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IS 4611 (1991): Metallic zinc powder (zinc dust) [CHD 1: Inorganic Chemicals]



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भारतीय मानक  
धात्विक जस्ता चूर्ण ( जस्ता धूल ) — विशिष्ट  
( दूसरा पुनरीक्षण )

*Indian Standard*  
**METALLIC ZINC POWDER ( ZINC DUST ) —  
SPECIFICATION**  
*( Second Revision )*

UDC 669.5 - 492.2

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## FOREWORD

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

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 electrogalvanizing 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.

This standard was originally published in 1967 and was revised in 1973. In this revision, the methods for determination of total zinc and metallic zinc have been modified to bring them in line with the methods given in ISO 3549 : 1976 'Zinc dust pigment for paints'. A new requirement for calcium oxide along with the test method has also been incorporated. The method for determination of lead has also been modified. Limits for various requirements for both the grades of metallic zinc have been modified. Additional requirement on free chloride for the material for use in explosive and pyrotechnic industry along with its method of test has been deleted.

Clause 4.3 of this standard calls for an agreement between the purchaser and the seller/manufacturers.

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 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**AMENDMENT NO. 1    FEBRUARY 2003**  
**TO**  
**IS 4611 : 1991   METALLIC ZINC POWDER**  
**(ZINC DUST) — SPECIFICATION**  
( *Second Revision* )

( *Page 2, clause A-2.1* ) — Substitute the following for the existing:

**A-2.1   Volumetric Method**

**A-2.1.0   *Outline of the Method***

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

**A-2.1.1   *Reagents***

**A-2.1.1.1   *Ammonium ferric sulphate solution*** — Dissolve 50 g of ammonium ferric sulphate in 200 ml of water.

**A-2.1.1.2   *Dilute sulphuric acid solution, 5N***

**A-2.1.1.3   *Standard potassium permanganate solution , 0.1N***

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

**A-2.1.2   *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 5N 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-2.1.3   *Calculation***

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

(CHD 1)

# *Indian Standard*

## **METALLIC ZINC POWDER ( ZINC DUST ) — SPECIFICATION**

### *( Second Revision )*

**1 SCOPE**

1.1 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**

2.1 The following Indian Standards are the necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
265 : 1987	Hydrochloric acid ( <i>third revision</i> )
1070 : 1977	Water for general laboratory use ( <i>second revision</i> )

**3 GRADES**

3.1 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 )**  
( *Clause 4.2* )

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
iv)	Iron as (Fe), percent by mass, <i>Max</i>	0.05	0.05	A-5
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
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.

## 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

**A-1.1** Unless otherwise specified, pure chemicals and distilled water (see IS 1070 : 1977) 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-2.0** Two methods are prescribed, namely, volumetric method and gas evolution method. In case of dispute, the volumetric method shall be the referee method.

#### A-2.1 Volumetric Method

##### A-2.1.0 Outline of the Method

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

##### A-2.1.1 Reagents

###### A-2.1.1.1 Ferric chloride solution

Dissolve 20 g of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) in 100 ml of water containing 20 ml of 20 percent sodium acetate solution.

###### A-2.1.1.2 Zimmerman-Reinhardt solution

Dissolve 67 g of manganese sulphate ( $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ ) in 130 ml of concentrated sulphuric acid and add 138 ml of 85 percent phosphoric acid and dilute to one litre with water.

###### A-2.1.1.3 Standard potassium permanganate solution, 0.1 N.

##### A-2.1.2 Procedure

Take 60 ml of the ferric chloride solution into a 500 ml of conical flask. Weigh accurately and transfer about 0.2 g of the sample into the solution in the flask without touching the side.

Close the flask with a rubber stopper tightly and agitate constantly for 15 minutes till all the particles are dissolved. Open the flask, add 30 ml of the Zimmerman-Reinhardt solution, dilute to 250 ml with water and titrate against standard potassium permanganate solution. Carry out a blank titration using 60 ml of the ferric chloride solution and also adding the same amount of reagents and keeping for the same time as in the test.

##### A-2.1.3 Calculation

Metallic zinc, percent by mass

$$= \frac{3.27 \times (V_1 - V_2) \times N}{M}$$

where

$V_1$  = volume, in ml, of standard potassium permanganate solution required for titration of the material;

$V_2$  = volume, in ml, of standard potassium permanganate solution required for the blank;

$N$  = normality of potassium permanganate solution; and

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

### A-2.2 Gas Evolution Method

#### A-2.2.1 Reagents

##### A-2.2.1.1 Hydrochloric acid/copper (I) chloride reagent

Dissolve 0.05 g of copper (I) chloride ( $\text{Cu}_2\text{Cl}_2$ ) in 700 ml of hydrochloric acid (relative density 1.18) and dilute to one litre with water.

##### A-2.2.1.2 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-2.2.2 Apparatus****A-2.2.2.1 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.

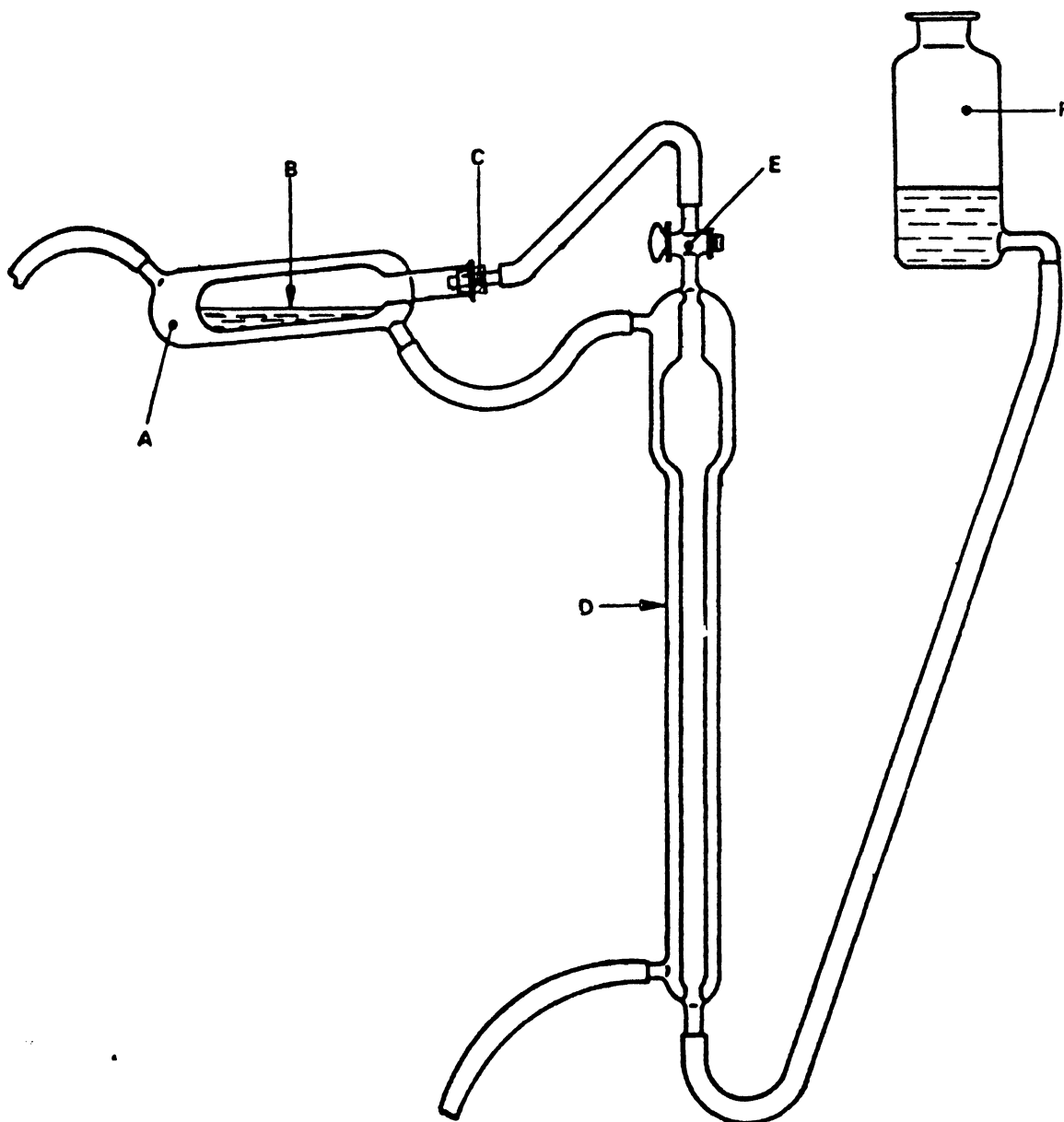
**A-2.2.2.2 Boat,** made of a suitable plastic material or porcelain with lid.

**A.2.2.3 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-2.2.3.1 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-2.2.3.2 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-2.2.1.1) 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.2.2.3.2 )

Temperature, °C	Barometer Reading, kPa( mmHg )						
	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**

- 1 Careful initial and vigorous final shaking of the reaction flask are essential.
- 2 Check that the apparatus is air-tight by opening the tap to connect the flask (A); levelling the solution in the levelling bottle stopping the bottle and lowering it. The level of the solution in the gas burette should remain constant.

**A-2.2.4 Calculation**

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  $t^\circ\text{C}$ ;  
 $m$  = 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 one 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 hydroxylammonium chloride ( $\text{NH}_2\text{OH} \cdot \text{HCl}$ ) 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 one litre with water.

**Table 3 Saturated Water Vapour Pressures, kPa ( mmHg )**  
( Clause A.2.2.3.2 )

Temperature	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)	0.80(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)
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)	0.93(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)	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)	1.07(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)	1.15(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)
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)	1.31(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)
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)	1.49(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)	1.59(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)	1.69(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)
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)	1.92(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)	2.05(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)	2.19(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)	2.32(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)	2.47(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)	2.63(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)	3.06(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 tetraacetate dihydrate (EDTA), in water and make up to one 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**

Total zinc ( as Zn ), percent by mass

$$= \frac{1\,000\, V\, T}{m}$$

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 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 one 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 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 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

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.5 DETERMINATION OF IRON

#### A-5.0 Outline of the Method

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

#### A-5.1 Apparatus

##### A-5.1.1 Nessler Cylinder

50-ml capacity.

#### A-5.2 Reagents

##### A-5.2.1 Hydrochloric Acid

1 : 1 (v/v).

##### A-5.2.2 Ammonium Persulphate

solid.

##### A-5.2.3 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.

#### A-5.2.4 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-5.3 Procedure

**A-5.3.1** Accurately weigh 10.0 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 minutes. Cool the solution, filter, if necessary, and make up to 500 ml in a volumetric flask.

**A-5.3.2** 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-5.3.3** 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-6 DETERMINATION OF CADMIUM

#### A-6.0 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-6.1 Reagents

##### A-6.1.1 Nitric Acid

concentrated ( see IS 264 : 1976 ).

##### A-6.1.2 Sulphuric Acid

approximately 12 N.

##### A-6.1.3 Hydrogen Sulphide

gas.

##### A-6.1.4 Sulphuric Acid

4 N.

##### A-6.1.5 Hydrochloric Acid

1 : 2 (v/v).

**A-6.1.6 Ammonium Hydroxide**

1 : 4 (v/v).

**A-6.1.7 Standard Iodine Solution**

0.1 N.

**A-6.1.8 Standard Sodium Thiosulphate Solution**

0.1 N.

**A-6.1.9 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 one litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

**A-6.2 Procedure**

**A-6.2.1** Weigh about 10 g of the material accurately into a porcelain dish. Add slowly concentrated nitric acid (A-6.1.1) 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-6.1.2) 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 minutes. Add ammonium hydroxide (A-6.1.6) 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-6.2.2** Dissolve the residue on filter paper in cold hydrochloric acid (A-6.1.5) collecting filtrate in a clean beaker. Dissolve any precipitate adhering to the original beaker also in hydrochloric acid (A-6.1.5) and transfer to the filter paper. Wash twice the filter paper with hydrochloric acid (A-6.1.5) and collect washing along with the filtrate.

**A-6.2.3** 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-6.1.6) 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-6.2.4** 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-6.1.5) and potassium ferrocyanide.

**A-6.2.5** 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-6.1.4). Stir for 5 minutes 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-6.3 Calculation**

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-7.0 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-7.1 Nessler Cylinders**

50 ml capacity.

**A-7.2 Reagents****A-7.2.1 Concentrated Nitric Acid**

concentrated (see IS 264 : 1976).

**A-7.2.2 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-7.2.3 Silver Nitrate Solution**

approximately 2 percent (m/v).

**A-7.3 Procedure**

Weigh accurately 5.0 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 minutes. -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 minutes.

**A-7.3.1** 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-8 DETERMINATION OF COPPER

### A-8.0 Outline of the Method

Copper is determined by visual comparison using sodium diethyldithiocarbamate solution.

#### A-8.1 Apparatus

##### A-8.1.1 Nessler Cylinders

50-ml capacity.

#### A-8.2 Reagents

##### A-8.2.1 Citric Acid Solution

10 percent ( m/v ).

##### A-8.2.2 Gum Arabic Solution

1 percent ( m/v ).

##### A-8.2.3 Ammonium Hydroxide

approximately 1 : 4 ( v/v ).

##### A-8.2.4 Hydrochloric Acid

concentrated ( see IS 265 : 1987 ).

##### A-8.2.5 Hydrochloric Acid

approximately 1 : 1 ( v/v ).

##### A-8.2.6 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-8.2.7 Standard Copper Solution

Dissolve 0.392 8 g of copper sulphate (  $\text{CuSO}_4 \cdot \text{SH}_2\text{O}$  ) 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-8.3 Procedure

**A-8.3.1** Weigh accurately 10.0 g of the material and transfer to a beaker. Heat to boiling with 50 ml of dilute hydrochloric acid ( **A-8.2.4** ). 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 2.5 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-8.3.2** 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-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 minutes. 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 hours under vacuum. Then remove the weighing bottle, cover it with the lid and weigh.

### A-9.2 Calculation

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 : 1987 ) with one volume of concentrated nitric acid ( see IS 264 : 1976 ).

### 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 to 110°C to a constant mass.

### A-10.3 Calculation

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

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 Buffer Solution ( pH 10 )

Dissolve 70 g of ammonium chloride in 200 ml of water. Add 570 ml of concentrated ammonia ( relative density 0.88-0.90 ) 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 - sulpho-naphthalzo ) - 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 5.0 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 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

$$\begin{array}{l} \text{Calcium oxide ( as GaO ),} \\ \text{percent by mass} \end{array} = \frac{0.11216 V}{M}$$

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 minutes 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.



**ANNEX B**

( Clause 6 )

**SAMPLING OF METALLIC ZINC POWDER ( ZINC DUST )****B-1 GENERAL REQUIREMENTS OF SAMPLING**

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall not be drawn in an exposed place.

**B-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.

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

**B-1.4** The sample shall be placed in clean, dry, airtight glass or other suitable containers which have no action on the material.

**B-1.5** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.6** 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-1.7** 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 4.

**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 4 Number of Containers to be Selected for Sampling**  
( Clause B-2.1.1 )

Lot Size $N$	Sample Size $n$
( 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-1.6** ).

**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-4.1** 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 (  $\bar{X}$  ) 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  $(\bar{X} - 0.6 R)$  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.

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