

DKS 2117:2013

DRAFT KENYA STANDARD

Acidic detergents for 'Cleaning-in-place' in food and beverage industry – Specification

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First Edition 2013

JULY 2013

PUBLIC REVIEW DRAFT

DKS 2117:2013

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Acidic detergents for 'Cleaning-in-place' in food and beverage industry – 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.

This standard is one of the standards dealing with detergents and sanitizers used in food and beverage industry. The standard specifies assessments for available acidity, corrosion, solubility and foaming characteristics in acidic detergents. These detergents are usually mixtures of mineral and organic acids, surface active agents and inhibitors. Acids commonly used are phosphoric, sulphamic, nitric, citric and acetic.

In the in food and beverage industry, acidic detergents are used to dissolve soils composed of water-insoluble salts of calcium, magnesium and iron. They are also used to degrade, modify or dissolve protein soils that have been chemically changed by the action of heat or calcium ions or both.

Certain acidic detergents may be used as rinse aids, the purpose being to prevent the deposition of hard water salts. As far as this standard is concerned, the test requirements relate to detergents for use in acid cleaning applications. Rinse aids as such are not covered.

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

AS NZS 1389:1997 Acidic detergents for use in the dairying industry

Acknowledgement is hereby made for assistance derived from this source.

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Acidic detergents for 'Cleaning-in-place' in food and beverage industry – Specification

1 Scope

This standard specifies requirements for acidic detergents used for cleaning-in-place in food and beverage industry.

Note: These detergents may be corrosive to metals other than stainless steel and could also adversely affect plastics.

2 Application

Acidic detergents may be used for the following cleaning operations:

- a) Removal of "milk stone" of the type formed during the cold treatment or hot processing of milk.

Note: Under these conditions, acidic detergents may be used in a sequence cleaning operation with alkaline detergents.

- b) Removal of rust and water scale.

- c) General cleaning.

Note: The information supplied in accordance with Clause 12 below should provide the essential instructions in respect to the particular application.

3 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 ISO 4314: *Surface active agents - Determination of free alkalinity or free acidity - Titrimetric method*

KS 92, part 3: *Synthetic detergent powders — Specification, Part 3: Determination of biodegradability of surfactants — Test method*

4. Definitions

4.1 Soil

Any residue, scale and other deposits to be removed from the food and beverage contact surface during the cleaning process.

4.2 Use dilution

The minimum concentration of the detergent, in water, that the manufacturer has recommended for acid cleaning.

3.4 Cleaning-in-place (CIP) — a method for cleaning equipment, using no direct mechanical aid to remove the soil but depending on solution flows, temperature and the properties of the detergent solution.

4.3 Use temperature

The temperature that the supplier/manufacturer has recommended for a specific application. Cold is between 1 °C and 35 °C, warm between 36 °C and 59 °C, and hot is 60 °C and above.

5 Requirements

5.1 General requirements

5.1.1 The product shall be a liquid or powder.

5.1.2 The product shall not contain any perfume or fragrant odorizer.

5.1.3 Any dyes used shall be approved food colouring substances.

5.1.4 When used in accordance with the manufacturer's/supplier's instructions, the product shall not impart any flavour to, or taint, food and beverage products.

5.1.5 The product shall be free from foreign matter.

5.1.6 When used in accordance with the manufacturer's/supplier's instructions the product shall not cause any residues harmful to humans or to the quality of food and beverage products.

5.1.7 The raw materials used in the manufacture of the product shall be biodegradable when tested against KS 92 part 3.

5.2 Specific quality requirements

The product shall comply with the specific quality requirements in Table 1.

Table 1. Specific quality requirements for cleaning-in-place acidic detergents

Sl. No.	Property	Requirement	Test Method
1)	Acidity, %, (m/m), minimum	7.0	Annex D
2)	Appearance	Colourless clear liquid or Off white, free flowing granular powder	Visual
3)	Solubility in water	Completely soluble	Visual

5.2 Available acid content

The available acid content of the detergent shall be such that, when 400 ml of the use dilution is used to digest 0.70 g of calcium carbonate in the manner described in Annex A, the pH of the resultant solution shall be not greater than 3.5.

5.3 Corrosion test

The corrosive effect of the detergent shall be such that test panels of stainless steel (316), when immersed in the manner described in Annex B for 3 days in the use dilution of the detergent at the maximum use temperature recommended by the manufacturer, will show no visible evidence of corrosion nor any discolouration.

5.4 Degree of foaming

The degree of foaming of the use dilution shall be expressed as either 'non', 'low', 'medium' or 'high' at the recommended working temperature. The degree of foaming shall be determined in the manner described in Annex C.

5.5 Freedom from grit

When a solution of the product is prepared at twice the concentration of the use dilution, using not less than 5 g of the product, any residue in the solution shall be free from grit.

Note: The requirement for this test is not applicable to those products designed and labeled as abrasive acidic detergents.

6 Packaging and marking

6.1 Packaging

The product shall be so packed as to prevent excessive drying out, leakage, or contamination.

The product shall be packed in containers that are strong enough to withstand normal usage and transportation.

6.2 Marking

Each package of detergent shall be legibly and indelibly marked with the following information:

- i) name of the product, e.g. 'acidic detergent' or 'abrasive acidic detergent'
- ii) manufacturer's name and physical address;

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

- iii) The net contents
- iv) Batch number or code number
- v) Storage instructions
- vi) Date of manufacture
- vii) Best before date
- viii) List of ingredients
- ix) Precautions
- x) Adequate and accurate directions for the application and use of the product.
- xi) Country of origin

10.2 The following information shall be provided to the consumer

- i) Adequate and accurate directions for the application and use of the product
- ii) List of ingredients
- iii) Degree of foaming
- iv) Precautions

Annex A (Normative)

Method of assessing available acid content

A.1 Principle

A known volume of specified detergent solution is used to digest completely a fixed quantity of calcium carbonate. The final pH of the solution is noted.

A.2 Apparatus and reagents

The following are required:

- a) Normal laboratory apparatus.
- b) Analytical grade calcium carbonate, anhydrous CaCO_3 .

A.3 Test solution

Using a representation sample of the detergent, prepare a 2000 ml sample stock at the use dilution, by using distilled water.

A.4 Procedure

The procedure shall be as follows:

- a) To a 600 ml beaker, add 0.70 ± 0.01 g of calcium carbonate, accurately weighed.
- b) To this, cautiously add 400 ± 2 ml of the sample stock solution at $20 \pm 5^\circ\text{C}$.

Note: Care should be taken to ensure that effervescence is minimized.

- c) Allow the reaction to proceed to completion. Stir the solution vigorously until all the calcium carbonate has dissolved, or until the effervescence has ceased.
- d) Determine the pH of the solution and note the result.
- e) Repeat steps (a) to (d). The difference in pH between replicate tests should not exceed 0.2.

5 Report

The report shall contain the following information:

- a) The pH of the solution.
- b) The use dilution.

Annex B (Normative)

Corrosion test

B.1 Apparatus and materials

The following special apparatus and materials are required:

- a) Squat and tall 1-L beakers of Pyrex glass and watch glasses to cover beakers.
- b) A water bath with a close-fitting lid capable of maintain the test temperature (see paragraph B.3 (b)) within $\pm 1^\circ\text{C}$.
- c) An oven capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$.
- d) Test panels made of stainless steel (316) of approximate dimension 125 x 63 x 1.5 mm. The panels shall have a '2B' finish on both faces. They shall be undamaged and unmarked, flat and their edges free from burrs.
- e) Stainless steel tongs for handling the panels.
- f) Panel holders made of inert material such as polypropylene for use during pre-cleaning and drying operations.
- g) Rubber bands of rectangular cross-section, measuring, when lying flat and unstretched, approximately 6 x 80 mm.
- h) Magnesium carbonate, LR grade, for use as an abrasive for cleaning the panels.
- i) Distilled water complying.

B.2 Pre-cleaning of test panels and rubber bands

B.2.1 Panels

The pre-cleaning procedure for panels shall be as follows:

- a) Swab the test panels, two for each test, with cotton wool using a warm 1% *m/V* solution of a general purpose dairy detergent.
- b) Scour the panels with cotton wool using water as lubricant and the magnesium carbonate as an abrasive.

Note: The scouring is to remove any film that is produced by reaction between the detergent and the abrasive, e.g. magnesium silicate.

- c) Without delay, thoroughly rinse the panels under hot tap water.
- d) Rinse the panels in boiling distilled water immersing each panel in turn in water contained in three 1 L beakers.
- e) Dry in a oven at $105 \pm 2^\circ\text{C}$.
- f) Allow to cool in a dry, dust-free position.

B.2.2 Rubber bands

The pre-cleaning procedure for rubber bands shall be as follows:

- a) Place the rubber bands in a hard-boiling 1 % *m/V* solution of general purpose dairy detergent.
- b) Rinse under hot tap water.
- c) Then rinse in distilled water and allow to dry.

B.3 Procedure

The procedure shall be as follows, carried out in duplicate:

- a) In a tall 1 L beaker, make up 950 ml of the use dilution of the detergent under test. Mark the level of the solution on the side of the beaker.
- b) Heat the solution to the maximum use temperature recommended by the manufacturer of the detergent. Place the beaker of solution in a water bath controlled at the maximum use temperature $\pm 1^{\circ}\text{C}$.
- c) Place a rubber band around the test panel in the direction of the panel's long axis, ensuring that the band is flat against both sides of the panel and that the panel is not touched by the fingers.
- d) Five minutes after placement of the band, immerse the panel on its end in the test solution so that there is at least 10 mm of solution above the panel. Note the time.
- e) Leave the panel in the test solution for 72 h. Each morning and evening, top up the solution, to the mark, with distilled water.
- f) At the end of the 72 h, remove the panel from the test solution and remove the rubber bands and rinse under hot, running tap water.
- g) Rinse three times as prescribed in paragraph B2.1 (c) above.
- h) Finally, dry the panel in the oven at 105°C . Allow to cool.
- i) Examine the panel for —
 - i) Evidence of corrosion (see note below); or
 - ii) Discoloration.

Note: Pitting is most likely to occur where the rubber band contacts the edges of the panel.

B.4 Interpretation of results

If the duplicate panels from the test detergent show the same characteristics, record the result. If the duplicates differ, repeat the test using fresh panels.

B.5 Report

The report shall contain the following information:

- a) Whether corrosion or discoloration of the panels has occurred.

Annex C

Determination of degree of foaming

(Normative)

C.1 Apparatus

The following is required:

- a) A 1000 ml one-mark volumetric flask.
- b) A stoppered 250 ml measuring cylinder.
- c) A thermostatically-controlled water bath capable of maintaining the test temperature (see paragraph C3 (c)) within ± 0.5 °C.

C.2 Test solution

Prepare 1000 ml of stock test solution by taking the required representative quantity of the detergent under test and making a use dilution with distilled water.

C.3 Procedure

The procedure shall be as follows:

- a) Rinse a clean 250 ml stoppered measuring cylinder with a suitable volume of the test solution. Allow the surplus to drain.

Note: Cylinders should be thoroughly cleansed before commencing each test.

- b) Transfer a 100 ml aliquot of test solution to the 250 ml cylinder, with a minimum of agitation.
- c) Using the water bath, adjust the temperature of the measuring cylinder and its contents to the minimum temperature recommended by the manufacturer of the detergent.

Note: During step (c) the entire length of the cylinder should be immersed in an enclosed water bath.

- d) Stopper the cylinder, and holding it vertically, shake it vigorously 10 times through a throw of approximately 30 cm in the vertical plane, the whole action taking approximately 3 s.

- e) Sixty seconds after the cessation of shaking, determine the volume of foam, recording this to the nearest milliliter.

- f) Repeat steps (a) to (e) using another 100 ml aliquot of test solution, and keep repeating until a variation between two results is obtained that is within the limit prescribed in paragraph C4 below. Record this mean volume of foam.

C.4 Expression of results

The degree of foaming shall be expressed as 'non', 'low', 'medium', or 'high' in accordance with the following:

Measured volume of foam mL	Degree of foaming	Maximum acceptable difference between two results mL
0 – 5	Non	2
6 – 30	Low	2
31 – 100	Medium	10
> 100	High	20

c.5 Report

The report shall contain the following information:

- a) The degree of foaming, i.e. 'non-foaming', 'low foaming', 'medium foaming' or 'high foaming'.
- b) The concentration of the test solution, i.e. the use dilution.
- c) The temperature at which the test was conducted.

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Annex D
(normative)

DETERMINATION OF ACIDITY

Accurately weigh 10g of sample in a 250-mL Erlenmeyer flask and add 25 mL of distilled water. Add approximately 15 ml of 5 percent aqueous calcium acetate solution and warm contents to 50 °C. Cool and filter into a beaker.

Carefully rinse the Erlenmeyer flask precipitate with 3 to 4 aliquots of distilled water collecting all rinsing with the filtrate. Titrate the filtrate with standard 1 mol/L NaOH solution.

Calculate as follows

$$\text{Percent acidity} = \frac{\text{concentration of NaOH } \left(\frac{\text{mol}}{\text{L}}\right) \times \text{mL of NaOH} \times C}{\text{mass of sample}}$$

Where,

C = 6.3 for Nitric acid

or

C = 3.132 for H₃PO₄

Or

C = 3.6 for HCl

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