IS 4955 (2001): Household Laundry Detergent Powders [CHD 25: Chemical]

भारतीय मानक

घरेलू धुलाई के लिये अपमार्जक पाउडर — विशिष्टि

(चौथा पुनरीक्षण)

Indian Standard

HOUSEHOLD LAUNDRY DETERGENT POWDERS — SPECIFICATION

(Fourth Revision)

ICS 71.100.40

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

December 2001 **Price Group 7**

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 sulphoriate, 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 tor the third time in 1991 During the third revision the requirement for *p*H 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 *p*H The limits for active detergents and sodium tnpolyphosphate (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 detergeney and ash built up has also been incorporated as it matches the performancc 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 I Method of test for irritant potential of synthetic detergents' and IS 11601 (Part 2) 1992 'Methods of safety evaluation of synthetic detergents Part 2 Methods of test for skin sensitization potential of synthetic detergents (Guinea pig maximazation 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 invironment 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 Gazatte 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 POWDERS — SPECIFICATION

(Fourth Revision)

(*Page* 11, *Annex* G, *clause* G-2) — Substitute the following tor 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)

Reprography Unit BIS New Delhi India

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 Indian Standards Art* 1986 and the Rules and Regultions made thereunder The details of conditons 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 POWDERS — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes requirements and methods of samphng 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 publiccation, 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

IS No	*Title*
199 1989	Textiles — Estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (*third revision*)
264 1976	Specification for nitric acid (*second revision*)
265 1993	Hydrochloric acid — Specificatio (*fourth revision*)
324 1959	Ordinary denatured spirit (*revised*)
1070 1992	Reagent grade water — Specification (*third revision*)
5785 (Part 4) 1976	Methods of performance tests for surface active agents Part 4 Relative detergency (*third revision*)
7597 1974	Glossary of terms relating to surface active agents
8401 1994	Alkyl benzene sulphonic acid (aci slurry) — Specification (*first revision*)
9458 1994	Synthetic detergents for washing woollen and silk fabrics — Specification (*first revision*)
9985 1992	Sodium alkyl benzene sulphonate, technical (*first revision*)
11601 (Part 1) 1986	Methods of safety evaluation of synthetic detergents Skin irritant potential
(Part 2) 1992	Skin sensitization potential (Guinea pig maximization test)
12795 1989	Specification for linear alkyl benzene
13933 1995	Method of test for biodegradabilit of surface active agents (modified Sturm test)

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 irrildnl 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 require mcnts tor quality salety 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 *All (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)

Sl No	Characteristice	Requirements for			Methods of Test (Ref to Annex)
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Active ingredient percent by mass *Min*	19 0	16 0	10 0	B
ii)	Total phosphates expressed as P_2O??? content percent by mass *Min*	11 0	7 0	—	*D*
iii)	Sodium tripolyphosphate (STPP) percent by mass *Min*	9 5	6	—	E
iv)	Active alkalnity (ml of 0 1 N HCI to litrate 50 ml of 1 percent product solution to phenolphthalcin end point) *Max*	15	20	30	F
V)	Percent detergency *Min*	65	55	45	G
VI)	Ash built up percent *Max*	1	5	10	H

1) Active ingredients,
2) Builders,
3) Soda ash,
4) Fillers, and
5) Lnzymes, 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 tor 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

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

Range (R) – 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 ($\bar{X}$ 0 4 R) is greater than or equal to minimum value given at [SI No (i), (iii) and (v)] of Table 1 and the expression ($\bar{X}$ +0 4 R) IS less than or equal to maximum value given at [SI No (vi)] of Table I

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 lor each characteristic shall satisfy the relevant requirement

8 TESTS

8 1 Tests to evaluate the characteristic prescribed in Tabic 1 shall be conducted as prescribed in Annexes B to J Reference to reltvant 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 alffect 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)]

DETERMINAl ION 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 amonic 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 sulphires interiere With the detection of the and point and should be destroyed by the addition of terious sulphite and hydrogen peroxide respectively

B-3 APPARALUS

B-3.1 Volumetric Flasks — 1 000 **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 REAGENES

B-4.1 Chloroform — chemically pure

B-4.2 Sulphuric Acid — 5 N

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 satfety implements (*see* IS 4262)

B-4.3 Standard Sulphuric Acid — 1 0 N

B-4.4 Standard Sodium Hydroxide Solution — 1 0 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 *Delermination 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 $= \frac{28{\cdot}84\,(V_1 \quad V_o)N_1}{m_1}$

where

V_o volume, in ml, of standard sodium hydroxide solution used tor the blank,

V_1 – volume, in ml, of standard sodium hydroxide solution used tor 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 m 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 lauyrl 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 Benzethoninm 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 wcigh 1???92 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 benzethomum 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 65-0 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-S.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 I owards 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 benzethonium chloride solution as follows

Molarity of benzethonium chloride solution $M_2 = \frac{10 M_1}{V_1}$

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-S 2 Determination of Anionic Active Matter

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

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

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 tor 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 S 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 Sliver 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 m 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 pha.ses 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 rhe 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*

$$\text{Mass in g of the sodium carbonate} = 0\,053 \times V_1 \times N_1 \times \frac{m_1}{m_2}$$

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, and

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*

$$\text{Mass in g of sodium chloride} = 0\,058\,5\,(20N_1 \quad V_1N_2) \times \frac{m_1}{m_2}$$

where

N_3 = normality of standard silver nitrate solution,

V_2 = 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

$$\text{Molecular mass of sodium salt of sulphonic acid} = \frac{100 \times m}{5 \times V_1 \times M_1}$$

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 hydrolyscd and precipitated as ammonium phosphomolybdate by addition of ammonium molybdate 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.S 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 Hidrochloric 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 1 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 tor 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 I drop of phenolphthalein does not require more than 3 to 4 drops of 0 1 normal caustic potash solution to produce pink colour Transter the filter paper with the precipitate to a 500 ml wide mouthed flat-bottom fiask 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 P_2O_5, percent by mass

$$\frac{V \times N \times 0.001349 \times 250 \times 100}{m \times 50} \times \frac{140}{62}$$

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 delermination or sodium tripolyphosphate (STPP) mvolve 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 $(en)_3$-$H_2P_3O_{10}$ $2H_2O$ at 3 5 *p*H 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 — *p*H 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_6P_3O??? 6H 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 tripoly-phosphate, using material of low metaphosphatc content Dissolve in 1 200 ml of water, and filler 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 tunnel 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 filtenng 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 ???out 60 g of (Na???P???O , $6H_2O$) Store thc hexahydrate in a tightly stoppered bottle The purification procedure should not be interrupted for any length of time unless the material is in a erystalline 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 ($COCl_2$ $6H_2O$)
d) Denatured spirit — *See* IS 324

E-3 S.2 *Procedure*

E-3.5.2 1 Weigh 261 g of diaminoethane hydrate into a 1 000-ml beaker Partly netrtralize 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 waler 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 ($Na_5P_3O_{10}.6H_2O$)

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 *p*H of about 3 5 Add 20 ml of isopropanol Warm the solution at 40°C in a water bath for 15 mm Add 10 ml of cobalt reagent in portions — first 4 ml, stir vigorously for 1 min, another 4 ml, stir again for 1 mm and finally add the last 2 ml and stir for 2 mm 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 mm Cool in a desiccator and weigh till constant mass is obtained

E-4.1.1 *Calculation*

Purity of recrystallized STPP, percent by mass = $\frac{M_2 \times 0\,694\,5}{M_1} \times 100$

where

M_2 – mass in g of the cobalt tripolyphosphate precipitate obtained, and

M_1 mass in g of recryslallired sample taken in the test

E-4.2 Determination of Tripolyphosphate 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 25 g), *(see* Note) and stirr 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 *p*H Add 15 ml of isopropanol and place the beaker in a warm water-bath (40°C) for 15 mm

NOTE In place of adding STPP 0 5 to 1 mg of this (ethylenediamine) cobalt tripotyphosphale 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 water-bath (40°C) for 10 mm 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_4) is obtained

E-5 CALCULATION

E-5.1 STPP in detergent powder,

percent by mass = $\frac{[(M_4 - M_3) \times 0\,694\,5] - M_2 \times 100}{M_1}$

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 *p*H 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 *p*H of the solution using a *p*H meter With the electrode of the *p*H meter dipping in the solution and keeping the *p*H meter 'ON add drop by drop 0 1 N hydrochloric acid from a burette till the *p*H 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 *p*H 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

AD		19
STPP		19
Soda		10
Alk silicate -		10
SCMC		1
Na_2SO_4		36
Moisture	=	5
		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 on as it is 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 mm at 27 ± 2°C

G-5.3 Remove the beaker from the water-bath and decant the solution and rinse 3 limes m 300 ppm water

G-5.4 Repeat with 5 more wash loads of 5 swatches each tor 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 Alt dry and measure the reflectance of washed swatches.

G-6 EVALUATION OF SOIL REMOVAL

G-6.1 Fold the desized unsoiled cloth, soiled swatches and washed swatches into four folds so as to minimize the effect of colour of background and take reflectance measurements Operate the rcflectomctcr 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

$$\text{Percentage of soil removal} = \frac{W_O}{S_O} \times 100$$

where

$$W_O = \frac{(100 - R_w)^2}{200R_w} - \frac{(100 - R_s)^2}{200R_s}$$

$$S_O = \frac{(100 - R_c)^2}{200R_c} - \frac{(100 - R_s)^2}{200R_s}$$

and R_c, R_w, and R??? are reflectances of clean, washed and soiled 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 Normalise the detergency values of products by applying a correction factor (that is, 70 percent detergency value for control powders) and report the normalised 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 $CaCO_3$), made by adding 2 96 g of $MgSO_4$ $7H_2O$ and 2 64 g of $CaCl_2$ $2H_2O$ to 10 litres of water

H-2 3 Bleached Cotton Long Cloth (125 ± 25 g/m^2)

H-3 EQUIPMENT

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

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 c m ' 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-tometer with sodium hexameta-phosphate 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 $CaCO_3$), at 27 ± 2°C for 20 minutes m a Terg-O-tometer Rinse the swatches in 1 litre water of 300 ppm hardness water by stirring in the Terg-O-tometer for 10 min 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

(*Clauses* 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 matenal 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)

Table 2 Scale of Sampling

(*Clause* J-2 2)

No or Packages in the Lot (*N*)	No of Packages to he 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 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 *r*th 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-3.1.1.1 From each one of the packages selected as in **J-2** draw at random one or more containers The material m 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 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

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(*Continued on page* **15**)

(*Continued form page* 14)

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BUREAU OF INDIAN STANDARDS

Headquarters

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones 323 01 31, 323 3375, 323 94 02

Telegrams Manaksanstha
(Common to all offices)

Regional Offices		Telephone
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Eastern	1/14C I T Scheme VII M, V I P Road Kankurgachi CALCUTTA 700054	337 84 99, 337 85 61 337 86 26 337 91 20
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TO
IS 4955 : 2001 HOUSEHOLD LAUNDRY DETERGENT POWDERS

(Fourth Revision)

[*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)

Reprography Unit, BIS, New Delhi, India