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Synthetic laundry detergent paste — Specification

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 the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS) and other deliverables. The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. 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 Principles and procedures for development of East African Standards.

East African Standards and other deliverables 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.

The committee responsible for this document is Technical Committee EASC/TC 074, *Surface active agents*

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

This second edition cancels and replaces the first edition (EAS 788:2013), which has been technically revised.

Synthetic laundry detergent paste — Specification

1 Scope

This Draft East African standard specifies the requirements, sampling and test methods for synthetic laundry detergent pastes based predominantly on alkyl aryl sulphonates for hand and machine wash.

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.

EAS 794, *Determination of the microbial inhibition of cosmetic soap bars and liquid hand and body washes - Test method*,

EAS 814 *Determination of biodegradability of surfactants — Test method*.

ISO 673, *Soaps — Determination of content of ethanol insoluble matter*

ISO 862, *Surface active agents — Vocabulary*

ISO 2271, *Surface active agents — Detergents — Determination of anionic active matter by manual or mechanical direct two-phase titration procedure*

3 Terms and definitions

For the purposes of this standard terms and definitions given in ISO 862 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <http://www.iso.org/obp>

synthetic detergent paste

thick, soft, moist substance formulated for removing stains, brightening and softening laundry.

4 Requirements

4.1 General requirements

4.1.1 The active ingredients used include salt of alkylaryl sulphononic acid, soap and non-ionics. The formulation may contain one or more of the builders or additives given in Annex A.

4.1.2 The active ingredient used shall be biodegradable when tested according to EAS 814.

4.1.3 The synthetic detergent paste shall be free from foreign matter and the components shall be homogeneously dispersed and shall not be separated.

4.1.4 The synthetic detergent paste shall not give any objectionable odour and shall have good cleaning and lathering properties.

4.2 Specific requirements

The synthetic detergent paste shall comply with the specific requirements given in Table 1 when tested in accordance with the corresponding test method.

Table 1 — Specific requirements for synthetic detergent paste

S/No.	Characteristic	Requirement	Test method
i.	Active ingredient, (on dry basis) % m/m, min.	18.0	ISO 2271
ii.	Moisture and volatile matter content at 105 °C, % m/m, max.	65	Annex B
iii.	Matter insoluble in ethanol (on dry basis), % m/m, max.	80.0	ISO 673
iv.	Matter insoluble in water, % m/m, max	5.0	Annex C
v.	pH of 1 % solution (m/v) at 25 °C ± 2 °C	9 - 11	Annex D
vi.	Non-Detergent Organic Matter (NDOM), (on dry basis) % m/m, max.	1.0	Annex E
vii.	Phosphate (expressed as sodium tripoly-phosphate), % m/m, max	15	ISO 4313
viii.	antibacterial activity ^{a)}	To pass the test	EAS 794

^{a)}This test is only applicable for synthetic laundry detergent paste with antibacterial claims

5 Packaging

Synthetic detergent paste shall be packaged in suitable well-closed containers/packages.

The containers/packages (including the closures) shall not impart any contamination to the synthetic detergent paste and shall be strong enough to protect the content adequately during normal handling, transportation and storage.

6. Labelling

Each package shall be legibly and indelibly labelled either in English, Kiswahili or French or combination or any other language as agreed to between the manufacturer and supplier with the following information:

- a) name of the product as “synthetic laundry detergent paste”;
- b) manufacturer’s name and physical address

NOTE The name, physical address of the distributor/supplier and trade mark may be added as required

- c) batch or code number;
- d) list of ingredients
- e) net content;
- f) country of origin;
- g) declare if antibacterial agent is incorporated;
- h) instructions for use;
- i) date of manufacture and best before date; and
- j) indicate mode of disposal of containers/packages

7 Sampling

Sampling shall be done in accordance with Annex F.

Annex A (informative)

List of suggested builders and additives

The following is a list of suggested builders and additives:

- a) sodium carbonate;
- b) sodium sulphate;
- c) sodium carboxymethyl cellulose;
- d) sodium silicate;
- e) optical brighteners;
- f) lather boosters;
- g) hydrotropes;
- h) perfume;
- i) preservatives;
- j) chelating agents (sequestering agents);
- k) colourants;
- l) perborates;
- m) enzymes;
- n) bactericides;
- o) common salt;
- p) magnesium sulphate;
- q) Sodium tripolyphosphate (STPP);
- r) zeolites; and
- s) any other internationally accepted builder cleared by respective partner states bureaux of standards.

Annex B (normative)

Determination of moisture and volatile matter content

B.1 Principle

The moisture and volatile matter content are determined by oven method.

B.2 Apparatus

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

B.2.2 Desiccator, containing an efficient desiccant, such as phosphorus pentoxide

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

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

B.4 Calculation

The moisture and volatile matter content are expressed as follows:

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

where

m_0 is the mass, in grams, of the material taken for test, and

m_1 is the mass, in grams, of the material upon drying

Annex C (normative)

Determination of matter insoluble in water

C.1 Apparatus

C.1.1 Beaker, 150-ml capacity

C.1.2 Steam bath

C.1.3 Buchner flask, 500-ml capacity, fitted with sintered glass filter funnel (porosity 4)

C.1.4 Air-oven, preferably electrically heated, with temperature control device

C.1.5 Vacuum pump

C.2 Reagents

C.2.1 Ethyl alcohol, freshly boiled, 96 % or higher (by volume)

C.2.2 Distilled water

C.3 Procedure

C.3.1 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 min. Stir and break up any hard lump with a glass rod flattened at one end.

C.3.2 Allow the solid matter to settle and decant the hot alcoholic solution through a dried and pre-weighed sintered glass filter funnel fitted to a Buchner flask, to which suction is applied

C.3.3 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.

C.3.4 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.

C.3.5 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.

C.3.6 Dry the sintered glass funnel with the residue in an air-oven at a temperature of 105 °C ± 2 °C for 1 h. Cool in a desiccator and weigh.

C.3.7 Repeat the process in C.3.6 for 30 min until a constant weight is obtained.

C.4 Calculation

The matter insoluble in water is expressed as follows:

$$\text{Matter insoluble in water (on dry basis), \% m/m} = 100 \frac{m_2 - m_1}{m} \times \frac{100}{(100 - \text{moisture})}$$

where

m_1 is the mass, in grams, of empty sintered glass filter funnel

m_2 is the mass, in grams, of sintered glass filter funnel with residue

m is the mass, in grams, of material taken for the test.

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Annex D (normative)

Determination of pH

D.1 General

pH determination shall 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 solution before use.

D.2.2 Volumetric flask, 100-ml capacity

D.3 Reagents

D.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/L.

D.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

D.4 Procedure

Weigh $10 \text{ g} \pm 0.001 \text{ g}$ material and transfer to a 1-L 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 $30 \text{ °C} \pm 0.5 \text{ °C}$, 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 2 h prior to measuring the pH. Measure the pH of the solution using a glass electrode at $25 \text{ °C} \pm 2 \text{ °C}$.

Annex E (normative)

Determination of Non-Detergent Organic Matter (NDOM)

E.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 alkylamide present in the material.

E.2 Apparatus

E.2.1 Evaporating basin

E.2.2 Separating funnels, 1 000-ml capacity.

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

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

E.2.5 Fume hood or well ventilated room

E.2.6 vacuum pump

E.3 Reagents

E.3.1 Ethyl alcohol, 50 %, 70 %, 90 % and 96% (by volume)

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

E.3.3 Acetone, non-volatile residue at 80 °C maximum 0.001 %

E.4 Procedure

E.4.1 For the removal of inorganic salts, 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.

E.4.2 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.

E.4.3 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.

E.4.4 Wash the combined ether extracts and rinsing (see E.4.3) 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 pre-weighed flask and recover the solvent. Add 10-ml of acetone and evaporate off the solvent using a stream of nitrogen. 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.

E.5 Calculation

The non- detergent organic matter shall be expressed as follows:

$$\text{Non- detergent organic matter (on dry basis), \% m/m} = 100 \frac{m_2 - m_1}{m} \times \frac{100}{(100 - \text{moisture})}$$

where

m_1 is the mass, in grams, of empty flask

m_2 is the mass, in grams, of the flask with residue

m is the mass, in grams, of material taken for the test.

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Annex F (normative)

Sampling procedure for detergent paste

F.1 General requirements

F.1.1 In drawing preparing, storing and handling samples, the precautions in F.1.2 – F.1.7 shall be observed.

F.1.2 Samples shall not be taken from places exposed to damp air, dust or soot.

F.1.3 The sampling instruments shall be clean and dry when used.

F.1.4 The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

F.1.5 The samples shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

F.1.6 Each container shall be sealed airtight after filling, and marked with full details of sampling which include, date of sampling, batch or code number, name of manufacturer, and other important particulars of the consignment.

F.1.7 The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature, and that they are protected from light.

F.2 Scale of sampling

F.2.1 In a single consignment, all the packages containing non-soapy detergents of the same type and form, and drawn from the same batch of manufacture, shall constitute a lot. If the consignment consists of packages containing non-soapy detergents of different types and forms, then the packages containing non-soapy detergents of the same type form and batch of manufacture shall be grouped together, and such group shall constitute a separate lot.

F.2.2 For ascertaining the conformity of the lot to the requirements prescribed in this standard, tests shall be carried out on each lot separately. The number (n) of packages to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table F.1

Table.1 — Scale of sampling

No. of packages in the lot (N)	No. of packages to be selected (n)
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

NOTE When the size of the lot is three packages or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specifications should be as agreed on between the purchaser/inspector and the supplier.

F.2.3 The packages shall be selected at random and to ensure randomness of selection, a random number table shall be used. In case such tables are not available, the procedure given below may be adopted.

Starting from any package, count all the packages in one order as 1, 2, 3, ..., up to r and so on, where r is the integral part of N/n, (N being the lot size and n the number of packages to be selected). Every rth package thus counted shall be withdrawn to give a sample for the purposes of test.

F.3 Preparation of gross samples, test sample and reference sample

F.3.1 Gross sample

From each one of the packages selected as in F.2.2, draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for purposes of test as indicated in F.4.

The material from the containers selected in F.3.1.2 shall be mixed by thoroughly to give the gross sample for the package.

F.3.2 Test sample

F.3.2.1 Segregate carefully the gross samples of paste. From the gross representing each form of synthetic detergent take a small but equal quantity of material and mix thoroughly into a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under F.4. The composite samples representing each form and type of synthetic detergent shall be divided into three equal parts, one for the purchaser/inspector, another for the supplier, and the third for the reference.

F.3.2.2 The remaining portion of the material in each of the gross samples shall be divided into three equal pans, each forming an individual sample. One set of individual samples, representing the n selected packages shall be for the purchaser/inspector, another for the supplier, and the third for reference.

F.3.2.3 All the composite and individual samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers, and labelled with full particulars of identification given in F.1.6.

F.3.3 Reference samples

F.3.3.1 The reference samples shall consist of a composite sample and a set of individual samples. All the containers shall bear the seals of both the purchaser/inspector and the supplier, and shall be kept at a place agreed to between the two parties.

F.3.3.2 Reference samples shall be used in case of any dispute between the purchaser/inspector and the supplier.

F.4 Number of tests

F.4.1 Tests for the determination of active ingredient shall be performed on each of the individual samples.

F.4.2 Tests for the determination of other requirements specified in Table 1 shall be conducted on the composite sample.

F.5 Criteria for conformity

F.5.1 For individual samples

F.5.1.1 For the characteristic, which has been determined on the individual sample, the mean (\bar{X}) and the range (R) of test results shall be calculated as follows:

$$\text{Mean}(\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range (R) is the difference between the maximum and the minimum value of test results.

F.5.1.2 If the specification limit for the characteristic is given as a minimum, the value of the expression ($\bar{X} - KR$) shall be calculated from the relevant test results (see also F.5.1.5). If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement for the characteristic.

F.5.1.3 If the specification limit for the characteristic is given as a maximum, the value of the expression ($\bar{X} + KR$) shall be calculated from the relevant test results (see also F.5.1.5). If the value so obtained is less than or equal to the maximum limit, the lot shall be declared as conforming to the requirement for the characteristic.

F.5.1.4 If the characteristic has two-sided specification limits, then the values of the expression ($\bar{X} \pm KR$) shall be calculated from the relevant test results (see also F.5.1.5). If the value so obtained lies between the two specification limits, the lot shall be declared as conforming to the requirement for the characteristic.

F.5.1.5 The value of the factor K referred to in F.5.1.2 - F.5.1.4 shall be chosen in accordance with Table F.2, depending upon the acceptable quality level, that is, the percentage, of non-conforming packages that may be tolerated reasonably.

Table F.2 — Value of 'K' for achieving different acceptable quality levels

Acceptable quality level	Value of 'K'
Not more than 3.0 % defectives	0.4
Not more than 1.5 % defectives	0.5
Not more than 0.5 % defectives	0.6

F.5.2 For composite sample

For declaring the conformity of the lot to the requirements of all the remaining characteristics determined on the composite sample, the test results for each one of the characteristics shall satisfy the relevant requirement given in Table 1 of this standard.

Bibliography

EAS 788: 2013 *Synthetic detergent paste – Specification*