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SPECIFICATION FOR POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

(Revised)

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INDIAN STANDARDS INSTITUTION
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Indian Standard

SPECIFICATION FOR POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

(Revised)

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Indian Standard

SPECIFICATION FOR POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

(Revised)

0. FOREWORD

0.1 This Indian Standard (Revised) was adopted by the Indian Standards Institution on 21 January 1964, after the draft finalized by the Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1953. The Sectional Committee responsible for the preparation of this standard reviewed it in the light of the progress made by the industry and decided to revise it. In this revised standard, limits for aluminium and sodium have been included and the limit for chloride has been reduced. Sampling procedure and some of the test methods have also been modified.

0.3 Flame photometric method has been prescribed for the determination of sodium. Since widely differing types of instruments are available for this determination, detailed instructions have not been given.

0.4 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 : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for potassium bichromate, technical and analytical reagent.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of orange-red crystals or granular powder, free from foreign matter and visible impurities, forming a clear solution when dissolved in water.

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

TABLE 1 REQUIREMENTS FOR POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

Sl No.	Characteristic	Requirement for Grade		Method of Test (Ref to Cl No. in Appendix A)
		Technical	Analytical Reagent	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by weight, *Max*	0·5	0·05	A-2
ii)	Matter insoluble in water, percent by weight, *Max*	0·30	0·005	A-3
iii)	Potassium bichromate ($K_2Cr_2O_7$), percent by weight, *Min*	99·0	99·9	A-4
iv)	Sulphates (as SO_4), percent by weight, *Max*	0·30	0·01	A-5
v)	Chlorides (as Cl), percent by weight, *Max*	0·50	0·000 5	A-6
vi)	Calcium (as $CaSO_4$), percent by weight, *Max*	0·10	0·005	A-7
vii)	Aluminium (as Al), percent by weight, *Max*	—	0·003	A-7
viii)	Sodium (as Na), percent by weight, *Max*	—	0·05	A-8

2.3 The *p*H of a 5 percent solution in carbon dioxide-free water of analytical reagent grade of the material when determined by a suitable *p*H meter shall be 3·7 to 3·9.

3. PACKING AND MARKING

3.1 Potassium bichromate, technical, shall be packed as mutually agreed to between the purchaser and the supplier.

3.2 Potassium bichromate, analytical reagent, shall be packed in glass or other suitable containers on which the material has no action.

3.3 The containers shall be securely closed against contamination, and marked with the manufacturer's name, weight and grade of the material in the container, recognized trade-mark, if any, and the year of manufacture of the material.

3.3.1 Containers for potassium bichromate, analytical reagent, shall also be labelled with full analytical data of the characteristics prescribed in Table 1.

3.4 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — Representative test samples of the material shall be prepared as prescribed in Appendix B.

4.2 Number of Tests

4.2.1 For both grades, tests for the determination of potassium bichromate shall be conducted on each of the individual samples constituting the set of test samples.

4.2.2 Tests for the remaining characteristics shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The mean and the range of the test results for the determination of potassium bichromate shall be calculated as follows:

Mean ($\bar{X}$) = The sum of the test results divided by the number of test results, and

Range (R) = The difference between the maximum and the minimum values of the test results.

4.3.1.1 If ($\bar{X} - 0{\cdot}6R$) is equal to or greater than the minimum requirement of potassium bichromate for the relevant grade, the lot shall be declared to have satisfied the requirement for potassium bichromate for the relevant grade.

4.3.2 *For Composite Sample* — The test results on the composite sample shall meet the corresponding requirements specified in **2** for the relevant grade.

4.3.3 A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristics listed in **2.**

4.3.4 If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of this specification.

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

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

A-2. DETERMINATION OF MOISTURE

A-2.1 Procedure — Weigh accurately about 1 to 2 g of the material, ground to fine powder, into a tared shallow dish and dry to constant weight at 110° ± 2°C.

A-2.2 Calculation

$$\text{Moisture, percent by weight} = 100\frac{W_1}{W_2}$$

where

W_1 = loss in weight in g, and

W_2 = weight in g of the material taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Weigh accurately about 10 g of the material and dissolve it in 100 ml of water. Heat on a water-bath for 30 minutes.

*Specification for water, distilled quality (*revised*).

Filter the solution through a tared Gooch (G No. 4) or sintered glass crucible. Wash the residue, if any, with hot water and dry to constant weight at 105° to 110°C.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by weight} = 100\,\frac{W_1}{W_2}$$

where

W_1 = weight in g of the residue, and

W_2 = weight in g of the material taken for the test.

A-4. DETERMINATION OF POTASSIUM BICHROMATE

A-4.0 Principle — Potassium dichromate is determined iodometrically.

A-4.1 Reagents

A-4.1.1 *Potassium Iodide*

A-4.1.2 *Dilute Sulphuric Acid* — 1 : 3.

A-4.1.3 *Standard Sodium Thiosulphate Solution* — 0·1 N, freshly standardized.

A-4.1.4 *Starch Solution* — Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

A-4.2 Procedure — Weigh accurately about 0·2 g of the material and dissolve it in 200 ml of freshly boiled and cooled water in a glass-stoppered flask. Add about 3 g of potassium iodide, and about 10 ml of dilute sulphuric acid. Allow to stand for 10 minutes in the dark and titrate the liberated iodine with standard sodium thiosulphate solution, using starch solution towards the end, until the blue colour produced just disappears.

A-4.3 Calculation

$$\text{Potassium bichromate } (K_2Cr_2O_7), \text{ percent by weight} = \frac{4{\cdot}904\,NV}{W}$$

where

N = normality of standard sodium thiosulphate solution,

V = volume in ml of standard sodium thiosulphate solution used in the titration, and

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

A-5. DETERMINATION OF SULPHATES

A-5.1 For Technical Grade

A-5.1.0 *Principle* — Sulphates are precipitated as barium sulphates with barium chloride ignited and weighed.

A-5.1.1 *Reagents*

A-5.1.1.1 *Concentrated hydrochloric acid* — conforming to IS : 265-1962*.

A-5.1.1.2 *Rectified spirit* — conforming to IS : 323-1959†.

A-5.1.1.3 *Barium chloride solution* — approximately 12 percent.

A-5.1.1.4 *Dilute hydrochloric acid* — approximately 1 : 99.

A-5.1.2 *Procedure* — Weigh accurately about 5 g of the material and dissolve in the minimum quantity of water. If insoluble matter is present, filter the solution into a 250-ml beaker. Add to the solution or filtrate 35 ml of concentrated hydrochloric acid and 25 ml of rectified spirit. Boil until all the chromate is reduced to the green trivalent form. Boil off most of the aldehydes formed. Dilute the solution to 150 ml with water and add slowly while stirring 20 ml of hot barium chloride solution. Continue boiling for 15 minutes and allow to stand for 4 hours at 60°C. Filter the solution containing barium sulphate through a tared Gooch crucible or a filter paper (Whatman No. 42 or equivalent). Wash twice with dilute hydrochloric acid and then with hot water till it is free from chlorides. Dry the precipitate and then ignite at 800°C to constant weight.

A-5.1.3 *Calculation*

$$\text{Sulphates (as } SO_4\text{), percent by weight} = 41{\cdot}15\,\frac{B}{W}$$

where

B = weight in g of the precipitate, and

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

*Specification for hydrochloric acid (*revised*).

†Specification for rectified spirit (*revised*).

A-5.2 For Analytical Reagent Grade

A-5.2.0 *Principle* — A solution of the material is tested with barium chloride for appearance of turbidity.

A-5.2.1 *Reagents*

A-5.2.1.1 *Dilute hydrochloric acid* — approximately 5 N.

A-5.2.1.2 *Barium chloride solution* — approximately 12 percent.

A-5.2.2 *Procedure* — Weigh 2·0 g of the material and dissolve in 45 ml of water. Filter, if any insoluble matter is present. Add to the solution or filtrate 7 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Allow to stand for 6 hours and then examine.

A-5.2.2.1 The limit given in Table 1 shall be taken as not having been exceeded if no turbidity or precipitate is produced.

A-6. DETERMINATION OF CHLORIDES

A-6.1 For Technical Grade

A-6.1.0 *Principle* — Chlorides are determined volumetrically by titration with standard silver nitrate solution.

A-6.1.1 *Reagents*

A-6.1.1.1 *Calcium carbonate*

A-6.1.1.2 *Standard silver nitrate solution* — 0·1 N.

A-6.1.2 *Procedure* — Weigh accurately about 2 g of the material, and dissolve in 50 ml of water. Neutralize with calcium carbonate till a pale yellow colour is obtained (usually 0·5 g is sufficient). Titrate with standard silver nitrate solution till a red colour is obtained.

A-6.1.3 *Calculation*

$$\text{Chlorides (as Cl), percent by weight} = \frac{3{\cdot}546\,(V_1 - V_2)\,N}{W}$$

where

V_1 = volume in ml of standard silver nitrate solution used in the titration with the material,

V_2 = volume in ml of standard silver nitrate solution used in the blank determination,

N = normality of standard silver nitrate solution, and

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

A-6.2 For Analytical Reagent Grade

A-6.2.0 *Principle* — A solution of the material is tested with silver nitrate for appearance of opalescence.

A-6.2.1 *Reagents*

A-6.2.1.1 *Dilute nitric acid* — approximately 5 N.

A-6.2.1.2 *Silver nitrate solution* — 5 percent.

A-6.2.2 *Procedure* — Weigh 2·0 g of the material and dissolve in 45 ml of water. Filter, if any insoluble matter is present. Add to the solution or filtrate 10 ml of dilute nitric acid and 0·5 ml of silver nitrate solution. Allow to stand for 5 minutes and then examine.

A-6.2.2.1 The limit given in Table 1 shall be taken as not having been exceeded if no opalescence is produced after 5 minutes.

A-7. DETERMINATION OF CALCIUM AND ALUMINIUM

A-7.0 The test given in **A-7.1** is for technical grade for calcium only. The test given in **A-7.2** is for analytical reagent grade for both calcium and aluminium.

A-7.1 For Technical Grade

A-7.1.0 *Principle* — Calcium is precipitated as calcium oxalate with ammonium oxalate and ammonium hydroxide. The precipitate is dissolved in dilute sulphuric acid and titrated with standard potassium permanganate solution.

A-7.1.1 *Reagents*

A-7.1.1.1 *Ammonium oxalate solution* — saturated.

A-7.1.1.2 *Methyl red indicator solution* — Dissolve 0·5 g of methyl red in water and dilute the solution to one litre.

A-7.1.1.3 *Dilute ammonium hydroxide* — approximately 5 N and 0·5 N.

A-7.1.1.4 *Dilute sulphuric acid* — 1 : 4.

A-7.1.1.5 *Standard potassium permanganate solution* — 0·01 N.

A-7.1.2 *Procedure* — Weigh accurately about 5 g of the material, and dissolve in 50 ml of water. Filter, if necessary. Heat the filtrate and washings to boiling and add, with constant stirring, an excess of hot ammonium oxalate solution. Add two to three drops of methyl red

indicator, and to the hot solution maintained at 70° to 80°C add, with constant stirring, dilute ammonium hydroxide (5 N) dropwise, until the colour changes from red to yellow. Allow the solution to stand at 70° to 80°C for one hour, and then filter through a filter paper (Whatman No. 40 or its equivalent). Test the filtrate for the presence of calcium with ammonium oxalate solution before throwing it away. Wash the precipitate a few times with dilute ammonium hydroxide (0·5 N) until it is free from oxalates. Pierce the apex of the filter paper with a stirring rod, and wash the bulk of the precipitate through the funnel into a conical flask with hot water. Then wash the filter paper a few times with warm dilute sulphuric acid. Finally, wash the filter paper thoroughly with hot water. Add about 30 ml of dilute sulphuric acid to the washings, dilute to 250 ml, heat to 60°C and titrate while hot with standard potassium permanganate solution.

A-7.1.3 *Calculation*

$$\text{Calcium (as } CaSO_4\text{), percent by weight} = 6{\cdot}807 \frac{VN}{W}$$

where

V = volume in ml of standard potassium permanganate solution required for the titration,

N = normality of standard potassium permanganate solution, and

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

A-7.2 For Analytical Reagent Grade

A-7.2.0 *Principle* — A solution of the material is tested with ammonium hydroxide and ammonium oxalate for appearance of turbidity.

A-7.2.1 *Reagents*

A-7.2.1.1 *Dilute ammonium hydroxide* — approximately 5 N.

A-7.2.1.2 *Ammonium oxalate solution* —3·5 percent.

A-7.2.2 *Procedure* — Weigh 2·0 g of the material and dissolve in 40 ml of water. Filter, if necessary. Add 5 ml of dilute ammonium hydroxide and 5 ml of ammonium oxalate solution. Allow to stand for 2 hours and then examine the solution.

A-7.2.2.1 The limit for calcium and aluminium given in Table 1 shall be taken as not having been exceeded if no turbidity or precipitate is produced.

A-8. DETERMINATION OF SODIUM

A-8.1 Procedure — Determine sodium by flame photometer at 589 mμ according to the directions of the manufacturer of the apparatus (*see* **0.3**).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

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 thoroughly by suitable means.

B-1.5 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 almost but not completely filled with the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

B-2. SCALE OF SAMPLING

B-2.1 Lot—All the containers in a single consignment of one grade 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 from each lot for ascertaining conformity of the material to the requirements of the specification.

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

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size	Number of Containers to be Selected
N	n
(1)	(2)
3 to 50	3
51 ,, 200	4
201 ,, 400	5
401 ,, 650	6
651 ,, 1 000	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them as 1, 2, 3,.....up to r and so on, where r is the integral part of N/n. Every rth container thus counted shall be taken out.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples and Referee Sample

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected for sampling. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **2.**

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 300 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 out) shall be divided into three equal parts, each part weighing not less than 10 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 in **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 for the purchaser, another for the supplier and the third shall be used as referee sample.

B-3.2 The referee sample consisting of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1**) shall be marked for this purpose and shall bear the seals of the purchaser and the supplier. It 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.

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