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भारतीय मानक मसौदा
**पोटेशियम कार्बोनेट, एनहाइड्रस और सेस्क्यूहाइड्रेट,
फोटोग्राफिक ग्रेड — विशिष्टि**
(IS 13381 का पहला पुनरीक्षण)

Draft Indian Standard

**POTASSIUM CARBONATE, ANHYDROUS
AND SESQUIHYDRATE, PHOTOGRAPHIC
GRADE—SPECIFICATION**

(First Revision of IS 13381)

(ICS 37.040.30)

Electroplating Chemicals And Photographic Materials Sectional
Committee, CHD 5

Last Date for Comments: **6th April 2024**

FOREWORD

(formal clause shall be added later)

Anhydrous potassium carbonate (K_2CO_3) has molecular mass of 138.21 and potassium carbonate, sesquihydrate ($K_2CO_3 \cdot 1.5H_2O$) has molecular mass of 165.22.

This standard was first published in 1992. In this revision, alternative instrumental test methods AAS, ICP-MS and ICP-OES have been incorporated for the determination of lead and iron and the standard has been updated based on the experience of last three decades and 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.

Indian Standard
**POTASSIUM CARBONATE, ANHYDROUS AND
SESQUIHYDRATE, PHOTOGRAPHIC GRADE — SPECIFICATION
(First Revision)**

1 SCOPE

This standard prescribes requirements and methods of sampling and test for potassium carbonate, anhydrous and sesquihydrate, photographic grade for use in processing of photographic materials.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provision 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 standards.

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third Revision</i>)
IS 1070 : 2023	Reagent Grade Water — Specification (<i>fourth 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	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	Random sampling and randomization procedures (<i>first Revision</i>)
IS 12074 : 1987	Method for determination of lead by atomic absorption spectrophotometer
IS 13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method

3 GRADES

The material shall be of following two grades:

- a) Grade 1 — Anhydrous
- b) Grade 2 — Sesquihydrate

4 REQUIREMENTS

4.1 Description

Anhydrous potassium carbonate, photographic grade shall be in the form of white granules or powder. Sesquihydrate potassium carbonate, photographic grade shall be in the form of colourless crystals.

4.2 Solution in Water

A freshly prepared aqueous solution of the material containing 20 g of the sample in 100 ml of water shall be clear and free from sediment other than slight flocculence.

4.3 The material shall also comply with the requirements prescribed in Table 1 when tested by the methods given in Annex A. The reference to relevant clauses of Annex A is given in col 4 of Table 1.

Table 1 Requirements for Potassium Carbonate, Anhydrous and Sesquihydrate, Photographic Grade

(Clause 4.3)

SI No.	Characteristic	Requirements		Methods of Test, Ref to CI No. in Annex A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Assay (as K_2CO_3), percent by mass	98.5, <i>Min</i>	98.5-102.0	A-1
ii)	Bicarbonate, percent by mass, <i>Max</i>	0.25	0.4	A-2
iii)	Free alkali (as KOH), percent by mass, <i>Max</i>	0.2	0.2	A-3
iv)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	0.001	A-4
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	0.001	A-5
vi)	Halides (as Cl), percent by mass, <i>Max</i>	0.03	0.03	A-6
vii)	Reaction to ammonical silver nitrate	To pass test	To pass test	A-7
viii)	Volatile matter, percent by mass, <i>Max</i>	2.0	—	A-8

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable airtight bottles or as agreed to between the purchaser and the supplier.

5.2 Marking

The container shall be securely closed and legibly and indelibly marked with the following information:

- Name of the material,
- Net mass of the material,
- Date of manufacture,
- Indication of the source of manufacture, and
- Batch or lot number in code or otherwise to enable the batch of manufacture to be traced from records.

5.2.1 BIS Certification Marking

The containers may also be marked with the BIS Certification Mark.

5.2.1.1 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 thereunder. The details of the conditions under which the licence for use of the BIS Certification Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative test samples of the material and their criteria for conformity to this standard shall be as prescribed in Annex B.

7 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.

ANNEX A
(*Clause 4.3*)

**METHODS OF TEST FOR POTASSIUM CARBONATE, ANHYDROUS AND
SESQUIHYDRATE, PHOTOGRAPHIC GRADE**

A-1 ASSAY

A-1.1 Reagents

A-1.1.1 *Hydrochloric Acid* — 1.0 M.

A-1.1.2 *Methyl Orange Indicator*

Add 0.1 g of methyl orange to 100 ml of water, stir until dissolved.

A.1.2 Procedure

Place about 3.0 g of the sample into a tared weighing bottle. Weigh to the nearest 0.000 1 g and dissolve the sample in approximately 50 ml of water. Add 2 drops of methyl orange indicator and titrate with hydrochloric acid until the colour changes from yellow to red.

A-1.3 Calculation

$$\text{Assay (as K}_2\text{CO}_3 \text{), percent by mass} = \frac{V M \times 6.91}{M_1}$$

$$\text{and Assay (as K}_2\text{CO}_3 \cdot 1.5\text{H}_2\text{O} \text{)} = \frac{V M \times 8.26}{M_1}$$

where

V = volume in ml of hydrochloric used;
 M = molarity of hydrochloric acid; and
 M_1 = mass in g of the sample taken for the test.

A-2 DETERMINATION OF BICARBONATE

A-2.1 Reagents

A-2.1.1 *Standard Barium Chloride Solution*

Weigh 24 g of barium chloride, dihydrate and transfer to a 100 ml volumetric flask. Dissolve in water and make upto the mark.

A-2.1.2 *Phenolphthalein Indicator*

Weigh 0.1 g of phenolphthalein and dissolve in 60 ml of ethyl alcohol. Dilute to 100 ml with water.

A-2.1.3 *Sodium Hydroxide Solution* — 0.1 M.

A-2.1.4 *Carbon dioxide Free Water*

Boil distilled water for 20 minutes, cool and store in a stoppered bottle.

A-2.2 Procedure

Dissolve (2.5 ± 0.1) g of the sample in approximately 30 ml of carbon-dioxide free water in a 100 ml stoppered conical flask. Add 25 ml of barium chloride solution neutral to phenolphthalein and add 10 drops of phenolphthalein indicator. Stopper and swirl. If the solution has a pink colour, proceed immediately to **A-3**. If the solution is colourless, titrate with 0.1 M sodium hydroxide solution. Not more than 1 ml of 0.1 M sodium hydroxide solution shall be required to produce the first appearance of a faint pink colour which persists for 30 seconds.

A-3 DETERMINATION OF FREE ALKALI

A-3.1 Reagents

A-3.1.1 *Hydrochloric Acid* – 0.1 M.

A-3.2 Procedure

If the solution has a pink colour after the addition of the indicator specified in **A-2.2**, titrate with 0.1 M hydrochloric acid. Not more than 4.5 ml of 0.1 M hydrochloric acid shall be required to discharge the pink colour.

A-4 DETERMINATION OF HEAVY METALS (as Pb)

A-4.1 General

Four methods are prescribed for the determination of heavy metals (as Pb). Either of these may be used for general routine purposes, but in case of a dispute Method C shall be used as referee method.

A-4.2 Method A

A-4.2.1 *General*

The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-4.2.2 *Reagents*

A-4.2.2.1 *Dilute Ammonium Hydroxide* — 10 percent (*m/m*).

A-4.2.2.2 *Dilute Hydrochloric Acid* — approximately 0.1 M.

A-4.2.2.3 *p-Nitrophenol Indicator* — 0.25 percent aqueous solution (*m/v*).

A-4.2.2.4 *Hydrogen Sulphide* — saturated aqueous solution.

A-4.2.2.5 *Standard Lead Solution*

Weigh accurately 1.60 g of lead nitrate and add 50 ml of nitric acid of analytical reagent grade. Dissolve in water and dilute to 1 000 ml with water. Pipette out 10 ml of this solution and dilute further to 1 000 ml with water, freshly when required. (1 ml of the solution is equivalent to 0.01 mg of lead (as Pb)).

A-4.2.3 *Procedure*

A-4.2.3.1 Dissolve 2.00 g of the material in 25 ml of water in a Nessler cylinder and add a few drops of *p* nitrophenol indicator and then add ammonium hydroxide dropwise, until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless again and then add 0.5 ml in excess. Add 5 ml of hydrogen sulphide solution and dilute to 50 ml.

A-4.2.3.2 At the same time transfer 2 ml of standard lead solution into another Nessler cylinder and render it neutral to p-nitrophenol indicator by using dilute ammonium hydroxide and dilute hydrochloric acid as described in **A-4.2.3.1**. Add 0.5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml.

A-4.2.3.3 The material shall be considered to have passed the requirement of the test if the colour produced in the sample solution is not more intense than that produced in the standard solution.

A-4.3 Method B

Determine heavy metals (as Pb) content by Atomic Absorption Spectrophotometer (AAS) in accordance with the method prescribed in IS 12074.

A-4.4 Method C

Determine heavy metals (as Pb) content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

A-4.5 Method D

Determine heavy metals (as Pb) content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

A-5 DETERMINATION OF IRON

A-5.1 General

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

A-5.2 Method A

A-5.2.1 Reagents

A-5.2.1.1 pH 5 Acetate Buffer

Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to 1 000 ml with water. Adjust the final pH of the solution to 5.0 ± 0.1 with acetic acid or 10 percent sodium hydroxide (NaOH) solution.

A-5.2.1.2 o-phenanthroline Mixture

Thoroughly mix equal parts of 0.1 percent aqueous solution of o-phenanthroline, 10 percent hydroxylamine hydrochloride solution in water and pH 5 acetate buffer.

A-5.2.1.3 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 (\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in 10 ml dilute sulphuric acid and dilute with water to 1 000 ml and mix thoroughly. Dilute 100 ml of this solution again to 1 000 ml with water. (1 ml of the solution is equivalent to 0.01 mg of iron (as Fe)).

A-5.2.2 Procedure

Dissolve 1 g of the sample in 40 ml of water. Add 5 ml of the o-phenanthroline mixture and let stand for 10 minutes. Dilute to 50 ml with water and mix well. Any colour produced shall not be greater than that produced by a freshly prepared solution of a soluble iron salt equivalent to 0.01 mg of iron containing 2 ml of 1 percent (v/v) hydrochloric acid, and treated in the same manner as that of sample. Use Nessler cylinders for comparison. The sample shall be considered to have passed the requirement of the test if the colour produced with the sample is not darker than that with the standard solution.

A-5.3 Method B

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

A-5.4 Method C

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

A-5.5 Method D

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

A-6 DETERMINATION OF HALIDES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 *Silver Nitrate Solution* — 10 percent.

A-6.2.2 *Nitric Acid* — concentrated (*see* IS 264)

A-6.2.3 *Standard Chloride Solution*

Dissolve 0.165 g of sodium chloride in water and make it to 100 ml. Dilute 10 ml of this solution to 100 ml with water. 1 ml of the diluted solution contains 0.000 1 g of chlorine (as Cl).

A-6.3 Procedure

Dissolve (1 ± 0.01) g of the sample in 40 ml of water in a 50 ml Nessler cylinder. Add 2 drops of concentrated nitric acid, 2 drops of 10 percent silver nitrate solution, dilute to the mark with water and mix thoroughly.

A-6.3.1 In another Nessler cylinder, take 3.0 ml of standard chloride solution and treat it similarly. Compare the turbidity produced in the two cylinders. The sample shall be considered to have passed the requirement if the turbidity produced with the sample is less than that with the standard solution.

A-7 AMMONIACAL SILVER NITRATE TEST

A-7.1 Reagent

Mix equal volumes of 10 percent silver nitrate solution and concentrated ammonium hydroxide solution.

A-7.2 Procedure

Dissolve 1 g of the sample in 20 ml of water. Add 10 ml of freshly prepared reagent and mix well. Let the mixture stand for 2 minutes and compare it with a blank prepared by adding 5 ml of concentrated ammonium hydroxide solution to a solution of 1 g of the sample dissolved in 25 ml of water. No colour or precipitate in excess of that produced in the blank should be noticeable.

A-8 DETERMINATION OF VOLATILE MATTER

A-8.1 Procedure

Take about 3 g of the sample in a glass stoppered weighing bottle and weigh to the nearest 0.000 1 g. Dry at 150°C for four hours, cool in a desiccator and weigh to constant mass to the nearest 0.000 1 g.

A-8.2 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M - M_1) \times 100}{M}$$

where

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

M_1 = mass in g of the material obtained on drying.

ANNEX B (Clause 6)

SAMPLING OF POTASSIUM CARBONATE, ANHYDROUS AND SESQUIHYDRATE, PHOTOGRAPHIC GRADE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Samples shall not be taken in an exposed place.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are nearly filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In any consignment of the material, all the containers of same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers (n) to be selected for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Scale of Sampling

(Clause B-2.2)

Lot Size	Number of Containers to be Selected
N	N
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

B-2.3 These containers shall be selected at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS 4905 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw a small portion of the material from different parts of each selected container with an appropriate sampling instrument, The total quantity of the material drawn from each container shall be sufficient to conduct the tests for the characteristics given in Table 1 and should be about 400 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 800 g. This composite sample 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-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as a referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for determination of assay (as Na_2CO_3), percent by mass, shall be conducted on each of the individual samples.

B-4.2 Tests for all other characteristics given in Table 1 shall be conducted on the composite sample.

