Synthetic industrial detergent powder — Specification
DKS 2292:2011

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The following organizations were represented on the Technical Committee:

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Foreword

This Kenya standard was developed by the Technical Committee on Soaps and Detergents under the guidance of the Standards Projects Committee and is in accordance with the procedures of the Bureau.

Synthetic detergents or non-soapy detergents (NSD), as they are normally termed, are products specially formulated to promote the development of detergency, and comprise essential components (surface active agents), and generally, complementary components like builders, e.t.c. Locally they are as known industrial detergent powders, (IDP) and they are meant for general purpose cleaning in the industry.

At present IDP produced mainly are of the alkyl aryl type, such as sodium salt of dodecyl benzene sulphonic acid and thus they are different from soaps, the other class of detergents, which are mainly sodium salts of higher fatty acids. The limitations of soaps for use in hard water areas have led to the development of synthetic detergents. The development of this standard was initiated by the industry.

This standard emphasizes on the biodegradability of the surfactants used to manufacture detergent powders and a method for determining the same has been developed.

During the development of this standard, reference was made to:

IS 4956: 2002, Synthetic detergents for industrial purposes - Specification

Acknowledgement is hereby made from the assistance derived from the above source.
Synthetic industrial detergent powder — Specification

1 Scope

This Kenya standard prescribes the requirements and methods of test for synthetic industrial detergent powders based predominantly on the use of alkyl aryl sulphonates.

This Kenya Standard covers synthetic industrial detergent powders only and does not cover synthetic detergent powders for household use and machine wash.

2 Requirements

2.1 Description

The active ingredients used may include, besides salt of alkyl aryl sulphonic acid, soap and non-ionsics. The active ingredient used must be biodegradable when tested against KS 92-3. The formulation may contain one or more of the builders or additives given in Annex A. The material shall be a free flowing powder, free from visible dirt and impurities. The material shall not give any unpleasant odour and shall have good cleaning and lathering properties. The material shall also comply with the requirements given in Table 1 when tested in accordance with the corresponding test method.

Table 1 — Requirements for industrial detergent powders

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3 Packaging and marking

3.1 Packing

The material shall be supplied in suitable well-closed packages.

3.2 Marking

The container shall be securely closed and marked legibly and indelibly with the following information:

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1) Determination of biodegradability of surfactants — Test Method

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a) Manufacturer’s name and address and/or registered trade mark if any.
b) Product name.
c) Batch or code number.
d) Net weight.
e) Country of origin.
f) Instructions for use.
g) Date of manufacture and best before date
h) Cautionary statement “this product is not recommended for hand wash”

4 Tests

Tests shall be carried out as prescribed in Annexes B to G. Due to the fact that detergent powder can absorb or lose moisture during storage, the analytical results found on samples in open markets shall be calculated to the original weight found out at the sampling stage, i.e.

\[
\frac{\text{Actual weight} \times \text{moisture determined}}{\text{specified weight}}
\]

Unless otherwise specified, chemicals of analytical grades and distilled water shall be used in tests.
Annex A
(normative)

List of suggested builders and additives

Sodium carbonate
Sodium sulphate
Sodium carboxymethyl cellulose
Sodium silicate
Optical brighteners
Lather boosters
Hydrotropes
Perfume
Preservatives
Chelating agents (sequestering agents)
Colourants
Perborates
Enzymes
Bactericides
Common salt
Urea
Magnesium sulphate
Sodium tripolyphosphate (STPP)
Zeolites
Any other internationally accepted builder cleared by the Kenya Bureau of Standards.
Annex B
(normative)

Determination of active ingredient

B.1 Principle
Active matter, namely, the sodium salt of sulphonated alkyl benzene, shall first be separated from inorganic salt and non-detergent organic matter. It shall then be neutralized with ethyl alcohol, dried and weighed. Finally the weighed extract shall be corrected for the presence of sodium chloride and alkali carbonates.

B.2 Apparatus

B.2.1 Beakers
150 mL and 100 mL capacity.

B.2.2 Buchner flask
500 mL capacity fitted with a sintered glass filter funnel.

B.2.3 Evaporating basin

B.2.4 Separating funnels
100 mL capacity.

B.2.5 Steam bath

B.2.6 Wide-mouthed flat-bottomed flask
200 mL capacity.

B.2.7 Air-oven
Preferably electrically heated, with temperature control device.

B.3 Reagents

B.3.1 Ethyl alcohol
30 per cent, 96 per cent and absolute (by volume).

B.3.2 Diethyl ether

B.3.3 Acetone

B.3.4 Standard sulphuric acid
Approximately 0.1N.

B.3.5 Standard silver nitrate solution
0.1N.

B.3.6 Phenolphthalein indicator
1 per cent solution in 95 per cent (by volume) ethyl alcohol.

B.3.7 Methyl orange indicator
0.1 per cent (m/v).

B.3.8 Nitric acid
Concentrated, sp. gravity 1.42.

B.3.9 Nitro-benzene.

B.3.10 Standard ammonium thiocyanate solution, approximately 0.1N.
B.3.11 Ferric ammonium sulphate indicator, saturated solution.

B.3.12 Caustic soda solution, 10 per cent (m/v).

B.4 Procedure

B.4.1 Removal of inorganic salts

Weigh accurately about 5 g of the material for products containing about 20 % active matter, and correspondingly less for products of higher active matter content. Proceed as described in the determination of matter insoluble in alcohol (Annex D). After filtering and washing the residue thoroughly with hot ethyl alcohol, evaporate the combined filtrate to a small bulk in an evaporating basin.

B.4.2 Determination of active matter

B.4.2.1 To ensure that the residue is completely anhydrous, add 300 mL of absolute alcohol and evaporate to dryness. Extract the residue with 30 mL of hot 96 % 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 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 96 per cent ethyl alcohol from the jet of a wash bottle.

B.4.2.2 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.4.2.3 Heat the flask for not more than five minutes 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 weighings does not exceed 3 mg. Record this weight as \( M_1 \).

B.4.2.4 The extract obtained in B.4.2.1 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.4.3 Determination of alkali carbonates

B.4.3.1 Dissolve the extract in B.4.2.4 in cold distilled water, add a few drops of methyl orange indicator and titrate with standard sulphuric acid to the methyl orange endpoint.

B.4.3.2 Calculation

Mass in g of sodium carbonate,

\[ M_2 = 0.053 \times V \times N \]

where

\( V \) = Volume in ml of standard sulphuric acid used and,

\( N \) = Normality of the standard sulphuric acid solution.

B.4.3.3 Reserve the solution for the estimation of chlorides.

B.4.4 Determination of chlorides

B.4.4.1 To the solution remaining after the estimation of alkali carbonates, 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 aluminium sulphate as indicator.

B.4.4.2 Calculation
Mass in g of sodium chloride,
\[ M_3 = 0.058 (20N - VN_1) \]

where,
- \( N \) = normality of the standard silver nitrate solution;
- \( V \) = volume in ml of standard ammonium thiocyanate used;
- \( N_1 \) = normality of the standard ammonium thiocyanate solution.

**B.4.4.3 Calculation**

Active matter, per cent by mass

\[ = \frac{100 (m_2 - m_2 - m_3)}{m} \]

where,
- \( m_1 \) = is the mass in g of alcohol extract (see B.4.2.4);
- \( m_2 \) = is the mass in g of sodium carbonate (see B.4.3.2);
- \( m_3 \) = is the mass in g of sodium chloride (see B.4.4.2);
- \( m \) = is the mass in g of the material taken for the test.
Annex C
(normative)

Determination of moisture and volatile matter content

C.1 Principle Moisture and volatile matter is determined by oven method.

C.2 Apparatus

C.2.1 Porcelain or silica dish 6 cm to 8 cm in diameter and 2 cm to 4 cm in depth.

C.2.2 Desiccator Containing an efficient desiccant, such as phosphorus pentoxide.

C.2.3 Air-oven, preferably electrically heated, with temperature control device.

C.3 Procedure

Weigh accurately about 5 g of the material into a dry tarred dish, and dry to constant mass in air-oven at a temperature of 105 ±1°C. Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for one-hour period shows a difference of not more than 5 mg in the net loss in mass.

C.4 Calculation

Moisture and volatile matter content (at 105°C), per cent by mass \( \frac{m_0 - m_1}{m_0} \times 100 \)

where

\( m_0 \) = mass in g of the material taken for test, and

\( m_1 \) = mass in g of the material upon drying.
Annex D
(normative)

Determination of matter insoluble in alcohol

D.1 Principle of method
Matter insoluble in alcohol, generally means the inorganic salts, such as phosphates, sulphates, silicates and carbonates which are usually present in non-soapy detergent preparations. These can be separated from active matter, non-detergent organic matter, e.t.c by extracting the material with 96 per cent ethyl alcohol.

D.2 Apparatus
D.2.1 Beaker
150 mL capacity.

D.2.2 Steam bath

D.2.3 Buchner flask
500 mL capacity, fitted with sintered glass filter funnel.

D.2.4 Air-oven
Preferably electrically heated, with temperature control device.

D.3 Reagents
D.3.1 Ethyl alcohol Freshly boiled, 96 per cent or higher (by volume).

D.4 Procedure
D.4.1 Weigh accurately about 5 g of the material into a beaker, and digest with 50 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 five 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 soluble matter.

Dry the sintered glass funnel with the residue in an air-oven at a temperature of 105 ±2°C until a constant weight is obtained.

D.4.2 Even after digestion with five 30 mL portions of boiling ethyl alcohol, the alcohol insoluble portion may sometimes be found to be sticky. In that case treat it further with more boiling ethyl alcohol until it is free from active matter and the alcohol insoluble portion is no longer sticky.

D.4.3 Calculation
Matter insoluble in alcohol, per cent by mass = \( \frac{100 \cdot m_0}{m_1} \)

Where,
- \( m_0 \) = in grams of matter insoluble in alcohol, and
- \( m_1 \) = in grams of material taken for the test.
Annex E
(normative)

Determination of hydrogen ion concentration

E.1 General
pH determination shall be made in acid free atmosphere.

E.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 solution before use.

E.2.2 Volumetric flask –100 mL capacity.

E.3 Reagents

E.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 30°C. The residue on evaporation when heated at 105°C for one hour shall not exceed 0.5 mg per litre.

E.3.2 Standard buffer solution
Any two suitable buffer solutions within the pH range of 9 to 11 at 30°C for calibrating the pH meter.

E.3.3 Procedure
Weigh 10±0.001 g material and transfer to a 1 litre volumetric flask. Partially fill the flask with distilled water and agitate until the sale is completely dissolved. Adjust the temperature of the solution and the distilled water to 30±0.5°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 30°C for two hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.
Annex F
(normative)

Determination of non-detergent organic matter (NDOM)

F.1 Principle of the method
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 alkylamide present in the material.

F.2 Apparatus
F.2.1 Evaporating basin
Wide-mouthed flat-bottomed flask, 200 mL capacity, fitted with a sintered glass filter funnel porosite.

F.3 Reagents
F.3.1 Ethyl alcohol
50 per cent and 96 per cent (by volume).

F.3.2 Petroleum ether
Boiling range 40 °C to 60 °C, non-volatile residue at 80°C, maximum 0.001 %.

F.3.3 Acetone
Non volatile residue at 80 °C, maximum 0.001 %.

F.4 Procedure
F.4.1 Removal of inorganic salts
Proceed as in D.4.1.

F.4.2 Transfer quantitatively all the combined filtrate from the Buchner flask in D.4.1 to a 1 litre separating funnel. 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 the second separating funnel, and extract with 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 of petroleum ether and add the rinsings to the combined ether extracts.

F.4.3 Wash the combined ether extracts and rinsing (F.4.2) 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. Rotate the flask on a steam bath during the operation. Cool the flask to about 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.

F.5 Calculation
Non-detergent organic matter, per cent by mass, 

\[ \frac{100m_0}{m_1} \]

where

- \( m_0 \) = the mass in g of the non-detergent organic matter in the flask, and
- \( m_1 \) = the mass in g of the material taken for the test.
ANNEX G
(normative)
Determination of matter insoluble in water

G.1 Procedure
Starting with a fresh portion of the material, proceed as described under D.4 but do not dry or weigh the matter insoluble in alcohol. After filtering and washing the residue thoroughly with hot ethyl alcohol, change the receiver, extract the residue with successive portions of distilled water at about 60 °C, and wash the residue several times to remove all the water solubles. Dry the sintered glass funnel with the residue in an air-oven at a temperature of 105 °C ± 2 °C until a constant mass is obtained.

G.2 Calculation

Matter insoluble in water, % by mass = \( \frac{m_1}{m} \times 100 \)

Where

\( m_1 \) is the mass in g of matter insoluble in water; and
\( m \) is mass in g of material taken for the test.