SRI LANKA STANDARD 860 : 1989

UDC 664. 8. 035

# SPECIFICATION FOR POTASSIUM METABISULFITE (FOOD GRADE)

**SRI LANKA STANDARDS INSTITUTION** 

- Blank Page -

# SPECIFICATION FOR POTASSIUM METABISULFITE (food grade)

SLS 860:1989

Gr. 7

Commight Reserved SRI LANKA STANDARDS INSTITUTION 53, Dharmapala Mawatha, Colombo 3, Sri Lanka.

# DRAFTING COMMITTEE ON FOOD ADDITIVES

# CONSTITUTION

# CHA IRMAN

REPRESENTING

Mr T. Kandasamy

Personal Capacity

## MEMBERS

Mr	Ρ.	Ν.	Υ.	Abevgunawardena

Mr D.A.M. Arsecularatne

- Mrs M.J. de Silva
- Mrs S. Durairatnam
- Miss P. Motha
- Dr (Mrs) S. Samarasinghe
- Dr V.K. Samuel
- Mr G.H. Sarath Kumara
- Mrs K. Selvarajah
- Mr W.A.P. Silva
- Mr K. Sivarajah

Lanka Canneries Limited Ceylon Cold Stores Limited City Analyst's Laboratory Milk Industries of Lanka Company Limited Motha Confectionery Works Limited University of Sri Jayawardenepura Medical Research Institute Pure Beverages Company Limited Ceylon Institute of Scientific and Industrial Research Food Processing Company (Ceylon) Limited Government Analyst's Department

# TECHNICAL SECRETARIAT SRT LANKA STANDARDS INSTITUTION

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This standard does not purport to include all the necessary provisions of a contract.

3

# SRI LANKA STANDARD SPECIFICATION FOR POTASSIUM METABISULFITE (FOOD GRADE)

#### FOREWORD

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.

Potassium metabisulfite is used as an antimicrobial preservative as well as an antibrowning agent 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 product.

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

All standard values used throughout 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 (FAO) and the Bureau of Indian Standards is gratefully acknowledged.

## **1** SCOPE

This specification prescribes the requirements, methods of sampling and test for food grade potassium metabisulfite (potassium pyrosulfite).

# 2 REFERENCES

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

# 3 REQUIREMENTS

# 3.1 General

3.1.1 Description

Potassium metabisulfite shall be in the form of white free flowing crystals, crystalline powder, or granules, usually having an odour of sulphur dioxide.

3.1.2 Solubility.

Potassium metabisulfite shall be soluble in water and insoluble in ethanol.

3.1.3 Identification

3.1.3.1 Potassium

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

3.1.3.2 Sulfite

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

# 3.2 Chemical requirements

Potassium metabisulfite shall also comply with the requirements given in Table 1 when tested in accordance with the methods given in Column 4 of the table.

OT A 12 T 13	- 1		cm · · ·		-			
JADLP.		-	Chemical	requiremente	For	Dotectium	makely and the	-
	-		ANTONITCOT	redurrements	101	LOCUSSIUM	mecadishittir	e
	-		ALL AND TO GT	requirements	TAT	TOLOSSIU	meranisuille	e

S1. No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Potassium metabisulfite, as		
	K <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , per cent by mass, min.	90	Appendix B
ii)	Water insoluble matter, per		
iii)	cent by mass, max. Thiosulfate, per cent by mass,	0.05	Appendix C
	max.	0.1	Appendix D
iv)	Iron, mg/kg, max.	10	Appendix E
• <b>v</b> ) •	Arsenic, mg/kg, max.	3	Appendix F
vi)	Heavy metals (as lead), mg/kg,		
4. A. A. J. A.	max.	10	Appendix G

## .4 PACKAGING AND MARKING

# 4.1 Packaging

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

h 1 ? The container 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 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 given 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 and G of this specification.

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

## 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.

7

# APPENDIX A IDENTIFICATION TESTS

and the second and and

# A.1 TEST FOR POTASSIUM

1 1 Programme

A.1.1.1 Sodium hydrogen tartrate solution, saturated.

a sector of a sector

A.1.1.2 Ethanol.

A.1.2 Procedure

To 1 per cent solution (m/m) of the sample add sodium hydrogen tartrate solution (A.1.1.1) and ethanol (A.1.1.2) in 1:1:1 ratio and shake well.

### A.2 TEST FOR SULFITE

A.2.1 Reagents

A.2.1.1 Hydrochloric acid, 100 g/l solution.

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, pour this into 100 ml of boiling water and boil for 1 minute. This solution should be prepared fresh before use.

# A.2.2 Procedure

Add hydrochloric acid (A.2.1.1) to the sample. Recognize the evolution of sulfur dioxide by

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

# APPENDIX B

# DETERMINATION OF POTASSIUM METABISULFITE CONTENT

## **B.1 REAGENTS**

Pol 1 Hydrochloric acid solution

Prepare by diluting 236 ml of concentrated hydrochloric acid (rel. den. = 1.18) with sufficient water to make 1000 ml.

B.1.2 Iodine solution, standard volumetric solution, 0.05 mol/1.

B.1.3 Sodium this sulfate solution,  $c(Na_2S_2O_3) = 0.1 \text{ mol}/1$ .

#### **B.2 PROCEDURE**

Weigh, to the nearest milligram, about 0.25 g of the sample and transfer into a glass stoppered flask containing 50.0 ml of iodine solution (B.1.2) and 150 ml of water. Stopper the flask and allow it to stand for 5 minutes. Add 1 ml of dilute hydrochloric acid solution (B.1.1) and titrate the excess iodine with the sodium thiosulfate solution (B.1.3), using starch solution (A.2.1.4) as the indicator. Carry out a blank determination without the sample, in similar manner under the same conditions.

# **B.3 CALCULATION**

1 ml of 0.05 mol/1 iodine is equivalent to 5.558 mg of potassium metabisulfite.

Potassium metabisulfite, as  $K_2S_2O_5$ , =  $5.558 \times c (V_0 - V)$ per cent by mass 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  $105 \pm 5$  °C. Wash the residue thoroughly with water. Dry the residue at  $105 \pm 5$  °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

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  $^{\rm O}$ C and cool. Dilute to 1 litre and allow to stand overnight. Filter if necessary to obtain a clear solution.

D.1.2 Sodium thiosulfate solution, standard volumetric solution, 0.0002 mol/1.

# D.2 PROCEDURE

## **D.2.1** Preparation of sample solution

Weigh, to the nearest 0.1 g, 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 test 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 test tube and add slowly 2 ml of sodium thiosulfate (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, 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,  $\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}_5$ 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 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 thiccyanate 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, but containing 0.5 ml of iron standard solution (5  $\mu$ g Fe) (E.1.3), instead of the sample.

# APPENDIX F DETERMINATION OF ARSENIC

**F:1 PROCEDURE F:1 Preparation of the 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

n de la sec Angle angles Naso de la

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 Hydrochloric acid, concentrated, rel. den. = 1.18

G.1.3 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.1.4 Standard lead solution, 1 ml of solution is equivalent to 10  $\mu$ g of lead ion (Pb<sup>2+</sup>).

Dilute 10.0 ml of lead nitrate stock solution (G.1.3), accurately measured, with water to 100 ml. Prepare freshly before use.

G.1.5 Dilute acetic acid, 60 g/l solution.

G.1.6 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 (shortrange pH indicator paper) by the addition of dilute acetic acid solution (G.1.5) or ammonia solution (G.1.1). Dilute with water to 40 ml and mix the solution.

# G.2.2 Preparation of Solution B

Weigh, to the nearest milligram, about 2 g of the sample and dissolve it in 10 ml of water. Add 5 ml of hydrochloric acid (G.1.2) and evaporate to dryness on a steam bath. 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 (shortrange pH indicator paper) by the addition of dilute acetic acid (G.1.5) solution or anmonia 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.1.6), mix, allow to stand for 5 min, and observe downward over a white surface. The colour of the Solution B, shall be not darker than that of Solution A.

# SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.

Printed at the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.

# SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI which includes surveillance inspection of the factory, testing of both factory and market samples.

Further particulars of the terms and conditions of the permit may be obtained from the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.

Printed at SLSI (Printing Unit)