

SRI LANKA STANDARD 603 : 2016
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**SPECIFICATION FOR
HYDROCHLORIC ACID**
(First Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 603 : 2016

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Sri Lanka Standard
SPECIFICATION FOR HYDROCHLORIC ACID
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FOREWORD

This Standard was approved by the Sectorial Committee on Chemical and Polymer Technology and was authorized for adoptions and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2016-03-23.

This Standard was first published in 1983 and this First Revision introduces the Chromatographic and Spectroscopic grades, limits for heavy metals of cadmium, mercury and chromium and alternative method for determination of metals and hydrochloric acid content. The limits for hydrochloric acid content of Analytical Reagent grade has been changed and additional marking requirements have been included.

This Specification covers five grades of hydrochloric acids, i.e. Technical grade, General Purpose Reagent (GPR)/ Laboratory Reagent Grade (LRG), Analytical Reagent (AR), Spectroscopic and Chromatographic grade. Technical grade acid is intended for use in industrial purposes such as pickling of iron, water purification, brine processing and for production of ferric chloride. Purity of GPR grade acid is higher than the technical grade acid and it could be used in industries and laboratories. Purity of AR grade acid is higher than Technical and GPR grades and is intended for use in analytical work where a high degree of purity is required. Chromatographic and Spectroscopic grades are the purest grades and Chromatographic grade is intended for use in HPLC and GC. Spectroscopic grade is intended for use in Atomic Absorption Spectroscopy (AAS), Spectroscopy and UV Spectroscopy.

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

All values in this Specification are given in SI units.

In the preparation of this Specification, the assistance derived from the following publications are gratefully acknowledged:

SS	8	Specification for Hydrochloric acid (Technical)
IS	265	Hydrochloric acid – Specification
JIS K	8180	Hydrochloric acid (Reagent)

1 SCOPE

1.1 This Specification prescribes the requirements, test methods and sampling procedure for hydrochloric acid used in industries and laboratories.

1.2 It does not specify requirements for hydrochloric acid intended for pharmaceutical use.

2 REFERENCES

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

3 GRADES

Hydrochloric acid shall be of the following five grades:

- a) Technical grade;
- b) General purpose reagent (GPR) grade/Laboratory Reagent grade(LRG);
- c) Analytical reagent (AR) grade;
- d) Chromatographic grade; and
- e) Spectroscopic grade.

4 REQUIREMENTS

4.1 General requirements

4.1.1 *Technical grade*

The material shall be a clear, colourless or light yellow liquid, free from dirt and other visible impurities.

4.1.2 *General purpose reagent (GPR) or Laboratory Reagent (LRG), Analytical Reagent (AR), Chromatographic and Spectroscopic grades.*

The material shall be a colourless fuming liquid, free from dirt and other visible impurities.

4.2 Other requirements

The material shall also comply with the requirements given in Table 1, when tested according to the relevant methods given in Column 6 of Table 1.

5 PACKAGING

The acid shall be packed in suitable clean, sound, dry and air-tight sealed containers that are resistant to the reactive property of the material. For bulk order, the arrangement for packaging and transportation shall be subject to agreement between the purchaser and supplier. In the case of packaging in containers, the manufacturer shall ensure that sufficient measures are taken to prevent breakage during handling and transportation.

TABLE 1 – Requirements for hydrochloric acid

Sl. No. (1)	Characteristic (2)	Requirement					Method of test (6)
		Technical grade (3)	GPR / LRG (4)	AR grade (5)	Spectroscopic grade (5)	Chromatographic grade (5)	
i	Specific gravity, 27 °C / 27 °C, min.	1.1453	1.1553	1.1553	1.1789	1.1789	Appendix B
ii	Hydrochloric acid content, per cent by mass, min.	30.0	32.0	35.0	36.0	36.0	Appendix C
iii	Residue on ignition, per cent by mass, max.	0.1	0.01	0.001	0.001	0.001	Appendix D
iv	Sulphates (as H ₂ SO ₄), mg/kg, max.	1 000	200	5	5	5	Appendix E
v	Iron (as Fe), mg/kg, max.	200	10	0.4	0.4	0.4	Appendix F
vi	Free chlorine and bromine (as Cl), mg/kg, max.	200	20	2	2	2	Appendix G
vii	Sulphites (as SO ₂), mg/kg, max.	500	400	1	1	1	Appendix H
viii	Lead (Pb), mg/kg, max.	10	5	0.8	0.8	0.8	Appendix J
ix	Cadmium (Cd), mg/kg, max.	1	0.05	0.01	0.01	0.01	Appendix J
x	Mercury (Hg), mg/kg, max.	1	0.05	0.01	0.01	0.01	Appendix J
xi	Chromium (Cr), mg/kg, max.	2	0.01	0.02	0.02	0.02	Appendix J
xii	Arsenic (as As), mg/kg, max.	4	2	0.02	0.02	0.02	SLS 312 or Appendix J
xiii	Ammonium compounds (as NH ₃), mg/kg, max.	10	6	3	3	3	Appendix K

6 MARKING

6.1 The following shall be marked legibly and indelibly on each container:

- a) Name and Grade of the acid;
- b) Strength of the acid in molarity;
- c) The manufacturer's /supplier's/distributor's name and address (see Note);
- d) Registered trade mark if any;
- e) Brand name if any;
- f) The net mass, in kilogram (kg), or net volume in liters (l) or in milliliters (ml);
- g) The batch or code number;
- h) Date of manufacture;
- j) Density of the acid;
- k) Pictorial diagram to indicate corrosive nature (see Figure 1); and
- l) The words, *AVOID BREATHING VAPOUR*.

NOTE: Additional details should be included as agreed to between the purchaser and the supplier.

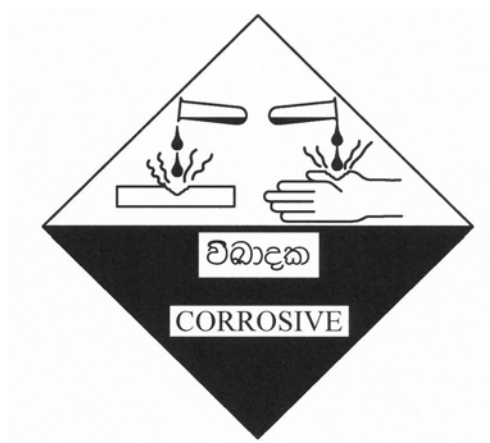


FIGURE 1 – Pictorial marking for corrosive nature

7 SAMPLING

Representative samples of the material shall be drawn as prescribed in Appendix A.

8 METHODS OF TEST

Tests shall be carried out as prescribed in Appendices B to K.

APPENDIX A SAMPLING

A.1 LOT

In a single consignment, all the containers of same size, containing material of same grade, drawn from one batch of manufacture shall constitute a lot.

A.2 SAMPLING INSTRUMENTS

A.2.1 Glass sampling tube

A.2.2 Pipette

A.3 GENERAL REQUIREMENTS OF SAMPLING

In drawing samples, the following precautions and directions shall be observed.

A.3.1 Precautions shall be taken to prevent the samples from adventitious contamination.

A.3.2 Sampling instrument when used shall be clean and dry.

A.3.3 To draw a representative sample, the contents of each container shall be mixed thoroughly by rolling, shaking or stirring by suitable means and with necessary caution.

A.3.4 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

A.3.5 Each sample container shall be sealed air-tight after filling and shall be marked with necessary details of sampling.

A.3.6 Samples shall be stored in such a manner that the conditions of storage do not affect the quality of the material.

A.4 SCALE OF SAMPLING

A.4.1 Samples shall be tested from each lot for ascertaining its conformity of the material to the requirements of this specification.

A.4.2 The number of containers to be selected from the lot shall be in accordance with Table 2.

A.4.3 The containers shall be selected at random. In order to ensure randomness of selection, random number tables as given in SLS 428 shall be used.

TABLE 2 – Scale of sampling

Number of containers in the lot (1)	Number of containers to be selected (2)
Up to 25	2
26 to 100	3
101 to 150	5
151 to 300	8
301 to 500	10
501 and above	13

A.5 COMPOSITE SAMPLE

Equal quantities of material shall be taken from each container selected as in **A.4.2** to make a sample and mix thoroughly to form a composite sample of sufficient size to carry out the tests for the requirements specified in **A.7.3**.

A.6 INDIVIDUAL SAMPLE

The remaining portion of material from each container shall form an individual sample for testing the requirement specified in **A.7.2**.

A.7 NUMBER OF TESTS

A.7.1 Each container selected as in **A.4.2** shall be inspected for packaging and marking requirements.

NOTE

This may be done at the place of inspection.

A.7.2 Each individual sample obtained as in **A.6** shall be tested for total acidity (Hydrochloric acid content) and specific gravity.

A.7.3 The composite sample obtained as in **A.5** shall be tested for requirements other than acidity and specific gravity given in Table 1.

A.8 CONFORMITY TO STANDARD

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

- A.8.1** Each container inspected as in **A.7.1** satisfies the relevant requirements.
- A.8.2** Each individual sample tested as in **A.7.2** satisfies the relevant requirements.
- A.8.3** The composite sample tested as in **A.7.3** satisfies the relevant requirements.

APPENDIX B

DETERMINATION OF SPECIFIC GRAVITY

All chemicals shall be of Analytical grade. Temperature tolerance shall be 27 ± 2 °C

B.1 PRINCIPLE

For the purpose of this Specification, the specific gravity of a material is the ratio of the mass of a given volume of the material at the specified temperature, to the mass of an equal volume of distilled water at the same temperature.

B.2 PROCEDURE

Fill the specific gravity bottle with the material to overflow, holding the specific gravity bottle on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper, immerse in the water bath maintained at 27 °C and keep for 30 minutes. Remove the specific gravity bottle from the bath, clean and dry it thoroughly.

B.3 CALCULATION

$$\text{Specific gravity at } 27 \text{ }^{\circ}\text{C} = \frac{m_1 - m_2}{m_3 - m_2}$$

where,

- m_1 is the mass, in g, of the specific gravity bottle with the material at 27 °C;
- m_2 is the mass, in g, of the specific gravity bottle at 27 °C; and
- m_3 is the mass, in g, of the specific gravity bottle with water at 27 °C.

NOTE : *All the weights (m_1 , m_2 , m_3) shall be taken at the same temperature*

APPENDIX C DETERMINATION OF HYDROCHLORIC ACID CONTENT

Two methods have been prescribed for the determination of hydrochloric acid. The method prescribed in C.1 shall be the reference method and shall be carried out in case of any dispute.

C.1 METHOD 1

C.1.1 Principle

Titration of the total acidity in a test portion with standard sodium hydroxide solution, in the presence of Methyl Orange as indicator.

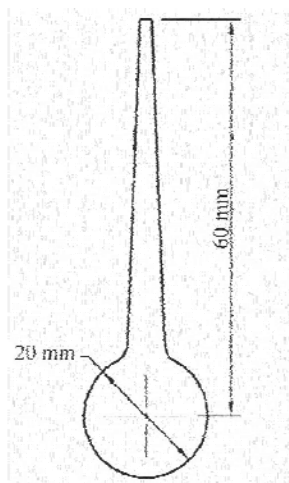


FIGURE 2 – Spherical glass ampoule

C.1.2 Apparatus

C.1.2.1 *Flask*, of capacity approximately 500 ml, with neck of diameter about 30 mm, with ground glass stopper.

C.1.2.2 *Spherical glass ampoule*, of suitable shape and capacity, for example of diameter 20 mm, having one capillary end of length about 50 mm (see Figure 2).

C.1.2.3 *Conical flask*, of capacity 500 ml, with ground glass stopper

C.1.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, neutral Methyl Orange.

C.1.3.1 *Sodium hydroxide*, 1 M standard volumetric solution.

C.1.3.2 *Methyl Orange indicator*, 0.05 per cent (V/V) aqueous solution.

C.1.4 Procedure

C.1.4.1 Test portion

Fill the flask (C.1.2.1) with the sample. Slightly heat in a flame, the bulb of the glass ampoule (C.1.2.2), previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the flask containing the test sample and ensure that the bulb is filled up to about two-thirds of its volume during cooling (2 ml to 3 ml approximately). Withdraw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in an oxidizing flame, without loss of glass. Remove from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper. Weigh the ampoule to the nearest 0.1 mg and calculate by difference, the mass of the test portion.

C.1.4.2 Determination

Carefully place the ampoule, containing the test portion (C.1.4.1) into the conical flask (C.1.2.3) containing 200 ml of cold water. Stopper the flask and while cooling, shake to break the ampoule containing the test portion. Keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the same flask. Grind the fragments of the ampoule by means of a glass rod. Withdraw the glass rod and wash it with water, collecting the washings in the same flask. Add 2 drops of the Methyl Orange indicator (C.1.3.2) and titrate with the standard volumetric sodium hydroxide solution (C.1.3.1) until the colour changes from yellow to red.

C.1.5 Calculation

$$\text{Hydrochloric acid content, percent by mass} = \frac{V \times 0.03646 \times 100}{m}$$

where,

V is the volume, in ml, of the standard volumetric sodium hydroxide solution (C.1.3.1) used for the titration; and

m is the mass, in g, of the test portion (C.1.4.1).

NOTE : One milliliter of 1M sodium hydroxide is equivalent to 0.0364g of hydrochloric acid.

C.2 METHOD 2

C.2.1 Principle

Titration of the total acidity in a test portion with standard sodium hydroxide solution, in the presence of Methyl orange as indicator.

C.2.2 Reagents

C.2.2.1 Sodium hydroxide, 1 M standard volumetric solution.

C.2.2.2 Methyl orange indicator, 0.05 per cent (V/V) aqueous solution.

C.2.3 Procedure

Take 250 ml glass stoppered Erlenmeyer flask containing approximately 20 ml of water and weighed (m_1) to the nearest 0.1 mg. Rapidly add 3 to 4 ml of the hydrochloric acid sample, replace the stopper and weight (m_2). Titrate with 1 M sodium hydroxide solution (C.2.2.1) using methyl orange (C.2.2.2) as the indicator and calculate the total hydrochloric acid in the sample according to (C.2.4) below.

C.2.4 Calculation

$$\text{Total acidity, percentage by mass} = \frac{0.03646 \times V \times 100}{m}$$

where,

V is the volume, in milliliters of the standard volumetric sodium hydroxide solution (C.2.2.1) used for the titration; and

m is the mass, in g, of the sample taken.

NOTE : *If the sodium hydroxide standard volumetric solution is exactly 1 M equivalent weight shall be used in calculating the result. One milliliter of 1 M sodium hydroxide is equivalent to 0.0364 g of hydrochloric acid*

APPENDIX D DETERMINATION OF RESIDUE ON IGNITION

D.1 PRINCIPLE

Evaporation of test portion; conversion of salts to sulphates by treatment with sulphuric acid ignition; at 800 ± 50 °C and weighing.

D.2 APPARATUS

D.2.1 Platinum dish, capacity approximately 200 ml.

D.2.2 Electric furnace, temperature controlled at 800 ± 50 °C.

D.3 REAGENTS

D.3.1 Sulphuric acid: Approximately 1.84 g/ml density, about 96 per cent (m/m) or approximately 18 M solution.

D.4 PROCEDURE

D.4.1 Ignite the platinum dish (D.2.1) at 800 °C and cool in a desiccator and weigh to the nearest 0.1 mg. Weigh to the nearest 0.01 g, about 100 g of the test sample in the platinum dish.

D.4.2 Evaporate the test portion to obtain the final volume about 5 ml to 10 ml by carefully heating the dish, containing the test portion (on a sand bath). Allow to cool to ambient temperature. Add 1 ml of the sulphuric acid solution (**D.3.1**) and heat to dryness. Place the dish containing residue in the electric furnace (**D.2.2**) at 800 ± 50 °C temperature and keep at this temperature for about 15 min. Remove the dish from the furnace, place in a desiccator and weigh to the nearest 0.1 mg, after cooling to ambient temperature.

D.5 CALCULATION

Sulphated ash, percentage by mass = $\frac{m_1 \times 100}{m_2}$

where,

m_1 is the mass, in g, of the residue; and

m_2 is the mass, in g, of sample of the test sample (**D.4.1**).

APPENDIX E DETERMINATION OF SULPHATES CONTENT

E.1 FOR TECHNICAL GRADE

E.1.1 Regents

E.1.1.1 *Analytical reagent grade Sodium carbonate*

E.1.1.2 *Barium chloride solution, approximately 10 per cent (V/V).*

E.1.2 Procedure

Weigh about 10 g of the sample in a porcelain dish to the nearest 0.1 mg, add 0.5 g of sodium carbonate and evaporate to dryness. Moisten the residue with about 1 g of the material, weighed to the nearest milligram. Add 200 ml of boiling water, mix well and filter if necessary. Bring the contents to boil over a low flame and add 5 ml of hot barium chloride solution slowly with stirring. Boil the contents for 2 minutes and allow the precipitate to settle for 4 hours. Filter the supernatant liquid through a tarred sintered glass crucible (G No. 4) or a tarred Gooch crucible and transfer the precipitate carefully into the crucible. Wash thoroughly with hot water till the washings are free from chlorides. Heat the crucible at 105 °C to 110 °C. Cool the contents in a desiccator and weigh, repeat the heating, cooling and weighing operations until the difference in mass between two successive weighings does not exceed 5 mg.

E.1.3 Calculation

Sulphates (as H_2SO_4), percentage by mass = $\frac{42.02 m}{(m_1 + m_2)}$

where,

m is the mass, in g, of the precipitate;

m_1 is the mass, in g, of the sample taken for the test initially; and

m_2 is the mass, in g, of the sample added subsequently.

E.2 FOR GENERAL PURPOSE REAGENT GRADE

E.2.1 Reagents

As given in **E.1.1**.

E.2.2 Procedure

Follow the procedure as given in **E.1. 2** using about 50.0 g of the material, weighed to the nearest 0.01 g , instead of 10 g.

E.2.3 Calculation

Calculate as in **E.1.3**

E.3 FOR ANALYTICAL REAGENT, CHROMATOGRAPHIC AND SPECTROSCOPIC GRADES

E.3.1 Reagents

E.3.1.1 *Sodium carbonate*, of analytical reagent grade.

E.3.1.2 *Hydrochloric acid*, approximately 1 M, sulphate free.

E.3.1.3 *Barium chloride solution*, approximately 10 per cent (V/V).

E.3.1.4 *Standard sulphate solution*, Dissolve 0.178 g of potassium sulphate in water and make up the volume to 1 000 ml. Dilute 100 ml of the solution in a graduated flask to 1 000 ml. 1 ml of the dilute solution contains 0.01 mg of sulphate (as H₂SO₄).

E.3.2 Procedure

To about 20 g of the material, weighed to the nearest mg, add 20 mg of sodium carbonate and evaporate to dryness. Add 5 ml of water and 0.5 ml of hydrochloric acid to the residue, Filter, wash with water to make up to 10 ml and add 1 ml of barium chloride solution. Carry out a control test in the same manner using standard sulphate solution (limits) in place of the sample. Stir the two solutions and compare the turbidity produced.

E.3.3 Expression of results

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

APPENDIX F DETERMINATION OF IRON CONTENT

Two methods have been prescribed for the determination of Iron. The method prescribed in F.1 shall be the reference method and shall be carried out in case of any dispute.

F.1 METHOD 1 (BIPYRIDYL METHOD)

F.1.1 Apparatus

Photometer, any spectrophotometer suitable for measurement at a wave length of about 522 nm or a photoelectric absorption meter.

F.1.2 Reagents

F.1.2.1 *Dilute hydrochloric acid.* 1 M approximately, iron free.

F.1.2.2 *Hydroxylammonium chloride solution.* Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

F.1.2.3 *2, 2'-Bipyridyl solution.* Dissolve 1 g of 2, 2' - bipyridyl in 10 ml of dilute hydrochloric acid (1 N) and dilute to 100 ml with water.

F.1.2.4 *Standard iron solution A.* Dissolve 0.7022 g of ferrous ammonium sulphate ($\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6\text{H}_2\text{O}$) in water in a 1 000 ml volumetric flask, add 4 ml of concentrated sulphuric acid and make up to the mark with water. 1 ml of this solution contains 0.1 mg of iron (as Fe).

F.1.2.5 *Standard iron solution B.* Take 100 ml of the standard iron solution A (see **F.1.2.4**) and dilute to 1 000 ml with water in a 1 000 ml volumetric flask. This diluted solution should be freshly prepared. 1 ml of this solution contains 0.01 mg of iron (as Fe).

F.1.2.6 *Ammonium acetate solution.* Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

F.1.3 Procedure

F.1.3.1 Weigh to the nearest milligram, about 50 g of the sample in a platinum or silica dish (100 ml capacity), place on a boiling water bath and carefully evaporate to dryness. Cool and add 2 ml of hydrochloric acid and 25 ml of water and heat to facilitate dissolution. Transfer quantitatively to a 100 ml one mark volumetric flask, dilute to the mark, mix and filter if necessary. Transfer an aliquot of the sample solution containing between 50 μg and 500 μg of iron (Table 3) to a 100 ml one mark volumetric flask. Dilute to approximately 50 ml if necessary and add successively 2 ml of hydrochloric acid solution, 2 ml of hydroxylammonium chloride solution and after 5 minutes, 5 ml of ammonium acetate solution and 1 ml of 2, 2' – bipyridyl solution. Dilute to the mark, mix and allow to stand for 10 minutes. Carry out the measurement on the spectrophotometer at a wavelength of about 522 nm, adjusting the instrument to zero optical density using as reference the blank test given in **F.1.3.2**.

F.1.3.2 At the same time of the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the test.

F.1.3.3 Prepare a calibration curve by taking the quantities of standard iron solution shown in Table 3, in a series of eleven 100 ml volumetric flasks.

TABLE 3 – Volumes of the standard iron solution and corresponding masses

Volume of standard iron Solution B ml (1)	Corresponding mass of iron (Fe) µg (2)
0*	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

* Compensation solution

F.1.3.4 Add to each volumetric flask an amount of water, sufficient to dilute to approximately 50 ml, then 2 ml of dilute hydrochloric acid solution, 2 ml of hydroxylammonium chloride solution and after 5 minutes, 5 ml of ammonium acetate solution and 1 ml of 2,2'-bipyridyl solution. Dilute to the mark, mix thoroughly, allow to stand for 10 minutes.

F.1.3.5 Carry out the photometric measurements as in **F.1.3.1** adjusting the instrument to zero optical density using as reference the compensation solution. Prepare a calibration curve having, for example, the iron content in µg per 100 ml of the standard matching solution as abscissa and the corresponding values of absorbance as ordinate.

F.1.4 Calculation

Iron content (as Fe), percentage by mass = $\frac{m \times 100 \times 100}{V \times M}$

where,

m is the mass, in g, of iron-determined in the aliquot of sample solution;

V is the volume, in ml, of the sample taken for colour reaction; and

M is the mass, in g, of the test portion.

F.2 METHOD 2 (AAS METHOD)

Suitable Atomic Absorption Spectroscopy (AAS) method shall be used. for determination of Iron

**APPENDIX G
DETERMINATION OF FREE CHLORINE AND BROMINE****G.1 FOR TECHNICAL GRADE****G.1.1 Reagents**

G.1.1.1 *Potassium iodide, crystals.*

G.1.1.2 *Starch solution.* Make a paste of 0.5 g of starch with 2.5 ml of water. Transfer the paste in small quantities into 200 ml of water. Boil for 15 minutes with stirring. Preserve in small containers which shall be previously sterilized in boiling water for 2 hours.

G.1.1.3 *Potassium iodide solution.* 10 per cent (V/V)

G.1.1.4 *Standard sodium thiosulphate solution.* 0.1 M.

G.1.2 Procedure

G.1.2.1 Place about 20 ml of the test sample in a 100 ml conical flask, add 50 ml of water, one crystal of potassium iodide, 0.5 ml of starch solution and stir. If a blue colour appears indicating the presence of iodine, follow the procedure prescribed in **G.1.2.2** for determination of free chlorine and bromine. If no colour appears, proceed to determination of sulphites as in Appendix **H**.

G.1.2.2 Fill a ground glass stoppered weighing bottle with the test sample and take a portion of approximately 50 g, weighing by difference to the nearest 10 mg. Transfer the test portion to a conical flask fitted with ground glass stopper of capacity 500 ml, containing 100 ml of water. Stopper the flask and cool. Add to the conical flask 10 ml of potassium iodide solution, stopper the flask and shake. Allow to stand for 2 minutes and then add 1 ml of starch solution. Using standard sodium thiosulphate solution, titrate the iodine liberated until the blue colour disappears.

G.1.2.3 Since iron may interfere in the determination, carry out a blank determination using 100 ml of water, adding a water soluble ferric iron salt equivalent to that, contained in 50 g of the sample. Add other reagents as added to the sample. Subtract the volume of sodium thiosulphate solution required for the titration from the volume required with the sample.

G.1.3 Calculation

Free chlorine and bromine (as Cl), per cent by mass = $\frac{V \times m \times 100}{M}$

where,

V is the volume, in ml, of 0.1 N sodium thiosulphate solution used in titration (corrected for the blank);

m is the mass, in g, of chlorine corresponding to 1 ml of 0.1 N sodium thiosulphate solution (theoretical value, 1 ml = 0.00355 g of Cl); and

M is the mass, in g, of test portion.

G.2 FOR GENERAL PURPOSE REAGENT, ANALYTICAL REAGENT, CHROMATOGRAPHIC AND SPECTROSCOPIC GRADES

G.2.1 Apparatus

G.2.1.1 *Microburette.*

G.2.2 Reagents

G.2.2.1 *Cadmium iodide solution.* Dissolve 9.2 g of cadmium iodide in 100 ml of water.

G.2.2.2 *Starch solution.* as given in **G.1.1.2**.

G.2.2.3 *Standard sodium thiosulphate solution* 0.05 M and 0.005 M.

G.2.3 Procedure

G.2.3.1 Dilute 4 ml of the sample with 20 ml of freshly boiled and cooled water. Add 1 ml of cadmium iodide solution and 1 ml of starch solution and allow to stand in the dark for 10 minutes.

G.2.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if any blue colour produced on treating the material as in **G.2.3.1** is discharged by the addition of not more than 0.05 ml of 0.05 M sodium thiosulphate solution for general purpose reagent grade and not more than 0.05 ml of 0.005 M sodium thiosulphate solution in the case of analytical reagent grade.

APPENDIX H DETERMINATION OF SULPHITES CONTENT

H.1 FOR TECHNICAL AND GENERAL PURPOSE REAGENT GRADES

H.1.1 Reagents

H.1.1.1 *Standard iodine solution.* 0.1 M, freshly standardized.

H.1.1.2 *Standard sodium thiosulphate solution.* 0.1 M, freshly standardized.

H.1.1.3 *Starch solution.* as given in **G.1.1.2**

H.1.2 Procedure

Introduce 100 ml of freshly boiled water into a 500 ml glass stoppered conical flask and 10 ml of Standard iodine solution. Add about 50 g of the material weighed to ± 0.01 g while cooling. Stopper the flask, mix and titrate the excess iodine with Standard sodium thiosulphate solution after 2 minutes, adding 5 ml of starch solution towards the end of the titration.

H.1.3 Calculation

Sulphites (as SO_2), per cent by mass = $\frac{0.3203 (10 - V)}{m}$

where,

V is the volume, in ml, of the standard sodium thiosulphate solution used in titration; and

m is the mass, in g, of the material taken for the test.

H.2 FOR ANALYTICAL REAGENT, CHROMATOGRAPHIC AND SPECTROSCOPIC GRADES

H.2.1 Reagents

H.2.1.1 *Potassium iodide solution.* 10 per cent (V/V)

H.2.1.2 *Standard iodine solution.* 0.01 M.

H.2.1.3 *Starch solution.* as given in **G.1.1.2**

H.2.2 Procedure

To 400 ml of freshly boiled and cooled water add 1.5 ml of potassium iodide solution, 5 ml of hydrochloric acid and 2 ml of starch solution and titrate with 0.01 M iodine solution to a faint permanent blue colour. Add 100 g of the sample and titrate with 0.01 M iodine solution to

the same end point. The limit prescribed in Table 1 shall be taken as not having been exceeded, if not more than 0.25 ml of 0.01 M iodine is required for the titration.

APPENDIX J DETERMINATION OF HEAVY METALS

Atomic Absorption Spectroscopy (AAS) method shall be used to determine the metals and **SLS 312** describes the conventional methods for metals determination.

APPENDIX K DETERMINATION OF AMMONIUM COMPOUNDS

K.1 REAGENTS

K.1.1 Sodium hydroxide solution. 5 M, ammonia free.

K.1.2 Standard ammonia solution. Dissolve 3.15 g of ammonium chloride in 1000 ml of water. Further dilute 10 ml to 1000 ml with water. 1 ml of the diluted solution corresponds to 0.01 mg of ammonia.

K.1.3 Nessler's reagent. Dissolve 35 g of potassium iodide and 12.5 g of mercuric chloride in 800 ml of water and add a saturated solution of mercuric chloride until a slight permanent precipitate is produced. Then add 120 g of sodium hydroxide and dissolve. Add a little more mercuric chloride solution and sufficient water to make up to 1000 ml. Shake the solution occasionally for several days. Allow to settle and decant the clear liquid.

K.2 PROCEDURE

K.2.1 Dilute 2.8 ml (3.3 g) of the material with water to about 20 ml and add sodium hydroxide solution, until alkaline to litmus paper (about 7 ml is required). Dilute to 50 ml with water. Prepare a standard by diluting 1 ml of standard ammonia solution to 50 ml with water. To both solutions add 2 ml of Nessler's reagent and compare after 1 minute.

K.2.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the yellow colouration produced, if any, in the test with the material is not deeper than that of the standard.

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