IS 44 (1991): Iron Oxide Pigments for Paints [CHD 20: Paints, Varnishes and Related Products]

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IS 44 : 1991
(Reaffirmed - 2012)

भारतीय मानक

रंग रोगन के लिए लौह ऑक्साईड वर्णक — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

IRON OXIDE PIGMENTS FOR PAINTS — SPECIFICATION

(Second Revision)

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BUREAU OF INDIAN STANDARDS
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Price Group 5

Raw Materials for Paint Industry Sectional Committee, CHD 021

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

The standards IS 44 : 1950 'Specification for black oxide of iron for paints', IS 45 : 1950 'Specification for manufactured red oxide of iron for paints', IS 46 : 1950 'Specification for natural red oxide of iron for paints', IS 47 : 1950 'Specification for ochre for paints', IS 48 : 1950 'Specification for natural sienna (raw and burnt) for paints' and IS 49 : 1950 'Specification for natural umber (raw and burnt) for paints' were first issued in 1950. These were based on the interim co-ordinated drafts produced with the assistance of the representatives of manufacturers and of various departments, and authorities of the Government of India by the Coordinating Subcommittee of No. 5 Standing Committee on specification for paints and allied stores of the General Headquarters, India (now Army Headquarters).

In 1969, all the above standards were amalgamated into IS 44 : 1969 'Specification for iron oxide pigments for paints *(first revision*)'. The revised specification stipulated requirements for 6 classes of the material corresponding to the materials covered in the earlier set of six standards (IS 44 — IS 49). The requirement for sulphur and sulphur compounds for class 2 material (manufactured red oxide) was made more stringent. Besides, requirements for volatile matter and water soluble matter had been modified and unified for class 3 to 6. The method of determination of carbonates as carbon dioxide was also modified. New requirements for limits for ferrous iron and calcium compounds were also introduced for class 2 material. Limits for manganese content had also been fixed for classes 5 and 6.

The standard had also been aligned with IND/ME 38 (a) 'Red oxide of iron, dry, lead-free' issued by the Department of Standardization, Ministry of Defence, Government of India.

In this second revision, classes have been designated as grades. Requirements for in additional grade, namely, iron oxide yellow, have been added. Requirements for total iron (as Fe_2O_3) for the Grades 2, 3 and 4; volatile matter for Grade 4 and matter soluble in water for Grade 1 have been modified. Three new requirements (a) sum of water soluble chloride and sulphate, (b) *p*H of aqueous extract, and (c) loss on ignition have been added. The requirements for sulphur and compounds of sulphur have been deleted.

In preparing this standard, assistance has been derived from the following standards:

1) ISO 1248-1974 'Iron oxide pigments for paints', issued by International Organization for Standardization (ISO)
2) ASTM D 768-81 'Standard specification for yellow iron oxide hydrated', issued by American Society for Testing and Materials (ASTM)
3) ASTM D 769-81 'Standard specification for black synthetic iron oxide', issued by American Society for Testing and Materials (ASTM)
4) ASTM D 3721-78 'Standard specification for synthetic iron oxide pigments', issued by American Society for Testing and Materials (ASTM)
5) ASTM D 3722-82 'Standard specification for natural red and brown iron oxide pigments', issued by American Society for Testing and Materials (ASTM)
6) ASTM D 3724-82 'Standard specification for synthetic brown iron oxide pigments', issued by American Society for Testing and Materials (ASTM)

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 accodance 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 SEPTEMBER 2000
TO
IS 44 : 1991 IRON OXIDE PIGMENTS FOR PAINTS — SPECIFICATION
(*Second Revision*)

(*Para* 2, *Table* 1, *col* 11) — Substitute 'Ref to Cl No. in IS 33 : 1992' *for* 'Ref to Cl No. in IS 33 : 1991'.

[*Page* 2, *Table* 1, *Sl No.* (iii)] — Substitute '± 10% of approved sample' *for* '± 10 of approved sample'.

[*Page* 2, *Table* 1, *Sl N*o. (iv)(a), *col* 11] — Substitute '11' for '—'.

[*Page* 2, *Table* 1*, Sl No.* (iv)(b), *col* 11]— Substitute '11' *for* '—'.

(CHD 21)

Reprography Unit, BIS, New Delhi, India

Indian Standard

IRON OXIDE PIGMENTS FOR PAINTS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for natural and synthetic iron oxide pigments.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
33 : 1991	Methods of sampling and test for inorganic pigments and extenders for paint (*third revision)*
1070 : 1977	Water for general laboratory use (*second revision)*
1303 : 1983	Glossary of terms relating to paints (*second revision)*
2263 : 1979	Methods of preparation of indicator solution (*first revision)*
2316 : 1968	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision)*

3 TERMINOLOGY

For the purpose of this standard, definitions given under 2 of IS 33 : 1991 and IS 1303 : 1983 shall apply.

4 GRADES AND TYPES

4.1 Grades

There shall be seven grades of material, namely,

Grade 1 Black oxide,
Grade 2 Synthetic red oxide,
Grade 3 Natural red oxide,
Grade 4 Ochre,
Grade 5 Natural sienna,
Grade 6 Natural umber, and
Grade 7 Synthetic iron oxide yellow hydrated.

4.2 Types

4.2.1 Grade 4 shall have two types:

Type A — Red
Type B — Yellow

4.2.2 Grade 5 and 6 material shall have two types:

Type C — Raw
Type D — Burnt

5 REQUIREMENTS

5.1 Composition

5.1.1 *Grade 1*

The material shall be synthetic ferro-ferric oxide and shall be free from added colouring matter and water soluble iron salts. The colour shall be entirely due to inorganic iron compounds.

5.1.2 *Grade 2*

The material shall consist of wholly manufactured oxide of iron and shall be free from added colouring matter and water soluble iron salts. The colour shall be entirely due to inorganic compounds of iron.

5.1.3 *Grade 3*

The material shall be a natural product, the colour of which shall be entirely due to inorganic compounds of iron.

5.1.4 *Grade 4*

The material shall be a naturally available hydrated oxide of iron. It shall be free from added colouring matter and impurities. The colour shall be entirely due to inorganic compounds of iron.

5.1.5 *Grade 5 and Grade 6*

5.1.5.1 Type (C) material shall be a hydrated oxide of iron together with the manganese salt that is naturally associated with it. The colour of the material shall be entirely due to inorganic compounds of iron and shall contain no organic colouring matter.

5.1.5.2 Type (D) material shall be produced by calcination of type (c) and shall be free from admixture of any other substance.

5.1.6 *Grade 7*

The material shall consist of synthetic oxide of iron by chemical reaction and shall be free from salts. The colour shall be entirely due to inorganic compounds of iron and shall be free from admixture of any other substance.

5.2 Form and Condition

The material shall be supplied in the form of dry powder or in such a condition that it may be reduced to powder form by crushing, without grinding action, under palette knife.

5.3 All grades and types of the material shall conform to the requirements given in Table 1.

5.4 Lead Free Material

When lead free material is required, it shall not contain more than 0.03 percent of lead or compounds of lead or a mixture of both, calculated as metallic lead (Pb), when tested as prescribed under **25** of IS 33 : 1991.

6 TEST METHODS

6.1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 :1977) shall be employed for tests.

NOTE – 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

7 PACKING AND MARKING

7.1 Packing

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

Table 1 Requirement for Iron Oxide Pigment for Paints

(*Clause* 5.3)

Sl No.	Characteristic	Requirements for Grade							Method of Test	
		1	2	3	4	5	6	7	Annex	Ref to Cl No. in IS 33 : 1991
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
i)	Volatile matter, percent by mass, *Max*	2.5	0.5	0.5	1.0	0.5	0.5	1.0	—	7
ii)	Residue on sieve, percent by mass, *Max*	0.5	0.5	0.5	0.5	0.5	0.5	0.5	—	8
iii)	Oil absorption	**± 10 of approved sample**							—	9
iv)	Colour									
	a) Staining power	**Not inferior to the approved sample**							—	—
	b) Undertone	— Close match to the approved sample —							—	—
v)	Matter soluble in water, percent by mass, *Max*	1.0	0.5	0.5	0.5	0.5	0.5	0.3	—	17
vi)	Sum of water soluble chloride and sulphate expressed as Cl and SO_4, percent by mass, *Max*	–	0.1	—	–	–	–	0.1	A	–
vii)	Acidity (as H_2SO_4) or alkalinity (as NaOH), percent by mass, *Max*	0.1	0.1	0.1	0.1	0.1	0.1	0.1	—	18
viii)	*pH* of aqueous extract	Within ± 1 of approved sample							—	—
ix)	Total iron (as Fe_2O_3), percent by mass, *Min*	79	94	80	45	—	—	83	B	—
x)	Ferrous iron (as FeO), percent by mass,	20 *Min*	1.5 *Max*	—	–	–	–	–	C	—
xi)	Carbonates (as CO_2), percent by mass, *Max*	0.5	—	—	—	—	0.5	—	D	—
xii)	Calcium compounds (as CaO), percent by mass, *Max*	—	2.25	—	5.0	2.5	2.5	—	E	—
xiii)	Manganese content (as MnO_2), percent by mass, *Max*	—	—	—	—	1	15	—	F	—
xiv)	Loss on ignition, percent by mass, *Max*	–	–		–	–	–	13	G	—

7.2 Marking

7.2.1 The containers shall be marked with the following information:

a) Name of the material;

b) Grade and type of the material;

c) Indication of the source of manufacture;

d) Net mass of the material;

e) Batch No. or Lot No. in code or otherwise; and

f) Month and year of manufacture.

7.2.2 The containers may also be marked with the Standard Mark.

8 SAMPLING

8.1 Representative samples of the material shall be drawn as prescribed under **4** of IS 33 : 1991.

ANNEX A

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF WATER SOLUBLE CHLORIDE AND SULPHATE

A-0 PREPARATION OF AQUEOUS EXTRACT

Weigh about 5 g of pigment in a beaker. Add 200 ml of water and stir and boil for 5 minutes. Cool rapidly to room temperature and transfer to 250 ml volumetric flask and dilute to mark with water. Mix thoroughly by shaking and inversion. Filter it through filter paper and preserve the solution for determination of sulphate and chloride.

A-1 DETERMINATION OF SULPHATE

A-1.0 Outline of Method

The solution is heated and barium chloride is added to the solution. The barium sulphate precipitate, thus obtained, is dried and weighed.

A-1.1 Reagents

A-1.1.1 *Hydrochloric Acid* — concentrated.

A-1.1.2 *Barium Chloride Solution* — 5 percent. Weigh about 25 g of barium chloride into a beaker. Add about 200 ml of water and stir and boil for 5 minutes. Cool to room temperature and transfer to 500-ml volumetric flask and dilute to mark with water. Mix thoroughly by shaking and inversion.

A-1.2 Procedure

Take 50 ml of the clear aqueous extract obtained in **A-1.0,** in a beaker, acidify with 3 ml of the hydrochloric acid and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add 5 percent barium chloride solution drop by drop, to the hot solution until in slight excess, and allow the solution to stand overnight. Decant the supernatant liquid through a tared sintered crucible (P 10 or P 16). Wash it free from chloride. Ignite it gently, and then at red heat. Cool it in a desiccator and weigh to the nearest 1 mg.

A-1.3 Calculation

Calculate the water soluble sulphate content (as SO_4) percentage by mass, as given below:

$$\text{Water soluble sulphate content, percent by mass} = \frac{206 \times M_1}{M_2}$$

where

M_1 = mass in g, of barium sulphate precipitate; and

M_2 = mass in g, of pigment in the aliquot.

Report the result in two decimal places.

A-2 DETERMINATION OF CHLORIDE

A-2.0 General

A-2.0.1 *Outline of the Method*

The aqueous extract is titrated with silver nitrate solution using potassium chromate solution as indicator.

A-2.1 Reagents

A-2.1.1 *Silver Nitrate Solution* — 0.01 N (*see* IS 2316 : 1968).

A-2.1.2 *Potassium Chromate Solution* — 5 percent.

A-2.2 Procedure

Take 50 ml of the clear aqueous extract obtained in **A-0**, in a beaker and add 1 ml of 5 percent potassium chromate solution. Titrate with 0.01 N silver nitrate solution, slowly and with vigorous shaking, until a faint reddish brown colour persists. Carry out a blank determination by adding 1 ml of 5 percent potassium chromate solution to

50 ml of water and titrate with silver nitrate solution until the colour matches to that of the previous titration making due allowance for any opalescence or turbidity.

NOTE — Alternatively the end point of the titration may be determined by potentiometric indication.

A-2.3 Calculation

Calculate the water soluble chloride content expressed as Cl, percentage by mass, as given below:

$$\text{Water soluble chloride content, percent by mass} = 0.177\,5 \times \frac{(V_1 - V_2)}{M}$$

where

V_1 = the volume, in millilitres of 0.01 N silver nitrate solution required by the test portion;

V_2 = the volume, in millilitres of 0.01 N silver nitrate solution required for the blank determination; and

M = the mass in g, of pigment in the aliquot.

Report the result in two decimal places.

A-3 SUM OF WATER SOLUBLE CHLORIDE AND SULPHATE

Add the water soluble sulphate content and chloride content, percent by mass

Sum of water soluble chloride and sulphates, percent by mass = Water soluble sulphate, percent by mass + Water soluble chloride, percent by mass

ANNEX B

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF TOTAL IRON

B-0 GENERAL

Two methods of determination, Method 1 — external indicator method and Method 2 — internal indicator method; have been prescribed In case of dispute, Method 1 shall be used.

B-1 METHOD 1 — EXTERNAL INDICATOR METHOD

B-1.0 Outline of the Method

The ferric iron is reduced to ferrous state by stannous chloride and solution titrated against standard potassium dichromate solution using potassium ferricyanide as external indicator.

B-1.1 Reagents

B-1.1.1 *Hydrochloric Acid* — concentrated.

B-1.1.2 *Standard Potassium Dichromate Solution* — 0.1 N (*see* IS 2316 : 1963).

B-1.1.3 *Stannous Chloride Solution*

Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

B-1.1.4 *Mercuric Chloride Solution* — saturated.

B-1.1.5 *Potassium Ferricyanide Indicator* — (*see* IS 2263 : 1962).

B-1.2 Procedure

B-1.2.1 Weigh accurately 0.25 to 0.40 g of the material, dried as prescribed under **6** of IS 33 : 1990 and transfer to a 500-ml conical flask. Add 25 ml of the hydrochloric acid and warm gently until clear solution is obtained. The addition of a few drops of stannous chloride solution after adding the acid gently assists dissolution of iron. If the solution of the sample is still not clear, digest the solution on a hot-plate, a evaporate to dryness and redissolve in hydrochloric acid as above. Filter into a long necked flask and dilute the solution to about 50 ml. Bring the solution to boil and keeping the solution hot, add stannous chloride solution in drops until the solution is colourless. It is best to keep a watch-glass on the flask while adding the stannous chloride solution with agitation of the hot solution after each addition. If too much stannous chloride is added by mistake, add potassium permanganate to the solution until a yellow colour appears, then adding one or two drops in excess.

NOTE — Only a very slight excess of stannous chloride solution should be added to form a slight silky white precipitate with mercuric chloride solution added subsequently. Reduction should be repeated if a black or grey precipitate is obtained.

Cool the solution rapidly and dilute to 150 to 200 ml with water. Add a slight excess of mercuric chloride solution (normally 5 ml) when a silky white precipitate will be formed.

B-1.2.2 Mix thoroughly and wash down the sides of the flask with cold water. Titrate the contents of the flask against standard potassium dichromate solution using potassium ferricyanide as external indicator.

B-1.2.3 *Calculation*

$$\text{Total iron (as } Fe_2O_8 \text{), percent by mass} = \frac{7{\cdot}984 \times N \times V}{M}$$

where

N = normality of standard potassium dichromate solution;

V = volume, in ml, of standard potassium dichromate solution used; and

M = mass, in g, of material taken.

B-2 METHOD 2 — INTERNAL INDICATOR METHOD

B-2.0 Outline of the Method

The ferric iron is reduced by stannous chloride to ferrous state titrated against standard potassium dichromate solution using sodium diphenylamine sulphonate solution as indicator.

B-2.1 Reagents

B-2.1.1 *Standard Potassium Dichromate* — 0.1 N (*see* IS 2316 : 1968).

B-2.1.2 *Stannous Chloride Solution* — Same as **B-1.1.3.**

B-2.1.3 *Mercuric Chloride Solution* — saturated.

B-2.1.4 *Sulphuric Acid – Phosphoric Acid Mixture*

Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this 150 ml of phosphoric acid (85 percent *w/w*).

B-2.1.5 *Sodium Diphenylamine Sulphonate Indicator Solution*

Dissolve 0.2 to 0.3 g of sodium diphenylamine sulphonate in 100 ml of hot water. Cool and store in amber-coloured bottle.

B-2.2 Procedure

B-2.2.1 Proceed exactly as given in **B-1.2.1.**

B-2.2.2 Add 15 ml of sulphuric acid-phosphoric acid mixture and dilute to 100 ml. Add 5 drops of sodium diphenylamine sulphonate indicator solution and titrate with standard dichromate solution with constant stirring until the solution begins to darken. Complete the titration by adding the dichromate solution in drops until a permanent violet colouration is obtained. Carry out a blank determination with the reagents alone using the same procedure.

B-2.2.3 *Calculation*

$$\text{Total iron (} Fe_2O_3 \text{), percent by mass} = \frac{(V_1 - V_2) \times N \times 7{\cdot}984}{M}$$

where

V_1 = volume, in ml, of the standard potassium dichromate solution required to titrate test solution;

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

N = normality of standard potassium dichromate solution; and

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

ANNEX C

[*Table* 1, *Sl No.* (x)]

DETERMINATION OF FERROUS IRON

C-0 OUTLINE OF METHOD

A solution of the material in concentrated hydrochloric acid is titrated with standard potassium dichromate solution using potassium ferricyanide as an external indicator.

C-1 REAGENTS

C-1.1 Hydrochloric Acid — concentrated.

C-1.2 Standard Potassium Dichromate Solution – 0.1 N(*see* IS 2316 : 1968).

C-1.3 Potassium Ferricyanide Indicator — (*see* IS 2263 : 1979).

C-2 PROCEDURE

C-2.1 Weigh accurately about 0.3 g of sample and transfer to a 250-ml conical flask. Pour 25 ml of hydrochloric acid and titrate immediately with standard potassium dichromate solution using potassium ferricyanide as an external indicator. Run a blank at the same time and under same condition.

C-3 CALCULATION

$$\text{Ferrous iron (FeO), percent by mass} = \frac{7{\cdot}185 \times (V_1 - V_2) \times N}{M}$$

where

V_1 = volume, in ml, of standard potassium dichromate solution required to titrate test solution;

V_2 = volume, in ml, of standard potassium dichromate solution required for blank;

N = normality of standard potassium dichromate solution; and

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

ANNEX D

[*Table* 1, *Sl No.* (xi)]

DETERMINATION OF CARBONATES AS CARBON DIOXIDE

D-0 OUTLINE OF THE METHOD

A known mass of the sample is made to react with the acid and the liberated carbon dioxide freed from impurities, is absorbed in previously weighed soda asbestos bulbs and weighed. From the difference in weights, the percentage of carbon dioxide is calculated.

D-1 APPARATUS

D-1.1 The assembly of apparatus is shown in Fig. 1. In order to minimize pressure (vacuum) loss through the train, all joints should be properly sealed.

D-2 REAGENTS

D-2.1 Hydrochloric Acid — 1 : 1 (*v\v*).

D-2.2 Sulphuric Acid — concentrated.

D-2.3 Ascarite or Soda Asbestos

D-2.4 Magnesium Perchlorate — solid.

D-2.5 Pumice Impregnated with Copper Sulphate — anhydrous. Crush pumice to approximately 5 mm size, sift free from dust and transfer 60 g to a casserole. Cover with a concentrated solution of 30 g to a 35 g of copper sulphate, evaporate to dryness while constantly stirring and then heat for 3 to 4 hours at 150° to 160°C in an air-oven. Cool in a desiccator and preserve in a glass-stoppered bottle.

D-3 PROCEDURE

D-3.1 Transfer about 5 to 10 g of the accurately weighed sample to the flask A and cover it with water. Insert the stopper carrying the separating funnel B and a condenser C. Connect the latter with D, E and F. Pass air that is free from carbon dioxide through the system until it is judged that all carbon-dioxide has been removed. Close the stop-cock in the separating funnel and insert the weighed absorption bulbs G and H in the train; the latter acts as a guard tube. Half fill the separating funnel with dilute hydrochloric acid, replace the stopper carrying the air and see that there is free passage for gases through the train. Open the stop cock in the separating funnel and run acid into the flask slowly if there is much effervescence and rapidly if there is little. When effervescence diminishes in the former case, at once in the latter, start the flow of water in the condenser and heat the flask slowly so as to secure steady but quite ebullition. When it is judged that carbon dioxide has been boiled out of the solution, remove the source of flame, increase current of air and sweep out all carbon dioxide. Disconnect the weighed bulbs, close their inlet and outlet tubes and place them in the balance case. When cooled, open the stopper momentarily and weigh.

D-4 CALCULATION

$$\text{Carbon dioxide, percent by mass} \quad \frac{M_2 - M_1}{M_3} \times 100 \text{ percent}$$

where

M_1 = mass, in g, of the bulbs before the test;

M_2 = mass, in g, of the bulbs after the test; and

M_3 = mass, in g, of the sample taken for the test.

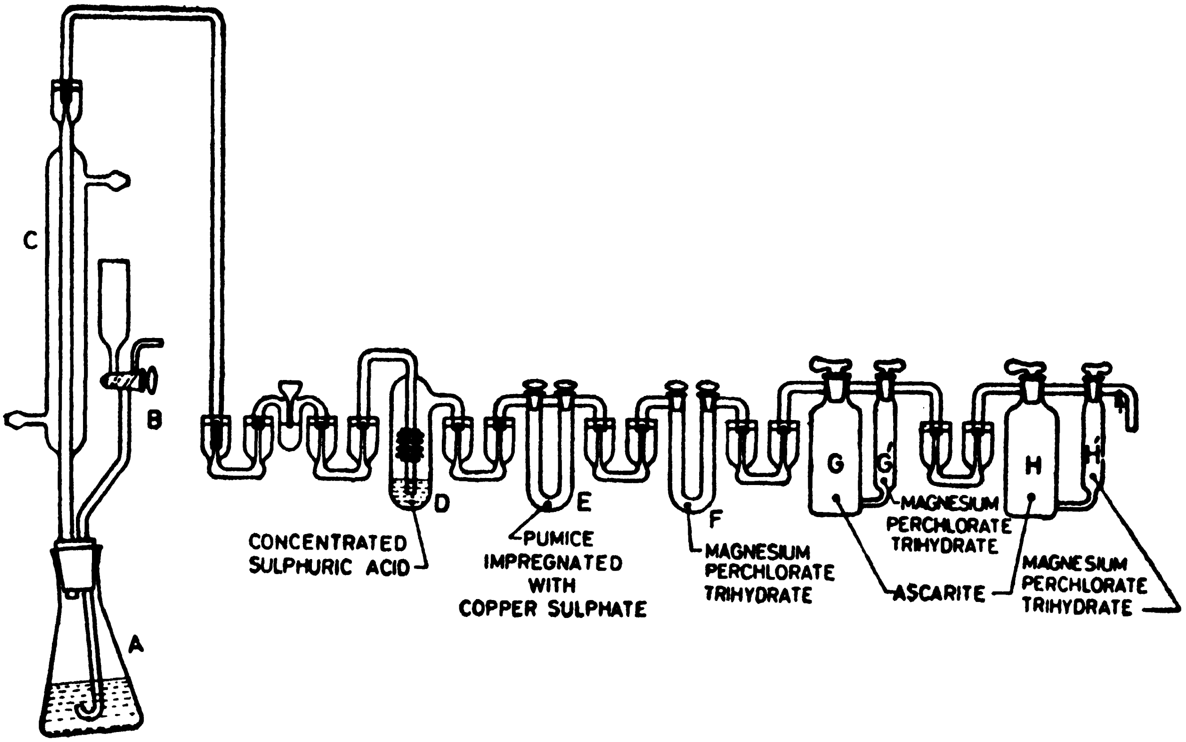

FIG. 1 ABSORPTION TRAIN FOR CARBON DIOXIDE

ANNEX E

[*Table* 1, *Sl No.* (xii)]

DETERMINATION OF CALCIUM COMPOUNDS

E-0 OUTLINE OF THE METHOD

The material is converted into calcium oxalate and dissolved in sulphuric acid. The oxalic acid liberated is titrated against standard potassium permanganate.

E-1 REAGENTS

E-1.1 Dilute Hydrochloric Acid — 1 : 1 (*v/v*).

E-1.2 Ammonium Hydroxide

E-1.3 Hydrogen Peroxide — 30 percent (*w\v*).

E-1.4 Ammonium Oxalate Solution — saturated.

E-1.5 Dilute Sulphuric Acid — 1 : 4 (*v\v*).

E-1.6 Standard Potassium Permanganate Solution – 0.1 N (*see* IS 2316 : 1976).

E-2 PROCEDURE

E-2.1 Weigh accurately about 2.5 g of the material, transfer to a 500-ml beaker and add about 100 ml of hydrochloric acid. Heat to boiling to bring into solution. Add ammonium hydroxide solution in slight excess and about 2 ml of hydrogen peroxide. Cool and transfer to a 500-ml volumetric flask and make up to mark. Mix thoroughly and filter. Pipette out 100 ml of the filtrate and transfer to a 400 ml beaker. Add a few drops of ammonium hydroxide solution, heat to boiling and add an excess of saturated ammonium oxalate solution. Continue boiling until the precipitate becomes granular. Keep aside for 30 minutes, filter and wash the precipitate with hot water till the washings are free from ammonium oxalate. Place a beaker under the filter, puncture the filter paper and wash the precipitate into the beaker with hot water. Pour warm sulphuric acid through the filter paper and wash for a few times. Ensure that all the precipitate is washed down into the beaker. Add about 25 ml of sulphuric acid, heat to about 60° to 70°C and titrate the hot solution with standard potassium permanganate.

E-2.2 Calculation

$$\text{Calcium compounds (as CaO), percent by mass} = \frac{2{\cdot}804 \times V \times N}{M} \times 5$$

where

V = volume, in ml, of standard potassium permanganate solution required for titration;

N = normality of standard potassium permanganate solution; and

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

ANNEX F

[*Table* 1, *Sl No.* (xiii)]

DETERMINATION OF MANGANESE CONTENT

F-0 OUTLINE OF THE METHOD

The material is dissolved in nitric acid and oxidized with sodium bismuthate to permanganate acid. The excess sodium bismuthate is removed by filtration and a measured excess of standard ferrous ammouium sulphuric solution added to reduce the permanganate to manganous salt. The excess ferrous iron is then determined by titration with standard potassium permanganate solution.

F-1 REAGENTS

F-1.1 Nitric Acid — L : 1 (*v/v*).

F-1.2 Sodium Bismuthate — solid.

F-1.3 Sulphurous Acid or Sodium Sulphite

F-1.4 Nitric Acid — 3 percent (*v/v*).

F-1.5 Standard Ferrous Ammouium Sulphate Solution — 0.03 N (*see* IS 2316 : 1976).

F-1.6 Standard Potassium Permanganate Solution — 0.03 N (*see* IS 2316 : 1976).

F-1.7 Phosphoric Acid

F-2 PROCEDURE

F-2.1 Weigh accurately about 0.25 g of the material and transfer it to a 250 ml conical flask. Add 50 ml of the nitric acid and dissolved, heating, if necessary. If any residue remains undissolved, filter using a sintered glass crucible of medium porosity. Boil the solution for 5 minutes, cool and add about 1 g sodium bismuthate and boil again for another 5 minutes. A pink colour or dark brown precipitate should result. If no colouration or precipitate results, add further 0.5 g of sodium bismuthate and boil for 5 minutes. Then, add drop by drop a concentrated solution of flreshly prepared sulphurous acid or sodium sulphite until the solution becomes clear. Boil to expel the oxides of nitrogen. Cool the solution. Add a further 0.5 g of sodium bismuthate, stir the contents well for 2 to 3 minutes. Add 50 ml of 3 percent nitric acid and filter through a suitable sintered glass crucible. Catch the filtrate in a 500 ml conical flask. Wash the residue well with 3 percent nitric acid till the washings are free from permanganic acid and catch the washings also in the conical flask containing the filtrate. Add standard ferrous ammonium sulphate solution from a burette until a slight excess indicated by the disappearance of pink colour, is present (*see* Note 1). Note the exact volume. Titrate immediately with standard potassium permanganate to the first appearance of faint pink colour (*see* Note 2). Addition of about 2 to 3 ml of phosphoric acid improves the end point.

NOTES

1 The standard ferrous ammonium sulphate solution be added as soon as the filtration is completed because the permanganic acid slowly decomposes.

2 The titration should be carried out immediately after the ferrous ammonium sulphate solution has been added as the ferrous ammonium solution reacts slowly with nitric acid.

F-2.2 Calculation

$$\text{Manganese (as MnO}_2\text{), percent by mass} = \frac{0{\cdot}010\,99 \times N \times V \times 100}{M}$$

where

V = volume, in ml, of standard potassium permanganate used in the titration;

N = normality of standard potassium permanganate solution; and

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

ANNEX G

[*Table* 1, *Sl No.* (xiv)]

TEST METHOD FOR LOSS ON IGNITION

G-1 PROCEDURE

Dry about 5 g of the sample at 105 ± 2°C for two hours. Transfer about 1 g of the dried pigment weighed accurately to the nearest mg (M_1) to a previously ignited and weighed, porcelain crucible, and ignite at 900 to 1 000°C for one hour. Cool and weigh to the nearest mg. Repeat till a constant mass is obtained. Calculate the loss of mass on ignition and let it be M_2.

G-2 CALCULATION

Calculate the percent of loss ignition, L, as follows:

$$\frac{M_2}{M_1} \times 100$$

where

M_1 = mass of the sample, in g; and
M_2 = loss of mass on ignition, in g.

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