

भारतीय मानक मसौदा
पोटेशियम क्रोमेट — विशिष्टि
(आईएस 6605 का पहला पुनरीक्षण)

Draft Indian Standard

POTASSIUM CHROMATE — SPECIFICATION

(First Revision of IS 6605)

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ICS 71.060

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

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

Potassium chromate is chiefly used in the manufacture of pigments. It is also used in leather finishing, as a mordant in textile industry, in corrosion inhibition and as analytical reagent. It has a limited application in the manufacture of enamels.

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

In this revision, Packing and marking clause has been updated. Also, Reference clause and instrumental test methods for the determination of aluminium, calcium, chlorides, iron, sodium and sulphates have been incorporated as alternate methods.

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 prescribes the requirements and the methods of sampling and test for potassium chromate.

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 revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 2021	Hydrochloric acid – Specification (<i>fifth revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
695 : 2020	Acetic Acid — Specification (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<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>)
4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

3 GRADES

3.1 The material shall be of the following three grades:

- a) Technical (TECH),
- b) Pure, and
- c) Analytical Reagent (AR).

4 REQUIREMENTS

4.1 Description

Potassium chromates shall be in the form of lemon yellow crystals with no water of crystallization. It shall be soluble in water.

4.2 The material when tested according to the methods prescribed in Annex A shall comply with the requirements laid down in Table 1. Reference to the relevant clauses of Annex A is given in col 6 of the table.

Table 1 Requirements for Potassium Chromate

Sl. No.	Characteristic	Requirement for Grade			Methods of Test (Ref to Cl. No. in Annex A)
		Tech	Pure	AR	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.01	0.005	0.005	A-2
ii)	Moisture, percent by mass, <i>Max</i>	0.20	—	—	A-3
iii)	Potassium chromate (as K_2CrO_4), percent by mass, <i>Min</i>	98.5	99.50	99.50	A-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.10	0.005	0.001	A-5 or A-11
v)	Sulphates (as SO_4), percent by mass, <i>Max</i>	0.30	0.05	0.01	A-6 or A-11
vi)	Iron and aluminium oxides (as R_2O_3), percent by mass, <i>Max</i>	—	—	0.002	A-7 or A-12
vii)	Calcium (as Ca), percent by mass, <i>Max</i>	—	0.005	0.005	A-8 or A-12
viii)	Sodium (as Na), percent, by mass, <i>Max</i>	—	—	0.025	A-9 or A-13
ix)	pH of aqueous solution	—	8.6 to 9.0	8.6 to 9.0	A-10

5 PACKING AND MARKING

5.1 Packing

Technical grade of the material shall be packed in mild steel drums lined with polyethylene or gunny bags with polyethylene lining. Pure grade shall be packed in polyethylene bags which shall be placed in cardboard cartons. Analytical reagent grade shall be packed in bottles.

5.2 Marking

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

- Name and grade of the material;
- Net mass;
- Year of manufacture;
- Manufacturer's name and/or -his recognized trade-mark, if any; and
- Lot number in code or otherwise to enable the batch of manufacture to be traced from records.
- Cautionary warning* — Potassium chromate is carcinogenic. The compound is also corrosive and exposure may produce severe eye damage or blindness. Human exposure further encompasses impaired fertility, heritable genetic damage and harm to unborn children.

5.2.1 In case of AR grade of the material, complete chemical analysis in respect of the characteristics

specified in Table 1 shall also appear on the label.

5.3 BIS Certification Marking

The product may also be marked with the Standard Mark.

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 drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2)

METHODS OF TEST FOR POTASSIUM CHROMATE

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 that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-2.1 Procedure

Weigh accurately about 20 g of the material and transfer to a beaker. Dissolve in about 200 ml of water and filter through sintered glass crucible No. G4. Wash with water till the filtrate is colourless and collect the washings. Weigh till constant mass is obtained. Dry the crucible at 105 ± 2 °C and weigh till constant mass is obtained.

A-2.1.1 Take the filtrate along with the washings in a 500 ml volumetric flask and make up the volume with water up to the mark. This shall be the prepared solution for analysis.

A-2.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue, and

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

A-3 DETERMINATION OF MOISTURE

A-3.1 Procedure

Weigh accurately about 5 g of the material in silica or porcelain dish of about 75 ml capacity. Dry the contents in an oven at 125 ± 5 °C to constant mass.

A-3.2 Calculation

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

where

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

M_1 = mass in g of the material after drying.

A-4 DETERMINATION OF POTASSIUM CHROMATE

A-4.1 Reagents

A-4.1.1 *Standard Potassium Permanganate Solution* — 0.05 N.

A-4.1.2 *Ferrous Ammonium Sulphate Solution* — approximately 0.1 N.

A-4.1.3 *Dilute Sulphuric Acid* — approximately 2 N.

A-4.2 Procedure

Pipette out 50 ml of the prepared sample solution (*see A-2.1.1*) in a 500 ml volumetric flask and make up the volume with water. Take out 25 ml of the solution in a conical flask. Add 25 ml of dilute sulphuric acid and 25 ml of ferrous ammonium sulphate solution. Titrate the excess of ferrous ammonium sulphate against standard potassium permanganate solution. Conduct a blank titration also.

A-4.3 Calculation

$$\text{Potassium chromate (as K}_2\text{CrO}_4\text{), percent by mass} = \frac{(V_1 - V_2) \times N \times 129.46}{M}$$

where

V_1 = volume in ml of standard potassium permanganate solution used in the blank titration,

V_2 = volume in ml of standard potassium permanganate solution used in the titration with the material,

N = normality of standard potassium permanganate solution, and

M = mass in g of the material present in the aliquot.

A-5 DETERMINATION OF CHLORIDES

A-5.1 For Technical Grade

A-5.1.1 Reagents

A-5.1.1.1 *Concentrated nitric acid* — *see* IS 264.

A-5.1.1.2 *Silver nitrate solution* — approximately 5 percent (m/v).

A-5.1.1.3 *Dilute nitric acid* — approximately 4 N.

A-5.1.2 Procedure

Take 200 ml of the prepared sample solution. Add 20 ml of concentrated nitric acid and heat to about 50°C. Add to this hot solution, sufficient volume of silver nitrate solution to precipitate chlorides completely. Protect the silver chloride precipitate from light by wrapping black paper around the container. Cool the solution and filter through a tared sintered glass crucible No. G4. Wash the precipitate first with dilute nitric acid to dissolve the adhering silver chromate and then with cold water. Dry the crucible and its contents to constant mass at 130 ± 2 °C.

A-5.1.3 Calculation

$$\text{Chlorides (as Cl), percent by mass} = \frac{24.7 \times M_1}{M}$$

where

M_1 = mass in g of silver chloride precipitate, and

M = mass in g of the material present in the aliquot.

A-5.2 For Pure and Analytical Reagent Grades**A-5.2.1 Apparatus**

A-5.2.1.1. Nessler cylinders — 50 ml capacity.

A-5.2.2 Reagents

A-5.2.2.1 Dilute nitric acid — approximately 4 N.

A-5.2.2.2 Silver nitrate solution — approximately 5 percent (m/v).

A-5.2.2.3 Standard chloride solution

Dissolve 0.164 9 g of ignited sodium chloride in 1000 ml of water. Dilute 100 ml of the solution to 1 l. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

A-5.2.2.4 Ammonium hydroxide — 20 percent (m/m).

A-5.2.3 Procedure

Weigh accurately 0.20 g of the material, dissolve in 10 ml of water in a Nessler cylinder and add 1 ml of ammonium hydroxide and 1 ml of silver nitrate solution. Add 5 ml of dilute nitric acid and dilute to the mark. Carry out a control test in another Nessler cylinder using 1 ml of standard chloride solution for pure grade and 0.2 ml for AR grade of the material and the same quantities of other reagents. Compare the turbidity produced in the two cylinders.

A-5.2.3.1 The colour in the sample can be compensated for by superimposing a cylinder containing 0.2 g of the sample, 1 ml of ammonium hydroxide, 5 ml of dilute nitric acid, made up to the mark with water, over the cylinder containing the standard, and placing a cylinder containing 1 ml of ammonium hydroxide and 5 ml of dilute nitric acid behind the cylinder containing the sample. Both the turbidities are thus viewed through the same depth and colour of the solution.

A-5.2.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced with the material is not greater than that produced in the control test.

A-5.3 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-11**.

A-6. DETERMINATION OF SULPHATES

A-6.1 For Technical Grade

A-6.1.1 Reagents

A-6.1.1.1 *Concentrated hydrochloric acid* — see IS 265.

A-6.1.1.2 *Rectified spirit* — see IS 323.

A-6.1.1.3 *Glacial acetic acid* — see IS 695.

A-6.1.1.4 *Barium chloride solution* — approximately 12 percent (*m/v*).

A-6.1.1.5 *Dilute hydrochloric acid* — approximately 1 percent (*m/v*).

A-6.1.2 Procedure

A-6.1.2.1 Concentrate 200 ml of the prepared sample solution to about 50 ml. Add 10 ml of concentrated hydrochloric acid, 15 ml of rectified spirit and 35 ml of glacial acetic acid. Boil for 15 min. Dilute to about 200 ml. To the hot solution add 20 ml of hot barium chloride solution in a slow stream with constant stirring, to precipitate sulphate. Continue boiling for 15 min and allow to stand for 4 h, Filter the solution containing barium sulphate through a tared sintered glass crucible No. G4, Gooch crucible or a filter paper (Whatman No. 42 or equivalent). Wash with dilute hydrochloric acid till the filtrate is free from chromium salts and then with water till it is free from chlorides.

A-6.1.2.2 If the filtrate has been done through a sintered glass crucible, dry the precipitate at 110 ± 5 °C. If through Gooch crucible, dry the precipitate and ignite it over a burner or in the muffle furnace at 600 to 700 °C for 30 min. In case of filter paper, after drying, ashing shall be done on a low flame and the precipitate then ignited over a burner or in a muffle furnace at 600 to 700 °C for 30 min. Cool, add 2 drops of concentrated nitric acid and gently heat to drive off fumes. Ignite till constant mass is obtained.

A-6.1.3 Calculation

$$\text{Sulphates (as SO}_4\text{) percent by mass} = \frac{41.2 \times M_1}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material present in the aliquot.

A-6.2 For Pure and Analytical Reagent Grades

A-6.2.1 Reagents

A-6.2.1.1 *Barium chloride solution* — approximately 12 percent (m/v).

A-6.2.1.2 *Concentrated hydrochloric acid* — see IS 265.

A-6.2.1.3 *Rectified spirit* — see IS 323.

A-6.2.1.4 *Sodium carbonate* — solid.

A-6.2.1.5 *Ammonium hydroxide* — 20 percent (m/m).

A-6.2.2 Procedure

A-6.2.2.1 Take 250 ml of the prepared sample solution. Add 5 ml, of concentrated hydrochloric acid. Heat to boiling and add 25 ml of barium chloride solution. Digest on a steam bath for 2 h and let it stand overnight. If a precipitate is formed proceed as in **A-6.2.2.2**.

A-6.2.2.2 Filter, wash the precipitate with water till the washings are colourless and ignite. Fuse the residue in a platinum crucible with 1 g of sodium carbonate. Cool extract with hot water and filter. Add 5 ml of concentrated hydrochloric acid to the filtrate, dilute to 75 ml, heat to boiling, add 10 ml of rectified spirit and heat on the steam bath till the solution is clear green or colourless. Dilute to 100 ml and add about 10 ml of glacial acetic acid. Heat to boiling, add 10 ml of hot barium chloride solution, digest on the steam bath for 2 h and let it stand overnight. Filter, wash with hot water till the washings show no chlorides. Ignite, cool and weigh. Continue ignition till constant mass is obtained.

A-6.2.2.3 Carry out a blank test simultaneously.

A-6.2.3 Calculation

$$\text{Sulphates (as SO}_4\text{), percent by mass} = \frac{41.2 (M_1 - M_2)}{M}$$

where

M_1 = mass in g of the ignited residue obtained in test with the material,

M_2 = mass in g of the ignited residue obtained in the blank test, and

M = mass in g of the material present in the aliquot.

A-6.3 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at **A-11**.

A-7 DETERMINATION OF IRON AND ALUMINIUM OXIDES (R₂O₃)

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — see IS 265.

A-7.1.2 *Ammonium Hydroxide* — 20 percent (m/m)

A-7.1.3 *Ammonium Chloride* — solid.

A-7.2 Procedure

Take 25 ml of the prepared sample solution, add 2 ml of concentrated hydrochloric acid. Further add 4 g of ammonium chloride and 0.5 ml of ammonium hydroxide and boil. Filter, wash and ignite the precipitate. Cool and weigh till constant mass is obtained. Preserve the filtrate for determination of calcium in **A-8.2**.

A-7.3 Calculation

$$\text{R}_2\text{O}_3, \text{ percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass in g of the ignited precipitate, and

M = mass in g of the material present in the aliquot of the solution taken.

A-7.4 Alternative Method

Iron may aluminum alternatively be determined by instrumental test method as prescribed at **A-12**.

A-8 DETERMINATION OF CALCIUM**A-8.1 Reagents**

A-8.1.1 Ammonium Hydroxide — 20 percent (*m/m*).

A-8.1.2 Ammonium Oxalate Solution — 15 percent (*m/v*).

A-8.1.3 Concentrated Sulphuric Acid — see IS 266.

A-8.1.4 Dilute Hydrochloric Acid — approximately 1 N.

A-8.2 Procedure

Acidify the filtrate preserved in **A-7.2** with dilute hydrochloric acid to about 4 *pH*. Boil and add 10 ml of ammonium oxalate solution. Bring the *pH* to 7 by adding ammonium hydroxide. Allow the precipitate to settle for 4 h. Filter any precipitate present, wash it with water and ignite in a silica crucible. After cooling, add a few drops of concentrated sulphuric acid and reignite at 500 °C. Cool and weigh till constant mass is obtained.

A-8.3 Calculation

$$\text{Calcium (as Ca), percent by mass} = 100 \times \frac{0.294 \times M_1}{M}$$

where

M_1 = mass in g of calcium sulphate precipitate, and

M = mass in g of the material taken for test in **A-7.2**.

A-8.4 Alternative Method

Calcium may alternatively be determine by instrumental test method as prescribed at **A-12**.

A-9 DETERMINATION OF SODIUM

A-9.1 Qualitative Test

Dissolve 1 g of the material in 10 ml of water. Dip a clean platinum wire to the edge of a colourless Bunsen flame. When it is found to be colourless cool and dip it in the solution of the sample. Again, introduce it into the edge of the colourless Bunsen flame.

The presence of sodium radical is established if the characteristic yellow colour is imparted to the flame.

A-9.2 If sodium is present, estimate it on a suitable flame photometer at 589 mμ according to the directions of the manufacturer of the apparatus.

A-9.3 Alternative Method

Sodium may alternatively be determined by instrumental test method as prescribed at **A-11**.

A-10 DETERMINATION OF pH

A-10.1 Procedure

Dissolve 10 g of the material in 100 ml of carbon dioxide free water. Determine pH of the solution by means of a suitable pH meter using glass and calomel electrodes.

A-11 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-11.1 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, 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-11.2 Equipment

A-11.2.1 *Anion guard column* — a protector of the separator column.

A-11.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

A-11.2.3 *Anion Suppressor device*

Anion micro membrane suppressor is used to analyse the data Detector: Conductivity detector.

A-11.2 .4 Software

Software suitable for control of various operating parameters, receiving inputs and analysis of all data. 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-11.3 Reagents and Standards

A-11.3.1 *Glass or polyethylene sample bottles.*

A-11.3.2 *Distilled water or deionized water free from the anions of interest.*

A-11.3.3 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. Micromembrane suppressor solution (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

A-11.4 Standard solutions

A-11.4.1 Chloride — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-11.4.2 Sulphate — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

A-11.5 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-11.6 Procedure

Dissolve between 1 to 5 g 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 and deviation of retention force shall not exceed ± 10 percent of RT of calibration. 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 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-11.7 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-12 DETERMINATION OF ALUMINIUM, CALCIUM, IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-12.1 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-12.2 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.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

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)
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
viii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

A-12.3 Reagents and Solutions

A-12.3.1. Nitric acid (65 percent) Suprapure**A-12.3.2 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 Iron, calcium, and aluminium in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-12.3.3 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- 12.3.4 Sample preparation

Weigh about 2.5 g of the 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-12.3.5 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-12.4 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-12.5 Procedure**A-12.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-12.3.5). 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-12.5.2 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/or Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) 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-12.6 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).

A-13 DETERMINATION OF SODIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-13.1 The solution under analysis is passed with the help of a peristaltic pump through a nebulizer inside a spray chamber. The aerosol formed is aspirated in the form of argon plasma where the atoms are 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 wavelength/intensity of emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-13.2 Reagents and Solutions

A-13.2.1 *Magnesium nitrate, 6.67 percent (w/v)* — Add 66.7 g of magnesium nitrate hexahydrate in 1000 ml of deionized water and mix well.

A-13.2.2 *HCl solution* — Dilute 83.0 ml concentration HCl to 1000 ml deionized water.

A-13.2.3 *50 percent Nitric Acid* — Dilute 500 ml of Nitric Acid in 500 ml of deionized water.

A-13.3 Standards

A-13.3.1 *Yttrium Internal Standard (to infuse in the instrument T junction), 2 $\mu\text{g/ml}$*

Prepare 1 000 $\mu\text{g/ml}$ of Yttrium stock standard. Transfer 2 ml of this standard stock solution in 1 l volumetric flask and dilute the solution using 1 N HCl.

A-13.3.2 *Yttrium Working internal standard:*

Pipette out 25 ml of 1 000 µg/ml Yttrium stock standard into 50 ml volumetric flask and dilute using 1N HCl. If Yttrium Internal Standard is used to infuse in the instrument, then there is no need to add Yttrium during the preparation of calibration standards.

Sodium — Calibration standard 0 µg/ml- Add 0 µl of 10 000 µg/ml sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 6 µg/ml- Add 30 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 12 µg/ml- Add 60 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 24 µg/ml- Add 120 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium — Calibration standard 48 µg/ml- Add 240 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask — To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Similarly, calibration standard solutions can be prepared for iron and calcium analysis.

A-13.4 Sample Preparation

Process the sample until homogeneous.

A-13.5 Procedure (Sample Preparation)

Take sample in a platinum crucible. Add magnesium nitrate (2 ml) to the sample and gently swirl it. Place the sample in a cool muffle furnace with temperature less than 80 °C. Set the furnace program as:

Step 1 Ramp=3°C/min	Level=100°C	Dwell=360 min
Step 2 Ramp=3°C/min	Level=150°C	Dwell=60 min
Step 3 Ramp=3°C/min	Level=500°C	Dwell=480 min
Step 4 Ramp=end		

Else, heat the sample in the furnace gradually as per the above steps by controlling the sample through the thermostat.

The sample must not be heated so rapidly that it ignites. Remove the sample and cool the furnace at room temperature. Add 2 ml 50 percent aqueous HNO₃ and wash with it the sides of the crucible. Dissolve all the ash and transfer in the beaker. Remove excess of acid by slow heating of the sample on a hot plate. Make sure that the sample is free from any black precipitate. If any precipitate is found, again transfer the dry mass of the beaker into the crucible using little water and evaporate the water and heat at 500-550°C for 1 h. Again repeat this procedure till the sample becomes free from black precipitate. Remove the sample from furnace and cool it to room temperature. Add HCl (5 ml, 1 N) and dissolve the ash/mass. Transfer the above solution to 50 ml graduated flask and make up the final volume to 50 ml using 1 N HCl. Fortify the sample with 200 µl of Yttrium working standard and mix it. This step is omitted if T junction is used on the instrument. Analyse the sample using ICP/OES

A-13.6 Instrument Settings

Set up the instrument as per the manufacturer's instructions. Relative to the internal standard concentration, the quantification of the ions is done by the system software. Calibrate the instrument using ICP reagent blank and the calibration standard. Monitor the wavelengths for the respective metal

ions.

Na=589.592 nm, Pb=220nm, Fe=238.2 nm

A-13.7 Manual Calculation

Use linear regression analysis to determine a standard curve (emission vs. concentration)

$$\text{ppm (analyzed)} = \frac{\mu\text{g/ml (evaluated from calibration curve)} \times \text{ml (final volume)}}{\text{g (sample weight)}}$$

ANNEX B

(Clause 6.1)

SAMPLING OF POTASSIUM CHROMATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

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

B-1.4 Precautions shall be taken to protect the sample, 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 thoroughly by suitable means.

B-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers, on which the material has no action.

B-1.7 The sample containers shall be of such a size that they are almost but not completely filled with the sample.

B-1.8 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 the material of the same grade from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or different -batches of manufacture, the containers belonging to the same grade and 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 3.

Table 3 Number of Containers to be selected for Sampling

Lot Size N (1)	Number of Containers to be Selected n (2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and over	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 in one order as 1, 2, 3,..... 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 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 4.

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, the second for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite 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, the second 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 sample (*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.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of potassium chromate shall be conducted on each of the individual samples constituting set of the test sample.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

The mean and the range of the test results for the determination of potassium chromate 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.

B-5.1.1 If $(\bar{X} - 0.6 R)$ is equal to or greater than the minimum requirement of potassium chromate for the relevant grade, the lot shall be declared to have satisfied the requirement for potassium chromate for the relevant grade.

B-5.2 For Composite Samples

The test results on the composite sample shall meet the corresponding requirements specified in **4**.

B-5.3 A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristics listed in **4**.

B-5.4 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.