

भारतीय मानक मसौदा
अमोनियम ब्रोमाइड, शुद्ध और विश्लेषणात्मक अभिकर्मक —
विशिष्टि
(दूसरा पुनरीक्षण)

Draft Indian Standard
**Ammonium Bromide, Pure and Analytical Reagent—
Specification**
(*Second Revision*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1964 and subsequently revised in 1985. In the first revision, one more method for the determination of iron which is more sensitive and requires less reagents was incorporated as a referee method. Method for the determination of heavy metals was modified, and a new requirement of arsenic for analytical reagent grade was also specified.

In this revision, instrumental test methods for the determination of chlorides, sulphates, iron, lead and arsenic have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated. Further, Packing and Marking clause has been updated.

Ammonium bromide is used in the preparation of silver bromide in the chemical industry, in medicine and as an analytical reagent.

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

Draft Indian Standard
**AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT—
 SPECIFICATION**
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for ammonium bromide, pure and analytical reagent.

2 REFERENCES

The standards 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>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2088 : 2023	Methods for determination of arsenic (<i>second revision</i>)
IS 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>)
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>)

3 GRADES

The material shall be of two grades, namely, pure and analytical reagent.

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of a small colourless crystals or a white crystalline powder, free from foreign matter and visible impurities. It is slightly hygroscopic and slowly becomes yellowish in air and should be kept in well-closed containers.

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

Table 1 Requirements for Ammonium Bromide, Pure and Analytical Reagent

(Clauses 4.2, 5.2, 6.2.2, 6.3.1.1, A-5.3.1, A-6.2.1, A-8.3.1, A-9.2.4.1 and A-10.3.1)

Sl. No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Annex A)
		Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)

i)	Ammonium bromide, percent by mass of dried material, <i>Min</i>	98.0	99.5	A-2
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	—	0.005	A-3
iii)	<i>pH</i> of aqueous solution, <i>Min</i>	—	4.8	A-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.6	0.2	A-5 or A-17
v)	Bromates (as BrO ₃) percent by mass, <i>Max</i>	0.001	0.001	A-6
vi)	Iodides	—	To pass Test	A-7
vii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	0.000 5	A-8 or A-16
viii)	Iron (as Fe), percent by mass, <i>Max</i>	0.008	0.000 2	A-9 or A-16
ix)	Sulphates (as SO ₄) percent by mass, <i>Max</i>	0.06	0.01	A-10 or A-17
x)	Moisture, percent by mass, <i>Max</i>	1.0	0.5	A-11
xi)	Sulphated ash, percent by mass, <i>Max</i>	0.1	0.01	A-12
xii)	Arsenic (as As ₂ O ₃), ppm, <i>Max</i>	10	1	A-13 or A-16
xiii)	Tarry matter	To pass Test	—	A-14
xiv)	Barium	To pass Test	—	A-15

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean galvanized iron drums lined with polyethylene, or in glass bottles.

5.2 Marking

The containers shall be marked with the name and grade of the material; name of the manufacturer and recognized trade-mark, if any, mass of the material in the container; and batch number and the date of manufacture. The containers of the analytical reagent grade material shall also be labelled with the analytical data for the characteristics given in col 4 of Table 1.

5.2.1 BIS Certification Marking

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

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the methods of finding out 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 AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

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 AMMONIUM BROMIDE

A-2.1 Reagents

A-2.1.1 *Standard Silver Nitrate Solution* — 0.1 N.

A-2.1.2 *Concentrated Nitric Acid* — *see* IS 264.

A-2.1.3 *Nitrobenzene*

A-2.1.4 *Ferric Alum Indicator Solution* — Saturated.

A-2.1.5 *Standard Ammonium Thiocyanate Solution* — 0.1 N.

A-2.2 Procedure

Dry the material to constant mass at $(105 \pm 2) ^\circ\text{C}$. Weigh accurately about 0.4 g of the dried material and dissolve in 50 ml of water. Add with a pipette 50 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid, 15 ml of nitrobenzene and finally 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

A-2.3 Calculation

$$\text{Ammonium bromide (as NH}_4\text{ Br), percent by mass of the dried material} = \frac{0.9796(50 - V)}{M}$$

where

V = volume in ml of standard ammonium thiocyanate solution used in the titration, and

M = mass in g of the dried material taken for the test.

NOTE — Presence of chlorides increases the results for ammonium bromide and the results shall therefore be corrected. One gram of chloride (as Cl) is equivalent to 2.76 g of ammonium bromide.

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure

Accurately weigh about 20 g of the material and dissolve it in about 150 ml of water in a beaker. Cover the beaker and digest for one hour on a water-bath. Filter the solution through a tared filter paper or tared Gooch or sintered glass crucible (G. No. 4) and wash the residue thoroughly with water. Dry the filter paper or the crucible along with residue to constant mass at $105 ^\circ\text{C}$ to $110 ^\circ\text{C}$.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-4 DETERMINATION OF pH OF AQUEOUS SOLUTION

A-4.1 Procedure

Dissolve 5 g of the material in 100 ml of freshly boiled and cooled water (the water shall be protected with a soda lime tube while cooling). Determine the *pH* of the solution using a suitable *pH* meter.

A-5 TEST FOR CHLORIDES**A-5.1 Apparatus**

A-5.1.1 Nessler Cylinders — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 Dilute Nitric Acid — 1 : 2.

A-5.2.2 Hydrogen Peroxide

Dilute one volume of 30 percent hydrogen peroxide with one volume of water.

A-5.2.3 Concentrated Nitric Acid — Same as in **A-2.1.2**.

A-5.2.4 Silver Nitrate Solution — 10 percent (*m/v*).

A-5.2.5 Standard Chloride Solution

Dissolve 0.164 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the solution contains 0.01 mg of chloride (as Cl).

A-5.3 Procedure

Dissolve 0.5 g of the material in 15 ml of dilute nitric acid in a small conical flask. Add 6 ml of hydrogen peroxide and digest on a steam bath until the solution is colourless. Wash down the sides of the flask with water, digest again for 15 min, cool and dilute to 250 ml with water. Pipette out 10 ml of the solution into a Nessler cylinder, dilute to about 25 ml with water, add 1 ml of concentrated nitric acid and 0.5 ml of silver nitrate solution. Dilute to 50 ml and mix well. Carry out a control test in another Nessler cylinder using 8 ml of standard chloride solution for pure grade and 4 ml of standard chloride solution for analytical reagent grade and the same quantities of the other reagents.

A-5.3.1 The material shall be taken as not having exceeded the limit given 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-17**.

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

A-6.1.1 Potassium Iodide Solution — 10 percent.

A-6.1.2 Starch Solution

A-6.1.3 Dilute Sulphuric Acid — 1 : 15.

A-6.2 Procedure

Dissolve 1 g of the material in 10 ml of water. Add 2 drops of potassium iodide solution, 1 ml of starch solution and 5 drops of dilute sulphuric acid. Mix thoroughly and allow to stand for 10 min.

A-6.2.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if no blue colour is produced.

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

A-7.1.1 Carbon Tetrachloride or Chloroform

A-7.1.2 Ferric Chloride Solution — 10 percent.

A-7.1.3 Dilute Sulphuric Acid — 1 : 9.

A-7.2 Procedure

Dissolve 1 g of the material in 20 ml of water. Add 1 ml of chloroform or carbon tetrachloride, 3 drops of ferric chloride solution and 5 drops of dilute sulphuric acid. Shake the mixture vigorously.

A-7.2.1 The material shall be taken to have passed the test if no violet colour is produced in the chloroform or carbon tetrachloride layer.

A-8 TEST FOR HEAVY METALS

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 *p-Nitrophenol Indicator Solution*

Dissolve 0.2 g of *p*-nitrophenol in hot water and dilute to 100 ml.

A-8.2.2 *Dilute Ammonium Hydroxide* — 1 : 9.

A-8.2.3 *Dilute Acetic Acid* — 1: 99.

A-8.2.4 *Hydrogen Sulphide Solution* — Saturated and freshly prepared.

A-8.2.5 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-8.3 Procedure

Dissolve 2 g of the material in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator solution. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute acetic acid dropwise until the solution becomes colourless and then add 0.5 ml of the acid in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler cylinder using 2 ml of standard lead solution for pure grade and 1 ml for analytical reagent grade.

A-8.3.1 The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-8.4 Alternative Method

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-16** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-9 TEST FOR IRON

A-9.1 Two methods are prescribed for determining iron, namely, Method A and Method B. In case of dispute, Method B shall be the referee method.

A-9.2 Method A

A-9.2.1 *Outline of the Method*

Iron is determined colorimetrically using *o*-phenanthroline. This method requires strict control on the *pH* of the solution.

A-9.2.2 *Apparatus*

A-9.2.2.1 *Nessler cylinders* — 50 ml capacity.

A-9.2.3 *Reagents*

A-9.2.3.1 *p*-Nitrophenol indicator solution — same as in **A-8.2.1**.

A-9.2.3.2 Dilute ammonium hydroxide — 1: 9.

A-9.2.3.3 Dilute hydrochloric acid — 1 : 99.

A-9.2.3.4 Acetate buffer solution

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

A-9.2.3.5 *o*-Phenanthroline reagent

Thoroughly mix equal volumes of *o*-phenanthroline solution (0.1 percent aqueous), hydroxylamine hydrochloride solution (10 percent aqueous) and acetate buffer solution.

A-9.2.3.6 Standard iron solution

Dissolve 0.70 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-9.2.4 Procedure

Dissolve 1 g of the material in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless and then add 2 ml in excess. Add 5 ml of *o*-phenanthroline reagent, mix well and let it stand for 10 min. Dilute to 50 ml and mix well. Simultaneously carry out a control test using 8 ml of standard iron solution for pure grade and 0.2 ml of standard iron solution for analytical reagent grade.

A-9.2.4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-9.3 Method B

A-9.3.1 Outline of the Method

Iron is determined colorimetrically using thioglycollic acid in an alkaline medium. Unlike method A, strict control on pH is not required in this case and the method is equally sensitive.

A-9.3.2 Reagents

A-9.3.2.1 Standard iron solution — Prepare in the same manner as under **A-9.2.3.6**.

A-9.3.2.2 Thioglycollic acid

A-9.3.2.3 Concentrated ammonia — relative density 0.90.

A-9.3.2.4 Citric acid solution — 20 percent (*m/v*).

A-9.3.3 Procedure

Dissolve 1 g of the material in 25 ml of water in a Nessler cylinder. Add 10 ml of citric acid solution and five drops of thioglycollic acid, mix and make alkaline with ammonium hydroxide. Dilute with water to 50 ml mark and allow to stand for 5 min. Carry out a control test using 8 ml of standard iron solution for pure grade and 0.2 ml of standard iron solution for analytical grade.

A-9.3.3.1 The limit prescribed in Table I shall be taken as not having been exceeded, if the intensity of colour produced with the material is not greater than that produced in the control test.

A-9.4 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-16** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-10 TEST FOR SULPHATES

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50 ml capacity.

A-10.2 Reagents**A-10.2.1** *Standard Sulphate Solution*

Dissolve 0.148 g of anhydrous sodium sulphate in water and dilute to one litre in a volumetric flask. One millilitre of the solution contains 0.1 mg of sulphate (as SO₄).

A-10.2.2 *Dilute Hydrochloric Acid* — 1: 99.

A-10.2.3 *Barium Chloride Solution* — 10 percent.

A-10.3 Procedure

Dissolve 1 g of the material in 25 ml of water and transfer to a Nessler cylinder. In another Nessler cylinder carry out a control test using 3 ml of standard sulphate solution for pure grade and 1 ml of standard sulphate solution for analytical reagent grade and 25 ml of water. To each tube add 1 ml of dilute hydrochloric acid and 5 ml of barium chloride solution. Dilute the contents of each tube to the mark and stir well.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than produced in the control test.

A-10.4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at **A-17**.

A-11 DETERMINATION OF MOISTURE**A-11.1 Procedure**

Weigh accurately about 5 g of the material in a low-form glass-stoppered weighing bottle. Dry at (105 ± 2) °C for 4 h, cool in a desiccator and weigh.

A-11.2 Calculation

$$\text{Moisture, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = loss in mass in g on drying, and

M_2 = mass in g of the material taken for the test.

A-12 DETERMINATION OF SULPHATED ASH**A-12.1 Reagent**

A-12.1.1 *Concentrated Sulphuric Acid* — see IS 266.

A-12.2 Procedure

Weigh accurately about 10 g of the material in a platinum dish. Moisten with 3 drops of concentrated sulphuric acid, ignite gently and moisten again with 3 drops of concentrated sulphuric acid. Reignite to constant mass.

A-12.3 Calculation

$$\text{Sulphated ash, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-13 TEST FOR ARSENIC

A-13.1 Dissolve 1 g of the material in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088, using 0.01 mg of arsenic trioxide for pure grade and 0.001 mg of arsenic trioxide for analytical reagent grade for preparing the comparison stain.

A-13.2 Alternative Method

Arsenic may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-16** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-14 TEST FOR TARRY MATTER**A-14.1 Reagent****A-14.1.1 Citric Acid****A-14.2 Procedure**

Mix 5 g of the material in a dish with 15 ml of water and add 7 g of citric acid. Stir until all solids have dissolved.

A-14.2.1 The material shall be taken to have passed the test if no tarry odour is observed.

A-15 TEST FOR BARIUM**A-15.1 Reagents**

A-15.1.1 Dilute Sulphuric Acid — approximately 4 N.

A-15.2 Procedure

Dissolve 0.5 g of the material in 10 ml of water and add 1 ml of dilute sulphuric acid.

A-15.2.1 The material shall be taken to have passed the test if no turbidity is produced within 5 min.

A-16 DETERMINATION OF ARSENIC, IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-16.1 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-16.2 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

(Clauses A-16.2 and A-16.4)

SI No.	Element	Wavelength	Approximately Achievable Limits	Interfering Elements
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(1)	(2)	(nm) (3)	Radial Viewing (µg) (4)	Axial Viewing (µg) (5)	(6)
i)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)		Cr, Fe
		217.00		(20)	

A-16.3 Reagents and Solutions

A-16.3.1. Nitric Acid (65 percent) Suprapure

A-16.3.2 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 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-16.3.3 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-16.3.4 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-16.3.5 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-16.4 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-16.5 Procedure

A-16.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-16.3.5). 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-16.5.2 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, arsenic) 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-16.6 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-17 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-17.1 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-17.2 Equipment

A-17.2.1 Anion Guard Column — a protector of the separator column

A-17.2.2 Anion Separator Column — suitable for selective separation of ions under analysis.

A-17.2.3 Anion Suppressor Device

Anion micro membrane suppressor is used to analyse the data

A-17.2.4 *Detector* — conductivity detector

A-17.2.5 *Software*

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

A-17.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-17.3 Reagents

A-17.3.1 *Glass or Polyethylene Sample Bottles.*

A-17.3.2 *Distilled Water or Deionized Water free from the Anions of interest.*

A-17.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litre of water.

A-17.3.4 *Micro membrane suppressor solution: (0.025 N of sulphuric acid)*

Dilute 2.8 ml of concentrated sulphuric acid in 4 litre of water

A-17.4 Standard solutions

A-17.4.1 *Chloride*

Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water

A-17.4.1 *Sulphate*

Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water

A-17.5 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 ml to 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-17.6 Procedure

Dissolve between 1 g to 5 g samples in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 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-17.7 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.

ANNEX B

(Clause 6.1)

PREPARATION OF TEST SAMPLES OF AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 The sampling instrument and the containers for samples shall be such that the material has no action on them. They shall be clean and dry.

B-1.3 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and, as far as possible, the material shall also be protected from light.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In a single consignment of one grade of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of one grade of the material is known to consist of different batches of manufacture or 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 this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 3.

Table 3 Scale of Sampling

(Clause B-2.2)

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

NOTE — In the case of very small lots where the selection of 3 containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection random number tables shall be used. In case the tables are not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and 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 r^{th} container thus counted shall be withdrawn to give sample for tests.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a small representative portion of the material of about 100 g shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of the material (not more than 90 g) shall be taken and mixed thoroughly to form a composite sample of about 250 g. The composite samples shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

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

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

B-4 NUMBER OF TESTS

B-4.1 Test for the determination of ammonium bromide shall be performed on each of the individual samples (*see B-3.3*).

B-4.2 Tests for the determination of all other characteristics given in Table 1 shall be performed on the composite sample (*see B-3.2*).

B-5 CRITERIA FOR CONFORMITY

B-5.1 *For Individual Samples*

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

B-5.1.1 The lot shall be declared as conforming to the requirements for ammonium bromide if the value of $(\bar{X} - 0.6R)$ as calculated from the test results is equal to or greater than the corresponding value given for the relevant grade in Table 1.

B-5.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified.