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IS 9458 (1994): Synthetic detergents for washing woolen and silk fabrics [CHD 25: Soaps and other Surface Active Agents]



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भारतीय मानक

ऊनी और रेशमी कपड़ों की धुलाई के लिये संश्लिष्ट  
अपमार्जक — विशिष्ट

( पहला पुनरीक्षण )

*Indian Standard*

SYNTHETIC DETERGENTS FOR WASHING  
WOOLLEN AND SILK FABRICS —  
SPECIFICATION

( *First Revision* )

UDC 661.187.762 : 677.31.074

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

## FOREWORD

This Indian Standard ( First 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.

Woollen, silken and other delicate fabrics, unlike cotton fabrics, are sensitive to alkalis. Conventionally, therefore, neutral soap without builders and practically free from caustic alkali has been used for washing woollens, silken and synthetic fabrics in households and laundries. With the introduction of synthetic detergent based products, the position has changed.

This Indian Standard was first published in 1980. In this revision, type I namely, powder has been deleted as it was not being manufactured or marketed in the country presently. With a view to ensure safety from skin irritation and skin sensitization hazards to consumers using this product and to ensure that this is also not injurious to the fabrics washed, a requirement for safety of the product on use has been introduced. The concerned technical committee is also actively considering to introduce a requirement for detergency for which suitable test methods are being developed.

A scheme for labelling environment friendly products to be known as ECO Mark is being introduced at the instance of the Ministry of Environment and Forests (MEF). 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 also carry the standard mark of BIS for quality besides meeting additional optional environment friendly (EF) requirements. The EF requirements for synthetic detergents for washing woollen and other delicate fabrics were added to this standard through Amendment No. 2 of May 1992 which has been incorporated in this revision.

The composition of the Technical Committee and Subcommittee responsible for formulation of this Indian Standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# SYNTHETIC DETERGENTS FOR WASHING WOOLLEN AND SILK FABRICS — SPECIFICATION

( *First Revision* )

## 1 SCOPE

This standard prescribes requirements and methods of sampling and test for synthetic detergents for washing woollen and silk fabrics.

## 2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
4955 : 1993	Household laundry detergent powders ( <i>third revision</i> )
4956 : 1977	Synthetic detergent for industrial purposes ( <i>first revision</i> )
5785 ( Part 4 ) : 1976	Methods for performance tests for surface active agents : Part 4 Relative detergency ( <i>first revision</i> )
7597 : 1974	Glossary of terms relating to surface active agents
8401 : 1977	Alkyl benzene sulphononic acid ( acid slurry )
11601 ( Part 1 ) : 1986	Method of safety evaluation of synthetic detergents : Part 1 Method of test for irritant potential of synthetic detergents
11601 ( Part 2 ) : 1992	Method of safety evaluation of synthetic detergents : Part 2 Method of test for skin sensitization potential of synthetic detergents ( Guinea pig maximization test )
12795 : 1989	Linear alkyl benzene

## 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 : 1974 shall apply.

## 4 REQUIREMENTS

### 4.1 Composition

The active ingredient used in the formulation of the material shall be one or more of the surface active chemicals namely, alkyl benzene sulphonate, secondary alcohol sulphate, fatty alcohol sulphate, fatty alcohol ethoxylate, salts of sulphated fatty alcohol ethoxylate, sodium alpha olefin sulphonate, and alkyl-phenol ethoxylate. The formulation may contain one or more of the builders and additives given in Annex A, or any other builders or additives keeping in view the end use of the product.

### 4.2 Description

The material shall be clear liquid and free from unpleasant odour. The material shall possess good cleaning and lathering properties.

### 4.3 Alkyl Benzene Sulphonic Acid

Alkyl benzene sulphononic acid from which alkyl benzene sulphonate is prepared shall conform to IS 8401 : 1977 for which linear alkyl benzene conforming to IS 12795 : 1989 shall be the starting material.

### 4.4 Safety Evaluation

The material shall pass the test for skin irritant potential when evaluated as per the method prescribed in IS 11601 ( Part 1 ) : 1986 and for skin sensitization potential when evaluated as per the method prescribed in IS 11601 ( Part 2 ) : 1992.

NOTE — This requirement has also been identified as one of the criteria for ECO Mark.

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

### 4.6 Optional Requirements for ECO Mark

#### 4.6.1 General Requirements

4.6.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under 4.1 to 4.5 except that for

phosphate content which shall be substituted with alternate environment friendly builder(s) to maintain similar detergency when tested according to IS 5785 ( Part 4 ) : 1976 ( *see also* 4.6.2.1 ).

**4.6.1.2** 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 ) *Act*, 1974 and Air ( Prevention and Control of Pollution ) *Act*, 1981 along with the authorization, if required under the Environment ( Protection ) *Act* 1986, while applying for ECO Mark.

#### **4.6.2 Specific Requirements**

**4.6.2.1** The material shall not contain any phosphate when tested as per the method prescribed in Annex D of IS 4955 : 1993. Any other substitute used shall be environment friendly but should be in sufficient quantity to ensure similar performance of the product as compared to that with phosphates.

**4.6.2.2** The active ingredient used in the manufacture of synthetic detergents for washing woollen and other delicate fabrics shall have a biodegradability of minimum 97 percent when tested as per the method prescribed in Annex B of IS 12795 : 1989.

### **5 PACKING AND MARKING**

#### **5.1 Packing**

**5.1.1** The material shall be packed in suitable, well-closed containers, as agreed to between the purchaser and the supplier.

**5.1.2** 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.

#### **5.2 Marking**

**5.2.1** The containers shall be securely closed and marked with the following information:

- a) Name of the material;
- b) Net mass in the container;
- c) Indication of source of manufacture;

- d) Batch number or lot number, in code or otherwise;
- e) Month and year of manufacture; and
- f) **CAUTION**: Detergent solutions can be skin irritants. Avoid prolonged contact. Rinse garments and hands thoroughly.

**5.2.2** The following additional information shall be marked on the labels for ECO Mark:

- a) The following identified critical ingredients in descending order of quantity, percent by mass:
  - 1) Active ingredients;
  - 2) Builders used;
  - 3) Soda ash, if added;
  - 4) Fillers; and
  - 5) Enzymes, if added.
- b) The criteria for which the product has been labelled as ECO Mark.

#### **5.2.3 BIS Certification Marking**

The product shall also be marked with the Standard Mark.

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

### **6 SAMPLING**

Representative samples of the material shall be drawn as prescribed in Annex H of IS 4956 : 1977.

### **7 TESTS**

**7.1** Tests shall be conducted as prescribed in IS 4955 : 1993, IS 4956 : 1977 and Annex B of this standard, reference to which has been given in col 4, 5 and 6 of Table 1.

#### **7.2 Quality of Reagents**

Unless specified otherwise, pure chemicals and distilled water ( *see* IS 1070 : 1992 ) shall be used in tests.

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

**Table 1 Requirements for Synthetic Detergents for Washing Woollen and Silk Fabrics**  
( Clause 4.5 )

SI No.	Characteristic	Requirement	Method of	Test,		Ref to
			Annex of this Standard	IS 4955 : 1993	Annex in IS 4956 : 1977	Annex in IS 4956 : 1977
(1)	(2)	(3)	(4)	(5)	(6)	(6)
i)	Active ingredient, percent by mass, <i>Min</i>	15.0	B	—	—	—
ii)	Total phosphates content, expressed as STPP, calculated from phosphorus pentoxide ( $P_2O_5$ ), percent by mass, <i>Min</i>	3.0	—	D	—	—
iii)	pH of 1 percent solution ( <i>m/v</i> ) at 27°C	7.0 to 8.5	—	—	—	F
iv)	Matter insoluble in water, percent by mass, <i>Max</i>	Nil	—	—	—	G
v)	Clear point, °C, <i>Max</i>	10	—	—	—	E

NOTE — In case non-ionics and soaps are absent, the active ingredients shall be determined by the method given in Annex B of IS 4955 : 1993.

## ANNEX A

( Clause 4.1 )

### LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

- |                                  |  |
|----------------------------------|--|
| 1. Sodium sulphate               | 9. Hydrotropes   |
| 2. Tetra sodium pyrophosphate    | 10. Perfume  |
| 3. Sodium tripolyphosphate       | 11. Preservatives  |
| 4. Carboxy methyl cellulose      | 12. Chelating agents                                       |
| 5. Polyvinyl pyrrolidone         | 13. Organic solvents such as isopropanol and ethyl alcohol |
| 6. Tetra potassium pyrophosphate | 14. Colour   |
| 7. Sodium silicate               |  |
| 8. Optical brightener            |  |



## ANNEX B

[ Clause 7.1, and Table 1, Sl No. (i) ]

## DETERMINATION OF ACTIVE INGREDIENTS

## B-0 GENERAL

**B-0.1** Active matter is first separated from inorganic salts, non-detergent organic matter and alkylamide. It is then neutralized to phenolphthalein, evaporated to dryness, extracted with ethyl alcohol, dried and weighed. Finally, the weighed extract is corrected for the presence of sodium chloride and alkali carbonates.

## B-1 APPARATUS

**B-1.1** Beakers — 150 ml and 1 000 ml capacity.

**B-1.2** Buchner Flask — 500 ml capacity, fitted with a sintered glass filter funnel ( porosity 4 ).

**B-1.3** Evaporating Basin

**B-1.4** Separating Funnels — 1 000 ml capacity.

**B-1.5** Steam-Bath

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

**B-1.7** Air-Oven — Preferably electrically heated, with temperature control device.

## B-2 REAGENTS

**B-2.1** Ethyl Alcohol — 30 percent, 90 percent, 96 percent and absolute ( by volume ).

**B-2.2** Diethyl Ether

**B-2.3** Acetone

**B-2.4** Standard Sulphuric Acid — approximately 0.1 N.

**B-2.5** Standard Silver Nitrate Solution — approximately 0.1 N.

**B-2.6** Phenolphthalein Indicator — 1 percent solution in 95 percent ( by volume ) ethyl alcohol.

**B-2.7** Methyl Orange Indicator — 0.1 percent ( *m/v* ).

**B-2.8** Nitric Acid — concentrated, relative density 1.42 ( conforming to IS 264 : 1976 ).

**B-2.9** Nitrobenzene

**B-2.10** Standard Ammonium Thiocyanate Solution — approximately 0.1 N.

**B-2.11** Ferric Ammonium Sulphate Indicator — saturated solution.

## B-3 PROCEDURE

## B-3.1 Removal of Inorganic Salts

Weigh accurately about 6 g of the material for products containing about 15 percent active matter and corresponding less or more for products of higher or lower active matter content. Digest with 60 ml of ethyl alcohol by heating on a steam-bath for about 2 minutes. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and, finally wash the residue several times with hot ethyl alcohol to remove all the alcohol solubles. After filtering and washing the residue thoroughly with hot ethyl alcohol, evaporate the combined filtrate to a small bulk in an evaporating basin.

## B-3.2 Removal of Non-detergent Organic Matter and Alkylolamide

**B-3.2.1** Dilute the evaporated filtrate ( *see B-3.1* ) with 50 ml of 90 percent ethyl alcohol, and transfer to a separating funnel. Rinse the evaporating basin once with 50 ml of 90 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.

**B-3.2.2** 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.

**B-3.2.3** Combine the washings and rinsing from **B-3.2.2** with the aqueous alcoholic phase obtained in **B-3.2.1**.

### B-3.3 Determination of Active Matter

**B-3.3.1** Transfer the combined aqueous alcoholic phase and washings from **B-3.2.3** to a porcelain basin. 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.

**B-3.3.2** To ensure that the residue is completely anhydrous, add 30 ml of absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with six further consecutive 30 ml portions of hot 96 percent ethyl alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 ml of hot 96 percent ethyl alcohol from the jet of a wash bottle.

**B-3.3.3** Transfer the filtrate and washings in the Buchner flask to a tared wide-mouthed flat-bottomed flask, evaporate nearly to dryness on a water-bath, and drive off the remaining solvent by directing a gentle stream of dry-air into the flask whilst continuously rotating the latter 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 described above, cool in a desiccator and weigh.

**B-3.3.4** Heat the flask for not more than 5 minutes in an air-oven at a temperature of  $100 \pm 1^\circ\text{C}$ , gently blow out with a current of air, cool and reweigh. Repeat this drying process until the difference between two successive weighings does not exceed 3 mg. Record this mass as  $M_1$ .

**B-3.3.5** The extract obtained in **B-3.3.4** contains the active matter, some sodium chloride and possibly a trace of alkali carbonates which may have passed through the filter in the presence of the detergent.

### B-3.4 Determination of Alkali Carbonates

**B-3.4.1** Dissolve the extract (see **B-3.3.4**) in cold water, add a few drops of methyl orange

indicator and titrate with standard sulphuric acid to the methyl orange end point.

### B-3.4.2 Calculation

Mass, in g, of sodium carbonate,  $M_2 = 0.053 VN$

where

$V$  = volume, in ml, of standard sulphuric acid solution used; and

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

**B-3.4.3** Reserve the solution for the estimation of chlorides.

### B-3.5 Determination of Chlorides

**B-3.5.1** To the solution remaining after the estimation of alkali carbonates (see **B-3.3.4**), 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.

### B-3.5.2 Calculation

Mass in g of sodium chloride,  $M_3 = 0.0585 (20A - BC)$

where

$A$  = normality of the standard silver nitrate solution;

$B$  = volume, in ml, of standard ammonium thiocyanate solution used; and

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

### B-3.6 Determination of Active Matter

Active matter, percent by mass =  $\frac{100 (M_1 - M_2 - M_3)}{M}$

where

$M_1$  = mass, in g, of the alcoholic extract (see **B-3.3.4**);

$M_2$  = mass, in g, of sodium carbonate (see **B-3.4.2**);

$M_3$  = mass, in g, of sodium chloride (see **B-3.5.2**); and

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

## ANNEX C

### ( Foreword )

#### COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 025

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### Revision of Indian Standards

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

This Indian Standard has been developed from Doc : No. CHD 025 ( 0246 ).

### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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LUCKNOW. PATNA. THIRUVANANTHAPURAM.

**AMENDMENT NO. 1 AUGUST 1999  
TO  
IS 9458 : 1994 SYNTHETIC DETERGENTS FOR  
WASHING WOOLLEN AND SILK FABRICS —  
SPECIFICATION**

*( First Revision )*

*( Page 1, clause 2 )* — Insert the following at the end:

<i>IS No.</i>	<i>Title</i>
13933 : 1995	Method of test for ready biodegradability of surface active agents ( modified sturm test )

*( Page 2, clause 4.6.2.2 )* — Substitute the following for the existing text:

‘The surfactants used in the manufacture of synthetic detergent for washing woollen and silk fabrics shall be readily biodegradable when tested by modified sturm test as prescribed in IS 13933 : 1995’.

( CHD 25 )

**AMENDMENT NO. 2 MARCH 2002**  
**TO**  
**IS 9458 : 1994 SYNTHETIC DETERGENTS FOR**  
**WASHING WOOLLEN AND SILK FABRICS —**  
**SPECIFICATION**

*( First Revision )*

[ Page 2, clause 5.2.1(f) ] — Insert the following after 5.2.1(f):

‘g) Critical ingredients mentioning the actual compound in descending order up to a limit of 0.5 percent by mass:

- 1) Active ingredients;
- 2) Builders used;
- 3) Fillers;
- 4) Soda ash;
- 5) Enzymes, if added; and
- 6) Bleach and bleach activators, if added.’

**AMENDMENT NO. 3 SEPTEMBER 2008**  
**TO**  
**IS 9458 : 1994 SYNTHETIC DETERGENTS FOR WASHING**  
**WOOLLEN AND SILK FABRICS — SPECIFICATION**

*( First Revision )*

*(Page 1, clause 4.6)* — Substitute 'Additional Requirements for ECO Mark' for 'Optional Requirements for ECO Mark'.

(CHD 25)