

**BUREAU OF INDIAN STANDARDS**

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भारतीय मानक मसौदा  
इलेक्ट्रोप्लेटिंग के लिए हाइड्रोफ्लुओसिलिक एसिड — विशिष्टि  
(IS 12201 का पहला पुनरीक्षण)

*Draft Indian Standard*

**Hydrofluosilicic Acid for Electroplating  
— Specification**  
(First Revision of IS 12201)

ICS 25.220.40

Electroplating Chemicals and Photographic Materials  
Sectional Committee, CHD 5

**Last Date for Comments: 22-09-2024**

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 5

FOREWORD

(Formal clause will be added later)

The hydrofluosilicic acid is transparent, colourless, corrosive, fuming and poisonous liquid. It is commercially and popularly known as fluosilicic acid or flourosilicic acid or hexa fluorosilicic acid or hydro flourosilicic acid or sand acid. It is also used in the manufacture of a large number of products, such as concrete flooring composition, refraction reducer of glass surfaces, wood preservatives, electrolytic refining of lead, electroplating of chromium (specially black and bright), hardening of ceramics product, etc. However, this standard covers the acid used for the manufacturer of tin free steel (high oxide) in electroplating process.

This standard was originally published in 1987. This revision has been brought out in order to incorporate alternative instrumental test methods AAS, ICP-MS and ICP-OES for the determination of iron and lead. The relevant clauses have been added and the references have been updated.

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*  
**HYDROFLOUSILICIC ACID FOR ELECTROPLATING —  
SPECIFICATION**  
*(First Revision)*

## 1 SCOPE

This standard prescribes the requirements, and method of sampling and test for hydroflousilicic acid for manufacturer of tin free steel (high oxide) in electroplating process.

## 2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed in Annex A.

## 3 REQUIREMENTS

### 3.1 Description

The material shall be transparent and clear colourless liquid without sediment. It is soluble in water and shall correspond essentially to the formula  $H_2SiF_6$ .

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col 4 of Table 1.

**Table 1 Requirements for Hydrofluosilicic Acid**

(Clause 3.2, C-3, C-4.1, C-4.2)

Sl. No	Characteristic	Requirements	Method of Test ( Ref to Clause No. of Annex B)
(1)	(2)	(3)	(4)
i)	Total acidity ( as $H_2SiF_6$ ), percent by mass, <i>Min</i>	30.0	B-2
ii)	Free acidity (as HF), percent by mass, <i>Max</i>	0.05	B-3
iii)	Heavy metals (as pb), ppm, <i>Max</i>	200.0	B-4
iv)	Iron (as Fe), ppm, <i>Max</i>	200.0	B-5
v)	Solubility in water	To pass the test	B-6

## 4 PACKING AND MARKING

### 4.1 Packing

The hydrofluosilicic acid shall be packed in polyethylene lined steel carbuoys or barrels. Each carbuoy/barrel shall contain 25 kg acid. An outage or space is provided above the liquid level to compensate for liquid expansion as temperature increases.

### 4.2 Marking

4.2.1 The containers as also packages of hydrofluosilicic acid shall be marked in red letters not less than 30 mm high showing:

- a) Name of the material;
- b) Name of the manufacturer and/or his recognized trade-mark, if any;
- c) Net mass of the material;
- d) Batch No.; and
- e) Month and year of manufacture.

**4.2.2** The containers shall be labelled as shown in Fig. 15 of IS 1260 (Part 1).

**4.2.3** *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

**5 SAMPLING**

The method of drawing representative samples of the material and the criteria for its conformity with the requirements of this standard shall be as prescribed in Annex C.

ANNEX A

(Clause 2)

**LIST OF REFERRED STANDARD**

<i>IS</i>	<i>Title</i>
IS1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 1260 : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 1575 : 2003	Laboratory glassware — Separating funnels and dropping funnels ( <i>second revision</i> )
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
Part 2 : 2019	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) ( <i>First Revision</i> )
Part 65 : 2022	Application of inductively coupled plasma mass spectrometry ( ICP-MS ) — Determination of selected elements including uranium isotopes ( <i>first revision</i> )
IS 4161 : 2023	Nessler cylinder — Specification ( <i>first revision</i> )
IS 12074 : 1987	Method for determination of lead by atomic absorption spectrophotometer
IS 13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method

## ANNEX B

(Clause 3.2, Table 1)

### METHODS OF TEST FOR HYDROFLUOSILICIC ACID

#### B-1 QUALITY OF REAGENTS

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

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

#### B-2 DETERMINATION OF TOTAL ACIDITY AS HYDROFLUOSILICIC ACID (as $\text{H}_2\text{SiF}_6$ )

##### B-2.1 Principle

Evaporation of the solution to dryness in presence of sodium chloride. Dilution with water and titration 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.

##### B-2.2 Reagents

**B-2.2.1** *Sodium Chloride* — 5 percent aqueous solution.

**B-2.2.2** *Potassium Chloride* — 10 percent aqueous solution.

**B-2.2.3** *Standard Sodium Hydroxide Solution* — 1 N.

**B-2.2.4** *Standard Sodium Hydroxide Solution* — 0.1 N.

**B-2.2.5** *Phenolphthalein Indicator Solution* — 10 g/l in ethanol.

##### B-2.3 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. The evaporation shall be carried out in an exhausthood with proper suction arrangement. 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 to 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 1 N sodium hydroxide solution 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.

##### B-2.4 Calculation

$$\text{Total acidity (as } \text{H}_2\text{SiF}_6\text{), Percent by mass} = 0.0072 \times V \times \frac{100}{M}$$

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

0.0072 = factor corresponding to g of hydrofluosilicic acid ( $\text{H}_2\text{SiF}_6$ ) per 1 ml of sodium hydroxide solution.

#### B-3 DETERMINATION OF FREE ACIDITY AS HYDROFLUORIC ACID (as HF)

##### B-3.1 Principle

Titration of a test portion with standard sodium hydroxide solution using phenolphthalein as indicator.

##### B-3.2 Reagents

**B-3.2.1** *Standard Sodium Hydroxide Solution* — 1 N and 0.1 N.

### **B-3.2.2 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.

### **B-3.3 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 carbon dioxide free water. Add 5 drops of phenolphthalein solution, titrate with sodium hydroxide solution (1 N) until the end point is approached. Then heat the solution to boiling and complete the titration in the hot with sodium hydroxide solution, 0.1 N, to the appearance of a permanent pink colour.

### **B-3.4 Calculation**

$$\text{Free acidity (as HF)} = \frac{V \times N \times 0.2001 \times 100}{M}$$

Where

$V$  = volume in ml, of standard sodium hydroxide solution used in titration,

$N$  = normality of standard sodium hydroxide,

$M$  = mass in g, of the test sample, and

0.2001 = factor corresponding to mass in g of hydrofluoric acid per ml of 0.1 N sodium hydroxide solution.

## **B-4 DETERMINATION OF HEAVY METALS (as Lead)**

### **B-4.1 General**

Five methods have been prescribed for the determination of heavy metals (as Lead). However, in case of dispute, Method D shall be the referee method.

### **B-4.2 Method A — Colorimetric Method**

#### **B-4.2.1 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.

#### **B-4.2.2 Reagents**

**B-4.2.2.1 Dilute hydrochloric acid** — 20 percent (v/v).

**B-4.2.2.2 Sodium acetate solution** — 20 percent (v/v).

**B-4.2.2.3 Acetic acid solution** — 20 percent (v/v).

**B-4.2.2.4 Hydrogen sulphide water** — saturated solution of the gas.

#### **B-4.2.2.5 Standard lead solution**

Weigh accurately 1.831 g of lead acetate  $(\text{CH}_3\text{COO})_2\text{Pb} \cdot 3\text{H}_2\text{O}$ . Dissolve in small volume of water and transfer quantitatively in 1 litre measuring flask. Dilute to the mark and mix. One milliliter of this solution contains 1 milligram of lead.

#### **B-4.2.2.6 Dilute lead standard solution**

Transfer 10 ml of the lead solution into a 1 liter measuring flask. Dilute to the mark and mix. One ml of this solution contains 10 micrograms of lead.

#### **B-4.2.3 Apparatus**

**B-4.2.3.1** *Platinum dish* — 100 ml capacity.

**B-4.2.3.2** *Nessler cylinder* — 100 ml capacity (*see* IS 4161)

**B-4.2.4** *Procedure*

**B-4.2.4.1** Weigh accurately about 250 g of the test sample in a polyethylene weighing bottle of 500 ml capacity with screwable cap. Transfer about 60 ml of the sample in to a platinum dish kept on a water bath and evaporate to a small bulk. The evaporation shall be carried out in an exhausthood with proper suction arrangement. Transfer some more volume of the sample and evaporate again. Thus evaporate the total mass of the sample to dryness.

**B-4.2.4.2** Add 1 ml of dilute hydrochloric acid the residue in the dish and evaporate nearly to dryness on a water bath.

**B-4.2.4.3** 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 Nessler cylinder with stopper.

**B-4.2.4.4** Add 3 ml of sodium acetate solution and 0.3 ml of acetic acid into the tube and mix thoroughly.

**B-4.2.4.5** 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.

**B-4.2.4.6** Note the concentration of the standard lead solution, the brown colour of which matches the most with that of the sample solution.

**B-4.2.4.7** *Treatment of standard lead solution*

Add by means of a graduated pipette 10 ml of hydrochloric acid (**B-4.2.2.1**) 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 ml, 2 ml, 3 ml, 4 ml, 5 ml and 6 ml of the standard lead solution in series. Dilute to the mark and mix. Transfer the solution in mentioned in **B-4.2.4.4** and **B-4.2.4.5** above. Match the brown colouration developed in the standard solution with that of the sample solution.

**B-4.2.5** *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.

**B-4.3 Method B — Spectrophotometric Method (Alternative Method)**

**B-4.3.1** *Principle*

Lead reacts with diphenyl thiocarbazon to form a pink coloured complex in chloroform or carbon tetrachloride solution. The complex is separated, by extraction with  $\text{CHCl}_3$  or  $\text{CCl}_4$  from an aqueous, ammonium-citrate-cyanide solution. Photometric measurement is made at 520 nm.

**B-4.3.2** *Reagents*

**B-4.3.2.1** *Standard lead solution*

Dissolve 0.799 2 g of  $\text{Pb}(\text{NO}_3)_2$  in water and make the volume to 500 ml in a volumetric flask. One ml of this solutions is equivalent to 1 mg of lead.

**B-4.3.2.2** *Dilute lead standard solution*

Transfer 10 ml of the lead solution (**B-4.2.2.6**) into a 100 ml volumetric flask. Dilute to the mark and mix. One ml of this solution contains 0.1 mg of lead.

**B-4.3.2.3** *Citric acid solution* — 50 percent (m/v).

**B-4.3.2.4** *Potassium cyanide* — 10 percent (m/v).

**B-4.3.2.5** *Ammonium hydroxide solution* — 1 : 1 (v/v)

**B-4.3.2.6** *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.

**B-4.3.2.7** *Dilute nitric acid solution* — 1 percent (v/v)

**B-4.3.2.8** *Dithizone* — 10 mg/1 and 5 mg/1 in chloroform/carbon tetrachloride.

**B-4.3.3** *Apparatus*

**B-4.3.3.1** *Platinum dish* — 100 ml capacity.

**B-4.3.3.2** *Separating funnel* — see IS 1575.

**B-4.3.3.3** *Measuring flask* — 50 ml and 100 ml.

**B-4.3.4** *Procedure*

**B-4.3.4.1** Weigh accurately about 250 g of the test sample in 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. The evaporation shall be carried out in an exhaust hood with proper suction arrangement. 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 of water and filter through Whatman filter paper No. 42 or its equivalent. Collect the filtrate in 100 ml measuring flask, wash with water and make the volume of the filtrate to 100 ml and mix thoroughly.

**B-4.3.4.2** Take 25 ml of the filtrate in separating funnel (A) add 10 ml 50 percent citric acid and neutralize ( $pH = 7$ ) with 1: 1 ammonia using bromothymol blue indicator, cool, then add 10 ml of 10 percent potassium cyanide and 15 ml (10 mg/1) of dithizone and extract to separate lead. Transfer the lower layer in another funnel (B). Add 15 ml (5 mg/1) 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).

**B-4.3.4.3** 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.

**B-4.3.4.4** The 50 ml nitric acid solution containing lead or a suitable aliquot of it is poured into a small separatory funnel. (If an aliquot 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/1). 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.

**B-4.3.4.5** Following the same procedure for standard solution of lead, one for high concentration and the second for low concentration and take reading on colorimeter. Deduct the blank value from the sample result and standard lead solutions result is obtained and lead is calculated as follows.

**B-4.3.5** *Calculation*

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

where

$SR$  = sample reading,

*LSR* = lower standard reading,

*HSR* = higher standard reading,

*HS* = higher standard lead, and

*LS* = lower standard lead.

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

where

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

#### **B-4.4 Method C**

Determine lead content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 12074.

#### **B-4.5 Method D**

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

#### **B-4.6 Method E**

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

### **B-5 DETERMINATION OF IRON (as Fe)**

#### **B-5.1 General**

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

#### **B-5.2 Method A**

##### **B-5.2.1 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 (pH 3.5 to 4.2) Photometric measurement of colour intensity at 510 nm wavelength.

##### **B-5.2.2 Reagents**

**B-5.2.2.1 Dilute hydrochloric acid** — 10 percent (v/v)

**B-5.2.2.2 Hydroxyl ammonium chloride** (NH<sub>2</sub>OH.HCL) solution — 10 percent (m/v)

**B-5.2.2.3 1:10 Phenanthroline hydrochloride solution** — 0.5 percent.

Add 0.5 g of 1 : 10 phenanthroline hydrochloride, monohydrate (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub> HCl.H<sub>2</sub>O) 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 develop subsequently.

##### **B-5.2.2.4 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.

**B-5.2.2.5 Standard iron solution** (corresponding to 100 g of iron as Fe per litre)

Weigh accurately 0.863 g of ferric ammonium sulphate [FeNH<sub>4</sub>(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O], dissolve in little water. Add 10 ml of hydrochloric acid (32 percent). Transfer quantitatively to 1 litre measuring flask, dilute to the mark and mix. One ml of this solution contains 100 micrograms of iron (as Fe).

#### **B-5.2.2.6** *Standard iron solution*

Transfer 50 ml of standard solution (**B-5.2.1.5**) 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).

#### **B-5.2.3** *Apparatus*

**B-5.2.3.1** *Platinum dish* — 100 ml capacity.

**B-5.2.3.2** *pH meter with glass electrode* — see IS 5741

**B-5.2.3.3** *Photo- electric absorptiometer*

**B-5.2.3.4** *Ordinary laboratory apparatus*

#### **B-5.2.4** *Procedure*

**B-5.2.4.1** Weigh accurately about 250 g of test sample in polyethylene 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. The evaporation shall be carried out in an exhaust hood with proper suction arrangement. 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 to 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 min. 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 as to adjust the pH of the solution to about 4.

**B-5.2.4.2** *Blank test* — Carry out a blank test using exactly the same procedure as follows.

#### **B-5.2.4.3** *Procedure*

Weigh accurately about 300 g of test sample in a polyethylene bottle with screwable cap. Transfer about 60 ml to 70 ml of the sample into a platinum dish and evaporate to a small volume on water bath. Again transfer about 50 ml to 60 ml of the sample in 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 (**B-5.2.4.5**) corresponding to a reading of 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 (**B-5.2.4.6**) 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 reading. Always light the gas first keeping the air flow closed and then set the luminosity of the flame by adjusting the air flow.

#### **B-5.2.4.4** *Preparation of calibration graph*

Into a series of six, 25 ml measurement flasks, transfer 1 ml, 2 ml, 3 ml, 4 ml, 6 ml, 8 ml and 10 ml of standard iron solution (**B-5.2.2.6**). Add 1 ml of 1 : 10 phenolphthalein solution, dilute to the mark, shake well, allow to stand for 30 min and measure the absorbency of the solution as mentioned in **B-5.2.4.3**. Proceed exactly as mentioned in **B-5.2.4.1** 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 abscissa and the corresponding absorbance values as the ordinates.

**B-5.2.4.5 High standard solution**

Transfer 5 ml of potassium standard solution (B-5.2.4.7) into a 100 ml measuring flask, dilute with water to the mark and mix. This solution contains 50 micrograms of potassium (as K).

**B-5.2.4.6 Low standard solution**

Transfer 1 ml of potassium standard solution (B-5.2.4.8) into a 100 ml measuring flask. Dilute with water to the mark and mix. This solution contains 1 microgram of potassium per milliliter.

**B-5.2.4.7 Dilute standard potassium solution**

Transfer 50 ml of standard solution (B-5.2.4.8) into a 500 ml measuring flask and dilute to the mark with water. One ml of this solution contains 10 micrograms of potassium (as K).

**B-5.2.4.8 Standard potassium solution**

Weigh accurately 0.1907 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).

**B-5.2.4.9 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.

$$\text{Iron ( as Fe), parts per million} = \frac{(M_1 - M_2)}{10^6} \times \frac{10^6}{M} = \frac{(M_1 - M_2)}{M}$$

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.

**B-5.3 Method B**

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

**B-5.4 Method C**

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

**B-5.5 Method D**

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

**B-6 TEST FOR SOLUBILITY IN WATER**

**B-6.1 Procedure**

Weigh accurately about 0.5 g of the material and dissolve in 50 ml water. The material shall be taken as having satisfied the requirement of the test, if there is no turbidity.

ANNEX C

(Clause 5)

**SAMPLING OF HYDROFLUOSILICIC ACID FOR ELECTROPLATING**

**C-1 GENERAL REQUIREMENTS OF SAMPLING**

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

**C-1.2** Samples shall not be taken at a place exposed to weather.

**C-1.3** The sample shall be placed in suitable, dry and air-tight containers, preferably with replaceable closures.

**C-1.4** While opening a sample container, adequate care shall be taken to prevent foreign particles from falling into the material.

**C-1.5** Each sample containers shall be marked with full details of sampling, the date of sampling, the year of manufacture and the batch number, if any.

**C-2 SCLAE OF SAMPLING**

**C-2.1 Lot**

All the steel carboys or barrels of the same batch shall constitute a lot.

**C-2.2** The conformity of a lot to the requirements of the standard shall be determined on the basis of the tests carried out on the sample selected from the lot.

**C-2.3** Unless otherwise agreed to between the purchaser and the manufacturer, the number of carbuoys or barrels to be selected at random from the lot shall be in accordance with Table 2.

**Table 2 Number of Carbuoys or Barrels to be Selected**

(Clause C-2.3)

SI No.	Lot Size	Sample Size
(1)	(2)	(3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 150	5
iv)	151 and above	7

**C-3 NUMBER OF TESTS**

Test for determination of total acidity (percent by mass) shall be conducted on individual carbuoys and for the remaining other characteristics given in Table 1 shall be tested on composite sample.

**C-4 CRITERIA FOR CONFORMITY**

**C-4.1** The lot shall be declared as conforming to the requirements of total acidity if:

$$(\bar{X} - 0.6 R) \geq \text{the minimum value specified in Table 1.}$$

where

$$\bar{X} \text{ (average) } = \frac{\text{sum of the test results}}{\text{Number of tests}}, \text{ and}$$

$R$  = Difference between the maximum and minimum value of the test results.

**C-4.2** The lot shall be declared as conforming to the remaining characteristics if the composite sample satisfies the relevant requirements given in Table 1.