



DRAFT TANZANIA STANDARD

Sucralose — Specification

TANZANIA BUREAU OF STANDARDS

Sucralose — Specification

0. Foreword

Sucralose, (1,6-Dichloro-1,6-dideoxy- β -D-fructofuranosyl-4-chloro-4-deoxy- α -D-galactopyranoside) is a non-nutritive artificial sweetener, approximately 650 times sweeter than sucrose (table sugar), twice as sweet as saccharin, and four times as sweet as aspartame. It has an acceptable daily intake (ADI) of 15 mg/kg body weight.

Consumption of sucralose has increased as result of the growing trends to consume low calorie foods with less impact on blood glucose than sucrose. It also has no effect on tooth decay and is commonly found in oral health products. It possesses the following sweetener characteristics: non-nutritive, insipid and stability at high temperatures and in acidic medium. Its characteristics are preserved, even during pasteurisation, sterilisation and cooking at high temperatures. In addition, it cannot be hydrolysed during digestion or metabolism due to the high stability of its carbon-chlorine bonds and it does not interact chemically with other foods.

Sucralose-based products are found in a broad range of lower-calorie foods, including, fizzy drinks, chewing gum, baking mixes, breakfast cereals and salad dressings.

This standard has been formulated to provide the necessary guidelines to manufacturers, traders and consumers of sucralose and sucralose-based products and, for purposes of conformity assessment.

In preparation of this draft Tanzania standard assistance was drawn from India standard (IS 15969:2013) Specification for sucralose.

In reporting the result of a test or analysis made in accordance with the Tanzania standard, if the final value observed or calculated is to be rounded off, it shall be done in accordance with TZS 4.

1 Scope

This Draft Tanzania Standard specifies requirements, methods of sampling and test for food grade sucralose.

2 Normative references

The following referenced documents are referred to in the text in such a way that some or all their content constitutes requirements 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.

AOAC 972.25, *Lead in food. Atomic absorption spectrophotometric method*

AOAC 973.34, *Cadmium in food by atomic absorption spectroscopy (AAS)*

TZS 116, *General standard for the labelling of food additives when sold as such*

CAC/GL 50 – *General guidelines on sampling*

The Weights and Measures (Sales and Labelling of goods) Rules, 2007

TZS 4, *Rounding off numerical value*

TZS 538, *Labelling of pre-packaged foods — General requirements*

3 Terms and definitions

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

3.1 artificial/non-nutritive sweetener is artificial substance that has a similar taste to sugar other than sugar used to sweeten food or drink.

3.2 food grade material a material, made of substances which 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

Sucralose shall be, free from off odors, foreign matter and adulterants

Sucralose shall be presented as a white to off-white, practically odourless crystalline powder. It shall be freely soluble in water, methanol and ethanol, and slightly soluble in ethyl acetate.

4.2 Infrared Absorption

The infrared spectrum of a potassium bromide dispersion of a sucralose sample shall correspond with the reference infrared spectrum for sucralose.

4.3 Thin Layer Chromatography

The main spot in the test solution shall have the same retardation factor (*R_f*) as that of the main spot of Standard Solution A obtained in the test for other chlorinated disaccharides (see Annex B) when tested using thin layer chromatography.

4.3 Specific requirements

When tested in accordance with the specified or annexed methods, sucralose shall conform to the requirements in Table 1.

Table 1 — Requirements for Sucralose

S/N	Parameter	Requirement	Method of test
i	Assay (purity), percent by mass (dry basis)	98 – 102	Annex C
ii	Moisture , percent by mass, Max	2	ISO 760
iii	Sulphated ash, percent by mass, Max	0.7	Annex D
iv	Triphenylphosphine oxide, mg/kg, Max	150	Annex E
v	Methanol, percent by mass, Max	0.1	Annex F
vi	Chlorinated disaccharides	To pass	Annex A
vii	Chlorinated monosaccharides	To pass	Annex B

5. Hygiene

5.1 Food grade sucralose shall be prepared and packaged in premises built and maintained under hygienic condition in accordance with TZS 109.

5.2 Food grade sucralose shall be free from pathogenic organisms.

6 Contaminants

When tested following AOAC 972.25, the heavy metal contamination expressed as Lead (Pb) shall not exceed 1 mg/kg.

7. Sampling

Representative samples of the product shall be drawn in accordance with CAC/GL 50.

8. Packaging, marking and labelling.

8.1 The product shall be securely packaged in containers made of food grade materials conforming to TZS 539. The packages shall preserve the quality of the product, prevent entry of light and preclude contamination from the external environment.

8.2 Marking and labelling

8.2.1 In addition to the requirements of TZS 538 and TZS 116, the product label shall be legibly and indelibly labelled with the following:

- a) Name of the product as sucralose with the words 'Food Grade';
- b) Name and physical address of the manufacturer/distributor;
- c) Net weight in metric units;
- d) Batch/lot number;
- e) Directions for storage;
- f) Date of manufacture; and the
- g) Expiry date.
- h) Instruction for use refer to CODEXSTAN 107
- i) Statutory warnings (For industrial use)

8.2.2 The language on the label shall be "Kiswahili" or Kiswahili and English. A second language may be used depending on the designated market.

8.2.3 The packages of the buns may also be marked with the TBS certification mark.

NOTE - The TBS Standards Mark of Quality may be used by the manufacturers only under license from TBS. Particulars of conditions under which the licenses are granted, may be obtained from TBS

Annex A

(normative)

Determination of chlorinated disaccharides**B-1 REQUIREMENTS**

- **TLC Plates** — Reverse phase thin layer chromatography plates coated with 0.2 mm layer of silica gel absorbent (for example, Whatman LKC18).

B-2 REAGENTS

- a) **Mobile phase** — Mix 7 volumes of a 5.0% w/v aqueous solution of sodium chloride and 3 volumes of acetonitrile.
- b) **Spray reagent** — Use 15% v/v solution of concentrated sulphuric acid in methanol.
- c) **Standard solutions** — Dissolve 1.0 g of sucralose reference standard in 10 ml of methanol (Solution A). Dilute 0.5 ml of Solution A with methanol to 100 ml (Solution B).
- d) **Test solution** — Dissolve 1.0 g of sample in 10 ml of methanol.

B-3 PROCEDURE

Apply 5 µl each of solution A, solution B and test solution to the bottom of the chromatographic plate. Place the plate in a suitable chromatography chamber containing freshly prepared mobile phase and allow the solvent front to ascend 15 cm. Remove the plate from the chamber, allow it to dry and spray with spray reagent. Heat the plate in an oven at 125 °C for 10 min.

The sample is said to have passed the test when the main spot in the test solution has the same *R_f* value as the main in solution A and no other spot in the test solution is more intense than the 0.5 percent spot in solution B.

Annex B

(normative)

Test for chlorinated monosaccharides**C-1 REQUIREMENTS**

- **TLC plates** — Thin layer chromatography plates coated with 0.25 mm thickness of silica gel 60 or equivalent.

C-2 REAGENTS

- **Spray reagent** — Dissolve 1.23 g p-anisidine and 1.66 g phthalic acid in 100 ml methanol. Store the solution in darkness and refrigerate to prevent it from becoming decolourised. Discard if solution becomes discoloured.

NOTE — p-anisidine is toxic by skin absorption and inhalation and should be used with due caution.

- **Standard solution A** — Dissolve 10.0 ± 0.001 g of mannitol in 40 ml of distilled water in a 100 ml volumetric flask. Dilute to volume using the water.
- **Standard solution B** — Dissolve 10 g of mannitol and 40 mg of fructose (Analytical grade) in 25 ml of water in a 100 ml volumetric flask and make up to volume with the water.
- **Sample solution** — Dissolve 2.5 g of sample in 5 ml of methanol in a 10 ml volumetric flask and make up to volume with methanol.

C-3 PROCEDURE

Spot 5 μ l of each of standard solutions A and B onto the TLC plate, applying the solution slowly in 1 μ l aliquots and allowing the plate to dry between applications. Spot 5 μ l of the sample solution onto the plate in a similar manner. The three spots should be of similar size. Spray the plate with the spray reagent and heat at 100 ± 2 °C for 15 minutes. Immediately after heating, view the plate against a dark background.

The sample shall have passed the test when the spot from the sample solution is not more coloured than the spot from solution B (equivalent to a limit of 0.1% maximum total chlorinated monosaccharides).

Darkening of the mannitol spot from standard solution A indicates that the plate has been held too long in the oven, and a second plate should be prepared.

Annex C

(normative)

Assay of sucralose**D-1 CHROMATOGRAPHIC SYSTEM**

Fit a high-pressure liquid chromatograph, operated at room temperature, with a radial compression module containing a 10 cm 5 µm C18 reverse phase column. The mobile phase is maintained at a pressure and flow rate (typically 1.5 ml/min) capable of giving the required elution time (see D-5). An ultraviolet detector that monitors absorption at 190 nm, or a refractive index detector is used.

D-2 MOBILE PHASE

Add 150 ml of acetonitrile (HPLC grade, far UV, filtered through a 0.45 µm millipore filter or equivalent) to 850 ml of water (glass distilled, filtered through a 0.45 µm millipore filter or equivalent). Mix and degas thoroughly.

D-3 STANDARD SOLUTION

Weigh accurately about 250 mg of sucralose reference standard into a 25 ml volumetric flask. Dissolve and make up to volume using the mobile phase. Filter the solution through a 0.45 µm millipore filter or equivalent. Record the weight of reference standard as W_s .

D-4 TEST SOLUTION

Weigh accurately about 250 mg of sample into a 25 ml volumetric flask. Dissolve and make up to volume using the mobile phase. Filter the solution through a 0.45 µm millipore filter or equivalent. Record the weight of sample as W_t .

D-5 SYSTEM SUITABILITY TEST

Inject duplicate 20 µl portions of standard solution into the chromatograph. The retention time of the sucralose should be approximately 9 min. The co-efficient of variation (100 x standard deviation divided by mean peak area) for the peak areas should not exceed 2 percent.

NOTE — The retention time quoted is appropriate for a 10 cm 5 µm Rad-Pak C18 column. If a column of a different make or length is used it may be necessary to adjust the proportion of acetonitrile in the eluent to obtain the required retention time.

D-6 PROCEDURE

Analyse the test solution under the conditions described above, making duplicate 20 µl injections, and calculate the mean peak area. Calculate the percentage purity from the relative peak areas of the test (A_t) and standard solutions (A_s) per the following formula:

$$\text{Purity, percent} = \frac{A_t \times W_s}{A_s \times W_t} \times 100$$

Calculate the percentage purity on a water-free and methanol-free basis using the values obtained in the tests for water and methanol.

Annex D

(normative)

Determination of sulphated ash**E-1 REAGENT**

- Concentrated sulphuric acid (A.R. grade)

E-2 APPARATUS

- Crucible
- Desiccator
- Hot plate
- Furnace
- Weigh balance

E-3 PROCEDURE

Weigh accurately about 2 g of product 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 consumed. Cool the crucible in a desiccator and weigh.

NOTE — Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

E-4 CALCULATION

$$\text{Sulphated ash, percent by mass} = \frac{W_1}{W_2} \times 100$$

where,

W_2

W_1 = mass in grams of the residue; and

W_2 = mass in grams of the product tested.

Annex E

(normative)

Test for triphenylphosphine oxide

F-1 APPARATUS

Chromatographic system — Typically a high-pressure liquid chromatograph, operated at room temperature, is fitted with a radial compression module containing a 5 μm C18 Rad-Pak reverse phase column (10 cm x 8 mm). The mobile phase is maintained at a pressure and flow rate (typically 1.5 ml/min) capable of giving the required elution time. The chromatograph is equipped with a UV detector (220 nm).

F-2 REAGENTS

F-2.1 Mobile phase — Add 67 volumes of acetonitrile (HPLC grade, far UV, filtered through a 0.45 μm millipore filter or equivalent) to 33 volumes of water (glass distilled, filtered through a 0.45 μm millipore filter or equivalent). Mix and de-gas thoroughly.

F-2.2 Standard solution — Weigh accurately 100 mg of triphenylphosphine oxide into a 10 ml volumetric flask. Dissolve and make up to volume using the mobile phase. Take 1.0 ml of the resulting solution and make up to 100 ml with mobile phase. From this solution, prepare a further 100-fold dilution with mobile phase and use this as the standard solution. Filter through a 0.45 μm millipore filter or equivalent.

F-2.3 Test solution — Weigh accurately 100 mg of sample into a 10 ml volumetric flask. Dissolve and make up to volume with mobile phase. Filter through a 0.45 μm millipore filter or equivalent. Record the weight of sample as W_t mg.

F-3 PROCEDURE

Inject duplicate 25 μl portions of the standard and test solutions into the chromatograph. Under the conditions stated above the retention time for triphenylphosphine oxide is 6 min. Record the mean peak areas for the standard and test solutions as A_s and A_t respectively.

F-4 CALCULATION

Calculate the concentration of triphenylphosphine oxide (TPPO) in the sample from the following formula:

$$\text{TPPO (mg/kg)} = \frac{A_t}{A_s} \times \frac{10000}{W_t}$$

Annex F

(normative)

Test for methanol

G-1 APPARATUS/EQUIPMENT

Use a suitable gas chromatograph equipped with a hydrogen flame ionization detector containing a 2.1 m x 4.0 mm (id) glass column packed with Porapak PS 80-100 mesh or equivalent materials.

G-2 OPERATING CONDITIONS

The operating conditions may vary depending upon the particular instrument used but a suitable chromatogram may be obtained by using the following conditions:

- a) Column temperature: 150 °C (isothermal)
- b) Inlet temperature: 200 °C
- c) Detector temperature: 250 °C
- d) Carrier gas nitrogen: 20 ml/min

G-3 REAGENTS

G-3.1 Standard solution — Using a 2.0-ml class A volumetric pipette, pipette 2.0 ml of methanol into a 100 ml volumetric flask, dilute to volume with pyridine, and mix. Transfer 1.0 ml of this solution to a 100 ml volumetric flask, dilute to volume with pyridine, and mix.

G-3.2 Sample solution — Weigh accurately about 2 g of the sample into a 10-ml volumetric flask, dilute to volume with pyridine, and mix.

G-4 PROCEDURE

Inject a 1 µl portion of the standard solution onto a gas chromatography column, obtain the chromatogram, and measure the area of the peak produced. The relative standard deviation for replicate injections is not more than 2.0 percent. Calculate the mean peak areas for the standard solution. Similarly, inject a 1 µl portion of the sample solution into the gas chromatograph, and measure the areas of the peaks produced by methanol. Calculate the mean peak areas, and determine the methanol concentration using the following formula:

Methanol, percent =

where

SA = sample area;

$$\frac{S_A \times V_S \times C_S}{A_S \times W_S}$$

CS = percentage concentration of methanol in the standard (volume of methanol x dilution factor x density of solvent equals $2 \times 10^{-4} \times 0.79 \times 100$);

VS = volume of the sample solution;

AS = standard area; and

WS = mass in grams of the sample.

Bibliography

- [1] IS 15969: 2013, Sucralose, food grade — Specification
- [2] JECFA (1993), Sucralose

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