the referee.

H-3.2.3 All the composite and individual samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers, and labeled with full particulars of identification given in H-1.1.5

### H-3.3 Referee Samples

H-3.3.1 The referee samples shall consist of a composite sample and a set of  $n$  individual samples. All the containers shall bear the seals of both the purchaser and the supplier, and shall be kept at industry of authorized person.

H-3.3.2 Referee samples shall be used in cases of any dispute between the purchaser and the supplier.

### H-4 Number of Tests:

H-4.1 Test for the determination of active ingredient shall be performed on each of the individual samples.

H-4.2 Test for the determination remaining characteristics specified in Table 1 shall be conducted on the composite sample.

### H-5 Criteria for conformity

H-5.1 For Individual Samples – For the characteristics which has been determined on the individual sample, then mean ( $\bar{x}$ ) and the range ( $R$ ) of test results shall be calculated as follows:

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

Range ( $R$ ) = The difference between the maximum and minimum values of test results.

- a) The value of the expression  $(\bar{X} - KR)$  shall be calculated from the relevant test results [ see also H-5.1 (b) ]. If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement for that characteristic.

- b) The value of the factor K referred to in H-5.1. (a) shall be chosen in accordance with Table 3, depending upon the acceptable quality level, that is the percentage of non-conforming packages that may be tolerated reasonably.

**TABLE 3****VALUE OF 'K' FOR ACHIEVING DIFFERENT ACCEPTABLE QUALITY LEVELS**

[Clause H-5.1 (b)]

<b>Acceptable Quality Level</b> (1)	<b>Value of "K"</b> (2)
Not more than 3.0 percent defective	0.4
Not more than 1.5 percent defective	0.5
Not more than 0.5 percent defective	0.6

H-5.2

*For composite Sample* – For declaring the conformity of the lot to the requirements of all the remaining characteristics determined on the composite sample, the test results for each one of the characteristics shall satisfy the relevant requirement given in Table 1 of this standard.

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