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BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

इलेक्ट्रोप्लेटिंग के लिए सिल्वर साइनाइड और सिल्वर पोटेशियम साइनाइड — विशिष्टि

(*IS 6267 का पहला पुनरीक्षण*)

Draft Indian Standard

Silver Cyanide and Silver Potassium Cyanide for Electroplating — Specification

(*First Revision of IS 6267*)

(ICS 25.220.40)

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 5	Last Date for Comments: **10 July 2024**

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

FOREWORD

(*formal clauses will be added later*)

Silver is normally electrodeposited from a solution containing essentially a double cyanide of silver and an alkali metal, additional alkali cyanide (free cyanide) and other alkaline compounds. The solution is usually produced from silver cyanide and silver potassium cyanide. The anodes used are usually stainless steel or silver. Requirements of silver anodes are covered in IS 1959.

This standard was originally published in 1971. In this revision, alternative instrumental test methods AAS, ICP-MS and ICP-OES have been incorporated for determination of copper and the standard has been updated based on the experience of last five decades and on the currently available data. Amendment no. 1 has also been incorporated in this revision. The relevant clauses and references have been updated.

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

Draft Indian Standard

SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for silver cyanide and silver potassium cyanide for electroplating.

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 Indian Standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

2 REQUIREMENTS

2.1 Description

2.1.1 *Silver Cyanide*

The material shall be in the form of creamy white crystals, free from dirt, foreign matter and visible impurities and shall correspond essentially to formula AgCN.

2.1.2 *Silver Potassium Cyanide*

The material shall be in the form of white powder free from dirt, foreign matter and visible impurities and shall correspond essentially to the formula $KAg(CN)_2$.

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

2.2.1 For use in special electroplating applications like electrical and electronic engineering industries, the requirements of silver cyanide and silver potassium cyanide, permissible limits of copper (as Cu) and chloride (as Cl) and any other impurities, may be as agreed to between the purchaser and the manufacturer.

2.2.1.1 For guidance, copper (as Cu) may not exceed 0.005 percent by mass and chloride (as Cl) 0.02 percent by mass.

2.2.1.2 The methods of determination of these requirements shall be as agreed to between the purchaser and the manufacturer.

Table 1 Requirements for Silver Cyanide and Silver Potassium Cyanide for Electroplating

Sl No.	Characteristic	Requirements		Method of Test (Ref to Clause No. In Annex B)
		Silver Cyanide	Silver potassium Cyanide	
(1)	(2)	(3)	(4)	(5)
i)	Silver (as Ag), percent by mass (dry basis), *Min*	80.0	54.0	**B-2**
ii)	Matter insoluble in aqueous solution of potassium cyanide, percent by mass, *Max*	0.1	—	**B-3**
iii)	Matter insoluble in distilled water, percent by mass, *Max*	—	0.1	**B-4**

iv)	Copper (as Cu), percent by mass, *Max*	0.05	0.05	**B-5**

3 SAFETY PRECAUTIONS IN HANDLING SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

Since silver cyanide and silver potassium cyanide are highly poisonous, extreme caution is necessary in handling the same. Useful information on this subject is given in Annex C.

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed to between the purchaser and the vendor, the salts shall be supplied in airtight containers which shall not have any reaction with the contents.

4.2 Marking

The containers shall be marked with the following:

a) Name of material and its net weight;
b) Name of manufacturer and recognized trade-mark, if any;
c) Lot number and date of manufacture; and
d) The word 'POISON' and the appropriate symbol (*see* IS 1260).

4.2.1 *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.

5 SAMPLING

The method of preparing representative samples of the material and the criteria for conformity to this standard shall be as prescribed in Annex D.

ANNEX A

(*Clause* 2)

(**LIST OF REFERRED STANDARDS**)

IS No	*Title*
IS 264 : 2005	Nitric acid — Specification (*third 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 1260	
(Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
(Part 2) : 2020	Packaging — distribution packaging — graphical symbols for handling and storage of packages Part 2 General goods (*fourth revision*)
IS 4905 : 2015 ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
IS 11123 : 1984	Method for determination of copper by atomic absorption spectrophotometry

ANNEX B

(*Clause* 2.2, and *Table* 1)

ANALYSIS OF SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

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 SILVER IN SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

B-2.1 Reagents

B-2.1.1 *Concentrated Nitric Acid — see* IS 264

B-2.1.2 *Ferric Ammonium Sulphate Solution* — 20 percent (*w/v*).

B-2.1.3 *Potassium Thiocyanate Solution*

0.1 N, prepared by dissolving 9.717 0 g of potassium thiocyanate in 1 000 ml of water.

B-2.1.4 *Hydrogen Cyanide Detection Papers*

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

Solution A

0.1 percent (*w/v*) *o*-tolidine acetate. Dissolve 0.64 g of *o*-toluidine in 5 ml of 50 percent (*w/v*) glacial acetic acid solution and dilute to 1 000 ml with water.

Solution B

0.3 percent (*w/v*) copper acetate solution in water.

B-2.2 Procedure

Weigh accurately about 1 g of the sample and transfer to a 250 ml conical flask. Add 20 ml of concentrated nitric acid and boil in a fume cupboard until a clear solution is obtained, and test for complete destruction of cyanide with hydrogen cyanide detection papers. Dilute to approximately 100 ml with water, add 2 ml of ferric ammonium sulphate solution and titrate with standardized potassium thiocyanate solution till the colour changes to red-brown tint.

B-2.3 Calculation

$$\text{Silver (as Ag), percent by mass } = \frac{V \times N \times 10.79}{W}$$

where

V = volume in ml of standard potassium thiocyanate solution consumed,

N = normality of potassium thiocyanate solution, and

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

B-3 DETERMINATION OF MATTER INSOLUBLE IN AQUEOUS SOLUTION OF POTASSIUM CYANIDE

B-3.1 Reagent

B-3.1.1 *Potassium Cyanide Solution* — 10 percent (*w/v*).

B-3.2 Procedure

Weigh accurately about 10 g of silver cyanide and dissolve in 100 ml of warm potassium cyanide solution. Filter the solution through a tared sintered glass crucible (G No. 3). Wash thoroughly with hot water and dry the crucible at 110 °C. Weigh again and express the mass of the residue as percentage of the mass of the material taken for the test.

B-4 DETERMINATION OF MATTER INSOLUBLE IN DISTILLED WATER

B-4.1 Procedure

Weigh accurately about 10 g of silver potassium cyanide and dissolve it in 100 ml of water. Filter the solution through a tared sintered glass crucible (G No. 3). Wash thoroughly with hot water and dry at 110 °C. Weigh again and express the mass of the residue as percentage of the mass of the material taken for the test.

B-5 DETERMINATION OF COPPER

B-5.1 General

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

B-5.2 Method A

B-5.2.1 *Procedure*

Weigh 2.0 g material accurately. Add 10 ml nitric acid and 10 ml sulphuric acid. Titrate the mixture to sulphuric acid fumes for decomposition of cyanides in a hood. Cool the above acidic solution and add 20 ml water. Wash the dropper and dish. Collect all washings. Precipitate all silver by adding about 5 ml to 10 ml saturated solution of sodium chloride. Filter silver chloride through Whitman's filter paper No. 40 and wash with water. Collect all the filtrates and make it just alkaline with 25 percent caustic soda and then make it acidic with acetic acid. Add 1 g of potassium iodide and shake for 2 min to 3 min. Keep the flask aside for 5 min in the dark.

Titrate the liberated iodine with 0.01 N sodium thiosulphate solution using starch as indicator when the blue colour disappears.

B-5.2.2 *Calculation*

$$\text{Copper as (Cu), percent by mass} = 6.35 \times \frac{VN}{M}$$

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-5.3 Method B

Determine copper content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 11123.

B-5.4 Method C

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

B-5.5 Method D

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

ANNEX C

(*Clause* 3)

SAFETY PRECAUTIONS FOR HANDLING SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

C-1 SAFETY PRECAUTIONS

C-1.1 As silver cyanide and silver potassium cyanide are highly poisonous, they should never be touched with unprotected hands; gloves should always be worn during sampling operations, and during crushing, goggles fitted with a face cloth should also be worn.

C-1.2 Solutions containing cyanide should never be pipetted by mouth suction. An evacuating bulb should 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 the highly poisonous character of hydrogen cyanide all operations involving the decomposition of cyanides should be conducted in a well-ventilated fume cupboard.

C-2 HYDROCYANIC ACID AND CYANIDE POISONING

C-2.1 Symptoms

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

C-2.2 First Aid

C-2.2.1 Remove patient from the 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.

NOTE — Amyl nitrite is sensitive to light and warmth and should, therefore, be kept in the dark at a temperature less than 15°C. All ampoules should be discarded every 2 years.

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 useful when cyanide is swallowed:

a) *Solution A*
Dissolve 158 g of ferrous sulphate ($FeSO_4.7H_2O$) and 3 g of citric acid in 1 000 ml of water. The solution should be regularly inspected and replaced wherever any deterioration occurs.

b) *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 contents of bottles A and B and administer 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 per minute.

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 an hour 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 other with a total capacity of 50 ml, together with particulars of the treatment (as above).

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 it 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* 5)

SAMPLING OF SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.1 In drawing, preparing, storing and handling samples, the safety precautions prescribed in Annex C shall be strictly followed.

D-1.2 The sampling implements and the sample containers shall be clean and dry.

D-1.3 Each sample container shall be sealed airtight after filling and marked with full details of sampling.

D-2 SCALE OF SAMPLING

D-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

D-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for 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 In order to ensure randomness of selection random number tables shall be used. For random selection procedures, guidance may be taken from IS 4905

Table 2 Scale of Sampling

(*Clause* D-2.2)

Lot Size (the Number of Containers)	Sample Size
Up to 25	3
26 to 50	4
51 to 150	5
151 to 300	6
301 and above	8

D-3 INDIVIDUAL AND COMPOSITE SAMPLE

D-3.1 From each of the containers selected according to **D-2.2** a representative portion of the material sufficient for the tests shall be withdrawn. These samples representing each of the selected containers are termed as individual samples.

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

D-3.3 The material constituting each of the individual sample as well as the composite sample shall be stored separately with full identification particulars.

D-4 NUMBER OF TESTS

D-4.1 Test for silver content shall be conducted on individual samples.

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

D-5 CRITERIA FOR CONFORMITY

D-5.1 For Individual Samples — For silver content the test results shall be noted and their mean (X) and the range (R), being the difference between the maximum and minimum of test results, shall be computed. For declaring the conformity of the lot in respect of silver content, (X — 0.6R) shall be greater than or equal to the minimum specified in Table 1.

D-5.2 For declaring the conformity of the lot to the requirements of all other characteristics, the test results of the composite sample shall satisfy the relevant requirements.