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IS: 261 - 1982 (Reaffirmed 2010)

Indian Standard SPECIFICATION FOR COPPER SULPHATE (Second Revision)

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

Gr 4 December 1982

AMENDMENT NO. 1 JUNE 1986

TO

IS:261-1982 SPECIFICATION FOR COPPER SULPHATE

(Second Revision)

(Page 13, clauses B-4.1 and B-5.1.1) - The expression 'Copper sulphate' shall be read as 'Copper'.

(Page 13, clause B-5.1.1, last paragraph) - Substitute the following for the existing paragraph:

'The value of expression '(\overline{X} - 0.6 R)'-shall be calculated. If the value 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.'

(CDC 3)

Indian Standard SPECIFICATION FOR COPPER SULPHATE

(Second Revision)

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Indian Standard SPECIFICATION FOR COPPER SULPHATE

(Second Revision)

0. FOREWORD

- **0.1** This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 28 January 1982, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.
- **0.2** 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: 4847-1979*. Pharmaceutical use of the material is being excluded from this revision as the specifications of this grade are covered in the Indian Pharmacopoeia.
- **0.3** 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.
- **0.3.1** This revision provides for expression of the limits of copper sulphate in terms of copper content instead of CuSO₄, 5H₂O. It is hoped that this would help in overcoming controversies emanating from the loss of water of crystallization.
- **0.4** 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: 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

†Rules for rounding off numerical values (revised).

^{*}Specification for copper salts for electroplating (first revision).

1. SCOPE

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

2. GRADES

- **2.1** 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.

3. REQUIREMENTS

- **3.1 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.
- **3.2** The material, when tested as prescribed in Appendix A, shall comply with the requirements given in Table 1. Reference to relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR COPPER SULPHATE

(Clauses 3.2, B-5.1.1, B-5.2 and B-5.3)

CI NO	CHARACTERICTIC	REQUIREMENT		METHOD OF TEST,	
(1)	. CHARACTERISTIC (2)	Tech (3)	Pure (4)	REF TO CL NO. IN APPENDIX A (5)	
i)	Copper, percent by mass, Min	24.7	25.1	A-2	
ii)	Matter insoluble in water, percent by mass, Max	0.20	0.05	A-3	
iii)	Soluble iron and aluminium compounds (as Fe), percent by mass, Max	0.30	0.15	A-4	
iv)	pH value, not less than	3.0	3.5	A-5	
V)	Chloride (as Cl), percent by mass, Max	_	0.01	A-6	
vi)	Arsenic (as As_2O_3), mg/kg , Max	_	10	A-7	
vii)	Lead and zinc	_	to pass the test	A-8	

4. PACKING AND MARKING

- **4.1 Packing** The material shall be packed in well closed containers as agreed to between the purchaser and the supplier.
- **4.1.1** The material, when used as a pesticide, shall also comply, with the packing requirements as specified in IS: 8190 (Part I)-1976* under the *Insecticides Act*, 1968.
- **4.2 Marking** The packages shall be securely closed and bear legibly and indelibly the following information:
 - a) Name and grade of the material;
 - b) Name of the manufacturer and his recognised trade-mark, if any;
 - c) Gross and net mass;
 - d) Date of manufacture; and
 - e) Batch number.
 - **4.2.1** The packages may also be marked with the Standard Mark

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for The use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

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

^{*}Requirements for packing of pesticides (Part I) Solid pesticides

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR COPPER SULPHATE

A-0. QUALITY OF REAGENTS

A-0.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be, employed in tests.

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

A-1. PREPARATION OF TEST SAMPLE

A-1.1 Powder the material and sieve it through 1.00 mm IS sieve (see IS : 460-1977†) keeping the sieved material in a clean glass stoppered weighing bottle for test purposes.

A-2. COPPER

A-2.0 Two methods are prescribed for determining copper, namely, volumetric method and electrolytic method. The electrolytic method shall be regarded as the referee method.

A-2.1 Volumetric Method

A-2.1.0 Outline of the Method — Copper is determined with the addition of potassium iodide and titrating the liberated iodine against standard sodium thiosulphate solution.

A-2.1.1 Reagents

- **A-2.1.1.1** *Sodium carbonate see* IS : 296-1974‡.
- **A-2.1.1.2** *Potasium iodide* crystals.
- **A-2.1.1.3** Acetic acid—see IS: 695-1975§.
- **A-2.1.1.4** *Standard sodium thoisulphate solution* 0.1 N.

^{*}Specification for water for general laboratory use (second revision).

[†]Specification for test sieves: Part I Wire cloth test sieves (second revision).

Part II Perforated plate test sieves (second revision).

[‡]Specification for sodium carbonate, anhydrous (second revision).

[§]Specification for acetic acid (second revision).

A-2.1.1.5 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 one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

A-2.1.1.6 *Potassium thiocyanate* — crystals.

A-2.1.2 Procedure — Dissolve about 1 g of the test sample (see A-1) 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-2.1.3 Calculation

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 lor the test.

A-2.2 Electrolytic Method

A-2.2.1 Reagents

- **A-2.2.1.1** Dilute sulphuric acid 33 percent (v/v).
- **A-2.2.1.2** *Urea solution* 20 percent (m/v).
- A-2.2.2 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 minutes using a platinum gauze rotating anode and a platinum gauze stationary cathode as given in A-2.2.2.1 and A-2.2.2.2.
- A-2.2.2.1 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 ampere till copper starts depositing the gradually raise to 3 amperes to obtain a bright coherent deposit. Add 3 to 4 drops of urea solution half-way through the electrolysis.

A-2.2.2. 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 five minutes and about 110°C and weigh.

A-2.3 Calculation

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. MATTER INSOLUBLE IN WATER

A-3.1 Reagents

- **A-3.1.1** Concentrated Sulphuric Acid see IS: 266-1977*.
- A-3.2 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-3.3 Calculation

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-4. SOLUBLE IRON AND ALUMINIUM COMPOUNDS (as Fe)

A-4.0 Outline of the Method — Iron and aluminium are determined gravimetrically by precipitation with ammonium hydroxide.

A-4.1 Reagents

A.4.1.1 Concentrated Nitric Acid — see IS: 264-1976†.

^{*}Specification for sulphuric acid (second revision).

[†]Specification for nitric acid (second revision).

A-4.1.2 Ammonium Chloride

- A-4.1.3 Dilute Ammonium Hydroxide approximately 15 percent NH₃ (m/v).
 - **A-4.1.1** Dilute Hydrochloric Acid 33 percent (m/v).
- **A-4.2 Procedure** Take 10 g of the test sample (see **A-1**) 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-4.3 Calculation

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

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. pH VALUE

A-5.1 Procedure — Dissolve 5 g of the test sample (see **A-1**) in water and make up the volume to 100 ml. Determine the pH with glass electrodes using a suitable pH meter.

A-6. CHLORIDES

A-6.0 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-6.1 Apparatus

A-6.1.1 Nessler Cylinders — 50-ml capacity (see 18:4161-1967*).

^{*}Specification for Nessler cylinders.

A-6.2 Reagents

- **A-6.2.1** Dilute Nitric Acid approximately 4 N.
- **A-6.2.2** Silver Nitrate Solution approximately 5 percent (m/v).
- A-6.2.3 Standard Chloride Solution Dissolve 0.164 9 g of ignited sodium chloride in 1 000 ml of water. Dilute 100 ml of the solution again to one litre. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).
- **A-6.3 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-6.3.1** 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. ARSENIC

A-7.1 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: 2088-1971*, using for comparison the stain obtained with 0.001 mg of arsenic trioxide (as As_2O_3).

A-8. LEAD AND ZING

A-8.1 Reagents

- A-8.1.1 Citric Acid
- **A-8.1.2** Dilute Ammonium Hydroxide approximately 15 percent NH₃ (m/v).
 - **A-8.1.3** Potassium Cyanide Solution 10 per cent (m/v).
 - **A-8.1.4** Sodium Sulphide Solution 10 percent (m/v).
- **A-8.2 Procedure** Dissolve one gram of the test sample in 10 ml of water. Add one gram of citric acid and 10 ml of ammonium hydroxide followed by potassium cyanide solution, dropwise, until the blue colour is discharged. Add one 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.

^{*}Methods for determination of arsenic (first revision).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF COPPER SULPHATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

- **B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.
- **B-1.1** Precaution shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.
- **B-1.2** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.
- **B-1.3** 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-1.4** 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 rth container thus counted shall be taken out for drawing samples.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

(*Clause* B-2.2)

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(N)	(n)
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 he 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-1.4**. 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 sulphate 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 sulphate shall be recorded and the mean and the range for these test results shall be calculated as follows:
 - Mean (\overline{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 ($\overline{x} \pm 0.6 R$) shall be calculated. If the values of this expression lie within the limits specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirements 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.

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