DUS 1925

DRAFT UGANDA STANDARD

First Edition
2018-mm-dd

Saccharin (food grade) — Specification

PUBLIC REVIEW DRAFT

Reference number
DUS 1925: 2018

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Foreword

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

(b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and

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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.

The committee responsible for this document is UNBS/TC 2 [*Food and Agriculture standards*], Subcommittee SC 6, [*Food Additives and contaminants*].

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Introduction

Saccharin (1,2-Benzisothiazole-3(2H)-one-1,1-dioxide, 3-oxo-2,3-dihydrobenzo[d]isothiazol-1,1-dioxide) and its sodium, calcium and potassium salts are widely utilised in the food industry as non-nutritive artificial sweeteners. They are used as a substitute for cane sugar in the production of diet and low-calorie foods. They are colourless crystals or white crystalline powders with faint aroma and about 300 – 500 times sweeter than sucrose (table sugar). In the Codex Alimentarius Commission International Numbering System, saccharins are assigned as INS 954 and the FAO/WHO Joint Experts Committee on Food Additives (JECFA) established the Acceptable Daily Intake (ADI) for saccharin and its salts at 0 – 5 mg/kg body weight.

There are two main approaches to making saccharin: the Remsen-Fahlberg process (named after the two scientists who discovered the compound) and the Maumee or Sherwin-Williams process.

The Remsen-Fahlberg process requires reacting toluene, which has a natural sweet smell, with chlorosulfonic acid, which is a colourless liquid. This acid compound is then reacted with a series of compounds, including potassium permanganate and ammonia and heated to yield saccharin. Because this process takes a lot of compounds to produce a relatively low yield, improvements were sought-which is why the Maumee process was created.

The Maumee process begins with converting phthalic anhydride, an industrial compound used in creating plastics to anthranilic acid. The acid is reacted with several compounds, including nitrous acid, sulphur dioxide, chlorine and ammonia to produce saccharin.

Common food products containing saccharin and/or its Na, Ca or K salts include: soft drinks (sodas), low-calorie jams or jellies, biscuits, candies and dessert toppings such as flavoured syrups for ice cream, and salad dressings. These sweeteners are also used in pharmaceutical products and toothpaste.

The use of artificial sweeteners in food continues to be a controversial subject and this standard has been developed to ensure that the use of saccharin and its salts conforms to acceptable limits prescribed by scientists, health authorities, consumer organisations and the relevant international bodies.

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Saccharin (food grade) — Specification

1 Scope

This Draft Uganda Standard specifies requirements, sampling and test methods for food grade saccharin.

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*

US 277, *General standard for the labelling of food additives when sold as such*

US 1659, *Materials in contact with food — Requirements for packaging materials*

US CAC/GL 50, *General guidelines on sampling*

FDUS ISO 760, *Determination of water — Karl Fischer Method (General method)*

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 address:

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

food grade material
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

Food grade saccharin shall be:

- white crystals or white crystalline powder;
- odourless or with a faint aromatic odour,
- slightly soluble in water, soluble in basic solutions and sparingly soluble in ethanol. 1 g of product shall be soluble in 1.5 ml of water or basic solutions and in about 50 ml of ethanol.

4.2 Specific requirements

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

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Table 1 — Specific requirements for food grade saccharin

S/N	Characteristic	Requirement	Test method
i)	Purity as $C_7H_5NO_3S$, % m/m (dry matter basis)	99 – 101	Annex A
ii)	Moisture content, %m/m, max.	1	Annex B
iii)	Acidity and alkalinity at room temperature	To pass test	Annex C
iv)	Benzoic and salicylic acid	To pass test	Annex D
v)	Readily carbonizable substances	To pass test	Annex E

5 Contaminants

Food grade saccharin shall comply with the Maximum Levels of contaminants given in Table 2 when tested in accordance with the test methods specified therein.

Table 2 — Limits for contaminants for food grade saccharin

S/N	Contaminant	Maximum level	Test method
i)	Lead (Pb), mg/kg, max.	1	AOAC 972.25
ii)	Arsenic (As), mg/kg, max.	2	AOAC 952.13
iii)	Selenium (Se), mg/kg, max.	30	Annex F
iv)	Toluenesulfonamides, mg/kg, max.	25	Annex G

6 Packaging

The product shall be securely packaged in containers made of food grade materials conforming to US 1659. The packages shall preserve the quality of the product, prevent entry of light and preclude contamination from the external environment.

7 Weights and Measures

The weight of the product when packaged shall comply with the Weights and Measures Regulations.

8 Labelling

In addition to the requirements of US 277, the product label shall be legibly and indelibly labelled with the following:

i) Name of the product as Saccharin with the words ‘Food Grade’;

ii) Name and physical address of the manufacturer/distributor;

iii) Net weight in metric units;

iv) Batch/lot number;

v) Directions for storage; and the

vi) Date of manufacture

vii) Expiry date.

9 Sampling

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

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Annex A
(normative)

Assay: Test for purity

A.1 Procedure

Dry 10 g of product in a hot air oven at 120 °C for 4 hours. Accurately weigh about 0.5 g (*M*) of the dried sample and dissolve it in 75 ml of hot water. Quickly cool the mixture and add 2 – 3 drops of phenolphthalein indicator. Titrate the mixture with 0.1 N sodium hydroxide till the endpoint and record the volume of NaOH used (*V*).

Each ml of 0.1 N sodium hydroxide is equivalent to 18.32 mg of $C_7H_5NO_3S$.

Therefore, percentage purity (as $C_7H_5NO_3S$), m/m (dry matter basis) shall be calculated from:

$$\frac{18.32 \times V}{M \times 1000} \times 100$$

where

V is the volume, in millilitres, of 0.1N NaOH used and

M is the mass, in grams, of dried sample dissolved in 75 ml of hot water.

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Annex B
(normative)

Moisture content

B.1 Procedure

The moisture content shall be determined by drying a weighed sample of product (M_1) in an air oven at 102 °C for 2 hours. Thereafter, the sample shall be cooled in a desiccator and the weighed (M_2). The moisture content shall be computed from:

$$\text{Moisture content, (\%m/m)} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 is the mass, in grams, of the sample before drying and

M_2 is the mass, in grams, of the sample after drying and cooling.

In cases of dispute, the moisture content shall be determined following FDUS ISO 760.

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Annex C
(normative)

Test for acidity and alkalinity

C.1 Procedure

Dissolve 1 g of sample in 10 ml of freshly boiled and cooled water. Add one drop of phenolphthalein indicator. No pink colour shall appear.

Add one drop of 0.1 M sodium hydroxide. A pink colour shall appear.

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Annex D
(normative)

Test for benzoic and salicylic acid

D.1 Procedure

Add ferric chloride drop wise to 10 ml of a hot, saturated solution of the sample. No precipitate or violet colour shall appear.

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Annex E
(normative)

Test for readily carbonizable substances

E.1 Reagents

E.1.1 Sulphuric Acid

E.1.2 Matching Fluid; Composed of 0.1 ml of cobalt chloride solution (see E.1.2.1), 0.4 ml of ferric chloride solution (see E1.2.2), 0.1 ml cupric sulphate solution (see E.1.2.3) and 4.4 ml of distilled water.

E.1.2.1 Cobalt chloride solution: Dissolve 65 g of cobaltous chloride ($CoCl_2.6H_2O$) in a mixture of 25 ml of hydrochloric acid and 975 ml of distilled water. Place exactly 5 ml of this solution in a 250-ml iodine flask, add 5 ml of 3 percent hydrogen peroxide solution and 15 ml of 20 percent sodium hydroxide solution. Boil for 10 minutes, cool, and add 2 g of potassium iodide and 20 ml of 25 percent sulphuric acid. When the precipitate has dissolved, titrate the liberated iodine with 0.1N sodium thiosulphate solution, adding starch as indicator. Each millilitre of 0.1N sodium thiosulphate is equivalent to 23.8 mg of cobaltous chloride. Adjust the final volume of the solution by adding an adequate amount of the hydrochloric acid and distilled water mixture so that each millilitre contains 59.5 mg of cobaltous chloride.

E.1.2.2 Ferric chloride solution: Dissolve 55 g of ferric chloride ($FeCl_3.6H_2O$) in a mixture of 25 ml hydrochloric acid and 975 ml of distilled water. Place 10 ml of this solution in a 250-ml iodine flask. Add 15 ml of distilled water and 3 g of potassium iodide, and allow the mixture to stand for 15 minutes. Dilute with 100 ml of distilled water, and titrate the liberated iodine with 0.1N sodium thiosulphate solution, adding starch as indicator. Each millilitre of 0.1N sodium thiosulphate is equivalent to 27.03 mg of ferric chloride. Adjust the final volume of the solution by adding enough of the hydrochloric acid and distilled water mixture to make each millilitre contain 45.0 mg of ferric chloride.

E.1.2.3 Cupric sulphate solution: Dissolve 65 g of cupric sulphate ($CuSO_4.5H_2O$) in a mixture of 25 ml of hydrochloric acid and 975 ml of distilled water. Place 10 ml of this solution in a 250-ml iodine flask, add 40 ml of distilled water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1N sodium thiosulphate solution, adding starch as indicator. Each millilitre of 0.1N sodium thiosulphate is equivalent to 24.97 mg of cupric sulphate. Adjust the final volume of the solution by adding enough of the hydrochloric acid and distilled water mixture to make each millilitre contain 62.4 mg of cupric sulphate.

E.2 Procedure

Dissolve 0.2 g of the sample in 5 ml of sulphuric acid. Keep the solution at 48 °C – 50 °C for 10 minutes. The colour of the resultant solution shall not be darker than that of the matching fluid in E.1.2 (a very light brownish-yellow).

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Annex F
(normative)

Test for Selenium

F.1 Reagents

F.1.1 Selenium Stock Solution: Transfer 120 mg of metallic selenium (Se) into a 1000-ml volumetric flask, add 100 ml of dilute nitric acid (1 in 2). Gently warm the solution on a steam-bath and dilute to volume with distilled water. Transfer 5 ml of this solution into a 200-ml volumetric flask, dilute to volume with distilled water, and mix. Each millilitre of this solution contains 3 µg of selenium ions (Se).

F.1.2 Standard Selenium Solution: Just prior to the test, transfer 20 ml of selenium stock solution (60 µg Se) into a 200 mm x 25 mm test tube, add 20 ml of hydrochloric acid, and mix.

F.1.3 Sample Solution: Transfer 2 g of the sample to a 250-ml Erlenmeyer flask, and cautiously add 10 ml of 30 percent hydrogen peroxide solution. After the initial reaction has subsided, add 6 ml of 70 percent perchloric acid, heat slowly until white fumes of perchloric acid are copiously evolved and continue heating gently for a few minutes to ensure decomposition of any excess peroxide. If the solution is brownish in colour due to non-decomposed organic matter, add a small amount of hydrogen peroxide solution and heat again to white perchioric acid fumes, repeating, if necessary until complete decomposition of the organic matter when a colourless solution is obtained. Cool, add 10 ml of distilled water and filter into a 200 mm x 25 mm test tube. Wash the filter paper with hot water until the filtrate measures 20 ml, add 20 ml of hydrochloric acid and mix.

F.1.2 Procedure

Place the test tubes containing the standard selenium solution and the sample solution in a water-bath, and heat until the temperature of the solution reaches 40 °C. To each tube, add 400 mg of ascorbic acid, stir until dissolved and maintain at 40 °C for 30 minutes. Cool the solution, dilute with distilled water to 50 ml and mix.

Any pink colour produced by the sample shall not exceed that produced by the standard selenium solution.

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Annex G
(normative)

Test for toluenesulfonamides

G.0 Two methods have been specified. Any method may be used depending upon the facilities available.

G.1 Method 1 – Gas Chromatography

G.1.1 Reagents

G.1.1.1 Methylene chloride: Use a suitable pure grade, equivalent to the product obtained by distillation in all glass apparatus.

G.1.1.2 Internal standard stock solution: Transfer 100 mg of 95 percent n-ticosane into a 10-ml volumetric flask. Dissolve in n-heptane, dilute to volume with the same solvent, and mix.

G.1.1.3 Stock standard preparation: Transfer 20 mg each of reagent grade o-toluenesulfonamide and p-toluenesulfonamide into a 10-ml volumetric flask. Dissolve in methylene chloride, dilute to volume with the same solvent, and mix.

G.1.1.4 Diluted standard preparation: Pipette into five lo-ml volumetric flasks, 0.1, 0.25, 1.0, 2.5 and 5.0 ml respectively, of the 'stock standard preparation'. Pipette 0.25 ml of the 'internal standard stock solution' into each flask, dilute each to volume with methylene chloride and mix. These solutions contain 250 µg of n-tricosane, plus respectively, 20, 50, 200, 500 and 1000 µg per ml of each toluenesulfonamide, plus 250 mg of n-tricosane.

G.1.1.5 Test preparation: Dissolve 2 g of the sample in 8 ml of 5 percent sodium bicarbonate solution. Mix the solution thoroughly with 10 g of chromatographic siliceous earth (Celite 545 or equivalent). Transfer the mix into a 25 mm x 250 mm chromatographic tube having a fritted glass disk and a Teflon stopcock at the bottom, and a reservoir at the top. Pack the contents of the tube by tapping the column on a padded surface, followed by tamping firmly from the top. Place 100 ml of methylene chloride in the reservoir and adjust the stopcock so that 50 ml of eluate is collected in 20 to 30 minutes. To the eluate, add 25 µl of 'internal standard stock solution'. Mix, and then concentrate the solution to a volume of 1 ml in a suitable concentrator tube fitted with a modified Snyder column, by using a Kontes tube heater maintained at 90 °C.

G.1.2 Procedure

G.1.2.1 Inject 2.5 µl of the test preparation (see G.1.1.5) into a suitable gas chromatograph equipped with a flame-ionization detector. The column should be of glass, approximately 3 m in length and 2 mm in inside diameter and packed with 3 percent phenylmethyl silicone on l00 - to 120-mesh equivalent to 150- to 125-micron IS test sieve and silanised calcined diatomaceous silica.

NOTE: The glass column should extend into the injector for on-column injection and into the detector base to avoid contact with metal.

The carrier is helium flowing at a rate of 30 ml per minute. The injection port, column, and detector are maintained at 225°C, 180°C and 250°C respectively. The instrument attenuation setting should be such that 2.5 µl of the 'diluted standard preparation' containing 200 µg per ml of each toluenesulfonamide gives a response of 40 to 80 percent of full-scale deflection. Record the chromatogram, note the peaks for o-toluenesulfonamide, p-toluenesulfonamide, and the n-tricosane internal standard, and calculate the areas for each peak by suitable means. The retention times for o-toluenesulfonamide, p-toluenesulfonamide, and n-tricosane are about 5, 6 and 15 minutes, respectively.

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G.1.2.2 In a similar manner, obtain the chromatograms for 2.5 µl portions of each of the live 'diluted standard preparations' and for each solution, determine the areas of the o-toluenesulfonanude, p-toluenesulfonamide, and n-tricosane peaks.

From the values thus obtained, prepare standard curves by plotting concentration of each toluenesulfonamide, in µg per ml versus the ratio of the respective toluenesulfonamide peak area to that of n-tricosane. From the standard curve determine the concentration, in µg per ml, of each toluenesulfonamide in the 'test preparation'. Divide each value by 2 to convert the result to parts per million of toluenesulfonamide in the 2 g sample taken for analysis.

NOTE: If the toluenesulfonamide content of the sample is greater than 500 parts per million, the impurity may crystallize out of the methylene chloride concentrate (see G.1.1.1). Although this level of impurity exceeds that permitted by the specification, the analysis may be completed by diluting the concentrate (usually 1: 10 is satisfactory) with methylene chloride containing 250 µg of n-tricosane per ml, and by applying appropriate dilution factors in the calculation. Care shall be taken obtain a homogeneous solution by completely dissolving any crystalline toluenesulfonamide.

G.2 Method 2 – Thin Layer Chromatography

G.2.1 Apparatus

G.2.1.1 Flat glass plates (200 mm x 100 mm)

G.2.1.2 Micropipette

G.2.1.3 Developing chamber lined with filter paper

G.2.2 Reagents

G.2.2.1 Silica gel

G.2.2.2 Chloroform

G.2.2.3 Methyl alcohol

G.2.2.4 Ammonia solution – strong

G.2.2.5 4–Sulphamoylbenzoic acid – reference material

G.2.2.6 Toluene–2–sulfonamide acid – reference material

G.2.2.7 Sodium hypochloride solution: Diluted with water to contain 0.5 percent (m/v) of available chlorine.

G.2.2.8 Potassium iodide

G.2.2.9 Starch mucilage

G.2.2.10 Glacial acetic acid

G.2.2.11 Solution A: 4 volumes of methyl alcohol plus 1 volume of acetone plus 0.5 percent (m/v) of the sample

G.2.2.12 Solution B: 4 volumes of methyl alcohol plus 1 volume of acetone plus 0.005 percent (m/v) of the 4-sulphamoylbenzoic acid

G.2.2.13 Solution C: 4 volumes of methyl alcohol plus 1 volume of acetone plus 0.005 percent (m/v) of the toluene-2-sulfonamide.

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G.2.3 Procedure

G.2.3.1 Prepare suspension of silica gel G. Spread the suspension on the plates about 0.25 mm thick. Allow to stand until the coating sets and then dry the plates at 105 °C to 110 °C for one hour. Protect the plates from moisture. Pour into the developing chamber sufficient quantity of mobile phase (100 volumes of chloroform + 50 volumes of methyl alcohol + 11.5 volume of strong ammonia solution) to form a layer about 15 mm deep. Close the tank for one hour at 20 °C to 27 °C. Remove the narrow strips of the coating, about 5 mm inside from the margins of the chromatoplate. Using micropipette apply separately to the chromatoplates 2 ml each of solutions A, B and C. These spots should be about 25 mm from the bottom of the plates and not less than 25 mm from the sides of the plates. The diameter of the spots should not be more than 6 mm. Dry the spots and place the chromatoplates in the developing chamber at 20 °C to 27 °C until the mobile phase has ascended to the 150 mm line.

Remove the plates and dry them in current of warm air. Then heat at 105 °C for five minutes. Spray the hot plates with the sodium hypochlorite solution. Dry in a current of cold air until sprayed area of the plate below the line of application give at most a faint blue colour with a drop of a mixture, prepared by dissolving 0.5 percent (m/v) of potassium iodide in starch mucilage containing 1 percent (m/v) of glacial acetic acid. Avoid prolonged exposure to cold air. Spray the plates with the same mixture. The spots in the chromatograms obtained with solution (B) and (C) should be more intense than any corresponding spots in the chromatogram obtained with solution (A).

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