

**Household fabric softeners –
Specification**

PUBLIC REVIEW DRAFT

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Household fabric softeners — Specification

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Foreword

This Kenya Standard was developed by the Technical Committee on Surface Active Agents under the guidance of the Standards Projects Committee and it is in accordance with the procedures of the Bureau.

Household fabric softeners are stable aqueous dispersions of cationic materials used for providing fabric softening and static control benefits during laundering. Rinse-added fabric softening compositions contain, as the active component, di-hardened tallow dimethylammonium chloride and imidazolinium compounds substituted with two tallow groups. These materials are normally prepared in the form of a dispersion in water.

The dispersion of the base material contains other additives such as preservatives, colourant (dyes) and fragrances. Other fabric rinses will contain additives to prevent or control foaming, electrolyte salts such as calcium chloride and thickener materials to control the viscosity. Other ingredients added will modify the rheology and/or flow of the dispersion.

During the preparation of this standard, reference was made to the following document:

ASTM D 5237-05: Standard guide for evaluating fabric softeners.

Acknowledgement is hereby made for the assistance derived from this source.

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Household fabric softeners — Specification

1 Scope

This Kenya Standard specifies requirements and methods of test for household fabric softeners/conditioner/enhancer.

2 Normative references

This Kenya Standard incorporates by dated and undated reference, provisions from other publications. These normative references are cited at the appropriate place in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Kenya Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

KS ISO 4316: *Surface active agents — Determination of pH of aqueous solution — Potentiometric method*

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

KS EAS 217-1-1, *Methods for the microbiological examination of foods — Part 1-1: General procedures and techniques*

3 Definitions

For the purposes of this standard, the following definitions shall apply:

3.1

fabric softener

a laundry auxiliary product or laundry detergent ingredient that gives fabrics a soft feel, freshening, smooth surface, or reduces static electricity, or a combination thereof

3.2

laundering

the cleaning and restoring of textile materials to a serviceable condition using the washing and drying equipment commonly found in household

4 Requirements

4.1 General

4.1.1 The fabric softener shall have uniform dispersion with no lumps or phase separation, and shall not contain ingredients in quantities that are toxic to human beings.

4.1.2 It shall be uniform in colour with no un-dissolved or precipitated dyes that may stain the treated fabrics.

4.1.3 If the product is antibacterial, it shall pass antibacterial activity test when tested in accordance with KS EAS 794.

4.2 Odour

The product, both as received and when dissolved in water, shall possess a pleasant, fresh odour and shall not develop an objectionable one during storage at ambient temperature.

4.3 Stability

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The product shall remain stable for the declared shelf life period without breaking of the dispersion (phase separation), changes in colour and deterioration of the odour.

4.4 Dispersion

The product shall disperse effectively or easily in standard hard water (total hardness 150 ppm \pm 20 ppm) at 30 °C.

4.5 Activity

The product shall influence the properties of the fabric to improved wet-ability, softer and reduced static charge.

4.6 Specific quality requirements

The product shall comply with the specific quality requirements given in Table 1, when tested in accordance with the method prescribed therein.

Table 1 — Specific quality requirements

SI no.	Property	Requirement	Test method
i)	Cationic content, % (w/w), minimum	2.5	Annex A
ii)	pH, neat, range	2 – 7	KS ISO 4316
iii)	Total viable counts, c.f.u./g, max.	2 x 10 ²	KS EAS 217-1-1
iv)	Specific organisms	Absent	KS EAS 794

5 Packaging and labeling

5.1 Packaging

The product shall be packaged in suitable containers that are strong enough to withstand normal usage and transportation and that will prevent leaking, drying out and contamination of the product.

5.2 Labeling

Each container and each bulk package shall be legibly and indelibly marked with the following information:

- name of the product as “Fabric softener/Conditioner/Enhancer”;
- manufacturer's name and physical address;

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

- batch or lot number;
- net contents;
- antibacterial agents if used and their levels;
- instructions for use (which shall be in either English, Kiswahili or French or in combination as agreed between the manufacturer and supplier”);
- date of manufacture;
- List of ingredients
- Precautions
- best before date; and
- Country of origin.

Annex A (normative)

Determination of cationic surfactants in finished products by two-phase titration

A.1 Scope

Method A is applicable to the determination of quaternary ammonium compounds in raw materials and in finished products containing no significant amounts of long chain amines. Mixed indicator is used (acid medium).

Method B is applicable to the determination of quaternary ammonium compounds in raw materials and finished products, which may contain long chain amines. Bromocresol green indicator is used (alkaline medium).

Method C is applicable to the determination of quat and esterquat in raw material and in rinse cycle fabric softener finished products.

Anionic surfactants or anionic brighteners interfere. The above procedures are applicable, however, if the interfering substances are removed.

A.2 Method A

A.2.1 Principle

Cationic surfactants react with the disulphine blue – dimidium bromide indicator system to form a blue chloroform-soluble complex, while anionic surfactants react with the same dye system to form a pink chloroform-soluble complex. Thus, when a solution containing a cationic surfactant is titrated with a standard solution of an anionic surfactant, the chloroform layer changes from blue to pink at the endpoint.

A.2.2 Safety requirements

Chloroform is a suspected teratogen and carcinogen compound. Always wear safety glasses. Work in a fume cupboard and avoid contact with the skin. If necessary, remove from the skin with large volumes of water.

A.2.3 Reagents

A.2.3.1 0.005 N/0.004 N sodium lauryl sulphate solution (see A.4.7)

A.2.3.2 Disulphine blue, dimidium bromide indicator solution mixed indicator (see A.4.8).

A.2.3.3 0.1 N sodium hydroxide, NaOH.

A.2.3.4 Phenolphthalein indicator solution, dissolve 1 g of phenolphthalein in 100 mL of reagent alcohol. Thoroughly mixed and neutralized with alcoholic potassium hydroxide solution to a faint pink colour.

A.2.3.5 0.1 N sulphuric acid, H₂SO₄.

A.2.3.6 Chloroform, A.R.

A.2.4 Apparatus

NOTE Equivalent apparatus may be used.

A.2.4.1 Burette, 25 mL semi-micro, graduated in 0.05 mL.

A.2.4.2 Glass-stoppered, mixing cylinders, 100 mL.

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A.2.5 Procedure

A.2.5.1 Accurately weigh (to the nearest 0.1 mg) an appropriate weight of sample into a 100 mL volumetric flask. The sample size (containing 0.5 to 0.75 milliequivalents of cationic) should be such that between 10 mL and 15 mL of 0.005 N sodium lauryl sulphate will be required for the titration in A.2.5.3. It can be estimated as follows:

$$\text{Sample wt. required (in g)} = \frac{(0.0005 \text{ to } 0.00075) \times MW \times 100}{\% \text{ cationic expected}}$$

where,

MW is the molecular weight of the cationic surfactant.

NOTE If the sample does not require any dilution, weigh it directly into the mixing cylinder and follow A.2.5.2.

A.2.5.2 Depending on the nature of the sample proceed as following:

A.2.5.2.1 Dissolve the sample in the 100-mL volumetric flask with distilled water and dilute to mark with distilled water.

NOTE Normally finished products are soluble in water. If it is found that chloroform is a better solvent, use it instead of water. Make sure, however, to have 20 mL of chloroform and about 45 mL of aqueous layers in the mixing cylinder before titrating with sodium lauryl sulphate.

A.2.5.2.2 Pipette a 10-mL aliquot into a 100-mL glass stoppered mixing cylinder. Add two drops of phenolphthalein indicator solution and neutralize to a faint pink colour with 0.1 N NaOH or 0.1 N H₂SO₄ as required. Adjust the volume to 35 mL with distilled water. Add 10 mL of the mixed indicator solution and 20 mL of chloroform.

A.2.5.3 Shake the cylinder and let the layers separate. The chloroform layer should be blue. Titrate this solution with standard sodium lauryl sulphate solution. Shake the cylinder well between each addition and release the pressure. Near the endpoint the blue colour of the chloroform layer begins to fade and the endpoint is reached when the colour changes to a distinct pink. Note the volume corresponding to the endpoint.

A.2.6. Calculations

$$\% \text{ cationic} = \frac{V \times N \times (MW/1000) \times 100}{S}$$

where,

V is the volume (in mL) of the titrant (SLS) at the endpoint (step 3);

N is the normality of the titrant (SLS);

MW is the molecular weight of the cationic surfactant;

S is the weight of sample in the aliquot (A.2.5.1).

A.3 Method B

A.3.1 Principle

Cationic surfactants react with the bromocresol green indicator to form a blue chloroform-soluble complex. When a solution containing a cationic surfactant is titrated with a standard solution of an anionic surfactant, the chloroform layer changes from blue to colourless at the endpoint.

A.3.2 Safety requirements

Chloroform is a suspected teratogen and carcinogen compound. Always wear safety glasses. Work in a fume cupboard and avoid contact with the skin. If necessary, remove from the skin with large volumes of water.

A.3.3 Reagents

NOTE Equivalent reagents may be used.

A.3.3.1 0.005 N sodium lauryl sulphate solution (see A.4.7)

A.3.3.2 Buffered Na₂SO₄ Solution (pH 9.5), Prepare two litres of solution containing 140 g of anhydrous Na₂SO₄, 6.1 g of Boric Acid and 137.5 mL of exactly 0.5N NaOH solution. Dilute to mark in a 2-litre volumetric flask with distilled H₂O. Adjust the Ph to 9.5 using Boric Acid or NaOH.

A.3.3.3 Chloroform (analytical grade)

A.3.3.4 Bromocresol green (BCG) indicator solution pH 9.5, weigh 0.04 g of BCG powder into a one-litre volumetric flask. Dissolve with sufficient buffered Na₂SO₄ solution and dilute to one litre with the same buffer solution. This indicator should be kept no longer than one or two weeks since the green colour may fade after this period resulting in poor titration endpoints. In addition, do not heat the BCG to solubilize since this will also cause fading.

A.3.4 Apparatus

NOTE Equivalent apparatus may be used.

A.3.4.1 Burette, 25 mL semi-micro, graduated in 0.05 mL.

A.3.4.2 Glass-stoppered mixing cylinders, 100 mL.

A.3.5 Procedure

A.3.5.1 Accurately weigh (to the nearest 0.1 mg) an appropriate weight of sample into a 100 mL volumetric flask. The sample size (containing 0.5 to 1.75 milliequivalents of cationic) should be such that between 10 mL and 15 mL of 0.005 N sodium lauryl sulphate will be required for the titration in A.3.5.4. It can be estimated as follows:

$$\text{Sample weight required (in g)} = \frac{(0.0005 \text{ to } 0.00075) \times MW \times 100}{\% \text{ cationic expected}}$$

where,

MW is the molecular weight of the cationic surfactant.

NOTE If the sample does not require any dilution, weigh it directly into the mixing cylinder and follow A.3.5.2.

A.3.5.2 Dissolve the sample in the 100-mL volumetric flask with distilled water and dilute to mark with water.

NOTE Normally finished products are soluble in water. If it is found that chloroform is a better solvent, use it instead of water. Make sure, however, to have 20 mL of chloroform and about 45 mL of aqueous layers in the mixing cylinder before titrating with sodium lauryl sulphate.

A.3.5.3 Pipette a 10-mL aliquot into a 100-mL glass stoppered mixing cylinder. Add 20 mL of chloroform, 25 mL of bromocresol green solution (BCG) and 10 mL of water. This procedure should be run in replicate with at least two sample aliquots.

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A.3.5.4 Titrate the solution in the mixing cylinder with 0.005 N sodium lauryl sulphate solution. At the beginning of the titration, add the titrant in 1 mL and 2 mL increments and shake (release regularly the pressure). As the titration progresses, slowly add smaller increments with vigorous shaking, adding 0.05 mL increments as the endpoint is approached. Initially, the blue colour of the BCG concentrates in the Chloroform layer. Near the endpoint the blue colour gradually moves from the chloroform layer to the aqueous layer and at the endpoint the chloroform layer is colourless.

A.3.5.5 Calculations

$$\% \text{ cationic} = \frac{V \times N \times (MW/1\ 000) \times 100}{S}$$

where,

V is the volume (in mL) of titrant (SLS) at the endpoint (A.3.5.4);

N is the normality of the titrant (SLS);

MW is the molecular weight of the cationic surfactant;

S is the weight of sample in the aliquot (A.3.5.2).

A.4 Method C

A.4.1 Principle

Cationic surfactants react with the disulphine blue – dimidate bromide indicator system to form a blue chloroform-soluble complex, while anionic surfactants react with the same dye system to form a pink chloroform-soluble complex. Thus, when a solution containing a cationic surfactant is titrated with a standard solution of an anionic surfactant, the chloroform layer changes from blue to pink at the endpoint.

A.4.2 Safety requirements

Chloroform is a suspected teratogen and carcinogen compound. Always wear safety glasses. Work in a fume cupboard and avoid contact with the skin. If necessary, remove from the skin with large volumes of water.

A.4.3 Reagents

NOTE Equivalent reagents may be used.

A.4.3.1 Sodium lauryl sulphate 0.004 M solution (see A.4.7)

A.4.3.2 Mixed indicator solution (see A.4.8)

A.4.3.3 Chloroform, analytical grade.

A.4.3.4 Isopropyl alcohol (IPA), analytical grade.

A.4.4 Apparatus

NOTE Equivalent apparatus may be used.

A.4.4.1 250 mL graduated, stoppered cylinders

A.4.4.2 100 mL or 250 mL volumetric flasks

A.4.4.3 10 mL and 25 mL volumetric pipettes

A.4.4.4 Magnetic stirrers

A.4.4.5 Metrohm dosimat, with automatic burette containing the SLS solution such as metrohm 665 or 725 dosimat.

A.4.4.6 Oven, capable to heat at 60 °C.

A.4.5 Procedure

A.4.5.1 Accurately weigh (to the nearest 0.1 mg) an appropriate weight of sample into a 250-ml volumetric flask. The sample size has to be calculated so that it contains at about 0.2 milliequivalents of cationic. It can be estimated as follows:

$$\text{Sample weight required (in g)} = \frac{(0.0002) \times MW \times 100}{\% \text{ cationic expected}}$$

where,

MW is the molecular weight of the cationic surfactant.

NOTE *MW* to be issued by the manufacturer of the raw material.

A.4.5.2 Dilute to volume with distilled water. Add a magnetic stirring bar and allow stirring for 5 min.

A.4.5.3 Pipette accurately 25 mL of the sample solution into a 250-ml graduated, stoppered cylinder. Add 10 mL of water, 15 mL of chloroform and 10 mL of mixed indicator solution.

A.4.5.4 Titrate the solution with sodium lauryl sulphate solution, shaking well between each addition, until the lower chloroform layer changes in colour from blue to the grey end point. Note the volume of titrant required to reach the endpoint.

NOTE One drop of excess titrant should produce pink colouration.

A.4.6 Calculations

$$\text{Meq/g cationic surfactant} = \frac{V \times M \times V_2}{W \times V_1}$$

where,

W is the weight (in g) of sample (A.4.5.1);

V is the titration volume of the end point in mL (A.4.5.4);

M is the molarity of the sodium lauryl sulphate solution;

V₁ is the volume (in mL) of the aliquot (A.4.5.3).

$$\text{Cationic surfactant (\%)} = \frac{\text{meq cationic surfactant} \times MW}{10}$$

where,

MW is the molecular weight of the cationic surfactant.

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A.4.7 Preparation of the 0.004 N/0.005 N SLS standard solution

A.4.7.1 Reagents

A.4.7.1.1 Sodium lauryl (or dodecyl) sulphate (minimum 99 % purity)

A.4.7.1.2 Water

A.4.7.1.3 Formaldehyde solution (37 % w/w), analytical grade.

A.4.7.2 Safety requirements

Formaldehyde used in this procedure is toxic, and contact with the skin should be avoided. Safety glasses and disposable gloves should be worn when handling these reagents and during sample preparation. Reagent disposal should be carried out in accordance with safety and environmental regulations.

A.4.7.3 Procedure

A.4.7.3.1 Dry about 5 g of sodium lauryl sulphate in a 60 °C vacuum oven overnight and place in a desiccator to cool to ambient temperature.

A.4.7.3.2 **0.005 N solution**, this solution is considered to be stable for 1 month after preparation.

A.4.7.3.2.1 Accurately weigh 1.44 g (to the nearest 0.1 mg) of the dried sodium lauryl sulphate and transfer to a 600-mL beaker.

A.4.7.3.2.2 Add 300 mL of water and stir to dissolve the sample.

A.4.7.3.2.3 Quantitatively transfer the solution from 600-mL to a 1 000-mL volumetric flask and dilute to volume with water at room temperature.

A.4.7.3.3 **0.004 M solution**, this solution is considered to be stable for 1 month after preparation.

A.4.7.3.3.1 Accurately weigh 1.15 g (to the nearest 0.1 mg) of the dried sodium lauryl sulphate and transfer to a 250-mL beaker.

A.4.7.3.3.2 Add 100 mL of water and stir to dissolve the sample.

A.4.7.3.3.3 Add 1 mL of formaldehyde solution.

A.4.7.3.3.4 Quantitatively transfer the solution from a 600-mL to a 1 000-mL volumetric flask and dilute to volume with water at room temperature.

A.4.7 Calculations

$$\text{Normality or molarity} = \frac{\text{Sodium lauryl sulphate in g} \times \text{purity}}{MW \times 100}$$

where,

MW is the molecular weight of sodium lauryl sulphate normally 288.38.

Purity is the stated % of sodium lauryl sulphate. A certificate of analysis from the supplier will give the stated purity.

A.4.8 Preparation of the mixed indicator solution

A.4.8.1 Principle

A stock indicator solution of dimidium bromide, a pink dye, and disulphine blue VN 150, a blue dye, is diluted to prepare a working indicator solution. The working indicator solution is used, as the indicator, in volumetric two-phase titration procedures to measure cationic and/or anionic surfactants.

A.4.8.2 Safety requirements

CAUTION: disulphine blue is a cancer suspect agent, handle carefully. Avoid inhalation of dust and contact with skin. Dispose off properly.

A.4.8.3 Reagents

A.4.8.3.1 Dimidium bromide

A.4.8.3.2 Disulphine blue VN 150

A.4.8.3.3 Stock solution (dimidium bromide/disulphine blue VN 150), can be purchased and used instead of preparing it from the individual dyes.

A.4.8.3.4 Aqueous 10 % (v/v) alcohol

A.4.8.3.5 Aqueous 15 % (v/v) sulphuric acid

A.4.8.4 Procedure

A.4.8.4.1 Stock indicator solution preparation

- Weigh $0.5 \text{ g} \pm 0.005 \text{ g}$ of dimidium bromide into a 100-mL beaker and dissolve in 20 mL to 30 mL of hot 10 % alcohol.
- Weigh $0.25 \text{ g} \pm 0.005 \text{ g}$ of disulphine blue VN 150 into a second 100-mL beaker and dissolve in 20 mL to 30 mL of hot 10 % alcohol.
- Quantitatively transfer both solutions into a 250-mL volumetric flask and dilute to volume with 10 % alcohol. (Keep stock solution in amber coloured bottle. After two years this stock solution should be discarded.)

A.4.8.4.2 Working indicator solution preparation

- Add approximately 200 mL of distilled water to a 500-mL volumetric flask followed by 20 mL of the prepared or purchased stock indicator solution.
- Add 20 mL of 15 % sulphuric acid to the flask mix well and dilute to volume with distilled water. This diluted indicator solution is used in the titrations and if stored in an amber coloured bottle will keep for at least six months.