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Draft Indian Standard
SILVER OXIDE — SPECIFICATION

(First Revision of IS 2449)

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ICS 71.060

Inorganic Chemicals Sectional Committee, CHD 01 | **Last date of comments: 17th December 2022**

FOREWORD

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

The production of silvered mica plates has been firmly established in the country and silver oxide is an important raw material for this industry. This standard is intended to assist the indigenous manufacturers of silver oxide in producing a material of the required quality. Advances in ceramic capacitors led to the replacement of mica with ceramic in most applications and usage of silvered mica plates reduced drastically.

In this revision, instrumental test methods for the determination of copper, chlorides, iron, lead and sulphates have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause and amendment have been incorporated.

This standard is intended chiefly to cover the technical provisions relating to silver oxide, and it does not include all the necessary provisions of a contract.

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

1 SCOPE

1.1 This standard lays down requirements and methods of sampling and test for silver oxide.

2 REFERENCES

The Indian 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 revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards given below:

<i>IS No.</i>	<i>Title</i>
323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
3025 (Part 2) : 2019/ ISO 11885	Methods of sampling and test (Physical And Chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be dark brown in colour, and shall be free from dirt, reduced silver and other gritty matter.

3.2 Particle Size

The material shall completely pass through 63 micron IS Sieve.

NOTE — BS Sieve 240, ASTM Sieve 230 and Tyler Sieve 250 have their apertures within the limits specified for the IS test sieve mentioned above and may, therefore, be used as 03 micron IS Sieve.

3.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the table.

Table 1 Requirements for Silver Oxide

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Silver oxide (as Ag ₂ O), percent by weight, <i>Min</i>	99.3	A-2
ii)	Copper, lead and iron	To pass test	A-3 or A-9
iii)	Sulphates (as SO ₄), percent by weight, <i>Max</i>	0.005	A-4 or A-8
iv)	Chlorides (as Cl), percent by weight, <i>Max</i>	0.05	A-5 or A-8
v)	Nitrates (as NO ₃) , percent by weight, <i>Max</i>	0.01	A-6 or A-8
vi)	Alkalinity (as Na ₂ O) , percent by weight, <i>Max</i>	0.06	A-7

4 PACKING AND MARKING

4.1 Packing

The size and material of construction of the containers shall be subject to agreement between the purchaser and the supplier.

4.2 Marking

The containers shall be securely closed and marked with the name and weight of the material in the container; manufacturer's name and trade-mark, if any; batch number; and the year of manufacture.

4.2.1 BIS Certification Marking

The packages may also be marked with the Standard Mark.

4.2.1.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed there under, and the products may be marked with the standard mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Annex B.

Annex A

(Clause 3.3)

ANALYSIS OF SILVER OXIDE

A-1 QUALITY OF REAGENTS

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

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

A-2 DETERMINATION OF SILVER OXIDE

A-2.1 Reagents

A-2.1.1 *Dilute Nitric Acid* — 1:1.

A-2.1.2 *Sodium Chloride Solution* — 10 percent.

A-2.2 Procedure

Weigh accurately about 0.15 to 0.2 g of the material, previously dried at $105^{\circ} \pm 2^{\circ}\text{C}$ for 2 h and cooled. Dissolve in the minimum quantity of dilute nitric acid and boil to expel nitrous fumes. Dilute to about 100 ml with water. Filter off the insoluble matter and wash well with hot water. Collect the washings and the filtrate and heat to 60° to 70°C . Add sodium chloride solution slowly with constant stirring till no more precipitate of silver chloride develops. Boil for 5 min to coagulate the precipitate. Allow it to settle in a dark place for 5 to 6 h. Filter the precipitate through a weighed sintered crucible No. 3 or filter paper (Whatman No. 40 or equivalent). Wash well with cold water acidified slightly with nitric acid and finally with cold water alone. Dry the precipitate in an air oven to constant weight at 130° to 150°C .

A-2.3 Calculation

Silver oxide (as Ag_2O), percent by weight = $\frac{80.83 B}{A}$

where

B = weight in g of the precipitate, and

A = weight in g of the material taken for the test.

A-3 TEST FOR COPPER, LEAD AND IRON

A-3.1 Reagents

A-3.1.1 *Dilute Nitric Acid*— 1:1.

A-3.1.2 *Dilute Ammonium Hydroxide* — 1:1.

A-3.1.3 *Ammonium Molybdate Solution* — 10 percent.

A-3.2 Procedure

Dissolve 5.000 g of the material in dilute nitric acid and boil off the nitrous fumes. Dilute to 200 to 300 ml with water. Add dilute ammonium hydroxide until the precipitate which is formed just dissolves. Let it stand for 3 h. The material shall be taken to have passed the test for iron if there is no brown precipitate, and for copper if the solution does not show a blue colour. To the clear solution, add 1 ml of ammonium molybdate solution. The material shall be taken to have passed the test for lead if no turbidity or precipitate is formed.

A-3.3 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD

A-3.3.1 Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

A-3.4 Alternative Method

Iron and lead may alternatively be determined by instrumental test method as prescribed at A-9.

A-4 TEST FOR SULPHATES

A-4.1 Reagents

A-4.1.1 *Dilute Nitric Acid* — approximately 5 N.

A-4.1.2 *Barium Nitrate Solution* — 6 percent.

A-4.2 Procedure

Dissolve 2.000 g of the material in dilute nitric acid and boil to expel nitrous fumes. Dilute to 200 ml and add 5 ml of barium nitrate solution. Let it stand for 1 h. Prepare a blank for comparison.

A-4.2.1 The material shall be considered as not having exceeded the limit prescribed in Table 1 if no turbidity or precipitate is produced.

A-4.3 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at A-8.

A-5 TEST FOR CHLORIDES

A-5.1 Apparatus

A-5.1.1 Nessler Tubes — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 Dilute Nitric Acid — approximately 5 N.

A-5.2.2 Standard Chloride Solution

Dissolve 1.64 g of sodium chloride, previously dried at $105^{\circ} \pm 2^{\circ}\text{C}$, in water and dilute to 1000 ml in a volumetric flask. Further dilute 100 ml of the solution to 1000 ml. 1 ml of the solution contains 0.1 mg of chlorides (as Cl).

A-5.2.3 Silver Nitrate Solution — 10 percent.

A-5.3 Procedure

Dissolve 0.200 g of the material in 10 ml of dilute nitric acid. Boil to expel nitrous fumes. Cool and transfer to a Nessler tube and dilute to the mark. In another Nessler tube, take 1 ml of standard chloride solution, add 10 ml of dilute nitric acid and 1 ml of silver nitrate solution. Dilute to the mark. Compare the turbidity produced in the two Nessler tubes.

A-5.3.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if the turbidity produced with the material is not greater than that produced in the control test.

A-5.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-8**.

A-6 TEST FOR NITRATES**A-6.1 Reagents**

A-6.1.1 Dilute Hydrochloric Acid — approximately 5 N.

A-6.1.2 Standard Indigo Carmine Solution

Dissolve 0.2 g of indigo carmine in 500 ml of dilute sulphuric acid, add 20 ml of hydrochloric acid and sufficient dilute sulphuric acid to produce 1000 ml. Standardize the solution so that 10 ml added to 3.3 ml of M/1000 potassium nitrate solution is just decolourized on adding 13 ml of sulphuric acid and heating to boiling.

A-6.1.3 Dilute Sulphuric Acid — 5 N.

A-6.2 Procedure

Suspend 1.000 g of the material in 10 ml of water, mix well and allow to settle. Take the supernatant clear liquid, add 0.5 ml of standard indigo carmine solution and 5 ml of dilute sulphuric acid and heat to boiling.

A-6.2.1 The limit prescribed shall be taken as not having been exceeded if the blue colour produced does not entirely disappear.

A-7 TEST FOR ALKALINITY**A-7.1 Reagents**

A-7.1.1 *Standard Sulphuric Acid* — 0.05 N.

A-7.1.2 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit conforming to IS 323 and dilute with water to 100 ml.

A-7.2 Procedure

Take 10.0 g of the material in a flask and add 500 ml of water. Reflux on a hot water-bath for about 1 h. Filter 50 ml of the clear supernatant liquid. Add a few drops of phenolphthalein indicator solution and titrate against standard sulphuric acid.

A-7.3 Calculation

Alkalinity (as Na₂O), percent by weight = $\frac{31 V N}{W}$

where

V = volume in ml of standard sulphuric acid used,

N = normality of standard sulphuric acid, and

W = weight in g of the material taken for the test.

A-8 ION CHROMATOGRAPHY FOR CHLORIDES, NITRATES AND SULPHATES

A-8.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-8.2 Equipment

A-8.2.1 *Anion Guard Column* — A protector of the separator column.

A-8.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

A-8.2.3 *Anion Suppressor Device* — Anion micromembrane suppressor is used to analyse the data

A-8.2.4 *Detector* — Conductivity detector

A-8.2.5 *Software*

Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-8.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

A-8.3 Reagents

A-8.3.1 *Glass or Polyethylene Sample Bottles*

A-8.3.2 *Distilled water or deionized water free from the anions of interest.*

A-8.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-8.3.4 *Micromembrane Suppressor Solution, (0.025 N of sulphuric acid)*

Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water.

A-8.4 Standard solutions

A-8.4.1 *Chloride* — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-8.4.2 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

A-8.4.3 *Nitrate Stock Solution*

Dry approximately 2 g of sodium nitrate (NaNO₃) at 105 °C for 24 h. Dissolve exactly 1.3707 g of the dried salt in reagent water, and dilute to 1 l with reagent water in a volumetric flask.

A-8.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1-1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-8.6 Procedure

Dissolve between 1 to 5 grams sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This

membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-8.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

A-9 DETERMINATION OF LEAD AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-9.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-9.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

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

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

A-9.3 Reagents and Solutions

A-9.3.1 Nitric acid (65 percent) Suprapure

A-9.3.2 Standard stock solution

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1000 µg/ml of Lead and Iron in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-9.3.3 Standard solution

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask & make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

A-9.3.4 Sample preparation

Weigh about 2.5 g polyphosphoric acid sample in a 50 ml volumetric flask and add 1.0 ml Nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument

A-9.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-9.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument

A-9.5 Procedure

A-9.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-9.5.2). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary

adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-9.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the lead and Iron in the sample solution.

NOTE – It is recommended that IS 3025(Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

A-9.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

Annex B

(Clause 5.1)

SAMPLING OF SILVER OXIDE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Samples shall be taken in a protected place.

B-1.3 The sampling instrument shall be clean and dry.

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

B-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.

B-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.

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

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

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

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

B-2.2 The number of containers (n) to be selected from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 3.

Table 3 Number of Containers to be selected for Sampling

Lot Size N (1)	No. of Containers to be Selected n (2)
4 to 15	3
10 to 40	4
41 to 65	5
66 to 110	7
111 to above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criterion for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, the following procedure shall be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3... etc. up to r and so on, where r is the integral part of N/n . Every r^{th} container thus counted shall be taken out.

B-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 From each of the containers selected according to **B-2.3**, draw, with an appropriate sampling instrument, a representative portion of the material, about 25 g.

B-3.2 Out of these portions collected from all the selected containers, equal quantity of the material (about 15 g) shall be taken and mixed thoroughly to form a composite sample. The composite sample so formed shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts and each such part shall constitute an individual sample. One set of the individual samples shall be marked for the purchaser, another for the supplier and the third one for the referee.

B-3.4 All the individual and composite samples shall be immediately transferred to separate containers and labelled with full identification particulars.

B-3.5 The referee sample, consisting of a composite sample and a set of n individual samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of a dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of silver oxide shall be conducted on each of the individual samples separately.

B-4.2 Tests for the determination of all other characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to the requirements of this specification if the conditions given in **B-5.2** and **B-5.3** are satisfied.

B-5.2 For Individual Samples

From the individual test results for silver oxide, the mean (\bar{X}) and the range (R) of the test results shall be computed (range being defined as the difference between the maximum and minimum of the test results).

B-5.2.1 The value of the expression ($\bar{X} - 0.6 R$) calculated from the test results shall be greater than or equal to 99.3.

B-5.3 For Composite Sample

The test results for those characteristics which have been determined on the composite sample shall satisfy the relevant requirements given in the specification.