भारतीय मानक मसौदा

क्लोरोसल्फोनिक अम्ल, तकनीकी — विशिष्टि

(*IS 7130 का तीसरा पुनरीक्षण*)

Draft Indian Standard

Chlorosulphonic Acid, Technical — Specification

(*Third Revision of IS 7130*)

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ICS 71.060.30

Inorganic Chemicals Sectional Committee, CHD 01	Last date of comments: 30 November 2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*Formal clauses will be added later*).

Chlorosulphonic acid is a versatile chemical, widely used for the production of detergents; pharmaceuticals and pharmaceutical intermediates; dye-stuff intermediates and organic chemicals.

This standard was first published in 1973 and subsequently revised in 1985 and 1994. In the second revision, a new requirement for free sulphur trioxide along with the relevant test method was incorporated. Also an alternate method for determination of purity, free sulphuric acid and free hydrochloric acid was included in the standard.

In this revision, instrumental test method for the determination of iron has been added as alternate test method. In addition to this, editorial corrections have been made wherever required. Also, Reference clause and Packing & Marking clause has been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard

CHLOROSULPHONIC ACID, TECHNICAL — SPECIFICATION

(*Third Revision*)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for chlorosulphonic acid, technical.

2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 REQUIREMENTS

3.1 Description

Chlorosulphonic acid is a strong inorganic acid which may be considered as the mono acid chloride of sulphuric acid ($ClSO_2OH$). It is light yellow to light brown, fuming, very corrosive liquid with a pungent odour.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex B. Reference to relevant clauses of Annex B is given in co1 (4) of Table 1.

4 PRECAUTIONS IN HANDLING AND STORING

The precautions in handling and storing of the material as given in IS 6156 shall be observed.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in drums or in tank cars.

5.2 Marking

The containers shall be securely closed and marked with the following details:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material; and
d) Lot or batch number in code or otherwise.

5.2.1 The containers shall be clearly labelled as shown in Fig. 7 of IS 1260 (Part 1). The following caution note shall also be marked on the containers:

> DANGER — VERY CORROSIVE ACID. CAUSES SEVERE BURNS. REACTS VIOLENTLY WITH WATER, HARMFUL IF INHALVED. MAY CAUSE DELAYED LUNG INJURY. CONTACT WITH WATER OR MOIST AIR RELEASES IRRITATING GAS.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

The procedure for drawing representative samples of the material and their criteria for conformity shall be as prescribed in Annex C.

Table 1 Requirements for Chlorosulphonic Acid, Technical

(*Clause* 3.1, B-3.2.3.1.1)

Sl No. (1)	**Characteristic** (2)	**Requirement** (3)	**Method of Test, Ref to Clause No.** (4)
i)	Purity (as $ClSO_2OH$), percent by mass, *Min*	98.0	**B-2**
ii)	Sulphuric acid (as H_2SO_4) , and free SO_3 (if any) (as SO_3), percent by mass, *Max*	2.0	**B-2**
iii)	Free hydrochloric acid (as HCl), percent by mass, *Max*	0.5	**B-2**
iv)	Iron (as Fe), percent by mass, *Max*	0.01	**B-3**

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision)*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous Goods (*first revision*)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (*first revision*)
(Part 65) : 2025/ ISO 17294-2 : 2023	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*second revision*)
IS 4161 : 2023	Nessler cylinder — Specification (*first revision*)
IS 4905 : 2015/ISO 24153 : 2009	Random Sampling And Randomization Procedures (*first revision*)
IS 6156 : 1971	Code of Safety for chlorosulphonic acid

ANNEX B

(*Clause* 3.2)

METHODS OF TEST FOR CHLOROSULHPONIC ACID, TECHNICAL

B-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTES

1) 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

2) Since chlorosulphonic acid is corrosive and produces burns on skin and reacts violently with water, it should be very carefully handled observing the information given in IS 6156.

B-2 DETERMINATION OF CHLOROSULPHONIC ACID, FREE SULPHURIC ACID AND FREE HYDROCHLORIC ACID

B-2.1 General

Two methods are prescribed, namely, Method A and Method B. In case of dispute, Method A shall be the referee method.

B-2.2 Method A

The sample is first oxidized with hydrogen peroxide and then titrated against standard sodium hydroxide solution using methyl red as indicator.

B-2.2.1 *Apparatus*

B-2.2.1.1 A weighing bottle, or a glass ampoule of the type shown in Fig. 1 may be used.

B-2.2.1.2 *Dely weighing tube*

This form of weighing tube has proven to be of exceptional value, to the busy works-chemist, in the analysis of oleum and mixed acids. Both speed and accuracy are gained by its use. The apparatus shown in Fig. 2, consists of a long glass tube of small bore, wound in a spiral coil.

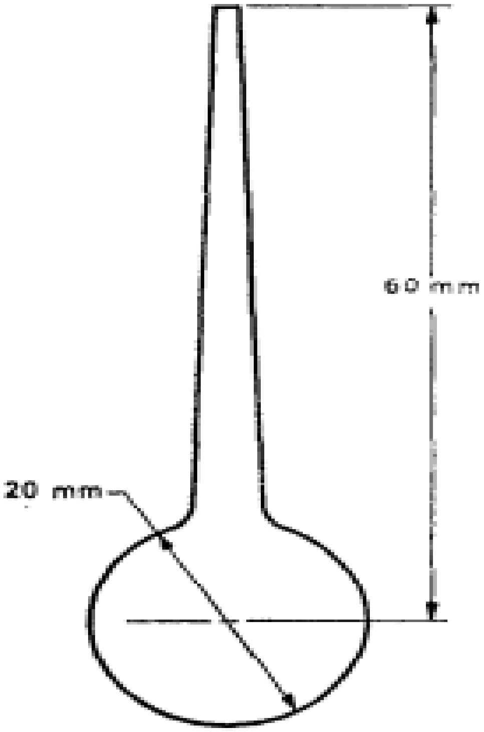
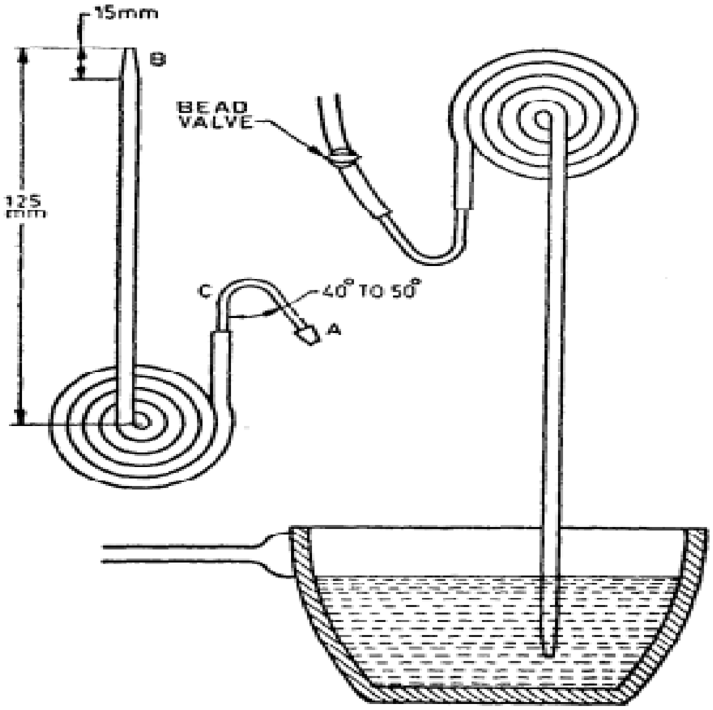


FIG 1 SPHERICAL GLASS AMPOULE

The sample of acid is drawn into a clean, dry and weighed Dely tube by applying suction through a rubber tube attached to *A* and drawing in the required amount of acid through tap *B*, a mark, ascertained by a previous run being made, to indicate the point to which the acid is drawn. The Dely tube is inverted, the tip *B* is carefully wiped off with tissue paper and the tube and sample weighed. The mass of the tube deducted gives the mass of the sample. The apparatus is now inclined so that the acid runs back in to the crook at *C* to a point marked on the wall of tube, in order to expel as much air as possible from this end. A rubber tube filled with water is attached to *A*, the other end of the rubber tube being connected to a bottle containing water. A glass bead, such as used in rubber tipped burettes, fitting snugly in this tube, regulates the flow of water. The Dely tube is now inverted, the tip being immersed in 150 ml to 200 ml of water in a 10 cm casserole (*see* Fig. 2). By pressing gently, on the bead, water is slowly admitted in the tube, forcing the acid before it. The acid and water are separated by a bubble of air. Before forcing out the last 10 to 15 ml of acid, the tube connected to the water supply is disconnected and the weak acid from the casserole drawn back 5 cm or 8 cm into the Dely tube by suction, then again the acid is almost entirely expelled by water from the reservoir and the procedure repeated. This is to absorb the sulphur trioxide gas that invariably is present in the bubble of air above mentioned, which would be lost if forced out directly by the water column. In order to facilitate this last step it is preferred to have a short rubber tube attached to the Dely tube, and a glass tip in the tube connected with the reservoir of water. The acid in the casserole, upon washing out the Dely tube, is titrated with standard sodium hydroxide solution according to the procedure for titration of acids to methyl orange end point. The tube is dried after washing with alcohol, followed by ether, by heating on an asbestos mat on a hot plate, dry air being aspirated through.



A — end of the tube;

B — tube of 5 mm bore and capillary drawn to 0.25 mm bore at *B*; and

C — mark on the tube.

FIG 2 DELY WEIGHING TUBE IN OPERATION

B-2.2.2 *Reagents*

B-2.2.2.1 *Hydrogen peroxide* — 60 g per litre solution, neutral to methyl red

B-2.2.2.2 *Standard sodium hydroxide solution* — 1 N, freshly standardized

B-2.2.2.3 *Methyl red indicator*

Dissolve 1 g of methyl red in 95 percent (*v*/*v*) ethanol and dilute to 100 ml with the same ethanol.

B-2.2.2.4 *Standard silver nitrate solution* — 0.1 N

B-2.2.2.5 *Potassium chromate indicator solution* —5 percent

B-2.2.3 *Procedure*

B-2.2.3.1 Accurately weigh about 2 g of the material in the weighing bottle or glass ampoule. If the glass ampoule is used, carefully mix the test sample by shaking the container. If the acid is partially crystallized, slightly heat the container until the sample is dissolved, then carefully mix again. Take the sample in a beaker or flask and slightly heat in a flame the bulb of glass ampoule previously weighed to the nearest 0.1 mg. Withdraw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in the oxidizing flame, without loss of glass. Remove the ampoule from 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.

Carefully place the ampoule containing the test portion into the conical flask containing 300 ml of cold water. Immerse the capillary end of the bulb if filled up to about two-third of its volume during cooling (2 ml to 3 ml approximately). Stopper the flask and shake to break the ampoule containing the test portion. Cool during this operation. Keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the conical flask. By means of a glass rod, grind the fragments of the ampoule and in particular the capillary which may have remained intact inspite of shaking. Withdraw the glass rod and wash it with water, collecting the washings in the conical flask.

Add 5 ml of hydrogen peroxide solution and gently boil for 10 min. Allow to cool, add two drops of methyl red solution and titrate to the end point by means of standard sodium hydroxide solution.

Neutralise this solution with dilute sulphuric acid and transfer to a 500 ml volumetric flask, dilute to the mark and mix. Pipette out 100 ml of the diluted solution into a 500 ml flask and titrate with silver nitrate solution using 1 ml of potassium chromate indicator.

B-2.2.4 Alternatively, one can also make use of Dely weighing tube (*see* **B-2.2.1.2**) for weighing instead of bulb. The weighed sample can be transferred with the help of cold water in a conical flask containing cold water. The titration then carried out as above.

B-2.2.5 *Calculation*

B-2.2.5.1 $\text{Total acidity (as SO}_3\text{), percent by mass}, a = \frac{VN}{M} \times 4.003$

where

V = volume in ml, of standard sodium hydroxide solution used,
M = normality of standard sodium hydroxide solution; and
M = mass in g, of the sample taken for the test.

B-2.2.5.2 $\text{Total chlorides (as HCI), percent by mass}, b = \frac{V_1 N_1}{M} \times 18.25$

where,

V_1 = volume in ml, of standard silver nitrate solution used,
N_1 = normality of standard silver nitrate solution; and
M = mass in g, of the material taken for the test.

B-2.2.5.3 Convert total chlorides (as HCI) to its equivalent sulphur trioxide. Total chlorides (as SO_3), b_1 = *b* [Total chlorides (as HCl), percent by mass] × 1.0967

B-2.2.5.4 Total sulphur trioxide (combined + free), percent by mass, *c* = *a* — b_1

B-2.2.5.5 Water, percent by mass *d* = 100 — (*c* + *b*)

B-2.2.5.6 This water (*see* **B-2.2.5.5**) is combined with a portion of sulphur trioxide to form sulphuric acid.

Sulphuric acid, percent by mass, *e* = *d* ×5.444 4

Sulphur trioxide combined with water, percent by mass, *f* = *d* ×4.444

B-2.2.5.7 Sulphur trioxide of chlorosulphonic acid and free sulphur trioxide, g = *c* — *f*

B-2.2.5.8 By inspection, it is possible to ascertain whether the product contains free sulphur trioxide or free hydrochloric acid since chlorosulphonic acid is in the proportion 31.29 percent hydrochloric acid and 68.71 percent sulphur trioxide.

a) *Case I* — If sulphur trioxide is in excess, that is, if g is greater than 68.71 then:
   Chlorosulphonic acid, percent by mass, *h.* = *b* × 3.195 6
   Free sulphur trioxide, percent by mass, *i* = 100 — (*h* + *e*)

b) *Case II* — If hydrochloric acid is in excess, that is, if b is greater than 31.29, then
   Chlorosulphonic acid, percent by mass, *j* = g × 1.455 5
   Free hydrochloric acid, percent by mass, *k* = 100 — (*j* + *e*).

B-2.2.5.9 Results shall be reported as follows:

a) Chlorosulphonic acid, percent by mass;
b) Sulphuric acid, percent by mass; and
c) Free sulphur trioxide or hydrochloric acid, if any.

B-2.3 Method B

The sample is titrated against standard sodium hydroxide solution using methyl red indicator and then sodium chloride solution formed in the same sample is titrated against standard silver nitrate solution, using potassium chromate as indicator.

B-2.3.1 *Apparatus*

A glass ampoule (as shown in Fig. 1) and a thick walled heavy glass bottle of capacity 250 ml to 300 ml fitted with a stopper.

B-2.3.2 *Reagents*

B-2.3.2.1 *Standard sodium hydroxide solution* — 1 N, freshly standardized

B-2.3.2.2 *Methyl red indicator*

Dissolve 1 g of methyl red in 95 percent (*v*/*v*) ethyl alcohol and dilute to 100 ml with the same ethyl alcohol

B-2.3.2.3 *Standard silver nitrate solution* — N 3 freshly standardized

B-2.3.2.4 *Potassium chromate indicator*

Dissolve 5 g of A.R. Grade potassium chromate in water and dilute to 100 ml.

B-2.3.3 *Procedure*

Weigh accurately 3 g of the material in a Dely tube or a small glass bulb. Place about 25 ml of water and a volume of 1 N standard sodium hydroxide solution, less than 10 ml is necessary to neutralize the sample in a heavy glass bottle. If the material is weighed in a Dely tube, run it into the standard sodium hydroxide solution according to the procedure prescribed in **B-2.2.1.2**. If the bulb is used, then stopper the bottle containing the bulb filled with the material, wrap in a towel and shake vigorously until the bulb breaks and acid mixes with water and standard sodium hydroxide. Titrate the excess acid with 1 N sodium hydroxide solution using methyl red as indicator.

Titrate the sodium chloride formed with N/3 standard silver nitrate solution, using potassium chromate solution as indicator. Calculate the volume of reagent used as equivalent to HCl. Since sodium hydroxide is apt to contain sodium chloride, carry out a blank titration and subtract the blank reading from the sample reading.

B-2.3.4 *Calculation*

Determine the composition of the acid as follows:

a) Total acid (as SO_3,), percent by mass = *a*
b) Make the sodium chloride solution obtained by titration of the total acidity by sodium hydroxide faintly acidic by adding dilute sulphuric acid and neutralize the solution by adding calcium carbonate. Titrate this solution with N/3 silver nitrate solution using potassium chromate indicator. Note the titre value as percent HCl to its equivalent SO_4 by multiplying by 1.097 8 (= *b*).

Total SO_3, percent by mass = $a — b$

Water, percent by mass = $100 – (a + b)$

Water combines with a portion of SO_3 to form sulphuric acid. Calculate to percent H_2SO_4 by multiplying by 5.444 4.

NOTE — Allowance should be given for impurities present as follows:

(100 — Impurities, non-titratable) — $(a + b)$ = Water, Percent by mass.

Subtract the SO_3 combined with water from the total SO_3. This value gives the SO_3 of the chlorosulphonic acid and free SO_3, if any.

NOTE — from the results it is possible to ascertain whether the product contains free SO_3 or free HCI since SO_3 and HCl are in the ratio 2.28:1.

If SO_3 is in excess, the HCl is calculated to SO_3 .HCI by multiplying by 3.195 6, the result is the chlorosulphonic acid, percent by mass in the sample.

Free SO_3, percent by mass = 100 — (SO_3. HCl + H_2SO_4).

If HCL is in excess, chlorosulphonic acid, percent by mass = 1.455 5 × SO_4 (percent by mass)

Free HCL, percent by mass = 100 – (SO_3.HCl + H_2SO_4).

Factors

HCl × 1.097 8 = SO_3,

HCl × 3.195 6 = SO_3. HCl, and

HCl × 2.195 9 = SO_3 in SO_3.HCl

SO_3 × 0.899 8 = HCl,

SO_3 × 1.455 5 = SO_3.HCl, and

SO_3 × 0.455 4 = HCl in SO_3.HCl

H_2O × 5.444 4 = H_2SO_4,

H_2SO_4 × 0.1837 = H_2O,

H_2SO_4 × 0.743 6 = HCl

NaCl × 0.623 8 = HCl,

SO_3.HCl = 31.29 percent by mass HCl and 68.71 percent by mass SO_3.

B-3 TEST FOR IRON

B-3.1 General

Three methods are prescribed for determining iron, namely, Method A (Bipyridyl Method), Method B (ICP-OES method) as prescribed at **B-4** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-3.2 Method A

B-3.2.1 *Apparatus*

B-3.2.1.1 *Nessler Cylinders* — 100 ml capacity (*see* IS 4161)

B-3.2.2 *Reagents*

B-3.2.2.1 *Concentrated hydrochloric acid* — *see* IS 265

B-3.2.2.2 *Ammonium persulphate*

B-3.2.2.3 *Potassium permanganate solution* — 0.1 N

B-3.2.2.4 *Butanolic potassium thiocyanate*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

B-3.2.2.5 *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4 . 6H_2O$] in 10 ml of dilute sulphuric acid, 10 percent (*v*/*v*). Add 0.1 N potassium permanganate solution drop wise till a faint colour of potassium permanganate persists and dilute with water to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of iron (as Fe).

B-3.2.3 Procedure

B-3.2.3.1 *Qualitative Test*

Weigh accurately 5 g of the sample and dissolve in about 175 ml of ice-cold water as described in **B-2.1.3.1**. Transfer the solution quantitatively to a 250 ml volumetric flask and dilute up to the mark with water at room temperature. Take 50 ml aliquot from the prepared solution in a 100 ml Nessler cylinder, add about 50 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Dilute with water to the mark. Mix well for 30 s and allow butanolic layer to separate. Carry out a control test in another Nessler cylinder with 1 ml of standard iron solution and dilute to 50 ml with water. Add 1 ml of hydrochloric acid, 50 mg ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Dilute with water to 100 ml. Mix well for 30 seconds.

B-3.2.3.1.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of red colour in the clear butanolic layer (upper) produced in the test with the material is not greater than that produced in the control test.

B-3.2.3.2 *Quantitative Test*

For the exact estimation of iron content, add to the control test (*see* **B-3.2.3.1**) standard iron solution slowly so as to match the colour with that of the sample colour. Note the amount of the standard iron solution required to match the colour.

B-3.2.3.3 *Calculation*

$$\text{Iron (as Fe), percent by mass} = \frac{0.05 \times A}{M}$$

where

A = volume in ml, of standard iron solution required to match the colour; and
M = mass in g, of the sample taken for the test (*see* **B-3.2.3.1**).

B-4 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-4.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

B-4.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* B-4.2 *and* B-4.4)

Sl No.	Element	Wavelength	Approximately Achievable Limits	Interfering Elements

		(nm)	Radial Viewing (µg/L)	Axial Viewing (µg/L)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

B-4.3 Reagents and Solutions

B-4.3.1. *Nitric Acid* (*65 Percent*) *Suprapure*

B-4.3.2 *Standard Stock Solution*

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of iron in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-4.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml and 0.5 µg/ml solution of respective elements under reference.

B-4.3.4 *Sample Preparation*

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument.

B-4.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

B-4.4 Instrument

Set up the instrument as per the manufacturer’s instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

B-4.5 Procedure

B-4.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-4.3.5**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

B-4.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the iron in the sample solution.

NOTE — It is recommended that IS 3025 (Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

B-4.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

a) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and

b) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

ANNEX C

(*Clause* 6)

SAMPLING OF CHLOROSULPHONIC ACID

C-1 GENERAL REQUIREMENTS FOR SAMPLING

C-1.1 Drawing, preparing, storing and handling of test samples shall be done according to IS 6156.

C-1.2 The samples shall be placed in suitable containers and each container shall be marked with full details of sampling giving the date of sampling, type of material, batch number, and indication of the source of manufacture.

C-2 SAMPLING INSTRUMENT

C-2.1 Sampling Tube

It is made of thick glass and is 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 3). The upper and lower ends are conical and reach 5 mm to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

C-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

C-3 SCALE OF SAMPLING

C-3.1 Lot

In any consignment, all the containers of the same size belonging to the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or different sizes of containers, the containers belonging to the same batch and same size shall be grouped together and each such group shall constitute a separate lot.

C-3.2 For ascertaining the conformity of the material in a lot to the requirement of this specification, tests shall be carried out for each lot separately.

C-3.3 The number of containers (n) to be chosen from the lot (*N*) shall depend on the size of in Table 3.

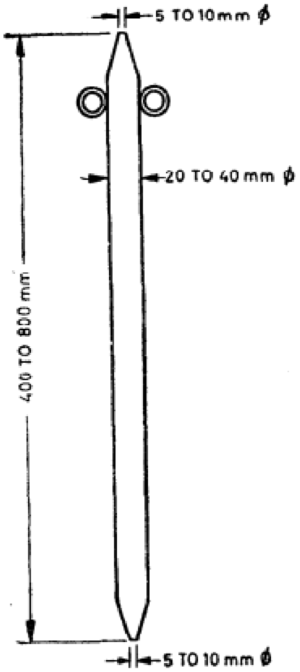


FIG. 3 SAMPLING TUBE

C-3.4 The containers to be selected for sampling shall be chosen at random from the lot. For this purpose, a random number table shall be used (*see* IS 4905). In case such a table is not available, the following procedure shall be adopted:

Starting from any container, count them as 1, 2,3 . . . . . . *r* , and so on in a systematic manner, where *r* is the integral part of *N/n*. Every r^{th} container thus counted shall be withdrawn to constitute the required sample.

Table 3 Scale of Sampling

(*Clause* C-3.3)

Sl No.	Lot Size *(n)*	Number of Containers to be Selected *N*
(1)	(2)	(3)
i)	2 to 15	2
ii)	16 to 50	3
iii)	51 to 100	4
iv)	101 to 300	5
v)	301 and above	6

C-4 NUMBER OF TESTS AND CRITERION FOR CONFORMITY

C-4.1 Tests for the determination of all the characteristics of this specification shall be carried out on the composite sample prepared according to **C-4.2.**

C-4.2 Draw with an appropriate sampling instrument (*see* **C-2**) equal portions of the material from each container selected in **C-3.3** and mix them thoroughly together to constitute a single composite sample. Divide this composite sample into three parts each sufficient for carrying out the intended tests and transfer them to thoroughly cleaned and dry sample containers. Send each one of these to the purchaser and the supplier. Reserve the third composite sample as referee sample bearing the seals of the purchaser and the supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

C-4.3 The material in any lot shall be accepted as conforming to the requirements of this specification if each of the test results satisfy the corresponding requirements.