# SRI LANKA STANDARD 80: 2019
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# SPECIFICATION FOR EDIBLE IODIZED SALT (POWDERED FORM)
## (Second Revision)

SRI LANKA STANDARDS INSTITUTION

# Sri Lanka Standard
# SPECIFICATION FOR EDIBLE IODIZED SALT (POWDERED FORM)
**(*Second Revision*)**

**SLS 80: 2019**

**Gr. 11**

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# Sri Lanka Standard
# SPECIFICATION FOR EDIBLE IODIZED SALT (POWDERED FORM)
## (*Second Revision*)

## FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Food products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2019-03-07.

Edible salt in powdered form consists predominantly of Sodium chloride. The remainder comprises natural secondary products, which are present in varying amounts depending on the origin and the method of production of the salt, are composed mainly of Calcium, Potassium, Magnesium and Sodium sulphates, Carbonates, Bromides and of Calcium, Potassium, Magnesium chlorides as well. Natural contaminants may also be present in amounts varying with the origin and the method of production of the salt.

Edible salt can be used as a carrier for food additives or nutrients for technological or public health reasons. Examples of such preparations are mixtures of salt with Nitrite (curing salt) and salt mixed with small amounts of Fluoride, Iodate, Iron, vitamins, etc., and additives used to carry or stabilize such additions. Edible salt is iodated to prevent iodine-deficiency disorders (IDD) for public health reasons. In Sri Lanka, edible salt has been identified as a suitable material for fortified by iodate as it is consumed in fairly uniform quantity by all categories of the population.

This Standard was first published in 1970 and subsequently revised in 2014. In this revision, requirements of Calcium, Magnesium, and Sulphates have been incorporated and limits for iodine content have been changed to align with the Food (Iodization of salt) Regulations. Subject to the provisions of this Standard more specific requirements for special needs may be applied. Edible salt in granular form is covered in **SLS 79**.

This Standard is subject to the restrictions imposed under the Sri Lanka Food Act No. 26 of 1980 and the regulations framed thereunder.

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

In revising this Standard, the assistance derived from the relevant publications of Codex Alimentarius Commission, EU and China Standards Institution is gratefully acknowledged.

## 1 SCOPE

**1.1** This Standard prescribes the requirements, methods of sampling and testing for edible iodized salt in powdered form used as an ingredient of food, both for direct sale to the consumer and for food manufacture.

**1.2** This Standard does not apply to salt from origins other than those specified in Clause **3**, notably the salt which is a by-product of chemical industries.

## 2 REFERENCES

| | |
|---|---|
| SLS 79 | Edible iodized/non-iodized salt (granular form) |
| SLS 102 | Rules for rounding off numerical values |
| SLS 124 | Test sieves |
| SLS 143 | General principles of food hygiene |
| SLS 428 | Random sampling methods |
| SLS 467 | Labelling of prepackaged foods |

Methods of Analysis of European Salt produces' Association (Eu salt) 2017

## 3 DEFINITIONS

For the purpose of this Standard, the following definitions shall apply:

**3.1** **refined iodized salt:** The product consisting predominately of Sodium chloride obtained from the sea or from natural brine and from which soluble and insoluble impurities are reduced by washing, centrifuging, milling, iodizing with food grade Potassium iodate, and drying. The product may contain permitted anti-caking substance.

**3.2** **vacuum evaporated iodized salt:** The product made by evaporating brine employing forced evaporators operating under vacuum, recrystalization, centrifuging, iodizing with food grade Potassium iodate, and drying. The product may contain permitted anti-caking substance.

## 4 TYPES

Edible iodized salt in powdered form shall be of the following types.

**4.1** Refined iodized salt

**4.2** Vacuum evaporated iodized salt

## 5 INGREDIENTS

Food grade Potassium iodate shall comply to requirements given in Food (Iodization of Salt) Regulations.

## 6 FOOD ADDITIVES

Food Additives used shall comply with the Sri Lanka Food Act No. 26 of 1980 and the regulations framed thereunder as amended from time to time or replaced. The limits set for the use of additives by the regulations in the said Food Act shall be adhered to.

### **6.1** Anti-caking substance

Silicon dioxide, amorphous (INS No. 551) - GMP

**NOTE**
*Ferrocyanides are not permitted as additives. Incase of disputes on addition, follow the method of test given in in Appendix* **P**

## 7 REQUIREMENTS

### 7.1 General requirements

**7.1.1** The product shall be produced, processed, packaged, stored and distributed under the hygienic conditions as prescribed in **SLS 143.**

**7.1.2** The product shall be white or off-white in colour and in powdered form. It shall be free from visible impurities such as clay, grit, shells and other extraneous matter.

**7.1.3** The production of iodized edible salt shall only be performed by manufacturers having the knowledge and the equipment requisite for the adequate production of iodized salt, and specifically, for the correct dosage and even for intermixing.

### 7.2 Particle size

Minimum 95 per cent by mass of the product shall pass through a sieve of aperture size 1000 µm (BS 16) when tested in accordance with the method prescribed in Appendix **N**.

### 7.3 Chemical requirements

The product shall also conform to the requirements given in Table **1,** when tested in accordance with the method given in Column **5** of the Table **1**.

**TABLE 1 – Chemical requirements**

| **SI No** | **Characteristic** | **Requirement** | | **Method of test** |
|---|---|---|---|---|
| | | **Refined iodized salt** | **Vacuum Evaporated iodized salt** | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Moisture, per cent by mass, max. | 0.5 | 0.5 | Appendix **B** |
| ii) | Matter insoluble in water, on dry basis, per cent by mass, max. | 0.5 | 0.5 | Appendix **D** |
| iii) | Sodium chloride, as NaCl, on dry basis, per cent by mass, min. | 98.0 | 98.5 | Appendix **E** |
| iv) | Matter soluble in water, other than sodium chloride, on dry basis, per cent by mass, max. | 1.5 | 1.0 | Appendix **L** |
| v) | Calcium as Ca, on dry basis, per cent by mass, max. | 0.2 | 0.2 | Appendix **G** |
| vi) | Magnesium as Mg, on dry basis, per cent by mass, max. | 0.1 | 0.1 | Appendix **G** |
| vii) | Sulphate as $SO_4$**,** on dry basis, per cent by mass, max. | 0.7 | 0.5 | Appendix **H** |
| viii) | Iodine content, as I, on dry basis, mg/kg | 20-40 | 20-40 | Appendix **M** |

## 7.4 Potentially toxic elements

The product shall not exceed the limits given in Table **2**, when tested according to the relevant methods given in Column **4** of the Table **2**

**TABLE 2 – Limits for Potentially toxic elements**

| **Sl No** (1) | **Potentially toxic elements** (2) | **Maximum limit** (3) | **Method of test** (4) |
|---|---|---|---|
| i) | Arsenic, as As mg/ kg | 0.5 | EuSalt/AS 011-2005 or EuSalt/ 015-2007 |
| ii) | Cadmium, as Cd mg/ kg | 0.5 | EuSalt/AS 014-2005 or EuSalt/ 015-2007 |
| iii) | Lead, as Pb mg/ kg | 1.0 | EuSalt/AS 013-2005 or EuSalt/ 015-2007 |
| iv) | Mercury, as Hg mg/ kg | 0.1 | EuSalt/AS 012-2005 or EuSalt/ 015-2007 |

## 8 PACKAGING, TRANSPORTATION AND STORAGE

**8.1** In order to avoid the loss of iodine, the iodized product shall be packaged in air tight food grade bags of either high density polyethylene (HDPE) or polypropylene (PP) (laminated or non-laminated) or linear low density polyethylene (LLDPE) and polyethylene terephthalate (PET) bags.

**8.2** For consumer packaging the product shall be packaged in food grade transparent containers. Where the packing material is plastic, the thickness of such material shall be not less than 75 micrometers.

### 8.3 Bulk packaging

Bulk packaging of edible salt (iodized or non iodized) shall be packaged in HDPE, PP or polysack bags with food grade inner liner.

**8.4** The product shall not be exposed to rain, excessive humidity or direct sunlight at any stage of storage, transportation or sale.

**8.5** The product shall be stored in covered rooms, having adequate ventilation.

**8.6** The distribution network should be streamlined so as to reduce the interval between iodization and consumption of salt.

## 9 MARKING AND/OR LABELLING

**9.1** The following shall be marked or labelled legibly and indelibly on each package/container:

a) Name of the product as “Refined iodized powdered edible salt” or “Vacuum dried iodized edible powdered salt” or “Refined iodized table salt” or “Vacuum dried iodized table salt” or “Free flowing table salt”
b) Brand name or Trade Mark, if any;
c) Net mass, in ‘g’ or ‘kg’;
d) Batch or Code number or a decipherable code marking;
e) Name and address of the manufacturer/ packer and distributor;
f) Date of packaging;
g) Date of expiry;
h) Instructions for storage “store in a cool and dry place, away from sunlight”;
j) The words, “use without washing”; and
k) Country of origin, in case of imported products.

**9.2** The marking and labelling shall also be in accordance with **SLS 467**.

## 10 SAMPLING

Representative samples of the material for testing its conformity to this Standard shall be drawn according to the method prescribed in Appendix **A**.

## 11 METHODS OF TEST

**11.1** During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water for tests.

**11.2** Tests shall be carried out as prescribed in Appendices **B** to **P** of this Standard and the methods given in Methods of Analysis of the European Salt Producers' Association (EuSalt).

## 12 CRITERIA FOR CONFORMITY

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

**12.1** Each container/ package examined as in **A.4.1** satisfies the packaging and marking/ labelling requirements.

**12.2** Each container/package tested as in **A.4.2** satisfies the moisture content, and iodine content as given in Table **1** and also the requirements given in **7.1.2**.

**12.3** The composite sample tested as in **A.4.3** satisfies the all the other characteristics given in Table **1** and requirements given in **7.2** and **7.4**.

# APPENDIX A
# SAMPLING

## A.1 GENERAL REQUIREMENTS OF SAMPLING

In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

**A.1.1** Samples shall not be taken at an exposed place.

**A.1.2** Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

**A.1.3** To draw a representative sample, the contents of each container/ package selected for sampling shall be mixed thoroughly by a suitable method.

**A.1.4** The sample shall be placed in suitable, clean, dry and air-tight glass containers.

**A.1.5** Each sample container shall be sealed air-tight after filling and marked with full details of sampling.

## A.2 SCALE OF SAMPLING

### A.2.1 Lot

All the packages/ containers in a single consignment of edible iodized salt drawn from a single batch of manufacture shall constitute a lot. If the consignment consists of different batches, the batches shall be marked separately and the packages/ containers in each batch shall constitute separate lots.

**A.2.1.1** The number of packages/ containers to be selected from each lot shall be in accordance with Table **3**.

**TABLE 3 – Scale of sampling**

| **No. of packages/containers in the lot** (1) | **No. of packages/containers to be selected** (2) |
|---|---|
| Up to 150 | 4 |
| 151 to 300 | 7 |
| 301 to 500 | 10 |
| 501 and above | 16 |

**A.2.1.2** These packages/containers shall be selected at random from the lot and to ensure randomness of selection, tables of random numbers as given in **SLS 428** shall be used.

## A.3 Preparation of test samples

**A.3.1** From each of the packages/containers selected in accordance with **A.2.1.1**, a portion of the material (about 200 g) shall be drawn with the help of a suitable sampling instrument.

**A.3.2** Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 600 g.

**A.3.3** All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

## A.4 Number of tests

**A.4.1** Each package/ container selected as in **A.2.1.1** shall be examined for packaging and marking/ labelling requirements.

**A.4.2** Each sample obtained as in **A.3.1** shall be tested for moisture content and iodine content, as given in Table **1** and also shall be examined for the requirements given in **7.1.2**.

**A.4.3** The composite sample prepared as in **A.3.2** shall be tested for all other characteristics given in Table **1** and for the requirements given in **7.2** and **7.4**.

# APPENDIX B
# DETERMINATION OF MOISTURE

## B.1 PROCEDURE

**B.1.1** Grind rapidly, where relevant, about 250 g of the sample in a mortar, to a powder. Keep in an air-tight container.

**B.1.2** Weigh, to the nearest 0.001 g, approximately 10 g of the material in a previously dried and weighed weighing bottle about 30 ml capacity and preferably wide mouth squat type. Dry in an oven at 140 $^{o}$C to 150 $^{o}$C for at least 4 hours. Cool in a desiccator and weigh. Repeat drying, cooling and weighing procedures until the difference between two successive weightings' does not exceed 0.001 g.

## B.2 CALCULATION

$$\textit{Moisture, per cent by mass} = \frac{m_1 - m_2}{m_1} \times 100$$

where,

$m_1$ is the initial mass, in g, of the sample taken for the test; and
$m_2$ is the final mass, in g, of the sample.

# APPENDIX C
# PREPARATION OF THE DRIED SAMPLE

Spread about 200 g of the sample obtained as in **B.1.1** on a petri dish and dry by the method given in **B.1.2**. The dried material shall be referred to as the dried sample and shall be used in the tests where so indicated.

# APPENDIX D
# DETERMINATION OF MATTER INSOLUBLE IN WATER

## D.1 PROCEDURE

**D.1.1** Weigh, to the nearest 0.001 g, approximately 20 g of the dried sample (*see* Appendix **C**), dissolve it in 200 ml of water and heat to boiling. Cool and filter the solution through a weighed sintered glass crucible (G. No. 4). Wash the residue with water until the washings do not give any opalescence when tested with silver nitrate solution. Collect the filtrate and washings in a one-litre graduated flask and dilute to the mark. Reserve the solution so obtained for subsequent tests.

**D.1.2** Dry the crucible along with the residue at 105 $^{o}$C to 110 $^{o}$C for 1 hour and weigh. Repeat drying and weighing procedures until the difference between two successive weighings does not exceed 0.001 g.

## D.2 CALCULATION

$$\text{Matter insoluble in water, per cent by mass} = \frac{m_1}{m_2} \times 100$$

where,

$m_1$ is the mass, in g, of the residue, and
$m_2$ is the mass, in g, of the dried sample taken for the test.

**NOTE**
*The residue will include anti-caking substances, if added.*

# APPENDIX E
# DETERMINATION OF SODIUM CHLORIDE

## E.1 DETERMINATION OF ANIONS AND CATIONS TO CALCULATE SODIUM CHLORIDE

Determine the anions and cations given in Column **2** of Table **4**, by the methods prescribed in Column **3** of the table.

**Table 4 – Anions and cations**

| **Sl No** (1) | **Anion or cation** (2) | **Method of test** (3) |
|---|---|---|
| i) | Carbonate, as $CO_3$ | Appendix **F** |
| ii) | Calcium, as Ca | Appendix **G** |
| iii) | Magnesium, as Mg | Appendix **G** |
| iv) | Sulphate, as $SO_4$ | Appendix **H** |
| v) | Total chloride, as Cl | Appendix **J** |
| vi) | Potassium, as K | Appendix **K** |

## E.2 CALCULATION OF RESIDUAL CHLORIDE

Express the Carbonate as Calcium carbonate or as Sodium carbonate in absence of Calcium and Magnesium. If there is excess of Calcium over the Calcium carbonate, combine all the Calcium with the sulphate and express the result as percentage of Calcium sulphate. If there is excess of sulfate over what is required for Calcium, combine the excess with Magnesium and express the result as percentage of Magnesium sulphate. If there is excess of calcium over the Calcium sulphate, express it as percentage of Calcium chloride and deduct the chloride corresponding to it from the total Chloride content. Calculate the balance of Magnesium as Magnesium chloride and deduct the Chloride corresponding to it from the Chloride content. Also, deduct from the Chloride content and amount of Chloride corresponding to Potassium content and express it as percentage of Potassium chloride. The remaining Chloride content is referred to as residual Chloride.

## E.3 CALCULATION OF SODIUM CHLORIDE

Sodium chloride, as NaCl, per cent by mass = Residual chloride, as Cl x 1.648

# APPENDIX F
# DETERMINATION OF CARBONATE

## F.1 REAGENTS

**F.1.1 Hydrochloric acid**, standard volumetric solution, c (HCl) = 0.1 mol/l

**F.1.2 Methyl orange** indicator solution

Dissolve 0.1 g of methyl orange in 100 ml of water.

## F.2 PROCEDURE

Pipette out 100 ml of the solution reserved in **D.1.1** and titrate against standard Hydrochloric acid (**F.1.1**) using methyl orange (**F.1.2**) as indicator.

## F.3 CALCULATION

Carbonate, as $CO_3$, per cent by mass $= \dfrac{M \times V \times 30}{m}$

where,

$M$ is the strength, in mol/l, g Hydrochloric acid used in the titration;
$V$ is the volume, in ml, of standard Hydrochloric acid used in the titration; and
$m$ is the mass, in g, of the dried sample taken for the test in **D.1.1**.

# APPENDIX G
# DETERMINATION OF CALCIUM AND MAGNESIUM

## G.1 REAGENTS

### G.1.1 Standard calcium solution

Weigh, 1.000 g of Calcium carbonate dried at 120 $^{o}$C and dissolve it in the minimum quantity of dilute Hydrochloric acid. Dilute the solution to one litre in a graduated flask. One milliliter of this solution is equivalent to 0.401 mg of Calcium.

### G.1.2 Ammonium chloride – Ammonium hydroxide buffer solution

Dissolve 67.5 g of Ammonium chloride in a mixture of 570 ml Ammonium hydroxide (rel. den. = 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of Disodium ethylene diamine tetra acetate dihydrate and 0.616 g of Magnesium sulfate ($MgSO_4.7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to one litre.

**NOTE**
*Five milliliters of buffer solution added to* 50 *ml of distilled water shall not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.*

### G.1.3 Eriochrome black T indicator solution

Dissolve 0.1 g of the dye in 20 ml of rectified spirit. This solution shall be prepared fresh every week.

### G.1.4 Standard EDTA solution

Dissolve 3.72 g of Disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against standard calcium solution following the procedure given in **G.2**

**G.1.5 Dilute Sodium hydroxide**, approximately 10 per cent (m/v) solution.

### G.1.6 Murexide indicator solution

Grind 0.2 g of murexide with 10 g of Sodium chloride until the mixture is homogeneous. Thus 0.2 g of the mixture is suitable for 100 ml of the sample solution.

### G.1.7 Calcein indicator

Grind thoroughly 0.1 g of calcein and 0.06 g of thymolphthalein with 10 g of Potassium chloride.

## G.2 PROCEDURE

### G.2.1 Standardization of EDTA solution

**G.2.1.1** Transfer 25 ml of standard Calcium solution (**G.1.1**) into a conical flask, add 25 ml of water, 10 ml of Ammonium chloride-ammonium hydroxide buffer solution (**G.1.2**), 5 drops of erichrome black T indicator solution (**G.1.3**) and titrate against the standard EDTA solution (**G.1.4**) to a pure blue end point.

**G.2.1.2** Titrate 25 ml of the buffer solution (**G.1.2**) with EDTA solution (**G.1.4**) using erichrome black T indicator (**G.1.3**). Substract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in (**G.2.1.1**) and note the final titre value. Calculate the Calcium equivalent to 1 ml of EDTA solution ( $m_1$).

### G.2.2 Determination of calcium and magnesium

**G.2.2.1** Transfer 100 ml of the solution reserved in **D.1.1** into a 250-ml conical flask, add 10 ml of Ammonium chloride-ammonium hydroxide buffer solution (**G.1.2**), and 5 drops of eriochrome black T indicator solution (**G.1.3**). Titrate against standard EDTA solution (**G.1.4**) till wine red colour of the solution changes to pure blue end point. Note the volume of the EDTA solution used in the titration.

**G.2.2.2** Transfer 100 ml of the solution reserved in **D.1.1** into a 250-ml conical flask, add 5 ml of Sodium hydroxide solution (**G.1.5**) and stir well. Add 0.2 g of murexide indicator (**G.1.6**) or 100 mg of calcein indicator (**G.1.7**) (*see* Note) and titrate against standard EDTA solution (**G.1.4**) till wine red colour of the solution changes to pure blue end point with murexide or green to purple with calcein indicator. Note the volume of the EDTA solution used in the titration.

**NOTE**
*Patton and Reader's indicator may also be used in place of murexide indicator or mixed calcein indicator.*

## G.3 CALCULATION

Calcium, as Ca, per cent by mass $= \dfrac{m_1 V_2}{2}$

Magnesium, as Mg, per cent by mass $= 0.6068 \dfrac{m_1(V_1 - V_2)}{m_2}$

where,

$m_1$ is the mass, in mg, of Calcium equivalent to 1 ml of EDTA solution (as determined in **G.2.1**);
$V_1$ is the volume, in ml, of standard EDTA solution used in **G.2.2.1**;
$V_2$ is the volume, in ml, of standard EDTA solution used in **G.2.2.2**; and
$m_2$ is the mass, in g, of the dried sample taken for the test in **D.1.1**.

# APPENDIX H
# DETERMINATION OF SULPHATE

## H.1 REAGENTS

**H.1.1 Standard Barium chloride,** 0.025 mol/l solution
Dissolve 6.108 g of Barium chloride dihydrate in water and make up to one litre.

**H.1.2 Dilute Hydrochloric acid,** approximately 1 mol/l solution

**H.1.3 Ammonium chloride – ammonium hydroxide** buffer solution, same as in **G.1.2.**

**H.1.4 Eriochrome black T indicator** solution, same as in **G.1.3**.

**H.1.5 Standard EDTA solution**

Weigh 3.72 g of Disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized for frequently against standard barium chloride solution (**H.1.1**) and follow the procedure given in (**G.2.2**). One millilitre of standard EDTA solution is equivalent 0.001374 g of barium or 0.00096 g of sulfate.

## H.2 PROCEDURE

**H.2.1** Pipette out 20 ml of standard Barium chloride solution (**H.1.1**) into a conical flask, add 2 drops of Hydrochloric acid (**H.1.2**) and 10 ml of Ammonium chloride – Ammonium hydroxide buffer solution (**H.1.3**). Dilute with water to about 50 ml, add five drops of eriochrome black T indicator solution (**H.1.4**) and titrate against standard EDTA solution (**H.1.5**) to pure blue end point. Note the volume of the EDTA solution used in the titration.

**H.2.2** Pipette out 100 ml of the solution reserved in **D.1.1** and add two drops of Hydrochloric acid (**H.1.2**) and heat to gentle boiling. To the hot solution, add with a pipette 20 ml of standard Barium chloride solution (**H.1.1**). Boil gently for about 5 minutes and then cool to room temperature. The solution will be neutral or slightly acid when tested with a litmus paper. Add 10 ml of Ammonium chloride – Ammonium hydroxide buffer solution (**H.1.3**) and five drops of eriochrome black T indicator solution (**H.1.4**). Titrate against standard EDTA solution to pure blue end point. Note the volume of the EDTA solution used in the titration.

### H.3 CALCULATION

$$\text{Sulphate, as } SO_4\text{, per cent by mass} = 0.96 \frac{V_1 + V_2 - V_3}{m}$$

where,

$V_1$ is the volume, in ml, of standard EDTA solution used in **H.2.1**;
$V_2$ is the volume, in ml, of standard EDTA solution used in **G.2.2.1** (total Ca and Mg);
$V_3$ is the volume in ml, of standard EDTA solution used in **H.2.2**; and
$m$ is the mass, in g, of the dried sample taken for the test in **D.1.1**.

## APPENDIX J
## DETERMINATION OF TOTAL CHLORIDE

### J.1 REAGENTS

**J.1.1 Potassium chromate indicator**, 5 per cent (v/v) solution

**J.1.2 Silver nitrate,** standard volumetric solution c ($AgNO_3$) = 0.1 mol/l

### J.2 PROCEDURE

Transfer 10 ml of the solution reserved in **D.1.1** into a conical flask and add 1 ml of potassium chromate indicator solution (**J.1.1**). Titrate against standard Silver nitrate solution (**J.1.2**) until the reddish brown tinge persists after brisk shaking. Carry out a blank determination.

### J.3 CALCULATION

$$\text{Total chloride, as Cl, per cent by mass} = 709.2 \frac{V \times C}{m}$$

where,

$V$ is the volume, in ml, of standard Silver nitrate solution used in the titration, corrected to blank;
$C$ is the concentration, in mol/l, of standard Silver nitrate solution used; and
$m$ is the mass, in g, of the dried sample taken for the test in **D.1.1**.

# APPENDIX K
# DETERMINATION OF POTASSIUM

## K.1 APPARATUS

**K.1.1 Simple flame photometer,** equipped with potassium filter corresponding to wave length of 767 nm

**K.1.2 Ordinary laboratory apparatus**

## K.2 REAGENTS

### K.2.1 Standard Potassium solution

Weigh 1.0 g of Potassium chloride, dissolve in distilled water and dilute to one litre with distilled water in a measuring flask. This solution contains 0.1 g of Potassium chloride per 100 ml.

### K.2.2 Standard dilute Potassium solutions

Dilute 10 ml, 20 ml, 40 ml, 60 ml and 80 ml of standard Potassium solution (**K.2.1**) to 100 ml with distilled water in different measuring flasks. Each flask now contains 0.01 g, 0.02 g, 0.04 g, 0.06 g and 0.08 g of Potassium chloride per 100 ml.

### K.2.3 Sample solution

Dissolve approximately 10 g of the dried sample (*see* Appendix **C**) in minimum quantity of distilled water and dilute to 100 ml in a measuring flask with distilled water.

## K.3 PROCEDURE

Adjust the galvanometer to read zero by distilled water and 100 by standard potassium solution (**K.2.1**). Spray various diluted solutions (**K.2.2**) and obtain the corresponding galvanometer readings. Plot the concentrations against galvanometer readings. Draw smooth curve over the points which give a calibration curve in the range of 0.01 g of Potassium chloride per 100 ml to 0.1 g of Potassium chloride per 100 ml.

After washing with distilled water, spray the sample solution (**K.2.3**) and obtain the galvanometer reading. From the graph read out the corresponding concentration of Potassium chloride in the solution.

## K.4 CALCULATION

$$\text{Potassium, as K, per cent by mass} = 52.4\ \frac{m_1}{m_2}$$

where,

$m_1$ is the mass, in g, of potassium chloride in the sample solution; and
$m_2$ is the mass, in g, of the dried sample taken for the test.

## APPENDIX L
## CALCULATION OF MATTER SOLUBLE IN WATER OTHER THAN SODIUM CHLORIDE

Matter soluble in water, other than sodium chloride, per cent by mass = $100 - (m_1 + m_2)$

where,

$m_1$ is the matter insoluble in water, calculated as in **D.2**; and
$m_2$ is the sodium chloride content; calculated as in **E.3**.

## APPENDIX M
## DETERMINATION OF IODINE

### M.1 REAGENTS AND MATERIALS

**M.1.1 Methyl orange indicator** solution

Dissolve 0.01 g of methyl orange in water and dilute to 100 ml.

**M.1.2 Phosphoric acid**, analytical reagent grade**,** approximately 85 per cent (v/v) solution.

**M.1.3 Bromine water,** saturated aqueous solution

Determine the approximate concentrate (milligram of bromine per millilitre) by adding from a burette a measured volume to a flask containing 5 ml of 10 per cent (v/v) solution of Potassium iodide and 5 ml of dilute Sulphuric acid and titrating the liberated iodine with 0.1 mol/l solution of Sodium thiosulfate. One millilitre of 0.2 mol/l solution of Sodium thiosulfate is equivalent to 8 mg of bromine or 12.7 mg of Iodine.

**M.1.4 Salicylic acid**

**M.1.5 Potassium iodide**

**M.1.6 Sodium thiosulphate**, standard volumetric solution - **c** ($Na_2 S_2 O_3$) = 0.005 mol/l

Prepare 0.1 mol/l solution by dissolving 25 g of analytical reagent grade Sodium thiosulphate in 1000 ml of water. Dilute 50 ml of this solution to 1000 ml.

**NOTE**
*Sodium thiosulphate solution should be standardized.*

**M.1.7 Starch solution,** freshly prepared 1 per cent (V/V) solution

**M.1.8 Sodium chloride** analytical reagent

**M.1.9 Potassium iodate**

**M.1.10 Sulphuric acid,** approximately 1 mol/l

## M.2 QUALITATIVE TEST FOR IODATE

### M.2.1 Procedure

**M.2.2** Transfer about 2 g of salt to a test tube and dissolve in 10 ml distilled water

**M.2.3** Add about 1 ml of 10 per cent KI solution and 2 ml of 1 M Sulphuric acid and few mililitre of starch solution.

**M.2.4** If iodate is present the solution turns blue due to the liberation of iodine

**M.2.5** Estimate the amount of Iodate as given in Procedure **M.3**

**M.2.6** If there is no blue colour, there is no iodate in the salt. The salt may have iodide or may not be iodised.

**M.2.7** To check for the presence of iodide

**M.2.8** Transfer about 2 g of salt to a test tube and dissolve in 10 ml distilled water.

**M.2.9** Add about 1 ml of 10 per cent $KIO_3$ solution, 2 ml Sulphuric acid and few ml of starch solution.

**M.2.10** If iodide is present the solution turns blue due to the liberation of iodine.

**M.2.11** If there is no blue colour, there is no iodide in the salt. The salt may have iodate or may not be iodised.

**M.2.12** If there is no blue colour by both tests then the salt does not have iodide or iodate.

**NOTE**
*These qualitative tests must be carried before an opinion is expressed.*

## M.3 PROCEDURE (If iodine is added as IODATE)

**M.3.1** Weigh 10 g of salt accurately and dissolve in 100 ml distilled water in a 250 ml conical flask which can be stoppered.

**M.3.2** Add 2 ml of 1 mol/l sulfuric acid to the solution. Add 5 ml 10% (v/v) Potassium iodide and stopper immediately. Shake it well and keep in dark for 5 minutes.

**M.3.3** Titrate this solution with a standard Sodium thiosulfate solution 0.005 mol/l, to a light straw colour and add 2 ml of 1 per cent starch solution and continue titration to a colourless end point.

**M.3.4** Carry-out a blank test using 10 g of analytical grade Sodium chloride.

## M.4 CALCULATION

$$\text{Iodine, as I, mg/kg} = 21150 \; \frac{(V_1 - V_2) \times C}{m}$$

where,

$V_1$ is the volume, in ml, of standard Sodium thiosulfate solution required for the sample;
$V_2$ is the volume, in ml, of standard Sodium thiosulfate requirement for blank determination;
$C$ is the concentration, in mol/l, of the Sodium thiosulfate solution; and
$m$ is the mass, in g, of the sample taken (dry basis)

## APPENDIX N
## DETERMINATION OF PARTICLE SIZE

## N.1 APPARATUS

**N.1.1 Sieve**, 1000 µm , conforming to **SLS 124**

**N.1.2 Balance**, with accuracy 0.01 g

## N.2 PROCEDURE

Weigh, to the nearest 0.1 g, about 100 g of sample into the sieve. Sieve for a minimum period of 5 min with occasional tapping on the sieve until all the sieveable particles are passed through the sieve. Transfer the material retained on the sieve quantitatively into a tared dish and weigh.

## N.3 CALCULATION

$$\text{Material passed through the sieve, per cent by mass} = 100 - \left[ \frac{m_2}{m_1} \times 100 \right]$$

where,

$m_1$ is the mass, in g, of the test portion;; and
$m_2$ is the mass, in g, of the material retained on the sieve.

# APPENDIX P
# DETERMINATION OF FERROCYANIDE
## (Ferrous Sulfate Method)

## P.1 PRINCIPLE

Under acidic conditions, ferrocyanide reacts with ferrous sulfate to form a white precipitate of ferrous ferrocyanide, which is oxidized by air to form Prussian blue and determined by spectrophotometry.

## P.2 APPARATUS

### P.2.1 Spectrophotometer

### P.2.3 General laboratory instruments

## P.3 REAGENTS

### P.3.1 Sodium chloride

Take 50 g of Sodium chloride; place into high temperature furnace; burn it at 800 $^{o}$C for 30 min; cool it off for later-use.

### P.3.2 Sulfuric acid solution (1+20)

Take 5 ml of concentrated Sulfuric acid; slowly place it into 100 ml of water and mix well.

### P.3.3 40 g/1000 ml ferrous sulfate solution

Take 4 g of Ferrous sulfate ($FeSO_4.7H_2O$); dissolve it in 100 ml of sulfuric acid solution (**P.3.2**); filter it. Store in the brown reagent bottle at low temperature.

### P.3.4 Potassium ferrocyanide standard stock solution (1 ml of solution contains 1.0 mg $[Fe(CN)_6]^{4-}$)

Accurately weigh 0.1993 g of Potassium ferrocysnide ($K_4[Fe(CN)_6].3H_2O$. Add a small amount of water to dissolve and dilute to 100 ml.

### P.3.5 Potassium ferrocyanide standard working solution (1 ml of solution contains 20 µg $[Fe(CN)_6]^{4-}$)

Pipette 2.00 ml of Potassium ferrocyanide standard stock solution (**P.3.4**) and dilute to 100 ml.

## P.4 PROCEDURE

### P.4.1 Determination of the limit method

Pipette the Potassium ferrocyanide standard working solution (**P.3.5**) corresponding to the limited amount into 50 ml colorimetric tube. Add 5 g of Sodium chloride (**P.3.1**). Add water to dissolve. Add 4 ml of Ferrous sulfate solution (**P.3.3**). Add water to dilute to the scale.Shake evenly; stand for 10 min. Take reagent blank as a reference at the wavelength of 670 nm. Then measure the absorbance. Take another 50 ml colorimetric tube. Take 5.0 g of specimen into the colorimetric tube. Add 40 ml of water to dissolve (if the solution is turbid, filter with 0.45µm filter membrane). Add 4 ml of ferrous sulfate solution (**P.3.3**). Add water to dilute to the scale. Shake evenly, stand for 10 min and then take reagent blank as a reference at the wavelength of 670 nm, measure the absorbance. If the absorbance of the specimen solution is lower than that of the standard solution, then it is qualified. Otherwise, it is disqualified.

### P.4.2 Determination of working curve method

#### P.4.2.1 Standard curve

Pipette standard working solution (**P.3.5**) containing 0 µg, 10 µg, 20 µg, 30 µg, 40 µg, 50 µg, 60 µg of Ferrocyanide in 50 ml colorimetric tubes, separately and add 5 g of sodium chloride (**P.3.1**). Add water to dissolve. Add 4 ml of Ferrous sulfate solution (**P.3.3**). Add water to dilute to the scale, shake evenly and stand for 10 min. Then take reagent blank as a reference at the wavelength of 670 nm and measure the absorbance in 4 cm cell. Take the Ferrocyanide mass as the abscissa and take the corresponding absorbance as the ordinate to draw the standard working curve.

#### P.4.2.2 Determination of specimen

Take 5.0 g of specimen into 50 ml colorimetric tube and add 40 ml of water to dissolve (if the solution is turbid, filter with 0.45 µm filter membrane). Add 4 ml of Ferrous sulfate solution. Add water to dilute to the scale, shake evenly and stand for 10 min. Then take reagent blank as a reference at the wavelength of 670 nm and measure the absorbance in 4 cm cell. The mass of the ferrocyanide can be checked out from the working curve based on the absorbance of the specimen.

## P.5 CALCULATION

The content of ferrocyanide in the specimen can be expressed in mass fraction, the value can be expressed by mg/ kg.

$$\text{Ferrocyanide content ,mg/ kg} = \frac{m_1}{m}$$

where

$m_1$ is the ferrocyanide mass checked out from the working curve, in µg;
$m$ is the specimen mass, in g.

The absolute difference between two independent tests carried out in the same laboratory, by the same operator using the same equipment, according to the same test method, against the same test object shall be no greater than the provisions of Table **5**.

**Table 5**

| **Ferrocyanide content / (mg/kg)** | **Absolute difference between results / (mg/kg)** |
|---|---|
| ≤ 10.0 | 1.0 |

**……………………….**

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