

## **DRAFT ZANZIBAR NATIONAL STANDARD**

### **Food grade citric acid— specification**

DRAFT FOR STAKEHOLDER'S COMMENT

**ZANZIBAR BUREAU OF STANDARDS**

## Foreword

This Draft Zanzibar National Standard has been prepared by Food Standard Technical committee (TCFA1). In accordance with the Zanzibar Bureau of Standards general procedures, this draft is here by presented to the public in order to receive any technical comment concerns.

## Technical Committee Representatives

This draft Zanzibar National Standard was prepared by Food Technical Standard committee which consists of representatives from the following organizations:

Chief Government Chemist Laboratory Agency (CGCLA)  
Ministry of Agriculture, Irrigation, Natural Resources and Livestock (MANRL)  
Ministry of Health Zanzibar (MoH)  
Said Salim Bakhresa & Company LTD  
The State University of Zanzibar (SUZA)  
Umoja wa Wazalishaji Wadogo Wadogo Zanzibar (UWAZI)  
Zanzibar Food and Drug Agency (ZFDA)  
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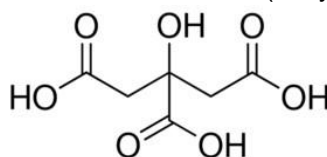
## Introduction

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. This standard would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances are added to the processed foods.

Citric acid is used as an antioxidant, synergist, sequestrant acidulant and flavourings agent.

Chemical names and formula — Citric acid is also known as 2-hydroxy-1,2,3-propanetricarboxylic acid; beta-hydroxytricarboxylic acid. Its empirical formulae, molecular weight and structural formulae are given below:

Structural formula of citric acid (anhydrous) is:

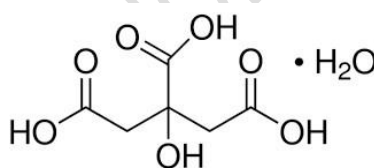


Anhydrous form

Empirical formula –  $C_6H_8O_7$

Molecular weight – 192.13

Structural formula of citric acid (monohydrate) is:



Monohydrate form

Empirical formula –  $C_6H_8O_7 \cdot H_2O$

Molecular weight – 210.15

# Food grade citric acid— specification

## 1 Scope

This draft Zanzibar national standard specifies requirements, method of sampling and test for food grade citric acid.

## 2 Normative references

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

ISO 2483, *Sodium chloride for industrial use — Determination of the loss of mass at 110 degrees C*

ISO 2480, *Sodium chloride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method*

ISO 2482, *Sodium chloride for industrial use — Determination of calcium and magnesium contents — EDTA complexometric methods*

AOAC 2013.06, *Arsenic, Cadmium, Mercury, and Lead in Foods*

EAS 39, *Hygiene in the food and drink manufacturing industry — Code of practice*

ZNS 61, *Packaging and labelling of food*

## 3 Terms and definitions

For the purposes of this standard, the following terms and definitions shall apply.

### 3.1

#### **citric acid**

product produced by recovery from sources such as lemon or pineapple juice or fermentation of carbohydrate solutions or other suitable media using *Candida spp.* or non- toxicogenic strains of *Aspergillus niger*

### 3.2

#### **food grade packaging material**

any material, made of substances that are safe and suitable for their intended use and which will not impart any toxic substance or undesirable odour or flavour to the product

## 4 Requirements

### 4.1 General requirements

Food grade citric acid shall be

- a) in the form of white or colourless, practically odourless crystals or crystalline powder; and
- b) soluble in water, freely soluble in ethanol and slightly soluble in ether.

### 4.2 Specific requirements

Food grade citric acids shall conform to the chemical requirements in Table 1 when tested in accordance with the methods prescribed therein.

Table 1: Specific requirements for food grade Citric Acid

SN	Characteristic		Requirement	Test method
i.	Purity, as $C_6H_8O_7$ , percent by mass, (calculated on the anhydrous basis), Min		99.5	Annex A
ii.	Sulphated ash, percent by mass, Max		0.01	Annex B
iii.	Moisture, percent by mass	Anhydrous, max	0.5	ISO 2483
		monohydrate	7.5 - 8.8	
iv.	Readily carbonizable substance		To pass the test	Annex C
v.	Sulphates (as $Na_2SO_4$ ), mg/kg, Max		150.0	ISO 2480
vi.	Water insoluble matter, ppm, Max		30.0	Annex D
vii.	Chloride (as Cl), ppm, Max		5.0	Annex E
viii.	Phosphate (as $P_2O_5$ ), ppm, Max		5.0	Annex F
ix.	Calcium, ppm, Max		25.0	ISO 2482
x.	Tridodecylamine, ppm, Max		0.1	Annex G

## 5 Contaminants

### 5.1 Pesticide residues

Food grade Citric acid shall conform to those maximum residue limits established by the Codex Committee on Pesticide Residues for this commodity.

### 5.2 Heavy Metals contaminants

S/N	Heavy Metal	Maximum Limit (mg/kg)	Test Method
i.	Arsenic (As)	0.5	AOAC 2013.06
ii.	Lead (Pb)	3.0	

## 6 Hygiene

Food grade citric acid shall be prepared under hygienic conditions in accordance with EAS 39.

## 7 Weights and measures

Citric Acid shall be packed in accordance with the weights and measures regulations of Zanzibar.

## 8 Packaging and Labelling

### 8.1 Packaging

Food grade citric acid shall be packaged in food grade materials that will safeguard the hygienic, nutritional and organoleptic qualities of the product.

## 8.2 Labelling

**8.2.1** In addition to the labelling requirements specified in ZNS 61, the containers shall be also legibly and indelibly labelled with the following: -

- a) name of the product as 'Food grade citric acid';
- b) brand name/trade name if any;
- c) name and address of producer/packer/distributor;
- d) storage condition and transportation;
- e) date of manufacturing;
- f) best before date;
- g) lot identification or batch or code number;
- h) country of origin;
- i) net weight in metric unit; and
- j) instruction for disposal of used packaged material.

**8.2.2** The language on the label shall be 'Kiswahili' and/or English. Additional language may be used depending on the designated market.

## 9 Sampling

Sampling of shall be done according to Annex H.

**Annex A**  
**(normative)**  
**Determination of Purity**

Dissolve about 3 g of the accurately weighed sample in 40 ml of water. Add phenolphthalein (1 g in 100 ml alcohol) and titrate with 1 N sodium hydroxide. Each ml of 1 N sodium hydroxide is equivalent to 64.04 mg of  $C_6H_8O_7$ .

**Annex B**  
**(normative)**  
**Determination of Sulphated Ash**

**B.1 Reagent**

concentrated sulphuric acid

**B.2 Procedure**

Weigh accurately 5 g of the material in a tared crucible. Ignite, gently at first, until the material is thoroughly charred. Cool, moisten the residue with 1 ml of sulphuric acid and ignite gently till the carbon is completely oxidized. Cool the crucible in a desiccator and weigh.

**B.3 Calculation**

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = mass, in g, of the residue; and

$M_2$  = mass, in g, of the material taken for the test.



## Annex C (normative) Method of readily carbonizable

### C.1 Reagents

**C.1.1** *Sulphuric Acid* —  $95 \pm 0.5$

**C.1.2** Mix 1 g of suitable starch with 10 mg of red mercuric oxide and sufficient cold water to make a thin paste. Add 20 ml of boiling water to this and boil for 1 min with continuous stirring and cool. Only clear solution shall be used.

**C.1.3** *Cobaltous Chloride*: Dissolve about 65 g of cobaltous chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 5 ml of this solution into a 250 ml iodine flask, add 5 ml hydrogen peroxide solution (containing between 2.5 g and 3.5 g of  $\text{H}_2\text{O}_2$  in each 100 ml) and 15 ml of sodium hydroxide solution (1 in 5). Boil for 10 min, cool and add 2 g of potassium iodide and 20 ml of dilute sulphuric acid (1 in 4). When the precipitate had dissolved, titrate the liberated iodine with 0.1 N sodium thiosulfate. The titration is sensitive to air oxidation and should be blanketed with carbon dioxide. Each ml of 0.1 N sodium thiosulfate is equivalent to 23.79 mg of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ . Adjust the final volume of the solution by the addition of enough of the mixture of hydrochloric acid and water to make each ml contain 59.5 mg of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ .

**C.1.4** *Ferric Chloride*: Dissolve about 55 g of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 10 ml of this solution into a 250 ml iodine flask, add 15 ml of water, 5 ml of hydrochloric acid, and 3 g of potassium iodide, and allow the mixture to stand for 15 min. Dilute with 100 ml of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate, adding starch solution (see C.1.2) as the indicator. Perform a blank determination with the same quantities of the same reagent and in the same manner, and make any necessary correction. Each ml of 0.1 N sodium thiosulphate is equivalent to 27.03 mg of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ . Adjust the final volume of the solution by the addition of the mixture of hydrochloric acid and water to make each ml contain 45.0 mg of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ .

### C.2 Procedure

Transfer 1.0 g, finely powdered material, to a 22 mm × 175 mm test tube, previously rinsed with 10 ml of sulphuric acid solution and allowed to drain for 10 min. Add 10 ml of sulphuric acid solution, agitate the tube until solution is complete, and immerse the tube in water bath at  $90 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$  for  $60 \text{ min} \pm 0.5 \text{ min}$ , keeping the level of the acid below the level of the water during the heating period. Cool the tube in a stream of water, and transfer the acid solution to a colour comparison tube. The colour of the acid solution shall not be darker than that of the same volume of a mixture of 0.5 parts cobaltous chloride solution (C.1.3) and 4.5 parts ferric chloride solution (C.1.4) (v/v)

**Annex D**  
(normative)  
**Determination of water insoluble matter**

**Procedure**

Treat 10 g of sample, accurately weighed, with 100 ml of hot water and filter through a tared filtering crucible. Wash the insoluble residue with hot water, dry at 105 °C for 2 h, cool and weigh.

## Annex E (normative)

### Determination of the chloride content calculated as sodium chloride

#### E.1 Apparatus

Normally available laboratory glassware

#### E.2 Reagents

##### E.2.1 Potassium chromate solution

Dissolve 5 g of potassium chromate ( $K_2CrO_4$ ) in 100 mL of water.

##### E.2.2 Standard 0.1 N silver nitrate solution

###### E.2.2.1 Preparation

Dissolve 17.0 g of silver nitrate ( $AgNO_3$ ) in 1 000 mL of water. Store the solution in the dark.

###### E.2.2.2 Standardization

###### E.2.2.2.1 Carry out the standardization in triplicate.

**E.2.2.2.2** Weigh out accurately 5.8 g of analytical reagent grade sodium chloride ( $NaCl$ ) (previously dried at  $200\text{ }^{\circ}C \pm 50\text{ }^{\circ}C$  for 2 h and cooled to room temperature in a desiccator) into a 1-L volumetric flask and dissolve it in approximately 200 mL of water. Adjust the temperature of this solution to  $20\text{ }^{\circ}C$  and dilute it to 1 000 mL with water at the same temperature. Pipette 25 mL of the sodium chloride solution at  $20\text{ }^{\circ}C$  into a 250 -mL conical flask, add 1 mL of potassium chromate solution, and titrate with the 0.1 N silver nitrate solution until a faint reddish-brown colour persists after brisk shaking.

**E.2.2.2.3** Carry out a blank titration using the same procedure but replacing the 25 mL sodium chloride solution with 25 mL water.

**E.2.2.2.4** Calculate the mean normality of the silver nitrate solution from the triplicate determinations.

$$N = \frac{Ax0.4277}{b-c}$$

where

$N$  is the normality of silver nitrate solution,

$A$  is the mass of sodium chloride, in grams, in 1 000 mL solution,

$b$  is the volume of silver nitrate solution, in millilitres, required to titrate 25 mL of sodium chloride solution, and

$c$  is the volume of silver nitrate solution, in millilitres, required to titrate the blank.

**NOTE** A commercially prepared volumetric solution may be used instead.

### E.3 Procedure

**E.3.1** Carry out the determination in triplicate on each of the test samples.

**E.3.2** Pipette 50 mL of the principal solution reserved in accordance with ISO 2479 at 20 °C, into a 250-mL volumetric flask and dilute to 250 mL with water at the same temperature. Mix well and pipette 25 mL of this solution at 20 °C into a 250-mL conical flask. If the solution is acid to litmus, neutralize with sodium bicarbonate solution; if the solution is alkaline, add dilute nitric acid (1:10) drop by drop until the solution is acid to litmus and then neutralize with sodium bicarbonate solution. Add 1 mL of potassium chromate solution and titrate with the standard 0.1 N silver nitrate solution until a faint reddish-brown colour persists after brisk shaking.

### E.4 Calculation

Calculate the chloride content as NaCl, on a moisture-free basis (and free-flowing agent-free basis, where relevant), as a percentage, as follows:

$$C = \frac{a \times N \times 1169}{B}$$

where

*C* is the chloride content, as NaCl, expressed as a percentage (by mass),

*a* is the volume of silver nitrate solution, in millilitres, used in the titration,

*N* is the normality of the silver nitrate solution,

*B* is the mass of sample, in grams, in 1 000 mL principal solution, corrected for moisture content and, where relevant, the drier content.

### E.5 Report

Report the chloride content of each test sample as the mean of its triplicate determinations

## Annex F (normative) Determination of Phosphate

### *Ferric Chloride*

Dissolve about 55 g of ferric chloride ( $\text{FeCl}_2 \cdot 6\text{H}_2\text{O}$ ) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 10 ml of this solution into a 250 ml iodine flask, add 15 ml of water, 5 ml of hydrochloric acid, and 3 g of potassium iodide, and allow the mixture to stand for 15 min. Dilute with 100 ml of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate, adding starch solution (See C-5.1.2) as the indicator. Perform a blank determination with the same quantities of the same reagent and in the same manner, and make any necessary correction. Each ml of 0.1 N sodium thiosulphate is equivalent to  $\times 175$  mm test tube, previously rinsed with 10 ml of sulphuric acid solution and allowed to drain for 10 min. Add 10 ml of sulphuric acid solution, agitate the tube until solution is complete, and immerse the tube in water bath at  $90\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$  for  $60\text{ min} \pm 0.5\text{ min}$ , keeping the level of the acid below the level of the water during the heating period. Cool the tube in a stream of water, and transfer the acid solution to a color comparison tube. The color of the acid solution shall not be darker than that of the same volume of a mixture of 0.5 parts cobaltous chloride solution (C-5.1.3) and 4.5 parts ferric chloride solution (C-5.1.4) (v/v)

### **Procedure**

Weigh accurately about 200 mg to 300 mg of sample, dissolve in 25 ml of water and 10 ml of diluted nitric acid test solution and boil for 30 min. Filter if necessary, and wash any precipitate, then dissolve the precipitate by the addition of 1 ml diluted nitric acid test solution. Adjust the temperature to about  $50\text{ }^\circ\text{C}$ , add 75 ml of ammonium molybdate test solution, and maintain the temperature at about  $50\text{ }^\circ\text{C}$  for 30 min, stirring occasionally. Allow to stand for 16 h or overnight at room temperature. Decant off the supernatant, through a filter paper, wash the precipitate once or twice with water by decantation using 30 ml to 40 ml each time, and pour the washings through the same filter. Transfer the precipitate to the same filter, and wash with potassium nitrate solution (1 in 100) until the filtrate is no longer acid to litmus paper.

Transfer the precipitate with filter paper to the original precipitation vessel, add 50.0 ml of 1 N sodium hydroxide, agitate and stir until the precipitate is dissolved, add 3 drops of phenolphthalein test solution and titrate the excess alkali with 1 N sulphuric acid. Each ml of 1 N sodium hydroxide consumed is equivalent to 3.088 mg of  $\text{P}_2\text{O}_5$ .

## Annex G (normative) Determination of Tridodecylamine

### Reagents

**Buffered Indicator Solution** — Prepare a mixture consisting of 700 ml of 0.01 M citric acid (anhydrous, reagent grade), 200 ml of 0.2 M disodium phosphate and 50 ml each of 0.2 percent bromophenol blue and of 0.2 percent bromocresol green in spectro-grade methanol.

**No Indicator Buffer Solution** — Prepare a mixture consisting of 700 ml of 0.1 M of citric acid (anhydrous, reagent grade), 200 ml of 0.2 M disodium phosphate and 100 ml of spectro-grade methanol.

**Amine Stock Solution** — Transfer between 40 mg and 45 mg of tridodecyl (trilauryl) amine, accurately weighed into a 500 ml volumetric flask, dilute to volume with isopropyl alcohol and mix. Discard after three weeks.

**Standard Amine Solution** — Using a graduated 5 ml pipette, transfer into a 100 ml volumetric flask an amount of Amine stock solution equivalent to 400 µg of tridodecylamine, dilute to volume with isopropyl alcohol and mix. Prepare this solution fresh on the day of use.

### Procedure

Dissolve 160 g of anhydrous reagent grade citric acid (not the sample to be tested) in 320 ml of water and divide the solution equally between two 250 ml separators,  $S_1$  and  $S_2$ . To  $S_1$  add 5 ml of no indicator buffer solution. To  $S_2$ , add 2.0 ml of standard amine solution and 5 ml of buffered indicator solution. To prepare solutions of the sample being tested, dissolve 160 g of anhydrous citric acid monohydrate sample in 306 ml of water. Divide the test solution equally between two 250 ml separators,  $S_3$  and  $S_4$ . Add 5 ml of no indicator buffer solution to  $S_3$  and 5 ml of buffered indicator solution to  $S_4$ . To each of the four separators, add 20 ml of 1 : 1 mixture (v/v) prepared from spectrograde chloroform and n-heptane. Shake for 15 min on a mechanical shaker and allow the phases to separate for 45 min. Drain all except the last few drops of the lower (aqueous) phases and discard. Hand-shake the organic phases with 25 ml each of 0.05 N sulphuric acid for 30 s and allow the phase to separate for 10 min. Drain all except the last few drops of the lower (organic) phase through a Whatman No. 40 (or equivalent) paper and collect the aqueous filtrates in separate small glassstoppered containers. Determine the absorbance of each solution in a 5 cm cell at 400 nm, with a suitable spectrophotometer standardized prior to analysis, against chloroform heptane (1 : 1 v/v). The net absorbance of the sample ( $S_4 - S_3$ ) is not greater than that of the standard ( $S_2 - S_1$ ).

## Annex H (normative)

### Method of sampling

#### H.1 General Requirements of Sampling

In drawing, preparing, storing and handling test samples, the precautions and directions as given below shall be observed:

**H.1.1** The samples shall be taken in a protected place which is not exposed to damp air, dust or soot.

**H.1.2** The sampling instrument shall be clean and dry.

**H.1.3** Precautions shall be taken to protect the sample and the material being sampled. The sampling instrument and the containers for samples from adventitious contamination.

**H.1.4** To draw a representative sample, the content of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**H.1.5** The sample shall be placed in clean, dry, air tight glass containers or other suitable containers on which the material has no reaction.

**H.1.6** The sample containers shall be of such a size that are almost completely filled by the sample.

**H.1.7** Each sample container shall be sealed air-tight with a stopper after filling, marked with full details of sampling, date of sampling, batch and code number.

**H.1.8** The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal atmospheric temperature.

**H.1.9** The sampling shall be carried out by a person agreed by the purchaser and the supplier and in the presence of the purchaser (or his representative) and the supplier (or his representative).

#### H.2 Scale of sampling

##### H.2.1 Lot

All the containers of the same material produced under the same conditions of manufacture shall be grouped together to constitute a lot.

The sample shall be tested from each lot so as to ascertain the conformity of the material to the requirements of the specification.

**H.2.2** The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table H.1.

**Table H.1: Number of containers to be selected for sampling**

Lot Size	No of containers to be selected
2-15	2
16-50	3
51-150	5
151 and above	9

NOTE: The containers shall be selected at random from the lot
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### H.3 Test and referee samples

Draw with an appropriate sampling instrument, small quantities of the material from different parts of each container selected according to col 2 of Table H.1. Mix all the portion thoroughly to form a composite sample weighing not less than 30 g. Divide the composite sample into three equal parts to form a test sample. Each part obtained shall constitute the test sample weighing not less than 10g and shall be sufficient to conduct all the tests.

The test samples shall be transferred immediately to thoroughly clean and dry containers which shall be sealed air-tight. These shall be labelled with the particulars given in H.1.7. One sample shall be for the purchaser, the second for the supplier and the third bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier and shall be kept at a place agreed to between the purchaser and the supplier or their representatives.

### H.4 Criterion for Conformity

**H.4.1** The test for the various characteristics shall be performed on the composite samples and shall meet the corresponding requirements specified in this Standard.

**H.4.2** In case any of the samples selected as per Table H.1 fail in any of the tests, two more samples shall be selected from the unopened containers from the sample lot for retest. If these two samples satisfy the requirements of the specification, the lot shall be deemed to comply with the requirements specified in the Standard. If either of the two samples fail in the retests, the lot shall be deemed as not conforming to the requirements specified in this Standard.