

भारतीय मानक मसौदा
फैरिक क्लोराइड, तकनीकी — विशिष्टि
) आईएस 711 का दूसरा पुनरीक्षण

Draft Indian Standard
FERRIC CHLORIDE, TECHNICAL — SPECIFICATION
(*Second Revision of IS 711*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

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

This standard was first published in 1955 and was subsequently revised in 1970. The sampling scheme prescribed in the original standard was modified in the first revision.

In this revision, instrumental test methods for the determination of sulphates, nitrates and copper have been added as alternate test methods. The requirements of cyanide, cadmium, chromium, lead, mercury, nickel and selenium along with their methods of test have been incorporated. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated.

Ferric chloride, technical, is produced generally in India by treating iron scraps with hydrochloric acid or chlorine. The material is obtained in the form of anhydrous powder, hydrated material as deliquescent crystalline lumps, and liquid of varying concentrations.

Ferric chloride is used as a coagulant for sewage and industrial wastes, also in glycerine manufacture, in etching copper, in photo-engraving as a mordant, and to produce decorative surface effects on ceramics. It is also an oxidizing, chlorinating and condensing agent, a disinfectant, a pigment and a medicine.

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

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ferric chloride, technical, chiefly used as coagulant in water purification, in the manufacture of glycerine, in etching glass plates and in photo-engravings.

2 REFERENCES

The Indian Standards given below 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.

<i>IS No.</i>	<i>Title</i>
265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
299 : 2012	Alumino-ferric — Specification (<i>fifth revision</i>)
323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)
2480 (Part 1) : 1983	General purpose glass thermometers part 1 solid-stem thermometers (<i>second revision</i>)
3025 (Part 2) : 2019/ ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
3025 (Part 27) : 1986	Methods of sampling and test (physical and chemical) for water and wastewater : part 27 cyanide (<i>first revision</i>)
3025(Part 41) : 1992	Methods of sampling and test (physical and chemical) for water and wastewater : part 41 cadmium (<i>first revision</i>)
3025(Part 42) : 1992	Methods of sampling and test (physical and chemical) for water and wastewater : part 42 copper (<i>first revision</i>)
3025(Part 56) : 2003	Methods of sampling and test (physical and chemical) for water and wastewater : part 56 selenium (<i>first revision</i>)
4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)
11124 : 1984	Method for atomic absorption spectrophotometric determination of arsenic
12574 : 1989	Fried banana chips

3 FORM

3.1 The material shall be in one of the following forms:

- a) Anhydrous powder,
- b) Hydrated, and
- c) Liquid.

4 REQUIREMENTS

4.1 Description

4.1.1 *Anhydrous Powder*

The material of this form shall be greenish black, crystalline powder, extremely hygroscopic, becoming orange coloured on hydration.

4.1.2 *Hydrated*—The material of this form shall be brownish yellow, deliquescent, crystalline lumps.

4.1.3 *Liquid*

The material of this form shall be a clear dark-brown, viscous liquid, free from sediments or suspended matter or other visible impurities.

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

5 PACKING AND MARKING

5.1 The material shall be packed in containers as agreed to between the purchaser and the supplier.

5.1.1 For anhydrous material, mild steel drums, for hydrated material, rubber and PVC lined drums, and for liquid material, carboys and stone jars, are recommended.

5.2 The containers shall be securely closed and marked with the name of the manufacturer; net weight and form of the material in the container; recognized trade-mark; if any, and the year of manufacture.

5.3 BIS Certification Marking

The product may also be marked with Standard Mark.

5.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and Rules and Regulations made thereunder. The details of the conditions under which a license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Table 1 Requirements for Ferric Chloride, Technical
(Clause 4.2)

Sl No.	Characteristic	Requirement For			Method of Test (Ref to Cl No. in Annex A)
		Anhydrous (3)	Hydrated (4)	Liquid (5)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Relative Density at 25°/25°C, <i>Min</i>	-	-	1.46	A-2
ii)	Ferric chloride (as FeCl ₃), percent by weight, <i>Min</i>	98.0	59.0	43.0	A-3
iii)	Free acid (HCl), percent by weight, <i>Max or</i>	—	0.03	0.02	A-4
	Basicity (as Fe ₂ O ₃), percent by weight, <i>Max</i> .	—	0.03	0.2	A-4
iv)	Ferrous salts (as FeCl ₂), percent by weight, <i>Max</i>	0.10	0.10	0.10	A-5
v)	Insoluble matter, percent by weight, <i>Max</i>	0.50	0.20	0.05	A-6
vi)	Free chlorine (as Cl), percent by weight, <i>Max</i>	0.01	0.01	0.01	A-7
vii)	Sulphates (as SO ₄), percent by weight, <i>Max</i>	0.30	0.30	0.30	A-8 or A-20
viii)	Nitrates (as NO ₃), percent by weight, <i>Max</i>	0.05	0.05	0.05	A-9 or A-20
ix)	Alkalis and alkaline earths (expressed as sulphates), percent by weight, <i>Max</i>	0.3	0.3	0.2	A-10
x)	Copper as (Cu), percent by weight, <i>Max</i>	0.015	0.015	0.015	A-11
xi)	Zinc (as Zn), percent by weight, <i>Max</i>	0.01	0.01	0.01	A-11
xii)	Arsenic (as As ₂ O ₃), percent by weight, <i>Max</i>	0.000 5 (5 ppm)	0.000 3 (3 ppm)	0.000 2 (2 ppm)	A-12
xiii)	Cyanide (as CN ⁻), ppm, <i>Max</i>	40	25	18	A-13
xiv)	Cadmium (as Cd), ppm, <i>Max</i>	2.4	1.5	1.1	A-14
xv)	Chromium (as Cr), ppm, <i>Max</i>	40	25	18	A-15
xvi)	Lead (as Pb), ppm, <i>Max</i>	8	5	3.6	A-16
xvii)	Mercury (as Hg), ppm, <i>Max</i>	0.8	0.5	0.36	A-17
xviii)	Nickel (as Ni), ppm, <i>Max</i>	16	10	7.2	A-18
xix)	Selenium (as Se), ppm, <i>Max</i>	8	5	3.6	A-19

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A (Clause 4.2)

ANALYSIS OF FERRIC CHLORIDE, TECHNICAL

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 RELATIVE DENSITY

A-2.1 Outline of the Method

The sample is brought to the prescribed temperature and transferred to a hydrometer cylinder at approximately the same temperature. The appropriate hydrometer is lowered into the sample and allowed to settle. The hydrometer is read and the temperature of the sample noted.

A-2.2 Apparatus

A-2.2.1 *Relative Density Hydrometer*

Of a suitable range. A relative density hydrometer calibrated at any temperature other than 25°C may also be used with appropriate correction (*see* Note under **A-2.2.5**).

A-2.2.2 *Hydrometer Cylinder*

Of clear glass or plastic, with a lip for convenience of pouring and having an inside diameter of at least 25 mm greater than the outside diameter of the hydrometer used. The height of the cylinder shall be such that the hydrometer floats in the sample with at least 25 mm clearance between the bottom of the hydrometer and the bottom of the cylinder.

A-2.2.3 Thermometer — of a suitable range and conforming to IS 2480 (Part 1).

A-2.2.4 *Constant Temperature Bath*

A-2.3 Procedure

A-2.3.1 Adjust the temperature of the sample to 25°C and bring the hydrometer, cylinder and thermometer to approximately that temperature.

A-2.3.2 Pour the sample into the clean hydrometer cylinder without splashing in order to avoid the formation of bubbles and to reduce to a minimum the evaporation of the sample. Remove any air bubbles formed before inserting the hydrometer.

A-2.3.3 Place the cylinder containing the sample in a constant temperature bath to ensure that the temperature of the sample does not change during the test.

A-2.3.4 Lower the hydrometer gently into the sample and when it has settled, determine the temperature of the sample which is thoroughly but cautiously stirred with the thermometer, the whole of the mercury being immersed. Remove the thermometer and allow the hydrometer to resettle. Depress it about two scale divisions into the liquid and then release it. Keep the portion of the stem above the level of the liquid dry, since failure to do this changes the effective weight of the instrument and thus affects the accuracy of the reading. Allow sufficient time for the hydrometer to come to rest and for all air bubbles to come to the surface.

A-2.3.5 When the hydrometer has come to rest, floating away from the walls of the cylinder, and the temperature of the sample is constant, read the hydrometer to the nearest 0.0005. The correct hydrometer reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. If a standardization certificate is provided, correct the hydrometer reading according to the data thereon. Report the value so obtained, to the nearest 0.001 as the relative density at 25°/25°C.

NOTE —When a relative density hydrometer calibrated at a temperature other than 25°C is used, the correction for the hydrometer reading is calculated as follows:

$$X = 0.00025 h (t - 25)$$

where

X = correction factor,

h = relative density of liquid at $t^{\circ}\text{C}$ as indicated by the hydrometer, and

t = temperature at which the hydrometer is calibrated.

A-3 DETERMINATION OF FERRIC CHLORIDE

A-3.1 Reagent

A-3.1.1 *Concentrated Hydrochloric Acid* — See IS 265

A-3.1.2 *Potassium Iodide* — Iodate-free.

A-3.1.3 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-3.1.4 *Starch Solution*

Triturate 5 g of pure starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 l of boiling water, boil for 3 min, allow the solution to cool and decant off the clear liquid.

A-3.1.5 *Cuprous Iodide* — solid.

A-3.2 Prepared Sample Solution

Weigh accurately about 10 g of the material and dissolve in 100 ml of 2 percent (w/v) hydrochloric acid. If there is any insoluble matter, filter through a sintered glass crucible or a Gooch crucible. Wash four times with 2 percent (w/v) hydrochloric acid and then with cold water. Make up the volume of the filtrate to 250 ml.

A-3.3 Procedure

Transfer 25 ml of the prepared sample solution into a 300 ml glass-stoppered flask and dilute to 50 ml. Add 3 ml of concentrated hydrochloric acid, 3 g of potassium iodide, a little solid cuprous iodide and allow to stand for 30 min. Then dilute with 100 ml of water and titrate with standard sodium thiosulphate solution using starch solution towards the end.

A-3.3.1 Carry out a blank test with all the reagents but without the material to be tested.

A-3.4 Calculation

$$\text{Ferric chloride (as FeCl}_3\text{), percent by weight} = \frac{162.22 (V_1 - V_2)N}{W}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in the test with the material,

V_2 = volume in ml of standard sodium thiosulphate solution used in the blank,

N = normality of standard sodium thiosulphate solution, and

W = weight in g of the material taken under **A-3.2**.

A-4 DETERMINATION OF FREE ACIDITY OR BASICITY

A-4.1 Reagents

A-4.1.1 Phenolphthalein Indicator

Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (*See* IS 323), 95 percent by volume that has been previously neutralized to the indicator.

A-4.1.2 *Standard Potassium Hydroxide Solution* — 0.5 N and 0.2 N.

A-4.1.3 *Standard Hydrochloric Acids* — 0.5 N and 0.2 N.

A-4.1.4 Potassium Fluoride Solution

Dissolve 100 g of potassium fluoride in 120 ml of hot water, freshly distilled. Add 1 ml of phenolphthalein indicator and neutralize with standard hydrochloric acid (0.5 N) or standard potassium hydroxide solution (0.5 N), as the case may be. (One millilitre of potassium fluoride solution in 10 ml of freshly distilled water shall give a faint pink colour.) Filter the solution, if necessary, and then dilute to 200 ml with freshly distilled water.

A- 4.2 Procedure

Pipette out 40 ml of standard hydrochloric acid (0.2 N) into a 100 ml volumetric flask. Add to the flask about 10 g of the material, accurately weighed, and shake well to dissolve. Make up the volume to the mark. Pipette out 25 ml of this, solution in a 250 ml conical flask. Add 50 ml of potassium fluoride solution and 0.5 ml of phenolphthalein indicator, and cool the contents to 15°C. Titrate the solution with standard potassium hydroxide solution (0.2 N) until a delicate pink colour persisting for 1 min is obtained.

A-4.2.1 Carry out a blank test using 10 ml of standard hydrochloric acid and other reagents.

A-4.3 Calculation

$$\text{Free acidity (as HCl), percent by weight} = \frac{14.60 (A - B)N}{W}$$

where

A = volume in ml of standard potassium hydroxide solution used in the test with the material,

B = volume in ml of standard potassium hydroxide solution used in the blank,

N = normality of standard potassium hydroxide solution, and

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

A-4.3.1 In case A is less than B, there is no free acid and the sample is basic. In such cases, calculate the basicity as under:

$$\text{Basicity (as Fe}_2\text{O}_3\text{), percent by weight} = \frac{10.646 (B-A)N}{W}$$

A-5 DETERMINATION OF FERROUS SALTS (as FeCl₂)

A-5.1 Reagents

A-5.1.1 *Concentrated Hydrochloric Acid* — See IS 265

A-5.1.2 *Potassium Ferricyanide Solution* — 5 percent (w/v), freshly prepared.

A-5.2 Procedure

Weigh accurately 0.5 g of the material and dissolve in water and make up the volume with water to 500 ml. Transfer 25 ml of this solution, add 1 ml of hydrochloric acid and one drop of potassium ferricyanide solution.

A-5.2.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if no blue colour is produced.

A-6 DETERMINATION OF INSOLUBLE MATTER

A-6.1 Reagents

A-6.1.1 *Dilute Hydrochloric Acid* — approximately 2 percent (v/v).

A-6.2 Procedure

Weigh accurately about 20 g of the material and dissolve in 100 ml of dilute hydrochloric acid. Filter through a tared sintered glass crucible (G No. 4). Wash twice with cold dilute hydrochloric acid and then thrice with water. Dry the crucible with its contents to constant weight at 105° to 110°C.

A-6.3 Calculation

$$\text{Insoluble matter, percent by weight} = 100 \times \frac{W_1}{W_2}$$

where

W_1 = weight in g of the residue, and

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

A-7 TEST FOR FREE CHLORINE

A-7.1 Reagents

A-7.1.1 *Starch Iodide Paper*

Impregnate ordinary filter paper with mucilage of starch, diluted with an equal volume of 0.4 percent (w/v) solution of potassium iodide in water.

A-7.2 Procedure

Dissolve 0.5 g of the material in 10 ml of water in a conical flask. Boil the solution and place a moist starch iodide paper on the mouth of the flask.

A-7.2.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if no blue colour is produced on the starch paper.

A-8 DETERMINATION OF SULPHATES**A-8.1 Reagent**

A-8.1.1 *Dilute Hydrochloric Acid* — approximately 10 percent (w/v).

A-8.1.2 *Barium Chloride Solution* — approximately 10 percent (w/v).

A-8.2 Procedure

Weigh accurately about 5 g of the material and add 100 ml of dilute hydrochloric acid. Filter, if necessary, and wash the filter paper thoroughly. Heat the solution to boiling and add drop by drop 10 ml of hot barium chloride solution. Boil the solution for 15 min and then allow to stand for 4 h. Filter through a tared Gooch or a sintered glass crucible (G No. 4). Wash the precipitate first with hot dilute hydrochloric acid and then with hot water till it is free from chlorides and dry to constant weight at 105° to 110°C.

A-8.3 Calculation

$$\text{Sulphates (as SO}_4\text{), percent by weight} = \frac{41.15 \times A}{W}$$

where

A = weight of barium sulphate, and
W = weight of the material taken for the test.

A-8.4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-9 TEST FOR NITRATES**A-9.1 Reagents**

A-9.1.1 *Dilute Sulphuric Acid*—approximately 5 N.

A-9.1.2 *Concentrated Hydrochloric Acid* — See IS 265.

A-9.1.3 *Standard Potassium Nitrate Solution* — 0.001 molar.

A-9.1.4 *Concentrated Sulphuric Acid* — See IS 266.

A-9.1.5 *Standard Indigo Carmine Solution*

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid, add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to produce 1000 ml. Standardize the solution so that 10 ml added to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heating to boiling. One millilitre of this solution is equivalent to 0.02 mg of nitrate (as NO_3).

A-9.1.6 Dilute Ammonium Hydroxide Solution—approximately 5 N.

A-9.2 Procedure

Weigh accurately 0.4 g of the material and dissolve in 100 ml of water. Add 50 ml of dilute ammonium hydroxide solution and filter by suction. To 15 ml of the filtrate, add 5 ml of dilute sulphuric acid, 1 ml of standard indigo carmine solution and 20 ml of concentrated sulphuric acid and heat to boiling.

A-9.2.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced does not entirely disappear.

A-10 DETERMINATION OF ALKALIS AND ALKALINE EARTHS

A-10.1 Reagents

A-10.1.1 Concentrated Hydrochloric Acid — See IS 265.

A-10.1.2 Hydrogen Peroxide — approximately 20 volumes.

A.10.1.3 Ether

A-10.1.4 Dilute Hydrochloric Acid — approximately 2 N.

A-10.1.5 Potassium Permanganate Solution — approximately 0.1 N.

A-10.1.6 Ammonium Thiocyanate Solution— approximately 10 percent (w/v).

A-10.1.7 Extraction Mixture — a mixture of equal volumes of amyl alcohol and amyl acetate.

A-10.1.8 Sulphuric Acid — See IS 266.

A-10.2 Procedure

A-10.2.1 Weigh accurately about 10 g of the material and treat it with 10 ml of water. Add 17 ml of concentrated hydrochloric acid and two drops of hydrogen peroxide. Shake the solution with four successive 20 ml portions of ether in a separating funnel. Evaporate the acid solution, which will be almost colourless, to dryness. Dissolve the residue in 2 ml of concentrated hydrochloric acid and 10 ml of water. Filter, if necessary, and dilute with water to 100 ml.

A-10.2.2 Evaporate a 35 ml aliquot to about 10 ml. Add 1 ml of dilute hydrochloric acid and one drop of potassium permanganate solution and mix well. Add 5 ml of ammonium thiocyanate solution and extract with two 10 ml portions of extraction mixture. Separate the aqueous layer and evaporate to dryness preferably in silica or platinum crucible of 25-30 ml capacity. Moisten with concentrated sulphuric acid. Ignite and weigh the residue to constant weight.

A-10.3 Calculation

Alkalis and alkaline earths
(expressed as sulphates) = $\frac{10\,000\,A}{35\,W}$

where,

A = weight of the residue, and

W = weight of the material taken for the test.

A-11 TEST FOR COPPER AND ZINC**A-11.1 Apparatus**

A-11.1.1 *Nessler* Cylinders — 50 ml capacity.

A-11.2 Reagents

A.11.2.1 *Citric Acid* — solid.

A-11.2.2 *Dilute Ammonium Hydroxide Solution* — approximately 5 N.

A-11.2.3 *Sodium Diethyl Dithiocarbamate Solution*

Dissolve 0.1 g of the salt [(C₂H₅)₂ NCS₂ Na] in 100 ml of water.

A-11.2.4 *Carbon Tetrachloride*

A-11.2.5 *Anhydrous Sodium Sulphate*

A-11.2.6 *Standard Copper Solution*

Dissolve 0.392 8 g of copper sulphate (CuSO₄.5H₂O) in 250 ml of copper free water. Pipette out 25 ml of this solution and make up the volume to 100 ml. One millilitre of the resulting solution is equivalent to 0.1 mg of copper (Cu).

A-11.2.7 *Resorcinol*

A-11.2.8 *Thymol Blue Indicator*

Warm 0.1 g of thymol blue with 4.3 g of 0.05 N sodium hydroxide solution and 5 ml of rectified spirit until dissolved. Dilute with ethyl alcohol, 20 percent by volume.

A-11.2.9 *Chloroform*

A-11.2.10 *Dithizone Solution*

Dissolve 0.1 g of diphenyl thiocarbazon (C₆H₅NH NH CS N:NC₆H₅) in 100 ml of chloroform.

A-11.2.11 *Standard Hydrochloric Acid* — 1 N.

A-11.2.12 *Ammonium Chloride* — solid.

A-11.2.13 *Potassium Ferrocyanide Solution* — approximately 1 percent freshly prepared solution in water.

A-11.2.14 Standard Zinc Solution

Dissolve 1.000 g of zinc in slight excess of hydrochloric acid and dilute with water to make up the volume to 1000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of this solution is equivalent to 0.1 mg of zinc.

A-11.3 Prepared Sample Solution

Treat 10 g of the material with 10 ml of water. Add 17 ml of concentrated hydrochloric acid and 2 drops of hydrogen peroxide (20 volumes). Shake the resulting solution with four successive 20 ml portions of ether. Evaporate the acid solution to dryness. Dissolve the residue in 2 ml of dilute hydrochloric acid and 10 ml of water and filter if necessary. Dilute with water and make up the volume to 100 ml.

A-11.4 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD

A-11.4.1 Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

A-12 DETERMINATION OF CYANIDE

Dissolve 1.0 g of material in about 50 ml distilled water and make it to 500 ml in a volumetric flask (Sample Solution). Test cyanide in this sample solution by following the method as prescribed in IS 3025 (part 27) and multiply value of result in this test by a factor of 500 to get cyanide content in the sample.

A-13 DETERMINATION OF ARSENIC

A-13.1 Procedure

Dissolve 1.0 g of material in 10 ml of water and carry out the test for Arsenic as prescribed in IS 2088, using for comparison a stain obtained with following amounts of arsenic trioxide.
For Anhydrous — 0.0105 mg; For Hydrated — 0.0066 mg; For Liquid — 0.0048 mg

A-13.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length and intensity of the stain is not greater than that produced in the control sample.

A-13.3 Referee Method

Follow the method by Atomic Absorption Spectrophotometer (AAS) as prescribed in IS 11124. Alternately, it can be also analyzed by ICP-OES.

A-14 DETERMINATION OF CADMIUM

Follow the method by AAS (referee method) as prescribed in IS 3025 (part 41). Alternatively it can be also analyzed by ICP-OES.

A-15 DETERMINATION OF TOTAL CHROMIUM

Follow the method given in **A-12** of IS 299 (but weighing 2 g of sample in water instead of 1 g in case of anhydrous form) as per procedure laid in **A-12.3** of IS 299.

A-16 DETERMINATION OF LEAD

Follow the method provided in **A-8** of IS 299, but weighing following quantities of sample in **A-8.3** instead of 1 g and comparing with control test using 2.0 ml of standard lead solution.

For Anhydrous — 2.5 g; For Hydrated — 4 g; For Liquid — 5.6 g

Alternatively, it can also be analyzed by ICP-OES.

A-16.1 Referee method

Follow the method by Atomic Absorption Spectrophotometer (AAS) as prescribed in IS 12074.

A-17 DETERMINATION OF MERCURY

Follow the method given in **A-10** of IS 299, but weighing 3 g of sample in water (instead of 5 g) as per procedure laid in **A-10.5.2** of IS 299.

A-18 DETERMINATION OF NICKEL

Follow the method by AAS (referee method) as prescribed in IS 3025 (Part 54).

Alternatively it can be also analyzed by ICP-OES.

A-19 DETERMINATION OF SELENIUM

Follow the method by AAS (referee method) as prescribed in IS 3025 (Part 56).

Alternatively it can be also analyzed by ICP-OES.

A-20 ION CHROMATOGRAPHY FOR NITRATES AND SULPHATES

A-20.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of Nitrates 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 nitrates 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-20.2 Equipment

A-20.2.1 Anion Guard Column — A protector of the separator column.

A-20.2.2 Anion Separator Column — Suitable for selective separation of ions under analysis.

A-20.2.3 Anion Suppressor Device — Anion micromembrane suppressor is used to analyse the data

A-20.2.4 Detector — Conductivity detector

A-20.2.5 Software

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

A-20.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

A-20.3 Reagents

A-20.3.1 Glass or Polyethylene Sample Bottles

A-20.3.2 Distilled water or deionized water free from the anions of interest.

A-20.3.3 Eluent

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-20.3.4 Micromembrane Suppressor Solution, (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water.

A-20.4 Standard solutions

A-20.4.1 Chloride — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-20.4.2 Sulphate — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

A-20.4.3 Nitrate Stock Solution

Dry approximately 2 g of sodium nitrate (NaNO₃) at 105°C for 24 h. Dissolve exactly 1.3707 g of the dried salt in reagent water, and dilute to 1 l with reagent water in a volumetric flask.

A-20.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1-1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-20.6 Procedure

Dissolve between 1 to 5 grams sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. 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-20.7 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)

SAMPLING AND CRITERIA FOR CONFORMITY OF FERRIC CHLORIDE, TECHNICAL

B.1 GENERAL

B-1.1 In drawing samples the following precautions and directions shall be observed.

B-1.2 Precautions shall be taken to protect the samples, the sampling implements and the container for samples from contamination. If the material is in anhydrous form, the sampling shall be carried out with minimum amount of exposure to atmosphere.

B-1.3 The sampled material shall be placed in suitable, dry, clean container.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling.

B-1.5 Samples shall be taken from different parts of the containers using suitable sampling implements in order that the collected material is truly representative of the container.

B-2 SCALE OF SAMPLING

B-2.1 All the containers in a consignment of ferric chloride of the same type and from a single batch of manufacture shall constitute a lot.

B-2.2 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.3 The number (*n*) of containers to be selected from the lot shall depend upon the size of the lot (*N*) and shall be in accordance with col 1 and 2 of Table 2.

Table 2 Number of Containers to be selected from a Lot

Sl. No	Lot Size	Number of Containers to be Selected In the Sample
(1)	N (2)	n (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 and above	7

B-2.4 These containers shall be selected at random from the lot. For random selection procedures, guidance may be had from IS 4905.

B - 3 NUMBER OF TESTS

B-3.1 From each of the selected containers about 200 g of ferric chloride shall be withdrawn with the help of a sampling implement. Out of the portions so collected equal quantity of the material shall be taken out and mixed together to form composite sample weighing about 300 g. The remaining portions corresponding to each of the selected containers shall be transferred to separate sample bottles and these sample bottles containing ferric chloride shall be termed individual samples.

B-3.2 Tests for ferric chloride content and relative density (in the case of liquid) shall be conducted on individual samples. Tests for all other characteristics shall be made on the composite sample.

B - 4 CRITERIA FOR CONFORMITY OF LOT

B-4.1 Ferric Chloride Content and Relative Density

From the individual test results, the mean (\bar{X}) and range (R) of the values shall be calculated for each of the two characteristics (range being defined as the difference between the maximum and minimum values). The values of the expression ($\bar{X} - 0.6R$) shall be greater than or equal to the corresponding value given in Table 1 if the lot is to be accepted under this clause.

B-4.2 For Other Characteristics

The test results for all other characteristics tested on the composite sample shall satisfy the relevant requirements for the acceptance of the lot.