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## **EAST AFRICAN STANDARD**

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Synthetic industrial detergent powder — Specification

DRAFT

**EAST AFRICAN COMMUNITY**

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## Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to meet the above objectives, the EAC Partner States have enacted an East African Standardization, Quality Assurance, Metrology and Test Act, 2006 (EAC SQMT Act, 2006) to make provisions for ensuring standardization, quality assurance, metrology and testing of products produced or originating in a third country and traded in the Community in order to facilitate industrial development and trade as well as helping to protect the health and safety of society and the environment in the Community.

East African Standards are formulated in accordance with the procedures established by the East African Standards Committee. The East African Standards Committee is established under the provisions of Article 4 of the EAC SQMT Act, 2006. The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

Article 15(1) of the EAC SQMT Act, 2006 provides that “Within six months of the declaration of an East African Standard, the Partner States shall adopt, without deviation from the approved text of the standard, the East African Standard as a national standard and withdraw any existing national standard with similar scope and purpose”.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

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## **Introduction**

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. They are sometimes known as industrial detergent powders, (IDP) and they are meant for general purpose cleaning in the industry.

IDP produced are mainly 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. This East African standard emphasizes on the biodegradability of the surfactants used to manufacture detergent powders.

## Synthetic industrial detergent powder — Specification

### 1 Scope

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

### 2. Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies

ISO 673, *Analysis of soap — Determination of ethanol insoluble matter*

ISO 862, *Surface active agents — Vocabulary*

ISO 2271 *Surface -Active agent - Determination of anionic active matter by manual or mechanical direct two phase -Phase procedure.*

### 3 Terms and definition

For the purposes of this standard terms and definitions specified under ISO 862 apply.

### 3 Requirements

The active ingredients used may include, besides salt of alkyl aryl sulphonic acid, soap and non-ionics.

The active ingredient used must be biodegradable when tested against EAS 127. 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. The fineness shall be as agreed between manufacturers and customer.

**Table 1 — Requirements for industrial detergent powders**

SI No	Characteristic	Requirement	Test method
i)	Active ingredient, %, m/m, min.	10.0	Annex B/ ISO 2271
ii)	Moisture and volatile matter content at 105 °C, %, m/m, max.	13.0	Annex C
iii)	Matter insoluble in alcohol, % m/m, max.	90.0	Annex D/ ISO 673
iv)	Matter insoluble in water, % by mass,	5.0	Annex G

	max		
v)	pH of 1 % solution (m/v) at 25 ± 2 °C	9 to 11	Annex E
vi)	Non-detergent organic matter (NDOM), %, m/m, max.	1.0	Annex F

#### 4 Packaging and marking

##### 4.1 Packing

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

##### 4.2 Marking

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

- a) Product name.
- b) Manufacturer's name and address and/or registered trade mark if any.
- c) Batch or code number.
- d) Net weight.
- e) Country of origin.
- f) Instructions for use (which shall be in either English, Kiswahili or French or in combination as agreed between the manufacturer and supplier”).
- 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.

$$\frac{\text{Actual weight x moisture determined}}{\text{specified weight}}$$

**Annex A**  
(Informative)

**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  
Magnesium sulphate  
Sodium tripolyphosphate (STPP)  
Zeolites  
Any other internationally accepted builder cleared by respective partner states Bureaux 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 treated 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  $100 \pm 1^\circ\text{C}$ , 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

**B.4.2.5** The extract obtained in B.4.2.4 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.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,  
 $M_2 = 0.053 VN$

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**  $= 100 \frac{(m_0 - m_2 - m_3)}{m}$

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  $105 \pm 1^\circ\text{C}$ . 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**

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

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 = 
$$\frac{100 m_0}{m_1}$$

Where,

$m_o$  = 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  $10 \pm 0.001$  g material and transfer to a 1 litre volumetric flask. Partially fill the flask with distilled water and agitate until the salt is completely dissolved. Adjust the temperature of the solution and the distilled water to  $30 \pm 0.5^\circ\text{C}$ , 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 flasks 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**

$$\text{Matter insoluble in water, \% by mass} = 100 \frac{m_1}{m}$$

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.



