

भारतीय मानक मसौदा
फ्रेंच चाक, तकनीकी – विशिष्टि
(आईएस 380 का तीसरा का पुनरीक्षण)

Draft Indian Standard
FRENCH CHALK, TECHNICAL — SPECIFICATION
(*Third Revision of IS 380*)

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ICS 71.060.50

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

FORWARD

This Indian Standard (Third 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.

French chalk is largely used in the preservation of rubber goods, in pyrotechnic compositions, as a filler for insecticides, in the manufacture of paper and as a sizing material in textile.

French chalk is also used by paint industry and in the production of cosmetics and high frequency ceramics but the material for these end uses is not covered in this standard.

This standard was first issued in 1952 and subsequently revised in 1967 and 1978. In this revision, instrumental test methods for the determination of chlorides, copper, iron and magnesium have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated.

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 French chalk; technical, used for various purposes.

1.1.1 This standard does not cover the material required for paint industry, cosmetics and high frequency ceramics.

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>
264:2005	Nitric acid — Specification (<i>third revision</i>)
265:1993	Hydrochloric acid — Specification (<i>fifth revision</i>)
266:1993	Sulphuric acid — Specification (<i>third revision</i>)
296:1986	Specification for sodium carbonate, anhydrous (<i>third revision</i>)
323:2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
1070:1992	Reagent grade water — Specification (<i>third revision</i>)
3025 (Part 2) : 2019/ ISO	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>)
11885	
4161: 1967	Specification for Nessler cylinders

3 REQUIREMENTS

3.1 Description

French chalk, technical, shall be obtained from the natural mineral (steatite, talc or soapstone). It shall be in the form of dry, fine, white powder, free from extraneous impurities, except small quantities of substances naturally occurring in the mineral. It shall have a smooth and soapy feel.

3.2 Free Moisture and Volatile Matter

The free moisture and volatile matter in the material, when determined according to the method prescribed in Annex A, shall not exceed 0.5 percent by mass.

3.3 Particle Size

The material shall have no residue when passed through 75 micron IS Sieve (*see* IS 460) or as mutually agreed to between the purchaser and the supplier.

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

Table 1 Requirements for French Chalk, Technical

Sl. No.	Characteristic	Requirement	Method Of Test, Ref to CI No.
(1)	(2)	(3)	(4)
i)	Loss on ignition, percent by mass, <i>Max</i>	4	B-3
ii)	Matter insoluble in hydrochloric acid, percent by mass, <i>Min</i>	95	B-4
iii)	Relative density at 27/27°C	2.7 to 2.9	B-5
iv)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.5	B-6 or Annex E
v)	pH (of 10 percent solution), <i>Max</i>	8.5	B-7

3.5 Additional Requirements

The material, required for use in textile, paper and pyrotechnic industries, shall, in addition to the requirements prescribed in Table 1, also comply with the requirements prescribed in Table 2 when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col 4 of the table.

Table 2 Additional Requirements for French Chalk, Technical, For Use in Textile Paper and Pyrotechnic Industries

Sl. No.	Characteristic	Requirement	Method of Test, Ref to CI No. in Annex B
(1)	(2)	(3)	(4)
i)	Grit, percent by mass, <i>Max</i>	0.02	B-8
ii)	Iron (as Fe ₂ O ₃), percent by mass, <i>Max</i>	0.3	B-9 & Annex D
iii)	*Whiteness, reflectance to blue light of wavelength 5040 Å, percent, <i>Min</i>	80	B-10

*This requirement shall be tested only when required by the purchaser.

3.6 Copper, Manganese and Their Compounds

The material, required for use in the preservation of rubber goods, shall contain not more than 0.05 percent by mass of either copper or manganese or their compounds calculated in terms of respective metals, when tested according to the method prescribed in Annex C.

4. PACKAGING AND MARKING

4.1 Packaging

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

4.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

- Name and grade of the material;
- Name of the manufacturer and his recognized trade mark, if any;

- c) Gross and net mass;
- d) Date of manufacture and;
- e) Batch number

4.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.2.1.1 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 there under, and the products may be marked with the standard mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be carried out and the criteria for conformity of the material in a lot to the requirements of the specification shall be as prescribed in Annex F.

ANNEX A (Clause 3.2)

DETERMINATION OF FREE MOISTURE AND VOLATILE MATTER

A-1 PROCEDURE

A-1.1 Introduce about 5 g of the material in a tared weighing bottle provided with a glass stopper and weigh. Remove the stopper and heat for 3 h at 105 to 110°C. Cool in a desiccator, replace the stopper and weigh. Repeat the process for one hour till constant mass is obtained.

A-2 CALCULATION

Free moisture and volatile matter, percent by mass = $\frac{M_1}{M_2} \times 100$

where

M_1 = loss in mass in g of the material after heating, and

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

ANNEX B (Clauses 3.4 and 3.5)

METHODS OF TEST FOR FRENCH CHALK, TECHNICAL

B-1 QUALITY OF REAGENTS

B-1.1 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 which affect the results of analysis.

B-2 PREPARED SAMPLE

B-2.1 Take about 50 g of the material in a china dish and dry to constant mass at 105 to 110°C. Cool and keep this prepared sample in a desiccator for subsequent tests.

B-3 DETERMINATION OF LOSS ON IGNITION

B-3.1 Procedure

Weigh accurately about 5 g of the prepared sample (*see B-2*) in a silica or porcelain crucible of 25 ml capacity and heat in a muffle furnace at 850 to 900°C. Cool to about 100°C and then finally transfer the crucible with its contents to a desiccator till it comes down to room temperature and weigh. Repeat the process until constant mass is obtained.

B-3.2 Calculation

$$\text{Matter insoluble in hydrochloric acid percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = loss in mass in g of the material after ignition, and

M_2 = mass in g of the prepared sample taken for the test.

B-4 DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

B-4.1 Reagents

B-4.1.1 *Rectified Spirit* — *See IS 323*

B-4.1.2 *Dilute Hydrochloric Acid* — Approximately 5 N.

B-4.2 Procedure

Weigh accurately about 0.5 g of the prepared sample (*see B-2*) and transfer to a 200 ml beaker. Add 5 ml of rectified spirit so that the material is thoroughly wetted, and then add 100 ml of dilute hydrochloric acid. Boil for 10 minutes. Filter off the insoluble matter through a tared sintered glass crucible (G No. 4). Wash the residue with water till it is free from chlorides and dry at $140 \pm 5^\circ\text{C}$ for 1 h. Cool in a desiccator and weigh till constant mass is obtained.

B-4.3 Calculation

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

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the prepared sample taken for the test.

B-5 DETERMINATION OF RELATIVE DENSITY

B-5.1 Apparatus

B-5.1.1 Relative Density Bottle — 50 ml capacity, having a ground-in capillary stopper with square end.

B-5.1.2 Vacuum Pump — with a receiver, capable of giving a vacuum of 100 mm.

B-5.2 Procedure

Weigh the empty, dry relative density bottle, introduce about 10 g of the material and weigh again, nearly fill the relative density bottle with water and do not insert the stopper. Place it on a small tared evaporating dish and keep the assembly in the exhausted receiver of the vacuum pump overnight (at least 16 h). Fill up the relative density bottle carefully with kerosine (*see* IS 1459) and insert the stopper. It is possible that a very small portion of the sample will run out on the sides of the stopper on inserting it. Wash down the particles thus forced out into the tared dish. Wipe off the excess of kerosine from the relative density bottle, bring it to 27°C and weigh.

B-5.2.1 Evaporate the contents of the tared dish on a boiling water-bath under cover, dry to constant mass at 105 to 110°C.

B-5.2.2 Empty the specific gravity bottle, fill it with kerosine and weigh after bringing it to 25°C.

B-5.3 Calculation

$$\text{Relative density at } 27/27^{\circ}\text{C} = \frac{M_2 - M_1 - M_4}{(M_5 - M_1) - (M_3 - M_2) - M_4} \times d$$

where

M_2 = mass in g of relative density bottle and sample,

M_1 = mass in g of empty relative density bottle,

M_4 = mass in g of residue in tared dish,

M_5 = mass in g of relative density bottle and water,

M_3 = mass in g of relative density bottle with sample and water, and

d = relative density of kerosine.

B-6 DETERMINATION OF CHLORIDES**B-6.1 Apparatus**

B-6.1.1 Nessler Cylinders — 50 ml capacity (*see* IS 4161).

B-6.2 Reagents

B-6.2.1 Dilute Nitric Acid — 5 N approximately.

B-6.2.2 *Silver Nitrate Solution* — 10 percent.

B-6.2.3 *Standard Hydrochloric Acid* — 0.0 1 N.

B-6.3 Procedure

Weigh accurately about 1 g of the *prepared sample* (see **B-2**) and shake with about 10 ml of water. Filter through a folded &her paper and wash thoroughly with water, collecting the filtrate in a Nessler cylinder. Add 10 ml of dilute nitric acid, dilute to 50 ml with water and add 1 ml of silver nitrate solution. Stir immediately with a glass rod and allow to stand for 5 min.

B-6.3.1 Place 1 ml of standard hydrochloric and 10 ml of dilute nitric acid in another Nessler cylinder. Dilute to 50 ml with water and add 1 ml of silver nitrate solution. Stir immediately with glass rod, and allow to stand for 5 min.

B-6.3.2 The material shall be considered to have not exceeded the limit for chloride prescribed in Table 1 if the opalescence produced in the test solution is not greater than that produced in the control test.

B-6.4 Alternate Method

Chlorides may alternatively be determined by instrumental test method as prescribed at Annex E.

B-7 DETERMINATION OF pH

B-7.1 Procedure

Shake 10.0 g of the prepared sample (see **B-2**) with 100 ml of freshly boiled and cooled water for one minute and determine the pH by means of a pH meter using glass electrode.

B-8 DETERMINATION OF GRIT

B-8.1 Procedure

Measure a depth of 50 mm from the bottom of a 500 ml beaker of about 8 cm diameter and make a mark on the beaker to indicate this height. Weigh accurately about 20 g of the material into the beaker. Break up any lump of the material with a rubber policeman but no hard object should be used for this purpose which may crush the particles of grit. Mix the material thoroughly with water and fill up to the mark. Allow to settle for exactly 1 min and pour off the milky water. Repeat the process until the supernatant water can be poured off practically clean at the end of one minute. Place the beaker on the water-bath until perfectly dry. Brush out the settled grit into a tared watch glass with camel hair brush and weigh. Examine the material under microscope after weighing to confirm the presence of grit.

B-8.2 Calculation — Express the mass of grit as the percentage on the mass of the material taken for the test.

B-9 DETERMINATION OF IRON

B-9.1 Apparatus

B-9.1.1 *Nessler Cylinders* — 50 ml capacity (see IS 4161).

B-9.2 Reagents

B-9.2.1 Concentrated Hydrochloric Acid — See IS 265

B-9.2.2 Ammonium Persulphate

B-9.2.3 Butanolic Potassium Thiocyanate Solution

Dissolve 10 g of potassium thiocyanate in 10 ml of water, Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

B-9.2.4 Standard Iron Solution

Dissolve 0.4911 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to 1000 ml. One millilitre of this solution is equivalent to 0.1 mg of iron (as Fe_2O_3).

B-9.3 Procedure

Digest 1.0 g of the prepared sample (*see* **B-2**) with 5 ml of water and 15 ml of concentrated hydrochloric acid for 1 h, on a hot-plate without boiling. Cover the beaker in which digestion is done, with a watch glass containing some water. After the digestion is over, wash the under-side of the watch glass with water into the beaker. Filter and wash the residue six times with hot water adding the washings to the filtrate. Make up the volume of the filtrate to 250 ml with water. Pipette out 25 ml of this solution in a Nessler cylinder, add 50 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution and make up the solution to 50 ml mark with water. Carry out a control test in another Nessler cylinder with 3 ml of standard iron solution and the same quantities of other reagents in exactly the same way as above.

B-9.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour obtained in the test with the material is not greater than that in the control test.

B-9.4 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at Annex D.

B-10 DETERMINATION OF WHITENESS**B-10.1 Principle**

The colour of the sample is compared with that of Lovibond magnesium oxide block with the help of a suitable apparatus.

B-10.2 Procedure

By means of a suitable arrangement, light falling on a Lovibond magnesium oxide block shall be reflected on a selenium cell connected to a galvanometer. The galvanometric deflection is proportional to light reflected. For comparison the reading obtained by magnesium oxide block is taken as 100. **B-10.1.1** Substitute the magnesium oxide block with a block prepared from a sample of the material. Measure the reflectance and report as whiteness (or reflectance) compared to that of magnesium oxide as 100.

ANNEX C
(Clause 3.6)
DETERMINATION OF COPPER AND MANGANESE

C-1 PREPARATION OF THE SOLUTION**C-1.1 Reagents**

C-1.1.1 *Anhydrous Sodium Carbonate* — See IS 296

C-1.1.2 *Concentrated Sulphuric Acid* — See IS 266

C-1.1.3 *Concentrated Nitric Acid* — See IS 264

C-1.2 Procedure

Grind the material fine enough to pass through a 106 micron IS Sieve. Weigh accurately 1 g of the finely ground material and mix it with 4 g of anhydrous sodium carbonate and fuse in a crucible until the melt is clear. Pour the hot fused mass in a dish and rotate so as to form a thin film, and then put the crucible in the dish with the fused mass. Add about 10 ml of water and heat on the water bath until the product is disintegrated. Remove the crucible and wash with water. Add 5 ml of concentrated hydrochloric acid in the crucible and digest hot, until the iron stain in it dissolves out completely. Add this acid to the contents in the dish. Evaporate to near dryness. Add a mixture of 6 ml of concentrated sulphuric acid and 1 ml of concentrated nitric acid. Evaporate to fumes of sulphur trioxide (SO₃). Cool and dilute to exactly 250 ml in a volumetric flask. Use this prepared solution for the tests under **C-2 and C-3**.

C-2 DETERMINATION OF COPPER**C-2.1 Apparatus**

C-2.1.1 *Nessler Cylinders* — 100 ml capacity see IS 4161

C-2.2 Reagents

C-2.2.1 *Ammonium Hydroxide* — approximately 1: 4 (v/v).

C-2.2.2 *Sodium Diethyldithiocarbamate Solution*

Dissolve 1 g of sodium diethyldithiocarbamate in 1000 ml of water. Keep in an amber coloured glass bottle and store in a dark place. This solution should not be used after two weeks.

C-2.2.3 *Gum Arabic Solution* — 1 percent (m/v).

C-2.2.4 *Standard Copper Solution*

Dissolve 0.3928 g of copper sulphate (CuSO₄.5H₂O) equivalent to 0.1000 g of copper in water. Make up the volume to 1000 ml with water. Pipette 100 ml of this solution into a 1000 ml volumetric flask and dilute up to the mark with water. One millilitre of this solution contains 0.01 mg of copper (as Cu). This dilute solution should be freshly prepared before use.

C-2.3 Procedure

Pipette 25 ml of the prepared solution into a small conical flask or beaker. Drop into it a small piece of litmus paper and make the solution just alkaline with ammonium hydroxide. Add 2.5 ml of ammonium hydroxide in excess and heat to boiling. Allow to stand on a water bath for 1 h to ensure complete precipitation of aluminium hydroxide and then filter through Whatman filter paper No. 1 or equivalent into a Nessler cylinder, washing the filter paper with two or three small portion of hot Water. To the solution in the Nessler cylinder add 1 ml of gum arabic solution, 10 ml of ammonium hydroxide and 10 ml of sodium diethyldithiocarbamate solution. Dilute to 100 ml mark and mix well. To the other Nessler cylinder containing a similar aliquot blank solution carried through the entire analysis in the same manner as the prepared solution, add equal amounts of the same reagents, dilute to about 90 ml and mix. Titrate this solution with standard copper sulphate solution from a 10 ml burette until its colour matches that of the prepared solution when diluted to the same volume. Mix well after each addition of standard copper sulphate solution.

C-2.3.1 If the colour produced with the prepared solution is too deep for comparison, a smaller aliquot portion of the prepared sample from the acid digestion shall be used.

C-2.3.2 In case the prepared solution is turbid, due to the presence of some interfering substances like iron, lead, bismuth or zinc, the following modified procedure is recommended.

C-2.3.2.1 If iron is present, precipitate the iron by adding a slight excess of ammonium hydroxide, filter and wash. Dissolve the ferric hydroxide precipitate in dilute nitric acid, precipitate with ammonium hydroxide, filter and wash. Combine the two filtrates, and dilute to a known volume. Follow rest of the procedure as under **C-2.3**.

C-2.3.2.2 If lead or bismuth is present, add a few drops of ferric chloride solution, boil and precipitate twice with ammonium hydroxide as under **C-2.3.2.1**. Follow rest of twice the procedure as under **C-2.3**.

C-2.3.2.3 If zinc is present, add sufficient ammonium hydroxide solution to prevent any turbidity developing after the addition of the reagent, and then follow the rest of the procedure as under **C-2.3**.

C-2.4 Calculation

$$\text{Copper, percent by mass} = \frac{0.01}{M} \times V$$

where

V = volume in ml of standard copper sulphate solution used for the titration, and

M = mass in g of the material taken under **C-1.2**

C-2.5 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD

C-2.5.1 Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

C-3 DETERMINATION OF MANGANESE

C-3.1 Apparatus

C-3.1.1 Nessler Cylinders — 100 ml capacity (see IS 4161).

C-3.2 Reagents

C-3.2.1 Phosphoric Acid — 85 percent by mass.

C-3.2.2 Potassium Periodate

C-3.2.3 Standard Potassium Permanganate Solution

Prepare a dilute solution of potassium permanganate by diluting to 50 ml in a volumetric flask a quantity of recently standardized solution in accordance with the following formula:

$$V = \frac{0.4551}{N}$$

where

V = volume in ml of standard potassium permanganate solution to be made up to a volume of 50 ml, and
 N = normality of the standard potassium permanganate solution.

NOTE — Do not keep the solution for more than one week. One millimetre of this solution is equivalent to 0.1 mg of manganese (as Mn).

C-3.3 Procedure

Transfer 100 ml of the prepared solution to a 250 ml beaker, evaporate to 75 ml and add 10 ml of phosphoric acid. Sprinkle 0.5 g of potassium periodate into the solution and bring it to boil. Go on adding 0.1 g more potassium periodate every 3 to 4 min until the permanganate does not seem to deepen any more and then add 0.1 g more. Place the beaker over a boiling water bath for 15 min. Cool and transfer to a Nessler cylinder and dilute to mark with water.

C-3.3.1 In the second Nessler cylinder, place 2 ml of standard potassium permanganate solution and 10 ml of phosphoric acid and dilute to mark with water. Mix the contents of the tubes well before matching.

C-3.3.2 The material shall be deemed to have passed the test if the colour produced by the test sample is not deeper than that produced by the standard solution.

ANNEX D**D-1 DETERMINATION OF IRON AND MANGANESE BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****D-1.1 Principle**

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get

excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

D-1.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 3. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Table 3 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

Sl. No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

D-1.3 Reagents and Solutions

D-1.3.1 Nitric acid (65 percent) Suprapure

D-1.3.2 Standard stock solution

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1000 $\mu\text{g}/\text{ml}$ of iron in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

D-1.3.3 Standard solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 $\mu\text{g}/\text{ml}$ solution. From this 5 $\mu\text{g}/\text{ml}$ solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

D-1.3.4 *Sample preparation*

Weigh about 2.5 g polyphosphoric acid sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument

D-1.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

D-1.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

D-1.5 Procedure**D-1.5.1** *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**D-1.5.2**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

D-1.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the iron and manganese in the sample solution.

NOTE – It is recommended that IS 3025(Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

D-1.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

ANNEX E

E-1 ION CHROMATOGRAPHY FOR CHLORIDES

E-1.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the *pH* of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

E-1.2 Equipment

E-1.2.1 *Anion guard column* — a protector of the separator column.

E-1.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

E-1.2.3 *Anion Suppressor device* — Anion micromembrane suppressor is used to analyse the data
Detector: Conductivity detector.

E-1.2.4 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

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

E-1.3 Reagents and Standards

E-1.3.1 *Glass or polyethylene sample bottles.*

E-1.3.2 *Distilled water or deionized water free from the anions of interest.*

E-1.3.3 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.

E-1.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid)

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

E-1.4 Standard solutions

E-1.4.1 Chloride — Dissolve NaCl, 1.6485 g in 1 l of reagent water

E-1.5 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.

E-1.6 Procedure

Dissolve between 1 to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard and deviation of retention force shall not exceed ± 10 percent of RT of calibration. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

E-1.7 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 F (Clause 5.1)

SAMPLING OF FRENCH CHALK, TECHNICAL

F-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

F-1.3 Sampling instrument shall be clean and dry when used.

F-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination and harmful contact and exposure.

F-1.5 The samples shall be placed in clean, dry, air-tight containers made of glass or other suitable material.

F-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.7 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.

F-1.8 The samples shall be stored in such a manner that during storage the properties of the material do not get affected.

F-1.9 Sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

F-2 SCALE OF SAMPLING FOR MATERIAL PACKED IN CONTAINERS

F-2.1 In a single consignment all containers of the material coming from the same source or supplier, shall constitute a lot.

F-2.2 Sub-lot

For the purpose of sampling, all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight .in accordance with col 1 and 2 of Table 4.

F-2.3 The number of containers to be sampled from each sub-lot is given in col 3 of Table 4. These containers shall be selected at random from the sub-lot. From each of these containers a small but equal increment of material shall be taken. In order to ensure the randomness of selection, necessary guidance may be obtained from IS 4905.

Table 4 Scale of Sampling for Containers
(Clauses F-2.2 and F-2.3)

No. of Containers in the Lot	No. of Sub-Lot	No. of Containers to be Sampled from Each Sub-Lot
(1)	(2)	(3)
Up to 10	1	All
11 to 200	2	5
201 to 500	3	10
501 to 2 001	4	15
2001 and above	5	20

F-2.4 All the increments from the sample containers in the same sub-lot shall be pooled together to constitute a gross sample so that there are as many gross samples as the sub-lots into which a lot has been

divided.

F-3 SAMPLING FROM BULK

F-3.1 Lot

The part or whole of the material in the bulk coming from the same source or supplier shall constitute a lot.

F-3.2 Sub-lot

For the purpose of sampling, the material in a lot shall be divided into a suitable member of homogeneous sub-lots of approximately equal weight in accordance with Table 5 by suitably marking the lines of demarcation on the surface of the bulk.

Table 5 Scale of Sampling for Bulk and Wagons
(Clauses F-3.2 and F-3.3)

Weight in the Bulk	No. of Sub-Lots	No. of Increments from Each Sub-Lot
(1)	(2)	(3)
Up to 200 tonnes	2	20
201 to 500	3	30
501 to 2000	4	40
2001 tonnes and over	5	50

F-3.3 Sampling from bulk shall be carried out as far as possible when the material is in motion. For this purpose the increments shall be drawn at regular intervals depending upon the rate of flow of the material of the sub-lot and the number of increments as given in col 3 of Table 5.

F-3.4 All the increments from a sub-lot shall be pooled together to constitute a gross sample.

F-4 SAMPLING FROM WAGONS

F-4.1 Lot

In a single consignment all the material coming from the same source or supplier shall constitute a lot.

F-4.2 Sub-lot

For the purpose of sampling all the wagons in a lot shall be divided into a suitable number, of homogeneous sub-lots of approximately equal weight in accordance with Table 5.

F-4.3 A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot.

F-4.4 All the increments taken from the selected wagons in sub-lot shall be pooled together to constitute a gross sample.

F-5 PREPARATION OF SAMPLES

F-5.1 All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the gross sample shall be about 3 kg. (If the gross sample is less than this, additional number of increments shall be drawn so as to make up the required quantity.

F-5.2 The gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory samples, one for the purchaser, another for the supplier and the third as the referee sample, each duly placed in the sample container.

F-6 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

F-6.1 One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory if the laboratory sample satisfies all the requirements of this specification.

F-6.2 The lot shall be declared to conform to the requirements of this specification if all the sub-lots are found satisfactory in **F-6.1**.