SRI LANKA STANDARD 760 : 2016

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SPECIFICATION FOR

SYNTHETIC LAUNDRY DETERGENT POWDER

(First Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 760 : 2016

Gr. 11

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SRI LANKA

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FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2016-06-23.

Synthetic detergent powders are intended for cleaning laundry with mixtures of chemicals including anionic, cationic and non-ionic surface active agents to improve the performance of cleaning even in hard or acidic water.

This standard focus on the detergent powders indicating mainly anionic surfactants, and hence the method involved in the determination of active ingredient content is a measure of same.

This Standard was first published in 1986. In this First Revision, the requirements for non detergent organic matter, available O_2 as sodium perborate and heavy metal limits are introduced. The requirements for total phosphates and matter insoluble in water have been revised. The requirement for sodium silicates has been withdrawn. A method for the evaluation of skin irritation of synthetic laundry detergent powder and a method for the determination of biodegradability have been included. The method of test for the determination of matter insoluble in water has been changed.

This standard is subject to the restrictions imposed by any applicable regulatory authority.

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 an analysis, shall be rounded off in accordance with **SLS 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 standard, the assistance derived from the following publications is gratefully acknowledged:

DEAS 127 -2: 2013	Synthetic detergent powders Part 2 : Machine wash
IS 4955 : 2001	Household Laundry detergent powders – Specification
IS 7597 : 2001	Surface active agents – Glossary of terms
IS 11601: 2002	Methods of safety evaluation of synthetic detergents – Tests for skin irritation and potential of synthetic detergents
IS 13933 : 1995	Method of test for ready biodegradability of surface active agents (Modified sturm test)
MS 553 : 2002	Detergent powder for hand wash household laundry
SS 231 : 2013	Laundry detergent powder for household use in manual washing

1 SCOPE

1.1 This Standard prescribes the requirements and methods of sampling and test for synthetic laundry detergent powder for use in domestic laundering machines and in hand laundering.

1.2 This is not applicable to soap-based laundry powders and nappy washing powders.

2 REFERENCES

ASTM	3049	Determination of synthetic anionic ingredient by cationic titration
SLS	102	Rules for rounding off numerical values
SLS	428	Random sampling methods
SLS	1348	Good manufacturing practices (GMP) for cleansing materials

3 DEFINITIONS

For the purpose of this standard the following definitions shall apply :

3.1 detergent : A product specially formulated for cleaning through the process of detergency.

NOTE:
A detergent comprises essential components (surface active agents) and generally complementary components (builders etc).

3.2 surface active agent : A chemical compound possessing surface activity which, dissolved in a liquid in particular in water, lowers the surface tension or interfacial tension, by preferred adsorption at the liquid/vapour surface, or other interfaces.

4 REQUIREMENTS

4.1.1 General requirements

4.1.1 The product shall be a homogeneous, free flowing powder which is readily soluble in water. It shall be free from any unpleasant odour and shall possess good lathering and cleaning properties.

4.1.2 The active ingredients used in the formulation of synthetic laundry detergent powder 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.

NOTES :

The product may contain builders and additives such as colouring matter, perfume, preservatives , opacifiers and optical brighteners.

In addition to the active ingredients specified above builders and additives which may be used in the formulation are given in Appendix **B**.

4.1.3 The product shall be non-injurious to the hands of consumers and to the fabric washed with it.

NOTES :

1. *It is recognized that some people may show an allergic reaction to common ingredients of detergent powder. It is not intended that this clause should cover such allergic reactions. Suitable protective measures should be taken against the degreasing action of detergents on the skin.*

2. *The method for the evaluation of skin irritation of synthetic laundry detergent powder is given in* Appendix **H**.

4.1.4 *Biodegradability*

The surfactants used in the manufacture of household laundry detergent powders shall be linear to be readily biodegradable.

NOTE :
The method for the determination of biodegradability is given in Appendix **J.**

4.1.5 The product shall be manufactured by a process adhering to Good Manufacturing Practices (GMP) complying with **SLS 1348**.

4.2 Other requirements

The product shall also comply with the requirements given in Table **1** when tested according to the relevant methods given in Column **4** of the Table.

TABLE 1 – Requirements for synthetic laundry detergent powder

SI. No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Active ingredient content, (see Note 1) per cent by mass, min.	12.0	ASTM D 3049
ii)	Total phosphates content expressed as sodium tripolyphosphate, per cent by mass, max. (See Note 2)	20.0	Appendix C
iii)	pH of 1 per cent solution at 27 ± 2 °C	8.0 to 11.0	Appendix D
iv)	Matter insoluble in water content, per cent by mass, max.	5.0	Appendix E
v)	Non detergent organic matter content, per cent by mass, max.	1.0	Appendix F
vi)	Available O_2 content as sodium perborate, per cent by mass, max.	1.0	Appendix G

NOTES :

1. *If the active ingredient is not declared in the label by the manufacturer, it is calculated as sodium alkyl benzene sulphonates.*

2. *Other substitutes for phosphate should be environmental friendly but should be in sufficient quantity to similar performance of the product as compared to that of phosphates.*

4.3 Limits for heavy metals (Optional)

The product shall also comply with the limits for heavy metals given in Table **2** when tested in accordance with Atomic Absorption Spectrophotometric method.

TABLE 2 – Limits for heavy metals

Sl. No. (1)	Heavy metal (2)	Limit (3)
i)	Lead (as Pb), mg/kg, max.	10
ii)	Arsenic (as As), mg/kg, max.	3
iii)	Mercury (as Hg), mg/kg, max.	1
iv)	Cadmium (as Cd), mg/kg, max.	3

5 PACKAGING AND MARKING

5.1 The detergent powder shall be well packed in a suitable container so as to retain its free flowing properties. Each container shall be marked legibly and indelibly with the following information:

a) Name of the product as "Synthetic laundry detergent powder";
b) Name and address of the manufacturer. In the case of imported products, name and address of the distributor / importer including the country of origin;
c) Registered trade mark / brand name, if any;
d) Net mass, in grams or kilograms;
e) Type of wash as "hand wash and/ or machine use";
f) Instructions for use, including any limitations on the type of fabric that can be washed and recommended usage / quantity;
g) Date of manufacture and date of expiry;
h) Batch or code number;
j) Warning statement as "Detergent solutions can be skin irritants. Avoid prolonged contact. Rinse garments and hands thoroughly"; and
k) The following critical ingredients (Chemical name) in descending order of quantity, per cent by mass:
    - Active ingredients;
    - Builders;
    - Soda ash;
    - Fillers;
    - Enzymes;
    - Optical brighteners;
    - Perfumes;
    - Preservatives; and
    - Perfumes, if any.

m) Precautuionary statement in mimimum of 1.5 mm font size as "Personnels with skin sensitive shall take necessary precautions to avoid direct contact." , if enzymes and bleaching agents (eg: perborate) are added.

5.2 A number of such containers as agreed to between the purchaser and the supplier may be suitably packed in a carton. Each carton shall be marked legibly and indelibly with the following information:

a) Name of the product as "Synthetic laundry detergent powder";
b) Name and address of the manufacturer. In the case of imported products, name and address of the distributor / importer including the country of origin;
c) Number of units per carton; and
d) Batch or code number.

6 METHODS OF TEST

6.1 Tests shall be carried out as prescribed in Appendices **C** to **G** of this standard and **ASTM D 3049**.

6.2 During the analysis, unless, otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

APPENDIX A
COMPLIANCE OF A LOT

The sampling scheme given in this Appendix should be applied where compliance of a lot to the requirements of this standard is to be assessed based on statistical sampling and inspection.

Where compliance with this standard, appropriate schemes of sampling and inspection shall be adopted based on manufacturer's control systems coupled with Type tests and testing procedures.

A.1 LOT

In a single consignment, all the containers of the same size and belonging to the same batch of manufacture shall constitute a lot.

A.2 SCALE OF SAMPLING

A.2.1 Samples shall be tested from each lot for ascertaining its conformity to the requirements of this specification.

A.2.2 The number of containers to be selected from each lot shall be in accordance with Table **3**.

A.2.3 If containers are packed in cartons 10 per cent of the cartons, subject to minimum of 2 cartons, shall be selected. As far as possible an equal number of containers shall be drawn from each carton so selected to get the required number of containers as given in Table **3**.

A.2.4 The cartons and containers shall be selected at random. In order to ensure randomness of selection, tables of random numbers as given in **SLS 428** shall be used.

TABLE 3 – Scale of sampling

No. of containers In the lot (1)	No. of containers To be selected (2)
Up to 100	3
101 to 300	4
301 to 500	5
501 to 1 500	8
501 to 3 000	9
3 001 and above	10

A.3 COMPOSITE SAMPLE

A sample, but equal quantity of material, shall be taken from each container selected as in **A.2.2** or **A.2.3.** The material so obtained shall be mixed thoroughly to form a composite sample which shall be of a sufficient size to carry out the tests for requirements specified in **A.5.4**.

A.4 INDIVIDUAL SAMPLE

The remaining portion of material from each container shall form an individual sample for testing the requirement specified in **A.5.3**.

A.5 NUMBER OF TESTS

A.5.1 Each carton selected as in **A.2.3** shall be inspected for packaging and marking requirements.

A.5.2 Each container selected as in **A.2.2** or **A.2.3** shall be inspected for packaging and marking requirements.

A.5.3 Each of the individual samples prepared as in **A.4** shall be tested separately for the active ingredient content as given in Table **1**.

A.5.4 The tests for the other requirements given in Table **1** shall be carried out on the composite sample prepared as in **A.3.**

A.6 CONFORMITY TO STANDARD

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

A.6.1 Each carton inspected as in **A.5.1** satisfies the relevant requirements.

A.6.2 Each container inspected as in **A.5.2** satisfies the relevant requirements.

A.6.3 The value of the expression ($\bar{x}$ - 0.4R), for active ingredient content tested as in **A.5.3** is not less than specified value.

NOTES:

1 *Mean (x)* $= \dfrac{\textit{Sum of the observed values}}{\textit{Number of values}}$

2 *Range R is the difference between the maximum and the minimum of a set of observed values.*

A.6.4 The composite sample tested as in **A.5.4** satisfies the relevant requirements.

APPENDIX B
LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

a) Trisodium phosphate;
b) Sodium carbonate;
c) Tetrasodium pyrophosphate;
d) Sodium tripolyphosphate;
e) Sodium hexametaphosphate;
f) Sodium carboxy methyl cellulose;
g) Sodium silicate;
h) Sodium bicarbonate;
j) Borax;
k) Optical brightners;
m) Foam boosters;
n) Sodium sulphate;
p) Hydrotropes;
q) Perfume;
r) Preservatives;
s) Chelating agents (sequestering agents);
t) Colours;
u) Bleaching agents
v) Anti caking agents; and
w) Enzymes.

APPENDIX C
METHOD OF TEST FOR
DETERMINATION OF TOTAL PHOSPHATES CONTENT

C.1 GENERAL

C.1.1 The sample is oxidized by gently heating with sodium nitrate. Silica is removed and the condensed phosphates are hydrolysed and precipitated as ammonium phosphomolybdate by the addition of ammonium molybdate. The precipitate is washed with dilute potassium nitrate solution and phosphorous in the washed precipitate is determined by titration with standard sodium hydroxide using phenolphthalein as indicator.

C.2 APPARATUS

C.2.1 Silica dish, 70-mm diameter

C.2.2 Beaker, 250-ml capacity

C.2.3 Buchner flask, 500-ml capacity with a sintered glass filter funnel

C.2.4 Volumetric flask, 500-ml capacity

C.2.5 Funnel, 75-ml diameter

C.2.6 Wide-mouthed flat-bottomed flask, 500-ml capacity

C.3 REAGENTS

C.3.1 Sodium nitrate

C.3.2 Hydrochloric acid, (a) concentrated and (b) 1:1 dilution (relative density =1.18).

C.3.3 Nitric acid, relative density 1.40

C.3.4 Ammonium molybdate reagent, dissolve 90 g of ammonium molybdate in hot water.

Add 240 g ammonium nitrate and stir to dissolve. Cool and add 30 ml of concentrated ammonium (rel .den. = 0.9). Dilute to 1 liter.

C.3.5 Potassium nitrate solution, 12.5 g/l

C.3.6 Sodium hydroxide, standard volumetric solution, c (NaOH) = 1 mol/1

C.3.7 Sulfuric acid, standard volumetric solution, c (H_2SO_4) = 0.5 mol/1

C.3.8 Potassium hydroxide solution, 5.6 g/l

C.3.9 Phenolphthalein solution, Dissolve 1 g phenolphthalein in 100 ml of ethyl alcohol.

C.4 PROCEDURE

C.4.1 Weigh, to the nearest milligram, approximately 1.5 g of the sample in a silica dish and add a small amount of 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 (**C.3.2**) 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 (**C.3.2**) and filter through a sintered glass crucible. Wash the crucible with 25 ml of dilute hydrochloric acid and then wash four times with 50 ml of water. Collect the filtrate and washings and make up to 250 ml in a volumetric flask.

C.4.2 Pipette a 50-ml portion from the volumetric flask into a 250-ml beaker. Add 10 ml of nitric acid (**C.3.3**) and boil for 15 minutes. Cool and add 100 ml of water and adjust the temperature of the solution to 40 ^{o}C to 45 ^{o}C. Add 50 ml of ammonium molybdate solution (**C.3.4**) (previously heated to 40 ^{o}C)slowly with constant stirring. Allow to stand for 30 minutes. Filter the precipitate through a quantitative filter paper. Wash the precipitate with potassium nitrate solution (**C.3.5.**) until 5 ml of the filtrate and to which a drop of phenolphathalein (**C.3.9**) does not require more than 3 to 4 drops of potassium hydroxide solution (**C.3.8**) to produce a pink colour. Transfer the filter paper with the precipitate to a 500-ml wide-mouthed flat-bottomed flask and add 100 ml of water. Heat over a water bath for 15 minutes, cool and titrate with sodium hydroxide solution (**C.3,6**) , using 1 ml of phenolphthalein until the pink colour just appears. Add 2 ml sodium hydroxide in excess. Shake well, heat to 60 ^{o} C in a water-bath. Cool and back titrate against sulfuric acid (**C.3.7**) until the pink colour just disappears. Calculate the volume of sodium hydroxide solution required to react with the precipitate.

C.5 CALCULATION

Total phosphates as sodium tripolyphosphate, per cent by mass

$$= \frac{V \text{ x } M \text{ x } 0.001349 \text{ x } 250 \text{ x } 100 \times 368}{m \text{ x } 50 \times 95}$$

where,

V is the volume, in ml, of sodium hydroxide solution required to react with the precipitate;
M is the morlarity of sodium hydroxide solution; and
m is the mass, in g, of sample taken for the test.

APPENDIX D
METHOD OF TEST FOR DETERMINATION OF pH VALUE

D.1 GENERAL

pH determination should be made in an acid free atmosphere.

D.2 APPARATUS

D.2.1 pH meter, any standard electrometric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solutions (see **D.3.2**) before use.

D.2.2 Volumetric flask, 1 000-ml capacity

D.2.3 Beaker, 100-ml capacity

D.3 REAGENTS

D.3.1 Distilled water, freshly boiled and cooled

D.3.2 Standard buffer solution, any two suitable buffer solutions within the pH range of 9 to 11 at 27 ^{0}C for calibrating the pH meter

D.4 PROCEDURE

Weigh, to the nearest milligram, approximately 10 g of the sample 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 ± 2 ^{o}C and fill to the calibration mark with the distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 27 ± 2 ^{o}C for 2 hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.

APPENDIX E
METHOD OF TEST FOR
DETERMINATION OF MATTER INSOLUBLE IN WATER

E.1 GENERAL

This method is intended for the determination of water insoluble matter in synthetic detergents containing silicates or phosphates or both. It is determined on the alcohol insoluble portion of the compound or mixture.

E.2 REAGENT

E.2.1 Ethanol, 95 % v/v

E.3 APPARATUS

E.3.1 Gooch crucible, having an asbestos filter

E.3.2 Oven, maintained at (105 ± 2) °C

E.4 PROCEDURE

E.4.1 Weigh, accurately 2 g sample of the material into a 250 ml beaker and digest with 100 ml ethanol (95 % v/v) on a steam bath until all soluble matter is in solution. Let the solution settle and filter the supernatant liquid through a weighed Gooch crucible with suction into a 250 ml Erlenmeyer flask retaining as much of the residue as possible in the beaker. Repeat this extraction five times with 25 ml portions of hot ethanol retaining as much of the residue as possible in the beaker.

E.4.2 Add 75 ml of hot water to the beaker containing the alcohol insoluble residue. Heat on a steam bath for about 15 min. Transfer the undissolved residue to the weighed Gooch crucible, washing with about 100 ml of hot water. The combined filtrate and washings should not exceed 200 ml. Dry the Gooch crucible to constant weight at 105 °C.

E.5 CALCULATION

E.5.1 $$\text{Water insoluble matter, \% } w/w = \frac{W_1}{W} \times 100$$

where,

W is the weight, in g, of material taken for the determination ; and
W_1 is the residue obtained by the procedure in **E.4.2**.

APPENDIX F
METHOD OF TEST FOR DETERMINATION OF NON- DETERGENT ORGANIC MATTER

F.1 GENERAL

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

F.2 APPARATUS

F.2.1 Evaporating basin

F.2.2 Separating funnels, 1 000-ml capacity

F.2.3 Wide mouthed flat-bottomed flask, 200-ml capacity

F.2.4 Buchner flask, 500 ml- capacity fitted with a sintered glass filter (porosity 4)

F.3 REAGENTS

F.3.1 Ethyl alcohol, 70 % and 90 % (by volume)

F.3.2 Petroleum ether, boiling rage 40 ^{0}C to 60 ^{0}C non-volatile residue at 80 ^{0}C maximum 0.001 %

F.3.3 Acetone, non-volatile residue at 80 ^{0}C maximum 0.001 %

F.4 PROCEDURE

F.4.1 Weigh accurately about 5 g of the material in a 150-ml squat beaker. Extract with 50 ml of hot 90 % ethanol by heating on the steam bath for about 2 min stirring and breaking up any hard lumps with a glass rod flattened at the end.

Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel (porosity 4) 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 % ethanol. Pass each extract in turn through the filter into the flask.

F.4.2 Transfer quantitatively all the combined filtrate from the Buchner flask to a 1-l 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 the third separating funnel with a further 75 ml of petroleum ether. Combine the three ether extracts in the first separating funnel. Rinse each of the two empty funnels with a few millilitres petroleum ether and add the rinsing to the combined ether extracts.

F.4.3 Wash the combined ether extracts and rinsing (see **F.4.2**) with four successive 50-ml portions of 70 % 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 about 60 ^{0} C to 65 ^{0} C, gently blow out the last traces of solvent with a current of dry air, cool in a desiccator and weigh.

F.5 Calculation

The non- detergent organic matter is expressed as follows:

$$\text{Non-detergent organic matter , \% by mass} = 100 \frac{m_1}{m}$$

where,

m_1 is the mass, in grams, of the non-detergent organic matter in the flask: and
m is the mass, in grams, of the material taken for the test.

APPENDIX G
METHOD OF TEST FOR DETERMINATION OF PERBORATE

G.1 REAGENTS

G.1.1 Sulfuric acid, 0.1 M

G.1.2 Standard potassium permanganate, 0.05 M

G.1 METHOD

Weigh 1.5 g – 2.0 g of sample, dissolve in water and make up to exactly 250 ml. Take an aliquot of 25 ml for titration and add 20 ml of 0.1M sulphuric acid. Titrate immediately with standard 0.05M potassium permanganate until a faint pink colour persists.

G.2 CALCULATION

$$\text{Available oxygen per cent by mass} = 8\frac{MV}{W}$$

where,

V is the titre;

M is the molality of potassium permanganate; and

W is the weight of material in aliquot.

NOTE :
When fresh contains approximately 95 % *corresponding to* 9.9 % *available oxygen.*

APPENDIX H
METHOD OF TEST FOR SKIN IRRITATION POTENTIAL OF SYNTHETIC DETERGENTS (HAND IMMERSION TEST)

H.1 PROCEDURE

H.1.1 Volunteers

Select 24 healthy adult volunteers (except pregnant ladies and breast feeding mothers) between the age of 18 and 55 years, with no known acute or chronic diseases or no adverse skin responses to cosmetics, personal wash or fabric wash products and no skin blemishes on their hands and forearms. They should not be under any medication, particularly anti inflammatory drugs. Inform the volunteers about the study in detail including the likely hazards and obtain a signed informed consent from each of them.

H.1.2 Products

Carry out the test on synthetic detergent products, prior to marketing. Store samples for the test properly to prevent contamination or spoilage. Do not use spoiled or contaminated sample. Preserve adequate quantity of the sample which is used for testing for at least 2 years for any re-testing. Identify each sample with an unique code number and make appropriate entry in the records. Ingredients used in the formulation should be of appropriate quality and have sufficient data to support safety at the level which is used in the product. In order to have a realistic approach for product dosing in the test, use the dosage regime given on the pack, however, in the event of not having such recommendation on the pack, a dosage of 7 g/l, 10 g/l or 14 g/l is recommended.

H.1.3 Positive control

Use 3 per cent *(w/v)* solution of sodium lauryl sulphate with 99.9 per cent purity (analytical grade) in later as positive control.

H.1.4 Method

Prepare fresh solution of the product and the positive control at the concentration mentioned above (see **H.1.2** and **H.1.3** respectively) using warm (37 ^{0}C) municipal water on each of the immersion days. Determine the pH and buffering capacity of the detergent solution and pH of Sodium lauryl sulphate solution on all the treatment (immersion) days. Transfer 500 ml of the respective solution into a suitable beaker/container for each volunteer for every immersion. Organize the seating arrangements of volunteers for hand immersion in a convenient/comfortable manner. 50 per cent of the volunteers in a test immerse their left hand in the test product/sample coded as 'A' and right hand in the positive control coded 'B', the other 50 per cent, vice versa. Each volunteer immerses his/her respective hand up to the wrist in the solution with constant movement of the fingers for agitating the liquid for 10 minutes twice a day (one in the morning and the other in the afternoon with a gap of about 4 h between two immersions) for 4 consecutive days. After each immersion, the volunteers wash the hands thoroughly with tap water and gently wipe with a dry towel.

H.1.5 Assessment and scoring

Assess the skin reactions and score them (double blind) as per the scale given in Table **4**, before each immersion under a constant light source by a trained assessor. The assessor should not know which hand received what product treatment. The final assessment and scoring shall be carried out on the morning after the last treatment. Add up the scores obtained at each assessment (total 9 assessments) and the mean of this score (total score divided by 9) is used for evaluating the product. Examine carefully the back of the hand, palm and the webs for scaling and glaze. Any sensation felt by the volunteer either during or after the immersion, and any other comments shall be taken into consideration in evaluating the product. Skin pH, hydration and trans epidermal water loss (TEWL) may also be monitored (purely optional) and recorded, to support the clinical observation.

H.1.6 Analysis of data and conclusion

Analyze the final score using student's paired t-test for determining the significance between the product and the positive control. If the product tested is found to cause adverse skin responses very similar to that manifested by the positive control, such a product could be judged to have unacceptable level of skin irritancy potential.

TABLE 4 - Scale for assessing the skin reaction

SI. No (1)	**Skin reactions (2)**	**Score (3)**
i)	No reaction	0
ii)	Very slight reaction (barely perceptible dryness and scales)	1
iii)	Slight reaction (perceptible dryness and scales usually in a small area)	2
iv)	Moderate reaction (perceptible dryness and scales in a major or whole area)	3
v)	Severe reaction (severe dryness and scales in the whole area with or without cracking)	4

H.1.7 Reporting

The report shall contain the following information:

a) Location of the study and date;
b) Identification of the sample;
c) Number of volunteers used;
d) Procedure followed and;
e) Result of the test in tabular form;
f) Any unusual findings;
g) Conclusion; and
h) Name of the investigator.

APPENDIX J
METHOD OF TEST FOR DETERMINATION OF BIODEGRADABILITY

J.1 APPARATUS

The apparatus shall be assembled as shown in Figure **1** consisting of the following:

J.1.1 Carbon dioxide scrubbing apparatus

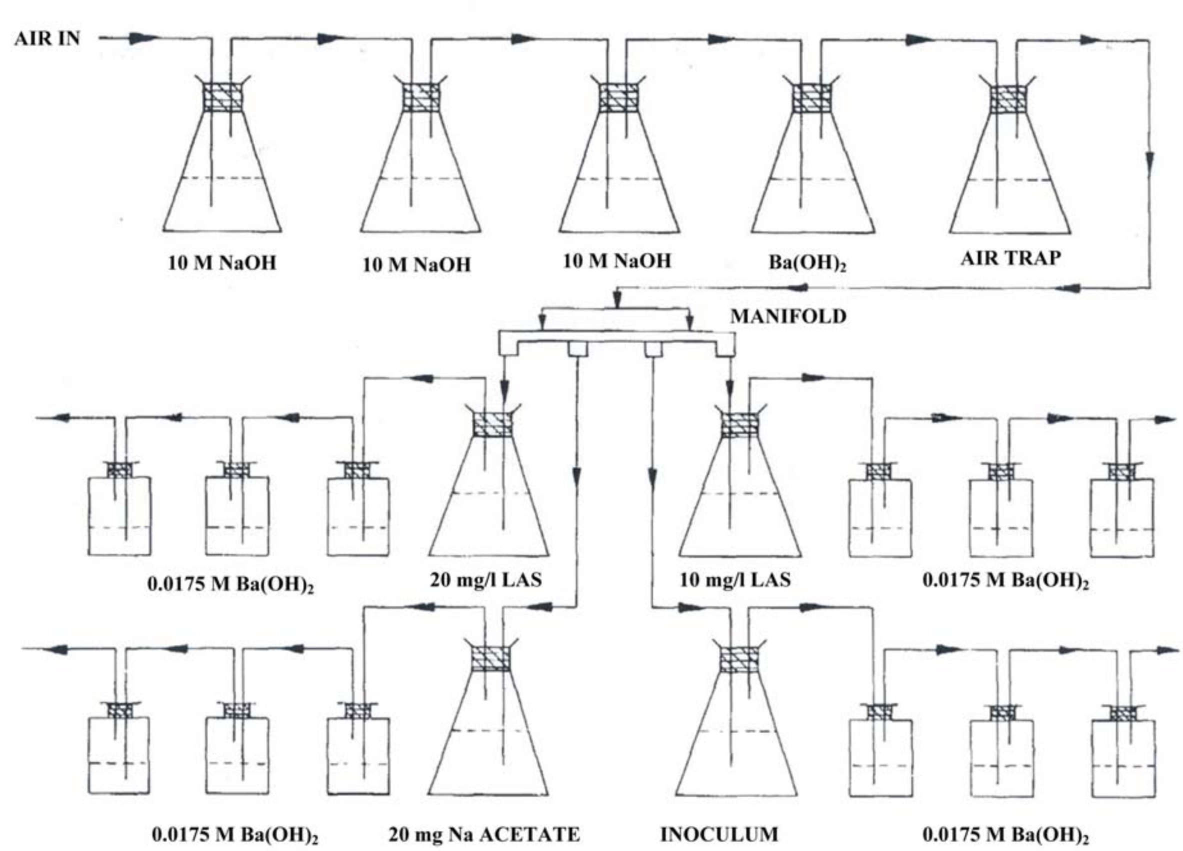

FIGURE 1 – OECD modified strum test for ready biodegradability

J.1.1.1 Four one-litre plastic bottles or glass flasks each containing 700 ml of 10 M Sodium hydroxide solution.

J.1.1.2 One one-litre Erlenmeyer flask containing 700 ml of 0.0175 M Barium hydroxide solution.

J.1.1.3 One empty one-litre Erlenmeyer flask as a trap to prevent liquid carry-over.

All these are connected in series, using suitable plastic tubing, to a pressurized air source, the air is sparged through the scrubbing solutions at a constant rate of flow.

J.1.2 Carbon dioxide production apparatus

J.1.2.1 Four 5-litre flasks for each test material.

J.1.2.2 Bubblers (100-ml Barium hydroxide absorbing solutions) - 18 numbers.

J.1.2.3 Stoppers and flexible plastic tubings.

J.2 REAGENTS

J.2.1 Purity of water

Sterilized distilled or deionised water free of compounds inhibitory to microbial survival must be used.

J.2.2 Mineral salts medium

The medium shall be prepared from the following stock solutions.
J.2.2.1 *Ferric chloride solution*

Dissolve 0.25 g Ferric chloride hexahydrate ($FeCL_3.6H2O$) in one litre of water.

J.2.2.2 *Magnesium sulphate solution*

Dissolve 22.5 g of Magnesium sulphate heptahydrate ($MgSO_4.7H_2O$) in one litre of water.

J.2.2.3 *Calcium chloride solution*

Dissolve 27.5 g of anhydrous calcium chloride in one litre of water.

J.2.2.4 *Phosphate buffer solution*

Dissolve 8.5 g of Potassium dihydrogen orthophosphate (KH_2PO_4), 21.75 g of di - potassium hydrogen orthophosphate (K_2HPO4), 33.4 g of disodium hydrogen orthophosphate heptahydrate ($Na_2HPO_4.7H_2O$) and 1.7 g of ammonium chloride (NH_4Cl) in one litre of water.

J.2.2.5 *Ammonium sulphate solution*

Dissolve 40 g of ammonium sulphate $(NH_4)_2SO_4$ in one litre of distilled water.

J.2.3 Test medium

Make up to one litre with water the following volumes of stock solution **(J.2.2):**
- 4 ml of ferric chloride solution,
- 1 ml of magnesium sulphate solution,
- 1 ml of calcium chloride solution,
- 2 ml of phosphate buffer solution, and
- 1 ml of ammonium sulphate solution.

The final solution will constitute the test medium.

J.2.4 **Barium hydroxide solution** - 0.0175 M.

Dissolve 4.0 g of barium hydroxide octahydrate $Ba(OH)_2.8H_2O$ in one litre of water. Filter through a filter paper and seal the clear solution to protect from air and carbon dioxide. When running a series, it is preferable to prepare more than 5 litres of solution.

J.2.5 Hydrochloric acid solution - 0.5 M.

J.2.6 Test organisms

J.2.6.1 *Source*

The source of the test organisms is activated sludge freshly sampled from a well-operated municipal sewage treatment plant receiving no or minimal industrial effluent.

J.2.6.2 *Preparation of microbial culture*

The activated sludge is aerated for 4 hours in the laboratory 500 ml of the mixed liquor is sampled and homogenized for 2 minutes at medium speed in a blender. It is then settled for 30 minutes.

If the supernatant still contains high levels of sludge solids at the end of 30 minutes, it may be settled for an additional 30 minutes to 60 minutes or adapted to laboratory conditions to obtain better settling.

The supernatant is decanted to provide sufficient volume for 1 per cent inoculum for each carbon dioxide test flask. Carry-over of sludge solids should be avoided, otherwise this would interfere with measurement of carbon dioxide produced.

J.2.6.3 *Measurement of viable count*

It is useful to perform viable counts on the supernatant fraction to determine microbial numbers; this is optional. The inoculum should normally contain 10^6 to 20×10^6 colony forming units per millilitre. It should be used the same day it was prepared.

J.3 PROCEDURE

J.3.1 Preparation of sample

Prepare a stock solution of sample by dissolving a homogeneous aliquot of the test material in water, the concentration would depend on the active content percentage. To obtain a homogeneous sample, it is necessary to mix well, at the same time avoiding forming which will tend to concentrate the active ingredient disproportionately. This step is extremely important for correct calculation of biodegradation based on the exact amount of carbon in test system.

J.3.2 Total organic carbon (TOC) of test compound

Determine the TOC of the test material by analyzing the stock solution (or the neutralized aliquot).

J.3.3 Reference substances

In order to check the activity of the inoculum, the use of control substances is desirable. Aniline sodium acetate or sodium benzoate are suggested as reference substances. The reference substance shall yield more than 60 per cent dioxide within 28 days, otherwise the test is regarded as invalid and must be repeated with an inoculum from a different source.

J.3.4 Test conditions

The following steps shall be done in sequence:

To each of the four 5-litre test flasks, add 2 470 ml of water followed by 3 ml of ammonium sulphate, 3 ml of magnesium sulphate buffer, 12 ml of ferric chloride (all stock solutions), and 30 ml of activated sludge inoculums. These additions now total 2 527 ml in each flask. Aerate the mixtures with carbon dioxide-free air (**3.1.1**) for 24 hours to purge the system of carbon dioxide.

After the aeration period, three carbon dioxide absorber bottles are each filled with 100 ml of 0.25 M barium hydroxide solutions and connected in series to the exit air line of each test flask.

Now, test material is added to two of the four flasks to begin with testing period. Each material is tested at two concentrations, 10 and 20 mg/ l. The amount of test material (stock solution) required in the flask is calculated as given below:

$$\text{Stock solution per flask, ml} = \frac{B \times C}{A}$$

where,

A is the test compound concentration in stock solution, mg/1;

B is the test compound concentration in test flask, mg/1; and

C is the final volume of test medium in the test flask (ml).

Sufficient stock solution to reach the desired test concentration, as calculated above, plus sufficient water to make 473 ml (stock solution + water) are added to the appropriate flasks. To the third flask, used as blank control and containing no test material, add 473 ml of water. The final volume in each flask is now 3 000 ml.

A control substance (**J.3.3**), at a concentration of 20 mg/1, is added to the fourth flask.

J.3.5 Performance of the test

To start with, bubble carbon dioxide-free air (**J.1.1**) through the solutions at a rate of 50 ml-100 ml/ min per flask (approximately 1 to 2 bubbles per second). The carbon dioxide produced in each flask reacts with the barium hydroxide and precipitates as barium carbonate. The amount of carbon dioxide produced is determined by titrating the unreacted barium hydroxide with standard 0.05 M Hydrochloric acid. Periodically, every 2 or 3 days, the carbon dioxide absorber nearest to the flask is removed for titration. The remaining two absorbers are moved closer to the flask, and a new absorber filled with 100 ml of fresh barium hydroxide solution is placed at the far end of the series. Titrations are made as needed (before any barium carbonate precipitate is evident in the second trap) approximately every alternate day for the first 10 days, and every fifth day until the 28th day.

On the 26th day, the pH of the flask contents is measured again, and then 1 ml of concentrated hydrochloric acid is added to each of the test flasks to decompose inorganic carbonate. The final titration is made on Day 28.

Titrations of the 100-ml barium hydroxide solution are made after removing the bottles closest to the flask. The barium hydroxide is titrated with standard hydrochloric acid, using phenolphthalein indicator.

The test is run at 25 + 2 °C temperature which is recorded throughout the duration of the test.

J.3.6 Reporting of the results

The amount of carbon dioxide produced by the test compound during the test is measured and expressed as per cent of the theoretical carbon dioxide it should have produced (TCO_2) as calculated from the carbon content of the test compound (TOC). Biodegradability is expressed as percentage TCO_2.

$$TC0_2 = \text{mg } CO_2 \text{ per mg of test material}$$

$$= \frac{(\text{number of carbon atoms in test material X molecular weight of } CO_2)}{\text{molecular weight of active test material}}$$

Example: sodium lauryl sulphate (SLS) contains 12 carbon atoms and therefore,

$$TCO_2 \text{ of SLS} = \frac{12 \times 44}{288.33} = 1.831 \text{mg } CO_2 / \text{mg SLS}$$

In the case of mixtures, TCO_2 of the total active material is weighed average of the TCO_2 of the individual compound.

J.3.7 Precaution

Evolution of a significant amount of carbon dioxide from the blank flask during test would indicate contamination or the medium, glassware or air supply. A total Carbon dioxide evolution in the blank at the end of the test exceeding 50 mg. Carbon dioxide per 3 litres medium should be considered as invalidating the test.

J.3.8 Calculations

The first step in calculating the amount of carbon dioxide produced is to correct the test material flasks for endogenous carbon dioxide production. The control flask serves as a “seed blank” to correct carbon dioxide which may be produced through endogenous respiration of the bacteria. The amount of carbon dioxide produced by a test material is determined by the difference (in ml of titrant) between the experimental and blank barium hydroxide traps.

Sample Calculation:

Blank	48.0	ml hydrochloric acid titrated
Experimental	45.0	ml hydrochloric acid titrated
Test Material	3.0	ml hydrochloric acid titrated

Then convert ml hydrochloric acid titrated into mg of carbon dioxide produced.

$$\text{Carbon dioxide, mg} = \frac{(0.5) \times \text{ml titrated} \times 44}{2}$$

$$= 1.1 \text{ X ml hydrochloric acid titrated}$$

The final step is calculating the theoretical carbon dioxide produced using the following formula

$$TCO_2\text{, percent} = \frac{\text{mg } CO_2 \text{ produced } \times 100}{(\text{mg test material added}) \times (TCO_2 \text{ of test material})}$$

$$\text{Biodegradability per cent} = \frac{\text{Amount of } CO_2 \text{ produced } \times 100}{\text{Theoretical amount of } CO_2}$$

J.3.9 Interpretation of the results

J.3.9.1 Test materials giving a result to greater than 60 per cent yield of carbon dioxide (within 28 days) are regarded as readily biodegradable. This level must be reached within 10 days of biodegradation exceeding 10 per cent.

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