SRI LANKA STANDARD 861 : 1989

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SPECIFICATION FOR
SODIUM BISULFITE AND SODIUM METABISULFITE (FOOD GRADE)

SRI LANKA STANDARDS INSTITUTION

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SLS 861:1989

Gr. 7

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SRI LANKA STANDARD

SPECIFICATION FOR SODIUM BISULFITE AND SODIUM METABISULFITE (FOOD GRADE)

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1989-10-11, after the draft, finalized by the Drafting Committee on Food Additives, had been approved by the Agricultural and Food Products Divisional Committee.

Sodium bisulfite and sodium metabisulfite are used as antimicrobial preservatives in the food industry. This specification is therefore formulated for the benefit of quality control exercises which are carried out to ensure the identity and purity of the products.

This specification is subject to the provisions of the Food Act No. 26 of 1980 and the regulations framed thereunder.

All standard values in this specification are given in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with the final value, observed or calculated, expressing the result of a test or an analysis shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification the assistance obtained from the publications of the Food and Agriculture Organization and the Bureau of Indian Standards is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements, methods of sampling and test for food grade sodium bisulfite (Sodium hydrogen sulfite, sodium acid sulfite) and food grade sodium metabisulfite (Sodium pyrosulfite).

2 REFERENCES

CS 102 Presentation of numerical values.
SLS 312 Determination of arsenic.
SLS 428 Random sampling methods.

3 REQUIREMENTS

3.1 General requirements

3.1.1 *Description*

3.1.1.1 Sodium bisulfite shall be in the form of white crystals or granular powder having an odour of sulfur dioxide.

3.1.1.2 Sodium metabisulfite shall be in the form of white crystals or crystalline powder having an odour of sulfur dioxide.

3.1.2 *Solubility*

3.1.2.1 Sodium bisulfite shall be freely soluble in water.

3.1.2.2 Sodium metabisulfite shall be freely soluble in water and slightly soluble in ethanol.

3.1.3 *Identification*

3.1.3.1 Sodium
A yellow crystalline precipitate shall be formed, when tested in accordance with the method described in A.1.

3.1.3.2 Bisulfite and sulfite
Sulfur dioxide shall be generated, when tested in accordance with the method described in A.2.

3.2 Chemical requirements

Sodium bisulfite and sodium metabisulfite shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Column 5 of the table.

TABLE 1 - **Chemical requirements for sodium bisulfite and sodium metabisulfite**

Sl. No.	Characteristic	Requirement		Method of test
		Sodium bisulfite	Sodium metabisulfite	
(1)	(2)	(3)	(4)	(5)
i)	Sodium bisulfite as $NaHSO_3$, per cent by mass, min.	95	-	Appendix B
ii)	Sodium metabisulfite as $Na_2S_2O_5$, per cent by mass, min.	-	95	Appendix B
iii)	pH value, at 27 ± 2 °C	2.5 to 4.5	4.0 to 4.5	Clause 6.3
iv)	Water insoluble matter, per cent by mass, max.	0.05	0.05	Appendix C
v)	Thiosulfate, per cent by mass, max.	-	0.1	Appendix D
vi)	Iron, mg/kg, max.	50	50	Appendix E
vii)	Arsenic, mg/kg, max.	3	3	Appendix F
viii)	Heavy metals (as lead), mg/kg, max.	10	10	Appendix G

4 PACKAGING AND MARKING

4.1 Packaging

4.1.1 Sodium bisulfite and sodium metabisulfite shall be securely packed in well-filled containers with minimum access to light and air.

4.1.2 The containers shall be such as to preclude contamination of the contents with metals or other impurities and strong enough to withstand pressure in handling.

4.2 Marking

Each container shall be marked or labelled legibly and indelibly with the following:

a) Name of the product, including the words `Food Grade';
b) Brand name or trade name, if any;
c) Net mass, in grams or in kilograms;
d) Name and address of the manufacturer and distributor, including the country of origin;
e) Batch or code number; and
f) Date of manufacture.

5 SAMPLING

5.1 Lot

In any consignment all the containers of the same product, same size and belonging to one batch of manufacture or supply shall constitute a lot.

5.2 Scale of sampling

5.2.1 Samples shall be tested from each lot for ascertaining its conformity to the requirements of this specification.

5.2.2 The number of containers to be selected from a lot shall be in accordance with the following table.

TABLE 2 - Scale of sampling

Number of containers in the lot (1)	Number of containers to be selected (2)
Up to 100	5
101 to 280	10
281 to 500	15
501 to 1 200	25
1 201 to 3 200	35
3 201 and above	50

5.2.3 The containers shall be selected at random. In order to ensure randomness of selection, tables of random numbers as given in SLS 428 shall be used.

5.3 Preparation of samples

Approximately an equal quantity of material shall be drawn from each of the containers selected as in 5.2.2, with the help of an appropriate sampling instrument. The material thus obtained shall be mixed to form a composite sample of approximately 150 g.

5.4 Reference sample

If a reference sample is required the size of the composite sample shall be approximately 450 g. The sample thus obtained shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for reference.

5.5 Number of tests

5.5.1 Each container selected as in 5.2.2 shall be inspected for packaging and marking requirements.

5.5.2 The composite sample obtained as in 5.3 shall be tested for all the requirements in 3.1 and 3.2.

6 METHODS OF TEST

6.1 Tests shall be carried out as prescribed in the Appendices A, B, C, D, E, F, G, and 6.3 of this specification.

6.2 Unless otherwise stated, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

6.3 Determine the pH value of 1 : 9 solution of the sample in distilled water at 27 ± 2 °C, using a pH meter.

7 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied.

7.1 Each container inspected as in 5.5.1 satisfies the relevant requirements.

7.2 The test results on the composite sample when tested as in 5.5.2 satisfy the relevant requirements.

APPENDIX A
IDENTIFICATION TESTS

A.1 TEST FOR SODIUM

A.1.1 *Reagents*

A.1.1.1 *Dilute acetic acid,* approximately 6 percent (V/V).

A.1.1.2 *Acetic acid,* approximately 5 mol/l.

A.1.1.3 *Uranyl zinc acetate solution*

Dissolve 10 g of uranyl acetate [($CH_3COO)_2$ $UO_2.2H_2O$] in 50 ml of water and 5 ml of acetic acid (A.1.1.2). Dissolve 30 g of Zinc acetate by heating with 30 ml of water and 3 ml of acetic acid. Mix the two solutions, allow to cool and filter.

A.1.2 *Procedure*

Acidify the sample with dilute acetic acid solution (A.1.1.1) and filter if necessary. Add dilute hydrochloric acid or dilute nitric acid and convert the bisulfite or metabisulfite into chloride or nitrate. Add uranyl zinc acetate solution (A.1.1.3).

A.2 TEST FOR BISULFITE AND SULFITE

A.2.1 *Reagents*

A.2.1.1 *Dilute sulfuric acid,* approximately 1 mol/l.

A.2.1.2 *Mercuric chloride solution,* saturated.

A.2.1.3 *Potassium iodate solution,* saturated

A.2.1.4 *Starch solution*

Disperse 1 g of soluble starch with a small quantity of water and pour this into 100 ml of boiling water and boil for 1 minute. Prepare freshly before use.

A.2.2 *Procedure*

Heat the sample with dilute sulfuric acid solution (A.2.1.1) and recognize the evolution of sulfur dioxide by:

i) its odour
ii) the blackening of filter paper moistened with mercuric chloride solution (A.2.1.2).
iii) the development of a blue colour on filter paper treated with potassium iodate (A.2.1.3) and starch solution (A.2.1.4).

APPENDIX B
DETERMINATION OF SODIUM BISULFITE AND SODIUM METABISULFITE

B.1 REAGENTS

B.1.1 *Iodine solution,* standard volumetric solution, 0.05 mol/l.

B.1.2 *Sodium thiosulfate solution,* $c(Na_2S_2O_3) = 0.1$ mol/l.

B.1.3 *Hydrochloric acid,* concentrated, rel. den. = 1.18.

B.2 PROCEDURE

Weigh, to the nearest milligram, about 0.2 g of the sample and add into a glass stoppered flask containing 50.0 ml of iodine solution (B.1.1). Stopper the flask and allow it to stand for 5 minutes. Add 1 ml of hydrochloric acid, and titrate the excess iodine with sodium thiosulfate solution (B.1.1) using starch solution (A.2.1.4) as an indicator. Carry out a blank determination without the sample in similar manner under the same conditions.

B.3 CALCULATION

B.3.1 *For sodium bisulfite*

1 ml of 0.05 mol/l iodine solution is equivalent to 5.203 mg of sodium bisulfite.

$$\text{Sodium bisulfite, as } NaHSO_3\text{, per cent by mass} = \frac{5.203 \times c\,(V_0 - V)}{m}$$

B.3.2 *For sodium metabisulfite*

1 ml of 0.05 mol/l iodine solution is equivalent to 4.753 mg of sodium metabisulfite.

$$\text{Sodium metabisulfite, as } Na_2S_2O_5\text{, per cent by mass} = \frac{4.753 \times c\,(V_0 - V)}{m}$$

Where,

m is the mass, in g, of the test portion;
c is the concentration, in mol/l, of the sodium thiosulfate solution;
V_0 is the volume, in ml, of sodium thiosulfate required for the blank determination; and
V is the volume, in ml, of sodium thiosulfate required for the sample.

APPENDIX C
DETERMINATION OF WATER INSOLUBLE MATTER

C.1 PROCEDURE

Weigh, to the nearest milligram, about 10 g of the sample and dissolve in 50 ml of water. Filter through a weighed sintered glass crucible (No. 4) previously washed and dried at 105 ± 5 °C. Wash the residue thoroughly with water. Dry the residue at 105±5 °C for 2 hours. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 1 mg.

C.2 CALCULATION

$$\text{Water insoluble matter, per cent by mass} = \frac{m_1 - m_0}{m_2} \times 100$$

where,

m_0 is the mass, in g, of the crucible ;
m_1 is the mass, in g, of the crucible with the residue ; and
m_2 is the mass, in g, of the sample taken for the test.

APPENDIX D
DETERMINATION OF THIOSULFATE

D.1 REAGENTS

D.1.1 *Potassium bromide and mercuric chloride mixture*

Dissolve 25 g of potassium bromide and 25 g of mercuric chloride in 900 ml of distilled water at about 50 °C and cool. Dilute to one litre and allow to stand overnight. Filter, if necessary to obtain a clear solution.

D.1.2 *Sodium thiosulfate solution*, standard volumetric solution, 0.000 2 mol/l.

D.2 PROCEDURE

D.2.1 *Preparation of sample solution*

Weigh, to the nearest milligram, about 5 g of the sample, dissolve in distilled water and dilute to 100 ml. Transfer 10 ml of potassium bromide and mercuric chloride mixture (D.1.1) into a 50-ml Nessler tube or any other suitable glass tube and add slowly 1 ml of the test solution using a pipette.

D.2.2 *Preparation of standard solution*

Transfer 10 ml of potassium bromide and mercuric chloride mixture (D.1.1) into a 50-ml Nessler tube or any other suitable glass tube and add slowly 2 ml of sodium thiosulfate solution (D.1.2) using a pipette.

Allow both tubes to stand for 15 minutes without agitation, then agitate carefully to distribute opalescence. The opalescence of the tube containing the sample solution shall not be more than that of the tube containing the standard solution.

APPENDIX E
DETERMINATION OF IRON

E.1 REAGENTS

E.1.1 *Bromine solution,* saturated

Dissolve 2 to 3 ml of bromine (Br_2) in 100 ml of cold water in a glass stoppered bottle. Store it in a cold place protected from light.

E.1.2 *Ammonium thiocyanate,* 76 g/l solution.

E.1.3 *Standard iron solution;* 1 ml of solution is equivalent to 0.01 mg of iron.

Dissolve 0.70 g of ferrous ammonium sulfate $FeSO_4.(NH_4)_2SO_4.6H_2O$, in 50 ml of water and add 20 ml of dilute sulfuric acid (1:15). Dilute to 1000 ml with water and mix thoroughly. Dilute 10 ml of this solution to 100 ml with water.

E.1.4 *Hydrochloric acid,* concentrated, rel. den. = 1.18.

E.1.5 *Ammonium persulfate*

E.2 PROCEDURE

Weigh, to the nearest milligram, about 0.5 g of the sample and add 2 ml of hydrochloric acid (E.1.4). Evaporate to dryness on a steam bath. Dissolve the residue in 2 ml of hydrochloric acid and 20 ml of water, and add few drops of bromine solution (E.1.1). Boil the solution to remove the bromine. Cool and dilute with water to 25 ml. Add 50 mg of ammonium persulfate and 5 ml of ammonium thiocyanate solution (E.1.2). Any red colour produced shall not exceed that of a control solution made in the same way as the test solution, containing 2.5 ml of iron standard solution (E.1.3), instead of the sample.

APPENDIX F
DETERMINATION OF ARSENIC

F.1 PROCEDURE

F.1.1 *Preparation of test solution*

Weigh, to the nearest milligram, about 2.5 g of the sample in a porcelain dish. Add 10 ml of water, 1.25 g of potassium chlorate and 16 ml of 32 per cent (V/V) hydrochloric acid and mix well. Heat to expel chlorine and remove the last traces of chlorine with a few drops of stannous chloride solution. Add 25 ml of water.

F.1.2 Proceed as given in Method 2.1 (Modified Gutzeit method) of SLS 312 : 1976.

APPENDIX G
DETERMINATION OF HEAVY METALS

G.1 REAGENTS

G.1.1 *Ammonia solution*

Dilute 400 ml of reagent grade ammonium hydroxide (rel. den = 0.91) to 1000 ml with water.

G.1.2 *Lead nitrate stock solution*

Dissolve 159.8 mg of lead nitrate, $Pb(NO_3)_2$, in 100 ml of water containing 1 ml of nitric acid, then dilute with water to 1000 ml and mix well. This solution shall be prepared and stored in a glass container which is free from lead salts.

G.1.3 *Standard lead solution*, 1 ml of solution is equivalent to 10 µg of lead (Pb^{2+}).

Dilute 10.0 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Prepare freshly before use.

G.1.4 *Dilute acetic acid*, 60 g/l solution.

G.1.5 *Hydrogen sulfide solution*, saturated.

Store in a small, dark, amber-coloured bottle, filled nearly to the top. It is unsuitable unless it possesses a strong odour of hydrogen sulfide. Store in a cold dark place.

G.2 PROCEDURE

G.2.1 *Preparation of Solution A (Control)*

Pipette 2.0 ml of standard lead solution into a 50-ml Nessler tube and add water to make 25 ml. Adjust the pH to between 3.0 and 4.0 (short-range pH indicator paper) by the addition of dilute acetic acid solution (G.1.4) or ammonia solution (G.1.1). Dilute with water to 40 ml and mix the solution.

G.2.2 *Preparation of Solution B (Test)*

Weigh, to the nearest milligram, about 2 g of the sample and dissolve it in 10 ml of water. Evaporate to dryness on a steam bath and dissolve the residue in 25 ml of water. Place this 25 ml in a 50 ml Nessler tube similar to the one used for the Solution A. Adjust the pH to between 3.0 and 4.0 (short-range pH indicator paper) by the addition of dilute acetic acid solution (G.1.4) or ammonia solution (G.1.1). Dilute to 40 ml with water and mix the solution.

To each tube add 10 ml of freshly prepared hydrogen sulfide solution (G.1.5), mix, allow to stand for 5 minutes and observe downward over a white surface. The colour of the Solution B, shall be not darker than that of the Solution A.

SRI LANKA STANDARDS INSTITUTION

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