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भारतीय मानक मसौदा
इलेक्ट्रोप्लेटिंग के लिए कॉपर साल्ट — विशिष्टि
(IS 4847 का तीसरा पुनरीक्षण)

Draft Indian Standard

Copper Salts for Electroplating — Specification

(Third Revision of IS 4847)

(ICS 25.220.40)

Electroplating Chemicals and Photographic Materials Sectional
Committee, CHD 5

Last Date for Comments: 25 June 2024

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

FOREWORD

(formal clauses will be added later)

This standard was first published in 1968 under the title 'Copper cyanide for electroplating'. The Committee responsible for this specification felt the necessity of enlarging its scope by covering other copper salts for plating purposes to make it more comprehensive. Accordingly, this was first revised in 1979 when the requirements for copper chloride, copper cyanide, copper potassium cyanide, copper sodium cyanide, copper pyrophosphate, copper sulphate were covered. In the second revision in 2006, requirement for copper fluoborate was added.

In this revision, alternative instrumental test methods AAS, ICP-MS and ICP-OES have been incorporated for the determination of iron and arsenic and the standard has been updated based on the experience of last two decades and on the currently available data.

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 SALTS FOR ELECTROPLATING — SPECIFICATION
(Third Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for the following copper salts for electroplating:

- a) Copper chloride,
- b) Copper cyanide,
- c) Copper potassium cyanide,
- d) Copper sodium cyanide,
- e) Copper pyrophosphate,
- f) Copper sulphate, and
- g) Copper fluoborate.

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 agreement based on this Indian Standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 REQUIREMENTS

3.1 Description

Copper salts shall conform to the following descriptions:

<i>Name of Salt</i>	<i>Description</i>
Copper chloride	Green crystalline powder, free from foreign matter and corresponding essentially to the formula $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$
Copper cyanide	Cream coloured powder, free from foreign matter corresponding essentially to the formula CuCN
Copper potassium cyanide	White crystalline powder, free from foreign matter and corresponding essentially to the formula $\text{K}_2\text{Cu}(\text{CN})_3$.
Copper sodium cyanide	White powder, free from foreign matter and corresponding essentially to the formula $\text{Na}_2\text{Cu}(\text{CN})_3$.
Copper pyrophosphate	Free flowing light blue powder, free from foreign matter having a slightly varying degree of hydration and corresponding essentially to the formula $\text{Cu}_2\text{P}_2\text{O}_7 \cdot 3\text{H}_2\text{O}$.

Copper sulphate	Crystalline or finely granulated powder, free from foreign matter and corresponding essentially to the formula $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.
Copper fluoborate	Blue coloured concentrate free from foreign matter and essentially containing $\text{Cu}(\text{BF}_4)_2$.

3.2 Copper salts shall also comply with the requirements prescribed in Table 1 when tested in accordance with the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in Col 10 of Table 1.

4 SAFETY PRECAUTIONS

Cyanide salts of copper are highly poisonous and shall be handled with extreme caution. Useful information on this subject is given in Annex C.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in airtight containers, preferably with replaceable closure. The plater usually needs to withdraw small quantities at a time from the container without the material being scattered about.

Table 1 Requirements for Copper Salts for Electroplating

(Clause 3.2, B-7.2.2.1, D-5.1.1, D-5.2)

Sl No.	Characteristics	Copper Chloride	Copper Cyanide	Copper Potassium Cyanide	Copper Sodium Cyanide	Copper Pyro-phosphate	Copper Sulphate	Copper Fluoborate	Method of Test Ref to clause No. of Annex B
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	Copper (as Cu), percent by mass, <i>Min</i>	36.7	69.0	28.5	32.0	32.5	24.9	13.2 (for a 50 percent concentrate)	B-2
ii)	Cyanide (as CN), percent by mass, <i>Min</i>	—	27.5	35.0	39.0	—	—	—	B-3
iii)	Pyrophosphate (as P_2O_7), percent by mass, <i>Min</i>	—	—	—	—	44.5	—	—	B-4
iv)	Fluoborate (as BF_4), percent by mass, <i>Min</i>	—	—	—	—	—	—	36.0	B-5
v)	Insoluble matter, percent by mass, <i>Max</i>	0.3	0.15	0.1	0.25	0.1	0.1	—	B-6

vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.015	0.05	—	0.02	—	0.075	0.03	B-7
vii)	Arsenic (as As), percent by mass, <i>Max</i>	—	—	—	—	0.002 5	0.001 5	—	B-8
viii)	Chloride (as Cl), percent by mass, <i>Max</i>	—	0.4	—	0.05	—	0.02	0.01	B-9
ix)	Sulphate (as SO ₄), percent by mass, <i>Max</i>	0.015	—	—	0.05	—	—	0.04	B-10

The container should, therefore, have an opening of such a size that the material may be easily withdrawn and then the opening may be resealed without difficulty.

5.2 Marking

The containers shall be marked with the following:

- Name of the material and its net mass;
- Name of manufacturer and his recognized trade-mark, if any;
- Batch number to enable the date of manufacture to be traced from records; and
- The Word 'POISON' and appropriate symbol [*see* IS 1260 (part)].

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 thereunder, and the products may be marked with the standard mark.

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to the standard shall be determined in accordance with the method given in Annex D.

ANNEX A

(Clause 2)

(LIST OF REFERRED STANDARDS)

IS No	Title
IS 264 : 2006	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>third revision</i>)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (<i>first revision</i>)
IS 2088 : 2023	Methods for determination of arsenic (<i>third revision</i>)
IS 3025 (Part 2) : 2019 ISO 11885 : 2007	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>)
IS 3025 (Part 65) : 2022 ISO 17294-2 : 2016	Methods of sampling and test physical and chemical for water and wastewater Part 65 Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including Uranium Isotopes (<i>first revision</i>)
IS 4905 : 2015 ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 11124 : 1984	Method for atomic absorption spectrophotometric determination of arsenic
IS 13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method

ANNEX B

(Clause 3.2 and Table 1)

METHODS OF TEST FOR COPPER SALTS FOR ELECTROPLATING

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 results of analysis.

B-2 DETERMINATION OF COPPER

B-2.1 General

Copper is determined by the electrolytic method.

B-2.2 Apparatus

B-2.2.1 Electrodeposition Apparatus

Provided with magnetic stirrer or rotation platinum gauze cathode.

B-2.2.2 pH Meter

B-2.3 Reagents

B-2.3.1 Concentrated Nitric Acid — relative density 1.42 (*see* IS 264).

B-2.3.2 Nitric Acid — relative density 1.20.

B-2.3.3 Dilute Sulphuric Acid — 50 percent (v/v) and 10 percent (v/v).

B-2.3.4 Acetone

B-2.3.5 Hydrogen Cyanide Detector papers

Prepare as required by dipping strips of filter paper in a mixture of equal volumes of solution A and B prepared as follows:

- a) *Solution A* — 0.1 percent (m/v) *o*-tolidine acetate. Dissolve 0.64 g of *o*-tolidine in 5 ml of 50 percent (m/v) glacial acetic acid and dilute to 1 000 ml with water.
- b) *Solution B* — 0.3 percent (m/v) copper acetate solution in water.

B-2.3.6 Tetrasodium Pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$)

B-2.3.7 Sodium Sulphide Solution

10 percent (m/v) freshly prepared solution of sodium sulphide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$).

B-2.4 Preparation of the Sample Solution

B-2.4.1 Copper Cyanide, Copper Potassium Cyanide and Copper Sodium Cyanide

Weigh accurately about 1 g of the material and transfer to a 250 ml beaker with 50 ml of water. Transfer the beaker to a well-ventilated fume chamber. Cautiously add 5 ml of concentrated nitric acid, cover the beaker with a watch-glass and boil until all hydrogen cyanide is expelled (Detector paper not turned blue in vapour from beaker). Rinse the watch-glass and sides of the beaker with water, add 5 ml of dilute sulphuric acid (50 percent v/v) and evaporate to a volume of 25 ml. Cool, dilute to 200 ml with water and proceed according to **B-2.5**.

B-2.4.2 Copper Chloride and Copper Sulphate

Weigh accurately about 1 g of the material and transfer to a 250 ml beaker with 50 ml of water. Add 5 ml of dilute sulphuric acid (50 percent v/v) and 5 ml of concentrated nitric acid, swirl until the sample has dissolved and evaporate to a volume of 25 ml. Cool, dilute to 200 ml with water and proceed according to **B-2.5**.

B-2.4.3 Copper Pyrophosphate

Weigh accurately about 4 g of tetrasodium pyrophosphate and transfer to a 400 ml beaker with 150 ml of water and stir until dissolved. Weigh accurately 1 g of the material and transfer to the solution of tetrasodium pyrophosphate. Stir until dissolved, heating if necessary. Adjust the *pH* between 8.5 to 9.0 using a few drops of dilute sulphuric acid (10 percent v/v) taking care to wash the electrodes of the *pH* meter with a jet of water and collecting the washings in the beaker. Heat to about 60 °C and proceed according to **B-2.5**.

B-2.4.4 Copper Fluoborate

Transfer with a graduated pipette an exactly weighed quantity of about 2 g of the concentrate to a 400 ml beaker, preferably of plastic, add 150 ml of water and proceed according to **B-2.5**.

B-2.5 Procedure

Transfer the beaker containing the prepared solution to the electro-deposition apparatus, fit the tarred cathode and cover with a split watch-glass. Electrolyze using a current of 2.5 A to 3.0 A (0.5 A for copper pyrophosphate) with a rotating cathode or a magnetic stirrer until the solution is colourless. Rinse the watch-glass and the inner walls of the beaker with water and continue the electrolysis for a further period of 10 min. Confirm complete deposition of copper by mixing 1 drop of electrolyte with 1 drop of sodium sulphide solution on a white marble tile. A black precipitate of copper indicates incomplete electrolysis.

B-2.5.1 When electrolysis is complete, rinse the electrodes and without interrupting the current, wash the cathode with a fine stream of water, collecting the rinsings in the beaker. Retain the electrolyte of copper pyrophosphate for the determination of pyrophosphate (*see B-4*) and those of other salts for iron, etc.

B-2.5.2 Switch off the current, remove the cathode, rinse it with water and then twice with acetone. Dry at 105 °C and weigh.

NOTE — after use, strip the deposit from the cathode by immersing it in nitric acid of relative density 1.20

B-2.6 Calculation

$$\text{Copper (as Cu), percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = increase in mass of cathode, in g; and

M = mass of the material taken for test, in g.

B-3 DETERMINATION OF CYANIDE

B-3.1 Apparatus

B-3.1.1 Distillation Apparatus

Provided with 500 ml distillation flask, water-cooled condenser and absorption Vessels; all provided with interchangeable conical ground glass joints.

B-3.2 Reagents

B-3.2.1 Sodium Hydroxide Solution — 3 percent (m/v).

B-3.2.2 Concentrated Hydrochloric Acid — see IS 265

B-3.2.3 Potassium Iodide Solution — 10 percent (m/v).

B-3.2.4 Standard Silver Nitrate Solution — approximately 0.05 N.

B-3.3 Procedure

Weigh accurately about 0.5 g of the material and transfer to a 500 ml distillation flask with 20 ml to 25 ml of water. Transfer 70 ml of sodium hydroxide solution in to the first absorption vessel and 30 ml into the second vessel. Assemble the apparatus and connect a steady stream of cooling water to the condenser. Add 80 ml of water containing 7 ml of hydrochloric acid through the tap funnel into the distillation flask containing the sample, carefully avoiding any loss of hydrogen cyanide. Quickly heat the contents of the flask to boiling and distill at such a rate that about 80 ml of distillate is collected in the absorbers in 10 min to 15 min. Disconnect the absorption vessels and transfer their solutions to a 500 ml volumetric flask rinsing the condenser and connections; dilute up to the mark with water and mix.

B-3.3.1 Transfer 250 ml of the solution prepared in **B-3.3** to a 600 ml beaker and dilute with water about 400 ml. Add 2 ml of potassium iodide solution and titrate with standard silver nitrate solution adding it rapidly at first with constant stirring and drop by drop as the precipitate formed first dissolves more slowly, until the first trace of permanent bluish opalescence can be seen in the liquid by transverse illumination against a black background.

B-3.4 Calculation

$$\text{Cyanide (as CN), percent by mass} = \frac{10.4 \times V \times N}{M}$$

where

V = volume of standard silver nitrate solution required, in ml;

N = normality of standard silver nitrate solution, and

M = mass of the material taken for the test, in g.

B-4 DETERMINATION OF PYROPHOSPHATE IN COPPER PYROPHOSPHATE

B-4.1 Apparatus

B-4.1.1 pH Meter

B-4.1.2 Stirrer

B-4.2 Reagents

B-4.2.1 Dilute Sulphuric Acid — approximately 1 N.

B-4.2.2 *Standard Sodium Hydroxide Solution* — approximately 0.3 N.

B-4.2.3 *Zinc Sulphate Solution* — 10 percent *m/v*.

B-4.2.4 *Tetrasodium Pyrophosphate* ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$)

B-4.3 Procedure

Take the electrolyte and washings from which copper has been removed electrolytically (*see B-2.4.1*) and make up to 250 ml in a volumetric flask. Transfer 100 ml of the well-mixed solution to a 400 ml beaker and dilute to 200 ml. Lower the stirrer and the electrodes of the *pH* meter into the beaker. Stir the solution and adjust the *pH* to 3.8 using dilute sulphuric acid. Add 30 ml of zinc sulphate and stir for 10 min. Run in standard sodium hydroxide solution from a burette, recording the volume (*V*) required to restore *pH* of the solution to 3.8.

B-4.3.1 Separately weigh 4.00 g of tetrasodium pyrophosphate, transfer to a 250 ml volumetric flask and dissolve in about 200 ml of water. Dilute the solution to the mark with water and pipette 100 ml of this solution into a 400 ml beaker. Dilute to about 200 ml with water and proceed as in **B-4.3**; finally record the volume V_1 of standard sodium hydroxide required to restore the *pH* of the solution to 3.8.

B-4.4 Calculation

$$\text{Pyrophosphate (as } \text{P}_2\text{O}_7\text{), percent by mass} = \frac{21.75 (V - V_1)N}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution required in **B-4.3**;

V_1 = volume in ml of the standard sodium hydroxide solution required in **B-4.3.1**;

N = normality of standard sodium hydroxide solution; and

M = mass of the materials taken for the test in **B-2.4.3**, in g;

NOTE — 1 ml of 1 N NaOH = 0.087 g of P_2O_7 .

B-5 DETERMINATION OF FLUOBORATE

B-5.1 Reagents

B-5.1.1 *Cetyltrimethyl ammonium chloride* — 25 percent in water.

B-5.2 Procedure

Make up the solution remaining after electrolysis to 500 ml in a volumetric flask. Transfer 100 ml of the same to a 250 ml glass beaker. Warm the solution and introduce approximately 4 ml of Cetyltrimethyl ammonium chloride solution. Let stand overnight with water covering the beaker with a watch glass. Filter through a weighed G No. 4 sintered glass crucible. Wash six times with small quantity of water each time. Weigh to constant weight after drying repeatedly at 105 °C.

B-5.3 Calculation

$$\text{Fluoborate (as } \text{BF}_4\text{) percent by mass} = \frac{M_1 \times 100 \times 5 \times 0.02337}{M}$$

where

M_1 = mass of precipitate, and

M = mass of the test sample initially taken for electrolysis

NOTE — The factor 0.02337 is the proportion of fluoborate in the precipitate

B-6 DETERMINATION OF INSOLUBLE MATTER

B-6.1 Reagents

B-6.1.1 *Potassium Cyanide Solution* — 20 percent (*m/v*), filtered.

B-6.1.2 *Potassium Pyrophosphate Solution* — 10 percent (*m/v*).

B-6.1.3 Sulphuric Acid — 10 percent (v/v).

B-6.2 Preparation of the Sample Solution

B-6.2.1 Copper Chloride, Copper Potassium Cyanide and Copper Sodium Cyanide

Dissolve about 10 g of the material in 100 ml of water and allow to stand for 1 h.

B-6.2.2 Copper Cyanide

Dissolve about 10 g of the material in 100 ml of potassium cyanide solution using a flat-ended glass rod to break up any lumps and allow to stand for 1 h.

B-6.2.3 Copper Pyrophosphate

Dissolve about 5 g of sample in 250 ml of potassium pyrophosphate solution at 50 °C, stirring until dissolved; and allow to stand for 1 h.

B-6.2.4 Copper Sulphate

Dissolve about 100 g of the sample in 250 ml of water and 1 ml of sulphuric acid. Warm to about 80 °C, stirring until solution is complete and allow to stand for 1 h.

B-6.3 Procedure

Filter the sample solution through tared sintered glass crucible (G No. 4). Wash the residue twice with appropriate reagent as above that is, KCN (*see B-6.2.1 and B-6.2.2*), potassium pyrophosphate (*see B-6.2.3*) and sulphuric acid (*see B-6.2.4*) and then thoroughly with water. Dry the crucible with residue at 105 °C. Cool and weigh.

CAUTION — Cyanide solution is very toxic. Take appropriate care in handling.

B-6.4 Calculation

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

where

M_1 = mass of the residue, in g; and

M = mass of the material taken for the test, in g.

B-7 DETERMINATION OF IRON

B-7.1 General

Four methods for the determination of iron are described. Either of these may be used for general routine purposes, but in case of a dispute Method C shall be the referee method.

B-7.2 Method A

B-7.2.1 Reagents

B-7.2.1.1 Concentrated hydrochloric acid — *see IS 265*

B-7.2.1.2 Ammonia — relative density 0.90.

B-7.2.1.3 Ammonium thiocyanate solution — 50 percent (m/v).

B-7.2.1.4 Standard iron solution

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 50 ml of 10 percent (v/v) sulphuric acid. Add 4 g of ammonium persulphate and boil for 5 min. Cool and dilute to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

B-7.2.2 Procedure

Take the electrolyte and washings from which copper has been removed electrolytically (*see B-2.4.1*) and boil for 15 min. Cool to room temperature and dilute with water to 250 ml in volumetric flask. Measure 25 ml of this solution into a Nessler cylinder, add 5 ml of ammonium thiocyanate solution and dilute to 50 ml with water. Carry out a control

test in a similar manner using the same amount of reagents and the following volumes of standard iron solution in place of the material:

Sl No.	Name of Material Under Test	Volume of Standard Iron Solution (in ml)
i)	Copper chloride	1.5
ii)	Copper cyanide	5.0
iii	Copper sodium cyanide	2.0
iv)	Copper sulphate	7.5

B-7.2.2.1 The material shall be taken as not having exceeded the limit prescribed for it in Table 1, if the intensity of colour produced with the material is not greater than that produced in the control test.

B-7.3 Method B

Determine iron content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 13320.

B-7.4 Method C

Determine iron content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-7.5 Method D

Determine iron content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

B-8 DETERMINATION OF ARSENIC

B-8.1 General

Four methods for the determination of arsenic are described. Either of these may be used for general routine purposes, but in case of a dispute Method C shall be the referee method.

B-8.2 Method A

B-8.2.1 General

Arsenic in the material is determined following the silver diethyldithiocarbamate method as prescribed in IS 2088.

NOTE — Because of noxious nature of pyridine, the evolution stage should be performed under a fume hood.

B-8.2.2 Procedure

Weigh accurately about 1 g of the material, transfer to a 100 ml conical flask provided with a conical socket. Add 10 ml of water and stir till the material is dissolved. Proceed further as prescribed in IS 2088, and determine the arsenic content of the material.

B-8.3 Method B

Determine arsenic content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 11124.

B-8.4 Method C

Determine arsenic content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-8.5 Method D

Determine arsenic content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

B-9 DETERMINATION OF CHLORIDE

B-9.1 Reagents

B-9.1.1 Concentrated Nitric Acid — see 1S 264

B-9.1.2 Dilute Nitric Acid — 1 percent (v/v).

B-9.1.3 Methyl Orange Indicator

Dissolve 0.1 g of methyl orange in 100 ml of water.

B-9.1.4 Hydrogen Cyanide Detector Papers — see **B-2.3.5**

B-9.1.5 Sodium Hydroxide Solution — 10 percent.

B-9.1.6 Silver Nitrate Solution — 2 percent (m/v).

B-9.2 Preparation of the Sample Solution

B-9.2.1 Copper Cyanide and Copper Sodium Cyanide

Weigh accurately about 10 g of the material and transfer to 600 ml beaker with 100 ml of water. Transfer to a well-ventilated fume chamber. Cover the beaker with watch-glass. Slowly add 20 ml of concentrated nitric acid and allow the reaction to subside. Prepare a blank of 100 ml of water and 20 ml concentrated nitric acid in a second beaker. Boil the contents of both the beakers until hydrogen cyanide cannot be detected in the vapour over the sample beaker (detector paper not turned blue). Filter both solutions through filter paper and wash well with hot water. Cool, dilute to 150 ml with water, add 10 drops of methyl orange and almost neutralize with sodium hydroxide solution. (In the sample solution the purple colour is just discharged and the solution is blue. The normal methyl orange colour change is observed in the blank.) Make both the solutions just acidic by dropwise addition of concentrated nitric acid and add 5 ml in excess. Dilute to 200 ml with water, cool to room temperature and proceed further as prescribed in **B-9.3**.

B-9.2.2 Copper Sulphate

Weigh accurately about 25 g of the material and dissolve in 100 ml of water containing 5 ml of concentrated nitric acid in a 400 ml beaker. Prepare a blank of 100 ml of water and 5 ml of concentrated nitric acid in a second beaker. Filter through filter paper and wash well with hot water. Dilute the filtrate to 200 ml with water, cool to room temperature and proceed further as prescribed in **B-9.3**.

B-9.3 Procedure

To the cold sample solution and blank, slowly add silver nitrate solution with constant stirring until precipitation is virtually complete and add 2 ml of silver nitrate solution in excess (From this stage carry out the operations in subdued light.). Cover the beakers with watch-glasses and warm until the precipitates coagulate. Add a few more drops of silver nitrate solution to confirm that precipitation is complete and allow to stand in dark for 1 h.

B-9.3.1 Filter through a tared sintered glass crucible (G No. 4) and wash the precipitates thoroughly with dilute nitric acid. Dry to constant mass at $(105 \pm 2)^\circ\text{C}$. Cool in a desiccator and weigh.

B-9.4 Calculation

$$\text{Chloride (as Cl), percent by mass} = \frac{(M_1 - M_2) \times 21.75}{M}$$

where

M_1 = mass of the precipitate obtained with material, in g;

M_2 = mass of the precipitate obtained with the blank, in g; and

M = mass of the material taken for the test, in g.

B-10 DETERMINATION OF SULPHATE

B-10.1 Reagents

B-10.1.1 Concentrated Hydrochloric Acid — see 1S 265

B-10.1.2 Barium Chloride Solution — 10 percent (m/v).

B-10.2 Procedure

Weigh accurately about 1 g of the material and transfer to a 500 ml beaker with 10 ml of water. Add 30 ml of concentrated hydrochloric acid and boil for 20 min in a fume chamber. Dilute the solution with water to about 400 ml, and heat to boiling. Add barium chloride solution drop wise until precipitation is virtually complete. Add 2 ml of barium chloride solution in excess. Allow to stand on a hotplate for 1 h and then overnight at room temperature. Filter through a tared sintered glass crucible (G No. 4) and wash well with water until free from chloride. Dry to constant mass at (105 ± 2) °C, cool and weigh.

B-10.3 Calculation

$$\text{Sulphate (as SO}_4\text{), percent by mass} = \frac{M_1 \times 41.15}{M}$$

where

M_1 = mass of the precipitate, in g; and

M = mass of the material taken for the test, in g.

ANNEX C

(Clause 4)

SAFETY PRECAUTIONS FOR HANDLING SALTS OF COPPER CYANIDE, COPPER POTASSIUM CYANIDE AND COPPER SODIUM CYANIDE

C-1 SAFETY PRECAUTIONS

C-1.1 As cyanides of copper are highly poisonous, they shall never be touched with unprotected hands; gloves shall always be worn during sampling operations and during crushing, goggles fitted with a face cloth shall also be worn.

C-1.2 Solutions containing cyanide shall never be pipetted by mouth suction. A safety pipette shall always be used to draw the liquid into the pipette. A burette may also be used for drawing measured quantity of the solution.

C-1.3 On account of highly poisonous character of hydrogen cyanide all operation involving the decomposition of copper cyanides shall be conducted in a well-ventilated fume chamber.

C-2 HYDROCYANIC ACID AND CYANIDE POISONING

C-2.1 Symptoms

The symptoms are giddiness, staggering and insensibility accompanied by panting respiration, followed by profound collapse with convulsions. The action is extremely rapid.

C-2.2 First Aid

C-2.2.1 Remove patient from cause of trouble, for example, fumes, etc, and take him to fresh air. Make the patient lie down, keep him warm and do not allow him to move more than necessary.

C-2.2.2 If breathing has ceased, apply artificial respiration.

NOTE — Mouth to mouth respiration should not be attempted.

C-2.2.3 Administer amyl nitrite. This is purchased in the form of small ampoules and one of these is broken and held under the nose so that the patient will inhale the vapour. It Should be administered for 15 s to 30 s every 2 min to 3 min.

C-2.2.4 If available, administer oxygen through a face mask and call for a qualified medical practitioner.

C-2.3 Antidote

C-2.3.1 The following antidote has been found used when cyanide is swallowed:

- Solution A** — Dissolve 158 g of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and 3 g of citric acid in 1 000 ml of water. The solution should be regularly inspected and replaced whenever any deterioration occurs.
- Solution B** — Dissolve 60 g of anhydrous sodium carbonate in 1 000 ml of water.

C-2.3.1.1 Take 50 ml from each of solutions A and B and keep in separate 175 ml wide-necked bottles with a polyethylene closure. Mark the bottles as 'Cyanide antidote A' and 'Cyanide antidote B'. Both the bottles should bear the legend. Mix the whole content of bottles A and B and swallow the mixture.

C-2.4 Medical Treatment

C-2.4.1 The treatment consists of the injection into the blood stream of sodium nitrite and sodium thiosulphate and should be carried out only by a qualified medical practitioner. Details are as follows:

- a) Intravenous injection of 0.3 g of sodium nitrite dissolved in 10 ml of sterile distilled water. This should be given slowly at the rate of 2.5 ml to 5 ml/min.
- b) Immediately following this and through the same needle an intravenous injection of 25 g of sodium thiosulphate dissolved in 50 ml of sterile distilled water is given at the same rate. Leakage of material outside the vein should be avoided.

C-2.4.2 A temporary improvement is not a criterion of recovery. If the symptoms persist or recur after 1 h, a second injection of the two substances should be given. It is suggested that electroplating shops using cyanide should keep a supply of these two substances in ampoule form and two sterilized syringes, one with a total capacity of 10 ml and the second with a total capacity of 50 ml, together with particulars of the treatment (*see C-2.4.1*).

C-2.4.3 Since it is unlikely that the average general practitioner or hospital would have such material ready for use, should the patient be sent to hospital these materials should accompany him in the ambulance, and during the journey the first-aid procedures already described should be continued.

C-2.4.4 Some patients may respond to the first-aid treatment alone, but in many cases it will be advisable if not necessary, to give the intravenous injection. This should be done as soon as possible, and in any case is desirable that it should be administered within 15 min. It is, therefore, essential that if medical help cannot immediately be obtained, the patient should be conveyed without delay to the nearest hospital.

ANNEX D

(Clause 6)

SAMPLING OF COPPER SALTS FOR ELECTROPLATING

D-1 GENERAL REQUIREMENTS OF SAMPLING

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

D-1.2 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

D-1.3 The sampling instruments shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

D-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

D-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

D-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.8 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, and the date of sampling.

D-2 SCALE OF SAMPLING

D-2.1 Lot

All the containers in a single consignment of the same copper salt and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

D-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested from each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

D-2.3 The containers shall be selected from the lot at random. In order to ensure the randomness of-selection, a random number table shall be used. For guidance and use of random number tables IS 4905 may be referred to. In the absence of a random number tables the following procedure may be adopted:

Starting from any container in the lot, count them as 1,2,3 ... etc., up to r and so on where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every r th container thus counted shall be withdrawn till the requisite number of containers is obtained.

D-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

D-3.1 From each of the containers selected according to **D-2.3** a representative portion of the material for carrying out the tests specified under **3.2** shall be drawn and these Shall constitute the individual samples

D-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

Table 2 Number of Containers to be Selected from Lots of Different Sizes

(Clause D-2.2)

Lot Size N (1)	Sample Size n (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 to 500	8
501 to 1 000	9
1 001 and above	10

D-3.3 The material in the individual and composite samples shall be transferred to separate bottles and labelled with full identification particulars of the samples.

D-4 NUMBER OF TESTS

D-4.1 Tests for the determination of copper content, cyanide content, and pyrophosphate content shall be carried out on each of the individual samples.

D-4.2 Tests for the remaining characteristics shall be done on the composite sample.

D-5 CRITERIA FOR CONFORMITY

D-5.1 For Individual Samples

For copper content, cyanide and pyrophosphate content, the mean and range of test results shall be computed as follow:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of individual test result}}{\text{Number of tests}}$$

Range (R) = Difference between the maximum and minimum value of the test results.

D-5.1.1 For declaring conformity of the lot to each of the characteristics, namely, copper content, cyanide content, and pyrophosphate content, the value of the expression $(\bar{X} - 0.6) R$ shall be greater than or equal to the corresponding minimum value specified in Table 1.

D-5.2 For composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics, the test results on the composite sample shall meet the corresponding requirements specified in Table 1.

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