IS 4956 (2002): Synthetic Detergent for Industrial Purposes
[CHD 25: Soaps and other Surface Active Agents]

BLANK PAGE

भारतीय मानक

औद्योगिक प्रयोजनों के लिए संशिलिष्ट अपमार्जक — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

SYNTHETIC DETERGENTS FOR INDUSTRIAL PURPOSES — SPECIFICATION

(Second Revision)

ICS 71.100.40

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2002

Price Group 7

Soaps and other Surface Active Agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Synthetic detergents or non-soapy detergents (NSD), as they are usually termed, are products specially formulated to promote the development of detergency, and comprise essential components (surface active agents), and generally, complementary components like builders, etc. The NSD produced in this country at present are mainly of the alkyl aryl type, such as sodium salt of dodecyl benzene sulphonic acid and thus they are different from soaps, the other class of detergents, which are mainly sodium salts of higher fatty acids. The limitations of soaps for use in hard water areas have led to the development of synthetic detergents.

This standard was first published in 1968 and subsequently revised in 1977, during which the requirement for matter insoluble in alcohol and non detergent organic matter were dropped, the limit for moisture content was reduced and the requirement for active ingredient for Type 2 of the material was increased from 30 to 40 percent and that for Type 1 was reduced from 40 to 35 percent based on that periods industrial practice. The method for determination of active matter by cationic titration was also incorporated.

This revision is taken up to incorporate the declaration of ingredients used in the manufacture of detergents in the marking clause and also to incorporate the existing six amendments to this standard.

This standard contains **5.1** which calls for agreement between the purchaser and the supplier.

A scheme for labelling environment friendly products known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF), Government of India. The ECO Mark would be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 1986 as the Resolutions No. 71 dated 21 February 1991 and No. 425 dated 28 October 1992 published in the Gazette of the Government of India. For a product to be eligible for marking with ECO logo, it shall also carry the ISI Mark of BIS besides meeting additional environment friendly requirements. The requirement to be satisfied for a product to qualify for the BIS Standard Mark for ECO friendliness, has been included in this revision. These requirements will be optional; manufacturing units will be free to opt for the ISI mark alone also.

It is necessary that the raw materials used in the production are such that in the concentrations in which they will be present in the finished product, after interaction with the raw material used in the formulation, are free from any harmful effects. For determining the suitability of a new formulation or of a new raw material used in old formulations in the skin, reference may be made to IS 11601 (Part 1).

The Composition of the Committee responsible for formulation of this standard is given in Annex J.

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

SYNTHETIC DETERGENTS FOR INDUSTRIAL PURPOSES — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for synthetic anionic detergents for industrial purposes.

2 REFERENCES

The Indian Standards 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 indicated below:

IS No.	*Title*
286 : 1978	Methods of sampling and test for soaps (*second revision*)
1070 : 1992	Reagent grade water(*third revision*)
4707 (Part 1) : 1988	Classification for cosmetics raw materials and adjuncts: Part 1 dyes, colours and pigments (*first revision*)
4905 : 1968	Methods for random sampling
7597 : 1974	Glossary of terms relating to surface active agents
8401 : 1994	Alkyl benzene sulphonic acid (acid slurry) (*first revision*)
11601	Method of safety evaluation of synthetic detergent:
(Part 1) : 1986	Method of test for irritant potential of synthetic detergents
(Part 2) : 1992	Method of test for skin sensitization potential of synthetic detergents (Guinea pig maximization test)
13933 : 1995	Method of test for ready biodegradability of surface active agents (Modified Strum test)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 shall apply.

4 TYPES

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

a) *Type 1* - Powder or Flakes,

b) *Type 2* - Paste, and

c) *Type 3* - Liquid.

5 REQUIREMENTS

5.1 Description

The active ingredient shall be the sodium salt of alkyl benzene sulphonic acid conforming to IS 8401. The formulation may contain one or more of the builders or additives given in Annex A or any other builders or additives keeping in view the end-use of the product.

5.2 The material shall be in the form of free flowing powders, paste or clear liquid, free from visible dirt and impurities. It shall not give any unpleasant odour. It shall have good cleaning and lathering properties.

5.3 The material shall pass the test for skin sensitization potential when evaluated as per the method prescribed in IS 11601(Part 1) and for skin sensitization potential when evaluated as per the method prescribed in IS 11601 (Part 2).

5.4 The material shall also comply with the requirements given in Table 1.

5.5 Additional Requirements for ECO Mark

5.5.1 *General Requirements*

5.5.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under **5.1** to **5.4**.

5.5.1.2 The manufacturer shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water (Prevention and Control of Pollution) Act,* 1974 and *Air (Prevention and Control of Pollution) Act,* 1981 along with the authorization, if required, under the *Environment (Protection) Act,* 1986 while applying for ECO Mark.

5.5.2 *Specific Requirements*

5.5.2.1 The material shall not contain any phosphate when tested as per the method given in Annex D of IS 4955.

5.5.2.2 The material shall pass the test when evaluated for irritant potential as per the method prescribed in IS 11601 (Part 1) and for skin sensitization potential when evaluated as per the method prescribed in IS 11601 (Part 2).

5.5.2.3 The active ingredient used in the manufacture of synthetic detergents for industrial purposes shall be readily biodegradable when tested as per IS 13933.

6 PACKING AND MARKING

6.1 Packing

6.1.1 The material shall be supplied in HDPE drums or in suitable, well-closed containers, as agreed to between the purchaser and the supplier.

6.1.2 For ECO Mark the product shall be packed in such packages which are made from recyclable/reusable or biodegradable material and declared by the manufacturer and may be accompanied with detailed instructions for proper use.

6.2 Marking

6.2.1 The containers shall be legibly and indelibly marked with the following information:

a) Name of the manufacturer;
b) Name, type and recognized trade-mark of the product, if any;
c) Net mass when packed;
d) Batch No. or lot No. in code or otherwise;
e) Month and year of manufacture; and
f) The following identified critical ingredients in descending order of quantity, percent by mass:
    1) Active ingredients,
    2) Builders used,
    3) Soda ash,
    4) Fillers, and
    5) Enzymes, if added.

6.2.2 *Additional Marking Requirements for ECO Mark*

The package shall also be marked with the criteria for which the product has been labelled as ECO Mark.

6.2.3 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

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

7 SAMPLING

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

8 TESTS

8.1 Tests shall be carried out as prescribed in Annexes B to G. Reference to the relevant annex is given in col 6 of Table 1.

8.2 Quality of Reagents

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

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

Table 1 Requirements For Synthetic Detergents for Industrial Purposes

(*Clauses* 5.4 *and* 8.1)

Sl No.	Characteristic	Requirements			Method of test, Ref to Annex
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture and volatile matter content, percent by mass, *Max*	5	—	—	B
ii)	Active ingredient, percent by mass, *Min*	35	40	20	C & D
iii)	Clear point, °C, *Max*	—	—	10	E
iv)	*p*H of 1 percent (*m/v*) at 30°C	7 to 9	7 to 9	6 to 8	F
v)	Matter insoluble in water, percent by mass, *Max*	0.5	0.5	Nil	G

ANNEX A

(*Clause* 5.1)

LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

1. Trisodium phosphate
2. Sodium carbonate
3. Sodium sulphate
4. Tetrasodium pyrophosphate
5. Sodium tripolyphosphate
6. Sodium hexametaphosphate
7. Sodium carboxymethyl cellulose
8. Sodium silicate
9. Optical brightener
10. Lather boosters
11. Hydrotropes
12. Perfume
13. Preservatives
14. Chelating agents (sequestering agents)
15. Colours

ANNEX B

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

DETERMINATION OF MOISTURE AND VOLATILE MATTER

B-1 APPARATUS

B-1.1 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 pentoxide.

B-1.3 Air-Oven — preferably electrically heated, with temperature control device for maintaining temperature at 105 ± 1°C.

B-2 PROCEDURE

Weigh accurately about 5 g of the material into a dry tared dish, and dry to constant mass in an air-oven at a temperature of 105 ± 1°C. Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for one-hour period shows a difference of not more than 5 mg in the net loss in mass.

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 in g of the material after drying.

ANNEX C

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF ACTIVE MATTER BY CATIONIC TITRATION

C-1 GENERAL

In the method prescribed, the molecular mass of active matter has been taken as 342. In practice, the molecular mass of sodium alkyl benzene sulphonate varies from 337 to 347 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 Annex D and then used in calculating the active matter content by the method.

C-2 OUTLINE OF THE METHOD

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 — Hypochlorites and sulphites interfere with detection of the end point and should be destroyed by the addition of ferrous sulphate and hydrogen peroxide respectively.

C-3 APPARATUS

C-3.1 Volumetric Flasks — 1 000, 500 and 250 ml.

C-3.2 Stoppered Graduated Cylinder — 100 ml.

C-3.3 Graduated Cylinder — 50 ml.

C-3.4 Burette — 25 ml.

C-3.5 Pipette — 10 ml.

C-3.6 Beaker — 250 ml.

C-4 REAGENTS

C-4.1 Chloroform — Chemically pure.

C-4.2 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 litre.

C-4.3 Standard Sulphuric Acid — 1.0 N.

C-4.4 Standard Sodium Hydroxide Solution — 1.0 N.

C-4.5 Standard Sodium Lauryl Sulphate Solution

0.001 M. Check the purity of the sodium lauryl sulphate as given in **C-4.5.1** and simultaneously prepare the standard solution.

C-4.5.1 *Determination of Purity of Sodium Lauryl Sulphate*

Weigh to the nearest 1 mg, 5 ± 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 or one hour. After 10 minutes the solution clarifies the foaming ceases. Reflux for 1½ h. 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.

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

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 g of sodium lauryl sulphate under test.

C-4.5.2 *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-4.6 Standard Benzethonium Chloride Solution[1]

0.004 M. Weigh to the nearest 1 mg between 1.75 g and 1.85 g of 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.

NOTES

1 In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105°C, weigh 1.792 to the nearest, 1 mg, dissolve in water and dilute to 1 litre. While drying take care not to raise the temperature beyond 105°C.

2 Other cationic reagents, such as cetyl trimethyl ammonium bromide and benzalkonium chloride, give results identical to those obtained using benzethonium 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-4.7 Phenolphthalein Solution

Dissolve 1 g of phenolphthalein in 100 mg of 95 percent (*v/v*) ethanol.

C-4.8 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-4.9 Sample Solution

Weigh a suitable quantity of the sample containing 100 to 160 mg of anionic active matter per 100 ml of solution. About 1 g 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-5 PROCEDURE

C-5.1 Standardization of Benzethonium Chloride Solution

C-5.1.1 Pipette 10 ml of standard sodium lauryl sulphate solution (*see* **C-4.5**) into a 100-ml graduated cylinder provided with a glass stopper. Add 15 ml of chloroform and 25 ml of methylene blue reagent to cylinder. Shake well. The chloroform layer (lower) shall be coloured blue or greenish blue.

C-5.1.2 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-5.1.3 Calculate the molarity of benzethonium chloride solution, T_2, as follows:

$$T_2 = \frac{10\, T_1}{V_1}$$

where

T_1 = molarity of sodium lauryl sulphate solution, and

V_1 = volume in ml of benzethonium chloride solution added.

C-5.2 Determination of Active Ingredient

Proceed as described in **C-5.1**, taking 10 ml of the sample solution (**C-4.9**) instead of sodium lauryl sulphate solution.

C-5.3 Calculation

Calculate the active ingredient as sodium alkyl benzene sulphate as follows:

$$\text{Active ingredient, percent by mass} = \frac{342\, V_2 T_2 \times 5}{M_2}$$

where

V_2 = volume in ml of benzethonium chloride solution added,

T_2 = molarity of benzethonium chloride solution (*see* **C-5.1.3**),

M_2 = mass in g of the sample taken, and

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

---

[1] The full name of this chemical is benzyl dimethyl 2-[2-*p*(1,1,3,3-tetramethyl-butyl) phenoxy-ethoxy] ethyl ammonium chloride, monohydrate $[(CH_3)_3\ C.CH_2.C.(CH_3)_2\,C_6H_4CH_2.OCH_2\,OCH_2\,CH_2N(CH_3)_2\,CH_2.C_6H_5]^+\,Cl^-\,H_2O$.

It is commercially sold under the name 'Hyamine 1622'

ANNEX D

[*Table* 1, *Sl No.* (ii) *and Clause* C-1]

DETERMINATION OF MOLECULAR MASS OF SULPHONIC ACID OR SODIUM SALT OF SULPHONIC ACID

D-1 APPARATUS

D-1.1 Beakers — 150 ml and 1 000 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 — 1 000 ml capacity.

D-1.5 Steam-Bath

D-1.6 Wide-Mouthed Flat-Bottomed Flask — 200 ml capacity.

D-1.7 Air-Oven — to maintain a temperature of 100 ± 1°C, 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 0.1 N.

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 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 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 portions of 30 percent ethyl alcohol and then with successive 50 ml portions 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 is reduced to about 25 ml. Add an equal 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 ingredient, 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 temperature of 100 ± 1°C, gently blow out with a current of air, cool and re-weigh. Repeat this drying process until the difference between two successive weighings does not exceed 3 mg.

D-3.4 The extract obtained contains the active ingredients, some sodium chloride and possible 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 as 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*

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

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 taken 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**), 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*

$$\text{Mass in g of sodium chloride} = 0.058\,5\,(20\, N_3 - V_2 N_2) \times \frac{M_1}{M_2}$$

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 the 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.

ANNEX E

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF CLEAR POINT TEMPERATURE

E-1 GENERAL

E-1.1 This method is not applicable to materials showing reverse solubility temperature effect.

E-1.2 The clear point is defined as the temperature at which the clarification occurs under the conditions of the test.

E-2 APPARATUS

E-2.1 Beaker — 2-litre capacity to serve as a water-bath.

E-2.2 Wide-Mouthed Bottle — 450-ml capacity, height 190 mm and inside diameter of neck 38 mm.

E-2.3 Test Tube — 100 mm in length and 25 mm in diameter.

E-2.4 Stirrer — Made of stainless steel or glass with one end bent in the form of a loop of 19 mm outside diameter.

E-2.5 Thermometer — Range –10°C to 100°C.

E-2.6 Cooling Mixture — *Iso*-propyl alcohol or ethanol to which solid carbon dioxide or ice-salt mixture is added as required.

E-3 PROCEDURE

E-3.1 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 separates, and the material under test becomes solid and opaque.

E-3.2 If no solid material separates after cooling up to 0°C, the test shall be discontinued and the sample shall be taken as passing the requirement of the test.

E-3.3 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.

ANNEX F

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF *p*H

F-1 GENERAL

*p*H determination shall be made in an acid-free atmosphere.

F-2 APPARATUS

F-2.1 *p*H 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-3.2**) before use.

F-2.2 Volumetric Flask — 1 000 ml capacity.

F-2.3 Beaker — 100 ml capacity.

F-3 REAGENTS

F-3.1 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 carbon dioxide, and shall be protected with soda lime or soda asbestos while cooling and in storage. The *p*H 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/l.

F-3.2 Standard Buffer Solution

Any two suitable buffer solutions within the *p*H range of 9 to 11 at 30°C for calibrating the *p*H meter.

F-4 PROCEDURE

Weigh 10 ± 0.001 g of the material and transfer to a one 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 ± 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 *p*H. Measure the *p*H of the solution using a glass electrode.

ANNEX G

[*Table* 1, *Sl No.* (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 Buchner 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 all the alcohol solubles.

G-1.2 Change the receiver, extract the residue with successive portions of distilled water at about 60°C, and wash the residue several times to remove all the water solubles. Dry the sintered glass funnel with the residue in an air-oven at a 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 the matter insoluble in water, and

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

ANNEX H

(*Clause* 7.1)

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 material 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 air-tight after filling, and marked with full details of sampling, date of sampling, batch or code number, name of manufacturer, and other important particulars of the consignment.

H-1.1.6 The samples shall be stored in such a manner that the temperature of the 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 material of the 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 the 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 the lot to the requirements prescribed in this standard, tests shall be carried out on each lot separately. The number (n) of the packages to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

(*Clause* H-2.2)

No of Packages in the Lot (N)	No. of Packages to be Selected (n)
(1)	(2)
4 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, IS 4905 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 given 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 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 air-tight with stoppers, and labelled 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 a place agreed to between the two parties.

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 Tests for the determination of characteristics of active ingredient shall be performed on each of the individual samples .

H-4.2 Tests for determination of the 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 samples, the mean (X) and the range (R) of the test results shall be calculated as follows:

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

Range (R) = the difference between the maximum and the minimum value of the test results.

a) The value of the expression ($X - K\,R$) 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 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 Quality Levels

[*Clause* H-5.1(b)]

Acceptable Quality Level (1)	Value of '*K*' (2)
Not more than 3.0 percent defectives	0.4
Not more than 1.5 percent defectives	0.5
Not more than 0.5 percent defectives	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 of the characteristics shall satisfy the relevant requirement given in Table 1 of this standard.

ANNEX J

(*Foreword*)

COMMITTEE COMPOSITION

Soaps and other Surface Active Agents Sectional Committee, CHD 25

Organization	*Representative(s)*
Drugs Controller General of India, New Delhi	Dr P. Dasgupta (***Chairman***) Shri B. R. Wadhawan (*Alternate*)
Association for Consumer Action on Safety & Health (ACASH), Mumbai	Shri Yogesh Kamdar Shri N. G. Wagle (*Alternate*)
Central Board of Excise & Customs, Ministry of Finance, New Delhi	Chief Chemist Deputy Chief Chemist (*Alternate*)
Central Pollution Control Board, Delhi	Dr S. K. Ghosh
Consumer Guidance Society of India (Regd), Mumbai	Shri N. G. Wagle Shrimati R. Talwani (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	Dr C. J. Shishoo Shri Santosh Yellore (*Alternate*)
Department of Industrial Development, Ministry of Industry, New Delhi	Shri Shaish Kumar Shri B. B. Sharma (*Alternate*)
Development Commissioner (Small Scale Industries), New Delhi	Shri J. S. Rekhi
Directorate General of Supplies and Disposals (Inspection Wing), New Delhi	Shri P. Jayakumaran Shri M. A. Khan (*Alternate*)
Federation of Associations of Small Scale Soap & Detergent Manufacturers of India, Delhi	Shri Santosh Kumar Shri R. C. Doshi (*Alternate*)
Godrej Soaps Limited, Mumbai	Shri A. Rangarajan
Gujarat Detergent Manufacturers Association, Ahmedabad	Shri S. A. Patel Shri Mahendra Vyas (*Alternate*)
Hindustan Lever Limited, Mumbai	Dr A. N. Bhat Dr V. R. Dhanuka (*Alternate*)
Indian Soaps and Toiletries Manufacturers Association, Mumbai	Shri V. P. Menon
Karnataka Soaps & Detergents Limited, Bangalore	Dr K. B. Patil Shri M. P. Naik (*Alternate*)
Khadi & Village Industries Commission, Mumbai	Shri A. A. Warsi
K. S. Krishnan Associates (P) Limited, New Delhi	Shri. K. S. Krishnan Shri S. Krishnan (*Alternate*)
Ministry of Defence (DGQA), Kanpur	Shri M. S. Sultania Shri S. S. Shukla (*Alternate*)
Mumbai Grahak Panchayat, Mumbai	Shri Bhimrao Bagalkote Shrimati Vasudha Chachad (*Alternate*)
Nand Kishore Khanna & Sons, Mumbai	Shri P. P. Khanna Shri A. C. Khanna (*Alternate*)
National Test House, Kolkata	Shri A. K. Chakravorty Shri P. K. Chakraborty (*Alternate*)
Nirma Limited, Ahmedabad	Shri K. K. Patel Shri M. A. Bhatt (*Alternate*)
Oil Technologists Association of India, Kanpur	Dr B. R. Gaikwad Shri P. K. Tiwari (*Alternate*)
Procter & Gamble Hygiene & Healthcare (India) Limited, Mumbai	Dr Arun Vishwanath Shrimati Shweta Purandre (*Alternate*)
Reliance Industries Limited, Mumbai	Representative
Research, Design and Standards Organization (Ministry of Railways), Lucknow	Shri A. K. Choudhuri

(*Continued on page* 13)

(*Continued from page* 12)

Organization	*Representative(s)*
Tata Chemicals, Pithampur	Representative
The Non-Power Soap Manufacturers Association, Mumbai	Shri R. C. Doshi Shri Y. R. Doshi (*Alternate*)
BIS Directorate General	Shri Lajinder Singh, Director & Head (Chem) [Representing Director General (*Ex-Officio Member*)]

Member-Secretary
Shrimati Chitra Gupta
Deputy Director (Chem), BIS

Non-Soapy Detergents Subcommittee, CHD 25:2

Nirma Limited, Ahmedabad	Shri M. A. Bhat (*Convener*) Shri. K. K. Patel (*Alternate*)
Central Board of Excise & Customs, Ministry of Finance, New Delhi	Chief Chemist Deputy Chief Chemist (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	Shri S. Yellore Shrimati Falguni Mazumdar (*Alternate*)
Consumer Guidance Society of India (Regd), Mumbai	Shri N. G. Wagle Shri A. R. Shenoy (*Alternate*)
Department of Industrial Development, Ministry of Industry, New Delhi	Shri Shaish Kumar Shri B. B. Sharma (*Alternate*)
Federation of Associations of Small Scale Soap & Detergent Manufacturers of India, Delhi	Shri R. C. Doshi Shri Santosh Kumar (*Alternate*)
Fena (P) Limited, New Delhi	Shri Dalip Jolly Shri Pradeep Jolly (*Alternate*)
Godrej Soaps Limited, Mumbai	Shri A. Rangarajan
Hindustan Lever Limited, Mumbai	Dr A. N. Bhat Dr V. R. Dhanuka (*Alternate*)
Khadi & Village Industries Commission, Mumbai	Chief Executive Officer Shri A. A. Warsi (*Alternate*)
Kopsons Surfactants (P) Limited, Mumbai	Shri S. S. Koppikar Shrimati Gita S. Koppikar (*Alternate*)
Office of Development Commissioner (Small Scale Industries), New Delhi	Shri S. R. Singh Shri R. Mukhopadhyay (*Alternate*)
Office of the Textile Commissioner, Mumbai	Shri M. R. Maji Shri R. A. Lal (*Alternate*)
Oil Technologists Association of India, Kanpur	Representative
Tata Chemicals, Pithampur	Representative

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. CHD 25 (910).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	323 76 17 323 38 41
Eastern	1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700 054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 38 43 60 20 25
Southern	C.I.T. Campus, IV Cross Road, CHENNAI 600 113	254 12 16, 254 14 42 254 25 19, 254 13 15
Western	Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	832 92 95, 832 78 58 832 78 91, 832 78 92

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM.

Printed at Prabhat Offset Press, New Delhi-2