

भारतीय मानक मसौदा
सोडियम बाईकारबोनेट — विशिष्टि
(आईएस 2124 का तीसरा पुनरीक्षण)

Draft Indian Standard
SODIUM BICARBONATE — SPECIFICATION
(Third Revision of IS 2124)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 17th December 2022

FOREWORD

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.

The specifications for pure and analytical reagent grade, and that for refined grade were first issued in 1954. Subsequently, in the year 1962, these were revised with a view to upgrade the refined grade and amalgamate the two standards.

In the first revision the requirements for heavy metals and arsenic content were modified for the refined grade, and for the pure grade, the requirement for matter insoluble in water and dilute ammonium hydroxide were deleted. Also the limits for copper content have been prescribed as per PFA rules for both the grades. Further, the analytical reagent grade of the material was modified on the basis of the prevailing standards for laboratory chemicals. In the second revision, calorimetric method for determination of chlorides, heavy metals, phosphates, copper, silicates and nephelometric method for determination of sulphate were incorporated as alternative methods, while some other modifications were made in other test methods also.

In this revision, instrumental test methods for the determination of arsenic, copper, chlorides, lead, sulphates, iron, magnesium and calcium have been added as alternate test methods. Also, Packing and Marking clause has been updated.

Sodium bicarbonate is used as baking powder, reagent in analytical chemistry; in the manufacture of effervescent salts and beverages. It also finds use in artificial mineral waters, gold and platinum plating, tanning industry, treating wool and silk, fire extinguishers and ceramics.

For particle size, sieves conforming to IS 460 is prescribed. Where IS sieves are not available, other equivalent standard sieves as judged by aperture size, may be used.

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 sodium bicarbonate.

2 REFERENCES

The Indian Standards listed 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 Indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 2021	Hydrochloric Acid — Specification (<i>fifth revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
323 : 2009	Rectified spirit for industrial use — Specification (second revision)
460 (Part 1) : 2020	Test Sieves — Specification Part 1 Wire Cloth Test Sieves (<i>fourth revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third 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>)
4905:2015/ ISO 24153: 2009	Random sampling and randomization procedures (<i>first revision</i>)

3 GRADES

3.1 There shall be three grades of the material, namely, Pure, Analytical Reagent and Refined.

3.1.1 *Pure Grade* — Suitable for the fine chemical and pharmaceutical industry.

3.1.2 *Analytical Reagent Grade Suitable as a reagent.*

3.1.3 *Refined Grade* — Suitable for aerated water and baking powder industry.

4 REQUIREMENTS

4.1 Description

The material of all the three grades shall be in the form of small, opaque, monoclinic crystals or as a white, minutely crystalline powder, free from dirt and other foreign matter.

4.1.1 The pure grade material suitable for the manufacture of effervescent powders shall have the particle size distribution as agreed to between the purchaser and the supplier.

4.2 The material, when tested according to the methods prescribed in Annexes A to R, shall also comply with the requirements specified in Table 1.

4.3 Criteria for ECO Mark for Refined Grade

4.3.1 General Requirements

4.3.1.1 Sodium bicarbonate shall meet the requirements as specified under **4.1** and **4.2**.

4.3.1.2 The product manufacturer must produce the consent clearance as per the provisions of Water (PCP) Act, 1974, Water (PCP) Cess Act, 1977 and Air (PCP) Act, 1981 along with the authorization if required under the Environment Act, 1986 and the rules made thereunder to Bureau of Indian Standards while apply for the ECO Mark and the product shall also be in accordance with the Prevention of Food Adulteration Act, 1954 and the rules made thereunder.

4.3.1.3 The product/packing may display in brief the criteria based on which the product has been labelled as Environment Friendly.

4.3.1.4 The material used for product packing shall be recyclable or biodegradable.

4.3.1.5 The date of manufacture and date of expiry shall be declared on the product package by the manufacturer.

4.3.1.6 The product package or leaflet accompanying it may display instructions of proper use and storage so as to maximize the product performance, safety and minimize wastage.

5 PACKING AND MARKING

5.1 Packing

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

5.2 Marking

The packages shall be securely closed and legibly marked with the following information:

- a) Name and grade of the material,
- b) Indication of the source of manufacture,
- c) Mass of the material in the packaging, and
- d) For analytical reagent grade material, the list of requirements given in col 4 of Table I

5.2.1 BIS Certification Marking

The packages may also be marked with the Standard Mark.

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

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to that standard shall be determined in accordance with the method prescribed in Annex S.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade 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.

Table 1 Requirements for Sodium Bicarbonate

(Clause 4.2)

Sl. No.	Characteristic	Pure Grade	Requirement		Method of test, Ref to Annex
			Analytical Reagent Grade	Refined Grade	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Total alkalinity (as NaHCO ₃) percent by mass, <i>Min</i>	99.0	99.8	99.0	A
ii)	pH (1 percent aqueous solution), <i>Max</i>	8.6	8.4	8.8	B
iii)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.01	0.001	0.06	C & T
iv)	Insoluble matter, percent by mass, <i>Max</i>	0.05	0.010	0.100	D
v)	Sulphates (as SO ₄) percent by mass, <i>Max</i>	0.05	0.003	0.07	E & T
vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.001 5	0.0005	0.004	F & S
vii)	Heavy metals (as Pb), ppm, <i>Max</i>	5	5	5	G & S
viii)	Arsenic (as As), ppm, box	1.5	0.4	1.5	H & S
ix)	Ammonium compounds (as NH ₄) ppm, <i>Max</i>	To pass test	5	—	J
x)	Phosphates (as P ₄). Percent by mass, <i>Max</i>	—	0.0005	—	K
xi)	Calcium and magnesium (as Ca), percent by mass, <i>Max</i>	—	0.015	—	L & S
xii)	Copper (as Cu), ppm, <i>Max</i>	30	—	30	M
xiii)	Nitrates (as NO ₃), percent by mass, <i>Max</i>	—	0.001	—	M
xiv)	Silicates (as SiO ₂), percent by mass, <i>Max</i>	—	0.005	—	N
xv)	Potassium (as K), percent by mass, <i>Max</i>	—	0.01	—	O
xvi)	Substances reducing iodine, percent by mass, <i>Max</i>	—	0.0065	—	P

ANNEX A*[Clause 4.2 and Table 1, Sl. No. (i)]***DETERMINATION OF TOTAL ALKALINITY****A-1 REAGENTS****A-1.1 Standard Hydrochloric Acid — 1.0 N.****A-1.2 Standard Hydrochloric Acid — 0.1 N.****A-1.3 Methyl Orange or Bromothymol Blue Indicator****A-2 PROCEDURE**

Weigh accurately 4.4 g of the material and dissolve in 100 ml of freshly boiled and cooled water contained in a conical flask. Add 5 drops of the methyl orange or bromothymol blue indicator and 50 ml of standard hydrochloric acid (1 N). Stir the contents thoroughly and further titrate with standard hydrochloric acid (0.1 N) until the colour of the indicator solution changes from orange to red (blue to green in case of bromothymol blue). Calculate the total volume in terms of hydrochloric acid (1 N).

A-3 CALCULATION

Total alkalinity (as NaHCO_3), percent by mass = $\frac{8.4 VN}{M}$

where

V = volume in ml of standard hydrochloric acid (1N) required for titration,

N = normality of standard hydrochloric acid used, and

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

ANNEX B*[Clause 4.2 and Table 1, Sl. No. (ii)]***DETERMINATION OF pH****B-1 GENERAL**

Two methods have been prescribed for determination of pH: electrometric method (Method A) and indicator method (Method B). However, in case of dispute, the electrometric method shall be considered as the referee method.

B-2 METHOD A (ELECTROMETRIC METHOD)**B-2.1 Procedure**

The determination shall be made on 1 percent (m/v) solution by any pH meter with glass electrode.

B-3 METHOD B (INDICATOR METHOD)**B-3.1 Reagents**

B-3.1.1 Bromothymol Blue Indicator

Dissolve 0.1 g in 100 ml of rectified spirit conforming to IS 323.

B-3.2 Procedure

Weigh 1 g of the material and dissolve in 100 ml of water. Take 10 ml of this solution in a hard glass test tube and add 0.5 ml of bromothymol blue indicator. Compare the colour produced with a series of buffer solutions of known *pH* in the range 8.0 to 9.0. Report as *pH*, the *pH* of the buffer solution which gives the closest match with the colour produced with the sample.

NOTE —Standard calibrated glass discs may also be used for the determination of *pH*.

ANNEX C

[Clause 4.2 and Table 1, Sl No. (iii)]

DETERMINATION OF CHLORIDES**C-1 GENERAL**

Two methods have been prescribed for determination of chlorides, namely, volumetric (Method A) and spectrophotometric (Method B). In case of dispute Method A shall be referee method

C-2 METHOD A (VOLUMETRIC METHOD)**C-2.1 For Pure and Refined trades****C-2.1.1 Reagents**

C-2.1.1.1 Concentrated nitric acid — See IS 264.

C-2.1.1.1 Standard silver nitrate solution — 0.1 N.

C-2.1.1.3 Nitrobenzene

C-2.1.1.4 Standard ammonium thiocyanate solution — 0.1 N.

C-2.1.1.5 Ferric ammonium sulphate indicator — Approximately 5 percent.

C-2.1.2 Procedure

Weigh accurately about 20 g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator. At the end point a permanent faint reddish brown colouration appears.

C-2.1.2 Procedure

Weigh accurately about 20 g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate with

standard ammonium thiocyanate solution using ferric ammonium sulphate indicator. At the end point a permanent faint reddish brown colouration appears.

C-2.1.3 Calculation

$$\text{Chlorides (as Cl) percent by mass} = \frac{3.547 (10 N_1 - V N_2)}{M}$$

where

N_1 = normality of standard silver nitrate solution,

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

N_2 = normality of standard ammonium thiocyanate solution, and

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

C-2.2 For Analytical Reagent Grade

Two methods have been prescribed for determination of chlorides, namely, volumetric (Method A) and spectrophotometric (Method B). In case of dispute Method A shall be referee method.

C-2.2.1 Apparatus

C-2.2.1.1 Nessler cylinders — 100 ml capacity.

C-2.2.2 Reagents

C-2.2.2.1 Concentrated nitric acid — See IS 264.

C-2.2.2.2 Silver nitrate solution — Approximately 4 percent.

C-2.2.2.3 Standard chloride solution

Dry sodium chloride at 250°C before making the solution. Dissolve 1.649 g of sodium chloride in water and make up the volume to 1000 ml. Pipette out 10 ml from this solution and dilute with water to 1000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

C-2.2.3 Procedure

Dissolve 1.000 g of the material in 30 ml of water and add slight excess of concentrated nitric acid. Boil the solution, cool and add 1 ml of silver nitrate solution. Transfer into a Nessler cylinder and make up the volume to 100 ml mark with water. Carry out a control test using 1 ml of standard chloride solution and the same quantities of other reagent in the same total volume of the reaction mixture. Compare the turbidity in both the cylinders after 5 min.

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

C-3 METHOD B (SPECTROPHOTOMETRIC METHOD)

C-3.1 Outline of the Method

To a neutralized solution of sodium bicarbonate, addition of mercuric thiocyanate and ferric ammonium sulphate, produces a highly coloured ferric thiocyanate complex, the intensity of which is proportional to the chloride content. The absorbance of this colour is measured at 450 nm wave length.

C-3.2 Apparatus

C-3.2.1 Standard Laboratory Apparatus

C-3.2.2 Spectrophotometer or Photocolorimeter

C-3.2.3 Platinum Evaporating Dish — 60 ml capacity.

C-3.3 Reagents

C-3.3.1 Ferric Ammonium Sulphate Solution (0.25 mol/l)

Dissolve 49.02 g of Ferric Ammonium Sulphate Dodecahydrate $[\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ in 203 ml of dilute nitric acid (about 1.5 mol/l) and dilute to 500 ml with distilled water.

C-3.3.2 Mercury Thiocyanate Solution — Saturated solution in absolute alcohol.

C-3.3.3 Standard Sodium Chloride Solution

Weigh accurately 0.1650 g of previously dried (at 500°C) and cooled A.R. sodium chloride, dissolve in a beaker in distilled water and quantitatively transfer into a 1 l volumetric flask, dilute to mark and mix well. 1 ml of this solution = 100 µg of Cl.

C-3.3.3.1 Dilute Standard Sodium Chloride Solution

Transfer 50 ml of standard sodium chloride solution into a 500 ml volumetric flask, dilute with distilled water to the mark and mix well. 1 ml of this solution = 10 µg of Cl.

C-3.3.4 Sulphuric Acid Solution

Approximately 1 mol/l — Pour 56 ml of concentrated sulphuric acid (AR quality) into 944 ml of distilled water slowly, mix and cool.

C-3.4 Procedure

C-3.4.1 Calibration

Into a series of six 50 ml volumetric flasks, transfer standard sodium chloride solution as given below:

<i>Standard Sodium Chloride ml</i>	<i>Corresponding Mass of Chloride (as Cl) µg</i>
0 (Compensation)	0
2	20
4	40
6	60
8	80
10	100

Add to each, 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in that order. Dilute to mark and mix well. Allow to stand for 15 min, transfer to the cell of spectrophotometer and measure absorbance of 450 nm wave length with compensation solution in reference cell.

Plot chloride content in micrograms as abscissa vs. corresponding absorbance values as ordinates.

C-3.4.2 Determination

Weigh suitable mass of sodium bicarbonate sample containing 50 to 150 g of chloride (as Cl) correct to 1 mg and transfer into the platinum evaporating dish wet this with 15 to 20 ml of distilled water and slowly neutralize with sulphuric acid solution add a few drops in excess. Evaporate to nearly dryness on hot water bath. Add 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml volumetric flask, cool and make up to the mark with distilled water and mix well.

Filter through Whatman No. 41 filter paper and discard a small volume of initial filtrate.

Transfer suitable volume (25 to 40 ml) into 50 ml volumetric flask add 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in that order, dilute to mark with distilled water and mix well.

Allow to stand for 15 min, transfer to the cell of spectrophotometer and measure absorbance at 450 nm wave length with distilled water in the reference cell.

C-3.4.3 Blank Test

Carry out a blank test using exactly the same procedure as used for determination using same quantities of all reagents but omitting the test solution. Instead of test solution use distilled water.

C-3.4.4 Calculations

The chloride content is calculated from the formula:

$$\text{Chloride percent by mass (as Cl), } \frac{M_1 - M_2}{M_1 - 10000}$$

where

M_1 = mass in micrograms of chloride corresponding to absorbance of test solution,

M_2 = mass in micro grams of chloride corresponding to absorbance of blank solution, and

M_0 = mass in grams of sodium bicarbonate in the test portion of the aliquot used for colour development.

C-6 Alternative Method

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

ANNEX D

[Clause 4.2 and Table 1, SI No. (iv)]

DETERMINATION OF INSOLUBLE MATTER

D-1 Procedure

Weigh accurately about 10 g of the material, transfer it to a 400 ml beaker, add about 200 ml of freshly boiled water and boil the resulting solution for 15 min. Filter any undissolved residue through a tared Gooch or a sintered glass crucible (G No.4) and wash the residue free from soluble salts with water. Collect the filtrate and washings in a 1 l graduated flask and dilute to mark. Preserve the solution so obtained for the test under Annex L.

Dry the crucible along with the residue to constant mass at 105 to 110°C.

D-2 CALCULATION

$$\text{Insoluble matter, percent by mass} = \frac{100 \times M_1}{M_2}$$

M_1 = mass in g of the residue, and

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

ANNEX E

[Clause 4.2 and Table 1, Sl No. (v)]

DETERMINATION OF SULPHATES

E-1 GENERAL

Two methods have been prescribed for determination of sulphates, namely, gravimetric method (Method A) and turbidimetric method (method B).

E-2 METHOD A (GRAVIMETRIC METHOD)

E-2.1 For Pure and Refined Grades

E-2.1.1 Reagents

E-2.1.1.1 *Concentrated hydrochloric acid* — See IS 265.

E-2.1.1.2 *Barium chloride solution* — Approximately 10 percent.

E-2.1.2 Procedure

Dissolve 10 to 15 g of the material, accurately weighed, in about 100 ml of water. Carefully add concentrated hydrochloric acid to make it acidic, and boil to drive off completely the carbon dioxide formed. Cool and filter, if necessary, through a folded filter paper. Wash the filter paper thoroughly with water, collecting the filtrate and washings in a 500 ml beaker. Dilute to 250 ml, boil and add 10 ml of hot barium chloride solution to the hot solution. Boil again for a few minutes. Allow the contents of the beaker to stand for 4 h and filter through a tared sintered glass crucible (G No. 4) or a Gooch crucible. Wash the precipitate with hot water till it is free from chlorides. In case of dispute Method A shall be referee method and dry to constant mass at 105 to 110°C.

E-2.1.3 Calculation

$$\text{Sulphates (as SO}_2\text{), percent by mass} = \frac{41.15 M_1}{M}$$

where

M_1 = mass in g of the precipitate, and

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

E-2.2 For Analytical Reagent Grade

E-2.2.1 Apparatus

E-2.2.1.1 *Nessler cylinders* — 50 ml capacity.

E-2.2.2 Reagents**E-2.2.2.1 Concentrated hydrochloric acid** — See IS 265.**E-2.2.2.2 Barium chloride solution**

Approximately 10 percent (m/v), prepared by dissolving 10 g BaCl₂ (GR) in 100 ml water or by dissolving proportionate amount of BaCl₂ in reduced volume of water. If any turbidity observed, filter through sintered glass crucible (4) under section and preserved the solution in glass bottle

E-2.2.2.3 Standard sulphate solution

Dissolve 0.1814 g of potassium sulphate in water and make up the volume to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of sulphate (as SO₄).

E-2.2.3 Procedure

Dissolve 2.000 g of the material in 20 ml of water, add 5 ml of concentrated hydrochloric acid and evaporate to dryness on a water-bath. Dissolve the residue in 20 ml of water and add a few drops of concentrated hydrochloric acid and 2 ml of barium chloride solution. Transfer to a Nessler cylinder and dilute to mark. Carry out a control test in another Nessler cylinder using 0.06 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

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

E-3 METHOD B (TURBIDIMETRIC METHOD)**E-3.1 Outline of the Method**

Neutralization of the test portion (for alkaline salts) with hydrochloric acid and making it acidic with slight excess of hydrochloric acid and precipitation of sulphates as Barium sulphate under well defined conditions. Measurement of turbidity using a spectrophotometer (or photocolourimeter) at 470 nm. This method is applicable when sulphate content is less than 0.1 percent by mass.

E-3.2 Reagents**E-3.2.1 Barium Chloride**

A.R. Barium chloride dihydrate of uniform particle size between 0.50 and 1.25 mm, standardized by screening. It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

E-3.2.2 Sodium Carbonate Solution

Approximately 0.5 mol/l.

E-3.2.3 Hydrochloric Acid Solution

Approximately 1 mol/l.

E-3.2.4 Hydrochloric Acid Solution

Approximately 6 mol/l.

E-3.2.5 Standard Sulphuric Acid Solution (0.05 mol/l)

Prepare approximately 1 mol/l solution by pouring 56 ml of concentrated sulphuric acid into 944 ml of distilled water. Dilute 50 ml of this solution to 1 000 ml in a volumetric flask which gives approximately 0.05 mol/l solution. Standardize this against standard sodium carbonate solution (0.05 mol/l) prepared by dissolving A.R. Sodium carbonate dried at 260-270°C. Dissolve 1.324 9 g in distilled water, transfer quantitatively into a 250 ml flask, make up to mark and mix well.

E-3.2.5.1 *Standard sulphuric acid solution*

0.100 g SO₂ per litre - Transfer 20.8 ml of 0.05 mol/l standard sulphuric acid solution into a 1 l volumetric flask, dilute to mark with distilled water and mix well. 1 ml of this solution = 0.100 mg SO₄.

E-3.3 **Apparatus**

E-3.3.1 *Standard Laboratory Apparatus*

E-3.3.2 *Platinum Evaporating Dish*

60 ml capacity.

E-3.3.3 *Spectrophotometer or Photoelectric Absorptiometer*

Fitted with filters giving only a negligible transmission below 450 nm and above 550 nm.

E-3.4 **Procedure**

E-3.4.1 *Calibration*

Into each of a series of nine 50 ml one-mark volumetric flasks, place the volumes of standard sulphuric acid solution as shown in the following table.

<i>Sulphuric Acid Solution, ml</i>	<i>Corresponding Mass of SO₄, mg</i>
0 (Compensation)	0
5.0	0.5
10.0	1.0
15.0	1.5
20.0	2.0
25.	2.5
30.0	3.0
35.0	3.5
40.0	4.0

To each flask, add 2 ml of sodium carbonate solution and 5 ml of hydrochloric acid solution, stir, dilute to mark and mix well. Transfer 25.0 ml of each of the solution except the first to separate 100 ml dry beaker each containing 0.15 g barium chloride. Stir by hand for 1 min at the rate of 2 revolutions per second. Allow to stand for 15 min at 27 ± 2°C.

NOTE — Stagger the test in order to adhere to the contact times indicated.

Stir by hand and transfer a sufficient quantity of each of the solutions to a cell of the spectrophotometer and measure the absorbance at 470 nm wavelength. Use compensation solution to adjust optical zero of spectrophotometer. Plot a graph with SO₄ content in mg as abscissae Vs corresponding absorbance values as ordinates. It should be noted that the calibration curve is linear only above 0.5 mg SO₄.

E-3.4.2 *Determination*

E-3.4.2.1 *Test portion*

Weigh to the nearest 1 mg a quantity of the test sample of sodium bicarbonate containing 1 to 4 mg of SO₄ and transfer into a platinum evaporating dish. Wet the sample with 10 to 20 ml of distilled water and neutralize with hydrochloric acid solution (**E-3.2.4**) and add a few drops in excess. Heat the evaporating dish on a water bath to almost dryness and add 5 ml of hydrochloric acid solution (**E-3.2.3**) and 20 to 30 ml distilled water. Heat again on water bath for a few minutes and transfer quantitatively into a 50 ml volumetric flask. Cool, make up to the mark and mix well. Filter through a Whatman No. 41 filter paper, discarding a small volume of the initial filtrate.

E-3.4.2.2 *Turbidimetric reaction*

Transfer 25 ml filtrate (**E-3.4.2.1**) into a 100 ml beaker containing 0.15 g of barium chloride stir by hand for 1 min at the rate of 2 revolutions per second. The barium chloride should then be in complete solution. Leave undisturbed for 15 min at 27±2°C.

E-3.4.2.3 *Turbidity measurement*

Transfer a sufficient quantity of the test solution to a cell of the spectrophotometer (same size as the one used for calibration) and use to adjust optical zero of the spectrophotometer. Stirr by hand the turbidity developed in the solution (**E-3.4.2.2**). Transfer it into a cell (same sizes as the one used for calibration) and measure absorbance at 470 nm wavelength.

NOTE — If the turbidity developed in the test portion is beyond the limits of the calibration range, dilute an aliquot filtrate (**E-3.4.2.1**) to 50 ml and use 25 ml of this diluted solution to develop turbidity. In this case use the diluted solution in the reference cell.

E-3.4.2.4 *Blank test*

At the same time, carryout a blank test following the same procedure and using the same quantities of all reagents used for determination, but by replacing the test portion by 5 ml of standard sulphuric acidsolution corresponding to 0.5 mg of SO₄ to allow operation in the linear part of the calibration curve.

E-3.5 Calculations

The sulphate content is obtained by the following formula:

$$\text{Sulphate (as SO}_4\text{), percent by mass} = \frac{M_1 - M_2 \times 1000}{M_0 \times 10}$$

where

M_1 = mass of sulphate in milligrams corresponding to absorbance of the test portion,

M_2 = mass of sulphate in milligrams corresponding to the absorbance of the blank solution after deduction of 0.5 mg of SO₄ added (that is Graph value — 0.5), and

M_0 = mass of the sodium bicarbonate in grams corresponding to aliquot used for turbidity development.

C-6 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at Annex T.

ANNEX F

[Clause 4.2 and Table 1, Sl No. (vi)]

TEST FOR IRON

F-1 APPARATUS

F-1.1 Nessler Cylinders — 100 ml capacity.

F-2 REAGENTS

F-2.1 Concentrated Hydrochloric Acid — See IS 265.

F-2.2 Ammonium Persulphate

F-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.

F-2.4 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid (10 percent v/v) and dilute with water to 1000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of iron (as Fe).

F-3 PROCEDURE

Dissolve 1.000 g of the material in 25 ml of water and neutralize with 5 ml of concentrated hydrochloric acid. Add 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer completely into a Nessler cylinder. Add 15 ml of butanolic potassium thiocyanate solution and shake vigorously for 30 s and allow it to separate. Carry out a control test in another Nessler cylinder using 0.2 ml of the standard iron solution for the pure grade 1.0 ml for the analytical reagent grade and 4 ml for the refined grade respectively in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced.

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

F-4 Alternative Method

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

ANNEX G

[Clauses 4.2 and U-5.1.2 and Table 1, Sl No. (vii)]

TEST FOR HEAVY METALS

G-1 GENERAL

Two methods have been prescribed for determination of heavy metals as lead, namely, visual colour comparison (Method A) and spectrophotometric method (Method B). In case of dispute Method A shall be referee method.

G-2 METHOD A (VISUAL COLOUR COMPARISON)

G-2.1 Apparatus

G-2.1.1 *Nessler Cylinders* — 50 ml capacity.

G-2.2 Reagents

G-2.2.1 *Concentrated Hydrochloric Acid* — See IS 265.

G-2.2.2 *Standard Lead Solution*

Dissolve 1.598 5 g of lead nitrate in water and make up the volume to 1000 ml. Transfer exactly 10 ml of the solution into a volumetric flask and dilute it against with water to 1000 ml mark. One millilitre of this solution contains 0.01 mg of lead (as Pb). The solution should be freshly prepared.

G-2.2.3 *Acetic Acid* — Approximately 1 N.

G-2.2.4 *Hydrogen Sulphide Solution* — Saturated.

G-2.3 Procedure

Weigh 4.000 g of the material and add 15 ml of water. Add 6 ml of concentrated hydrochloric acid and evaporate to dryness on a water-bath. Dissolve the residue in water and transfer this solution into a Nessler cylinder. Dilute it with water to 30 ml and add 1 ml of acetic acid and 10 ml of hydrogen sulphide solution. In a second Nessler cylinder, carry out a control test using 2 ml of standard lead solution for pure grade, analytical reagent grade and refined grade in place of the material, and the same quantities of other reagents. Dilute the contents of each tube to 50 ml and shake well. Compare the colour produced.

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

G-3 METHOD B (SPECTROPHOTOMETRIC METHOD)**G-3.1 Outline of the Method**

Lead reacts with dithizone (diphenylthio Carbazone) to form a pink coloured complex in chloroform solution. The complex is separated by extraction with chloroform from an aqueous Ammonia-Cyanide-Sulphite solution. The absorbance of the extracted complex is measured using a spectrophotometer at 510 nm wavelength.

G-3.2 Apparatus

G-3.2.1 *Standard Laboratory Apparatus*

G-3.2.2 *Separating Funnels* — 250 ml capacity.

G-3.2.3 *Spectrophotometer or Photocolorimeter*

G-3.3 Reagents**G-3.3.1** *Standard Lead Solution*

Dissolve 0.799 2 g of A.R. lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in distilled water in a beaker, transfer quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark and mix well. One ml of this solution contains 1 mg lead.

G-3.3.1.1 *Dilute Standard Lead Solution*

Transfer 10 ml of the above solution (**G-3.3.1**) into a 1 l volumetric flask, dilute to marked and mix well. 1 ml of this solution contains 10 µg of lead.

G-3.3.2 *Chloroform* — Reagent grade chloroform.

G-3.3.3 *Dithizone Reagent* — 50 mg/l solution in chloroform. Dissolve 5 mg of the reagent in 100 ml of chloroform.

G-3.3.4 Ammonia-Cyanide Sulphite Reagent to 3 ml of 10 percent potassium cyanide solution

CAUTION- Potassium cyanide is highly poisonous) add 35 ml of concentrated ammonia and dilute to 100 ml. Add 0.15 g of sodium sulphite and mix well.

G-3.3.5 *Dilute Hydrochloric Acid Solution* —Approximately 1 mol/l.

G-3.3.6 Hydrochloric Acid Solution —Approximately 6 mol/l.

G-3.4 Procedure**G-3.4.1 Calibration**

Transfer dilute standard lead solution to a series of six 250 ml separating funnels as given below:

<i>Standard Lead Solution, ml</i>	<i>Corresponding Lead Content μg</i>
0 (Compensation)	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

Add to each funnel 75 ml of ammonia-cyanide sulphite reagent solution and cautiously adjust pH to 9.5 using a pH meter (equipped with a combined electrode). This operation must be carried out slowly because, if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume cupboard during this operation is necessary. Then add 7.5 ml of dithizone reagent to each separating funnel. Maintain time difference among each such that the time after addition of dithizone reagent and final absorbance measurement is same for all. Then add 17.5 ml of chloroform and shake for 1 min to extract the lead complex into the chloroform layer, allow the phases to separate. Transfer the chloroform layer (lower one) through a cotton plug (placed in the stem of the separating funnel) into a 1 cm cell of the spectrophotometer. Immediately replace the lid of the cell. Measure absorbance (using chloroform extract of compensation solution, in reference cell) at 510 nm wavelength.

Plot a graph using 20-25 ml of lead (in 25 ml of chloroform) as abscissae and corresponding absorbance values as ordinates.

G-3.4.2 Determination

Weigh accurately correct to 1 mg about 10 g of Sodium bicarbonate sample (the quantity to be weighed, should be such that it contains 20 to 100 μg of lead) and transfer to 50 ml beaker dissolve in about 20-25 ml distilled water and transfer quantitatively into a 250 ml separating funnel and rinse the beaker twice with 5 ml portions of distilled water. 75 ml of Ammonia-Cyanide-Sulphite reagent and then by the cautious addition of dilute hydrochloric acid solution adjust pH to 9.5. Use a pH meter with combined electrode to adjust pH. This operation must be carried out slowly because if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume cupboard during this operation is necessary. Now add 7.5 ml of dithizone reagent to the separating funnel followed by 17.5 ml of chloroform. Shake for 1 min to extract the lead into the chloroform layer. Allow the phase separate. Transfer the chloroform layer through a cotton plug (placed in the separating funnel stem) into a 1 cm cell of the spectrophotometer and measure

the absorbance at 510 nm with chloro form in the reference cell. Read the corresponding lead content in micrograms from the calibration graph.

G-3.4.3 Blank Test

Carry out a blank test exactly as described in **G-3.4.2** but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph.

G-3.5 Calculations

Calculate the lead content in the material from the relation given below:

$$\text{Heavy metals (as Pb), ppm} = \frac{M_1 - M_2}{M_0}$$

where

M_1 = mass of lead in the test solution as obtained from graph in μg ,

M_2 = mass of lead in blank as obtained from graph in μg , and

M_0 = mass of sample taken for analysis in grams.

G-4 Alternative Method

Lead may alternatively be determined by instrumental test method as prescribed at Annex S.

ANNEX H

[Clauses 4.2 and U-5.1.2 and Table 1, Sl No. (Viii)]

TEST FOR ARSENIC

H-1 PROCEDURE

Dissolve 1 .000 g of the material in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088 using 0.001 98 mg of arsenic trioxide (As_2O_3) for pure grade and refined grade and 0.000 528 mg for analytical reagent grade for preparing the comparison stain.

G-4 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed at Annex-S

ANNEX J

[Clause 4.2 and Table 1, Sl No. (ix)]

TEST FOR AMMONIUM COMPOUNDS

J-1 APPARATUS

J-1.1 Nessler Cylinders — 50 ml capacity.

J-2 REAGENTS

J-2.1 Sodium Hydroxide Solution — 10 percent.

J-2.2 Devarda's Alloy Containing 45 Parts Aluminium, 50 Parts Copper and 5 Parts Zinc Heat the aluminium in a Hessian crucible in a furnace until the aluminium begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible and heat the mixture for a few moments and stir with an iron rod. Allow it to cool slowly with the cover on and pulverize the crystallized mass.

J-2.3 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly, while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

J-2.4 Standard Ammonium Chloride Solution

Dissolve 0.296 g of ammonium chloride (NH_4Cl) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this diluted solution is equivalent to 0.01 mg of ammonium (as NH_4).

J-3 PROCEDURE**J-3.1 For Pure Grade**

Dissolve 1.0 g of the material in 10 ml of water, add 20 ml of sodium hydroxide solution and warm the reaction mixture. The material shall be considered to have passed the test if no ammonia is detected in the vapours on testing with moistened litmus paper.

J-3.2 For Analytical Reagent Grade

Dissolve 2.0 g of the material in 45 ml of water and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible, previously washed with sodium hydroxide solution. Take 6 ml of the filtrate, dilute with water to 50 ml and add 2 ml of Nessler solution. In a control test, having an equal volume of the solution and containing 1 ml of standard ammonium chloride solution and 1.5 ml of sodium hydroxide solution, add 2 ml of Nessler solution.

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

ANNEX K

[Clause 4.2 and Table 1, SI No. (x)]

TEST FOR PHOSPHATES**K-1 GENERAL**

Two methods have been prescribed for determination of phosphates, namely, visual colour comparison (Method A) and spectrophotometric method (Method B). In case of dispute Method A shall be referee method.

K-2 METHOD A (VISUAL COLOUR COMPARISON)**K-2.1 Apparatus**

K-2.1.1 *Nessler Cylinders* —50 ml capacity.

K-2.2 Reagents

K-2.2.1 *Dilute Sulphuric Acid* —Approximately 5 N and 1 N.

K-2.2.2 *Phosphate Reagent A*

Dissolve without heating 5 g of powdered ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

K-2.2.3 *Phosphate Reagent B*

Dissolve without heating 0.2 g of methyl-p- aminophenol sulphate (metal) and 20 g of sodium metabisulphite in 100 ml of water.

K-2.2.4 *Standard Phosphate Solution*

Dissolve 0.286 0 g of potassium dihydrogen phosphate (KH_2PO_4) in water and make up the volume to 1000 ml. Dilute 10 ml of this solution to 100 ml with water. One millilitre of the diluted solution is equivalent to 0.02 mg of phosphate (as PO_4). Keep the solution in a waxed bottle.

K-2.3 Procedure

Dissolve 2.00 g of the material in 20 ml of water, add 7 ml of dilute sulphuric acid (5 N) and 1 ml each of phosphate reagents A and B. Dilute with water to 50 ml and place in a water-bath at 60 °C for 10 min. Carry out a control test using 0.5 ml of standard phosphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

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

K-1 METHOD B (SPECTROPHOTOMETRIC METHOD)**K-3.1 Outline of the Method**

Silica is eliminated by precipitation and filtration. Formation of phosphomolybdic acid complex in acidic solution and reduction of yellow complex to molybdenum blue complex. Measurement of the intensity of the blue colour (which is proportional to phosphate concentration) using a spectrophotometer or photocolourimeter at 550 nm wavelength.

K-3.2 Apparatus

K-3.2.1 *Standard Laboratory Apparatus*

K-3.2.2 *Spec Iropholom eler or Photoelecrlc Colorimeier*

K-3.2.3 *Platinum Evaporating Dish* —Approximately 60 ml capacity.

K-3.3 Reagents

K-3.3.1 *Perchloric Acid* — 60 to 70 percent solution (*m/m*).

K-3.3.2 *Ammonium Molybdate Solution*

Dissolve 10 g of ammonium molybdate tetra hydrate ($(\text{NH}_4)_6 \text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in 100 ml of water. Add

slowly 220 ml of sulphuric acid (1:1 solution) and 70 ml distilled water Mix and allow to cool.

K-3.3.3 Reducing Solution

Dissolve 0.25 g of 1-amino 2-naphthol 4-sulphonic acid and 0.5 g of anhydrous, sodium sulphite (Na_2SO_3) in about 70 ml distilled water. Dissolve separately 24 g of anhydrous sodium bisulphite (Na_2SO_3) in about 100 ml distilled water. Mix both solutions and dilute to 200 ml with distilled water, filter, if necessary and store in a cool dark place.

K-3.3.4 Standard Phosphate Solution

Weigh accurately 1.433 g of finely powdered and dried A.R. potassium dihydrogen phosphate (KH_2PO_4) dissolve in 100 to 150 ml distilled water in a 250 ml beaker, transfer quantitatively to 1000 ml volumetric flask, dilute to mark and mix well. Transfer 50 ml of this solution to a 500 ml volumetric flask, dilute to mark and mix well. One millilitre ml of this solution contains 100 μg of phosphate (as PO_4).

K-3.3.4.1 Dilute Standard Phosphate Solution

10 μg PO_4 per litre — Transfer 50 ml of standard phosphate solution into a 500 ml volumetric flask, dilute to mark and mix well.

K-3.3.5 Hydrochloric Acid Solution

Approximately 6 mol/l — Add slowly 535 ml of concentrated hydrochloric acid (sp gr 1.18) into 465 ml of distilled water and mix.

K-3.4 Procedure

K-3.4.1 Calibration

Into a series of six 100-ml volumetric flasks, transfer dilute standard phosphate solution as given below:

<i>Standard Phosphate Solution, ml</i>	<i>Corresponding Mass of Phosphate as PO_4 μg</i>
0(Compensation)	0
2	20
4	40
6	60
8	50
10	100

Add to each flask 2 ml of perchloric acid, 20 ml of distilled water, 10 ml of ammonium molybdate solution and finally 5 ml of reducing solution. Make up to the mark and mix well. Place flasks in boiling water-bath for 15 min, cool in running water and measure absorbance using 9 cm cell in a spectrophotometer (or photocolormeter) at 620 nm wavelength using compensation solution in the reference cell.

Plot a graph with g of PO_4 as abscissae and corresponding absorbance values as ordinates.

K-3.4.2 Determination

Weigh about 1 g of sodium bicarbonate accurately to 1 mg (the quantity to be weighed should be such that it contains 20 to 100 g of phosphate) and transfer into a 60 ml platinum evaporating dish, wet it with some distilled water and neutralize with hydrochloric acid solution and add a few drops in excess. Evaporate to nearly dryness and add 2 ml of perchloric acid and again evaporate to nearly dryness. Add a few drops of hydrochloric acid solution and about 20 ml of distilled water and transfer quantitatively into a 100 ml volumetric flask. Cool and dilute to mark and mix well.

Filter through a Whatman No. 41 filter paper, discard a small volume of initial filtrate and then transfer 50 ml of filtrate into 100 ml volumetric flask and develop colour as described under calibration (**K-3.4.1**).

Measure absorbance at 620 nm wavelength using same size cell as used for calibration, with distilled water in the reference cell.

K-3.4.3 Blank Test

Carry out a blank test using the same procedure and same quantities of all reagents as in the case of determination (**K-3.4.2**) except omitting the sample solution. Use distilled water instead of sample solution.

K-3.5 Calculations

Phosphate (as PO_4), percent by mass = $M_1 - M_2 / M_0 \times 10\,000$

Where

M_1 = mass in micrograms of phosphate corresponding to the absorbance of the test portion,

M_2 = mass in micrograms of phosphate corresponding to the absorbance of the blank, and

M_0 = mass in grams of the sodium bicarbonate content corresponding to the test portion of the aliquot solution used for colour development.

ANNEX L

[Clause 4.2 and Table 1, Sl No. (xi)]

DETERMINATION OF CALCIUM AND MAGNESIUM

L-1 REAGENTS

L-1.1 Standard Calcium Solution

Weigh 1.000 g of calcium carbonate dried at 120 °C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 l in a graduated flask. One millilitre of this solution is equivalent to 0.400 08 mg of calcium (as Ca).

L-1.2 Standard EDTA Solution

Dissolve 3.72 g of disodium ethylene diamine tetra-acetate dihydrate in water and dilute in a graduated flask to 1 l. The solution shall be standardized frequently against the standard calcium solution, following the procedure given in **L-2.1**.

L-1.3 Eriochrome Black-T Indicator Solution

Dissolve 0.1 g of the dye in 20 ml of rectified spirit conforming to IS 323. This solution shall be prepared fresh every week.

L-1.4 Ammonium Chloride-Ammonium Hydroxide Buffer Solution

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0.90) and 2.50 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in about 50 ml of water. Mix the two solutions and dilute to one litre.

NOTE — Five millilitres of the buffer solution added to 50 ml of distilled water should not consume more than a drop of EDTA solution to change to distinct blue with Eriochrome black-T indicator.

L-2 PROCEDURE**L-2.1 Standardization of EDTA Solution**

Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the Eriochrome black-T indicator solution and titrate against the EDTA solution to a pure blue end point.

L-2.2 Titrate 25 ml of the buffer solution with EDTA solution using Eriochrome black-T indicator. Subtract the buffer correction for 10 ml (usually, it will be 0.1 ml) from the reading obtained in **L-2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

L-2.3 Transfer exactly 100 ml of solution preserved in **D-1** into a 250 ml conical flask. Add 20 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of Eriochrome black-T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end-point. Note the volume of EDTA solution used in the titration.

L-3 CALCULATION

Calcium and magnesium (as Ca), percent by mass = $\frac{A V_1}{M} \times 100$

A = calcium equivalent calculated in g of 1 ml of EDTA solution determined in **L-2.2**,

V_1 = volume in ml of standard EDTA solution used in **L-2.3**, and

M = mass in g of the material taken for the test in **D-1**.

L-4 Alternative Method

Calcium and magnesium may alternatively be determined by instrumental test method as prescribed at Annex S.

ANNEX M

[Clause 4.2 and Table 1, Sl No. (xii)]

TEST FOR COPPER**M-1 GENERAL**

Two methods have been prescribed for determination of copper visual colour comparison (Method A) and spectrophotometric method (Method B). In case of dispute Method A shall be referee method.

M-2 METHOD A (VISUAL COLOUR COMPARISON)**M-2.1 Apparatus**

M-2.1.1 *Nessler Cylinders* — 50 ml capacity.

M-2.2 Reagents

M-2.2.1 *Concentrated Hydrochloric Acid*— See to IS 265.

M-2.2.2 *Concentrated Nitric Acid* — See to IS 264.

M-2.2.3 *Citric Acid*

M-2.2.4 Dilute Ammonium Hydroxide — Approximately 5 N.

M-2.2.5 Sodium Diethyldithiocarbamate Solution

Dissolve 1.0 g of sodium diethyldithiocarbamate in 1000 ml of copper free water. Filter and keep in an amber bottle and protect from strong light.

M-2.2.6 Standard Copper Solution

Dissolve 0.392 g of copper sulphate pentahydrate in copper-free water and make up the volume to 1000 ml. Take 100 ml of this solution and dilute again to 1000 ml. One millilitre of the diluted solution contains 0.01 mg of copper (as Cu).

M-2.2.7 Chloroform

M-2.3 Procedure

Weigh 1.000 g of the material and dissolve it in about 50 ml of water. Neutralize with concentrated hydrochloric acid and add 4 to 5 drops of concentrated nitric acid. Boil and cool. Add 1 g of citric acid and adjust pH to 9 by adding dilute ammonium hydroxide. Add 10 ml of sodium diethyldithiocarbamate solution and extract the yellow colour produced four times with 2.5 ml portions of chloroform. Collect the chloroform extracts and filter through a dry filter into a Nessler cylinder. Carry out a control test using 3 ml of standard copper solution in place of the material.

The material shall be considered not to have exceeded the limit prescribed in Table 1 If the intensity of the colour produced with the material is not greater than that produced in the control test.

M-3 METHOD B (SPECTROPHOTOMETRIC METHOD)

M-3.1 Outline of the Test Method

The material is neutralized with hydrochloric acid. The copper in the soluble form is treated with diethyldithiocarbamate solution to produce a coloured copper complex. This is extracted with carbon tetrachloride. The absorbance of the CCl₄ extracts measured at 440 nm using a spectrophotometer or a photocolormeter

M-3.2 Apparatus

M-3.2.1 Spectrophotometer or a Photocolormeter—Suitable for measuring absorbance at 440 nm.

M-3.2.2 Standard Laboratory Glass Apparatus

M-3.2.3 Platinum Dish, of about 60 ml capacity.

M-3.3 Reagents

M-3.3.1 Distilled Water

M-3.3.2 Hydrochloric Acid

Approximately 6 mol/l — Dilute 500 ml conc. HCl (sp. gr. 1.19) with 500 ml distilled water.

M-3.3.3 EDTA Solution

5 percent solution — Dissolve 50 g Ethylene diamine tetra-acetic acid, disodium salt (C₁₀H₁₄O₈Na₂·2H₂O) in distilled water, dilute to 1000 ml and keep the solution in a polyethylene bottle.

M-3.3.4 Sodium Diethyldithiocarbamate Solution (DEDTC)

Dissolve 0.1 g of DEDTC $[(C_2H_5)_2N.CS_2Na.3H_2O]$ in 100 ml of distilled water and keep the solution in a dark bottle.

M-3.3.5 Standard Copper Solution

Dissolve 0.100 g pure copper metal in the smallest possible quantity nitric acid in a 1 000 ml volumetric flask, cool and dilute to mark. One millilitre of this solution contains 100 µg copper.

Transfer 10 ml aliquot of this solution (**M-3.3.5**) in to 100 ml volumetric flask, dilute to mark and mix. One millilitre of this solution is equivalent to 10 µg of copper.

M-3.3.6 Ammonium Hydroxide Solution —Concentrated.**M-3.3.7 Carbon Tetrachloride —Reagent grade CCl_4 .****M-3.3.8 Phenolphthalein Solution**

Dissolve 0.5 g phenolphthalein powder in 500 ml of 50-70 percent ethanol or methanol. Neutralize the solution by adding 0.1 mol/l NaOH until a faint pink colour appears and then remove this colour with a drop or two of 0.1 mol/l hydrochloric acid.

M-3.3.9 Hydrochloric Acid Solution — Approximately 1 mol/l.**M-3.4 Procedure****M-3.4.1 Calibration**

Into a series of six 250 ml separatory funnels, transfer standard dilute copper solution as given below:

<i>Standard Copper Solution, ml</i>	<i>Corresponding Mass of Copper, µg</i>
0 (Compensation)	0.0
2.0	20.0
4.0	40.0
6.0	60.0
8.0	80.0
10.0	100.0

Add to each 20 ml hydrochloric acid solution, 10 ml EDTA solution and then 5 drops of phenolphthalein solution and mix well. Neutralize each with concentrated ammonium hydroxide solution to a faint pink colour and cool to room temperature. Add distilled water to each to a final volume of 45 ml.

Then add to each 5 ml DEDTC solution mix well and add 20 ml carbon tetrachloride Shake the funnel vigorously for at least 3 min and allow the phases to separate.

Filter CCl_4 phase (the lower phase) through a cotton plug inserted in the outlet stem of the separatory funnel into photometer cells. Apply the lid of the cell and measure absorbance at 440 nm with extract of copper free solution (compensation solution) as reference.

Plot copper content (µg/20 ml) as abscissae vs corresponding absorbance values as ordinates.

M-3.4.2 Determination

Weigh accurately correct to 1 mg an amount of sodium bicarbonate sample containing 200 to 1000 µg of copper and transfer into a platinum dish. Wet it with minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid solution and add a few drops in excess. Evaporate to dryness on a water-bath. Wet it with a few drops of dilute hydrochloric acid solution and then dissolve in 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml one mark volumetric flask, cool, make up to the mark and mix well. If the solution is turbid, filter through a Whatman No. 54 1 filter paper discarding 10 to 15 ml of initiate filtrate. Transfer 10.0 ml of the aliquot volume (or the filtrate if filtered) into a 250 ml separatory funnel, and proceed further exactly as described under calibration.

M-3.4.3 Blank Test

Carry out a blank test exactly as described in **M-3.4.2** but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph.

M-3.5 Calculations

$$\text{Copper (as Cu), parts per million} = \frac{M_1 - M_2}{M_0}$$

By means of the calibration graph determine the mass of copper in micrograms corresponding to the absorbance value of the test solution.

M_1 = mass of copper in micrograms in the test portion,

M_2 = mass of copper in micrograms in the blank, and

M_0 = mass of the sodium bicarbonate content in grams in the test portion used for colour development.

M-4 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD

M-4.1 Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

ANNEX N

[Clause 4.2 and Table 1, Sl No. (xiii)]

TEST FOR NITRATES

N-1 REAGENTS

N-1.1 Dilute Sulphuric Acid — Approximately 5 N.

N-1.2 Concentrated Hydrochloric Acid — See to IS 265.

N-1.3 Potassium Nitrate Solution — 0.001 N.

N-1.4 Concentrated Sulphuric Acid — See to IS 266.

N-1.5 Standard Indigo Carmine Solution

Diphenyl amine / Brucine sulphate solution may be used as indicator for determination of nitrates in place of indigo carmine solution

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid and 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).

N-2 PROCEDURE

Dissolve 2.000 g of the material in 10 ml of dilute sulphuric acid, and 10 ml of standard indigo carmine solution followed by 10 ml of concentrated sulphuric acid and heat to boiling. The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced in the test with the material does not entirely disappear.

ANNEX P

[Clause 4.2 and Table 1, Sl No. (xiv)]

TEST FOR SILICATES

P-1 GENERAL

Two methods have been prescribed for determination of silicates visual colour comparison (Method A) and spectrophotometric method (Method B). In case of dispute Method A shall be referee method.

P-2 METHOD A (VISUAL COLOUR COMPARISON)

P-2.1 Apparatus

P-2.1.1 Nessler Cylinders —50 ml capacity.

P-2.2 Reagents

P-2.2.1 Dilute Sulphuric Acid —Approximately 1 N.

P-2.2.2 Ammonium Molybdate Solution

Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

P-2.2.3 Sodium Citrate Solution

Dissolve 8 g of sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$) in 100 ml of water

P-2.2.4 Potassium Metabisulphite Solution

Dissolve 20 g of potassium metabisulphite and 1 g of hydroquinone in 100 ml of water.

P-2.2.5 Anhydrous Sodium Carbonate

P-2.2.6 Standard Silicate Solution

Fuse 0.100 g of pure, precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the melt with water, neutralize with dilute sulphuric acid and dilute to 1000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of silica (as SiO_2). Store in a polyethylene bottle.

P-2.3 Procedure

Dissolve 2.00 g of the material in 20 ml of water in a platinum dish, neutralize with about 10 ml of dilute sulphuric acid. Add 2 ml of the acid in excess and dilute to 50 ml. Transfer 10 ml of this solution into a Nessler cylinder. Add 1 ml of dilute sulphuric acid and 5 ml of ammonium molybdate solution. Allow to stand for 5 min and then add 5 ml of sodium citrate solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Stand both the cylinders in a boiling water-bath for 5 min, cool and compare the colour produced in the two cylinders after 5 min.

The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the material is not deeper than that produced in the control test.

P- 3 METHOD B (SPECTROPHOTOMETRIC METHOD)

P-3.1 Outline of the Test Method

This method specifies a reduced molybdosilicate spectrophotometric method for the determination of soluble silica. This method is applicable for materials with silica content in presence of phosphate.

Depolymerization of any polymerized silica by treatment with hydro fluoric acid and boric acid formation of the oxidized molybdosilicate complex (yellow) under well defined conditions of acidity ($pH 1 \pm 0.05$).

Selective reduction of the complex in a strong sulphuric acid medium in the presence of oxalic acid to eliminate interference of phosphate.

Spectrophotometric measurement of the coloured complex at wavelength of maximum absorption (about 795 nm).

P-3.2 Reagents

P-3.2.1 Distilled Water

P-3.2.2 Sulphuric Acid

Approximately 2 mol/l — Add 112 ml of concentrated A.R. Sulphuric acid (sp. gr. 1.84) in 888 ml of distilled water slowly by constant stirring cool and mix well.

P-3.2.3 Sulphuric Acid

Approximately 8 mol/l — Add 448 ml of concentrated A.R. Sulphuric acid (sp gr 1.84) in 552 ml of water slowly by constant stirring, cool and mix well.

P-3.2.4 Sodium Molybdate

274 g/l Solution — Dissolve 27.4 g of sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$) in warm distilled water in a beaker of material free from silica of suitable capacity and after cooling dilute to 100 ml. Store, if necessary, in a bottle of material free from silica and filter, if necessary, before use.

P-3.2.5 Oxalic Acid

100 g/l Solution — Dissolve 10 g of oxalic acid dihydrate ($COOH \cdot COOH \cdot 2H_2O$) in 100 ml distilled water.

P-3.2.6 Ascorbic Acid

25 g/l Solution — Dissolve 2.5 g of ascorbic acid in water and dilute to 100 ml. Store the solution in a bottle of material free from silica and protect from light. Prepare this solution at the time of use or as an alternative, prepare reduction solution.

P-3.2.7 Reduction Solution

P-3.2.7.1 Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 50 ml distilled water. Then add 1.5 g of 4-amino-3-hydroxyl naphthalene-1-sulphonic acid ($\text{C}_{10}\text{H}_7\text{NO}_3\text{S}$) and dissolve by grinding.

P-3.2.7.2 Dissolve 90 g of anhydrous metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 900 ml of water.

P-3.2.7.3 Mix (**P-3.2.7.1** and **P-3.2.7.2**) and dilute to 1 000 ml. Filter if turbid and store in a opaque bottle free from silica.

P-3.2.8 *Sodium Fluoride Solution* — 20 g/l solution.

P-3.2.9 *Boric Acid Solution* —Saturated solution at ambient temperature.

P-3.2.10 *Standard Silica Solution*

0.200 g SiO_2 per litre — Weigh to the nearest 1 mg 0.200 g SiO_2 obtained by heating pure silicic acid (H_2SiO_3) at 1000 °C until constant mass (consecutive weighings should not differ by more than 1 mg) and cooling in a desiccator into a platinum crucible of convenient capacity. Add 2 g of anhydrous Na_2CO_3 to the crucible, mix well preferably with a platinum spatula and carefully fuse the mixture. Add warm water directly to the crucible, heat gently until the contents are completely dissolved and transfer quantitatively to a beaker of material free from silica of suitable capacity.

Cool, dilute the solution to about 500 ml, transfer quantitatively to a 1000 ml volumetric flask, dilute to mark and mix well. Transfer the solution immediately to a bottle of material free from silica. 1 ml of this solution = 0.2 mg SiO_2 .

P-3.2.10.1 *Standard Silica Solution*

2.0 mg SiO_2 per litre — Dilute 20.0 ml of standard silica solution (**P-3.2.10**) to 1000 ml in a 1000 ml volumetric flask. Prepare this solution at the time of use. 1 ml of this solution = 2.0 μg SiO_2 .

P-3.3 Apparatus

P-3.3.1 *Standard Laboratory Apparatus*

P-3.3.2 *pH Meter*

P-3.3.3 *Specrophotometer or Photocolorimeter*

P-3.3.4 *Platinum Crucible with Lid of Suitable Size*

P-3.4 Procedure

P-3.4.1 *Preparation of the Calibration Graph*

P-3.4.1. 1 *Preliminary Test for Control and Correction of pH*

Place 10.0 ml of the standard silica solution (**P-3.2.10.1**) in a beaker. Add the distilled water to bring volume to 15 ml and then add 5 ml of sodium molybdate solution, 1 ml of sodium fluoride and 5 ml of boric acid solution. Mix and measure pH with a pH meter. Adjust pH to 1.0 ± 0.05 by adding slowly using a graduated pipette or a burette drop by drop, mixing after each addition, the necessary quantity of sulphuric acid (about 4.5 ml would be required).

Record the actual volume of sulphuric acid required and discard the solution.

P-3.4.1.2 *Preparation of the Standard Colorimetric Solution*

In to a series of six 50 ml beakers, transfer standard silica solution as given below:

<i>Standard Silica Solution</i>	<i>Corresponding Mass of Silica</i>
0 (Compensation)	0.0
2.0	4.0
4.0	8.0
6.0	12.0
8.0	16.0
10.0	20.0

Add to each beaker distilled water to make up volume to 15.0 ml. Then add the volume of sulphuric acid solution as calculated above (**P-3.4.1.1**) for pH correction, mix well. Add 1 ml of sodium fluoride solution, mix and allow to stand for 5 min.

P-3.4.1.3 *Colour Development*

Add to each beaker 5 ml of boric acid solution and 5 ml of sodium molybdate solution, mix and allow to stand for 10 min. Add 5 ml of oxalic acid solution and 8 ml of sulphuric acid solution (**P-3.2.3**) Allow to stand for 2 min, add 2 ml of ascorbic acid solution or as an alternative 2 ml of reduction solution and allow to stand for 10 min. Transfer the solutions quantitatively to a series of 50 ml volumetric flasks, dilute to marks and mix.

P-3.4.1.4 *Measurements*

Carry out the spectrophotometric measurements with spectrophotometer (or photocolorimeter) at 795 nm wavelength, after having adjusted the instrument zero absorbance with the compensation solution, use cells of 4 to 5 cm optical path length.

Plot a graph with kg of SiO₂ contained in 50 ml as abscissae and corresponding absorbance values as ordinates.

P-3.4.2 *Determination*

P-3.4.2.1 *Test solution*

Weigh accurately correct to 1 mg an amount of Sodium bicarbonate (containing between 4 to 20 kg of SiO₂) and transfer into a 100 ml beaker, dissolve in minimum

P-3.4.2.2 *Blank test*

Carry out a blank test following test same procedure and using the same quantities of all reagents used for the determination but replacing the test solution by the same volume of distilled water.

P-3.4.2.3 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements by the procedure specified in (**P-3.4.1.4**) at 795 nm wave length after adjusting the instrument to zero absorbance against water, use the same size of the cell used for calibration.

P-3.4.3 *Calculation*

By means of the calibration graph determine the mass of SiO₂ content corresponding to the aliquot used for colour development. The silica content is expressed by the relation given below:

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10\,000}$$

M_1 = mass in μg of SiO_2 in the test portion of the aliquot,

M_2 = mass in μg of SiO_2 in the blank, and

M_0 = mass in μg of SiO_2 in the blank, and mass of original material in g the aliquot portion used for colour development.

ANNEX Q

[Clause 4.2 and Table 1, SI No. (xv)]

DETERMINATION OF POTASSIUM

Q-1 APPARTUS

Q-1.1 Flame Photometer

Equipped with interference filter.

Q-2 REAGENTS

Q 2.1 Standard Potassium Solution

Weigh exactly 1 g of potassium chloride, dissolve in distilled water and dilute to 1 l with distilled water in a measuring flask. This solution contains 0.1 g KCl per 100 ml.

Q-3 PROCEDURE

Q-3.1 Calibration Graph

Take 10, 20, 40, 60 and 80 ml of standard potassium solution and dilute to 100 ml with distilled water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g KCl per 100 ml.

Use these dilute solutions to obtain a corresponding galvanometer reading as given in **Q-3.2.1** and plot the concentration against galvanometer reading in a rectangular co-ordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.01 to 0.1 percent KCl.

Q-3.2 Sample Solution

Dissolve approximately 10 g of the sample in minimum quantity of distilled water and dilute to 100 ml in measuring flask with distilled water.

Q-3.2.1 Determination

Use a flame photometer equipped with atomizer, burner, optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element. Insert the potassium filter corresponding to wavelength 767 nm, light the Burner fed by illuminating gas (laboratory gas), adjust the specified air pressure between 0.5 to 0.6 kg/cm and maintain the above air pressure constant, such that flame is non luminous by turning the control knob. First spray distilled water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the KCl standard solution and adjust the deflection to maximum 100 by using sensitivity control knob. Again spray water to see that the pointer comes to zero; then spray a standard solution reads 100 with same adjustment during both the operations. A reading zero by water and with the same adjustment 100 by standard solution indicate that the instrument has been made ready for measurement. Without altering the earlier adjustment of the instrument spray various diluted solutions prepared in **Q-3.1** and obtain a calibration graph in the range 0.01 to 0.1 percent KCl. After washing with distilled water, spray the

sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of KC1 in the solution (say A).

Q-4 CALCULATION

$$\text{Potassium (as K), percent by mass} = \frac{52.35 \times A}{M}$$

Where

A = concentration of KC1 in the sample solution, and

M = mass of dried sample taken for the test.

ANNEX R

[Clause 4.2 and Table 1, Sl No. (xvi)]

TEST FOR SUBSTANCES REDUCING IODINE

R-1 REAGENTS

R-1.1 Starch Solution

Titrate 5 g of 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 and boil for 3 min allow the solution to cool and decant off the clear liquid.

R-1.2 *Standard Iodine Solution* — 0.01 N.

R-2 PROCEDURE

Dissolve 10 g of the material in 200 ml of freshly boiled and cooled water. Add 2 ml of starch solution and titrate with standard iodine solution. The material shall be taken to have passed the test if not more than 0.5 ml of iodine solution is required to produce a permanent blue colour.

ANNEX S

S-1 DETERMINATION OF LEAD, IRON, CALCIUM, MAGNESIUM AND ARSENIC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

S-2.1 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.

S-2.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 1 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
vi)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti

S-2.3 Reagents and Solutions**S-2.3.1 Nitric acid (65 percent) Suprapure**

S-2.3.2 *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 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

S-2.3.3 *Standard solution*

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

S-2.3.4 *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

S-2.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

S-2.4 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

S-2.5 Procedure**S-2.5.1** *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**S-2.5.2**). 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.

S-2.5.2 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 µg/ml of the lead (and/or Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) 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.

S-2.6 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 T

T-1 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

T-1.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions, 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.

T-1.2 Equipment

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

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

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

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

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

T-1.3 Reagents and Standards

T-1.3.1 *Glass or polyethylene sample bottles.*

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

T-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.

T-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

T-1.4 Standard solutions

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

T-1.4.2 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

T-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.

T-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.

T-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 U

(Clause 6)

SAMPLING OF SODIUM BICARBONATE

U-1 GENERAL REQUIREMENTS OF SAMPLING

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

U-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

U-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

U-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

U-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

U-1.6 Samples shall not be taken in an exposed place.

U-2 SCALE OF SAMPLING

U-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

U-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

U-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (and shall be in accordance with col 1 and 2 of Table 3.

U-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random sampling procedures given in IS 4905 may be followed.

U-3 TEST SAMPLES AND REFEREE SAMPLE

U-3.1 Test Samples

U-3.1.1 Draw with an appropriate sampling instrument

Table 3 Scale of Sampling
(Clause U-2.2)

Lot Size <i>N</i> (1)	Number of Containers to be Selected <i>n</i> (2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7

a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **U-3** and shall not exceed 1 kg.

U-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

U-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **U-1.5**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

U-3.2 Referee Sample

The referee sample shall consist of the composite sample (**U-3.1.2**) and a set of individual samples (*see* **U-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

U-4 NUMBER OF TESTS

U-4.1 Tests for the determination of total alkalinity shall be conducted on each of the individual samples for all the three grades. For refined grade, tests for heavy metals and arsenic also shall be conducted on each of the individual samples.

U-4.2 Test for the remaining characteristics shall be conducted on the composite sample.

U-5 CRITERION FOR CONFORMITY

U-5.1 For Individual Samples

U-5.1.1 For Total Alkalinity

The test results for total alkalinity shall be recorded and the mean and the range for these test results shall be calculated as follows:

Mean (\bar{X}) Sum of the test results divided by the number of test results.

Range (R) = The difference between the maximum and the minimum values of the test results.

and the value of expression ($X - 0.6 R$) shall be calculated. If the value of this expression is equal to or greater than the limit specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirements for this characteristic.

U-5.1.2 *For Heavy Metal and Arsenic*

The lot shall be declared as conforming to the requirements for these characteristics if each of the samples satisfies the test laid down in Annexes G and H.

U-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements specified in Table 1.

U-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.