DRAFT TANZANIA STANDARD

Calcium hypochlorite used for disinfection of water intended for human consumption – Specification

TANZANIA BUREAU OF STANDARDS

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Foreword

This Draft Tanzania Standard is being developed by the Water Quality Technical Committee under supervision of the Chemical Division Standards Committee and it is in accordance with the procedures of the Bureau.

This Tanzania Standard has been prepared with assistance drawn from:

BS EN 900:1999, *Chemicals used for treatment of water intended for human consumption – Calcium hypochlorite*, published by the European Community (EC);

SABS 296:1998, *Specification for calcium hypochlorite*, published by the South African Bureau of Standards;

KS 1290(Part 4): 2007, *Chemicals used for treatment of water intended for human consumption – Part 4: Calcium Hypochlorite*, published by the Kenya Bureau of Standards.

In reporting the result of a test or analysis made in accordance with this Tanzania Standard, if the final value, calculated or observed is to be rounded off, it shall be done in accordance with TZS 4 (see clause 2).

DRAFT TANZANIA STANDARD **CDC 6(121) CD2**

Calcium hypochlorite used for disinfection of water intended for human consumption – Specification

1 Scope

This Draft Tanzania Standard specifies requirements, sampling and tests method for calcium hypochlorite used in disinfection of water intended for human consumption.

2 Normative references

The following referenced documents are indispensable for the application of this document. The latest edition of the referenced document (including any amendments) applies;

TZS 59 / ISO 3696 Water *for analytical laboratory use – Specification and test methods*

TZS 4 *Rounding off numerical values*

3 Terms and definitions

3.1. water intended for human consumption
all water either in its original state or after treatment, intended for drinking, cooking, food preparation or other domestic purposes, regardless of its origin and whether it is supplied from a distribution network, from a tanker, or in bottles or containers

4 Requirements

4.1 General requirements

4.1.1 Descriptions

4.1.1.1 *Chemical name*; Calcium hypochlorite

4.1.1.2 Relative *molecular mass*; 142.99

4.1.1.3 Empirical *formula*; $Ca(OCl)_2$.

4.1.1.4 Chemical formula; $Ca(OCl)_2$ or $CaCl_2O_2$

4.1.2 Physical properties

4.1.2.1 Calcium hypochlorite shall be in the form of white or yellowish-white free-flowing granules, powder or tablets.

4.1.2.2 Calcium hypochlorite granular powder or granules shall be substantially free from lumps. Not more than 10% of the powder shall pass a 100-mesh µm screen. It shall not contain any dirt or other foreign material.

4.1.2.3 Calcium hypochlorite tablets shall be uniform in shape. The weight of the tablets shall not vary by more than 5% from the average value stated on the label. Not more than 2% of the tablets shall be broken.

4.1.3 Calcium hypochlorite shall contain no soluble mineral or organic substances in a quantity that would be deleterious or injurious to anyone consuming water disinfected with acceptable quantities of the hypochlorite.

4.2 Specific requirements

4.2.1 The material, when tested according to the methods prescribed in Annexes A, B, and C shall comply with the specific requirements given in Table 1.

Table 1 Specific requirements for Calcium hypochlorite

S/N	Characteristic	Requirement	Method of test
i.	Available chlorine content, %, (m/m), min	65	annex A
ii.	Sodium chloride content %, (m/m), *max*	18	annex B
iii.	Water Insoluble matter, % (m/m), *max*	4	annex C

4.2.2 Toxic substances

The content of toxic substances shall comply with the requirements specified in Table 2.

Table 2 — Toxic substances

Element	Maximum limit mg/kg of product
Arsenic (As)	10
Cadmium (Cd)	1
Chromium (Cr)	6
Mercury (Hg)	0.02
Nickel (Ni)	3
Lead (Pb)	4
Antimony (Sb)	5
Selenium (Se)	1

NOTE For the purpose of this standard, “toxic substances” are those defined in the EU Directive 80/778/EEC of 15 July, 1980 (*see* [1])

[1)] Chemical Abstracts Service Registry Number

5 Packing and marking

5.1 Packing

Calcium hypochlorite shall be packed in a suitable opaque airtight container that withstand normal handling and transportation and that will prevent leakage, contamination and loss of percentage purity of the product.

In order that purity of the product is not affected, the means of delivery shall not have been used previously for any different product or it shall have been specially cleaned and prepared before use.

5.2 Marking

The following information shall be marked legibly and indelibly on the container at minimum;

a) name and formula of the product as 'CALCIUM HYPOCHLORITE' ($Ca(OCl)_2$ or $CaCl_2O_2$)
b) the name and address of manufacturer.
c) net content.
d) batch or code number.
e) nominal available chlorine content.
f) dates of manufacture and best before.
g) instruction for use and Handling.
h) first aid instructions.
i) the words 'STORE IN COOL PLACE AWAY FROM DIRECT SUNLIGHT'
j) hazard warning in symbol or words

5 Sampling

Sampling for Calcium hypochlorite shall be done as per Annex D

6 Testing

Methods of tests for Calcium hypochlorite shall be as prescribed in the annexes.

Annex A
(normative)
Determination of available chlorine and calcium hypochlorite content

A.1 General

This method applies to products with available chlorine contents within the range of 40% (m/m) to 70% (m/m).

A.2 Principle

Calcium hypochlorite reacts with potassium iodide to release iodine in the presence of acetic acid. The iodine is titrated with sodium thiosulphate standard volumetric solution in the presence of starch indicator solution.

A.3 Reagents

All reagents shall be of analytical grade and the water used shall be distilled water.

A.3.1 Potassium iodide crystals (KI)

A.3.2 Glacial acetic acid

A.3.3 Sodium thiosulphate standard volumetric solution, c ($Na_2S_2O_3.5H_2O$) = 0.1 mol/L

A.3.3.1 Dissolve 24.8 g of $Na_2S_2O_3.5H_2O$ in water (as per TZS 59). Add 0.5 mL of chloroform as preservative, dilute to volume with water in a 1000 mL one-mark volumetric flask and mix thoroughly.

A.3.3.2 To standardize; weigh, to the nearest 0.1, mg (160 ± 10) mg (m) of primary standard potassium dichromate into a tarred glass beaker. Place the contents of the beaker in a 500 mL stoppered conical flask, add 100 mL of water and (2 ± 0.5) g of potassium iodide and stir to dissolve. Add (15 ± 1) mL of hydrochloric acid solution (diluted 1:1 by volume), swirl and allow to stand for 5 min. Titrate with the sodium thiosulphate solution until the solution is pale yellow. Add (5 ± 1) mL of starch solution and titrate to the end point, i.e. to the disappearance of the blue-black colour. Record the volume (V) used.

A.3.3.3 The concentration, C, of the sodium thiosulphate standard volumetric solution ($Na_2S_2O_3.5H_2O$), expressed in moles per litre is given by the following equation:

$$C = \frac{m}{V \; X \; 49.0317}$$

where

m is the mass, in milligrams, of potassium dichromate ($K_2Cr_2O_7$) weighed;

V is the volume, in millilitres, of the sodium thiosulphate standard volumetric solution used.

A.3.4 Starch solution, 1% (m/m)

Make a slurry with (1 ± 0.1) g starch and (5 ± 1) mL water. Add (90 ± 5) mL boiling water to the slurry. Stir to dissolve it and cool the solution. This solution needs refrigeration to avoid the decomposition of the starch, which results in a vague end point. Keep the solution cool and use it within one week.

NOTE – Commercial indicators for iodine titration exist and can be used in place of the described starch solution provided that their efficiency has been previously tested.

A.4 Apparatus

Ordinary laboratory apparatus glassware, together with the following: Laboratory sonic vibrator.

A.5 Procedure

A.5.1 Test portion

Weigh to the nearest 0.1 mg, 3.5 g of the laboratory sample (m_1) into a tarred stoppered weighing bottle.

A.5.2 Determination

Transfer the test portion to a 500 mL volumetric flask with 300 mL of water, stopper and place in the sonic vibrator for 10 min, swirling it occasionally until the test portion is in solution. Dilute to the mark with water.

Place a magnetic stirring bar into the volumetric flask and begin mixing. Transfer 25 mL, while the test portion is being stirred and without allowing any insoluble matter to settle out, into the 500 ml conical flask.

Add 100 mL of water (as per TZS 59) and 2 g of potassium iodide and mix to dissolve. Add 8 mL of glacial acetic acid, stir and titrate immediately with the sodium thiosulphate standard volumetric solution to a light yellow colour. Add 3 mL of the starch solution and continue titration to the disappearance of the blue-black colour. Record the volume V_1, of the sodium thiosulphate standard volumetric solution used.

A.6 Expression of results

The chlorine (Cl_2) content, C_1, expressed as a percentage by mass, is given by the following equation:

$$C1 = \frac{V1\ X\ C\ X\ 35.453\ X\ 20\ X\ 100}{m_1}$$

where

V_1 is the volume, in millilitres, of the sodium thiosulphate solution used for the titration;

C is the concentration, in moles per litre, of the sodium thiosulphate standard volumetric solution;

m_1 is the mass, in milligrams, of the test portion;

35.453 is the mass in milligrams of chlorine (Cl_2) corresponding to 1,00 mL of sodium thiosulphate solution c ($Na_2S_20_3.5H_20$) = 1.000 mol/L.

The Ca $(ClO)_2$ content, C_2, expressed as a percentage by mass of product, is given by the following equation:

$$C_2 = \frac{C_1 \, x \, 3.5746}{3.5453}$$

Annex B
(normative)
Determination of sodium chloride

B.1 General

This method applies to products with sodium chloride contents in the range 15% (m/m) to 25% (m/m).

B.2 Principle

Calcium hypochlorite is acidified, digested to remove all traces of available chlorine, and diluted to a known volume. Sodium is analyzed in solution by atomic absorption spectrometry.

B.3 Reagents

All reagents shall be of analytical grade and the water used shall be distilled water.

B.3.1 Hydrochloric acid, concentrated, HCl

B.3.2 Sodium standard solution (1000 µg/mL)

Weigh 2.5421 g of sodium chloride that has been previously dried for 1 h at 110°C. Dissolve in water and dilute to 1 l. Mix well.

B.3.3 Commercial iodide starch test paper

NOTE – Provided that its efficiency has been previously tested.

B.4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

B.4.1 Atomic absorption spectrometer equipped with sodium hollow cathode lamp

B.4.2 Hot plates

B.4.3 Laboratory sonic vibrator

B.5 Procedure

B.5.1 Test portion

Weigh, to the nearest 0.1 mg, 3.5 g of the laboratory sample (m_3) into a tarred stoppered weighing bottle.

B.5.2 Determination

Transfer the test portion to a 500 mL volumetric flask with 300 mL of water, stopper and place in the sonic vibrator for 10 min, swirling occasionally until the sample is in solution. Dilute to the mark with water.

From the prepared solution, transfer accurately 25 mL to a 150 mL beaker. Add 10 mL of the concentrated hydrochloric acid and place on a hot plate.

Boil to reduce the volume to 25 mL or, until the salt begins to precipitate, rinse the side of the beaker with water and test for available chlorine using the iodide-starch test paper. If chlorine is still present add 50 mL of water and reduce the volume again to 25 mL. Rinse the side of the beaker with water and test for available chlorine.

Repeat the procedure until available chlorine is not present, then cover the beaker and digest for 1 min, allow cooling then transferring to a 100 mL volumetric flask and diluting to the mark with water.

Measure the absorbance of the solution on the Atomic Absorption Spectrometer (AAS) with sodium hollow cathode lamp at a wavelength of 330.2 nm with slit setting at “4” using an air-acetylene flame.

B.5.3 Calibration

Transfer accurately from the sodium standard solution 1.0 mL, 2.0 mL, 3.0 mL, 4.0 L, 5.0 mL, 10.0 ml and 20.0 ml portions to a series of 100 ml volumetric flasks, dilute each to the mark with water and mix well. Prepare a calibration blank.

Measure the absorbance of each calibration solution as described in B.5.2 and prepare a calibration graph.

B.6 Expression of results

The sodium chloride content, (C_4), expressed as a percentage by mass of product, is given by the following equation:

$$C_4 = \frac{C\ x\ 100\ x\ 20\ x\ 2.5421}{m_3\ x\ 10\ 000}$$

where

m_3 is the mass in grams, of the test portion;

C is the concentration, in micrograms per millilitre, of sodium in the test portion as determined from the calibration graph.

Annex C
(normative)
Determination of water-insoluble matter

C.1 Principle

A representative sample of calcium hypochlorite is dissolved in water. The insoluble matter is separated by filtration then dried and weighed.

C.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

C.2.1 Sintered glass crucible of porosity P40 (pores size between 16 µm to 40 µm).

C.2.2 Oven capable of being controlled at (105 ± 3) °C.

C.3 Procedure

Weigh approximately 10 g of the representative sample (m_4) to the nearest ± 0.01 g and dissolve in 1000 ml of water by stirring for 30 min. Then filter the solution under vacuum through a dried and weighed glass filter. After the filtration, wash the residue with 20 ml of water and remove excess water by filtering under vacuum. Dry the residue at (105 ± 3)°C in the oven until the mass remains constant and weigh it (m_5) after cooling in a dessicator.

C.4 Expression of results

The insoluble matter expressed as percentage by mass (% (m/m)), C_5 in the product is given by the following equation:

$$C_5 = \frac{m_5}{m_4} X\ 100$$

where

m_5 is the mass, in grams, of the residue;
m_4 is the mass, in grams, of the test portion.

Annex D
(Normative)

Sampling

D.1 Sampling.

The following sampling procedure shall be applied in determining whether a lot complies with the relevant requirements of the specification. The samples so drawn shall be deemed to represent the lot for the respective properties.

D-2 From the lot take at random at least four containers and, after inspecting each of them, use one container for testing for compliance with the requirement for shelf life. After thoroughly mixing the contents of each of the remaining containers, by taking an approximately equal quantity of material from each container, and immediately placing it in one clean, dry, air-tight glass or plastic container obtain enough material to provide a composite sample of total mass at least 1.5 kg.

D-3 Clearly mark the name of the product, the manufacturer's name or trademark, the batch identification and the date of sampling on the sample container.

D-4 Compliance with the specification

The lot shall be deemed to comply with the requirements of the specification if, after inspection and testing of the samples taken in accordance with D.1, no defective is found.

D-5 Inspection
Inspect the containers taken in accordance with D.1 for compliance with the requirements in clause 4.

Annex E
(Informative)

Labelling – Transportation – Marking

E.1 Risk and safety labelling

The following labelling requirements applies to calcium hypochlorite:

a) Symbols indications of danger.

O: Oxidizing
C: Corrosive

b) Nature of special risks attributed to dangerous substances

R 8: Contact with combustible material may cause fire
R 31: Contact with acids liberates toxic gas
R 34: Causes burns

c) Safety advice concerning dangerous substances

S 2: Keep out of children
S 26: In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
S 43: In case of fire, use air-independent respiratory equipment for fighting

E.2 Transportation regulations and labelling

Calcium hypochlorite is listed as;

. UN number 1748[2)] for granular, calcium hypochlorite mixture, dry
. UN number 1748[2] for tablets, calcium hypochlorite, dry

NOTE – The above products contain less than 5.5% (m/m) water and more than 39 % (m/m) available chlorine.

RID[3]: class 5.1
ADR[4]; class 5.1
IMDG[5]: class 5.1
IATA[6]: class5.1

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[2] United Nations Number
[3] Regulations concerning carriage of dangerous goods by rail
[4] European agreement concerning the international carriage of dangerous goods by road
[5] International Maritime Transport of Dangerous Goods
[6] International Air Transport Association