DUS DEAS 994

DRAFT UGANDA STANDARD

First Edition
2019-mm-dd

Food grade sucralose — Specification

Reference number
DUS DEAS 994: 2019

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National foreword

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(a) a member of International Organisation for Standardisation (ISO) and

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The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of representatives of consumers, traders, academicians, manufacturers, government and other stakeholders.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

This Draft Uganda Standard, DUS DEAS 994: 2019*, Food grade sucralose — Specification*, is identical with and has been reproduced from an East African Standard, DEAS 994: 2019, *Food grade sucralose — Specification,* and is being proposed for adoption as a Uganda Standard.

The committee responsible for this document is Technical Committee UNBS/TC 2, *Food and agriculture.*

Wherever the words, “East African Standard " appear, they should be replaced by "Uganda Standard."

DEAS 994: 2019

ICS 67.220.20

DRAFT EAST AFRICAN STANDARD

Food grade sucralose — Specification

EAST AFRICAN COMMUNITY

First Edition 2019

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 005 *Food additives*.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

Introduction

Sucralose, (1,6-dichloro-1,6-dideoxy-β-D-fructofuranosyl-4-chloro-4-deoxy-alpha-D-galactopyranoside) is a non-caloric 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.

Its consumption 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-caloric, 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 therefore been developed to prescribe requirements for food grade sucralose for use in food industry.

Food grade sucralose — Specification

1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for sucralose intended for use in food products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of 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*

CAC/GL 50, *General guidelines on sampling*

CODEX STAN 107, *General standard for the labelling of food additives when sold as such*

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
food grade material
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

3.2
sucralose
low calorie, artificial, derivative substance of chlorination of sucrose, used for sweetening food and drink

3.3
artificial sweetener
synthetic substance used as a sugar substitute to sweeten food and drink

4 Requirements

4.1 General requirements

Sucralose shall be;

a) a white to off-white;

b) practically odourless crystalline powder;

c) freely soluble in water, methanol and ethanol; and

d) slightly soluble in ethyl acetate.

4.2 Specific requirements

Sucralose shall comply with the specific requirements given in Table 1 when tested in accordance with the test methods specified therein.

Table 1 — Specific requirements for sucralose

S/N	Characteristic	Requirement	Test method
i)	Chlorinated disaccharides*	To pass the test	Annex B
ii)	Chlorinated monosaccharides	To pass the test	Annex C
iii)	Assay: percentage purity, % m/m (dry basis)	98 – 102	Annex D
iv)	Loss on drying, % m/m, max.	2	Annex E
v)	Sulphated ash, % m/m, max.	0.7	Annex F
vi)	Triphenylphosphine oxide, mg/kg, max.	150	Annex G
vii)	Methanol, % m/m, max.	0.1	Annex H

* 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 when tested using thin layer chromatography.

4.3 Infrared absorption

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

5 Hygiene

Food grade sucralose shall be manufactured and handled in accordance with EAS 39.

6 Contaminants

Lead (as Pb), shall not exceed 1 mg/kg when tested in accordance with AOAC 972.25.

7 Packaging

Food grade sucralose shall be securely packaged in containers made of food grade materials. The packages shall preserve the safety and quality of the product, prevent entry of light and preclude contamination from the external environment.

8 Weights and measures

The products shall comply with the Weights and Measures Regulations of the respective Partner States.

9 Labelling

In addition to the requirements of CODEX STAN 107, the product packages shall be legibly and indelibly labelled with the following information:

a) name of the product as “Food grade sucralose”;

b) name and physical address of the processor/packer/importer;

c) date of manufacture;

d) expiry date; and

e) net weight of the product in metric units.

10 Sampling

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

Annex A
(normative)

Infrared spectrum of sucralose

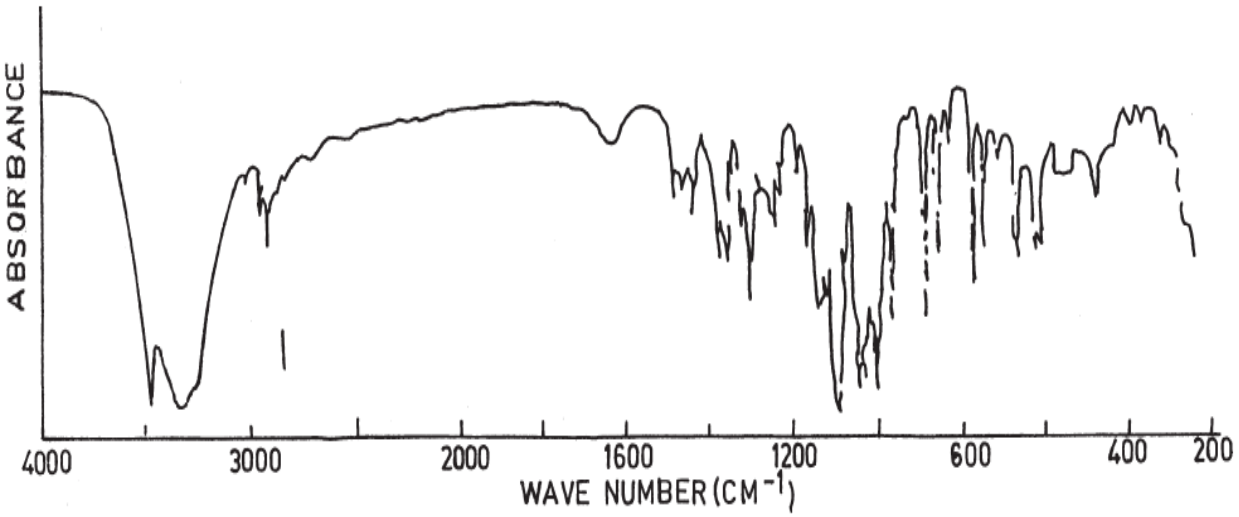

Figure 1 — Reference infrared spectrum of a potassium bromide dispersion of sucralose

Annex B
(normative)

Determination of chlorinated disaccharides

B.1 Apparatus

TLC plates. Reverse phase thin layer chromatography plates coated with 0.2 mm layer of silica gel absorbent (for example, Whatman LKC_{18})

B.2 Reagents

B.2.1 Mobile phase. Mix 7 volumes of a 5.0 % w/v aqueous solution of sodium chloride and 3 volumes of acetonitrile.

B.2.2 Spray reagent. Use 15 % v/v solution of concentrated sulphuric acid in methanol.

B.2.3 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).

B.2.4 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 % spot in solution B.

Annex C
(normative)

Test for chlorinated monosaccharides

C.1 Apparatus

TLC plates coated with 0.25 mm thickness of silica gel 60 or equivalent.

C.2 Reagents

C.2.1 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.

C.2.2 Standard solution A. Dissolve 10.0 g ± 0.001 g of mannitol in 40 ml of distilled water in a 100 ml volumetric flask. Dilute to volume using the water.

C.2.3 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.

C.2.4 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 µl of each of standard solutions A and B onto the TLC plate, applying the solution slowly in 1 µl aliquots and allowing the plate to dry between applications. Spot 5 µl 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 °C ± 2 °C for 15 min. View the plate against a dark background immediately after heating.

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 D
(normative)

Assay: Percentage purity

D.1 Apparatus

D.1.1 Chromatographic system

Fit a high-pressure liquid chromatograph, operated at room temperature, with a radial compression module containing a 10 cm 5 µm C_{18} 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.1.2 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.2 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 mass of reference standard as W_s.

D.3 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 mass of sample as W_t.

D.4 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 %.

NOTE The retention time quoted is appropriate for a 10 cm 5 µm Rad-Pak C_{18} 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.5 Procedure

Analyse the test solution under the conditions described above, making duplicate 20 µl injections, and calculate the mean peak area.

The percentage purity from the relative peak areas of the test (A_t) and standard solutions (A_s), expressed as percent, shall be calculated as follows:

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

where

A_t is the percentage purity of the test solution;

W_s is the mass, in milligrams, of the reference standard;

A_s is the percentage purity of the standard solution; and

W_t is the mass, in milligrams of the sample

The percentage purity on a water-free and methanol-free basis shall be calculated using the values obtained in the tests for water and methanol, respectively.

Annex E
(normative)

Loss on drying

E.1 Requirements

E.1.1 Weighing bottle with a stopper

E.1.2 Air oven

E.1.3 Desiccator

E.1.4 Weighing scale

E.2 Sample preparation

Weigh 1 to 2 g of sample (M_1). Tare a glass-stoppered, shallow weighing bottle that has been dried for 30 minutes at 105 ºC. Transfer the sample into the bottle, replace the cover, and weigh the bottle and the sample (M_2). Distribute the sample as evenly as practicable to a depth of about 5 mm, and not over 10 mm.

E.3 Procedure

Place the bottle with its contents in the drying chamber, removing the stopper and leaving it also in the chamber, and dry the sample at the 105 ºC for 2 hours. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature in a desiccator. Weigh the cool bottle and its contents (M_3).

Calculate the loss on drying from the following equation:

$$\text{Loss on drying (\%w/w)} = \frac{M_2 - M_3}{M_1} \times 100$$

where

M_1 is the mass of sample in grams;

M_2 is the mass of sample and weighing bottle in grams before drying; and

M_3 is the mass of sample and weighing bottle in grams after drying and cooling in a desiccator.

If the sample melts at a temperature lower than 105 ºC, prepare the sample as described above, then place it in a vacuum desiccator containing sulfuric acid. Evacuate the desiccator to 130 Pa (1 mm of mercury), maintain this vacuum for 24h, and then weigh the dried sample. Calculate the loss on drying using the same equation above.

Annex F
(normative)

Determination of sulphated ash

F.1 Apparatus

F.1.1 Crucible

F.1.2 Heating plate

F.1.3 Desiccator

F.1.2 Analytical balance

F.2 Reagents

Concentrated sulphuric acid (A.R. grade)

F.3 Procedure

Weigh accurately about 2 g of product in a tared crucible (W_2). Ignite, gently at first, until the material is thoroughly charred. Cool and 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 (W_1).

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.

Sulphated ash, expressed as percent by mass, shall be calculated using the formula below:

$$\frac{W_1}{W_2} \text{ X } 100$$

where

W_1 is the mass, in grams, of the residue; and

W_2 is the mass, in grams, of the product tested.

Annex G
(normative)

Test for triphenylphosphine oxide

G.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 C_{18} 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).

G.2 Reagents

G.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.

G.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.

G.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 mass of sample as W_t in milligrams

G.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.

The concentration of triphenylphosphine oxide (TPPO) in the sample, expressed in milligrams per kilogram, shall be calculated using the formula below:

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

where

A_t is the mean peak area for the standard solution;

A_s is the mean peak area for the test solution; and

W_t is the mass, in milligrams, of the sample.

Annex H
(normative)

Test for methanol

H.1 Apparatus

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.

H.1.2 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:

- Column temperature: 150 °C (isothermal);
- Inlet temperature: 200 °C;
- Detector temperature: 250 °C; and
- Carrier gas nitrogen: 20 ml/min.

H.2 Reagents

H.2.1 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.

H.2.2 Sample solution. Weigh accurately about 2 g of the sample (W_s) into a 10 ml volumetric flask, dilute to volume with pyridine, and mix.

H.3 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 should not be more than 2.0 %. Calculate the mean peak areas for the standard solution (A_s). Similarly, inject a 1 µl portion of the sample solution (V_s) into the gas chromatograph, and measure the areas of the peaks produced by methanol (S_A).

Calculate the mean peak areas, and determine the methanol concentration, expressed as a percentage, using the following formula:

$$\text{Methanol, percent} = \frac{S_A \times V_S \times C_S}{A_S \times W_S}$$

where

S_A is the area for the sample;

C_S is the percentage concentration of methanol in the standard (volume of methanol X dilution factor X density of solvent equals 2 x 10^{-4} x 0.79 x 100);

V_S is the volume of the sample solution;

A_S is the standard area; and

W_S is the mass, in grams, of the sample.

Bibliography

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[3] Food chemicals Codex (5th Edition)

[4] JECFA (2006), Analytical methods, test procedures and laboratory solutions used by and referenced in the food additive specifications, Volume 4.

