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भारतीय मानक मसौदा

# इलेक्ट्रोप्लेटिंग के लिए पोटैशियम और सोडियम साइनाइड — विशिष्टि

(IS 6358 का दूसरा पुनरीक्षण)

Draft Indian Standard

## Potassium and Sodium Cyanides for Electroplating — Specification

(Second Revision of IS 6358)

(ICS 25.220.40)

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

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

#### FOREWORD

(formal clauses will be added later)

Potassium and sodium cyanides find considerable use in the electroplating industry. In view of their highly poisonous nature, it is necessary that due precautions be observed in handling them. Due care should also be taken in storing and drawing out the materials as they are very hygroscopic and deteriorate on exposure to the atmosphere.

This standard was originally published in 1971. In this revision, amendment no. 1 has been incorporated and the standard has been updated based on the experience of last five 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 POTASSIUM AND SODIUM CYANIDES FOR ELECTROPLATING — SPECIFICATION

(Second Revision)

#### **1 SCOPE**

This standard prescribes requirements and methods of sampling and test for potassium and sodium cyanides for electroplating.

#### **2 REFERENCES**

The standards listed below 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 revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of these standards indicated below:

IS No	Title
IS 264 : 2005	Nitric acid — Specification (third revision)
IS 265 : 2021	Hydrochloric acid — Specification (fifth revision)
IS 321 : 1964	Specification for absolute alcohol (first revision)
IS 1070 : 2023	Reagent grade water — Specification (fourth revision)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 4161 : 2023	Nessler cylinder — Specification (first revision)

#### **3 REQUIREMENTS**

#### 3.1 Description

#### 3.1.1 Potassium Cyanide

The material (frequently known as single salt 97/99 percent) shall be in the form of pellets or balls free from foreign matter and visible impurities and shall correspond essentially to the formula KCN.

#### 3.1.2 Sodium Cyanide

The material (frequently known as single salt, KCN value 128/130 percent) shall be in the form of pellets free from foreign matter and visible impurities and shall correspond essentially to the formula NaCN.

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

#### Table 1 Requirements of Potassium and Sodium Cyanides for Electroplating

S1	Characteristic	Requirement		Methods of Test (Ref to Clause No. in Annex A)
No.				
		Potassium Cyanide	Sodium Cyanide	
(1)	(2)	(3)	(4)	(5)
i)	Cyanide ( as KCN ), percent by mass, <i>Min</i>	97.0	127.5	A-2
ii)	Sodium ( as Na ), percent by mass, <i>Max</i>	1.0	_	A-3
iii)	Sulphides ( as S ), percent by mass, <i>Max</i>	0.001	0.005	A-4
iv)	Chlorides ( as Cl ), percent by mass, <i>Max</i>	0.05	0.05	A-5
v)	Water insoluble matter, percent by mass, <i>Max</i>	0.02	0.01	A-6

(Clause 3.2, A-4.4.1, A-5.5, C-5.1, C-5.2)

\*When expressed as NaCN, not to be less than 90.0 percent by mass.

#### 4 SAFETY PRECAUTIONS IN HANDLING POTASSIUM AND SODIUM CYANIDES

Potassium and sodium cyanides are extremely poisonous and should be handled with extreme care. Useful information on this subject is given in Annex B.

#### **5 PACKING AND MARKING**

#### 5.1 Packing

The material shall be packed in air-tight resealable containers with an opening large enough to allow the contents to be easily removed.

#### 5.2 Marking

The containers shall be marked legibly and indelibly with the following:

- a) The name of the 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).

#### **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**

**6.1** The method of preparing representative samples of the material and the criteria for its conformity to this specification shall be as prescribed in Annex C.

**6.2** Potassium and sodium cyanides are very hygroscopic and lose strength rapidly on exposure to the atmosphere due to hydrolysis. To minimize errors due to this, packages to be sampled should only be opened for the minimum time

necessary to extract a sample and then resealed. All sampling equipment should be dry and preferably warmed to approximately 50  $^{\circ}$ C.

**6.2.1** To sample powdered and granulated products, remove the lid from the container and tilt it. Wearing gloves, force a stainless steel sampling spear almost to the bottom of the container, withdraw it and transfer the contents to a dry 250 g glass or plastics screw-capped bottle. Repeat as necessary until the bottle is full, replace the cap and seal with adhesive tape.

**6.2.2** To sample moulded pieces, remove the container lid, and wearing gloves, rapidly select sufficient pieces at random to provide a 250 g sample. Scoop away the top layers to give access to the product well inside the container. Place the selected pieces in a large dry screw-capped bottle and reseal the container. Wearing a face shield and gloves, rapidly crush the sample to pieces 3 mm or less in size with a warm dry steel mortar and pestle and fill a dry 250 g glass or plastics screw-capped bottle. Seal the cap with adhesive tape.

#### ANNEX A

#### (*Clause* 3.2)

#### ANALYSIS OF POTASSIUM AND SODIUM CYANIDES FOR ELECTROPLATING

#### A-1 QUALITY OF REAGENTS

A-1.1 unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be used.

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

#### **A-2 DETERMINATION OF CYANIDE**

#### A-2.1 Reagents

A 2.1.1 Potassium Iodide Solution — 10 percent (*m/v*).

A-2.1.2 Standardized Silver Nitrate Solution — 0.1 N approximately.

A-2.1.3 Sodium Hydroxide Solution — 5 percent (*m*/*v*).

A-2.1.4 Dilute Ammonium Hydroxide — 1:9

#### A-2.2 Procedure

Weigh accurately about 5 g of the sample in a tared stoppered weighing bottle. Dissolve in water containing 10 ml of sodium hydroxide solution per litre, transfer to a 500 ml one-mark graduated flask, dilute to volume with the same

water containing sodium hydroxide and mix.

**A-2.2.1** Using a suction bulb (not suction by mouth), pipette 25 ml of the solution into a 500 ml beaker containing 300 ml of water and 3 ml of sodium hydroxide solution. Add 2 ml of potassium iodide solution and 5 ml of dilute ammonium hydroxide and titrate with silver nitrate solution with constant stirring against a black-background and transverse illumination to the first faint turbidity.

#### A-2.3 Calculation

Cyanide (as KCN), percent by mass = 
$$\frac{260.4 \times V \times N}{M}$$
  
Cyanide (as NaCN), percent by mass =  $\frac{196.4 \times V \times N}{M}$ 

where

V = volume in ml, of standardized silver nitrate solution required in titration,

N = Normality of standard silver nitrate solution, and

M = Mass in g, of the sample taken for the test.

#### A-3 DETERMINATION OF SODIUM IN POTASSIUM CYANIDE

#### A-3.1 General

Two methods are prescribed for the determination of sodium. One uses a flame photometer or flame spectrophotometer and the other is gravimetric. In the case of a dispute, the gravimetric method shall be used as the referee method.

#### A-3.2 Photometric Method

A-3.2.1 Apparatus — a flame photometer or a spectrophotometer.

A-3.2.2 Reagents

A-3.2.2.1 *Hydrochloric acid solution* — 4 percent (*m/v*).

#### A-3.2.2.2 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 *o*-tolidine acetate

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

Solution B = 0.3 percent (m/v) copper acetate solution in water.

#### A-3.2.2.3 Standard sodium solution

Dissolve 2.543 g of dry sodium chloride in water. Add 50 ml of hydrochloric acid solution and dilute to 1 000 ml in a one-mark graduated flask. One millilitre of this solution is equivalent to 1 mg of sodium (as Na).

#### A-3.2.3 Procedure

Dissolve 1 g of sample in 25 ml of water in a 250 ml beaker. Transfer to an efficient fume chamber and add 40 ml of hydrochloric acid-solution. Boil until hydrogen cyanide can no longer be detected with detector paper (no blue coloration).

**A-3.2.3.1** Cool, add 25 ml of hydrochloric acid solution and dilute to 1 000 ml in a one-mark graduated flask. Obtain reading on the flame photometer or spectrophotometer and read off the sodium content of the sample from the calibration graph.

#### A-3.2.4 Calibration

Prepare a series of standard sodium solutions containing 0 ml, 5 ml, 10 ml and 15 ml of standard sodium solution and 50 ml of hydrochloric acid in one litre of solution. (These correspond to 0, 0.5, 1.0 and 1.5 percent sodium respectively for a 1 g sample.) Obtain reading for each of these solutions using a flame photometer with a sodium filter or a flame spectrophotometer at 589 nm adjusting the instrument sensitivity to give approximately full scale deflection on the highest standard. Plot a graph of percent sodium against instrument reading. This should be linear and should pass through the origin.

#### A-3.3 Gravimetric Method

A-3.3.1 Reagents

A-3.3.1.1 Absolute alcohol — see IS 321

A-3.3.1.2 Ammonium carbonate

**A-3.3.1.3** *Barium chloride solution* — 10 percent (m/v)

A-3.3.1.4 Concentrated hydrochloric acid — see IS 265

A-3.3.1.5 Concentrated nitric acid — see IS 264

A-3.3.1.6 Perchloric acid

A-3.3.1.7 *Sulphuric acid* — 0.5 N approximately.

A-3.3.2 Procedure

Weigh accurately about 1 g of the sample and transfer to a 100 ml beaker with 10 ml of water. Add 5 ml of concentrated hydrochloric acid and place in an efficient fume chamber. Add 5 ml of concentrated nitric acid, cover the beaker with a watch glass and boil until hydrogen cyanide can no longer be detected (no blue coloration), with detection paper (*see* **A-3.2.2.2**). Rinse the watch glass and the sides of the beaker, add 1 ml of hot barium chloride solution dropwise. Filter and wash the precipitate, if any, with hot water. Reject the precipitate, if any, and evaporate the filtrate and washings in a silica dish to a volume of about 10 ml. Cool, add 12 ml of perchloric acid and continue evaporation till dense fumes of perchloric acid are given off. Cool, add 10 ml of water and 2 ml of perchloric acid and repeat evaporation as above.

NOTE — Organic matter, if any, must be removed before the addition of perchloric acid to avoid explosion.

**A-3.3.2.1** Cool, add 10 ml of absolute alcohol and crush the crystals of potassium perchlorate thus formed with a flatheaded glass rod. Filter through a sintered glass crucible (G No. 4) and reserve the filtrate. Dissolve potassium perchlorate in a minimum volume of hot water, and 2 ml of perchloric acid and fume again. Cool, add 10 ml of absolute alcohol and separate the crystals of potassium perchlorate by filtering again through the sintered glass crucible and washing with absolute alcohol containing 0.2 percent (m/v) of Perchloric acid.

NOTE — The presence of ammonium salts and sulphates should be avoided as ammonium perchlorate and sodium sulphate are sparingly soluble in absolute alcohol.

**A-3.3.2.2** Combine the alcoholic filtrate and washings, dilute with an equal volume of water and concentrate to about 5 ml. Add 20 ml of hot water and 5 ml of sulphuric acid to the concentrate and boil. Filter, wash and reject the precipitate of barium sulphate, if any, collecting the filtrate in a tared platinum dish.

**A-3.3.2.3** Add 5 ml of sulphuric acid and evaporate to dryness. Heat the dish first at low heat and finally at 500 °C. Add 2 g of solid ammonium carbonate and heat to a constant weight.

A-3.3.3 Calculation

Sodium (as Na), percent by mass =  $\frac{32.38 \times M_1}{M}$ 

where

 $M_1$  = mass in g, of residue obtained; and

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

#### **A-4 DETERMINATION OF SULPHIDE**

#### A-4.1 General

The colour produced by the addition of alkaline lead acetate solution to the solution of the material is compared with that produced in the standard sulphide solution.

#### A-4.2 Apparatus

A-4.2.1 Nessler Cylinders — 50 ml capacity (see IS 4161)

#### A-4.3 Reagents

A-4.3.1 Alkaline Lead Acetate Solution

Dissolve 1.7 g of lead acetate and 35 g of sodium hydroxide in water and dilute to 100 ml.

#### A-4.3.2 Sodium Sulphide Solution A

Dissolve 0.75 g of sodium sulphide (Na<sub>2</sub>S.9H<sub>2</sub>O) in freshly boiled and cooled water and dilute to 1 000 ml. Pipette 100 ml of this solution into a 250 ml conical flask containing 10 ml of 0.1 N iodine solution and 25 ml of 0.1 N hydrochloric acid. Back titrate with 0.1 N sodium thiosulphate solution adding 1 percent starch solution as indicator before the end point. Let the volume of 0.1 N iodine solution absorbed be *a* ml.

#### A-4.3.2.1 Sodium sulphide solution B

Fill a burette with sodium sulphide solution A and measure 100/1.6 a ml into a 1 000 ml one-mark graduated flask and dilute to volume with freshly boiled and cooled water. Prepare immediately before use. One millilitre of this solution is equivalent to 1  $\mu$ g of sulphide (as S).

#### A-4.4 Procedure

Weigh accurately and as rapidly as possible 2 g of potassium cyanide or 0.40 g of sodium cyanide sample, as the case may be, dissolve in 100 ml of freshly boiled and cooled water and fill two 50 ml Nessler cylinders with the solution. Add 3 drops of alkaline lead acetate solution to each of the cylinders and mix. Carry out a control test in a similar Nessler cylinder using 10 ml of sodium sulphide solution B in place of the material along with the same quantity of alkaline lead acetate solution as with the material and diluting to the mark with freshly boiled and cooled water.

**A-4.4.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

#### **A-5 DETERMINATION OF CHLORIDE**

#### A-5.1 General

The turbidity produced by the addition of silver nitrate solution to the solution of the material is compared with that produced in standard chloride solution.

#### A-5.2 Apparatus

A-5.2.1 *Nessler Cylinders* — 100 ml capacity (*see* IS 4161)

#### A-5.3 Reagents

A-5.3.1 Hydrogen Cyanide Detection Papers — same as prescribed in A-3.2.2.2.

**A-5.3.2** *Dilute Nitric Acid* -10 percent (v/v).

A-5.3.3 Silver Nitrate Solution — 0.05 N.

#### A-5.3.4 Standard Chloride Solution

Weigh accurately 0.165 0 g of sodium chloride (NaCl) and dissolve in water in a 1 000 ml one-mark graduated flask and make up the volume. One millilitre of this solution is equivalent to 0.1 mg of chloride (as Cl).

#### A-5.4 Method

#### A-5.4.1 Procedure

Weigh accurately 0.50 g of the sample, dissolve in 75 ml of water in a 150 ml beaker and place in an efficient fume chamber. Add 15 ml of dilute nitric acid cover with a watch glass and boil until hydrogen cyanide can no longer be detected (no blue coloration) with detection paper (*see* **A-5.3.1**). Cool, rinse the watch glass, transfer the solution to the Nessler cylinder and dilute with water to the mark. Finally add 1 ml of silver nitrate solution and mix. Carry out a control test in another similar Nessler cylinder using 2.5 ml of the standard chloride solution and the same quantities of other reagents in the same total volume as with the material. Compare the turbidity produced in both the cylinders against a black background.

**A-5.5** The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the sample is not greater than that produced in the control test.

#### A-6 DETERMINATION OF WATER INSOLUBLE MATTER

**A-6.1 Procedure** — Weigh accurately about 100 g of the sample and dissolve in 500 ml of water. Filter the solution through a tared sintered glass crucible (G No. 4) and wash the residue thoroughly with water. Dry at 105 °C for 30 min and weigh to calculate the percentage of residue by mass of the material taken for the test.

#### ANNEX B

#### (Clause 4)

#### SAFETY PRECAUTIONS FOR HANDLING POTASSIUM AND SODIUM CYANIDES

#### **B-1 SAFETY PRECAUTIONS**

**B-1.1** As potassium and sodium cyanides 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.

**B-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.

**B-1.3** On account of highly poisonous character of hydrogen cyanide all operations involving the decomposition of potassium or sodium cyanides should be conducted in a well-ventilated fume cupboard

#### **B-2 HYDROCYANIC ACID AND CYANIDE POISONING**

#### **B-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.

#### **B-2.2 First Aid**

**B-2.2.1** Remove the 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.

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

 $\operatorname{NOTE}-\operatorname{Mouth}$  to mouth respiration should not be attempted.

**B-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 dark at a temperature less than 15  $^{\circ}$ C. All ampoules should be discarded every 2 years.

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

#### **B-2.3** Antidote

B-2.3.1 The following antidote has been found useful when cyanide is swallowed:

Solution A

Dissolve 158 g of ferrous sulphate (FeSO<sub>4</sub>.7H<sub>2</sub>O) and 3 g of citric acid in 1 000 ml of water. The solution should be regularly inspected and replaced wherever any deterioration occurs.

Solution B

Dissolve 60 g of anhydrous sodium carbonate in 1 000 ml of water.

**B-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.

#### **B-2.4 Medical Treatment**

**B-2.4.1** The treatment consists of the injection into the bloodstream 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 should be given at the same rate. Leakage of materials outside the vein should be avoided.

**B-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 second with a total capacity of 50 ml, together with particulars of the treatment (as above).

**B-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.

**B-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 C

#### (*Clause* 6.1)

#### SAMPLING OF POTASSIUM AND SODIUM CYANIDES FOR ELECTROPLATING

#### C-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

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

#### C-2 SCALE OF SAMPLING

#### C-2.1 Lot

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

**C-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.

Table 2 Scale of Sampling				
Lot Size	Sample Size			
(Number of Containers)				
N	n			
Up to 25	3			
26 to 50	4			
51 to 150	5			
151 to 300	6			
301 and above	8			

#### Table 2 Scale of Sampling

**C-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.

#### C-3 INDIVIDUAL AND COMPOSITE SAMPLES

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

**C-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 weighing about 20 g.

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

#### **C-4 NUMBER OF TESTS**

C-4.1 Test for cyanide content shall be conducted on individual samples.

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

#### **C-5 CRITERIA FOR CONFORMITY**

C-5.1 For Individual Samples

For cyanide 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 cyanide content, (X - 0.6R) shall be greater than or equal to the minimum specified in Table 1.

**C-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 given in Table 1.