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# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RUBBER COMPOUNDING INGREDIENTS, PART I

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**BUREAU OF INDIAN STANDARDS**
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# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RUBBER COMPOUNDING INGREDIENTS, PART I

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( *Continued on page* 2 )

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# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR RUBBER COMPOUNDING INGREDIENTS, PART I

## 0. FOREWORD

**0.1** This Indian Standard ( Part I ) was adopted by the Indian Standards Institution on 27 September 1973, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

**0.2** Part I of this standard includes the test methods for some of the common tests carried out to evaluate the quality of the rubber compounding ingredients, such as zinc oxide, calcium carbonate, barytes, red oxide of iron, whiting, etc. If necessary, other parts of this standard will be formulated later.

**0.3** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

## 1. SCOPE

**1.1** This standard ( Part I ) prescribes methods of sampling and test for rubber compounding ingredients. The following tests are covered:

a) Fineness,
b) Relative density,
c) *p*H,
d) Acidity,
e) Moisture,
f) Matter soluble in water,
g) Matter insoluble in acid,
h) Loss on ignition,
j) Manganese,
k) Copper,
m) Iron, and
n) Lead.

**1.2** Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

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*Rules for rounding off numerical values ( *revised* ).

## 2. QUALITY OF REAGENTS

**2.1** Unless otherwise specified, pure chemicals and distilled water ( *see* IS : 1070-1960* ) freshly boiled and cooled, shall be employed in tests.

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

## 3. DETERMINATION OF FINENESS

**3.0 Outline of the Method** — Measure the fineness of the material by sieving a known quantity of it through a sieve of specified mesh.

### 3.1 Apparatus

**3.1.1** *Gallie Porritt Sieve* — The apparatus ( Fig. 1 ) consists of a metal funnel terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, a wire gauze of the required size of 25 mm diameter is soldered. Water under pressure of 150 to 350 kN/m$^2$ ( approximately 1·5 to 3·5 kgf/cm$^2$ ) is supplied by a tube fitted with a nozzle designed to discharge a spreading jet throughout the wire gauze, and the tube is so arranged that the distance of the orifice in relation to the wire gauze may be adjusted. The tube is provided with a filter to ensure removal of any solid particles from the water. A second tube which is also provided with a filter is improvized to supply a gentle stream of water for wetting the powder and for supplying more water to the funnel during the progress of the test.

**3.1.1.1** The area of the wire gauze shall be sufficiently large to prevent undue loss of pressure head.

### 3.2 Procedure

**3.2.1** Weigh accurately about 50 ± 1 g of the material. Half fill the funnel with water from the subsidiary supply, then bring the central jet ( *see* Fig. 1 ) into operation. Transfer the weighed material to the metal funnel either dry or in the form of a slurry with a suitable wetting agent. Turn off the subsidiary supply and, if necessary, adjust the height of the central jet so that the level of water in the funnel commences to fall. When the level has fallen within 50 mm of the sieve turn on subsidiary supply until the level is within 50 mm from the top of the funnel and maintain there until the liquid appears clear and then for a further period of one minute. Turn off the subsidiary supply and allow the level to fall again. When the funnel is empty, turn off the main jet and allow to drain. Remove the cup and examine for any large soft agglomerates. If present, break up gently with the finger, replace the cup and wash with water from the subsidiary supply for 2 minutes. Transfer the grit on the sieve to a watch-glass, dry at 100°C and weigh. If the fineness at any other sieve size is also to be determined, the same can be done by employing the removable cup provided with the desired wire gauze at the bottom.

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*Specification for water, distilled quality ( *revised* ).

# AMENDMENT NO. 1 JULY 1990
# TO
# IS 7086 ( Part 1 ) : 1973 METHODS OF SAMPLING AND TEST FOR RUBBER COMPOUNDING INGREDIENTS, PART 1

( *Page* 4, *clause* 3.1.1 ) — Substitute the following for the existing clause:

'**3.1.1** *Gallie Porritt Sieve* — The apparatus ( Fig. 1 ) consists of a funnel made of copper terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, a wire gauze of the required size of 25 mm diameter is soldered. Water under pressure of 150 to 350 kN/$m^2$ ( approximately 1·5 to 3·5 kgf/$cm^2$ ) is supplied by a tube fitted with a nozzle designed to discharge a spreading jet throughout the wire gauze, and the tube is so arranged that the distance of the orifice in relation to the wire gauze may be adjusted. The tube should be made of brass or any other material which is not likely to corrode. The tube is provided with a filter to ensure removal of any solid particles from the water. A second tube which is also provided with a filter is improvized to supply a gentle stream of water for wetting the powder and for supplying more water to the funnel during the progress of the test.'

( *Page* 4, *clause* 3.2.1, *line* 15 ) – Substitute '$105 + 2$°C' *for* '100°C.'

( *Page* 6, *clause* 3.2.2 ) — Insert the following at the end:

'The wetting agent should be free from grit.'

( PCD 13 )

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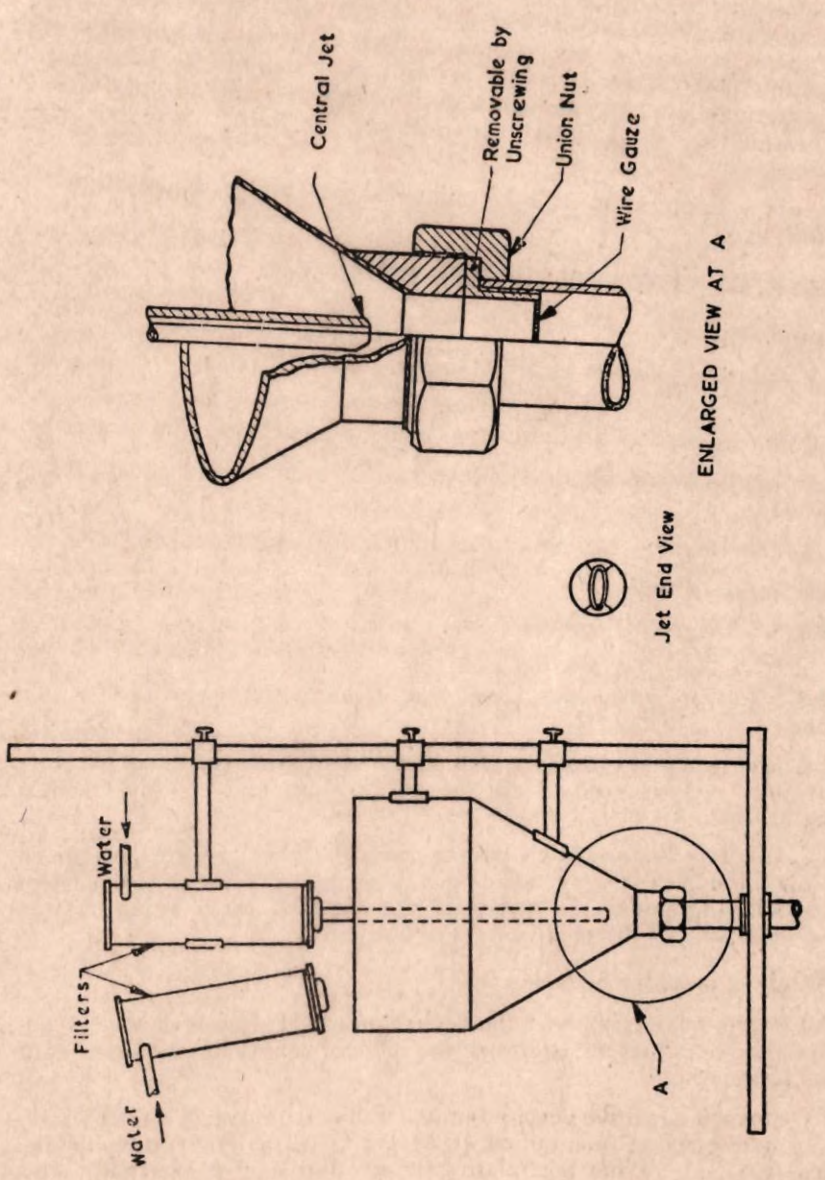

FIG. 1 GALLIE-PORRITT SIEVE

**3.2.2** Powders, which are very light, are difficult to be wetted at one time in large quantities, such as 50 g used in this test. To facilitate wetting of these powders, either a small amount of wetting agent may be added or the material may be added in small quantities into the apparatus. The powder may also be made into a slurry with water and wetting agent before transferring the weighed material into the funnel of the sieving apparatus.

**3.3 Result** — Express the mass of residue as percentage of the material taken for the test.

## 4. DETERMINATION OF RELATIVE DENSITY

### 4.1 Apparatus

**4.1.1** *Desiccator* — made of glass, constructed with heavy walls to withstand a vacuum of 3 mm of mercury.

**4.1.2** *Manometer* — of a suitable type to be used with vacuum oil pumps.

**4.1.3** *Relative Density Bottle* — with a capillary stopper and a cap, capacity 50 ml.

**4.1.4** *Thermometer* — having a range of 0 to 60°C graduated in 0·1°C.

**4.1.5** *Vacuum Pumps*

**4.1.5.1** *Oil pump* — provided with a motor to give a vacuum of at least 3 mm of mercury.

**4.1.5.2** *Water pump* — to expel the greater portion of air in the desiccator.

**4.1.6** *Water-Bath* — consisting of a vessel filled with sufficient water to permit only a very gradual rise in temperature and equipped with a stirring device.

**4.1.7** *Weighing Bottle* — fitted with a cork, the neck being small enough to fit inside the neck of the relative density bottle. This latter requirement is essential, since small quantities of the material easily adhere to the ground-glass joint of the relative density bottle.

### 4.2 Relative Density of Kerosine

**4.2.0** Before proceeding with the determination of relative density of the material, it is necessary to determine the relative density of the kerosine to be used in the test.

**4.2.1** Clean the relative density bottle. Fill it with freshly boiled water, place in a thermostat maintained at 27 ± 2°C till a temperature equilibrium is reached. Wipe the relative density bottle, dry externally and weigh as described in **4.3.2** and **4.3.3**. Empty the contents, clean and dry the bottle and weigh. Repeat the operation at 27 ± 2°C with kerosine to be used for the test and calculate as follows:

$$\text{Relative density of kerosine at } 27°\text{C}/27°\text{C} = \frac{\text{Mass of kerosine}}{\text{Mass of water}} = S$$

### 4.3 Procedure

**4.3.1** Dry about 5 g of the material at 105 ± 2°C for 2 hours in a weighing bottle. Cool to room temperature. Pour sufficient kerosine into the relative density bottle to form a 6-mm layer at the bottom of the relative density bottle and add about 4 g of the dried material from the previously weighed weighing bottle. Weigh the weighing bottle again. Stir the sample with a rounded and polished glass rod until completely covered by the kerosine adding more kerosine, if necessary. Wash the rod with kerosine. Place the relative density bottle in the desiccator, close and connect the desiccator to the water pump and evacuate most of the air from the system. ( This may take about 5 to 10 minutes. ) Close the system with a pinch-cock and connect the desiccator to the oil pump for the removal of small amount of air given off at the low pressures obtainable with the oil pump. Read the manometer to see that proper vacuum is maintained and when it becomes constant at 3 mm of mercury, cut off the oil pump for short periods, taking precautions to prevent leakage. It will be noticed that bubbles of air come out from the material very rapidly at first and that this action gradually decreases and finally stops altogether, which may require from half to two hours. When no more bubbles are seen, assume that all the occluded air has been given off and the material is thoroughly wet with kerosine. Admit air into the desiccator slowly by means of the pinch-cock.

**4.3.2** Take out the relative density bottle and fill it with kerosine, taking care to prevent the formation of air bubbles. Place the thermometer in the water-bath which is maintained at about 25°C. Carefully place the relative density bottle in the bath and permit it to come to constant temperature. Then insert the capillary stopper. Add warm water to the water-bath to raise the temperature quickly to 26°C in order to expand the kerosine and prevent it from keeping down the capillary and admitting a small quantity of air. Add more warm water and gradually raise the temperature of the water-bath to 27°C. Wipe the capillary stopper with filter paper and put the cap on. Remove the relative density bottle from the bath and dry. Allow it to stand for 30 minutes to enable it to come to room temperature and weigh.

**4.3.3** Allow the relative density bottle to stand approximately for the same length of time before each weighing so as to compensate for slight errors due to evaporation at the joints.

### 4.4 Calculation

$$\text{Relative density of the material at } 27°\text{C}/27°\text{C} = \frac{P \times S}{(P + K) - F}$$

where

$P$ = mass in g of the material used,
$S$ = relative density of the kerosine,
$K$ = mass in g of the bottle filled with kerosine only, and
$F$ = final mass in g of the bottle with the material and kerosine.

## 5. DETERMINATION OF *p*H

**5.1** Weigh 25·00 g of the material and transfer it completely to a 250-ml beaker. Add 100 ml of carbon dioxide-free distilled water and stir for 30 minutes. Allow the solid material to settle and then filter. Collect the filtrate and determine its *p*H by any suitable method. Use of a glass electrode system is preferable.

**5.1.1** In case of dispute, the method using glass electrode system shall be adopted.

## 6. DETERMINATION OF ACIDITY

### 6.1 Reagents

**6.1.1** *Standard Sodium Hydroxide Solution* — 0·01 N.

**6.1.2** *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit ( conforming to IS : 323-1959* ) and make up to 100 ml with water.

**6.2 Procedure** — Weigh accurately about 5 g of the material into a 400-ml beaker and boil with 200 ml of ammonia-free water for 5 minutes. Cool and make up to 250 ml in a volumetric flask. Filter through a double filter paper ( Whatman No. 40 or its equivalent ) and reject the first 100 ml of filtrate, collect the next 50 ml and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

### 6.3 Calculation

$$\text{Acidity as ( as } H_2SO_4 \text{ ), percent by mass} = \frac{24{\cdot}5\, V N}{M}$$

where

$V$ = volume in ml of standard sodium hydroxide solution,
$N$ = normality of standard sodium hydroxide solution, and
$M$ = mass in g of the material taken for the test.

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*Specification for rectified spirit ( *revised* ).

## 7. DETERMINATION OF MOISTURE CONTENT

**7.1 Procedure** — Weigh accurately about 5 g of the material in a previously weighed flat-bottom dish about 80 mm in diameter. Keep the dish with the material in an oven maintained at 105 ± 2°C for 2 hours. At the end of this period, transfer the dish to a desiccator and cool. Remove the dish and weigh. Repeat the heating and weighing till the mass is constant.

**7.2 Result** — Express the loss in mass as the percentage of the material taken for the test.

## 8. DETERMINATION OF MATTER SOLUBLE IN WATER

### 8.1 Reagent

**8.1.1** *Rectified Spirit* — conforming to IS : 323-1959*.

**8.2 Procedure** — Weigh accurately about 10 g of the material, previously dried at 105 ± 2°C to costant mass. Wet it thoroughly with 3 ml of rectified spirit and 200 ml of freshly boiled and cooled water. Boil for 5 minutes and cool to room temperature. Make up to 250 ml with water, shake and filter. Reject about 50 ml of the first portion of the filtrate and pipette out a 50-ml portion from the rest of the filtrate into a previously weighed porcelain dish and evaporate to dryness on a water-bath. Dry the residue so obtained to constant mass in an oven at 105 ± 2°C.

### 8.3 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{500\,m}{M}$$

where

$m$ = mass in g of the residue, and
$M$ = mass in g of the dried material taken for the test.

## 9. DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

### 9.1 Reagent

**9.1.1** *Dilute Hydrochloric Acid* — approximately 5 N.

**9.2 Procedure** — Weigh accurately about 2 g of the material, previously dried at 105 ± 2°C to constant mass into a 250-ml beaker, add 50 ml of dilute hydrochloric acid. Boil for 10 minutes, dilute to 100 ml and filter through a filter paper ( Whatman No. 40 or its equivalent ). Wash the filter paper with hot water until the filtrate is free from chlorides. Dry the filter paper and transfer it into a weighed crucible previously ignited.

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*Specification for rectified spirit ( *revised* ).

Heat the crucible on a burner and subsequently in a muffle furnace at 850 ± 25°C. Weigh the crucible and contents after cooling. Repeat heating, cooling and weighing until the mass is constant.

**9.3 Result** — Express the mass of the ignited residue as a percentage of the mass of the dried material taken for the test.

## 10. LOSS ON IGNITION

**10.1 Procedure** — Weigh accurately about 3 g of the material, previously dried at 105 ± 2°C to constant mass in a previously ignited porcelain crucible of 50 ml capacity and ignite at a temperature of 850 ± 25°C. Cool to about 100°C and then finally transfer the crucible with its contents to a desiccator till the temperature comes down to room temperature, and weigh. Repeat the process until constant mass is obtained.

**10.2 Result** — Express the loss on ignition as the loss in mass calculated as a percentage of the mass of the dried material taken for the test.

## 11. DETERMINATION OF MANGANESE

**11.0** Determine the content of manganese either by colorimetric comparison ( Method A ) or by volumetric estimation ( Method B ). In case of dispute, Method B shall be used as the referee method.

### 11.1 Method A

**11.1.1** *Apparatus*

**11.1.1.1** *Nessler cylinders* — *see* IS : 4161-1967*.

**11.1.2** *Reagents*

**11.1.2.1** *Sodium carbonate* — anhydrous, free from manganese.

**11.1.2.2** *Concentrated sulphuric acid* — conforming to IS : 266-1961†.

**11.1.2.3** *Concentrated nitric acid* — conforming to IS : 264-1968‡.

**11.1.2.4** *Phosphoric acid* — approximately 85 percent by mass.

**11.1.2.5** *Potassium periodate* — solid.

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

$$A = \frac{0{\cdot}551}{N}$$

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*Specification for Nessler cylinders.

†Specification for sulphuric acid ( *revised* ).

‡Specification for nitric acid ( *first revision* ).

where

$A$ = volume in ml of standard potassium permanganate solution to be made up to a volume of 50 ml, and

$N$ = normality of the standard potassium permanganate solution.

Do not keep the solution for more than one week, in a dark bottle. One millilitre of this solution is equivalent to 0·1 mg of manganese ( as Mn ).

**11.1.3** *Procedure*

**11.1.3.1** *Prepared sample solution* — Weigh accurately about 1·0 g of the material, previously dried at 105 ± 2°C to constant mass and mix it with 4 g of anhydrous sodium carbonate free from manganese and fuse in a platinum crucible until the melt is clear. Pour the hot molten mass on a platinum dish and rotate so as to form a thin film and then place the crucible in the dish with the fused mass. Add about 10 ml of water and heat on a water-bath until the product is disintegrated. Remove the crucible and wash. Evaporate to dryness. Add a mixture of 6 ml of concentrated sulphuric acid and 1 ml of concentrated nitric acid. Evaporate the fumes of sulphur trioxide ( $SO_3$ ). Dilute to about 200 ml and filter through a dry filter paper to remove silica. Collect the filtrate to make up to 250 ml in a volumetric flask. Call this as *prepared sample solution.*

**11.1.3.2** Transfer 125 ml of the *prepared sample solution* to a 250-ml beaker and evaporate to 75 ml. If the manganese content of the solution is very low, a separate sample should be digested as described under **11.1.3.1** for manganese determination and the whole quantity should be used. Add 10 ml of phosphoric acid and 0·5 g potassium periodate into the solution and bring it to boil. Cool slightly. Add again 0·1 g potassium periodate and boil, when the colour appears to have developed to the maximum, place the beaker on a water-bath and keep for 15 minutes. If there is any doubt about the completeness of the reaction, more potassium periodate should be added. After the sample has stood on the water-bath for 15 minutes, remove the beaker and cool. If the colour is too deep for a good comparison, transfer the solution to a 250-ml flask, dilute to a definite volume and use an aliquot for comparison. Place a suitable aliquot ( or the whole solution ) in a Nessler cylinder and dilute to the mark with water. Place a similar aliquot of a blank solution carried through the entire analysis in the same manner as the *prepared sample solution*, in the other Nessler cylinder and dilute almost to the mark. Titrate this solution with standard potassium permanganate solution from a 10-ml burette until its colour matches that of the *prepared sample solution* when diluted to the same volume.

**11.1.4** *Calculation*

$$\text{Manganese, percent by mass} = \frac{0{\cdot}02\,A}{M}$$

where

$A$ = volume in ml of standard potassium permanganate solution used for 125 ml of the *prepared sample solution*, and

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

### 11.2 Method B

**11.2.1** *Reagents*

**11.2.1.1** *Concentrated sulphuric acid* — relative density 1·84 ( conforming to IS : 266-1961* ).

**11.2.1.2** *Concentrated nitric acid* — relative density 1·42 ( conforming to IS : 264-1968† ).

**11.2.1.3** *Sodium bismuthate* — 75 percent, *Min.*

**11.2.1.4** *Standard ferrous ammonium sulphate solution* — 0·02 N.

**11.2.1.5** *Standard ceric sulphate solution* — 0·02 N.

**11.2.1.6** *Oxidation-reduction type indicator* — 1 : 10 *o*-phenanthroline ferrous complex solution.

**11.2.2** *Procedure* — Weigh 5 g of the material previously dried at 105 ± 2°C to constant mass into a 600-ml beaker, mix into a paste with 10 to 20 ml of water, and add slowly 10 ml concentrated sulphuric acid. Cover with a watch-glass, maintain at boiling point for 15 minutes. Cool and dilute to 150 ml. Filter through a prepared asbestos pad, wash the filter with 50 ml of water, and transfer the filtrate and washings to the original beaker. After boiling for 5 minutes with 10 ml of concentrated nitric acid, remove the beaker momentarily from the burner and cautiously add sodium bismuthate in small quantities at a time until pink colour persists after 4 to 5 minutes of boiling. Reduce the permanganate with a slight excess of ferrous ammonium sulphate solution. Boil for about 30 seconds and remove from the burner. Add about 1 g of sodium bismuthate, stir until effervescence subsides and cool to 15 to 20°C. Filter through a prepared asbestos pad into 10 ml of standard ferrous ammonium sulphate solution. Wash with cold water, dilute the filtrate and washings to 200 to 250 ml and titrate immediately with standard ceric sulphate solution using 2 drops of the indicator. The colour change at the second end point is red to pale blue.

Carry out a blank determination by titrating 10 ml of the standard ferrous ammonium sulphate solution diluted to 200 to 250 ml with standard ceric sulphate solution.

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*Specification for sulphuric acid ( *revised* ).

†Specification for nitric acid ( *first revision* ).

**11.2.3** *Calculation* — Express the results on the basis that 1 ml of normal ceric sulphate solution is equivalent to 0 010 98 g of manganese ( as Mn ) and calculate the percentage of manganese using the following formula:

$$\text{Manganese ( as Mn ), percent by mass} = \frac{1\cdot10\ (V_1 - V_2)\ N}{M}$$

where

$V_1$ = volume in ml of standard ceric sulphate solution required in the blank determination,

$V_2$ = volume in ml of standard ceric sulphate solution required in the test with the material,

$N$ = normality of standard ceric sulphate solution, and

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

## 12. TEST FOR COPPER

### 12.1 Apparatus

**12.1.1** *Nessler Cylinders* — *See* IS : 4161 - 1967*.

### 12.2 Reagents

**12.2.1** *Ammonium Hydroxide Solution* — r. d. 0·92.

**12.2.2** *Sodium Diethyldithiocarbamate Solution* — Dissolve 1·0 g of sodium diethyldithiocarbamate in 1 000 ml of copper-free water. Keep in an amber-coloured bottle and protect from strong light.

**12.2.3** *Gum Arabic* — Prepare a 5 percent solution of gum arabic in water.

**12.2.4** *Stock Copper Solution* — Dissolve exactly 0·392 8 g of copper sulphate ( $CuSO_4 \cdot 5H_2O$ ) in water and make up the volume to one litre.

**12.2.5** *Standard Copper Solution* — Prepare a fresh solution by diluting 10 ml of the stock solution ( **12.2.4** ) to one litre with water and adjust the *p*H between 5·5 and 6. Each millilitre of this solution contains 0·000 001 g of copper ( as Cu ).

**12.2.6** *Ferric Chloride Solution*

**12.3 Procedure** — Pipette 25 ml of the *prepared sample solution* ( **11.1.3.1** ) into a small conical flask or beaker. Drop into it a small piece of litmus paper and make the solution just alkaline with ammonium hydroxide solution. Add 2·5 ml of ammonium hydroxide solution in excess and heat to boiling. Allow to stand on a water-bath for one hour to ensure complete precipitation of aluminium hydroxide and then filter through filter

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*Specification for Nessler cylinders.

paper ( Whatman No. 1 or its equivalent ) into a Nessler cylinder washing the filter paper with two or three small portions of hot water. To the solution in the Nessler cylinder add 1 ml of gum arabic solution, add 10 ml of ammonium hydroxide solution and 10 ml of sodium diethyldithiocarbamate solution in the order named. Dilute to 100-ml mark and mix well.

**12.3.1** In case the *prepared sample solution* is turbid due to the interfering substances like iron, lead or bismuth, the following modified procedure is recommended.

**12.3.1.1** Add a few drops of ferric chloride solution and then a slight excess of ammonium hydroxide solution, filter and wash. Lead and bismuth, if present, will coprecipitate with ferric hydroxide. Dissolve the ferric hydroxide precipitate in dilute nitric acid, precipitate with ammonium hydroxide solution filter and wash. Combine the two filtrates and dilute to a known volume. Follow rest of the procedure as under **12.3.**

**12.3.2** Carry out a control test using 2 ml of the standard copper solution.

**12.3.3** The material shall be considered not to have exceeded the limit prescribed if the intensity of the colour produced with the material is not deeper than that produced in the control test.

## 13. TEST FOR THE PRESENCE OF IRON

### 13.1 Apparatus

**13.1.1** *Nessler Cylinders* — *See* IS : 4161-1967*.

### 13.2 Reagent

**13.2.1** *Thiocyanate Indicator Solution* — 2 percent ( *m/v* ).

**13.3 Procedure** — Weigh 1·000 g of the material and bring it into solution as described under **11.1.3.1**. Transfer, without filtering, to a 200-ml volumetric flask, make up to the mark and allow to settle. Filter and reject the first few millilitres of the filtrate and transfer some of the filtrate into Nessler cylinder. Add 10 ml of thiocyanate indicator and dilute to 100 ml with water. Mix thoroughly. In the other Nessler cylinder prepare a blank solution carried through the entire analysis in the same manner as done with the material and make up the volume to 100-ml mark.

**13.3.1** The material shall be regarded to have satisfied the requirement if the colour of the test solution is not deeper than that obtained in the blank.

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*Specification for Nessler cylinders.

## 14. DETERMINATION OF LEAD

### 14.1 Reagents

**14.1.1** *Hydrochloric Acid* — approximately 4 N.

**14.1.2** *Ammonium Acetate Solution* — approximately 2 N.

**14.1.3** *Congo Paper*

**14.1.4** *Potassium Dichromate* — approximately 5 percent ( *m/v* ) solution.

**14.1.5** *Acetic Acid* — 2 percent ( *m/v* ) solution ( *see* IS : 695-1967* ).

**14.2 Procedure** — Weigh accurately about 25·0 g of the material, previously dried at 105 ± 2°C to constant mass, transfer to a 300-ml beaker, and dissolve by heating in 35 ml of hydrochloric acid. Add to this solution ammonium acetate solution until the liquid gives no acid reaction on Congo paper. Filter off any insoluble matter present, and wash thoroughly with the ammonium acetate solution. Dilute the filtrate, combined with the washings, to about 200 ml with water, and then heat to boiling. Precipitate the lead from the boiling solution as lead chromate, by adding an excess of potassium dichromate solution. Keep the liquid boiling until the precipitate has turned to dark orange-red. Then keep the liquid hot ( on a water-bath ) for 1½ to 2 hours. After cooling the liquid, filter the precipitate through a weighed sintered glass crucible, wash with acetic acid, then with water. Dry in an oven at a temperature of 105 ± 2°C or in a vacuum desiccator to constant mass.

### 14.3 Calculation

**14.3.1** One milligram of lead chromate is equivalent to 0·637 mg of lead.

**14.3.2** Express the lead content as a percentage of the originally weighed amount of the material as follows:

$$\text{Percentage of lead ( as Pb )} = \frac{63{\cdot}7\, M_1}{M_2}$$

where

$M_1$ = mass of lead chromate, and

$M_2$ = mass of the material taken for the test.

## 15. SAMPLING

### 15.1 General Requirements of Sampling

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

**15.1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

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*Specification for acetic acid (*first revision*).

**15.1.2** The sampling instrument shall be clean and dry when used.

**15.1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the container for samples, from adventitious contamination.

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

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

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

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

**15.1.8** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

### 15.2 Scale of Sampling

**15.2.1** *Lot* — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**15.2.1.1** For ascertaining the conformity of the lot to the requirements of specification, tests shall be carried out for each lot separately.

**15.2.2** The number ( $n$ ) of containers to be selected from the lot shall depend upon the size ( $N$ ) of the lot and shall be in accordance with Table 1.

TABLE 1 NUMBER OF CONTAINERS TO BE SELECTED

| LOT SIZE<br>$N$ | NUMBER OF CONTAINERS TO BE SELECTED<br>$n$ |
|---|---|
| (1) | (2) |
| Up to 100 | 5 |
| 101 to 300 | 6 |
| 301 „ 500 | 7 |
| 501 „ 800 | 8 |
| 801 „ 1 300 | 9 |
| 1 301 and above | 10 |

**15.2.3** The sample containers shall be selected at random. In order to ensure the randomness of selection, the procedures given in IS : 4905-1968* may be followed.

### 15.3 Test Samples and Referee Samples

**15.3.1** Draw with an appropriate sampling instrument small portions of the material from different parts of the container selected from the lot ( *see* Table 1 ). The total quantity of the material so drawn should be sufficient to conduct the tests for all the requirements but shall not exceed 300 g.

**15.3.2** Mix thoroughly the portions drawn from the same container to form an individual sample representative of the container. In addition mix together equal portions from the individual samples to form the composite sample representative of the lot.

**15.3.3** Divide all the individual samples ( **15.3.1** ) and the composite sample ( *see* **15.3.2** ) into three equal parts forming three sets of test samples. Transfer these test samples immediately to thoroughly dried sample containers which shall then be sealed air-tight. Label them with all the particulars of sampling given under **15.1.7**. Send one of these sets to the purchaser and one to the supplier.

**15.3.3.1** *Referee samples* — The third set of test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee samples to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place as agreed to between the purchaser and the supplier.

**15.4 Number of Tests** — Tests for important characteristics to be specified in the relevant specifications shall be made on each of the individual sample while tests for all other characteristics shall be made on the composite sample.

**15.5 Criteria for Conformity** — Criteria for ascertaining the conformity of the lot to the requirements of specification shall be given in individual specifications.

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*Methods for random sampling.