

भारतीय मानक मसौदा
कॉपर सल्फेट – विशिष्टि
)आईएस 261 का तीसरा पुनरीक्षण

Draft Indian Standard
COPPER SULPHATE — SPECIFICATION
(*Third Revision of IS 261*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Copper sulphate is used in the manufacture and processing of various Industrial products, such as fungicide mixtures, dyeing cotton and silk, preserving hides and wood, tanning leather, electric batteries, process engraving, destroying algae in pools for rot proofing of jute bags; and for the manufacture of pigments. It is also used in fine chemicals, electroplating, pharmaceuticals and as a reagent in chemical analysis. This standard, however, does not cover the analytical reagent grade of the material and the material used for electroplating, as it is covered by IS 4847. Pharmaceutical use of the material is being excluded from this revision as the specifications of this grade are covered in the Indian Pharmacopoeia.

So far, copper sulphate has been manufactured by and large from copper scrap only. Recently, its production has started also from an indigenously available ore, namely, chalcopyrite. While preparing this revision, it has been ensured that specification for technical grade of the material would apply also to copper sulphate produced from chalcopyrite.

In this revision, instrumental test methods for the determination of aluminium, arsenic, copper, chlorides, iron and lead have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Amendment no. 1 and Reference clause have been incorporated.

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.

1 SCOPE

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

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provision 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 the standards Indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
296 : 1986	Specification for sodium carbonate, anhydrous (<i>third revision</i>)
460 (Part 1) : 2020	Test Sieves — Specification Part 1 wire cloth test sieves (<i>fourth revision</i>)
695 : 2020	Acetic Acid — Specification (<i>fourth revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)
3025(Part 2) : 2019 /ISO 11885 :	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
4161 : 1967	Specification for Nessler cylinders
8190 (Part 1) : 1988	Requirements for packing of pesticides : Part 1 solid pesticides (<i>second revision</i>)

3 GRADES

3.1 The material shall be of the following two grades:

a) *Technical (TECH)* — Used in dyeing cotton and silk; preserving hides and woods; tanning leather; electric batteries; process engraving, destroying algae in pools; rot proofing of jute bags; and manufacture of green and blue pigments; and fungicide mixtures (Bordeaux mixture).

b) *Pure* — Used in fine chemicals.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of blue, triclinic crystals, blue granules or light blue powder. The material slowly effloresces in dry air, turning to white blue powder.

4.2 The material, when tested as prescribed in Annex A, shall comply with the requirements given in Table 1. Reference to relevant clauses of Annex A is given in col 5 of the table.

Table 1 Requirements for Copper Sulphate
(Clauses 4.2, B-5.1.1, B-5.2 and B-5.3)

Sl. No.	Characteristic	Requirement		Method of Test, Ref to Cl No. in Annex A
		Tech (3)	Pure (4)	
(1)	(2)	(3)	(4)	(5)
i)	Copper, percent by mass, <i>Min</i>	24.7	25.1	A-3
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.20	0.05	A-4
iii)	Soluble iron and aluminium compounds (as Fe), percent by mass, <i>Max</i>	0.30	0.15	A-5 or A-11
iv)	pH value, not less than	3.0	3.5	A-6
v)	Chloride (as Cl), percent by mass, <i>Max</i>	—	0.01	A-7 or A-10
vi)	Arsenic (as As ₂ O ₃), mg/kg, <i>Max</i>	—	10	A-8 or A-11
vii)	Lead and zinc	—	to pass the test	A-9 or A-11

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in well closed containers as agreed to between the purchaser and the supplier.

5.1.1 The material, when used as a pesticide, shall also comply, with the packing requirements as specified in IS 8190 (Part 1) under the *Insecticides Act*, 1968.

5.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

- Name and grade of the material;
- Name of the manufacturer and his recognised trade-mark, if any;
- Gross and net mass;
- Date of manufacture; and
- Batch number.

5.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1.1 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

6.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A
(Clause 4.2)
METHODS OF TEST FOR COPPER SULPHATE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be, employed in tests.

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

A-2 PREPARATION OF TEST SAMPLE

A-2.1 Powder the material and sieve it through 1.00 mm IS sieve (*see* IS 460) keeping the sieved material in a clean glass stoppered weighing bottle for test purposes.

A-3 COPPER

A-3.1 Two methods are prescribed for determining copper, namely, volumetric method and electrolytic method. The electrolytic method shall be regarded as the referee method.

A-3.2 Volumetric Method

A-3.2.1 *Outline of the Method*

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

A-3.2.2 *Reagents*

A-3.2.2.1 *Sodium carbonate* — *see* IS 296.

A-3.2.2.2 *Potassium iodide* — crystals.

A-3.2.2.3 *Acetic acid* — *see* IS 695.

A-3.2.2.4 *Standard sodium thiosulphate solution* — 0.1 N.

A-3.2.2.5 *Starch indicator solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 l of boiling water. Boil for 3 min. Allow to cool and decant off the supernatant clear liquid.

A-3.2.2.6 *Potassium thiocyanate* — crystals.

A-3.2.3 *Procedure*

Dissolve about 1 g of the test sample (*see A-2*) accurately weighed, in 50 ml of water. Add a pinch of sodium carbonate till a slight turbidity appears. Then add 5 ml of acetic acid, 3 g of potassium iodide and titrate the liberated iodine with sodium thiosulphate solution, using starch as an indicator, until only a faint blue colour remains. Add about 2 g of potassium thiocyanate, shake and continue the titration until the blue colour disappears.

A-3.2.4 Calculation

$$\text{Copper, percent by mass} = 6.35 \times \frac{V N}{M}$$

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.

A-3.3 Electrolytic Method

A-3.3.1 Reagents

A-3.3.1.1 Dilute sulphuric acid — 33 percent (v/v).

A-3.3.1.2 Urea solution — 20 percent (m/v).

A- A-3.3.2 Procedure

Dissolve about 1 g of the test sample, accurately weighed in 200 ml of water. Add 5 ml of dilute sulphuric acid and heat to 60 - 70°C. Electrolyse for 50 min using a platinum gauze rotating anode and a platinum gauze stationary cathode as given in **A-3.3.2.1** and **A-3.3.2.2**.

A-3.3.2.1 Connect the anode and the weighed cathode to a 10-volt electro-analyser. Rotate the anode with the current on and test for 'short' across the electrodes. Stop the rotation and raise the beaker containing the solution until the electrodes are completely immersed. Restart the rotation, adjust the current to 0.5 A till copper starts depositing the gradually raise to 3 A to obtain a bright coherent deposit. Add 3 to 4 drops of urea solution half-way through the electrolysis.

A-3.3.2.2 At the end of electrolysis, the current being still on, lower the beaker slowly, washing the electrode with a jet of cold water from a wash bottle. Switch off the current and stop rotation. Remove the electrodes, wash the cathode first in rectified spirit and then in solvent ether and dry the cathodes for 5 min and about 110°C and weigh.

A-3.4 Calculation

$$\text{Copper, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the copper deposited on cathode, and

M_2 = mass in g of the copper sulphate taken for the test.

A-3.5 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD

A-3.5.1 Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

A-4 MATTER INSOLUBLE IN WATER

A-4.1 Reagents

A-4.1.1 *Concentrated Sulphuric Acid* — see IS 266.

A-4.2 Procedure

Weigh accurately about 10 g of the test sample and dissolve in 100 ml of water. Add 3 ml of sulphuric acid and stir thoroughly at room temperature. Filter through a tared filter paper or tared Gooch or sintered glass crucible (G No. 4). Wash the residue with water till it is free from acid. Dry the filter paper or crucible in an oven maintained at a temperature of 105 to 110°C till constant mass is obtained. For pure grade, use about 20 g of the test sample.

A-4.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the material taken for the test.

A-5 SOLUBLE IRON AND ALUMINIUM COMPOUNDS (as Fe)

A-5.1 Outline of the Method

Iron and aluminium are determined gravimetrically by precipitation with ammonium hydroxide.

A-5.2 Reagents

A-5.2.1 *Concentrated Nitric Acid* — see IS 264.

A-5.2.2 *Ammonium Chloride*

A-5.2.3 *Dilute Ammonium Hydroxide* — approximately 15 percent NH_3 (m/v).

A-5.2.4 *Dilute Hydrochloric Acid* — 33 percent (m/v).

A-5.3 Procedure

Take 10 g of the test sample (*see A-2*) and add 25 ml of water, 2 ml of nitric acid and 5 g of ammonium chloride. Make the solution alkaline by adding ammonium hydroxide solution. Keep it on a water bath until the precipitate has flocculated, keeping the solution alkaline by the addition of more ammonium hydroxide, if necessary. Filter and wash the residue with dilute ammonium hydroxide. Dissolve the residue in hot dilute hydrochloric acid. Make the solution again alkaline by adding ammonium hydroxide and allow the precipitate to settle. Filter and wash the residue with water. Dry the residue, ignite and weigh till a constant mass is obtained.

A-5.4 Calculation

$$\text{Soluble iron and aluminium compounds (as Fe) percent by mass} = \frac{70 \times M_1}{M_2}$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the sample taken for the test.

A-5.5 Alternative Method

Aluminium and iron may alternatively be determined by instrumental test method as prescribed in **A-11**.

A-6 pH VALUE

A-6.1 Procedure

Dissolve 5 g of the test sample (*see A-2*) in water and make up the volume to 100 ml. Determine the pH with glass electrodes using a suitable pH meter.

A-7 CHLORIDES

A-7.1 Outline of the Method

Chlorides are determined by comparison of the opalescence produced in a solution of the material with silver nitrate solution against the opalescence produced by a solution containing known amount of chloride.

A-7.2 Apparatus

A-7.2.1 Nessler Cylinders — 50 ml capacity (*see IS 4161*).

A-7.3 Reagents

A-7.3.1 Dilute Nitric Acid — approximately 4 N.

A-7.3.2 Silver Nitrate Solution — approximately 5 percent (*m/v*).

A-7.3.3 Standard Chloride Solution

Dissolve 0.1649 g of ignited sodium chloride in 1000 ml of water. Dilute 100 ml of the solution again to 1 l. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

A-7.4 Procedure

Dissolve 0.2 g of the test sample in 50 ml of water in a Nessler cylinder. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution and mix. Carry out a control test in the other Nessler cylinder in the same manner using 2 ml of standard chloride solution.

A-7.4.1 The limit as prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced in the test solution is not greater than that produced in the control test.

A-7.5 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed in **A-10**.

A-8 ARSENIC**A-8.1 Procedure**

Dissolve 1.0 g of the test sample in water and make up the volume to about 50 ml. Carry out the determination for arsenic as prescribed in IS 2088, using for comparison the stain obtained with 0.001 mg of arsenic trioxide (as As_2O_3).

A-5.5 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-11**.

A-9 LEAD AND ZINC**A-9.1 Reagents****A-9.1.1 Citric Acid**

A-9.1.2 Dilute Ammonium Hydroxide — approximately 15 percent NH_3 (m/v).

A-9.1.3 Potassium Cyanide Solution — 10 per cent (m/v).

A-9.1.4 Sodium Sulphide Solution — 10 percent (m/v).

A-9.2 Procedure

Dissolve one gram of the test sample in 10 ml of water. Add 1 g of citric acid and 10 ml of ammonium hydroxide followed by potassium cyanide solution, dropwise, until the blue colour is discharged. Add 1 drop of sodium sulphide solution. The material shall be taken to have passed the test if not more than a slight darkening and no opalescence is produced.

A-9.3 Alternative Method

Lead may alternatively be determined by instrumental test method as prescribed in **A-11**.

A-10 ION CHROMATOGRAPHY FOR CHLORIDES

A-10.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides. 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, 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 *pH* 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.

A-10.2 Equipment

A-10.2.1 *Anion guard column* — a protector of the separator column.

A-10.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

A-10.2.3 *Anion Suppressor device* — Anion micromembrane suppressor is used to analyse the data
Detector: Conductivity detector.

A-10.2.4 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 μl , 200 μl , 500 μl or 1000 μl be used to determine ionic concentration as per instrument manual and practice.

A-10.3 Reagents and Standards

A-10.3.1 *Glass or polyethylene sample bottles.*

A-10.3.2 *Distilled water or deionized water free from the anions of interest.*

A-10.3.3 *Eluent* — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-10.3.4 *Micromembrane suppressor solution* (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

A-10.4 Standard solutions

A-10.4.1 *Chloride* — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-10.5 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 0.1-1.0 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.

A-10.6 Procedure

Dissolve between 1 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 (0.1-1.0 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 and deviation of retention force shall not exceed ± 10 percent of RT of calibration. 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 clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-10.7 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.

A-11 DETERMINATION OF ALUMINIUM, ARSENIC IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-11.1 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.

A-11.2 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.

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

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti
iv)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

A-11.3 Reagents and Solutions**A-11.3.1 Nitric acid (65 percent) Suprapure****A-11.3.2 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 1000 $\mu\text{g}/\text{ml}$ of aluminium, arsenic, iron and lead in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-11.3.3 Standard solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask & make up volume with 2 percent nitric acid to prepare 5 $\mu\text{g}/\text{ml}$ solution. From this 5 $\mu\text{g}/\text{ml}$ 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 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

A-11.3.4 Sample preparation

Weigh about 2.5 g polyphosphoric acid 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

A-11.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (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.

A-11.4 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

A-11.5 Procedure

A-11.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-11.5.2). 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.

A-11.5.2 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 $\mu\text{g/ml}$ of the aluminium, arsenic, iron and lead 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.

A-11.6 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 B
(Clause 6.1)
SAMPLING OF COPPER SULPHATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

B-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.

B-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.

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

B.2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material of one 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.

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

B-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 col 1 and 2 of Table 2.

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables 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 on in a systematic manner, where r is the integral part of N/n . Every r^{th} container thus counted shall be taken out for drawing samples.

Table 3 Number of Containers to be selected
(Clause B-2.2)

Lot Size	Number of Containers
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(<i>N</i>) (1)	to be selected (<i>n</i>) (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-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 3 and shall not exceed 1 kg.

B-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, another for the supplier and the third to be used as referee sample.

B-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 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 **B-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 of samples in such a way that each set has an individual test sample representing each container selected. One of these sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-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.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of copper shall be conducted on each of the individual samples for all the grades.

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

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 *For Copper Sulphate*

The test results for copper 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 value of expression ($\bar{X} - 0.6 R$) shall be calculated. If the values of this expression is greater than or equal to the minimum limit specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirement for this characteristic.

B-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.

B-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1 for the relevant grade.