

DRAFT UGANDA STANDARD

Second Edition
2017-mm-dd

Sodium bicarbonate — Specification



Reference number
DUS 572: 2017

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Foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Trade, Industry and Cooperatives established under Cap 327, of the Laws of Uganda, as amended. UNBS is mandated to co-ordinate the elaboration of standards and is

- (a) a member of International Organisation for Standardisation (ISO) and,
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- (c) the National Enquiry Point on TBT Agreement of the World Trade Organisation (WTO).

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 key stakeholders including government, academia, consumer groups, private sector and other interested parties.

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 Technical Committee UNBS/TC 2 [Technical Committee on Food and Agriculture standards], Subcommittee SC 6, [Food Additives & Contaminants Subcommittee].

This second edition cancels and replaces the first edition (US 572: 2006) which has been technically revised.

DRAFT UGANDA STANDARD

Introduction

Sodium bicarbonate (NaHCO_3), also known as bicarbonate of soda or baking soda is used as a leavening agent for baked products; an ingredient of baking powder; a reagent in analytical chemistry, and as an ingredient in the manufacture of effervescent salts and beverages. It also finds use in gold and platinum plating, the tanning industry, treating wool and silk, fire extinguishers and in the production of ceramics.

This standard has been revised to set requirements for the commonly used grades of sodium bicarbonate and to provide for methods of test that were lacking in the previous edition. This will facilitate fair trade, customer satisfaction and consumer safety.

DRAFT UGANDA STANDARD

Sodium bicarbonate — Specification

1 Scope

This standard specifies the requirements and methods of sampling and test for the grades of sodium bicarbonate defined herein.

2 Normative references

The following referenced 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.

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

AOAC 965.18 – *Phosphorus in baking powder – Gravimetric method*

AOAC 999.11 – *Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods*

AOAC 920.46 – *AOAC Official method: Metals and Other Constituents in Baking Powder*

US ISO 10523 – *Water quality – Determination of pH*

DUS ISO 2199 – *Sodium hydrogen carbonate for industrial use – Determination of Sodium hydrogen carbonate content – Titrimetric method*

DUS ISO 2460 – *Sodium hydrogen carbonate for industrial use – Determination of Iron content – 1, 10-phenanthroline photometric method*

US ISO 2482 – *Sodium chloride for industrial use - Determination of calcium and magnesium contents – EDTA complexometric methods*

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

3 Terms and definitions

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

Food grade packaging

packaging 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 Grades

There shall be three grades of the product;

- a) Pure grade: suitable for the fine chemical and pharmaceutical industry,
- b) Analytical reagent grade: suitable as a chemical reagent, and
- c) Refined grade: the food grade product, suitable for the baking powder industry and other food industries.

5 Requirements

5.1 General requirements

For all the three grades, the products shall be:

- i) in the form of small, opaque, monoclinic crystals or, as white, minutely crystalline powder
- ii) free from dirt and other foreign matter

5.2 Specific requirements

The respective product grades, when tested per the specified methods or methods elaborated in the respective annexes shall comply with the requirements specified in Table 1.

Table 1 — Requirements for Sodium bicarbonate

S/N	Characteristic	Requirements for			Method of Test / Reference to Annex
		Pure Grade	Analytical Reagent Grade	Refined Grade	
1.	Total alkalinity (as NaHCO ₃) percent by mass, <i>Min</i>	99.0	99.8	99.0	DUS ISO 2199
2.	pH (1 percent aqueous solution), <i>Max</i>	8.6	8.4	8.8	US ISO 10523
3.	Iron (expressed as Fe ₂ O ₃), percent by mass, <i>Max</i>	0.0015	0.0005	0.004	DUS ISO 2460
4.	Calcium and magnesium (as Ca), percent by mass, <i>Max</i>	-	0.015	-	US ISO 2482
5.	Phosphorus (as PO ₄), percent by mass, <i>Max</i>	-	0.0005	-	AOAC 965.18
6.	Insoluble matter, percent by mass, <i>Max</i>	0.05	0.010	0.100	AOAC 920.46 (C)
7.	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.05	0.003	0.07	AOAC 920.46 (I)
8.	Ammonium compounds	To pass test	To pass test	To pass test	AOAC 920.46 (A)
9.	Potassium (as K), percent by mass, <i>Max</i>	-	0.01	-	AOAC 920.46 (H)
10.	Substances reducing iodine, percent by mass, <i>Max</i>	-	0.0065	-	AOAC 920.44
11.	Chlorides (as Cl), percent by mass, <i>Max</i>	0.01	0.001	0.06	A
12.	Nitrates (as NO ₃), percent by mass, <i>Max</i>	-	0.001	-	B
13.	Silicates (as SiO ₂), percent by mass, <i>Max</i>	-	0.005	-	C

6 Contaminants

When tested in accordance with the cited methods, the contaminant levels shall conform to the requirements specified in Table 2.

Table 2 — Requirements for Sodium bicarbonate

Contaminant	Requirements for			Method of Test
	Pure Grade	Analytical Reagent Grade	Refined Grade	
Heavy metals (as Pb), mg/kg, <i>Max</i>	5	5	5	AOAC 999.11
Arsenic (as As) mg/kg, <i>Max</i>	1.5	0.4	1.5	AOAC 920.46 (B)

7 Packaging

The product shall be packaged in containers that maintain its integrity. The refined grade product shall be packaged in food grade containers.

8 Weights and Measures

The weight of the packages shall comply with the Weights and Measures regulations of Uganda.

9 Labelling

The packages shall be securely closed and legibly marked with the following information:

- a) Name of the product as “Baking soda”/ “Sodium hydrogen carbonate”/ “Sodium bicarbonate”/ “Bicarbonate of soda”
- b) The product grade
- c) Name and physical address of the manufacturer/distributor,
- d) Net weight in metric units,
- e) The list of requirements given in Table 1 depending on the product grade,
- f) The expiry date
- g) The batch number

10 Sampling

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

Annex A (normative)

Determination of Chlorides

A-1 General

Two methods have been prescribed for determination of chlorides; volumetric (Method A) and spectrophotometric (Method B). In case of any dispute, Method A shall be the reference method.

A-2 Method A (Volumetric Method)

A-2.1 For Pure and Refined Grades

A-2.1.1 Reagents

- Concentrated nitric acid (A.R. grade: 65 – 70%)
- Standard silver nitrate solution (0.1 N)
- Nitrobenzene
- Standard ammonium thiocyanate solution (0.1 N)
- Ferric ammonium sulphate indicator (5 percent)

A-2.1.2 Procedure

Weigh accurately 20 g of the product, dissolve it in distilled water and neutralise with concentrated nitric acid. Add 5 ml of nitric acid in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator until a permanent faint reddish brown colouration appears, signalling the end point.

A-2.1.3 Calculation

$$\text{Chlorides (as Cl) percent by mass} = \frac{3.547(10N_1 - VN_2)}{M}$$

Where, N_1 = normality of standard silver nitrate solution,

V = volume in ml of standard ammonium thiocyanate solution consumed in the titration,

N_2 = normality of standard ammonium thiocyanate solution, and

M = mass of product tested in grams.

A-2.2 For Analytical Reagent Grade

A-2.2.1 Apparatus

- Nessler cylinders (100 ml capacity).

A-2.2.2 Reagents

- Concentrated nitric acid (A.R grade: 65 – 70%)
- Silver nitrate solution (4 percent)
- Standard chloride solution

Dry sodium chloride at 250 °C before making the solution. Dissolve 1.649 g of sodium chloride in distilled water and make up the volume to 1000 ml. Pipette 10 ml from this solution and dilute to 1000 ml using distilled water. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

A-2.2.3 Procedure

Dissolve 1 g of the product in 30 ml of distilled water and add slight excess of concentrated nitric acid. Boil the solution, cool and add 1 ml of silver nitrate solution. Transfer into a Nessler cylinder and make up the volume to the 100 ml mark with distilled water. Carry out a control test using 1 ml of standard chloride solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity in both cylinders after 5 minutes.

The limit prescribed in Table 1 shall be taken as not exceeded if the turbidity produced in the test with the product is not greater than that produced in the control test.

A-3 Method B (Spectrophotometric method)**A-3.1 Outline of the Method**

To a neutralised solution of sodium bicarbonate, addition of mercuric thiocyanate and ferric ammonium sulphate, produces a highly coloured ferric thiocyanate complex, the intensity of which is proportional to the chloride content. The absorbance of this colour is measured at 450 nm wave length.

A-3.2 Apparatus

- Standard laboratory apparatus
- Spectrophotometer or Photocolorimeter
- Platinum evaporating dish (60 ml capacity).

A-3.3 Reagents

- Ferric ammonium sulphate solution (0.25 mol/l)

Dissolve 49.02 g of Ferric Ammonium Sulphate dodecahydrate [$\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] in 203 ml of dilute nitric acid (about 1.5 mol/l) and dilute to 500 ml with distilled water.

- Mercury (II) thiocyanate solution; Saturated solution in absolute alcohol.
- Standard sodium chloride solution

Accurately weigh 0.165 g of previously dried (at 500°C) and cooled A.R. grade sodium chloride into a beaker. Dissolve it in distilled water and quantitatively transfer into a one-litre volumetric flask, dilute to the mark and mix well. 1 ml of this solution = 100 µg of Cl.

- Dilute standard sodium chloride solution

Transfer 50 ml of standard sodium chloride solution into a 500 ml volumetric flask, dilute with distilled water to the mark and mix well. 1 ml of this solution = 10 µg of Cl.

- Sulphuric Acid Solution (Approximately 1 mol/l)

Pour 56 ml of concentrated sulphuric acid (A.R. grade) into 944 ml of distilled water slowly. Mix and cool.

A-3.4 Procedure

A-3.4.1 Calibration

Into a series of six 50 ml volumetric flasks, transfer standard sodium chloride solution as given below.

Standard Sodium Chloride Solution, ml	Corresponding Mass of Chloride (as Cl), µg
0(compensation)	0
2	20
4	40
6	60
8	80
10	100

To each volumetric flask, add 2 ml of ferric ammonium sulphate solution followed by 2 ml of mercury thiocyanate solution. Dilute with distilled water to the mark and mix well. Allow to stand for 15 minutes, transfer to the cell of spectrophotometer and measure absorbance at 450 nm wave length with compensation solution in reference cell.

Plot chloride content in micrograms as abscissa against the corresponding absorbance values as ordinates.

A-3.4.2 Determination

Weigh suitable mass of sodium bicarbonate sample containing 50 to 150 µg of chloride (as Cl) correct to 1 mg and transfer into the platinum evaporating dish. Wet it with 15 to 20 ml of distilled water and slowly neutralise with sulphuric acid solution. Add a few drops of the acid in excess. Using a hot water bath, evaporate the mixture close to dryness. Add 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml volumetric flask, cool and make up to the mark with distilled water and mix well.

Filter through Whatman No. 41 filter paper and discard a small volume of initial filtrate. Transfer a suitable volume of the filtrate (25 to 40 ml) into a 50 ml volumetric flask. Add 2 ml of ferric ammonium sulphate solution followed by 2 ml of mercury thiocyanate solution. Dilute to the mark with distilled water and mix well.

Allow the mixture to stand for 15 minutes, transfer to the spectrophotometer cell and measure absorbance at 450 nm wave length with distilled water in the reference cell.

A-3.4.3 Blank Test

Carry out a blank test using exactly the same procedure in **A-3.4.2** using similar quantities of reagents, with distilled water serving as the test solution.

A-3.4.4 Calculations

$$\text{The chloride content (as Cl), percent by mass} = \frac{M_1 - M_2}{10000M_0}$$

where, M_1 = mass in micrograms of chloride corresponding to absorbance of test solution

M_2 = mass in micrograms of chloride corresponding to absorbance of blank solution, and

M_0 = mass in grams of sodium bicarbonate in the test portion of the aliquot used for colour development.

ANNEX B (normative)

Test for Nitrates

B-1 Reagents

- Dilute Sulphuric Acid (5 N)
- Concentrated hydrochloric acid (A.R. grade: 35 – 37%)
- Potassium nitrate solution (0.001 N)
- Concentrated sulphuric acid (A.R. grade: 98%)
- Standard Indigo carmine solution

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid and add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to produce 1000 ml. Standardise the solution so that 10 ml added to 3.3 ml of potassium nitrate solution is just decolourised on adding 13 ml of concentrated sulphuric acid and heating to boiling. One millilitre of this solution is equivalent to 0.02 mg of nitrate (as NO_2).

B-2 Procedure

Dissolve 2.0 g of the sample in 10 ml of dilute sulphuric acid and 10 ml of standard indigo carmine solution followed by 10 ml of concentrated sulphuric acid and heat to boiling.

The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced in the test with the sample does not entirely disappear.

ANNEX C (normative)

Test for Silicates

C-1 General

Two methods have been prescribed for determination of silicates; visual colour comparison (Method A) and spectrophotometric method (Method B). In case of a dispute, Method A shall be reference method.

C-2 Method A (Visual Colour Comparison)

C-2.1 Apparatus

- Nessler cylinders (50 ml)

C-2.2 Reagents

- Dilute sulphuric acid (1 N)
- Ammonium molybdate solution

Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

- Sodium citrate solution

Dissolve 8 g of sodium citrate ($\text{Na}_3\text{C}_6\text{H}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$) in 100 ml of distilled water.

- Potassium metabisulphite solution

Dissolve 20 g of potassium metabisulphite and 1 g of hydroquinone in 100 ml of distilled water.

- Anhydrous sodium carbonate
- Standard silicate solution

Fuse 0.10 g of pure, precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the mixture with water. Neutralise with dilute sulphuric acid and dilute to 1000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of silica (as SiO_2). Store in a polyethylene bottle.

C-2.3 Procedure

Dissolve 2.0 g of the product in 20 ml of distilled water in a platinum dish, neutralise with about 10 ml of dilute sulphuric acid. Add 2 ml of the acid in excess and dilute to 50 ml with distilled water. Transfer 10 ml of this solution into a Nessler cylinder. Add 1 ml of dilute sulphuric acid and 5 ml of ammonium molybdate solution. Allow to stand for 5 minutes and then add 5 ml of sodium citrate solution followed by 5 ml of potassium metabisulphite solution. Dilute to 50 ml. Carry out a control test in another Nessler cylinder using 2 ml of standard silicate solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Stand both the cylinders in a boiling water bath for 5 minutes, cool and compare the colour produced in the two cylinders after 5 minutes.

The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the product is not deeper than that produced in the control test.

C-3 Method B (Spectrophotometric Method)

C-3.1 Outline of the Test Method

This method specifies a reduced molybdosilicate spectrophotometric method for the determination of soluble silica. It is applicable for materials with silica content in presence of phosphate.

Depolymerisation of any polymerised silica by treatment with hydrofluoric acid and boric acid. Formation of the oxidized molybdosilicate complex (yellow) under well-defined conditions of acidity (PH 1 ± 0.05).

Selective reduction of the complex in a strong sulphuric acid medium in the presence of oxalic acid to eliminate interference of phosphate.

Spectrophotometric measurement of the coloured complex at wavelength of maximum absorption (about 795 nm).

C-3.2 Reagents

- Distilled water
- Sulphuric acid (2 mol/l)

Add 112 ml of concentrated Sulphuric acid (A.R. grade) in 888 ml of distilled water slowly by constant stirring cool and mix well.

- Sulphuric acid (8 mol/l)

Add 448 ml of concentrated Sulphuric acid (A.R grade) in 552 ml of distilled water slowly by constant stirring, cool and mix well.

- Sodium molybdate (274 g/l Solution)

Dissolve 27.4 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in warm distilled water in a beaker of material free from silica of suitable capacity and after cooling, dilute to 100 ml. Store, if necessary, in a bottle of material free from silica and filter, if necessary, before use.

- Oxalic acid (100 g/l solution)

Dissolve 10 g of oxalic acid dihydrate ($\text{COOH} \cdot \text{COOH} \cdot 2\text{H}_2\text{O}$) in 100 ml distilled water.

- Ascorbic acid (25 g/l Solution)

Dissolve 2.5 g of ascorbic acid in water and dilute to 100 ml. Store the solution in a bottle of material free from silica and protect from light. Prepare this solution at the time of use or as an alternative, prepare reduction solution.

- Reduction solution

Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 50 ml distilled water. Then add 1.5 g of 4-amino-3-hydroxyl naphthalene-1-sulphonic acid ($\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$) and dissolve by grinding.

Dissolve 90 g of anhydrous metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 900 ml of distilled water.

Mix the two prepared solutions and dilute to 1000 ml. Filter if turbid and store in an opaque bottle free from silica.

- Sodium fluoride solution (20 g/l solution)
- Boric acid solution (Saturated solution at ambient temperature)
- Standard Silica Solution (0.20 g SiO_2 per litre)

Weigh to the nearest 1 mg 0.20 g SiO₂ obtained by heating pure silicic acid (H₂SiO₃) at 1000 °C until constant mass (consecutive weighings should not differ by more than 1 mg) and cooling in a desiccator into a platinum crucible of convenient capacity. Add 2.0 g of anhydrous Na₂CO₃ to the crucible, mix well preferably with a platinum spatula and carefully fuse the mixture. Add warm water directly to the crucible, heat gently until the contents are completely dissolved and transfer quantitatively to a beaker of suitable capacity made of silica free material. Cool, dilute the solution to about 500 ml, transfer quantitatively to a 1000 ml volumetric flask, dilute to the mark and mix well. Transfer the solution immediately to a bottle of material free from silica. 1 ml of this solution = 0.2 mg SiO₂.

- Standard silica solution (2.0 mg SiO₂ per litre)

Dilute 20.0 ml of standard silica solution above to 1000 ml volumetric flask using distilled water. Prepare this solution at the time of use. 1 ml of this solution = 2.0 µg SiO₂.

C-3.3 Apparatus

- Standard laboratory apparatus
- pH meter
- Spectrophotometer or Photocolorimeter
- Platinum crucible with lid of suitable size

C-3.4 Procedure

C-3.4.1 Preparation of the calibration graph

C-3.4.1.1 Preliminary test for control and correction of pH

Place 10.0 ml of the standard silica solution (2.0 mg SiO₂ per litre) in a beaker. Add the distilled water to bring volume to 15 ml and then add 5 ml of sodium molybdate solution, 1 ml of sodium fluoride and 5 ml of boric acid solution. Mix and measure pH with a pH meter. Adjust pH to 1 ± 0.05 by adding slowly using a graduated pipette or a burette drop by drop, mixing after each addition, the necessary quantity of 2 mol/l sulphuric acid solution (about 4.5 ml would be required).

Record the actual volume of sulphuric acid required and discard the solution.

C-3.4.1.2 Preparation of the standard calorimetric solution

In to a series of six 50 ml beakers, transfer standard silica solution as given below:

Standard silica solution, ml	Corresponding mass of silica, µg
0(compensation)	0.0
2.0	4.0
4.0	8.0
6.0	12.0
8.0	16.0
10.0	20.0

To each beaker, add distilled water to make up volume to 15.0 ml. Then add the volume of sulphuric acid solution as calculated above (C-3.4.1.1) for pH correction, mix well. Add 1 ml of sodium fluoride solution, mix and allow to stand for 5 min.

C-3.4.1.3 Colour Development

To each beaker, add 5 ml of boric acid solution and 5 ml of sodium molybdate solution, mix and allow to stand for 10 minutes. Add 5 ml of oxalic acid solution and 8 ml of sulphuric acid solution (8 mol/l). Allow to stand for 2 minutes, add 2 ml of ascorbic acid solution or 2 ml of reduction solution and allow to stand for 10 minutes. Transfer the solutions quantitatively to a series of 50 ml volumetric flasks, dilute to marks and mix.

C-3.4.1.4 Measurements

Carry out the spectrophotometric measurements with a spectrophotometer (or photocolourimeter) at 795 nm wavelength, after having adjusted the instrument zero absorbance with the compensation solution, use cells of 4 to 5 cm optical path length.

Plot a graph with μg of SiO_2 contained in 50 ml as abscissae and corresponding absorbance values as ordinates.

C-3.4.2 Determination

C-3.4.2.1 Test solution

Weigh accurately (to the nearest 1 mg) an amount of Sodium bicarbonate containing between 4 to 20 μg of SiO_2 and transfer into a 100 ml beaker, dissolve in minimum amount of distilled water and neutralise with 1 mol/l sulphuric acid (about 6 ml) and further add 0.5 ml acid in excess. Heat on a water bath to expect all CO_2 liberated and cool. Ensure that the total volume is around 15 ml. Then add the volume of sulphuric acid solution (2 mol/l) used for pH correction in the preliminary test and 1 ml of sodium fluoride solution, mix and allow to stand for 5 minutes. Develop colour by the procedure given in (C-3.4.1.2).

C-3.4.2.2 Blank test

Carry out a blank test following the same procedure and using the same quantities of all reagents used for the determination, replacing the test solution by the same volume of distilled water.

C-3.4.2.3 Spectrophotometric measurements

Carry out the spectrophotometric measurements by the procedure specified in (C-3.4.1.4) at 795 nm wave length after adjusting the instrument to zero absorbance against water, use the same size of the cell used for calibration.

C-3.4.3 Calculation

By means of the calibration graph, determine the mass of SiO_2 content corresponding to the aliquot used for colour development.

$$\text{The silica content is expressed as: Silica (as SiO}_2\text{), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10000}$$

where,

M_1 = mass in micrograms of SiO_2 in the test portion of the aliquot,

M_2 = mass in micrograms of SiO_2 in the blank, and,

M_0 = mass of product sample in grams in the aliquot portion used for colour development.

Bibliography

- [1] IS 2124 (2000): *Sodium Bicarbonate (Second Revision)*
- [2] AOAC (2000): *Official methods of analysis of AOAC International (17th ed.)*, Gaithersburg, Md.

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