

भारतीय मानक मसौदा
धात्विक जिंक पाउडर (जिंक धूल) — विशिष्टि
(IS 4611 का तीसरा पुनरीक्षण)

Draft Indian Standard
Metallic Zinc Powder (Zinc Dust) — Specification
(Third Revision of IS 4611)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(Formal clauses will be added later)

This standard was first published in 1967 and subsequently revised in 1973 and 1991. In previous revision, the methods for determination of total zinc and metallic zinc were modified to bring them in line with the methods given in ISO 3549 'Zinc dust pigments for paints — Specifications and test methods'. A new requirement of calcium oxide along with the test method was also incorporated. The method for determination of lead was also modified. Limits for various requirements for both the grades of metallic zinc were modified. Additional requirement of free chloride for the material for use in explosive and pyrotechnic industry along with its method of test was deleted.

In this revision, instrumental test methods for the determination of chlorides, iron, lead and copper have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Amendment No. 1 has been incorporated. Further, Reference clause and Packing & Marking clause have been updated.

Zinc dust is used extensively in various chemical industries as well as for protective coatings. In chemical industry, it is used as a reducing agent in the manufacture of synthetic dyestuffs, a substitute for magnesium powder in Grignard reactions and in several other organic reactions. It is used in textile industry in the printing and application of dyestuffs to cotton, silk and wool and in pyrotechnic industry as an ingredient of smoke producing composition. Other uses of zinc dust are in conditioning of ships boiler water, as an anticorrosive agent in oil fired turbines and as a component of special greases and in silver-zinc storage batteries.

Zinc dust is used in metallurgical industry as a precipitating agent, in gold mining and for electro galvanizing where it is used to remove undesirable metal impurities especially cadmium, lead and copper from zinc sulphate solution. Since zinc will corrode preferentially to many common metals, zinc dust finds its largest single application in preventing corrosion of steel and other metals.

A certain amount of lead in zinc dust is considered to be necessary particularly when zinc dust is used in the manufacture of sodium hydrosulphite. In the first stage of the process, where a suspension of zinc dust in water is reacted with liquid sulphur dioxide, the presence of lead makes the end point of the completed reaction clearly visible.

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

*Draft Indian Standard***METALLIC ZINC POWDER (ZINC DUST) — SPECIFICATION**
*(Third Revision)***1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for metallic zinc powder commercially known as zinc dust for use in various industries.

2 REFERENCES

The standards contain provisions, which through reference in this text, constitute provisions 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>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>third revision</i>)
IS 3025 (Part 2) : 2019/ISO 11885 : 2007	Methods of sampling and test (physical and chemical) for water and wastewater Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
(Part 65) : 2022/ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including Uranium Isotopes (<i>first revision</i>)

3 GRADES

The material shall be of the following two grades:

- a) *Grade 1* — for use in the manufacture of hydrosulphites and related products; and
- b) *Grade 2* — for chemical and protective coating.

4 REQUIREMENTS**4.1 Description**

The material shall consist of very fine, uniform, smooth, dark grey metallic powder free from aggregates and visible impurities.

4.2 The material shall also comply with the requirements given in Table 1.

4.3 The limits and tolerances, if any, for the material passing through 45 micron IS sieve and 75 micron IS sieve shall be as agreed to between the purchaser and the seller/manufacturer and shall be tested as described in **A-12**.

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in sound, clean and dry airtight metal drums or double polyethylene lined hessian bags having another polyethylene bag inside.

Table 1 Requirements for Metallic Zinc Powder (Zinc Dust)*(Clauses 4.2, A-5.4.3, A-7.4.1, A-8.4.2 and B-5.2)*

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No.in Annex A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Metallic zinc, percent by mass, <i>Min</i>	94.0	94.0	A-2
ii)	Total zinc (as Zn), percent by mass, <i>Min</i>	98.0	98.0	A-3
iii)	Lead as (Pb), percent by mass, <i>Max</i>	0.15	0.05	A-4 or A-14
iv)	Iron as (Fe), percent by mass, <i>Max</i>	0.05	0.05	A-5 or A-14
v)	Cadmium (as Cd), percent by mass, <i>Max</i>	0.05	0.02	A-6
vi)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.01	0.01	A-7 or A-13
vii)	Copper (as Cu), percent by mass, <i>Max</i>	0 005	0.005	A-8
viii)	Moisture and other volatile matter, percent by mass, <i>Max</i>	0.05	0.05	A-9
ix)	Matter insoluble in aqua regia, percent by mass, <i>Max</i>	0.05	0.05	A-10
x)	Calcium oxide, percent by mass, <i>Max</i>	—	0.7	A-11

5.2 Marking

Each package shall be legibly and indelibly marked with the following information:

- Name and grade of the material;
- Indication of the source of manufacture;
- Mass of the material in the package;
- Year of manufacture; and
- Identification Mark in code or otherwise to enable the batch of manufacture to be traced from records.

5.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

The method of drawing representative samples of the material, the number of tests to be performed and the criteria of conformity of the material to the requirements of this specification, shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2)

METHODS OF TEST FOR METALLIC ZINC POWDER (ZINC DUST)

A-1 QUALITY OF REAGENTS

Unless otherwise specified, 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.

A-2 DETERMINATION OF METALLIC ZINC

A-2.1 Two methods are prescribed, namely, volumetric method and gas evolution method. In case of dispute, the volumetric method shall be the referee method.

A-2.2 Volumetric Method**A-2.2.1 Outline of the Method**

Metallic zinc reduces ferric sulphate into ferrous state which is reoxidized to ferric state using potassium permanganate solution.

A-2.2.2 Reagents**A-2.2.2.1 Ammonium ferric sulphate solution**

Dissolve 50 g of ammonium ferric sulphate in 200 ml of water.

A-2.2.2.2 Dilute sulphuric acid solution — 5 N**A-2.2.2.3 Standard potassium permanganate solution — 0.1 N**

NOTE — Only distilled water shall be used for solution preparation and dilution.

A-2.2.3 Procedure

Take 100 ml ammonium ferric sulphate solution into 500 ml of conical flask. Weigh about 1 g of zinc powder sample accurately and transfer it carefully into the solution in the flask. Displace the air inside the flask by carbon dioxide. Close the flask with a rubber stopper tightly and agitate constantly until the zinc is completely dissolved. Open the flask. Add 50 ml of 5 N sulphuric acid into the flask and dilute the resulting solution with water to a volume of 500 ml. Titrate 50 ml of the above solution against 0.1N potassium permanganate solution.

A-2.2.4 Calculation

1 ml of 0.1 N potassium permanganate solution = 0.003 269 g of metallic zinc.

A-2.3 Gas Evolution Method**A-2.3.1 Reagents****A-2.3.1.1 Hydrochloric acid/copper (I) chloride reagent**

Dissolve 0.05 g of copper (I) chloride (Cu_2Cl_2) in 700 ml of hydrochloric acid (relative density 1.18) and dilute to 1 litre with water.

A-2.3.1.2 Solution for levelling bottle

Acidify some water with a few drops of hydrochloric acid and add sufficient methyl orange indicator to render the colour of the water in the apparatus easily visible.

A-2.3.2 Apparatus**A-2.3.2.1 Gas measuring apparatus**

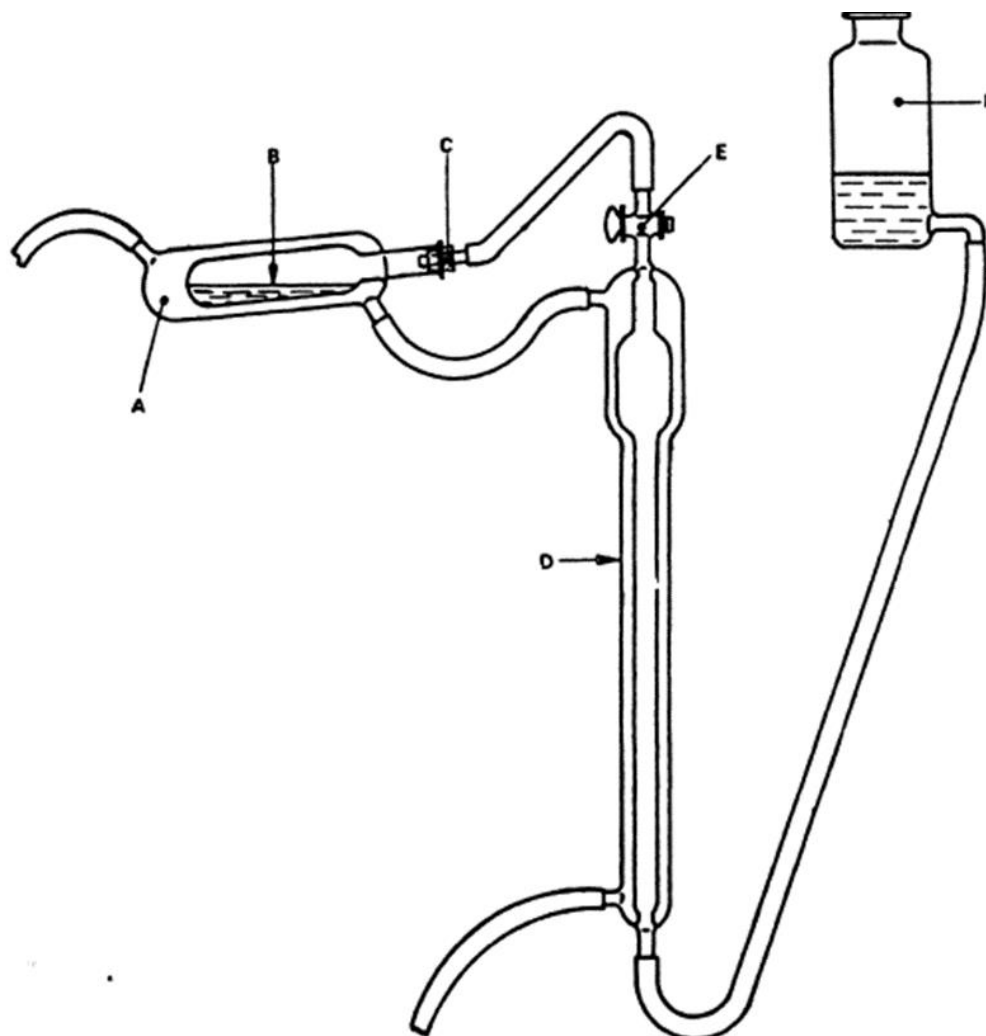
Consisting of a water jacketed gas burette of capacity 400 ml, graduated from 300 ml, and a water-cooled reaction flask of capacity 400 ml. Any suitable apparatus may be used. Fig. 1 illustrates one such convenient type of the apparatus.

A-2.3.2.2 Boat — made of a suitable plastic material or porcelain with lid.**A-2.3.3 Procedure**

NOTE — For convenience, the procedure given below refers to the apparatus illustrated in Fig. 1. Other suitable apparatus may require modification of the procedure described.

A-2.3.3.1 *Test portion*

Weigh, to the nearest 0.001 g, about 1 g of the sample into the boat and cover with the lid.



A — Reaction flask

B — Position of boat

C — Rubber stopper and connection

D — Gas burette

E — Three-way tap

F — Levelling bottle

FIG. 1 APPARATUS FOR THE DETERMINATION OF METALLIC ZINC BY THE GAS EVOLUTION METHOD

A-2.3.3.2 *Determination*

Pass a steady flow of tap water through the two water jackets. Introduce 30 ml of the hydrochloric acid/copper (I) chloride reagent (A-2.3.1.1) into the reaction flask (A) and clamp it in an almost horizontal position. Introduce the covered boat into the neck of the flask. Take care that the zinc does not come in contact with the acid. Fit the connecting stopper (C) to the flask (A). After the circulating tap water has been running long enough to establish a constant temperature, raise the level of the water in the gas burette (D) by opening the three-way tap (E) to atmosphere and raising the levelling bottle (F) until the level is at a convenient height (about eye level). Turn the three-way tap to connect flask (A) with the gas burette and move the levelling bottle until the liquids in the tube and bottle are at the same level. Keep the bottle steady until the tap is turned to atmosphere. Raise the bottle and

expel all the air from the gas burette. Turn the tap to connect the flask with the gas burette and place the bottle on the bench. Take the flask out of the clamp and allow the boat to drop into the acid. Shake the flask well until the reaction has virtually ceased. When the volume of gas evolved becomes constant and the temperature returns to the initial value, level off the liquids in the gas burette and bottle and record the volume V . Record the barometric pressure and the temperature of the cooling water. Correct the barometer reading for the temperature of the brass scale (only valid for a special barometer) by using Table 2. Read the saturated water vapour pressure at the temperature of the cooling water from Table 3.

Table 2 Barometer Corrections for Braes Scales*(Clause A.2.3.3.2)***Barometer Reading, kPa (mmHg)**

Temperature, °C	82.6 (620)	86.6 (650)	90.6 (680)	93.3 (700)	97.3 (730)	101.3 (760)	104.0 (780)
1	0.01 (0.1)	0.01 (0.1)	0.01 (0.1)	0.01 (0.1)	0.01 (0.1)	0.01 (0.1)	0.01 (0.1)
2	0.03 (0.2)	0.03 (0.2)	0.03 (0.2)	0.03 (0.2)	0.03 (0.2)	0.03 (0.2)	0.04 (0.3)
3	0.04 (0.3)	0.04 (0.3)	0.04 (0.3)	0.04 (0.3)	0.05 (0.4)	0.05 (0.4)	0.05 (0.4)
4	0.05 (0.4)	0.05 (0.4)	0.05 (0.4)	0.07 (0.5)	0.07 (0.5)	0.07 (0.5)	0.07 (0.5)
5	0.07 (0.5)	0.07 (0.5)	0.08 (0.6)	0.08 (0.6)	0.08 (0.6)	0.08 (0.6)	0.08 (0.6)
6	0.08 (0.6)	0.08 (0.6)	0.09 (0.7)	0.09 (0.7)	0.09 (0.7)	0.09 (0.7)	0.11 (0.8)
7	0.09 (0.7)	0.09 (0.7)	0.11 (0.8)	0.11 (0.8)	0.11 (0.8)	0.12 (0.9)	0.12 (0.9)
8	0.11 (0.8)	0.12 (0.9)	0.12 (0.9)	0.12 (0.9)	0.13 (1.0)	0.13 (1.0)	0.13 (1.0)
9	0.12 (0.9)	0.12 (0.9)	0.13 (1.0)	0.13 (1.0)	0.14 (1.1)	0.14 (1.1)	0.14 (1.1)
10	0.13 (1.0)	0.14 (1.1)	0.14 (1.1)	0.14 (1.1)	0.16 (1.2)	0.16 (1.2)	0.17 (1.3)
11	0.14 (1.1)	0.16 (1.2)	0.16 (1.2)	0.17 (1.3)	0.17 (1.3)	0.18 (1.4)	0.18 (1.4)
12	0.16 (1.2)	0.17 (1.3)	0.17 (1.3)	0.18 (1.4)	0.18 (1.4)	0.20 (1.5)	0.20 (1.5)
13	0.17 (1.3)	0.18 (1.4)	0.18 (1.4)	0.20 (1.5)	0.21 (1.6)	0.21 (1.6)	0.23 (1.7)
14	0.18 (1.4)	0.20 (1.5)	0.20 (1.5)	0.21 (1.6)	0.23 (1.7)	0.23 (1.7)	0.24 (1.8)
15	0.20 (1.5)	0.21 (1.6)	0.23 (1.7)	0.23 (1.7)	0.24 (1.8)	0.25 (1.9)	0.25 (1.9)
16	0.21 (1.6)	0.23 (1.7)	0.24 (1.8)	0.24 (1.8)	0.25 (1.9)	0.27 (2.0)	0.27 (2.0)
17	0.23 (1.7)	0.24 (1.8)	0.25 (1.9)	0.25 (1.9)	0.27 (2.0)	0.28 (2.1)	0.29 (2.2)
18	0.24 (1.8)	0.25 (1.9)	0.27 (2.0)	0.28 (2.1)	0.28 (2.1)	0.29 (2.2)	0.31 (2.3)
19	0.25 (1.9)	0.27 (2.0)	0.28 (2.1)	0.29 (2.2)	0.31 (2.3)	0.32 (2.4)	0.32 (2.4)
20	0.27 (2.0)	0.28 (2.1)	0.29 (2.2)	0.31 (2.3)	0.32 (2.4)	0.33 (2.5)	0.33 (2.5)
21	0.28 (2.1)	0.29 (2.2)	0.31 (2.3)	0.32 (2.4)	0.33 (2.5)	0.35 (2.6)	0.36 (2.7)
22	0.29 (2.2)	0.31 (2.3)	0.32 (2.4)	0.33 (2.5)	0.35 (2.6)	0.36 (2.7)	0.37 (2.8)
23	0.31 (2.3)	0.32 (2.4)	0.33 (2.5)	0.35 (2.6)	0.36 (2.7)	0.39 (2.9)	0.39 (2.9)
24	0.32 (2.4)	0.33 (2.5)	0.36 (2.7)	0.36 (2.7)	0.39 (2.9)	0.40 (3.0)	0.41 (3.1)
25	0.33 (2.5)	0.35 (2.6)	0.37 (2.8)	0.39 (2.9)	0.40 (3.0)	0.41 (3.1)	0.43 (3.2)
26	0.35 (2.6)	0.36 (2.7)	0.39 (2.9)	0.40 (3.0)	0.40 (3.0)	0.43 (3.2)	0.44 (3.3)
27	0.36 (2.7)	0.37 (2.8)	0.40 (3.0)	0.41 (3.1)	0.41 (3.1)	0.45 (3.4)	0.45 (3.5)
28	0.37 (2.8)	0.40 (3.0)	0.41 (3.1)	0.43 (3.2)	0.44 (3.4)	0.47 (3.5)	0.48 (3.6)

29	0.39 (2.9)	0.41 (3.1)	0.43 (3.2)	0.44 (3.3)	0.47 (3.5)	0.48 (3.6)	0.49 (3.7)
30	0.40 (3.0)	0.43 (3.2)	0.44 (3.3)	0.45 (3.4)	0.48 (3.6)	0.49 (3.7)	0.51 (3.8)

The value given in the table shall be subtracted from the barometric pressure reading if the temperature of the gas in the gas burette is above room temperature and added to the reading if the temperature is below room temperature.

PRECAUTIONARY NOTES

Note 1 — Careful initial and vigorous final shaking of the reaction flask are essential.

Note 2 — Check that the apparatus is air-tight by opening the tap to connect the flask (A); levelling the solution in the levelling bottle stoppering the bottle and lowering it. The level of the solution in the gas burette should remain constant.

A-2.3.4 Calculation

$$\text{Metallic zinc, percent by mass} = \frac{1.048 V (P_b - P_s)}{m (t + 273)}$$

where

V = volume, in ml, of hydrogen liberated;

P_s = saturated water vapour pressure, in kilopascals at t °C;

m = mass, in g, of the material taken for the test;

P_b = barometric pressure, in kilopascals; and

t = temperature in degree Celsius of the cooling water.

A-3 DETERMINATION OF TOTAL ZINC

A-3.1 Reagents

A-3.1.1 Hydrochloric Acid — relative density 1.18.

A-3.1.2 Hydrochloric Acid — 1 : 4 solution (v/v).

Dilute 200 ml of the hydrochloric acid (A-3.1.1) to 1 litre with water.

A-3.1.3 Sulphuric Acid — 1 : 1 solution (v/v)

Slowly add 1 part by volume of sulphuric acid (relative density 1.84) to 1 part by volume of water.

A-3.1.4 Nitric Acid — 1.42 g/ml.

A-3.1.5 Ammonia Solution — 0.880 g/ml.

A-3.1.6 Buffer Solution

Dissolve 200 g of hydroxyl ammonium chloride (NH₂OH.HCl) in approximately 300 ml of water. Dissolve 28 g of sodium hydroxide in approximately 300 ml of water. Mix the two solutions, cool and dilute to 1 litre with water.

Table 3 Saturated Water Vapour Pressures, kPa (mmHg)

(Clause A.2.3.3.2)

Tem- per- atur- e	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	0.61 (4.6)	0.61 (4.6)	0.61 (4.6)	0.62 (4.7)	0.62(4.7)	0.62(4.7)	0.63(4.8)	0.63(4.8)	0.63(4.9)	0.63(4.9)
1	0.65(4.9)	0.66 (5.0)	0.66 (5.0)	0.66 (5.0)	0.67(5.1)	0.67(5.1)	0.67(5.1)	0.69(5.2)	0.69(5.2)	0.71(5.3)
2	0.71(5.3)	0.71 (5.3)	0.72 (5.4)	0.72 (5.4)	0.72(5.4)	0.73(5.5)	0.73(5.5)	0.75(5.6)	0.75(5.6)	0.75(5.6)

3	0.76(5.7)	0.76(5.7)	0.77(5.8)	0.77(5.8)	0.77(5.8)	0.78(5.9)	0.78(5.9)	0.78(5.9)	0.80(6.0)	0.80(6.0)	0.83(6.2)
4	0.81(6.1)	0.81(6.1)	0.83(6.2)	0.83(6.2)	0.84(6.3)	0.84(6.3)	0.85(6.4)	0.85(6.5)	0.87(6.5)	0.87(6.5)	0.87(6.5)
5	0.87(6.5)	0.88(6.6)	0.88(6.6)	0.89(6.7)	0.89(6.7)	0.90(6.8)	0.90(6.8)	0.92(6.9)	0.92(6.9)	0.92(6.9)	0.93(7.0)
6	0.93(7.0)	0.95(7.1)	0.95(7.1)	0.96(7.2)	0.96(7.2)	0.97(7.3)	0.97(7.3)	0.99(7.4)	0.99(7.4)	0.99(7.4)	1.00(7.5)
7	1.00(7.5)	1.01(7.6)	1.01(7.6)	1.03(7.7)	1.04(7.8)	1.04(7.8)	1.05(7.9)	1.05(7.9)	1.05(7.9)	1.05(7.9)	1.07(8.0)
8	1.07(8.0)	1.08(8.1)	1.09(8.2)	1.09(8.2)	1.14(8.3)	1.11(8.3)	1.12(8.4)	1.12(8.4)	1.13(8.5)	1.13(8.5)	1.15(8.6)
9	1.15(8.6)	1.16(8.7)	1.16(8.7)	1.17(8.8)	1.17(8.8)	1.19(8.9)	1.20(9.0)	1.20(9.0)	1.21(9.1)	1.21(9.1)	1.21(9.1)
10	1.23(9.2)	1.24(9.3)	1.24(9.3)	1.25(9.4)	1.27(9.5)	1.27(9.5)	1.28(9.6)	1.29(9.7)	1.29(9.7)	1.29(9.7)	1.31(9.8)
11	1.31(9.8)	1.32(9.9)	1.33(10.0)	1.33(10.0)	1.35(10.1)	1.36(10.2)	1.36(10.2)	1.37(10.3)	1.39(10.4)	1.39(10.4)	1.39(10.4)
12	1.40(10.5)	1.41(10.6)	1.43(10.7)	1.43(10.7)	1.44(10.8)	1.45(10.9)	1.45(10.9)	1.47(11.0)	1.48(11.1)	1.48(11.1)	1.49(11.2)
13	1.49(11.2)	1.51(11.3)	1.52(11.4)	1.53(11.5)	1.53(11.5)	1.55(11.6)	1.56(11.7)	1.57(11.8)	1.57(11.8)	1.57(11.8)	1.59(11.9)
14	1.60(12.0)	1.61(12.1)	1.61(12.1)	1.62(12.2)	1.64(12.3)	1.65(12.4)	1.66(12.5)	1.66(12.5)	1.68(12.6)	1.68(12.6)	1.69(12.7)
15	1.71(12.8)	1.72(12.9)	1.73(13.0)	1.73(13.0)	1.75(13.1)	1.76(13.2)	1.77(13.3)	1.79(13.4)	1.80(13.5)	1.80(13.5)	1.80(13.5)
16	1.81(13.6)	1.83(13.7)	1.84(13.8)	1.85(13.9)	1.86(14.0)	1.87(14.1)	1.89(14.2)	1.91(14.3)	1.91(14.3)	1.91(14.3)	1.92(14.4)
17	1.93(14.5)	1.95(14.6)	1.96(14.7)	1.97(14.8)	1.99(14.9)	2.00(15.0)	2.01(15.1)	2.03(15.2)	2.04(15.3)	2.04(15.3)	2.05(15.4)
18	2.07(15.5)	2.08(15.6)	2.09(15.7)	2.11(15.8)	2.12(15.9)	2.13(16.0)	2.15(16.1)	2.16(16.2)	2.17(16.3)	2.17(16.3)	2.19(16.4)
19	2.20(16.5)	2.21(16.6)	2.23(16.7)	2.24(16.8)	2.25(16.9)	2.27(17.0)	2.28(17.1)	2.29(17.2)	2.31(17.3)	2.31(17.3)	2.32(17.4)
20	2.33(17.5)	2.35(17.6)	2.36(17.7)	2.39(17.9)	2.40(18.0)	2.41(18.1)	2.43(18.2)	2.44(18.3)	2.45(18.4)	2.45(18.4)	2.47(18.5)
21	2.49(18.7)	2.51(18.8)	2.52(18.9)	2.53(19.0)	2.55(19.1)	2.56(19.2)	2.57(19.3)	2.60(19.5)	2.61(19.6)	2.61(19.6)	2.63(19.7)

22	2.64(19.8)	2.65(19.9)	2.68(20.1)	2.69(20.2)	2.71(20.3)	2.72(20.4)	2.75(20.6)	2.76(20.7)	2.77(20.8)	2.79(20.9)
23	2.81(21.1)	2.83(21.2)	2.84(21.3)	2.87(21.5)	2.88(21.6)	2.89(21.7)	2.91(21.8)	2.93(22.0)	2.95(22.1)	2.96(22.2)
24	2.99(22.4)	3.66(22.5)	3.03(22.7)	3.04(22.8)	3.05(22.9)	3.08(23.1)	3.10(23.2)	3.10(23.2)	3.13(23.5)	3.15(23.6)

A-3.1.7 Standard EDTA Solution

Dissolve about 20 g of disodium ethylene diamine tetra acetate dihydrate (EDTA), in water and make up to 1 litre with water. Store in a polyethylene bottle. Standardize against 0.5 M zinc sulphate solution.

A-3.1.8 Xylenol Orange — 1 g/l indicator solution of the sodium salt in water.

A-3.1.9 Bromothymol Blue — 1 g/l indicator solution in ethanol 96 percent (v/v),

A-3.2 Procedure

A-3.2.1 Weigh, to the nearest 0.001 g, about 1.5 g of the sample into a 250 ml beaker. Dissolve the sample in 20 ml of the hydrochloric acid (**A-3.1.1**) and add 1 or 2 ml of the nitric acid (**A-3.1.4**) to ensure that any lead present is dissolved. Add 5 ml of the sulphuric acid (**A-3.1.3**) and evaporate until strongly fumes. Cool, then add 100 ml of water, boil for a short time and allow to stand until the following day. Filter the solution through a sintered glass or silica crucible; wash the crucible, collecting the filtrate in a 500 ml volumetric flask. Make the solution up to the mark and mix thoroughly.

A-3.2.2 By means of a pipette, transfer 50 ml of the solution to a 500 ml conical flask; add 200 ml of water and 3 drops of the bromothymol blue indicator (**A-3.1.9**), then add ammonia solution (**A-3.1.5**) drop by drop, until the colour turns blue. Then add the hydrochloric acid (**A-3.1.2**) until a yellow colour returns. Then add 20 ml of the buffer solution (**A-3.1.6**) and 3 drops of the xylenol orange indicator (**A-3.1.8**) and titrate with the disodium EDTA solution (**A-3.1.7**) until the red colour changes to pale yellow.

A-3.3 Calculation

$$\text{Total zinc (as Zn), percent by mass} = \frac{1\,000 \times V \times T}{m}$$

where

V = volume, in ml, of the disodium EDTA solution used;

T = zinc factor, in g/ml, of the disodium EDTA solution; and

m = mass, in g, of the material taken for the test.

A-4 DETERMINATION OF LEAD**A-4.1 Reagents**

A-4.1.1 Potassium Cyanide Solution — 10 percent (m/v).

A-4.1.2 Nitric Acid — 1 : 1 (v/v).

A-4.1.3 Ammonium Hydroxide — 18 N.

A-4.1.4 Chloroform

A-4.1.5 Ferrous Sulphate Solution

10 percent (m/v). This is made alkaline either with sodium hydroxide or ammonium hydroxide and is used to make cyanide solutions harmless before being discharged into the drain.

A-4.1.6 Standard Lead Nitrate Solution

Weigh accurately 0.160 g to 0.165 g of lead nitrate and dissolve it in water. Add a few drops of concentrated nitric acid to make the solution clear. Dilute the solution to 1 litre in a volumetric flask. Mark it as a *Stock Solution*.

A-4.1.6.1 Pipette out 10 ml of *Stock Solution* and dilute to 100 ml in a volumetric flask. Concentration of this standard solution is:

$$\text{Lead (g/ml)} = \frac{M \times 207.19}{331.23 \times 10\,000}$$

where

M = mass, in g, of lead nitrate taken.

A-4.1.7 Dithizone Solution

Weigh accurately about 45 mg to 50 mg of dithizone reagent. Dissolve it in chloroform and dilute it to 500 ml in a volumetric flask in the chloroform.

A-4.1.7.1 Standardization of dithizone solution

Pipette out 25 ml of the standard lead nitrate solution and transfer it to a separating funnel. Add, in the order given, 2 to 3 drops of phenol red indicator; 15 ml of potassium cyanide solution followed by drop by drop addition of dilute nitric acid till the pink colour of the indicator turns yellowish brown. Then, add, drop by drop, strong ammonium hydroxide till the solution becomes distinctly pink again. From a burette, add 5 ml of dithizone solution. Stopper the funnel and shake vigorously, care being taken to release the pressure developed during shaking. Allow to separate the two layers and transfer the pink chloroform layer into alkaline ferrous sulphate solution. The above operation is continued till the last 5 ml portion fails to give the pink coloured complex. Note this pilot reading. Carry out the second titration as described above, adding only 0.2 ml portions of dithizone solution near the end point. Note the exact volume of dithizone solution used. Lead equivalent per millilitre of dithizone solution is calculated by the formula given below:

$$E = \frac{C \times 25}{V}$$

where

E = lead equivalent, in g/ml, of dithizone solution;

C = concentration of lead, in g/ml; and

V = volume, in ml, of the dithizone solution used.

A-4.2 Procedure

A-4.2.1 Weigh accurately 1 g to 1.2 g of zinc dust (*see Note*). Transfer it to 500 ml beaker. Add about 100 ml of water and 10 ml of dilute nitric acid. Heat to boiling. If some zinc remains insoluble, a few drops of concentrated nitric acid may be added to effect complete solution. Cool to room temperature and transfer it to a 250 ml volumetric flask. Add water up to the mark and mix well.

NOTE — Avoid excess weighing of zinc dust and also excessive use of nitric acid. This may give erroneous results.

A-4.2.2 Use 25 ml of this solution for estimation of lead. Add 15 ml of potassium cyanide solution and follow exactly the procedure given for standardization of dithizone solution in **A-4.1.7.1**.

A-4.3 Calculation

$$\text{Lead, (as Pb), percent by mass} = \frac{E \times V \times 1\,000}{M}$$

where

E = lead equivalent in g/ml of dithizone solution;

V = volume, in ml, of dithizone solution; and

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

A-4.4 Alternative Method

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-14** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-5 DETERMINATION OF IRON

A-5.1 Outline of the Method

Iron is determined by visual comparison of colour using potassium thiocyanate.

A-5.2 Apparatus

A-5.2.1 Nessler Cylinder — 50 ml capacity.

A-5.3 Reagents

A-5.3.1 Hydrochloric Acid — 1 : 1 (v/v).

A-5.3.2 Ammonium Persulphate — solid.

A-5.3.3 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.

A-5.3.4 Standard Iron Solution

Dissolve 0.70 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water and add 10 ml of 10 percent (v/v) sulphuric acid. Dilute the solution to exactly one litre. Transfer 10 ml of this solution to a 100 ml volumetric flask and make up the volume to mark with water. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-5.4 Procedure

A-5.4.1 Accurately weigh 10 g of the material, transfer into a beaker and dissolve in about 60 ml of hydrochloric acid. Heat to dissolve with at least 10 ml of the acid in excess, dilute to 150 ml and boil for 5 min. Cool the solution, filter, if necessary, and make up to 500 ml in a volumetric flask.

A-5.4.2 Pipette out 5 ml of this solution into a Nessler cylinder, add 50 mg of ammonium persulphate and 10 ml of butanolic potassium thiocyanate solution. Make up the volume to 50 ml mark. In another Nessler cylinder, carry out a control test without the test solution, but with all the other reagents added in the same amount as to the test solution, and to which 10 ml of the standard iron solution has been added and make up to the same volume.

A-5.4.3 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour of the butanolic layer in the test solution is not darker than the colour produced in the control test.

A-5.5 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-14** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-6 DETERMINATION OF CADMIUM

A-6.1 Outline of the Method

Cadmium is selectively precipitated as cadmium sulphide free from other metallic sulphides and then cadmium sulphide is reacted with excess iodine.

A-6.2 Reagents

A-6.2.1 Nitric Acid — concentrated (*see* IS 264).

A-6.2.2 Sulphuric Acid — approximately 12 N.

A-6.2.3 Hydrogen Sulphide — gas.

A-6.2.4 Sulphuric Acid — 4 N.

A-6.2.5 Hydrochloric Acid — 1 : 2 (v/v).

A-6.2.6 Ammonium Hydroxide — 1 : 4 (v/v).

A-6.2.7 Standard Iodine Solution — 0.1 N.

A-6.2.8 Standard Sodium Thiosulphate Solution — 0.1 N.

A-6.2.9 Starch Indicator Solution

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

A-6.3 Procedure

A-6.3.1 Weigh about 10 g of the material accurately into a porcelain dish. Add slowly concentrated nitric acid (**A-6.2.1**) in small portions at regular intervals with simultaneous stirring and heating till the reaction ceases. Add 10 ml of the nitric acid in excess. Boil for a few minutes with stirring and evaporate carefully on a low flame to reduce the volume to about one-third. Cool, add 15 ml of sulphuric acid (**A-6.2.4**) and stir well. Evaporate slowly with continuous stirring till thick white fumes of sulphuric acid evolve profusely so that all the nitric acid is expelled. Cool, add 50 ml of water, heat and transfer to a 400 ml beaker. Wash with hot water transferring completely the residue and the solution into the beaker. Heat to boiling and filter through Whatman No. 40 filter paper (or equivalent), wash 3 times with warm water collecting the filtrate and the washings in a clean beaker. Dilute this to 200 ml, heat to 70 °C and pass a slow stream of hydrogen sulphide through the solution till it is saturated with hydrogen sulphide. Keep for 5 min. Add ammonium hydroxide (**A-6.2.6**) drop by drop, till a white precipitate just begins to form. Filter through a Whatman filter paper No. 40 (or equivalent) and wash twice with warm water. Discard the filtrate.

A-6.3.2 Dissolve the residue on filter paper in cold hydrochloric acid (**A-6.2.5**) collecting filtrate in a clean beaker. Dissolve any precipitate adhering to the original beaker also in hydrochloric acid (**A-6.2.5**) and transfer to the filter paper. Wash twice the filter paper with hydrochloric acid (**A-6.2.5**) and collect washing along with the filtrate.

A-6.3.3 Dilute the filtrate to 200 ml. Pass hydrogen sulphide gas slowly till the point of saturation at 60 °C. As before, add ammonium hydroxide (**A-6.2.6**) drop by drop just to start precipitation. Again pass hydrogen sulphide and filter through Whatman No. 1 filter paper (or equivalent). Wash twice with warm water. Redissolve the residue this time in hot dilute sulphuric acid. Collect the filtrate and washings.

A-6.3.4 Repeat the precipitation and dissolution till filtrate is shown to be free from zinc on testing a few millilitres of filtrate with hydrochloric acid (**A-6.2.5**) and potassium ferrocyanide.

A-6.3.5 Transfer the final washed residue along with the filter paper into a beaker containing 10 ml of standard iodine solution (accurately measured) and 15 ml of sulphuric acid (**A-6.2.4**). Stir for 5 min and titrate excess of iodine against the standard sodium thiosulphate solution using starch indicator solution towards the end. Titrate 10 ml of iodine against the standard sodium thiosulphate solution.

A-6.4 Calculation

$$\text{Cadmium (as Cd), percent by mass} = \frac{(V_1 - V_2) \times N \times 5.621}{M}$$

where

V_1 = volume, in ml, of thiosulphate required for 10 ml of iodine solution;

V_2 = volume, in ml, of thiosulphate used in the test titration for reacting with excess of iodine;

N = normality of standard sodium thiosulphate solution; and

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

A-7 DETERMINATION OF CHLORIDES**A-7.1 Outline of the Method**

Chlorides are determined by comparing the turbidity produced with silver nitrate solution against that produced by a standard sodium chloride solution.

A-7.2 Apparatus

A-7.2.1 *Nessler Cylinders* — 50 ml capacity.

A-7.3 Reagents

A-7.3.1 *Concentrated Nitric Acid* — concentrated (*see* IS 264).

A-7.3.2 *Standard Chloride Solution*

Dissolve 1.649 g of sodium chloride in water and make up the volume to one litre. Pipette out 10 ml of the solution, dilute with water and make up the solution to 100 ml. One millilitre of this solution contains 0.1 g of chloride (as Cl).

A-7.3.3 *Silver Nitrate Solution* — approximately 2 percent (*m/v*).

A-7.4 Procedure

Weigh accurately 5 g of the material and transfer to a beaker, add 50 ml of water and then cautiously 20 ml of concentrated nitric acid. Cover with a watch glass and heat carefully to boil on a hot plate and keep boiling for 5 min. Cool, filter, if necessary, and make up the filtrate and washing to 250 ml. Transfer 2 ml of this solution to a Nessler cylinder. Add 2 ml of silver nitrate solution and dilute to 50 ml mark. Carry out a control test in the other Nessler cylinder using 1 ml of the standard chloride solution and the same quantities of the other reagents and diluting finally to 50 ml mark. Stir both the solutions with glass rods and compare the turbidity produced in the two cylinders after 5 min.

A-7.4.1 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.

A-7.5 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-13**.

A-8 DETERMINATION OF COPPER**A-8.1 Outline of the Method**

Copper is determined by visual comparison using sodium diethyldithiocarbamate solution.

A-8.2 Apparatus

A-8.2.1 *Nessler Cylinders* — 50 ml capacity.

A-8.3 Reagents

A-8.3.1 *Citric Acid Solution* — 10 percent (m/v).

A-8.3.2 *Gum Arabic Solution* — 1 percent (m/v).

A-8.3.3 *Ammonium Hydroxide* — approximately 1 : 4 (v/v).

A-8.3.4 *Hydrochloric Acid* — concentrated (*see* IS 265).

A-8.3.5 *Hydrochloric Acid* — approximately 1 : 1 (v/v).

A-8.3.6 Sodium Diethyldithiocarbamate Solution

Dissolve 0.1 g of sodium diethyldithiocarbamate in 100 ml of water. Filter, if necessary. Preserve in an amber coloured glass bottle and store in a dark place. This solution should not be used after two weeks.

A-8.3.7 Standard Copper Solution

Dissolve 0.3928 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and make up the volume to one litre with water. Take 100 ml of this solution and further dilute to one litre with water. One millilitre of this solution contains 0.01 mg of copper (as Cu). This dilute solution should be prepared afresh.

A-8.4 Procedure

A-8.4.1 Weigh accurately 10 g of the material and transfer to a beaker. Heat to boiling with 50 ml of dilute hydrochloric acid (**A-8.3.4**). Filter and make up the filtrate to 100 ml. Transfer 10 ml of this solution to a Nessler cylinder. Add 2.5 ml of citric acid solution. Neutralize the excess of acid with ammonium hydroxide and add 10 ml in excess. Add 2 ml to 5 ml of gum arabic solution followed by 10 ml of sodium diethyldithiocarbamate solution. Make up the volume to 100 ml mark, and mix well. Carry out a control test using 50 ml standard copper solution proceeding exactly in the same way as for the material under test. Compare the intensity of the colour produced in the two tests.

A-8.4.2 The limit laid down in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that in the control test.

A-8.5 Determination of Copper Using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) Method

A-8.5.1 Copper may alternatively be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2) or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-9 DETERMINATION OF MOISTURE AND OTHER VOLATILE MATTER**A-9.1 Procedure**

Dry a flat-bottomed weighing bottle and lid separately in an oven maintained at about 100 °C for 30 min. Cool the bottle with lid over concentrated sulphuric acid in a vacuum desiccator and weigh. Introduce in the bottle about 5 g of the material, weighed accurately and place the bottle, containing the material and the lid separately over concentrated sulphuric acid in the vacuum desiccator for 18 h under vacuum. Then remove the weighing bottle, cover it with the lid and weigh.

A-9.2 Calculation

$$\text{Moisture and volatile matter, percent by mass} = 100 \times \frac{M_1 - M_2}{M_1}$$

where

M_1 = mass, in g, of the material before drying; and

M_2 = mass, in g, of the material after drying.

A-10 DETERMINATION OF MATTER INSOLUBLE IN AQUA REGIA**A-10.1 Reagents****A-10.1.1 Aqua Regia**

Prepared by mixing 3 volumes of concentrated hydrochloric acid (*see* IS 265) with one volume of concentrated nitric acid (*see* IS 264).

A-10.2 Procedure

Weigh accurately about 10 g of the material and dissolve by stirring in 30 ml of aqua regia. Heat to boiling and dilute with water to 100 ml. Filter through a tared filter paper wash the residue thoroughly with water till free from chlorides, dry at 105 °C to 110 °C to a constant mass.

A-10.3 Calculation

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

where

M_1 = mass, in g, of the dried residue; and

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

A-11 DETERMINATION OF CALCIUM OXIDE**A-11.1 Reagents****A-11.1.1 Hydrochloric Acid — 5 N.****A-11.1.2 Potassium Hydroxide Solution**

Approximately 10 N. The solution should be freshly prepared from AR quality potassium hydroxide.

A-11.1.3 Buffet Solution (pH 10)

Dissolve 70 g of ammonium chloride in 200 ml of water. Add 570 ml of concentrated ammonia (relative density 0.88 to 0.90) and dilute to one litre with water.

A-11.1.4 Patton and Reader's Indicator (P and R Indicator)

Mix 0.1 g of hydroxy 1-(2-Hydroxy - 4 – sulphonaphthalzo) - 3 - naphthoic acid with 10 g of potassium chloride and grind into a fine mixture in a glass mortar. Preserve in a well stoppered dry amber coloured glass bottle.

A-11.1.5 Eriochrome Black T Indicator

Dissolve 0.5 g of Eriochrome black T indicator in 100 ml of rectified spirit and preserve in amber coloured glass bottle. This indicator should preferably be freshly prepared.

A-11.1.6 Standard EDTA Solution

Weigh 7.444 g of disodium ethylene diamine tetraacetate dihydrate, (EDTA) and dissolve in water. Make up the volume to one litre. Mix thoroughly. The solution is 0.02 M.

A-11.1.7 *Potassium Cyanide* — pure.

A-11.1.8 *Hydroxylamine Hydrochloride* — pure.

A-11.2 Procedure

Weigh accurately 5 g of the material and transfer to a beaker. Heat to boiling with 50 ml of hydrochloric acid (**A-11.1.1**). Filter and make up the filtrate to 100 ml. Take two portions of 25 ml each in two conical flasks or white porcelain dish of 100 ml capacity. Add about 0.2 g to 0.4 g P and R indicator and quickly titrate till the colour changed to violet blue. Note the volume of EDTA consumed. Add the EDTA volume less 1.0 ml directly to the second flask. Add 5 ml of potassium hydroxide solution and about 50 g each of potassium cyanide and hydroxylamine hydrochloride. Add 0.4 g indicator and immediately titrate with continuous stirring until no further colour change occurs and the test solution is clear blue without any tinge of violet. Volume of EDTA consumed in the final titration should be taken for calculation.

A-11.2.1 Calculation

$$\text{Calcium oxide (as CaO), percent by mass} = \frac{0.112 \ 16 \ V}{M}$$

where

V = volume, in ml, of 0.02 M EDTA solution required for final titration; and

M = mass, in g, of the material contained in the aliquot of solution taken for the titration.

A-12 DETERMINATION OF PARTICLE SIZE

A-12.1 Procedure

Place 75 micron IS Sieve on top of the 45 micron IS Sieve, together with bottom receiver. Weigh about 100 g of the material and transfer it on the uppermost sieve. Shake the sieves for 15 min by means of a mechanical sieve shaker. Lightly brush with a clean, dry paint brush or camel hair brush after shaking.

A-12.1.1 Weigh separately the material passing through each sieve and express it as percentage of the mass of the material taken for the test.

A-13 ION CHROMATOGRAPHY FOR CHLORIDES

A-13.1 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.

A-13.2 Equipment

A-13.2.1 *Anion Guard Column* — a protector of the separator column.

A-13.2.2 *Anion Separator Column* — suitable for selective separation of ions under analysis.

A-13.2.3 *Anion Suppressor Device*

Anion micromembrane suppressor is used to analyse the data Detector: Conductivity detector.

A-13.2.4 *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 1 000 μl be used to determine ionic concentration as per instrument manual and practice.

A-13.3 Reagents and Standards

A-13.3.1 *Glass or Polyethylene Sample Bottles.*

A-13.3.2 *Distilled Water or Deionized Water Free from the Anions of Interest.*

A-13.3.3 *Eluent* — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litres of water.

A-13.3.4 *Micromembrane Suppressor Solution (0.025 N of sulphuric acid)*

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

A-13.4 Standard Solutions

A-13.4.1 *Chloride* — Dissolve NaCl, 1.648 5 g in 1 litre of reagent water

A-13.4.2 *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 to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-13.5 *Procedure*

Dissolve between 1 g to 5 g samples in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 to 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.

A-13.5.1 *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.

A-14 DETERMINATION OF IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-14.1 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.

A-14.2 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 4. 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 4 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 4 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences*(Clauses A-14.2 and A-14.4)*

SI 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)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe

A-14.3 Reagents and Solutions**A-14.3.1. Nitric Acid (65 percent) Suprapure****A-14.3.2 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 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-14.3.3 Standard Solution

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and 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.

A-14.3.4 Sample preparation

Weigh about 2.5 g of the 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.

A-14.3.5 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, polytetrafluoroethylene (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.

A-14.4 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 4.

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.

A-14.5 Procedure

A-14.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-14.3.5). 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.

A-14.5.2 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 lead (and/or Iron) 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.

A-14.6 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 B

(Clause 6)

SAMPLING OF METALLIC ZINC POWDER (ZINC DUST)

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Samples shall not be drawn in an exposed place.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B 1.5 The sample shall be placed in clean, dry, airtight glass or other suitable containers which have no action on the material.

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

B-1.7 Each sample container shall be sealed airtight with a stopper after filling, and marked with all particulars of the material (*see 5.2*) and the date of sampling.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2 SCALE OF SAMPLING**B.2.1 Lot**

B-2.1.1 In any consignment of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different sizes of batches of manufacture, the containers belonging to the same size and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out from each lot separately. The number of containers to be selected from a lot shall be in accordance with Table 5.

B-2.1.3 In order to ensure randomness of selection, random number tables shall be used. In case such tables are not available the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3, up to r , where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every r^{th} container thus counted shall be withdrawn to constitute the required number of containers in the samples.

Table 5 Number of Containers to be Selected for Sampling

(Clause B-2.1.2)

Lot Size	Sample Size
N	n
(1)	(2)
Up to 15	3
16 to 50	4
51 to 100	5
101 to 150	7
151 and above	10

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.1.2** and **B-2.1.3**, a small representative portion of the material not less than 100 g shall be taken out.

B-3.2 Out of these portions, small but equal quantities of the material shall be taken and thoroughly mixed to form a composite sample not less than 200 g. The 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.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of the individual samples representing the n containers shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual samples and composite samples shall be transferred to separate sample containers. The sample containers shall be sealed and labelled with full identification particulars (*see B-1.7*).

B-3.5 The referee samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two parties.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of metallic zinc and particle size shall be performed on the individual samples.

B-4.2 Test for all other characteristics given in **4** shall be performed on the composite sample.

B-5 CRITERIA FOR CONFORMITY**B-5.1 For Individual Samples**

From each set of test results for metallic zinc the mean (X) and range (R) of test results shall be computed separately (the range being defined as the difference between the maximum and the minimum values of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of metallic zinc if $(X - 0.6 R)$ as calculated from the relevant test results is greater than or equal to 94.

B-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite samples, the test result for each of the characteristics shall satisfy the relevant requirements specified in **4** and Table 1.