भारतीय मानक मसौदा

कॉपर नाइट्रेट — विशिष्टि

(*IS 10895 का पहला पुनरीक्षण*)

Draft Indian Standard

Copper Nitrate — Specification

(*First Revision of IS 10895*)

(*Not to be reproduced without the permission of BIS or used as an Indian Standard*)

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01	**Last date of comments: 9th December 2024**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(formal clauses will be added later)

This standard was first published in 1984. In this revision, instrumental test methods for the determination of sulphate, chloride, lead and iron have been incorporated. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated and Packing & Marking clause has been updated.

Copper nitrate, popularly known as cupric nitrate, is an important salt of copper and finds use in light-sensitive reproductive papers, as ceramic colour, as mordant and oxidant in textile dyeing and printing, as reagent for burnishing iron, for giving a black ‘antique’ finish to copper, for colouring zinc brown, in nickel plating baths, in aluminium brighteners, in wood preservatives, in fungicides and herbicides, in pyrotechnic compositions, as catalyst component in solid rocket fuel, as nitrating agent for aromatic organosilicon compounds and as catalyst for organic reactions.

Copper nitrate is of two types: $Cu(NO_3)_2.6H_2O$ and $Cu(NO_3)_2.3H_2O$. The first one being unstable above 26.4 °C converting to the $Cu(NO_3)_2.3H_2O$. This specification is, therefore, for the stable trihydrate form.

Copper nitrate is prepared from copper scrap by reaction with nitric acid and crystallizing from the solution. For various uses different purity standards may be necessary. But there is no available standard on this item, except as a reagent grade chemical for use in laboratory for analysis. This standard is intended to be a step towards establishing a workable standard for use by industries and consumers.

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

COPPER NITRATE — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for copper nitrate.

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 editions of these standards.

3 GRADES

There shall be two grades of the material, namely.

a) *Technical Grade* — suitable for all other uses where pure grade is not required, and
b) *Pure Grade* — suitable for electroplating, catalyst for rocket fuels, catalyst for organic reaction, and analytical applications.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of blue deliquescent rhombic crystals or plates having the formula $Cu(NO_3)_2.3H_2O$. The material shall be free from visible contamination from dust or black particles and other extraneous adulterants and impurities.

4.2 The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in Annex B. Reference to relevant clauses of Annex B is given in co1 **5** of the table 1.

Table 1 Requirements for Copper Nitrate

(*Clause* 4.2, B-9.4.1, C-5.1.1, C-5.2)

SL No	Characteristic	Requirement		Method of Test (Ref to Cl No. in Annex A)
		Technical Grade	Pure Grade	
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, *Min*	98.0	99.0	**B-2**
ii)	Matter insoluble in acid, percent by mass, *Max*	0.5	0.01	**B-3**
iii)	Chloride (as Cl), percent by mass, *Max*	—	0.002	**B-4 or B-11**
iv)	Sulphate (as SO_4), percent by mass, *Max*	—	0.01	**B-5 or B-11**
v)	Substances not precipitated by hydrogen Sulphide (as sulphate) percent by mass, *Max*	—	0.05	**B-6**
vi)	Lead (as Pb), percent by mass, *Max*	0.2	0.005	**B-8 or B-12**
vii)	Iron (as Fe), percent by mass, *Max*	0.2	0.005	**B-9 or B-12**

viii)	Ammonium Sulphide metals other than iron (as Ni), percent by mass, *Max*	—	0.01	**B-10**

5 PACKING AND MARKING

5.1 Both technical and pure grade materials shall be packed in polyethylene lined hessian/HDP bags taking care to see that they are properly closed/ sealed to avoid contact with air and water.

5.2 Marking

The packages shall be marked legibly and indelibly with the following information:

a) Name and grade of material;
b) Mass of the material;
c) Lot or batch number in code or otherwise; and
d) Name of the manufacturer and his recognized trade-mark, if any.

5.2.1 *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 there under, and the products may be marked with the standard mark.

6 SAMPLING

The method of drawing representative samples from a lot, number of tests and the criteria for conformity shall be as prescribed in Annex C.

ANNEX A

(*Clause* 2)

(LIST OF REFERRED STANDARDS)

IS No	*Title*
IS 261 : 2023	Copper sulphate — Specification
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth 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) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)
IS 4905 : 2015 ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
IS 7017 : 1973	Method for colorimetric determination of traces of heavy metals by dithizone

ANNEX B

(*Clause* 4.2)

METHODS OF TEST FOR COPPER NITRATE

B-1 QUALITY OF REAGENTS

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

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

B-2 ASSAY

B-2.1 Two methods are prescribed for determining copper, namely, volumetric method and electrolytic method. The electrolytic method shall be regarded as the referee method.

B-2.2 Volumetric Method

B-2.2.1 *Outline of the Method*

Copper is determined with the addition of potassium iodide and titrating the liberated iodine against standard sodium thiosulphate solution.

B-2.2.2 *Reagents*

B-2.2.2.1 *Concentrated hydrochloric acid* **—** *see* IS 265.

B-2.2.2.2 *Glacial acetic acid*

B-2.2.2.3 *Potassium iodide — solid***.**

B-2.2.2.4 *Standard sodium thiosulphate* — 0.1 N.

B-2.2.3 *Procedure*

Weigh accurately about 0.7 g in a 250 ml beaker. Dissolve in 5 ml of water, add 5 ml of hydrochloric acid and evaporate to dryness. Cool, dissolve the residue with 5 ml of water and re-evaporate with 5 ml of hydrochloric acid to dryness on the steam bath. Dissolve the residue in water, transfer the solution to it glass-stoppered 250 ml conical flask and dilute with water to 75 ml. Add 2 ml of glacial acetic acid and 3 g of potassium iodide and then titrate the liberated iodine with 0.1 N sodium thiosulphate, shake and continue the titration until the blue colour disappears.

B-2.2.4 *Calculation*

$$\text{Copper nitrate [as Cu (NO}_3)_2.3\text{H}_2\text{O], percent by mass} = 24.16 \times \frac{VN}{M}$$

where

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

B-2.3 Electrolytic Method — Proceed as per the method given in IS 261.

B-3 MATTER INSOLUBLE IN ACID

B-3.1 Reagents

B-3.1.1 *Dilute Nitric Acid* — 1: 200 (*v/v*).

B-3.2 Procedure

Weigh accurately about 10 g of the material and dissolve in 100 ml of dilute nitric acid. Heat to boil, cool to room temperature. Filter through tared sintered glass crucible. Wash the residue with hot water till precipitate is free from acid. Dry the crucible at 105 °C to 110 °C and weigh to constant mass.

B-3.3 Calculation

$$\text{Matter insoluble in acid, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue; and
M_2 = mass in g of the material taken for the test.

B-4 CHLORIDES

B-4.1 Reagents

B-4.1.1 *Dilute Nitric Acid* **—** 30 percent (*v/v*).

B-4.1.2 *Silver Nitrate Solution* **—** 0.1 N.

B-4.1.3 *Standard Chloride Solution*

Dissolve 1.648 4 g of pure dry sodium chloride in water and dilute to 1 litre. Dilute 10 ml of this solution to one litre So that one ml contains 0.01 mg of chloride (as Cl).

B-4.2 Apparatus

B-4.2.1 *Nessler Cylinders* — 20 ml capacity.

B-4.3 Procedure

Weigh 1 g of the material in a 250 ml beaker and dissolve in 20 ml of water, add 1 ml of nitric acid and divide them into two equal portions in different cylinders. To one portion add 1 ml of silver nitrate solution, bring to a boil, let stand for 10 min. Cool, filter until clear and use this solution for the control test. To the other portion in the second cylinder add 1 ml of silver nitrate solution. If a turbidity is produced, it is not greater than is produced by adding 1 ml of standard chloride solution to the control and adjusting both to the same volume.

B-4.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **B-11**.

B-5 SULPHATES

B-5.1 Reagents

B-5.1.1 *Dilute Hydrochloric Acid* — 1: 2 (*v/v*).

B-5.1.2 *Dilute Hydrochloric Acid* — 1: 99 (*v/v*).

B-5.1.3 *Barium Chloride Solution* — 0.5 M (122.1 g of barium chloride dissolved in water and made up to 1 000 ml).

B-5.2 Procedure

Dissolve 5 g of the material in 15 ml of hot dilute hydrochloric acid (1:2) and evaporate to dryness on the steam bath. Dissolve the residue in about 15 ml of hot dilute hydrochloric acid (1:2) and again evaporate to dryness. Dissolve the residue in 100 ml of dilute hydrochloric acid (1:99) and filter. Heat to boiling, add 5 ml of barium chloride solution, digest in a covered beaker on the steam bath for 2 h and allow to stand overnight.

B-5.2.1 If a precipitate is formed, filter, wash thoroughly and ignite. The mass of the precipitate should not be more than 0.001 2 g greater than the mass obtained in the blank.

B-5.3 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at **B-11**.

B-6 SUBSTANCES NOT PRECIPITATED BY HYDROGEN SULPHIDE

B-6.1 Reagents

B-6.1.1 *Hydrogen Sulphide* — gas**.**

B-6.1.2 *Dilute Sulphuric Acid* — 1:95 (*v/v*).

B-6.2 Procedure

B-6.2.1 Dissolve 4 g of the material in about 190 ml of dilute sulphuric acid, heat to about 70 °C, and pass in hydrogen Sulphide until all the copper is precipitated. Dilute to 200 ml and filter. Evaporate about 150 ml of the filtrate to dryness in a tared dish and ignite.

B-6.2.2 The mass of the residue shall not exceed 0.001 5 g. Reserve the residue for the preparation of sample solution A (**B-7**).

B-7 SAMPLE SOLUTION A

To the residue obtained in the test for substances not precipitated by hydrogen Sulphide (**B-6.2.2**), add 3 ml of dilute hydrochloric acid (1:1) and 0.15 ml of nitric acid, cover with a watch glass, and digest on the steam bath for 15 to 20 min. Remove the watch glass and evaporate to dryness. Dissolve the residue in 1 ml of hydrochloric acid and dilute to 30 ml (1 ml = 0.1 g).

B-8 LEAD

B-8.1 For Pure Grade

B-8.2 Reagents

B-8.2.1 *Dilute Nitric Acid* — 1:99 (*v/v*).

B-8.2.2 *Lead* — Foil.

B-8.2.3 *Hydroxylamine Hydrochloride* — Solution.

B-8.2.4 *Dilute Ammonium Hydroxide* **—** 1 N, approximately.

B-8.2.5 *Ammonium Citrate*

B-8.2.6 *Potassium Cyanide Solution*

B-8.2.7 *Dithizone Solution* — 0.01 percent in chloroform. This solution is to be freshly prepared.

B-8.2.8 *Dilute Dithizone Solution* — 0.001 percent. Dilute 10 ml of the solution (**B-8.2.7**) to 100 ml with chloroform.

B-8.3 Procedure

In a well ventilated hood dissolve 1.5 g in 15 ml of dilute nitric acid in a glass-stoppered cylinder. Prepare a control containing 0.5 g of sample and 0.01 mg of lead ion (Pb) in 50 ml of dilute nitric acid in a glass-stoppered cylinder. To each solution add 2 ml of hydroxylamine hydrochloride reagent solution for Dithizone test. Add ammonium hydroxide dropwise until a permanent precipitate forms, shaking well after the addition to each drop. Add 5 ml of ammonium citrate reagent solution and 25 ml of potassium cyanide reagent solution. Cool in ice water for 15 min. To each add 3 ml of dithizone standard reagent solution in chloroform, shake for 2 min, and allow the layers to separate. Any pink colour of the chloroform laver from the solution of the sample should not exceed that of the chloroform layer from the control.

B-8.4 For Technical Grade

B-8.4.1 Carry out the test as prescribed in IS 7017.

B-8.5 Alternative Method

Lead may alternatively be determined by instrumental test method as prescribed at **B-12** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

B-9 IRON

B-9.1 For Pure Grade

B-9.2 Apparatus

B-9.2.1 *Nessler Cylinders* — 50 ml capacity.

B-9.3 Reagents

B-9.3.1 *Concentrated Hydrochloric Acid* **—** *see* IS 265.

B-9.3.2 *Ammonium Persulphate* **—** Solid.

B-9.3.3 *Butanol Potassium Thiocyanate Solution*

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

B-9.3.4 *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$Fe (NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to exactly 1 000 ml. Pipette out 10 ml of the solution and dilute with water to make up the volume to exactly 100 ml. One milliliter of this solution contains 0.01 mg of iron (Fe).

B-9.4 Procedure

Dissolve 1 g of the material in 20 ml of water and transfer to a Nessler cylinder. Add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up the volume to 50 ml. Shake vigorously for 30 s and allow the butanol layer to separate. Carry out a control test in the other Nessler cylinder using 5 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the two cylinders after 2 min.

B-9.4.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

B-9.5 For Technical Grade

B-9.6 Reagents

B-9.6.1 *Nitric Acid* — *see* IS 264.

B-9.6.2 *Hydrochloric Acid* — *see* IS 265.

B-9.6.3 *Ammonia*

B-9.6.4 *Ammonium Chloride*

B-9.7 Procedure

Weigh accurately about 10 g of the material and dissolve it in 10 ml nitric acid and 100 ml of water by heating. Precipitate iron by adding ammonia till the colour of the solution turns deep blue, heat to boil and filter. Wash with dilute solution of ammonia till the washings are colourless. Dissolve the residue in 10 ml of hydrochloric acid and 50 ml of water taken in a beaker. Add 5 g of ammonium chloride and stir to dissolve. Add, 20 ml of ammonia, boil, cool, filter. Wash the precipitate with water, dry the residue, ignite, and weigh to constant mass.

B-9.8 Calculation

$$\text{Iron content, percent by mass} = \frac{M_1 \times 70}{M_2}$$

where

M_1 = mass in g of the residue; and
M_2 = mass in g of the material taken for the test.

B-9.9 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at **B-12** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

B-10 AMMONIUM SULPHIDE METALS OTHER THAN IRON

B-10.1 Apparatus

B-10.1.1 *p*H meter.

B-10.2 Reagents

B-10.2.1 *Dilute Ammonium Hydroxide* — 15 percent NH_3 (*v/v*).

B-10.2.2 *Dilute Hydrochloric Acid* **—** 1: 1 (*v/v*)

B-10.2.3 *Hydrogen Sulphide Water* **—** saturated**.**

B-10.2.4 *Standard Nickel Solution*

Dissolve 6.73 g of nickel ammonium sulphate [$NiSO_4$ $(NH_4)_2SO_4.6H_2O$] in water and dilute to 1 000 ml. Dilute 10 ml of the solution to 1 000 ml. One millimeter of the solution contains 0.01 mg of nickel (as Ni).

B-10.3 Procedure

Ammonium Sulphide metals other than iron. To 10 ml of Sample Solution A (**A-7**) add a slight excess of ammonium hydroxide, boil for 1 min, filter, and wash with a small quantity of hot water. Dilute the filtrate and washings to 25 ml. adjust the *p*H of 5 ml of this solution to 7 (using a *p*H meter) with dilute hydrochloric acid, and dilute to 20 ml. Add 0.10 ml of ammonium hydroxide and 1 ml of hydrogen Sulphide water. Any colour should not exceed that produced by 0.02 mg of nickel ion (Ni) in an equal volume of solution containing the quantities of reagents used in the test.

B-11 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

B-11.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the *p*H of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

B-11.2 Equipment

B-11.2.1 *Anion Guard Column* — A protector of the separator column.

B-11.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

B-11.2.3 *Anion Suppressor Device* — Anion micro membrane suppressor is used to analyse the data.

B-11.2.4 *Detector* — Conductivity Detector.

B-11.2.5 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

B-11.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

B-11.3 Reagents

B-11.3.1 *Glass or Polyethylene Sample Bottles.*

B-11.3.2 *Distilled Water or Deionized Water free from the Anions of interest*.

B-11.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litres of water.

B-11.3.4 *Micro Membrane Suppressor Solution*: (0.025 N *of sulphuric acid*)

Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

B-11.4 Standard Solutions

B-11.4.1 *Chloride* — Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water.

B-11.4.2 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

B-11.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

B-11.6 Procedure

Dissolve between 1g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

B-11.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

B-12 DETERMINATION OF IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-12.1 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-12.2 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-12.2 and B-12.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing	Axial viewing	

			(µg)	**(µg)**	
(1)	(2)	(3)	(4)	(5)	(6)
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)		Cr, Fe
		217.00		(20)	

B-12.3 Reagents and Solutions

B-12.3.1. *Nitric Acid (65 percent) Suprapure*

B-12.3.2 *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, 100 or 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-12.3.3 *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, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

B-12.3.4 *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-12.3.5 *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-12.4 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-12.5 Procedure

B-12.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-12.3.5**). 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-12.5.2 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 lead (and/or Iron, calcium, arsenic) 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-12.6 Calculation

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

i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
ii) 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 COPPER NITRATE

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 In drawing, preparing, storing and handling test samples the following precautions and directions shall be observed.

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

C-1.5 Each sample container shall be sealed air-tight after filling and marked with filling details of sampling, the date of sampling and the year of manufacture of the material.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the material of same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

C-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

C-2.2 The number (*n*) of containers to be chosen from a lot shall depend on the size of the lot (*N*) and shall be in accordance with co1 **(2)** and (**3)** of Table 3.

C-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables (*see* IS 4905) shall be used. In case such tables are not available, the following procedure may be adopted:

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

Table 3 Number of Containers to be Selected

(*Clause* C-2.2)

SI No. (1)	Lot Size (*N*) (2)	Number of Containers to be Selected (*n*) (3)
i)	3 to 50	3
ii)	51 to 200	4
iii)	201 to 450	5
iv)	401 to 650	6
v)	651 and above	7

C-3 TEST SAMPLES AND REFEREE SAMPLE

C-3.1 Preparation of Test Sample

C-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **4** and shall not exceed 1 kg.

C-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, second for the supplier and the third to be used as referee sample.

C-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **C-1.5.** The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets or samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

C-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **C-3.1.2**) and a set of individual samples (*see* **C-3.1.3**) marked for this purpose. It shall also bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Test for the determination of copper nitrate shall be conducted on each of the individual samples for all the grades.

C-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples

C-5.1.1 *For Copper Nitrate*

The test results for copper nitrate shall be recorded, and the mean, and the range for these test results shall be calculated as follows;

Mean $(\bar{X})$ = Sum of the test results divided by the number of test results.

Range (R) = The difference between the maximum and the minimum values of the test results.

The lot shall be declared to have satisfied the requirement for this characteristic if the expression ($\bar{X} - 0.4\ R$) is greater than or equal to the prescribed limits in Table 1.

C-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements specified in Table 1 for the relevant grade.

C-5.3 The lot shall be declared as conforming to the requirements of the specification if **C-5.1** and **C-5.2** are satisfied.