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SPECIFICATION FOR SYNTHETIC ORGANIC LIQUID DETERGENTS FOR HOUSEHOLD USE

SRI LANKA STANDARDS INSTITUTION

SPECIFICATION FOR SYNTHETIC ORGANIC LIQUID DETERGENTS FOR HOUSEHOLD USE

SLS 687:1985

(Attached AMD No.1)

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FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1985-04-02, after the draft, finalized by the Drafting Committee on Synthetic Detergents, had been approved by the Chemicals Divisional Committee.

This specification refers to non soapy organic detergents and in particular to anionic detergents. These may contain non-ionic active ingredients. Inorganic compounds such as phosphates and silicates generally termed as builders may also be included in the product.

The standard values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the result of a test or an analysis shall be rounded off in accordance with CS 102. The number of figures to be retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification, the assistance obtained from the publications of the Indian Standards Institution and the Federal Supply Service of the General Services Administration of USA is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements, and methods of sampling and test, for synthetic organic liquid detergents for household use.

2 REFERENCES

CS 102 Presentation of numerical values

SLS 428 Random sampling methods

SIS 535 Methods of test for paints

SLS 540 Enamel paints for interior use

3 REQUIREMENTS

3.1 General

The detergent shall be a homogeneous liquid free from sediments, suspended particles and objectionable odour. It shall contain synthetic organic active matter and foam stabilizing agents.

3.2 Active ingredient content

The active ingredient content shall be not less than 12 per cent (m/m) when tested as prescribed in Appendix B.

3.3 pH

The pH at 27 $^{\circ}$ C of a one per cent (V/V) distilled water solution of the detergent shall be between 7.0 and 9.0 when tested as prescribed in Appendix C.

3.4 Total non-detergent organic matter

The total non-detergent organic matter content shall be not more than 0.5 per cent (m/m) when tested as prescribed in Appendix D.

3.5 Solubility

The detergent shall be completely soluble when tested as prescribed in 6.1.

3.6 Rinsing properties

The detergent when tested as prescribed in 6.2 shall satisfy the requirements of the test.

3.7 Effect on painted surfaces

A 0.2 per cent solution of the detergent shall cause not more than half the loss of specular gloss caused by a 0.2 per cent solution of trisodium phosphate when tested in accordance with Appendix E.

4 PACKAGING AND MARKING

4

4.1 The detergent shall be well packed in suitable containers, and the containers shall in turn be packed in cartons.

4.1.1 Each container shall be marked legibly and indelibly with the following information:

a) Name of the product as Household detergent;

b) Name and address of manufacturer;

c) Active ingredient content;

d) Volume of contents, in millilitres; and

e) Instructions for use.

4.1.2 Each carton shall be marked legibly and indelibly with the following information;

a) Date of manufacture; and

b) Batch or code number.

4.2 The containers may also be marked with the Certification Mark of the Sri Lanka Standards Institution illustrated below on permission being granted for such marking by the Sri Lanka Standards Institution.



NOTE - The use of the Sri Lanka Standards Institution Certification Mark (SLS Mark) is governed by the provisions of the Sri Lanka Standards Institution Act and regulations framed thereunder. The SLS mark on products covered by a Sri Lanka Standard is an assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control, which is devised and supervised by the Institution and operated by the producer SLS marked products are also continuously checked by the Institution for conformity to that standard as a further safeguard. Details of conditions under which a permit for the use of the Certification Mark may be granted to manufacturers or processors may be obtained from the Sri Lanka Standards Institution.

5 SAMPLING

Representative samples of liquid detergent shall be drawn as specified in Appendix A.

6 METHODS OF TEST

Tests shall be carried out as in 6.1, 6.2 and Appendices B, C, D and E.

6.1 Test for solubility

6.1.1 Preparation of synthetic hard water (1.8 millimoles per litre calcium hardness)

Weigh to the nearest 0.001 gram, about 0.264 g of $CaCl_2.2H_2O$ and 0.295 g of $MgSO_4$. $7H_2O$. Transfer quantitatively to a 1-litre volumetric flask, dissolve in a small portion of distilled water and make up to the mark with distilled water.

6.1.2 Procedure

Using a pipette, transfer 5.0 ml of the detergent into a test tube and add sufficient synthetic hard water prepared as in 6.1.1 to give a volume of 50 ml. Stir vigorously for 5 min, and observe for solubility.

6.2 Test for rinsing properties

Dissolve 2.0 ml of the liquid detergent as completely as possible in 98 ml of synthetic hard water (see 6.1.1) at ambient temperature, in a clean 250-ml erlenmeyer flask. Stopper the flask and stir vigorously for 1 min. Pour out the solution. Rinse the flask by the same procedure, using three 75-ml portions of synthetic hard water alone. Invert the flask, allow to dry, and examine for any residue not rinsed from the interior. The flask shall contain no more residue after being dried than a similar flask allowed to dry after rinsing with synthetic hard water alone.

7 CONFORMITY TO STANDARD

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied.

7.1 Each container examined as in A.5.1 satisfies the requirements.

7.2 The test sample tested as in A.5.2 satisfies the relevant requirements.

APPENDIX A

SAMPLING

A.1 LOT

In any consignment, all the containers of same size containing liquid detergent of one batch of manufacture or supply, shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, preparing and handling samples the following precautions and directions shall be observed.

A.2.1 Samples shall not be taken in an exposed place.

A.2.2 The sampling instruments shall be clean and dry when used.

A.2.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible, by suitable means.

A.2.4 Samples shall be taken from freshly opened containers.

A.2.5 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the container for samples from adventitious contamination.

A.2.6 The samples shall be placed in suitable, clean, dry, and air tight glass containers.

A.2.7 The sample container shall be sealed after filling and marked with necessary details of sampling.

A.3 SCALE OF SAMPLING

A.3.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirement of this specification.

A.3.2 The number of containers to be selected from a lot shall be in accordance with Table 1.

	of c the l	ontainers ot	Number of containers to be selected
Up	to	100	02
101	to	300	03
301	to	500	05
501	to	1 000	07
1 001	to	3 000	10
3 001	and	above	13

TABLE 1

A.3.3 10 per cent of cartons, subject to a minimum of 2 shall be selected. As far as possible an equal number of containers shall be drawn from the cartons so selected to get the required number of containers as given in Table 1.

A.3.4 The cartons and containers shall be selected at random. In order to ensure randomness of selection, random number tables as given in SLS 428 shall be used.

A.4 PREPARATION OF TEST SAMPLE

The contents of each container selected as in A.3.2 shall be mixed thoroughly and sufficient quantity of material shall be poured from each container to a sample container to form a test sample of not less than 100 ml.

A.5 NUMBER OF TESTS

A.5.1 Each container selected as in A.3.2 shall be examined for packaging, and marking requirements.

A.5.2 The test sample prepared as in A.4 shall be tested for the requirements given in 3.2 to 3.7.

APPENDIX B

DETERMINATION OF ACTIVE INGREDIENT CONTENT

B.1 PRINCIPLE

Active ingredient is first separated from inorganic salts, non-detergent organic matter and alkanolamides. It is then neutralized with phenolphthalein as indicator evaporated to dryness, extracted with ethyl alcohol, dried and weighed. Finally the weighed extract is corrected for the presence of sodium chloride and alkaline carbonates.

B.2 REAGENTS

B.2.1 Ethyl alcohol, 30 per cent, 90 per cent, 96 per cent, and absolute (V/V).

B.2.2 Diethyl ether

B.2.3 Acetone

B.2.4 Sulfuric acid, standard volumetric solution $c(H_2SO_A) = 0.055 \text{ mol}/1$.

B.2.5 Silver nitrate, standard volumetric solution c(AgNO₃) = 0.05 mol/1.

B.2.6 Phenolphthalein indicator, 1 per cent solution in 96 per cent ethyl alcohol, (V/V).

B.2.7 Methyl orange indicator, 0.1 per cent, (m/V).

B.2.8 Nitric acid, concentrated, rel. density 1.42.

B.2.9 Nitrobenzene

B.2.10 Ammonium thiocyanate, standard volumetric solution $c(NH_ACNS) = 0.1 \text{ mol}/1.$

B.2.11 Ferric ammonium sulfate indicator

B.2.12 Caustic soda solution, 10 per cent (m/V)

B.3 PROCEDURE

B.3.1 Removal of inorganic salts

Weigh to the nearest 0.001 g, approximately 5 g of the material for products containing about 15 per cent active matter and correspondingly less for products of higher active matter contents. Place the material in a beaker and digest with 50 ml of 96 per cent ethyl alcohol by heating on a steam bath for about 2 minutes. Stir with a glass rod and allow the solid matter to settle. Decant the hot alcoholic solution through a sintered glass funnel into a buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with five further consecutive 30 ml portions of boiling 96 per cent ethyl alcohol. Filter each extract in turn, through the sintered glass funnel and wash the residue several times with hot ethyl alcohol to remove all the alcohol solubles. Combine the filtrate and evaporate to a small bulk in an evaporating basin.

B.3.2 Removal of non-detergent organic matter and alkanolamides

B.3.2.1 Dilute the evaporated filtrate (see B.3.1) with 50 ml of 90 per cent ethyl alcohol and transfer to a separating funnel. Rinse the evaporating basin once with a 50-ml portion of 90 per cent ethyl alcohol and then four times with 50-ml portions of distilled 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 alcohol into a second separating funnel and extract twice with 75-ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker. Combine the three diethyl ether extracts.

B.3.2.2 Take the combined ether extracts in a clean, separating funnel and wash three times with successive 50-ml portions of 30 per cent ethyl alcohol and then with successive 50-ml portions of distilled water until the other phase is free from alcohol; usually, 7 to 10 water washings are necessary.

B.3.2.3 Combine the washings and rinsings 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 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.

6.3.3.2 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 per cent 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 per cent 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 per cent 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 five minutes in an air-oven at a temperature of 100 \pm 1 °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 the 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 **B.3.3.4** in cold distilled water, add a few drops of methyl orange indicator and titrate with standard sulfuric acid to the methyl orange end point.

B.3.4.2 Calculation

Mass, in g, of sodium carbonate, $m_2 = 0.106 V_1 c_1$

where,

 V_1 = volume, in ml, of sulfuric acid solution (B.2.4) used, and c_1 = concentration, of the sulfuric acid solution (B.2.4), in mol/1. 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 (**B.3.4.3**) 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 sulfate as indicator.

B.3.5.2 Calculation

Mass, in g, of sodium chloride, $m_3 = 0.0585 (c_1 - Vc_2)$

where,

01	=	concentration of the silver nitrate solution (B.2.6) in mol/1;
0,	=	concentration of the ammonium thiocyanate solution (B.2.11),
2		in mol/1; and
V	=	volume in ml of the ammonium thiocyanate solution (B.2.11).

B.3.6 Calculation of active ingredient content

Active ingredient, per cent by mass = $\frac{100 (m_1 - m_2 - m_3)}{m_0}$

where,

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^m o		mass, i	in g,	of material taken for the test;
^m 1	-	mass, i	in g,	of the alcoholic extract (B.3.3.4);
^m 2		mass, i	in g,	of sodium carbonate (B.3.4.2); and
^m 3	-	mass, i	in g,	of sodium chloride (B.3.5.2).

APPENDIX C

DETERMINATION OF pH

C.1 GENERAL

pH determination should be made in an acid-free atmosphere.

C.2 APPARATUS

C.2.1 pH meter

Any standard pH meter, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard j buffer solutions (see C.3.2) before use.

(C.2.2 Volumetric flask, 1 000-ml capacity.

C.2.3 Beakers, 100-ml.

C.3 REAGENTS

C.3.1 Distilled water

Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 27 °C. The residue on evaporation when heated at 105 °C for one hour shall not exceed 0.5 ml per litre.

C.3.2 Standard buffer solution

Any two suitable buffer solutions within the pH range of 9 to 11 at 27 °C for calibrating the pH meter.

C.4 PROCEDURE

Weigh to the nearest milligram approximately 10 g of the material and transfer to a 1-litre volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to 27 \pm 2 °C, and fill to the calibration mark with distilled water, stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 27 \pm 2 °C for two hours prior to measuring the pH. Measure the pH of the solution at 27 \pm 2 °C using a glass electrode.

APPENDIX D

DETERMINATION OF NON-DETERGENT ORGANIC MATTER

D.1 PRINCIPLE

The term *non-detergent organic matter* includes hydrocarbons, fatty alcohols and perfumes. Using petroleum ether and under the conditions prescribed, only non-detergent organic matter is extracted leaving any alkanolamide present in the material.

D.2 REAGENTS

D.2.1 Ethyl alcohol, 70 per cent, and 90 per cent, (V/V)

D.2.2 Petroleum ether, boiling range 40 $^{\circ}$ C to 60 $^{\circ}$ C : non-volatile residue at 80 $^{\circ}$ C maximum 0.001 per cent.

D.2.3 Acetone, non-volatile residue at 80 °C, maximum 0.001 per cent.

D.3 PROCEDURE

D.3.1 Removal of inorganic salts

Weigh to the nearest milligram approximately 5 g of the material in a 150-ml beaker. Extract with 50-ml of hot 90 per cent ethanol by heating on a steam-bath for about 2 minutes stirring and breaking up any hard lumps with a glass rod.

D.3.1.1 Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a 500-ml buchner flask to which suction is applied. Repeat the extraction in a similar manner with five further consecutive 30-ml quantities of boiling 90 per cent ethanol. Pass each extract in turn through the filter into the flask.

D.3.2 Transfer quantitatively all the combined filtrate from the buchner flask to a 1-litre separating funnel and rinse the flask four times with 40-ml quantities of distilled water, transferring each wash in turn to the separating funnel. Add 100 ml of petroleum 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 with 75 ml of petroleum ether. Repeat the extraction of the aqueous alcoholic phase in a third separating funnel with a further 65-ml of petroleum ether. Combine the three other extracts in the first separating funnel. Rinse each of the two empty funnels with a few millilitres of petroleum ether and add the rinsings to the combined ether extracts.

D.3.3 Wash the combined ether extracts and rinsings (see **D.3.2**) with four successive 50-ml portions of 70 per cent ethyl alcohol, shaking and removing the alcoholic phase each time. Transfer the ether layer in stages to a tared flask and evaporate off the solvent. Add 10 ml of acetone and evaporate off the solvent. Rotate the flask on a steam-bath during the operation. Cool the flask to between 60 °C to 65 °C, gently blow out the last traces of solvent with a current of dry air, cool in a desiccator and weigh.

D.3.4 Calculation

Non-detergent organic matter, per cent by mass = $100 \frac{m_1}{m_2}$

where,

 m_1 = mass, in g, of the non-detergent organic matter in the flask; and m_2 = mass, in g, of the material taken for the test.

APPENDIX E

EFFECT ON PAINTED SURFACES

E.1 APPARATUS

E.1.1 Glass panels, two glass panels as described in Clauses 3.2.1 and 3.2.2 of SLS 535:Part 3:Section 3.2.

E.1.2 Film applicator, as described in Clause 6 of SLS 535:Part 3: Section 3.3.

E.1.3 Glossmeter, as described in Clause 3.3 of SLS 535:Part 4: Section 4.3.

E.2 MATERIALS AND REAGENTS

E.2.1 High gloss enamel paint, complying with the requirements of SLS 540.

E.2.2 Trisodium phosphate solution, prepare 1 litre of a 0.2 per cent (m/V) aqueous solution of anhydrous trisodium phosphate (Na_3PO_4) , in a volumetric flask.

E.3 PREPARATION OF TEST PANELS

Two glass panels shall be prepared for painting as described in Clause 3.2.3 of SLS 535:Part 3:Section 3.2.

The paint shall be applied on one side of each of the two glass panels by means of the film applicator (see E.1.2) so that the paint film shall be uniform, 0.05-mm thick in the wet state and at least 75 mm x 75 mm in size. Allow the film to dry horizontally for 12 hours to 24 hours. Apply a second coat of paint on each panel using the applicator as described above and dry for a minimum of 24 hours.

NOTE - Use the panels within 48 hours.

E.4 PROCEDURE

Measure the specular gloss of each test panel in accordance with the method described in SLS 535:Part 4:Section 4.3. Prepare, using a volumetric flask, a 0.2 per cent (m/V) aqueous solution of the test sample and immerse one test panel in this solution, and the other in the trisodium phosphate solution for 1 hour at room temperature. Remove the panels from the solutions, rinse thoroughly with water and allow to dry at room temperature for at least 1 hour. Again measure the specular gloss of each panel.

Calculate the percentage decrease in gloss of the two panels after immersion in the test solutions.

Amendment No: 01 approved on 2013-10-02 to SLS 687 : 1985

SRI LANKA STANDARD SPECIFICATION FOR SYNTHETIC ORGANIC LIQUID DETERGENTS FOR HOUSEHOLD USE

3.3 pH

Delete the text and substitute the following :

"The pH at 27 ± 2 ⁰C of one per cent (*V/V*) solution of the detergent in distilled water shall be between 6.5 and 9.0 when tested as prescribed in Appendix C."

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SRI LANKA STANDARDS INSTITUTION

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