IS : 10332 - 1982
(Reaffirmed 2003)
Edition 1.4
(2004-10)

Indian Standard

SPECIFICATION FOR HYDROFLUORIC ACID, AQUEOUS

(Incorporating Amendment Nos. 1, 2, 3 & 4)

UDC 661.487

B U R E A U O F I N D I A N S T A N D A R D S
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 8

Indian Standard

SPECIFICATION FOR HYDROFLUORIC ACID, AQUEOUS

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Indian Standard

SPECIFICATION FOR HYDROFLUORIC ACID, AQUEOUS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 October 1982, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Aqueous hydrofluoric acid as the name indicates, is a water solution of hydrogen fluoride available commercially in concentration varying between 30 and 80 percent HF content. It is a colourless corrosive liquid which fumes at concentrations above 48 percent HF. However, it is not required to be stored or transported in pressure containers.

0.3 Aqueous hydrofluoric acid finds wide application. It is used in the manufacture of a large number of inorganic fluorine chemicals like fluorides, bifluorides, silica fluorides, fluoborates, etc. It is also used in ceramics and enamel industry, etching and frosting of glassware, electroplating and electropolishing, fluorination and fluoridation work, metal cleaning and pickling. Other major uses are in ore floatation process, oil-well acidizing, manufacture of microelectrical circuits, semi-conductor grade silicon metal, and in analytical work.

0.4 Aqueous hydrofluoric acid, technical is available in different concentrations, namely, 30, 40, 50, 60, 70 and 80. The physical constants of different dilutions are given below for information:

Hydrofluoric Acid Concentration	*Relative Density at 0°C*	*Boiling Point* (± 1.0°C)	*Freezing Point* (± 0.5°C)
30	1.120	110.1	– 70.2
40	1.160	112.2	– 48.2
50	1.200	106.1	– 36.4
60	1.235	87.0	– 41.7
70	1.258	66.6	– 70.18
80	1.259	48.3	– 101

0.5 This edition 1.4 incorporates Amendment No. 1 (March 1984), Amendment No. 2 (January 1998), Amendment No. 3 (December 1999) and Amendment No. 4 (October 2004). Side bar indicates modification of the text as the result of incorporation of the amendments.

0.6 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.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for aqueous hydrofluoric acid for use in fluorine based chemical industries, for analytical work and for use in electronic industry.

2. GRADES

2.1 There shall be three grades of the material, namely:

a) *Technical* (Tech),

b) *Analytical Reagent* (AR), and

c) *Electronic* (EL).

2.1.1 The technical grade material shall be further of three sub-grades corresponding to different concentrations as follows:

Sub grade A . . 40 percent

Sub grade B . . 60 ,,

Sub grade C . . 70 ,,

3. REQUIREMENTS

3.1 The material shall be a clear colourless liquid without sediment.

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

4. PACKING AND MARKING

4.1 Packing

4.1.1 Aqueous hydrofluoric acid technical grade shall be packed in polyethylene lined steel drums. An outage or space is provided above the liquid level to compensate for liquid expansion as temperature increases.

In case of acid between 60 percent and 70 percent strength, the filling ratio is kept at 0.85.

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

TABLE 1 REQUIREMENTS FOR AQUEOUS HYDROFLUORIC ACID

(*Clause* 3.2)

SL NO.	CHARACTERISTIC	REQUIREMENT FOR GRADE			METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Tech	AR	EL	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Total acidity (as HF), percent by mass	a) 40.0 (*Min*) b) 60.0 (*Min*) c) 70.0 (*Min*)	40.0 (*Min*)	50.0 (*Min*)	A-2
ii)	Residue on ignition parts per million, *Max*	a) 50.0 b) 75.0 c) 100.0	10.0	5.0	A-3
iii)	Hydroflusilicic acid (as H_2SiF_6), percent by mass, *Max*	a) 0.02 b) 0.03 c) 0.04	0.50	100 (ppm)	A-4 or A-15 (*see* Note 1)
iv)	Heavy metals (as Pb), ppm, *Max*	—	1.0	0.1	A-5
v)	Arsenic (as As), ppm, *Max*	—	0.1	0.05	A-6
vi)	Iron (as Fe), ppm, *Max*	—	0.5	0.05	A-7
vii)	Potassium (as K), ppm, *Max*	—	—	1.0	A-8
viii)	Sodium (as Na), ppm, *Max*	—	—	1.0	A-9
ix)	Calcium (as Ca), ppm, *Max*	—	—	1.0	A-10
x)	Chloride (as Cl), ppm, *Max*	—	10.0	5.0	A-11
xi)	Phosphate (as PO_4), ppm, *Max*	—	10.0	0.5	A-12
xii)	Sulphite (as SO_3), ppm, *Max*	—	5.0	2.0	A-13
xiii)	Sulphate (as H_2SO_4), ppm, *Max*	a) 0.40 b) 0.60 c) 0.70	5.0	1.0	A-14

NOTE 1 — In case of dispute volumetric method prescribed in **A-4** shall be the referee method.

4.1.2 AR grade and EL grade shall be packed in high density poly-ethylene bottles of 500 ml and 1 litre capacity with screwed cap and also an inner lid for safety. The bottle shall be further packed in suitable wooden boxes providing adequate packing medium inside the box to keep the containers in upright position.

4.1.3 For details regarding unloading of the material and other safety precautions, reference shall be made to IS : 5184-1969*.

4.2 Marking

4.2.1 The containers and also packages of aqueous hydrofluoric acid shall be suitably marked in red letters not less than 25 mm high (in case of packages and bulk containers) showing:

a) Name of the acid;

b) Manufacturer's name and/or recognized trade-mark, if any;

c) Grade and mass of the material; and

d) Year of manufacture.

4.2.2 The containers shall prominently display the cautionary notice as in **6.2** of IS : 5184-1969*.

HYDROFLUORIC ACID, AQUEOUS

DANGER: EXTREMELY HAZARDOUS

LIQUID AND VAPOUR CAUSES SEVERE BURNS WHICH MAY NOT BE IMMEDIATELY PAINFUL OR VISIBLE

'DO NOT GET IN EYES, ON SKIN AND ON CLOTHING'

DO NOT BREATHE VAPOUR

STORE AWAY FROM SUN AND ALSO FROM DIRECT HEAT.

4.2.3 The packages shall be labelled as shown in Fig. 7 of IS : 1260 (Part I)-1973†.

*Code of safety for hydrofluoric acid.

†Pictorial markings for handling and labelling of goods : Part I Dangerous goods (*first revision*).

4.2.4 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. The ISI 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 which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. 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.

5. SAMPLING

5.1 General Requirements of Sampling

5.1.1 In drawing samples, precautions shall be taken to protect the samples, the material being sampled, sampling equipment and the sample containers from contaminations.

5.1.1.1 While drawing and analyzing the samples, precautions should be taken to protect the person from special hazards associated with the handling of the acid (*see also* IS : 5184-1969*).

5.1.2 To draw a representative sample the contents of each of the selected containers shall be mixed thoroughly by shaking or stirring by suitable means and with caution.

5.1.3 The samples shall be taken in suitable, clean, dry air-tight containers, of high density polyethylene make keeping necessary space above the sample.

5.1.4 Each sample container shall be sealed air-tight after filling and shall be marked with full details of sampling, the date of sampling, and the year of manufacture of the material.

5.2 Scale of Sampling

5.2.1 *Lot* — All the containers in a single consignment of the material of same grade shall constitute a lot.

5.2.2 Samples from each lot shall be tested separately for judging the conformity of the material to the requirements of the specification. The number of containers to be selected from lots of different sizes shall be as per Table 2.

*Code of safety for hydrofluoric acid.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* 5.2.2)

LOT SIZE (*N*)	NO. OF CONTAINERS TO BE SELECTED (*n*)
Up to 20	2
21 to 30	3
31 to 50	4
51 to 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 and above	9

5.3 The containers for sampling, shall be selected at random from the lot. Procedure given in IS : 4905-1968* may be followed to ensure the randomness of selection.

5.4 Preparation of Laboratory Sample

5.4.1 Test samples obtained from the material with 48-50 percent strength and below do not require any dilution with water for carrying out the tests and can be used directly as laboratory sample. But test samples with strength above 50 percent require dilution with water.

5.4.2 *Preparation of Laboratory Sample for Acid Containing More Than 50 Percent of Total Acidity (as HF)*.

5.4.2.1 *Procedure* — Weigh to the nearest 0.01 g, 30 to 35 g of mixture of ice and water into a tared polyethylene bottle of 250 ml capacity provided with screwed cap with mark at 100 ml. Fill the sample bottle carefully to the mark with the test sample; cool, if necessary, and reweigh. Agitate the bottle to mix the acid thoroughly.

5.4.2.2 *Calculation of concentration, that is, dilution factor* — Concentration of the dilute hydrofluoric acid solution expressed as g of test sample per 100 g of the solution is given by the formula:

$$\frac{M_1}{M_1 + M_2} \times 100$$

where

M_1 = mass in g of test sample, and

M_2 = mass in g of ice and water.

*Methods for random sampling.

5.5 Test Samples

5.5.1 Two sets of test samples of 1 000 g each shall be prepared from the composite sample obtained from each lot. These samples shall be placed in a clean dry PE bottles with screwed-stopper closed tightly, sealed and labelled with all the particulars of sampling.

5.5.2 The supplier shall retain one set of sealed sample and the second set of sample bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR AQUEOUS HYDROFLUORIC ACID

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

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

A-2. DETERMINATION OF TOTAL ACIDITY AS HYDROFLUORIC ACID

A-2.0 Principle — Titration of a test portion with standard sodium hydroxide solution using phenolphthalein as indicator.

A-2.1 Reagents

A-2.1.1 *Standard Sodium Hydroxide Solution* — 1 N and 0.1 N.

A-2.1.2 *Phenolphthalein Indicator Solution* — Dissolve 1 g of phenolphthalein in 100 ml of (95 percent *v/v*) ethanol. Add standard sodium hydroxide solution (0.1 N) drop by drop till very faint pink colouration.

A-2.2 Procedure — Weigh to the nearest 0.001 g about 2 g of the sample in a tared 100 ml polyethylene bottle with lid. Transfer quantitatively to a 250-ml polyethylene beaker, containing about 50 ml of water. Add 5 drops of phenolphthalein solution, titrate with sodium hydroxide solution (1 N) until the end point is approached. Complete the titration with sodium hydroxide solution, 0.1 N, to the appearance of a faint pink colour.

*Specification for water for general laboratory use (*second revision*).

A-2.3 Calculation

$$\text{Total acidity (as HF), percent by mass} = \frac{0.200\,1 \times (10\,V_1 + V_2)}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution (1 N) used in titration,

V_2 = volume in ml of standard sodium hydroxide solution (0.1 N) used to complete the titration,

M = mass in g of the test sample, and

0.200 1 = factor corresponding to mass in g of hydrofluoric acid per ml of 0.1 N sodium hydroxide solution.

A-3. DETERMINATION OF RESIDUE ON IGNITION

A-3.0 Principle — Evaporation of the sample to dryness on a water bath and ignition of the dried sample at 800°C.

A-3.1 Procedure — Weigh to the nearest 0.1 g about 400 g of the sample in a 500-ml polyethylene bottle with screwed cap. Transfer carefully about 60 ml of the sample into the pre-weighed platinum dish kept on a water bath and evaporate the mass to a small bulk in a fume cup-board. Add some more volume of the material from the bottle into the dish and evaporate as above. Transfer in this way the sample quantitatively into the dish and evaporate the mass to dryness. Place the dish in a furnace at a temperature of 800°C and remove it from the furnace after 30 minutes. Allow the dish to cool in a desiccator and reweigh.

A-3.2 Calculation

$$\text{Residue on ignition, ppm by mass} = \frac{(M_1 - M_2)}{M} \times 10^6$$

where

M_1 = mass in g of platinum dish with residue,

M_2 = mass in g of platinum dish alone, and

M = mass in g of the test portion.

A-4. DETERMINATION OF HYDROFLUOSILICIC ACID (H_2SiF_6)

A-4.0 Principle — Evaporation of the solution to dryness in presence of sodium chloride. Dilution with water and tiration of the ice-cold solution in presence of potassium chloride with standard sodium hydroxide solution, using phenolphthalein as an indicator followed by titration after heating.

A-4.1 Reagents

A-4.1.1 *Sodium Chloride* — 5 percent aqueous solution.

A-4.1.2 *Potassium Chloride* — 10 percent aqueous solution.

A-4.1.3 *Standard Sodium Hydroxide Solution* — 1 N.

A-4.1.4 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-4.1.5 *Phenolphthalein Indicator Solution* — 10 g/l in ethanol.

A-4.2 Procedure — Weigh accurately about 5 g of the test sample into a polyethylene weighing bottle and transfer quantitatively into the platinum dish. Add 5 ml of sodium chloride solution stir well and evaporate the solution to dryness on a water bath. Add 20 ml of potassium chloride solution and wash the inside of the dish with about 20 ml of water, stir to mix thoroughly. Add 3-4 drops of phenolphthalein solution. Cool the dish from outside with ice to cool the solution below 10°C and while keeping the solution ice-cold neutralize first with sodium hydroxide solution 1 N until the end-point is approached. Complete the neutralization with sodium hydroxide solution (0.1 N) to the appearance of a faint pink colour. Transfer the contents quantitatively to a 400 ml glass beaker, heat the solution to boil and titrate immediately with sodium hydroxide solution (0.1 N) to the appearance of a faint pink colour.

A-4.3 Calculation — Calculate hydrofluosilicic acid (H_2SiF_6) as parts per million, by mass by the formula:

$$0.003\,603 \times V \times \frac{10^6}{M}$$

where

V = volume in ml of standard sodium hydroxide solution (0.1 N) used in hot titration,

M = mass in g of the test portion, and

0.003 603 = factor corresponding to g of Hydrofluosilicic (H_2SiF_6) per 1 ml of sodium hydroxide solution.

A-5. DETERMINATION OF HEAVY METALS (as Pb)

A-5.1 Colorimetric Method

A-5.1.0 *Principle* — Evaporation of the sample to dryness. Acidifying with hydrochloric acid and formation of lead sulphide coloured solution with hydrogen sulphide. Measurement of the intensity of the coloured solution.

A-5.2 Reagents

A-5.2.1 *Dilute Hydrochloric Acid* — 20 percent (*v/v*).

A-5.2.2 *Sodium Acetate Solution* — 20 percent (*m/v*).

A-5.2.3 *Acetic Acid Solution* — 20 percent (*v/v*).

A-5.2.4 *Hydrogen Sulphide Water* — Saturated solution of the gas.

A-5.2.5 *Standard Lead Solution* — Weigh accurately 1.831 g of lead acetate $(CH_3.COO)_2Pb.3H_2O$. Dissolve in small volume of water and transfer quantitatively in a 1-litre measuring flask. Dilute to the mark and mix. 1 ml of this solution contains 1 milligram of lead.

A-5.2.6 *Dilute Lead Standard Solution* — Transfer 10 ml of the lead solution (**A-5.2.5**) into a 1-litre measuring flask. Dilute to the mark and mix. One ml of this solution contains 10 micrograms of lead.

A-5.3 Apparatus

A-5.3.1 *Platinum Dish* — 100-ml.

A-5.3.2 *Nessler Cylinders* — 100-ml capacity.

A-5.4 Procedure

A-5.4.1 Weigh accurately about 250 g of the test sample in a poly-ethylene weighing bottle of 500 ml capacity with screwable cap. Transfer about 60 ml of the sample into a platinum dish kept on a water bath and evaporate to a small bulk. Transfer some more volume of the sample and evaporate again. Thus evaporate the total mass of the sample to dryness.

A-5.4.2 Add 1 ml of dilute hydrochloric acid to the residue in the dish and evaporate nearly to dryness on a waterbath.

A-5.4.3 Add about 10 ml of water to the dish and transfer the mass quantitatively into a 25-ml measuring flask. Dilute to the mark and mix. Transfer the solution to a Nessler cylinder with stopper.

A-5.4.4 Add 3 ml of sodium acetate solution and 0.3 ml of acetic acid into the tube and mix thoroughly.

A-5.4.5 Add 10 ml of hydrogen sulphide water, mix, and compare the brown colour developed with that of the standard lead solution processed in the same manner by viewing the depth of the solution from above.

A-5.4.6 Note the concentration of the standard lead solution, the brown colour of which matches the most with that of the sample solution.

A-5.4.7 *Treatment of Standard Lead Solution* — Add by, means of a graduated pipette 10 ml of hydrochloric acid (**A-5.2.1**) to a platinum dish and evaporate to dryness on a water bath. Add exactly 20 ml of water to the dish to dissolve the residue and mix. Transfer 2 ml of water from the dish into each of the six numbered 25 ml measuring flasks followed by addition of 1, 2, 3, 4, 5 and 6 ml of the standard lead solution in series. Dilute to the mark and mix. Transfer the solutions in sequence to the six numbered Nessler cylinders. Proceed exactly, as mentioned in **A-5.4.4** and **A-5.4.5** above. Match the brown colouration developed in the standard solution with that of the sample solution.

A-5.4.8 *Calculation*

$$\text{Heavy metals (as Pb), parts per million} = \frac{a}{10^6} \times \frac{10^6}{m} = a/m$$

where

a = mass of metal in micrograms in the standard lead solution, the colour of which matches with that of sample solution; and

m = mass in g of the test solution.

A-5.5 Spectrophotometric Method (Alternative Method)

A-5.5.0 *Principle* — Lead reacts with diphenylthiocarbazone to form a pink coloured complex in a chloroform or carbon tetrachloride solution. The complex is separated by extraction with $CHCl_3$ or CCl_4 from an aqueous, ammonium-citrate-cyanide solution. Photometric measurement is made at 520 nm.

A-5.6 Reagents

A-5.6.1 *Standard Lead Solution* — Dissolve 0.799 2 g of Pb $(NO_3)_2$ in water and make the volume to 500 ml in a volumetric flask. One ml of this solution is equivalent to 1 mg of lead.

A-5.6.2 *Dilute Lead Standard Solution* — Transfer 10 ml of the lead solution (**A-6.6.1**) into a 100 ml volumetric flask. Dilute to the mark and mix. One ml of this solution contains 0.1 mg of lead.

A-5.6.3 *Citric Acid Solution* — 50 percent (*m*/*v*).

A-5.6.4 *Potassium Cyanide* — 10 percent (*m*/*v*).

A-5.6.5 *Ammonium Hydroxide Solution* — (1 : 1) *v*/*v*.

A-5.6.6 *Bromothymol Blue Indicator* — Dissolve 0.1 g of the dye in 8 ml of 0.02 N sodium hydroxide solution and dilute to 250 ml with water.

A-5.6.7 *Dilute Nitric Acid Solution* — 1 percent (*v*/*v*).

A-5.6.8 *Dithizone* — 10 mg/l and 5 mg/l in chloroform/carbon tetrachloride.

A-5.7 Apparatus

A-5.7.1 *Platinum Dish* — 100 ml.

A-5.7.2 *Separating Funnel*

A-5.7.3 *Measuring Flask* — 50 ml and 100 ml.

A-5.8 Procedure

A-5.8.1 Weigh accurately about 250 g of the test sample in a polyethylene weighing bottle of 500 ml capacity with screwed cap. Transfer about 50 ml of the sample into a platinum dish kept on a water bath and evaporate to a small bulk. Transfer some more volume of the sample and evaporate again. Thus evaporate the total mass of the sample to dryness. Add 10 ml of concentrated nitric acid and 25 ml of perchloric acid and 50 ml of water. Evaporate the mass to fumes, cool, add about 50 ml water and filter through Whatman No. 42 filter paper. Collect the filtrate in 100 ml measuring flask, wash with water and make the volume of the filtrate to 100 ml and mix thoroughly.

A-5.8.2 Take 25 ml of the filtrate in separating funnel (*A*) add 10 ml 50 percent citric acid and neutralize (*p*H = 7) with 1 : 1 ammonia using bromothymol blue indicator, cool, then add 10 ml of 10 percent potassium cyanide and 15 ml (10 mg/l) of dithizone and extract to separate lead. Transfer the lower layer in another funnel (*B*). Add 15 ml (5 mg/l) of dithizone in funnel (*A*) and extract. Collect the dithizone layer in funnel (*B*). Repeat the process till colour with dithizone is obtained (means all the lead is extracted).

A-5.8.3 The dithizone-chloroform extraction solution is run directly into 25 ml of 1 percent nitric acid in a small separating funnel and shake to extract the lead from the dithizone into the nitric acid solution. The green dithizone layer is drawn off into other separatory funnel, also containing 25 ml of 1 percent nitric acid and well shaken. The chloroform layer is discharged. The solution is filtered through a wet cotton plug in a funnel stem into a 50 ml volumetric flask. The volume is made to the mark if necessary with a few drops of 1 percent nitric acid.

A-5.8.4 The 50 ml nitric acid solution containing lead or a suitable aliquot of it is poured into a small separatory funnel. (If an aliquot is used, the volume is made to 50 ml with 1 percent nitric acid). Add 10 ml of ammoniacal potassium cyanide solution and mix well. The lead is immediately extracted with two successive 20 ml portion of standard dithizone solution in chloroform (5 mg/l). The chloroform extracts are combined and absorbance is measured at 520 nm against chloroform in the reference cell.

Carry out a blank by following the same procedure but omitting the sample.

A-5.8.5 Following the same procedure for standard solution of lead one for high concentration and second for low concentration and take reading on calorimeter. Deduct the blank value from the sample result and standard lead solutions result obtained and calculate lead as follows.

A-5.9 Calculation

$$\text{mg Pb} = \frac{SR - LSR}{HSR - LSR} \times (HS - LS) + LS$$

where

SR = sample reading,

LSR = lower standard reading,

HSR = higher standard reading,

HS = higher standard lead, and

LS = lower standard lead.

Therefore, lead (as Pb), percent by mass = $\frac{\text{mg Pb} \times 0.4}{M}$

where

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

A-6. DETERMINATION OF ARSENIC (As)

A-6.1 Procedure — Determine arsenic by silver diethyl dithiocarbomate method as prescribed in **5.2** of IS : 2088-1971*, after initial treatment of the material with concentrated sulphuric acid.

*Method for determination of arsenic (*first revision*).

A-7. DETERMINATION OF IRON (Fe)

A-7.0 Principle — Evaporation of test portion to dryness to remove fluorine. Preliminary reduction of iron by means of hydroxyl ammonium chloride. Formation of the complex iron with 1 : 10 phenanthroline in buffered medium (*p*H 3.5 to 4.2). Photometric measurement of colour intensity at 510 nm wavelength.

A-7.1 Reagents

A-7.1.1 *Dilute Hydrochloric Acid* — 10 percent (*v/v*).

A-7.1.2 *Hydroxyl Ammonium Chloride* ($NH_2OH.HCl$) *Solution* — 10 percent (*m/v*).

A-7.1.3 *1:10 Phenanthroline Hydrochloride Solution* — 0.5 percent. Add 0.5 g of 1:10 phenanthroline hydrochloride, monohydrate ($C_{12}H_8N_2.HCl.H_2O$) in little water and warm the solution to dissolve the salt. Cool and dilute to 100 ml, keep in the dark and discard if any colour develops subsequently.

A-7.1.4 *Buffer Solution* — Dissolve 136 g of sodium acetate, trihydrate in about 250 ml of water. Add 120 ml of glacial acetic acid approximately 17.4 N and dilute to 500 ml.

A-7.1.5 *Standard Iron Solution* — Weigh accurately 0.702 g of ferrous ammonium sulphate and dissolve in 10 ml of dilute sulphuric acid. Transfer quantitatively to a 1-litre measuring flask, dilute to the mark and mix. One ml of this solution contains 100 microgram (as Fe).

A-7.1.6 *Standard Iron Solution* — Transfer 50 ml of standard solution (**A-7.1.5**) to a 500-ml measuring flask, dilute to the mark with water and mix. One ml of this solution contains 10 micrograms of iron (as Fe).

A-7.2 Apparatus

A-7.2.1 *Platinum Dish* — 100 ml capacity.

A-7.2.2 p*H Meter with Glass Electrode*

A-7.2.3 *Photo-Electric Absorptiometer*

A-7.2.4 *Ordinary Laboratory Apparatus*

A-7.3 Procedure

A-7.3.1 Weigh accurately about 250 g of test sample in polyethylens bottle with screwable cap. Transfer carefully about 60 ml of the same sample into a platinum dish and evaporate on water bath to a small volume. Transfer some more volume of the sample from the bottle and evaporate again to a small bulk. Evaporate in this manner the

complete mass of the sample from the bottle to dryness in the platinum dish. Add 3 ml of dilute hydrochloric acid and heat to dissolve the residue. Add little water and cool. Transfer the solution quantitatively to a 25 ml measuring flask. Add 1 ml of hydroxyl ammonium chloride solution about 5 ml of buffer solution. Add 1 ml of 1:10 phenanthroline solution dilute to the mark, shake well and allow to stand for 30 minutes. Transfer the solution to the cell of photoelectric absorptiometer and measure the absorbancy at a wavelength of approximately 510 nm, after adjusting the instrument to zero absorbancy against water as reference solution.

NOTE — Quantity of buffer solution should be such so as to adjust the *p*H of the solution to about 4.

A-7.3.2 *Blank Test* — Carry out a blank test using exactly the same procedure (**A-8.3.1**) and the same quantities of all reagents but omitting the sample.

A-7.3.3 *Preparation of Calibration Graph* — Into a series of six 25 ml measuring flasks, transfer 1, 2, 3, 4, 6, 8 and 10 ml of standard iron solution (**A-7.1.6**). Add 1 ml of hydroxyl ammonium chloride solution, 5 ml of buffer solution. Add 1 ml of 1:10 phenolphthalein solution, dilute to the mark, shake well, allow to stand for 30 minutes and measure the absorbancy of the solution as mentioned in **A-8.3.1**. Proceed exactly as mentioned in **A-7.3.1** and measure the absorbancy of the standard solution on the photoelectric absorptiometer. Plot a graph having the quantities in micrograms of iron (Fe), in the standard solutions as abscissae and the corresponding absorbance values as the ordinates.

A-7.3.4 *Calculation* — Determine the quantities of iron (Fe) corresponding to the absorbance reading of the test sample solution and also that of the blank test solution by means of the graph.

$$\text{Iron (as Fe), parts per million} = \frac{(M_1 - M_2)}{10^3} \times \frac{1\,000}{M} = (M_1 - M_2)/M$$

where

M_1 = mass in micrograms of iron (as Fe) in the test solution,

M_2 = mass in micrograms of iron (as Fe) in the blank test solution, and

M = mass in g of the test sample.

A-8. DETERMINATION OF POTASSIUM (as *K*)

A-8.0 Principle — Evaporation to dryness for removal of fluorine. Dissolution of residue in water. Solution drawn into a non-luminous flame of the flame photometer with a suitable filter to impart a colour characteristic of the radical. Measurement of intensity of the colour on the instrument.

A-8.1 Reagents

A-8.1.1 *Potassium Chloride Powder*

A-8.1.2 *Standard Potassium Solution* — Weigh accurately 0.190 7 g of potassium chloride (KCl), dissolve in little water and transfer quantitatively into a 1-litre standard flask. Dilute to the mark with water and mix thoroughly. One ml of this solution contains 100 micrograms of potassium (as *K*).

8.1.3 *Dilute Standard Potassium Solution* — Transfer 50 ml of standard solution (**A-8.1.2**) in to a 500-ml measuring flask and dilute to the mark with water. One ml of this solution contains 10 micrograms of potassium (as *K*).

A-8.2 Apparatus

A-8.2.1 *Platinum Dish* — 100 ml capacity.

A-8.2.2 *Flame Photometer with a Filter for Potassium*

A-8.3 Procedure

A-8.3.1 Weigh accurately about 300 g of test sample in a polyethylene bottle with screwable cap. Transfer about 60-70 ml of the sample into a platinum dish and evaporate to a small volume on water bath. Again transfer about 50-60 ml of the sample into the dish and evaporate to a small bulk. Transfer in this manner the complete mass of sample to the platinum dish and evaporate to dryness. Add little water to the dish and warm to dissolve the residue completely. Transfer quantitatively to a 100 ml measuring flask, dilute with water to the mark and mix. Switch on the flame photometer, unclamp the galvanometer, open the gas control and light on the flame. Turn on air supply and adjust the air-control to a pressure of 68 947.6 pascals. Slide a beaker of water into the sample recess and slowly close the gas control until separate blue cones of flame are formed. Set the galvanometer to zero by 'zero' control against distilled water fix the appropriate filter (*K*) in position. Replace water sample by a potassium standard solution of higher concentration high standard solution (**A-8.3.2.1**) corresponding to a reading to 100 on the scale. Adjust sensitivity control to give approximately full scale deflection set at 90 percent. Set the galvanometer scale to zero again, using the distilled water. Replace distilled water by a potassium standard solution of low concentration — low standard solution

(**A-8.3.2.2**) and note the galvanometer scale reading. Again set the galvanometer scale to zero against distilled water. Replace distilled water beaker by the beaker containing sample solution and note down the reading on the galvanometer scale.

NOTE — Always take care to rinse the atomizer system with distilled water after taking each reading. Also check the zero and 100 reading of the scale in between the readings. Always light the gas first keeping the air flow closed and then set the luminasity of the flame by adjusting the air flow.

A-8.3.2 *Preparation of High Standard and Low Standard Solutions of Potas*sium

A-8.3.2.1 *High standard solution* — Transfer 5 ml of potassium standard solution (**A-8.1.3**) into a 100 ml measuring flask, dilute with water to the mark and mix. This solution contains 50 micrograms of potassium (as *K*).

A-8.3.2.2 *Low standard solution* — Transfer 1 ml of potassium standard solution (**A-8.1.2**) into a 100 ml measuring flask dilute with water to the mark and mix. This solution contains 10 micrograms of potassium (as *K*).

A-8.3.3 *Calculation*

$$\text{Potassium (as } K\text{), parts per million} = \frac{(S_r - L_r)}{H_r - L_r} \times (M_1 - M_2) + L \times \frac{1}{10^3} \times \frac{1\,000}{M}$$

where

S_r = galvanometer scale reading for sample,

H_r = galvanometer scale reading for high standard solution,

L_r = galvanometer scale reading for low standard solution,

M_1 = mass in micrograms of potassium in high standard solution,

M_2 = mass in micrograms of potassium in low standard solution, and

M = mass in g of the test sample.

NOTE — Always take care to rinse the atomizer system with distilled water after taking each reading. Also check the zero and 100 readings of the scale in between the readings.

A-9. DETERMINATION OF SODIUM (as Na)

A-9.0 Principle — Evaporation of dryness to remove fluorine. Dissolution of residue in water. Salt solution drawn into a non-luminous flame of the flame photometer and the intensity of the characteristic colour imparted to the flame is measured, using the appropriate filter.

A-9.1 Reagents

A-9.1.1 *Sodium Chloride Powder*

A-9.1.2 *Standard Sodium Solution* — Weigh accurately 0.254 2 g of dried sodium chloride powder, dissolve in little water and transfer quantitatively to a 1 litre measuring flask. Dilute to the mark with water and mix. 1 ml of this solution contains 100 micrograms of sodium (as Na).

A-9.1.3 *Dilute Standard Sodium Solution* — Transfer 10 ml of standard solution (**A-9.1.2**) in a litre measuring flask, dilute to the mark with water and mix. 1 ml of this solution contains 1 microgram of sodium (as Na).

A-9.2 Apparatus

A-9.2.1 *Platinum Dish* — 100 ml capacity.

A-9.2.2 *Flame Photometer with a Filter for Sodium*

A-9.3 Procedure

A-9.3.1 Weigh accurately about 200 g of the test sample in a polyethylene bottle with screwed cap. Transfer about 60-70 ml of the sample in a platinum dish and evaporate the mass to a small bulk on water bath. Transfer again about 50-60 ml of the sample from the bottle to the platinum dish and evaporate to a small volume. Transfer in this manner the complete volume of the sample in the dish and finally evaporate the mass to dryness. Add little water and heat to dissolve, cool, transfer quantitatively to a 100 ml measuring flask and dilute with water to the mark.

A-9.3.2 Switch on the flame photometer unclamp the galvanometer, open the gas controls and light on the flame, turn on air supply and then proceed exactly as mentioned in **A-8.3.1** in the same sequence.

NOTE — Always take care to rinse the atomizer system with water after taking each reading. Also check the zero and the 100 on the scale in between the reading.

A-9.3.3 *Preparation of High Standard Solution and Low Standard Solution of Sodium*

A-9.3.3.1 *High standard solution of sodium* — Transfer 10 ml of sodium standard solution (**A-9.1.3**) into a 100-ml measuring flask, dilute to the mark with water and mix. This solution contains 100 micrograms of sodium (as Na).

A-9.3.3.2 *Low standard solution of sodium* — Transfer 1 ml of sodium standard solution (**A-9.1.3**) into a 100-ml measuring flask, dilute to the mark with water and mix. This solution contains 10 micrograms of sodium (as Na).

A-9.4 Calculation

$$\text{Sodium (as Na), parts per million} = \frac{S-L}{HR-LR} \times (M_1 - M_2) + M_2 \times 1/M$$

where

S = galvanometer scale reading for sample,

H = scale reading for high standard solution of sodium,

L = galvanometer scale reading for low standard solution of sodium,

M_1 = mass in micrograms of sodium in high standard solution of sodium,

M_2 = mass in micrograms of sodium in low standard solution of sodium, and

M = mass in g of the test sample.

A-10. DETERMINATION OF CALCIUM (as Ca)

A-10.0 Principle — Evaporation to dryness for removing hydrofluoric acid. Dissolution of the residue in water. Solution drawn into a non-luminous flame of the flame photometer to impart a characteristic colour, measurement of the intensity of colour using suitable filter.

A-10.1 Reagents

A-10.1.1 *Standard Calcium Solution* — Weigh 2.497 5 g of calcium carbonate and transfer it to a 400 ml beaker. Add 50 ml of water and dissolve in 5.3 ml of concentrated hydrochloric acid, added in small increments. When the dissolution is complete, boil off carbon dioxide and cool to room temperature. Transfer to a 1 litre measuring flask. Dilute to the mark and mix. Transfer 50 ml of the solution into a 500 ml measuring flask, dilute to the mark and mix. One ml of this solution contains 100 micrograms of calcium (as Ca).

A-10.1.2 *Dilute Standard Calcium Solution* — Transfer 50 ml of standard solution (**A-10.1.1**) into a litre measuring flask, dilute to the mark with water and mix. One ml of this solution contains 50 micrograms of calcium (as Ca).

A-10.2 Apparatus

A-10.2.1 *Platinum dish* — 100 ml capacity.

A-10.2.2 *Flame Photometer* — with a filter for calcium.

A-10.3 Procedure

A-10.3.1 Weigh accurately about 400 g of the test sample in a polyethylene bottle with screwable cap. Transfer 60 to 70 ml of the sample into a platinum dish and evaporate on a water bath to about 10 ml. Add nearly 50-60 ml of the sample from the bottle and evaporate further to a small volume. Transfer in this manner the whole bulk of the sample from the bottle into the dish, and evaporate completely to dryness. Add little water, heat to dissolve the residue and cool. Transfer into a 100-ml measuring flask, dilute to the mark with water and mix. Switch on the flame photometer and proceed in sequence exactly as mentioned in (**A-8.2**), using high standard solution and low standard solution for calibration. Note down the high standard reading, low standard reading and sample reading of the galvanometer scale.

NOTE — Always take care to rinse the atomizer system with water after taking each reading. Also check the zero and the 100 markings on the scale in between the reading.

A-10.3.2 *Preparation of High Standard Solution and Low Standard Solution of Calcium*

A-10.3.2.1 *High standard solution of calcium* — Transfer 5 ml of standard solution (**A-10.1.1**) into a 100-ml measuring flask, dilute to the mark with water and mix. This solution contains 50 micrograms of calcium (as Ca).

A-10.3.2.2 *Low standard solution* — Transfer 10 ml of standard solution (**A-10.1.2**) into 100-ml measuring flask, dilute to the mark with water and mix. This solution contains 5 micrograms of calcium (as Ca).

A-10.4 Calculation

$$\text{Calcium (as Ca), parts per million} = \frac{SR - L}{H - L} \times (M_1 - M_2) + M_2 \times 1/M$$

where

S = scale reading for sample solution,

H = scale reading for high standard solution,

L = scale reading for low standard solution,

M_1 = calcium (as Ca) in micrograms in high standard solution,

M_2 = calcium (as Ca) in micrograms in low standard solution, and

M = mass in g of the test sample.

A-11. DETERMINATION OF CHLORIDE

A-11.0 Principle — Evaporate the sample to a small volume to remove hydrofluoric acid. Replacement of thiocyanate ion by chloride ion from mercury thiocyanate in presence of ferric salt and formation of highly coloured ferric thiocyanate complex. Measurement of the intensity of the coloured complex.

A-11.1 Reagents

A-11.1.1 *Ferric Ammonium Sulphate Solution — 0.25 Molar* — Dissolve 49.02 of ferric ammonium sulphate [$Fe\ NH_4\ (SO_4)_2.12H_2O$] in 203 ml of dilute nitric acid (70 percent). Dilute with water to 500 ml.

A-11.1.2 *Mercury Thiocynate* [$Hg\ (SCN)_2$] — Saturated solution. Add enough quantity of the salt to 250 ml of ethanol (absolute) so that few crystals remain undissolved, shake well and filter.

A-11.1.3 *Standard Chloride Solution* — Weigh accurately 0.165 0 g previously ignited (at 500°C) and cooled sodium chloride, dissolve in little water and transfer into one-litre measuring flask, dilute to the mark with water and mix. One ml of this solution contains 100 micrograms of chloride (as Cl).

A-11.1.4 *Standard Chloride Solution* — Transfer 50 ml of standard solution (**A-11.1.3**) into a 500-ml measuring flask, dilute with water to the mark and mix. 1 ml of this solution contains 10 micrograms of chloride (as Cl).

A-11.2 Apparatus

A-11.2.1 *Platinum Dish* — 100 ml capacity.

A-11.2.2 *Photo-electric Colorimeter*

A-11.3 Procedure

A-11.3.1 Weigh accurately about 100 g of the test sample in a polyethylene bottle with screwed cap. Transfer about 60 ml of the sample from the bottle into a platinum dish and evaporate to a small volume on a water-bath. Add the remaining quantity of sample from the bottle into the platinum dish and evaporate to a small volume of about 5 ml. Add about 100 milligrams of calcium carbonate powder, little water and evaporate the mass to dryness.

A-11.3.2 Cool and add about 20 ml of water, heat the contents to dissolve the chloride salts and filter the solution into a 50-ml measuring flask. Rinse the residue on filter paper with small portions of water thrice collecting the washings in the measuring flask. Cool and dilute with water to the mark and mix. Transfer 25 ml of the

solution into a 50-ml measuring flask. Add 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in sequence. Dilute with water to the mark and mix thoroughly.

A-11.3.3 Allow to stand for 15 minutes. Transfer the solution into the cell of the colorimeter and measure the absorbancy at a wavelength of about 450 nm, after having adjusted the instrument to zero absorbancy with water as reference solution.

A-11.3.4 *Blank Test* — Carry out a blank test using exactly the same procedure (**A-11.2.1** to **A-11.2.3**) and the same quantities of all the reagents, but omitting the sample.

A-11.3.5 *Preparation of Calibration Graph* — Transfer 1, 2, 3, 6, 8, 10 ml of standard solution (**A-11.1.4**) into a series of six, 50-ml measuring flasks. Proceed in sequence exactly as mentioned in (**A-11.3.2** and **A-11.3.3**). Measure the absorbancy of the chloride standard solutions on the instrument. Plot a graph having the quantities in micrograms of chloride (Cl) in the standard solution as the abscissae and the corresponding absorbance values as the ordinates.

A-11.4 Calculation — Determine the quantities of chloride (as Cl) corresponding to absorbance readings of the test sample solution and blank test solution by means of the graph.

$$\text{Chloride (as Cl), parts per million} = \frac{(m_1 - m_2)}{M}$$

where

m_1 = mass in micrograms of chloride (as Cl) in the test sample solution,

m_2 = mass in micrograms of chloride (as Cl) in the blank test solution, and

M = mass in g of the test sample.

A-12. DETERMINATION OF PHOSPHATE (AS PO_4)

A-12.0 Principle — Removal of fluorine and silica by evaporation. Formation of phosphomolybdic acid complex in acidic solution. Reduction to molybdenum blue complex. Photometric measurement of the intensity of blue colour at 580 nm wavelength.

A-12.1 Reagents

A-12.1.1 *Perchloric Acid* — 60 to 70 percent.

A.12.1.2 *Ammonium Molybdate Solution* — Dissolve 10 g of ammonium molybdate tetrahydrate ($(NH_4)_6.Mo_7.O_{24}.4H_2O$) in 100 ml of water. Add slowly 220 ml of sulphuric acid (1 : 1 solution) and 70 ml of water. Allow to cool and mix thoroughly.

A-12.1.3 *Reducing Solution* — Dissolve 0.25 g of 1-amino, 2-naphthol, 4-sulphonic acid and 0.5 g of anhydrous sodium sulphite (Na_2SO_3) in about 70 ml of water. Dissolve separately 24 g anhydrous sodium bi-sulphite ($Na_2S_2O_5$) in about 100 ml of water. Mix both the solutions and dilute to 200 ml with water, filter, if necessary, and store in a cool dark place.

A-12.1.4 *Standard Phosphate Solution* — Weigh accurately 1.433 g of pulverized, dry potassium dihydrogen phosphate (KH_2PO_4), dissolve in little water and transfer into a 1 litre standard flask dilute to mark with water and mix. Transfer 50 ml of this solution to a 500-ml measuring flask, dilute to the mark with water and mix 1 ml of this solution contains 100 micrograms of phosphate (as PO_4).

A-12.1.5 *Dilute Standard Phosphate Solution* — Corresponding to 10 milligrams of phosphate (as PO_4) per litre. Transfer 50 ml of solution (**A-12.1.4**) to a 500-ml measuring flask, dilute to the mark with water and mix. One ml of this solution contains 10 micrograms of phosphate (as PO_4).

A-12.2 Apparatus

A-12.2.1 *Platinum Dish* — 100 ml capacity.

A-12.2.2 *Photo-electric Colorimeter*

A-12.3 Procedure

A-12.3.1 Weigh accurately about 250 grams of the test sample in a PE bottle with screwable cap.

NOTE — In case of the sample, with higher phosphorus content, the quantity of test sample taken should be relatively reduced.

A-12.3.2 Transfer about 60 ml in a platinum dish and evaporate on water bath to a small volume. Add again about 60 ml of the sample from the bottle into the dish and evaporate the mass to about 10 ml. In this manner, evaporate the complete mass of the weighed sample nearly to dryness. Add 2 ml of perchloric acid and evaporate again nearly to dryness. Add about 20 ml of water and transfer the mass quantitatively into a 100 ml standard flask. Add 10 ml of ammonium molybdate solution and 5 ml of reducing solution and dilute with water to the mark. Place the flask in boiling water for 15 minutes and cool it with running water. Transfer the solution into a cell of 2 cm optical path length and carry out photometric measurement at 620 nm after having adjusted the instrument on zero absorbance against water.

A-12.3.3 *Blank Test* — Carry out a blank test at the same time using the same procedure and the same quantities of all reagents as in the case of the test sample but omitting the sample.

A-12.3.4 *Preparation of Calibration Graph* — Transfer 1, 2, 4, 6, 8 and 10 ml of the standard phosphate solution (**A-12.1.5**) into a series of six, 10 ml measuring flasks. Add 2 ml of perchloric acid, about 20 ml of water and proceed in sequence exactly as mentioned in **A-12.3.2**. Plot a graph having the quantities in micrograms of phosphate (as PO_4) in the standard phosphate solutions as abscissae and the corresponding absorbance values as the ordinates.

A-12.3.5 *Calculation* — Determine the quantities of phosphate (PO_4), in micrograms corresponding to the absorbance reading of the test sample solution and also that of the blank test solution by means of the calibration graph.

$$\text{Phosphate (as } PO_4\text{), parts per million} = \frac{(M_1 - M_2)}{M}$$

where

M_1 = mass in micrograms of phosphate (as PO_4) in the test sample solution,

M_2 = mass in micrograms of phosphate (as PO_4) in the blank test solution, and

M = mass in g of the test sample.

A-13. DETERMINATION OF SULPHITE (AS SO_3)

A-13.0 Principle — An addition of known volume of standard iodine solution to oxidize the sulphite (SO_3) and back titration of the excess iodine solution with standard sodium thiosulphate solution to find the iodine consumed in oxidation.

A-13.1 Reagents

A-13.1.1 *Iodine Solution* — approximately 0.1 N, prepared fresh.

A-13.1.2 *Iodine Solution* — approximately 0.01 N, prepared afresh.

A-13.1.3 *Sodium Thiosulphate Standard Solution* — 0.01 N.

A-13.1.4 *Starch Indicator Solution* — 0.05 percent.

A-13.2 Apparatus

A-13.2.1 *Polyethylene Weighing Bottle* — With screw cap, 250 ml capacity.

A-13.2.2 *Polyethylene Beaker* — 500 ml capacity.

A-13.2.3 *Burette* — 25 ml.

A-13.2.4 *Bulb Pipette* — 50 ml capacity.

A-13.3 Procedure

A-13.3.1 Weigh accurately about 100 g of the test sample in a polyethylene weighing bottle with screwable cap and transfer to a 500 ml capacity polyethylene beaker.

NOTE — In case of the test sample containing higher sulphite the quantity of the test sample may be relatively reduced.

A-13.3.2 Transfer 50 ml of the iodine solution into the beaker, stir and allow to stand for few minutes. Titrate the solution with standard sodium thiosulphate solution till the solution is straw yellow in colour. Add 2 ml of starch indicator solution and complete the titration indicated by disappearance of the blue colour formed.

A-13.3.3 *Standardization of 0.01 N Iodine Solution* — Take 100 ml of water into the polyethylene beaker, transfer 50 ml of the iodine solution and carry out the titration with standard sodium thiosulphate solution, using starch solution as indicator. Note down the volume of sodium thiosulphate solution used in the titration.

A-13.3.4 *Calculation*

$$\text{Sulphite (as } SO_3\text{), parts per million} = \frac{0.4 \times (V_1 - V_2)\ 1\,000}{M}$$

where

V_1 = volume in ml of the standard sodium thiosulphate solution used in the standardization of iodine solution,

V_2 = volume in ml of the standard sodium thiosulphate solution used in the titration of the test sample solution,

M = mass in g of the test sample, and

0.4 = factor corresponding to milligrams of sulphite (as SO_3) per 1 ml of standard sodium thiosulphate solution.

A-14. DETERMINATION OF SULPHATE (AS SO_4)

A-14.0 Principle — Evaporation of the sample to remove fluorine, and dilution with water and precipitation of sulphate by barium chloride solution. The measurement of the turbidity formed nephelometrically gives an estimate of sulphate.

A-14.1 Reagents

A-14.1.1 *Sodium Chloride Acidified Solution* — Dissolve 60.0 g of dried sodium chloride in about 200 ml of water, add 5 ml of hydrochloric acid (30-32 percent) and dilute the solution to 250 ml.

A-14.1.2 *Barium Chloride, Crystals, Size* — 600 to 850 microns.

A-14.1.3 *Glycerol-Alcohol Solution* — (1 : 2 *v/v*). Add 100 ml of glycerol to 200 ml of ethyl alcohol (absolute) and mix thoroughly.

A-14.1.4 *Standard Sulphate Solution* — Dissolve 1.814 1 g of dried potassium sulphate (K_2SO_4) in little water, transfer the solution to a one litre standard flask and dilute with water to the mark and mix. Transfer 50 ml of the solution to a 500 ml measuring flask, dilute with water to the mark and mix. One ml of this solution contains 100 micrograms of sulphate (as SO_4).

A-14.1.5 *Dilute Standard Sulphate Solution* — Transfer 100 ml of standard sulphate solution (**A-14.1.4**) into a litre standard flask, dilute with water to the mark and mix. One ml of this solution contains 10 micrograms of sulphate (as SO_4).

A-14.2 Procedure

A-14.2.1 Weigh accurately about 100 g of the test sample in poly-ethylene bottle with screwed cap. Transfer about 60 ml of the sample from the bottle into a platinum dish and evaporate to a small volume about 10 ml on a water bath. Add, the remaining volume of the sample from the bottle into the dish and evaporate nearly to dryness on a water bath. Add about 40 ml of water into the dish and evaporate, nearly to dryness in order to remove fluorine completely. Add about 20 ml of water and filter the solution through Whatman No. 1 filter paper into a 100-ml standard flask. Rinse the platinum dish from inside two times with little water and transfer the washings to the standard flask, through the filter paper.

A-14.2.2 Add in sequence, 0.3 g of barium chloride crystals, 20 ml of glycerol-alcohol solution into the measuring flask shaking the flask after each addition. Dilute with water to the mark. Mix thoroughly and immediately transfer the solution into the cell of the photoelectric colorimeter and measure the turbidity at a wavelength of about 430 nm, after adjusting the zero absorbance against water.

NOTE — Nephelometric reading should be taken without any loss of time after transferring the test solution into the cell to avoid settling of the precipitate.

A-14.2.3 *Blank Test* — Take about 20 ml of water in a 100 ml measuring flask and proceed in sequence exactly as mentioned in **A-15.2.2**. Note the nephelometric reading of the blank test solution.

A-14.2.4 *Preparation of Calibration Graph* — Transfer 1, 2, 4, 6, 8, 10 ml of standard sulphate solution (**A-14.1.5**) into a series of six 100 ml measuring flasks. Add about 20 ml water into each of the flasks and proceed exactly as mentioned in **A-14.2.2**. Plot a graph having the quantities in micrograms of sulphate (as SO_4) in the standard sulphate solutions as abscissae and the corresponding nephelometric readings as the ordinates.

A-14.3 Calculation — Determine the quantities of sulphate (as SO_4) in micrograms, corresponding to the nephelometric readings of the test sample solution and also that of the blank test solution from the calibration graph.

$$\text{Sulphates (as } SO_4\text{), parts per million} = \frac{(m_1 - m_2)}{M}$$

where

m_1 = mass in micrograms of sulphate (as SO_4) in the sample test solution,

m_2 = mass in micrograms of sulphate (as SO_4) in the blank test solution, and

M = mass in g of the test sample.

A-15. DETERMINATION OF SILICA AS HYDROFLUOSILICIC ACID (ALTERNATIVE METHOD)

A-15.1 Principle of the Method

A-15.1.1 Formation of molybdosilicate complex (yellow) in weakly acidic medium in presence of boric acid (to suppress fluorine interference). Reduction of the yellow complex, after addition of sulphuric acid and oxalic acid (to eliminate phosphate interference). Photometric measurement of the intensity of blue colour complex formed at 580 nm wavelength.

A-15.2 Reagents

A-15.2.1 *Dilute Sulphuric Acid* — approximately 9 N,

A-15.2.2 *Dilute Hydrochloric Acid* — approximately 2 N.

A-15.2.3 *Boric Acid Solution* — 40 g/l.

A-15.2.4 *Oxalic Acid Solution* — 100 g anhydrous acid per litre.

A-15.2.5 *Ammonium Molybdate Solution* — 100 g/l.

A-15.2.5.1 Dissolve 25 g of ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24},4H_2O$] in 200 ml of water at about 50 to 60°C. Allow to cool to ambient temperature. Filter, if necessary. Transfer to a 250-ml measuring flask and dilute to the mark with water. Mix thoroughly and transfer immediately to a polyethylene bottle.

A-15.2.5.2 Discard the solution when precipitates are formed.

A-15.2.6 *Reducing Solution*

A-15.2.6.1 Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in about 50 ml of water. Add 1.5 g of 4-amino 3-hydroxynapthalene 1-sulphonic acid (also known as 1-amino-2-naphthol-4-sulphonic acid) and dissolve by stirring.

A-15.2.6.2 Dissolve 90 g of sodium meta bisulphite ($Na_2S_2O_5$) in 900 ml of water.

A-15.2.6.3 Mix both the above solutions and dilute to 1 000 ml. Filter, if necessary and store in a cool dark place.

A-15.2.7 *Silica Standard Solutions*

A-15.2.7.1 *Standard solution A* — (corresponding to 1 000 ppm of hydrofluosilicic acid). Weigh accurately 0.417 g of pure quartz finely ground and dried powder in a 25-ml platinum crucible, add 5 g of anhydrous sodium carbonate and mix thoroughly with a thick platinum wire. Fuse the mixture gently till a clear solution is obtained.

Allow to cool, add warm water and heat moderately till the silicate dissolves completely. Cool, transfer quantitatively to 1 000 ml measuring flask, dilute to the mark and mix. Transfer immediately to a polyethylene bottle. One millilitre of this solution contains the equivalent of 1.00 mg of hydrofluorosilicic acid.

A-15.2.7.2 *Standard solution B* — (corresponding to 20 ppm of hydrofluorosilicic acid). Transfer 20.0 ml of standard solution *A* into a 1 000-ml measuring flask, dilute to the mark and mix. Transfer to a polyethylene bottle. One ml of this solution contains the equivalent of 20 micrograms. Prepare this solution at the time of use.

NOTE — Store all these reagents in polyethylene containers and carry out all operations in apparatus of this material except where specified otherwise.

A-15.3 Apparatus

A-15.3.1 *Platinum Crucible* — 25 ml capacity and platinum rod.

A-15.3.2 *Photoelectric Absorptiometer* — Fitted with cells of 2 cm optical path and with filters allowing maximum transmission at about 610 nm wavelength.

A-15.4 Procedure

A-15.4.1 Introduce a quantity of test solution prepared by the method specified in **B-5.3** of IS : 10271-1982* containing not more than 0.5 g of hydrogen fluoride into a tared 100-ml polyethylene beaker and weigh again to the nearest 0.001 g. Dilute to about 20 ml with water and add while stirring 4 ml of dilute hydrochloric acid and 5 ml of boric acid solution.

A-15.4.2 Allow to stand for 5 minutes, add 10-ml ammonium molybdate solution, mix well and allow to stand for 15 minutes (*p*H of the solution at this stage is 1.0 to 1.2). Add while stirring 5 ml of oxalic acid solution and then 20 ml of sulphuric acid.

A-15.4.3 Transfer quantitatively to a 100-ml measuring flask. Mix, add 2 ml of the reducing solution, dilute to the mark and mix. Allow to stand for 20 minutes. Using the photoelectric absorptiometer fitted with suitable filter carry out photometric measurement using cells of optical path length 2 cm, after having adjusted the instrument to zero absorbance against water.

A-15.4.4 Carry out a blank test at the same time as the test portion using the same procedure and the same quantities of all reagents as in the case of test portion but omitting the sample.

A-15.4.5 *Preparation of Calibration Graph*

A-15.4.5.1 Into a series of six 100-ml measuring flasks, transfer 0, 1, 2, 4, 6 and 8 ml of the standard silica solution *B*.

A-15.4.5.2 Add in each flask 20 ml of water and while stirring 4 ml of dilute hydrochloric acid and 35 ml of boric acid solution. Allow to stand for 5 minutes, then add 10 ml of ammonium molybdate solution, mix and allow to stand for 15 minutes. Add, while stirring 5.0 ml of oxalic acid solution, 20 ml of dilute sulphuric acid and mix.

A-15.4.5.3 Add 2 ml of reducing solution, dilute to the mark and mix well. Allow to stand for 20 minutes and then carry out the photometric measurement exactly in the same manner as described above in case of the test portion. Deduct the absorbance reading of the blank test on the reagents from those of the standard silica solutions.

A-15.4.5.4 Plot a graph having the quantities in micrograms of hydrofluosilicic acid (H_2SiF_6) in the standard silica solutions as abscissae and the corresponding absorbance values as the ordinates.

A-15.4.5.5 Determine the quantities of hydrofluosilicic acid (H_2SiF_6) corresponding to the absorbance reading of the test solution and that of the blank solution by means of the calibration graph.

*Specification for anhydrous hydrogen fluoride, technical.

A-15.5 Calculation

$$\text{Silica (as } H_2SiF_6\text{), percent by mass} = \frac{(M_1 - M_2)}{100\ M \times C}$$

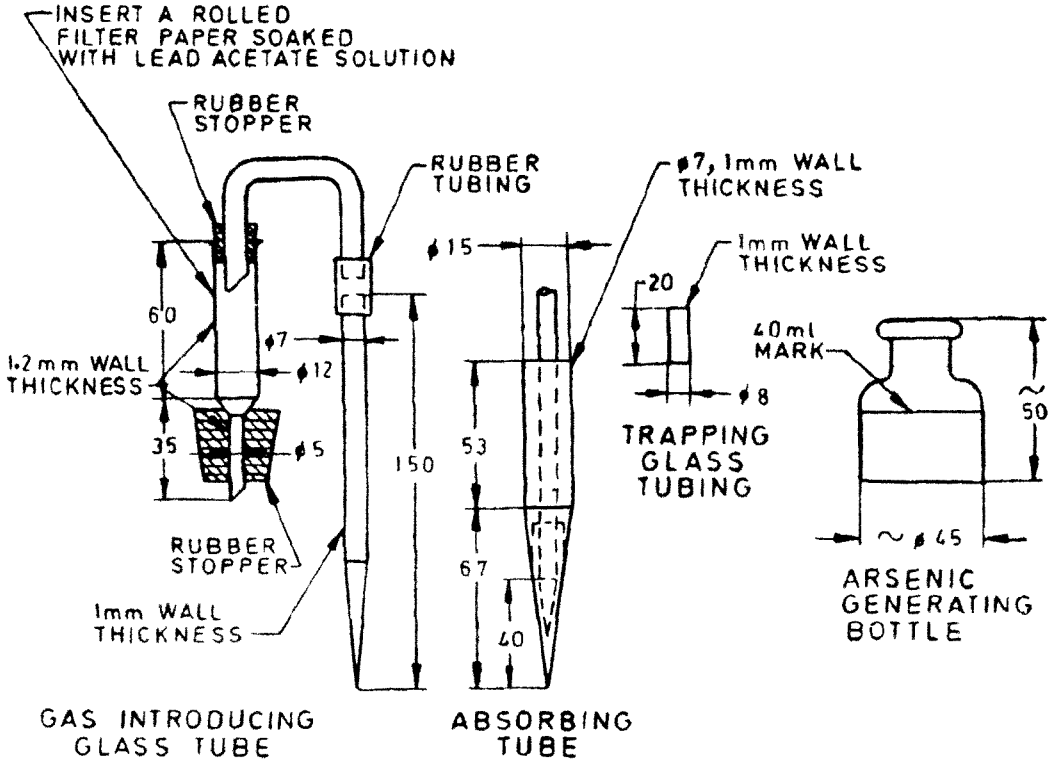
where

M_1 = mass in μg of the hydrofluosilicic acid in test solution,

M_2 = mass in μg of the hydrofluosilicic acid and blank test solution,

M = mass in g of the test portion, and

C = concentration of hydrogen fluoride expressed as percent by mass in the test sample (*see* **B-5.4** of IS : 10271-1982*).



All dimensions in millimetres.

FIG. 1 APPARATUS FOR ARSENIC DETERMINATION

*Specification for anhydrous hydrogen fluoride, technical.

(*Continued from page* 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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This Indian Standard has been developed by Technical Committee : CDC 56 and amended by CHD 1

Amendments Issued Since Publication

Amend No.	Date of Issue
Amd. No. 1	March 1984
Amd. No. 2	January 1998
Amd. No. 3	December 1999
Amd. No. 4	October 2004

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