Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.


"जानने का अधिकार, जीने का अधिकार"  
Mazdoor Kisan Shakti Sangathan  
"The Right to Information, The Right to Live"

"पुराने को छोड़ नये के तरफ"  
Jawaharlal Nehru  
"Step Out From the Old to the New"

"ज्ञान से एक नये भारत का निर्माण"  
Satyanarayan Gangaram Pitroda  
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"  
Bhartrhari—Nitisatakam  
"Knowledge is such a treasure which cannot be stolen"
Indian Standard

HOUSEHOLD LAUNDRY DETERGENT POWDERS — SPECIFICATION

( Fourth Revision )

ICS 71.100.40
Soaps and Other Surface Active Agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard (Fourth 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 produced in the country at present are mainly of the alkyl aryl type, such as, sodium salts of alkyl benzene sulphonic acid for which a separate Indian Standard, IS 9985 1981 'Specification for sodium alkyl benzene sulphonate, technical' is available.

This Indian Standard was originally published in 1968 and it covered primarily the spray dried powders produced by large scale sector. It was revised for the first time in 1978 incorporating another grade of the material produced by dry mixing of the ingredients, such powders have higher bulk density as compared to those produced by spray drying process. Thereafter, the standard was revised in 1982 incorporating two additional grades of the material in accordance with prevailing manufacturing practice and for the third time in 1991. During the third revision the requirement for pH has been substituted with 'active alkalinity' as it was felt that this characteristic would more explicitly reflect the effect of alkaline materials used in the formulation of detergents and their skin irritation potential from the physico-chemical angle as compared to the requirement for pH. The limits for active detergents and sodium tripolyphosphate (SIPP) were modified.

Based on the experience gained over the years and demand for incorporating performance requirements, Committee decided to revise it again. Hence in the present revision, Grade 3 has been deleted as it is not possible to distinguish the current Grade 3 and Grade 4 for performance like cleaning and ash built up. Further performance requirements like detergency and ash built up has also been incorporated as it matches the performance defined by the current Grades 1, 2 and 3 formulations of the standard. For the modified Grade 3 limit for active ingredient has been changed.

It is necessary that the raw materials used in the formulation of detergents are such that in the concentration in which they will be present in the finished product, after interaction between them are free from any harmful effects for determining the suitability of a new formulation or of a new raw material used in old formulations on the skin. Necessary tests as prescribed in IS 11601 (Part 1) 1986 'Methods of safety evaluation of synthetic detergents Part 1 Method of test for irritant potential of synthetic detergents' and IS 11601 (Part 2) 1992 'Methods of safety evaluation of synthetic detergents Part 2 Method of test for skin sensitization potential of synthetic detergents (Guinea pig maximization test)' need to be followed.

A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of Ministry of Environment and Forest (MEI). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the BIS Act 1986 as per the Resolution No 71 dated 20 February 1991 published in the Gazette of the Government of India. For a product to be eligible for ECO Mark, it shall carry the standard mark of BIS for quality besides meeting additional optional environment friendly (EF) requirements.

This standard covers clause 6.1 which calls for an agreement between the purchaser and the supplier.

The composition of the Committee responsible for the formulation of this standard is given in Annex K.

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.
AMENDMENT NO. 2 SEPTEMBER 2008
TO
IS 4955 : 2001 HOUSEHOLD LAUNDRY DETERGENT
POWDER — SPECIFICATION

( Fourth Revision )

( Page 11, Annex G, clause G-2) — Substitute the following for the
existing

'G-2 APPARATUS

G-2.1 Photoelectric Reflection Meter — With built-in galvanometer and
tungsten lamp as an illuminant.

G-2.2 Terg-O-tometer — With a battery of four or six agitator washers in 2-
litre stainless steel beakers. The angle through which the agitators are oscillated
IS 350°. The speed of rotation is adjustable and set at 100 strokes per minute,
each back and forth movement representing one stroke. The beakers are fully
immersed in an electrically controlled water-bath. The agitators and beakers are
removable

G-2.3 Cloth Soiling Machine — Electrically operated mangle with variable
pressure arrangements which can be recorded and variable speed drive with
attached air-drymg chamber fitted with exhaust'

(CHD 25)
AMENDMENT NO. 1  SEPTEMBER 2004
TO
IS 4955 : 2001  HOUSEHOLD LAUNDRY
DETERGENT POWDERS — SPECIFICATION
( Fourth Revision )

( Page 2, clause 6.2 ) — Insert the following after 6.2 1(g)

'h)  BIS Certification Marking

The packages may also be marked with the Standard Mark

The use of the Standard Mark is governed by the provisions of the Bureau of
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.'

( CHD 25 )

Reprography Unit BIS New Delhi India
Indian Standard

HOUSEHOLD LAUNDRY DETERGENT
POWERS — SPECIFICATION
( Fourth Revision )

1 SCOPE
This standard prescribes requirements and methods of sampling and tests for laundry detergent powders for household use

2 REFERENCES
The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard At the time of publication, the editions indicated were valid All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below

<table>
<thead>
<tr>
<th>IS No</th>
<th>Title</th>
</tr>
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<tr>
<td>199</td>
<td>Textiles — Estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (third revision)</td>
</tr>
<tr>
<td>264</td>
<td>Specification for nitric acid (second revision)</td>
</tr>
<tr>
<td>265</td>
<td>Hydrochloric acid — Specification (fourth revision)</td>
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<td>324</td>
<td>Ordinary denatured spirit (revised)</td>
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<td>1070</td>
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<td></td>
<td>Relative detergency (third revision)</td>
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<tr>
<td>7597</td>
<td>Glossary of terms relating to surface active agents</td>
</tr>
<tr>
<td>8401</td>
<td>Alkyl benzene sulphonate acid (aci slurry) — Specification (first revision)</td>
</tr>
<tr>
<td>9458</td>
<td>Synthetic detergents for washing woollen and silk fabrics — Specification (first revision)</td>
</tr>
<tr>
<td>9985</td>
<td>Sodium alkyl benzene sulphonate, technical (first revision)</td>
</tr>
<tr>
<td>11601</td>
<td>Methods of safety evaluation of synthetic detergents</td>
</tr>
</tbody>
</table>

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

4 GRADES
The material shall be of the following three grades
a) Grade 1,
b) Grade 2, and
c) Grade 3

5 REQUIREMENTS
5.1 Description
The material shall be in the form of a free flowing powder, free from any unpleasant odour, and shall possess good lathering and cleaning properties In addition to moisture, it may contain substances such as colouring matter, preservatives, powder conditioners, opacifiers and optical brightening agents

5.2 Active Ingredients
The active ingredients used in the formulation of synthetic detergent powders shall comprise one or more of the surface active agents, namely, linear alkyl benzene sulphonate, secondary alcohol sulphate, fatty alcohol sulphate, fatty alcohol ethoxylate, salts of sulphated fatty alcohol ethoxylate sodium alpha sulpho fatty acid esters, alpha olefin sulphonate, soap, sugar esters and other non-ionic detergents

5.2.1 If sodium alkyl benzene sulphonate is used as the active ingredient it shall be manufactured from alkyl benzene sulphonic acid conforming to IS 8401 for which linear alkyl benzene conforming to IS 12795 shall be the starting material

5.2.2 All other active ingredients shall conform to the relevant Indian Standards, as and when available

5.3 Formulation
In addition to the active ingredients specified in 5.2 and 5.2.2 the formulation may contain one or more of conventional builders or additives as given in Annex A

5.3.1 In case non-ionic active detergent is used, the total active ingredient shall be determined by the method given in Annex B of IS 9458 If soap is
present in the detergent formulation the above result will include non-ionic detergent and soap

5.4 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)

NOTE This requirement has also been identified as specific requirement for ECO Mark

5.5 The synthetic detergent powders shall also be non injurious to the fabrics washed with it

5.6 The material shall also comply with the requirements given in Table I

5.7 Additional Requirements for ECO Mark

5.7.1 General Requirements

5 7 1 1 The product shall conform to the requirements for quality safety and performance prescribed under 5 1 to 5 6 except that for phosphate content which shall be substituted with alternate environment friendly builder(s) to maintain similar delergency when tested according to IS 5785 (Part 4)

5 7 1 2 The manufacturers shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the Water (Prevention and Control of Pollution) for 1974 and Air (Prevention and Control of Pollution) Act 1981 along with the authorization, if required under Environment (Protection) Act, 1986 while applying for ECO Mark

5.7.2 Specific Requirements

5.7.2.1 The material shall not contain any phosphate when tested as per the method prescribed in Annex D Any other substitute used shall be environmental friendly but should be in sufficient quantity to ensure similar performance of the product as compared to that of phosphates

5.7.2.2 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)

5.7.2.3 The surfactants used in the manufacture of household laundry detergent powders shall be readily biodegradable when tested by modified sturm test as prescribed in IS 13931

6 PACKING AND MARKING

6 1 Packing

The material shall be suitably packed 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 materials and declared by the manufacturer and may be accompanied with detailed instructions for proper use

6 2 Marking

6 2 1 Each package shall be securely closed and marked with the following information

a) Name and grade of the material
b) Indication of the source of manufacture,
c) Net mass of the material,
d) Batch number or lot number in code or otherwise,
e) Month and year of manufacture
f) CAUTION Detergent solutions can be skin irritants Avoid prolonged contact Rinse garments and hands thoroughly, and
g) The following critical ingredients in descending order of quantity, percent by mass

| Table 1 Requirements for Household Laundry Detergent Powders  
**(Clauses 5 6, 7 2 1 7 2 2, 7 3 1 and 8 1)** |
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Sl No</td>
<td>Characteristics</td>
<td>Requirements for</td>
<td>Methods of Test</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Grade 1</td>
<td>Grade 2</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i) Active ingredient percent by mass Min</td>
<td>10 0</td>
<td>16 0</td>
<td>10 0</td>
</tr>
<tr>
<td>ii) Total phosphates expressed as P₂O₅ content percent by mass Min</td>
<td>11 0</td>
<td>7 0</td>
<td>—</td>
</tr>
<tr>
<td>iii) Sodium tripolyphosphate (STPP) percent by mass Min</td>
<td>9 5</td>
<td>6</td>
<td>—</td>
</tr>
<tr>
<td>iv) Active alkalinity (ml of 0 1 N HCl to titrate 50 ml of 1 percent product solution to phenolphthalein end point) M₄₅</td>
<td>15</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>v) Percent detergency Min</td>
<td>65</td>
<td>55</td>
<td>45</td>
</tr>
<tr>
<td>vi) Ash build up percent Max</td>
<td>1</td>
<td>5</td>
<td>10</td>
</tr>
</tbody>
</table>
1) Active ingredients, 
2) Builders, 
3) Soda ash, 
4) Fillers, and 
5) Enzymes, if any

6.2.2 The following information shall be marked on the label for ECO Mark
a) List of identified critical ingredients in descending order of quantity, percent by mass [(see 6.2.1(g)], and
b) The criteria for which the product has been labelled as ECO Mark

7 SAMPLING
7.1 General
General precautions for drawing samples, its scale and preparation of test samples shall be as prescribed in Annex J

7.2 Number of Tests
7.2.1 Tests for the determination of characteristics given at SI No (i), (iii), (v) and (vi) of Table 1 shall be conducted on each of the individual samples separately

7.2.2 Tests for the determination of the remaining characteristics given in Table 1 shall be conducted on the composite sample

7.3 Criteria for Conformity
7.3.1 For Individual Samples
For each of the characteristics which has been determined on the individual samples (see 7.2.1) the mean (\( \bar{X} \)) and the range (R) of the test results shall be calculated as follows

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

\[\text{Range (R)} = \text{The difference between the maximum and minimum value of the test results}\]

The lot shall be deemed as conforming to the requirement if the expression (\( \bar{X} \pm 0.4 \times R \)) is greater than or equal to minimum value given at [SI No (i), (iii) and (v)] of Table 1 and the expression (\( \bar{X} \pm 0.4 \times R \)) IS less than or equal to maximum value given at [SI No (vi)] of Table 1

7.3.2 For Composite Sample
For declaring the conformity of the lot to the requirements of the other characteristics determined on the composite sample, the test results for each characteristic shall satisfy the relevant requirement

8 TESTS
8.1 Tests to evaluate the characteristic prescribed in Table 1 shall be conducted as prescribed in Annexes B to J Reference to relevant clauses of 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

ANNEX A
(Clause 5.3)
LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

1 Trisodium phosphate
2 Sodium carbonate
3 Tetra sodium pyrophosphate
4 Sodium tripolyphosphate
5 Sodium hexametaphosphate
6 Sodium carboxymethyl cellulose
7 Sodium silicate
8 Sodium bicarbonate
9 Borax
10 Optical brighteners
11 Foam boosters
12 Sodium sulphate/sodium chloride
13 Perfume
14 Preservatives
15 Chelating agents (sequestering agents)
16 Colours
17 Crystalline sodium aluminosilicate (zeolite)
18 Enzymes
19 Bleach and bleach activators
ANNEX B

[Clause 8.1, and Table 1, Sl No. (i)]

DETERMINATION OF ACTIVE INGREDIENT BY CATION TITRATION

B-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 shall therefore, be used for routine analysis. In case of any dispute or doubt, the molecular mass of sodium alkyl benzene sulphonate shall be determined by the method prescribed in Annex C and then used in calculating the active ingredient content by this method.

B-2 OUTLINE OF THE METHOD

A solution of the amonic detergent with methylene blue IS shaken with chloroform, which dissolves the methylene blue salt of the detergent. The mixture is titrated with a catronic active agent which, after it has combined with all the free amionic 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 amionic detergent similar in nature to the unknown.

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

B-3 APPARATUS

B-3.1 Volumetric Flasks — 1000 ml, 500 ml and 250 ml capacity

B-3.2 Stoppered Graduated Cylinder — 100 ml capacity

B-3.3 Graduated Cylinder — 50 ml capacity

B-3.4 Burette — 25 ml capacity

B-3.5 Pipette — 10 ml capacity

B-3.6 Beaker — 250 ml capacity

B-4 REAGENTS

B-4.1 Chloroform — chemically pure

B-4.2 Sulphuric Acid — 5 %

Carefully add 134 ml of sulphuric acid (relative density 1.84) to 300 ml of water and dilute to 1 litre.

CAUTION: Highly corrosive, causes severe burns; use necessary safety implements (see IS 4262).

B-4.3 Standard Sulphuric Acid — 10 N

B-4.4 Standard Sodium Hydroxide Solution — 10 N

B-4.5 Standard Sodium Lauryl Sulphate Solution — 0.004 M

Check up purity of sodium lauryl sulphate as given in B-4.5.1 and simultaneously prepare the standard solution.

B-4.5.1 Determination of Purity of Sodium Lauryl Sulphate

Weigh, to the nearest 1 mg, 5 ± 0.2 g of sodium lauryl sulphate into a 250-ml round bottom flask with ground glass neck. Add exactly 25 ml of standard sulphuric acid (see B-4.3) and reflux under water condenser. During the first 5 to 10 mm, 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 the After a further 10 min the solution clarifies and foaming ceases. Reflux for a further one and half hours. 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 indicator solution and titrate with standard sodium hydroxide solution. Carry out a blank test by titrating 25 ml of standard sulphuric acid (see B-4.3) with standard sodium hydroxide solution.

Sodium lauryl sulphate content, percent by mass = \[ \frac{V_1 V_a}{m_1} N_1 \]

where

- \( V_0 \) — volume, in ml, of standard sodium hydroxide solution used for the blank,
- \( V_1 \) — volume, in ml, of standard sodium hydroxide solution used for the sample,
- \( N_1 \) — normality of standard sodium hydroxide solution, and
- \( m_1 \) — mass, in g, of sodium lauryl sulphate taken for the test.

B-4.5.2 Procedure

Weigh, to the nearest 1 mg, 1.14 to 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, \( M_1 \), of the solution by means of the formula.
**m_2** × sodium lauryl sulphate.

\[ M_1 = \frac{\text{percent by mass}}{288.4 \times 100} \]

where

\( m_2 = \) mass in g of sodium lauryl sulphate taken

**B-4.6 Standard Benzethonium Chloride Solution — 0.004 M**

Weigh to the nearest 1 mg, 1.75 to 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 1.7792 g to the nearest 1 mg, dissolve in water and dilute to 1 litre. While drying, care shall be taken not to raise the temperature beyond 105°C.
2. Other cationic reagents such as cetyl trimethyl ammonium bromide 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 shall be used.

**B-4.7 Phenolphthalein Indicator Solution**

Dissolve 1 g of phenolphthalein in 100 ml of 95 percent v/v ethanol.

**B-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.

**B-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 0.65 to 0.70 g of sodium alkyl benzene sulphonate (based on 100 percent purity) or 4.5 g of the sample having around 16 percent anionic active matter content per 500 ml of the solution is suitable.

**B-5 PROCEDURE**

**B-5.1 Standardization of Benzethonium Chloride Solution**

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

**B-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.

**B-5.1.3** Calculate the molarity of the benzethonium chloride solution as follows:

\[ M_2 = \frac{10M_1}{V_1} \]

where

\( M_1 = \) molarity of sodium lauryl sulphate solution (**B-4.5.2**), and

\( V_1 = \) volume in ml of benzethonium chloride solution added.

**B-5.3** Calculate the anionic active matter as sodium alkyl benzene sulphonate as follows:

\[ \text{Anionic active matter, percent by mass} = \frac{342 \times V_2 \times M_2 \times 5}{m_2} \]

where

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

\( V_2 = \) volume in ml of benzethonium chloride solution added,

\( M_2 = \) molarity of benzethonium chloride solution (**B-5.1.3**), and

\( m_2 = \) mass in g of the sample taken.
ANNEX C

(Clauses 8.1 and B-1)

DETERMINATION OF MOLECULAR MASS OF SODIUM SALT OF ALKYL BENZENE SULPHONIC ACID

C-1 APPARATUS
C-1.1 Beakers — 150 and 1 000 ml capacity
C-1 2 Buchner Flask — 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4)
C-1 3 Separating Funnels — 1 000 ml capacity
C-1 4 Wide-Mouthed Flat-Bottom Flask — 200 ml capacity
C-1.5 Air-Oven — Preferably electrically heated with temperature control device

C-2 REAGENTS
C-2.1 Caustic Soda Solution — 10 percent (m/v)
C-2.2 Ethyl Alcohol — 30 percent, 96 percent and absolute (v/v)
C-2.3 Diethyl Ether
C-2.4 Acetone
C-2.5 Phenolphthalein Indicator — 1 percent solution in 95 percent (v/v) ethyl alcohol
C-2.6 Methyl Orange Indicator — 0.1 percent (m/v)
C-2.7 Ferric Ammonium Sulphate Indicator — saturated solution
C-2.8 Standard Sulphuric Acid — Approximately 0.1 N
C-2.9 Standard Silver Nitrate Solution — Approximately 0.1 N
C-2.10 Standard Ammonium Thiocyanate Solution — Approximately 0.1 N
C-2.11 Nitric Acid — Concentrated, relative density 1.42
C-2.12 Nitrobenzene

C-3 PROCEDURE
C-3.1 Weigh about 3 g of the material into a 150-ml beaker. Dissolve in minimum quantity of water and neutralize with caustic soda solution, if required. 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 mm. Stir and break up any hard lumps 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 five times with hot ethyl alcohol to remove all the alcohol soluble. Evaporate the combined filtrate to a small bulk of approximate 50 ml 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.

C-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 hot 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 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.

C-3.3 Transfer the filtrate and washing in the Buchner flask to a wide-mouthed flat-bottom 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 on 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 prescribed above, cool in a desiccator and weigh. Heat the flask for not more than 5 min in an air oven at a 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 weighing does not exceed 3 mg.

C-3.4 The extract obtained contains active matter, some sodium chloride and possible traces of alkali carbonates which may have passed through the filter in the presence of the detergent. Find the percentage of sodium carbonate and sodium chloride in the extract by using a portion of the extract as prescribed in C-3.4.1 and C-3.4.2 respectively.

C-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.

C-3.4.1.1 Calculation

Mass in g of sodium carbonate = \( \frac{0.053 \times V_1 \times N_1 \times m_1}{m_2} \)

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,
- \( m_2 \) = mass in g of the extract taken for analysis.

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

C-3.4.2 Determination of Chlorides
To the solution remaining after the estimation of alkali carbonate (see C-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.

C-3.4.2.1 Calculation

Mass in g of sodium chloride = \( 0.0585 \times (20N_1 \times V_1) = \frac{m_2}{m_1} \)

where

- \( N_1 \) = normality of standard silver nitrate solution,
- \( V_1 \) = volume in ml of standard ammonium thiocyanate solution used
- \( N_2 \) = normality of standard ammonium thiocyanate solution,
- \( m_1 \) = mass in g of the total extract (see C-3.3), and
- \( m_2 \) = mass in g of the extract taken for analysis (in C-3.4.1.1).

C-3.5 Weigh accurately about 0.65-0.70 g of the extract. Dissolve in water and make up to 500 ml. Follow the titration procedure given in B-5.1 taking 10 ml of the solution for titration.

C-4 Calculation

Molecular mass of sodium salt of sulphonate acid = \( \frac{100 \times m}{5 \times V_1 \times M_1} \)

where

- \( m \) = mass in g of the extract taken after correcting for sodium carbonate and sodium chloride,
- \( V_1 \) = volume in ml of benzethonium chloride solution added, and
- \( M_1 \) = molarity of benzethonium chloride solution.
ANNEX D

[Clause 8.1, and Table 1, Sl No. (ii)]

DETERMINATION OF TOTAL PHOSPHATES

D-1 GENERAL

The sample is oxidized by gently heating with sodium nitrate. Silica is removed and the condensed phosphates are hydrolysed and precipitated as ammonium molybdate by addition of ammonium molybdate. The precipitate is washed with dilute potassium nitrate solution and phosphorus in the washed precipitate is determined by titration with standard sodium hydroxide using phenolphthalein as indicator.

D-2 APPARATUS

D-2.1 Silica Dish — 7 cm diameter
D-2.2 Beaker - 250 ml capacity
D-2.3 Buchner Flask — 500 ml capacity with a sintered glass filter funnel
D-2.4 Volumetric Flask — 500 ml capacity
D-2.5 Funnel — 7.5 cm diameter
D-2.6 Wide-Mouthed Flat Bottom Flask — 500 ml capacity

D-3 REAGENTS

D-3.1 Sodium Nitrate
D-3.2 Hydrochloric Acid — concentrated (see IS 265)
D-3.2.1 Dilute Hydrochloric Acid
D-3.3 Nitric Acid — see IS 264
D-3.4 Ammonium Molybdate Reagent

Dissolve 90 g of ammonium molybdate in hot water. Add 240 g of ammonium nitrate and stir to dissolve. Cool and add 30 ml of concentrated ammonia (0.9 relative density). Dilute to 1 litre

D-3.5 Potassium Nitrate — 1.25 percent solution in water

D-3.6 Standard Sodium Hydroxide Solution — 1 N
D-3.7 Standard Sulphuric Acid — 1 N
D-3.8 Phenolphthalein Solution — 1 percent solution (m/v) in ethyl alcohol
D-3.9 Potassium Hydroxide Solution — 0.1 N

D-4 PROCEDURE

D-4.1 Accurately weigh about 1.5 g of the sample in a silica dish and add about twice sample weight of a mixture of equal parts of sodium carbonate and sodium nitrate, mix well and heat gently over a Bunsen burner until the sample is completely oxidized. Cool and add 15 ml of concentrated hydrochloric acid and evaporate to dryness. Add further 15 ml of concentrated hydrochloric acid and repeat the evaporation procedure. Finally extract the residue in 25 ml of 1 N hydrochloric acid and then wash 4 times with 50 ml of water. Collect the filtrate and washings and make up to 250 ml in a volumetric flask.

D-4.2 Pipette 50 ml aliquot from the volumetric flask in a 250 ml beaker. Add 10 ml of nitric acid and boil 15 mm. Cool and add 100 ml of water and adjust the temperature of the solution to 40 to 45°C. Add 50 ml of ammonium molybdate solution (previously heated to 40°C) slowly with constant stirring. Allow to stand for 30 mm. Filter the precipitate through a quantitative filter paper and wash with 1.25 percent potassium nitrate solution till 5 ml of the filtrate with 1 drop of phenolphthalein does not require more than 3 to 4 drops of 0.1 normal caustic potash solution to produce pink colour. Transfer the filter paper with the precipitate to a 500 ml wide mouthed flat-bottom flask and add 100 ml water. Heat over a water-bath for 15 mm. cool and titrate with 1 N sodium hydroxide solution using 1 ml of phenolphthalein till the pink colour just appears. Add 2 ml excess. Shake well, heat to 60°C in a water-bath. Cool and back titrate against 1 N sulphuric acid till the pink colour just disappears. Find the volume of normal sodium hydroxide solution required to react with the precipitate.

D-5 CALCULATION

D-5.1 Total phosphates as P2O5, percent by mass

\[ \frac{l \times A \times 0.001349 \times 250 \times 100}{m \times 50} \times 140 \]

\[ \frac{62}{V} \]

Where

- \( V \) = volume in ml of sodium hydroxide solution required to react with the precipitate,
- \( N \) = normality of sodium hydroxide solution, and
- \( m \) = mass in g of sample taken for test.
ANNEX E

[Clause 8 1, and Table 1, Sl No (iii)]

DETERMINATION OF SODIUM TRIPOLYPHOSPHATE (STPP) CONTENT

E-1 GENERAL
Methods usually recommended for determination or sodium tripolyphosphate (STPP) involve time consuming and elaborate procedure based on ion-exchange and paper chromatography. The gravimetric method prescribed here is simple, quick and requires no special facilities

E-2 OUTLINE OF THE METHOD
It is based on the quantitative precipitation of STPP by tris (ethylenediamine) cobalt (III) chloride as Co(en)$_2$H$_2$PO$_4$·H$_2$O at 3.5 pH after removing the active detergent, by dissolution in alcohol, and water insoluble silica/silicates

E-3 REAGENTS
E-3.1 p-Nitrophenol Indicator Solution
Dissolve 0.1 g of p-nitrophenol in 100 ml of water

E-3.2 Dilute Hydrochloric Acid — 0.5 N approximately

E-3.3 Acetate Buffer Solution — pH 3.5
Dissolve 52.6 ml of glacial acetic acid and 6.16 g sodium acetate anhydrous in water and dilute to 500 ml

E-3.4 Sodium Tripolyphosphate Hexahydrate
(Na$_5$P$_3$O$_{10}$·7H$_2$O)
Crystallize from commercial STPP as STPP Hexahydrate as given in F-3.4.1 and E-3.4.2

E-3.4.1 Procedure
Weigh 150 g of commercial sodium tripolyphosphate, using material of low metaphosphate content. Dissolve in 200 ml of water, and filter to remove any suspended material. Add 300 ml of denatured spirit slowly, with mechanical stirring, over a period of 2 h. Separate the crystals of hexahydrate by filtration using a Buchner funnel fitted with a glass disc of medium porosity. Wash the crystals successively with 100 ml each of 25 percent, 50 percent, 75 percent and 90 percent (v/v) solutions of denatured spirit in water

E-3.4.2 Dissolve the hexahydrate in 900 ml of water and salt out with 225 ml of denatured spirit using the same procedure as above again, filtering and washing the crystals with the same volumes of dilute denatured spirit. Repeat the recrystallization using 675 ml of water and 170 ml of denatured spirit. Recrystallize once more using 500 ml of water and 150 ml of denatured spirit. After filtering and washing air dry the crystals of hexahydrate for several days.

NOTE — The yield is 75% of (Na$_5$P$_3$O$_{10}$·7H$_2$O). Store the hexahydrate in a tightly stoppered bottle. The purification procedure should not be interrupted for any length of time unless the material is in a crystalline form to avoid hydrolysis. Do not allow it to remain in solution overnight

E-3.5 This (Ethylenediamine) Cobalt (III) Chloride
Prepare as given in E-3.5.2.1 to E-3.5.2.4 Use 15 to 20 percent solution of the reagent

E-3.5.1 Reagents
a) Diaminoethane hydrate (ethylene diamine)
b) Concentrated hydrochloric acid — See IS 265
c) Cobalt chloride (COCl$_2$·6H$_2$O)
d) Denatured spirit — See IS 324

E-3.5.2 Procedure
E-3.5.2.1 Weigh 261 g of diaminoethane hydrate into a 1 000-ml beaker. Partially neutralize with 85 ml of concentrated hydrochloric acid in 535 ml of water. Pour the mixture with good agitation into a solution of 250 g of cobalt chloride in 750 ml of water contained in a conical flask. Pass a vigorous stream of air into the solution through a gas distribution tube for 8 h.

E-3.5.2.2 Transfer the solution to a 2 000-ml beaker and evaporate on a steam-bath under a stream of air until crystals form on the surface of the solution. Cool the solution and add 150 ml of concentrated hydrochloric acid with good mechanical stirring. Warm until the crystals re-dissolve. Salt out the tris diaminoethane cobaltic chloride by slowly adding over a period of 1 h. 300 ml of denatured spirit to the mechanically stirred solution. Cool and filter through a Buchner funnel and suction. Discontinue suction and wash by stirring with 150 ml of denatured spirit. Re-apply suction and filter. Repeat the washings three more times. Suck dry, spread out the crystals in an evaporating dish and allow the denatured spirit to evaporate.

E-3.5.2.3 Dissolve the crystals obtained in F-3.5.2.2 above in 200 ml of boiling water in 1000-ml beaker on a hot-plate using mechanical stirring. If the crystals do not dissolve in this volume of water, add 20 ml portion of water, reheating to boiling after each addition, until complete solution is obtained. Remove from the source of heat and salt out with 300 ml of the denatured spirit as above. Filter and wash as
before. If four washings do not give a colourless filtrate, continue washing until the filtrate is colourless

E-3 5.2.4 Spread out the crystals in a thin layer in large evaporating dishes and allow to air-dry overnight. Then dry in an oven at 100°C overnight.

NOTE — These crystals are hygroscopic and should be freshly dried each time they are weighed to make a new solution. An yield of about 300 g of the dried crystals is obtainable.

E-4 PROCEDURE

E-4.1 Determination of STPP in Recrystallized Sodium Tripolyphosphate Hexahydrate (Na₃P₃O₁₀·6H₂O)

To the solution containing 0.2 to 0.4 g of STPP (accurately weighed) in 75 ml and 2 to 3 drops of p-nitrophenol indicator, add dilute hydrochloric acid dropwise with constant stirring till the indicator changes to colourless. Add 10 ml of the buffer solution to get the desired pH of about 3.5. Add 20 ml of isopropanol. Warm the solution at 40°C in a water bath for 15 min. Add 10 ml of cobalt reagent in portions — first 4 ml, stir vigorously for 1 min, another 4 ml, stir again for 1 min and finally add the last 2 ml and stir for 2 min. Allow the precipitate to stand at 40°C for 15 min. Filter through a weighed sintered glass crucible. Wash with water till the washings are free of chloride ions. Dry the crucible in an oven at 110°C for 45 min. Cool in a desiccator and weigh till constant mass is obtained.

E-4.1.1 Calculation

Purity of recrystallized STPP, percent by mass -

$$\text{P} = \frac{M_1 \times 0.6945}{M_2} \times 100$$

where

- $M_2$ = mass in g of the cobalt tripolyphosphate precipitate obtained, and
- $M_1$ = mass in g of recrystallized sample taken in the test.

E-4.2 Determination of Triphosphatic Content in Detergent Powder

E-4.2.1 Separation of Active Detergent

Treat an accurately weighed quantity, about 3 g of detergent powder with 10 ml of water in a 500 ml beaker. Add 75 ml of denatured spirit and stir for 10 min. Filter through a fluted filter paper (Whatman No. 41 or equivalent) by decantation. Treat the residue left in the beaker with three more portions of 75 ml denatured spirit, followed by stirring and filtration by decantation after each addition. Discard the filtrate.

E-4.2.2 Preparation of STPP Sample Solution

Wash down the residue on the filter paper into the beaker containing most of the inorganic material using a jet from a wash bottle. Also wash down the material sticking to the sides of the beaker with water, using in all about 75 ml of water. Stir and keep aside for 15 mm with occasional stirring. Filter through filter paper (Whatman No. 4 or equivalent) transferring all solids into the filter with the help of minimum water. Wash the residue with 25 ml of water and collect the washings and the filtrate in a 500-ml beaker for STPP determination. Add a known amount of the recrystallized STPP (0.150 to 0.250 g), (see Note) and stir to dissolve. Add 3 to 4 drops of p-nitrophenol indicator. A green colour will appear. Add dilute hydrochloric acid dropwise with stirring till the indicator colour disappears. Add 10 ml of acetic acid buffer to get 3.5 pH. Add 15 ml of isopropanol and place the beaker in a warm water-bath (40°C) for 15 min.

NOTE: In place of adding STPP 0.5 to 1 mg of this (ethylenediamine) cobalt triphosphate crystals obtained from previous analysis if any may be added before adding the cobalt reagent and finally subtracted from the mass of the precipitate for the purpose of calculation.

E-4.2.3 Precipitation of STPP by the Cobalt Reagent

Add 10 ml of cobalt reagent solution in portions in three instalments, first add 4 ml and stir vigorously for 2 mm, then add 4 ml and again stir vigorously for 2 mm and finally add 2 ml and stir for 1 mm. Place the beaker containing the precipitate in a water-bath (40°C) for 10 min. Add 0.150 to 0.250 g of filter aid and stir. Filter through a weighed sintered glass crucible. Wash the precipitate with water till washings are free of chloride ions. Dry the precipitate at 110°C for 1 h. Weigh till constant mass ($M_3$) is obtained.

E-5 CALCULATION

E-5.1 STPP in detergent powder, percent by mass

$$\text{P} = \frac{\left( M_4 - M_3 \right) \times 0.6945}{M_1} - \frac{M_2}{100}$$

where

- $M_4$ = mass in g of the filter-aid and precipitate obtained,
- $M_3$ = mass in g of filter-aid added,
- $M_2$ = mass in g of STPP added (calculated as anhydrous STPP), and
- $M_1$ = mass in g of sample of powder taken in E-4.2.1.
ANNEX F
[Clause 8 1, and Table 1, Sl No (IV)]

DETERMINATION OF ACTIVE (RESERVE) ALKALINITY

F-1 APPARATUS
F-1.1 pH Meter
F-1.2 Beaker — 100-ml capacity
F-1.3 Magnetic Stirrer
F-1.4 Burette

F-2 REAGENTS
F-2.1 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

F-2.2 Hydrochloric Acid - 0 1 N
F-3 PROCEDURE
Weigh accurately 0 5 g of detergent in powder form
in a tared 100-ml beaker and add distilled water to
make up the weight to 50 g (1 percent solution m/m)
Place the beaker containing 1 percent solution of
detergent product in aqueous vehicle on a magnetic
stirrer and mix contents thoroughly Note down the
pH of the solution using a pH meter With the
electrode of the pH meter dipping in the solution and
keeping the pH meter 'ON' add drop by drop 0 1 N
hydrochloric acid from a burette till the pH of the
solution drops to 8 While adding hydrochloric acid
stir the solution continuously Note the amount of
0 1 N hydrochloric acid required to bring down the
pH of the solution to 8 which is a measure of the
active alkalinity of the test sample

Mean of 2 replicate measurements will give active
(reserve) alkalinity expressed as amount in ml of
0 1 N hydrochloric acid

ANNEX G
[Clause 8 1, and Table 1, Sl No (V)]

PROCEDURE FOR DETERGENCY TEST (POWDERS)

G-1 OUTLINE OF THE METHOD
The method prescribed here is based on the use of
Terg-O-tometer Cloth is artificially soiled and the
soil IS removed by washing the soiled swatches of
cloth with a solution of the detergent powder to be
evaluated under standard conditions A control
detergent powder (see below for details) is also run
simultaneously The reflectance of the unsoiled,
soiled and washed swatches is measured
instrumentally using a standard photoelectric
reflection meter The detergency is expressed as
percentage of soil removal

G-2 APPARATUS
G-2.1 Photoelectric Reflection Meter
G-2.2 Terg-O-tometer
G-2.3 Cloth Soiling Machine — Electrically
operated mangle with variable pressure arrangements
which can be recorded and variable speed drive with
attached air-drying chamber fitted with exhaust

G-3 PREPARATION OF STANDARD SOILED
CLOTH TEST SPECIMENS
Prepare soiled cloth swatches as per 6 of IS 5785
(Part 4)

G-4 CONTROL POWDER FORMULATION

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
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</thead>
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<tr>
<td>AD</td>
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</tr>
<tr>
<td>STPP</td>
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<tr>
<td>Soda</td>
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<tr>
<td>Alk silicate</td>
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<tr>
<td>Na₂SO₄</td>
<td>36</td>
</tr>
<tr>
<td>Moisture</td>
<td>5</td>
</tr>
</tbody>
</table>

100
G-5 WASHING PROCEDURE

G-5.1 The ratio of cloth (in g) to the volume of solution (in ml) shall be 1:100.

G-5.2 Prepare 1 litre solution of 0.5 percent of the product basis in 300 ppm water at which concentration the washing shall be carried out. Introduce 5 soiled swatches into beakers and agitate. Wash the specimens for exactly 10 minutes at 27 ± 2°C.

G-5.3 Remove the beaker from the water-bath and decant the solution and rinse 3 times with 300 ppm water.

G-5.4 Repeat with 5 more wash loads of 5 swatches for each formulation and control powder. A total of six replicate washings is given so that at the end all the formulations are washed in all beakers.

G-5.5 Air-dry and measure the reflectance of washed swatches.

G-6 EVALUATION OF SOIL REMOVAL

G-6.1 Fold the desired unsoiled cloth, soiled swatches and washed swatches into four folds so as to minimize the effect of color of background and take reflectance measurements. Operate the reflectometer in accordance with the instruction supplied with the instrument.

G-7 CALCULATION AND REPORTING

The detergency value expressed as percentage soil removed is calculated from the following equation:

\[
\text{Percentage of soil removal} = \frac{W_0}{S_0} \times 100
\]

where

\[
W_0 = \frac{(100 - R_c)^2}{200R_c} \quad \frac{(100 - R_s)^2}{200R_s}
\]

and \( R_c, R_s \) and \( R_p \) are reflectances of clean, washed and soil fabric pieces respectively.

Analysis of variance to be used for the handling of data and obtaining significant differences.

G-8 TREATMENT OF DATA AND REPORTING

Assign a value of 70 percent detergency to control powder. Normalize the detergency values of products by applying a correction factor (that is, 70 percent detergency value for control powders) and report the normalized detergency value.

ANNEX H

[Clause 8.1, and Table 1, Sl No (VI)]

DETERMINATION OF ASH BUILT UP ON FABRICS

H-1 GENERAL

This test determines the built-up of ash on a fabric under the condition of test.

H-2 REAGENTS

H-2.1 Sodium Hexametaphosphate

H-2.2 Hard water of 300 ppm (as CaCO₃), made by adding 2.96 g of MgSO₄ 7H₂O and 2.64 g of CaCl₂ 2H₂O to 10 litres of water.

H-2.3 Bleached Cotton Long Cloth (125 ± 25 g/m²)

H-3 EQUIPMENT

H-3.1 Washing Appliance Like Standard Terg-O-meter

H-3.2 Air oven with arrangements for suitably hanging the washed swatches for drying.

H-4 PROCEDURE

H-4.1 Cut the cotton cloth into swatches of 10 cm x 10 cm size. 10 swatches are required for each set.

H-4.2 Number the raw swatches and wash them 2 times in a Terg-O-meter with sodium hexametaphosphate solution (3 g/l) to remove the original salts present in the swatches. Rinse the swatches thoroughly with distilled water. Dry the swatches in an air oven at 105°C.

H-4.3 Stir 10 swatches in 1 litre solution of 0.2 percent concentration of the test detergent product prepared in 300 ppm hard water (as CaCO₃), at 27 ± 2°C for 20 minutes in a Terg-O-meter. Rinse the swatches in 1 litre water of 300 ppm hardness water by stirring in the Terg-O-meter for 10 minutes at 27 ± 2°C.

H-4.4 Dry the swatches at 50°C in an air oven. Repeat the washing, rinsing and drying operations 25 times. After 25 washes, dry the swatches in an air oven at 105°C. Determine the ash content of the fabric at 800°C as per IS 199.
ANNEX J

(Clause 7.1 and 8.1)

SAMPLING PROCEDURE FOR SYNTHETIC DETERGENTS

J-1 GENERAL REQUIREMENTS

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

J-1.1.1 Samples shall be taken in a protected place not exposed to atmospheric air

J-1.1.2 The sampling instruments shall be clean and dry

J-1.1.3 The samples, the material being sampled, the sampling instruments and the containers for such samples shall be protected from adventitious contamination

J-1.1.4 The sample 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

J-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

J-1.1.6 The samples shall be stored in such a manner that the temperature of the material docs not vary unduly from ambient temperature, and that they are protected from light

J-2 SCALE OF SAMPLING

J-2.1 Lot

In a single consignment, all the packages containing material of the same grade and drawn from the same batch of manufacturer, shall constitute a lot. If the consignment consists of packages containing material of different grades or batch of manufacture, then the packages containing detergent of the same grade and batch of the manufacture shall be grouped together, and each such group shall constitute a separate lot

J-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 packages to be selected for drawing samples shall depend upon the size (N) of the lot and shall be in accordance with Table 2

J-2.3 The packages shall be selected at random and, to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the procedure given in J-2.3.1 may be adopted

J-2.3.1 Starting from any package, count all the packages 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)

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<tr>
<th>No. of Packages in the Lot</th>
<th>No. of Packages to be Selected</th>
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</thead>
<tbody>
<tr>
<td>(N)</td>
<td>(n)</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
</tr>
<tr>
<td>4 to 15</td>
<td>3</td>
</tr>
<tr>
<td>16 to 40</td>
<td>4</td>
</tr>
<tr>
<td>41 to 65</td>
<td>5</td>
</tr>
<tr>
<td>66 to 110</td>
<td>7</td>
</tr>
<tr>
<td>111 and above</td>
<td>10</td>
</tr>
</tbody>
</table>

NOTE — When the size of the lot is 3 packages or less, the number of packages to be selected and the ??? for judging the conformity of the lot the specification shall be as agreed between the purchaser and the supplier

Every rth package thus counted shall be drawn to give a sample for the test

J-3 PREPARATION OF GROSS SAMPLES, TEST SAMPLES AND REFEREE SAMPLES

J-3.1 Gross Samples

J-3.1.1 From each one of the packages selected as in J-2 draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for the test as indicated in 7.2

J-3.1.1.1 The material from the containers selected as in J-3.1.1 shall be mixed thoroughly to give the gross sample for the package

J-3.2 Test Samples

J-3.2.1 Segregate carefully the gross samples (see J-3.1.1.1) of powders. From the 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 7.2. The composite sample shall be divided into three equal parts — one for the purchaser, another for the supplier, and the third for the referee

J-3.2.2 The remaining portion of the material in each one of 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

J-3.2.3 All the composite and individual samples shall be transferred to separate containers. These containers shall be sealed air-tight with stoppers, and
labelled with full particulars of identification given in J-1.1.5

J-3.3 Referee Samples

J-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

J-3.3.2 Referee samples shall be used in case of any dispute between the purchaser and the supplier

ANNEX K

(Foreword)

COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

<table>
<thead>
<tr>
<th>Organization</th>
<th>Representative(s)</th>
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<tbody>
<tr>
<td>Drugs Controller General of India New Delhi</td>
<td>DR P. Dasgupta (Chairman)</td>
</tr>
<tr>
<td>Association for Consumer Action on Safety &amp; Health (ACASH) Mumbai</td>
<td>Shri B.K. Waghwan (Alternate)</td>
</tr>
<tr>
<td>Central Board of Excise &amp; Customs Ministry of Finance New Delhi</td>
<td>Shri Yogesh Kambard</td>
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<tr>
<td>Central Pollution Control Board Delhi</td>
<td>Dr. N. G. Wagle (Alternate)</td>
</tr>
<tr>
<td>Consumer Guidance Society of India (Regd) Mumbai</td>
<td>Chief Chemist</td>
</tr>
<tr>
<td>Consumer Education and Research Centre Ahmedabad</td>
<td>Dr. S.K. Ghosh</td>
</tr>
<tr>
<td>Department of Industrial Development, Ministry of Industry New Delhi</td>
<td>Shri N. G. Wagle</td>
</tr>
<tr>
<td>Development Commissioner (Small Scale Industries) New Delhi</td>
<td>Shri P. Talwani (Alternate)</td>
</tr>
<tr>
<td>Directorate General of Supplies and Disposals (Inspection Wing) New Delhi</td>
<td>Dr. C.J. Shishoo</td>
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<tr>
<td>Federation of Associations of Small Scale Soap &amp; Detergent Manufacturers of</td>
<td>Shri Santosh Yelow (Alternate)</td>
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<tr>
<td>India Delhi</td>
<td>Shri S. B. Sharma (Alternate)</td>
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<td>Godrej Soaps Ltd Mumbai</td>
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<td>Shri M. A. Khan (Alternate)</td>
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<td>Shri S. Santosh Kumar</td>
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<td>Khadi &amp; Village Industries Commissioner Mumbai</td>
<td>Shri R. C. Doshi (Alternate)</td>
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<td>Shri A. Rangarajan</td>
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<td>Karnataka Soaps &amp; Detergents Limited Bangaluru</td>
<td>Shri S. A. Patel</td>
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<tr>
<td>Ministry of Defence (DGQA) Kanpur</td>
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### Organization

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<tr>
<td>Oil Technologists Association of India Kanpur</td>
<td>DR B R GAIKWAD (Alternate)</td>
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<td>Procter &amp; Gamble India Hygiene &amp; Healthcare Limited Mumbai</td>
<td>DR ARUN VISHWANATH (Alternate)</td>
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<td>Reliance Industries Ltd Mumbai</td>
<td>SHIRMATI SHWETA PURANDRI (Alternate)</td>
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<td>Research Design and Standards Organization (Ministry of Railways) Lucknow</td>
<td>SHRI A K CHOUDHURI (Alternate)</td>
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<td>The Non-Power Soap Manufactures Association Mumbai</td>
<td>SHRI R C DOSHI (Alternate)</td>
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<tr>
<td>Tata Chemicals Pithampur Directorate General BIS</td>
<td>REpresentative</td>
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<tr>
<td>Consumer Education and Research Centre Ahmedabad</td>
<td>SHRI RAJINDER SINGH Director and H??? (Chem)</td>
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<td>Federation of Associations of small scale soap &amp; Detergent Manufacturers India</td>
<td>[Representing Director General (Ex officio Member)]</td>
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<td>Godrej Soaps Limited Mumbai</td>
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### Non Soapy Detergents Subcommittee, CHD 25 2

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<td>Central Board of Excise &amp; Customs Ministry of Finance New Delhi</td>
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<td>DUPY CHIEF CHEMIST; Alternate</td>
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<td>SHRI S YELLOR</td>
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<td>SHRI SHAISH KUMAR (Alternate)</td>
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Amendments Issued Since Publication

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<th>Amend No</th>
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Headquarters
Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
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<th>Region</th>
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<td>Eastern</td>
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<td>C I T Campus, IV Cross Road, CHENNAI 600113</td>
<td>254 12 16, 254 14 42, 254 25 19, 254 13 15</td>
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Printed at Sunco Printing Press Delhi
[Page 3, clause 6.2.1(c)] — Substitute the following for the existing:

‘c) Net mass of the material, when packed;’

(Page 3, clause 6.2.2) — Insert the following note at the end of clause:

‘NOTE — Loss in mass, even in packed condition, may occur on account of moisture loss due to environmental conditions.’

(CHD 25)