

PRIMARY PLASTICIZERS

- 1.1 **APPEARANCE:** Clear liquid free from contamination on visual inspection.

DETERMINATION OF COLOUR

1.2

- 1.2.1 **Outline of the Method :** The colour of the sample is compared with that of permanent colour standards and expressed in terms of Hazen colour units.

The Hazen colour unit is defined as the colour of an aqueous solution containing one part per million of platinum in the form of the chloroplatinic acid and 2 parts per million of cobalt in the form of Cobaltous Chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

- 1.2.2 **Procedure:**

Pour into the colorimetric tube/ beaker a quantity of the sample sufficient to fill it up to the graduation mark. Similarly, pour the colour standard selected into the other tube/ beaker to the graduation mark. Compare the colour of the sample with that of the standard by looking down the tubes/ beaker against a white background, taking care to avoid side illumination. Where matching proves impossible give a description of the colour of the sample.

- 1.2.3 **Expression of Result:** Report the result to the nearest 10 Hazen colour units

- 1.2.4 **Preparation of Colour Standards:**

- 1.2.4.1 **Apparatus/Equipments:**

- 1.2.4.2 **50 ML Burette**

- 1.2.4.3 **Reagents:** - Cobaltous Chloride Hexahydrate AR Grade.
- Hydrochloric Acid A.R. Grade

- **Chloroplatinic Acid:** Dissolve 1.000 g of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a water-bath. When the metal had dissolved, evaporate the solution to dryness. Add 4 ml of hydrochloric acid and again evaporate to dryness.

- **Potassium Chloroplatinate (K_2PtCl_6):** 1.25 g of the salt containing 0.5 g of platinum.

- 1.2.4.4 **Procedure:**

Dissolve 2.0 g of Cobaltous chloride hexahydrate and either the chloroplatinic acid or 2.490 g of potassium chloroplatinate in 200 ml. of conc. hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into 2000 ml volumetric flask. Dilute with water up to the graduation mark.

From this solution, prepare a series of permanent colour standards ranging from zero Hazen colour unit upwards, at intervals of 10 units. For each 10 units, pipette 5.0 ml of the solution into a 250 ml volumetric flask and dilute with water up to the graduation mark.. Transfer each standard solution to a storage bottle. Check these standards at intervals against freshly prepared standards.

DETERMINATION OF COLOUR STABILITY

1.3.1 **Outline of the Method:** The colour of the material is measured after a specified heat treatment.

1.3.2 **Apparatus/Equipments:**

- Oven
- Beaker 100 ml.

1.3.3 **Procedure:**

Take about 70 ml of the material in a 100 ml. beaker. Place the beaker in an oven maintained at $180 \pm 2^{\circ}\text{C}$ for two hours. Remove the beaker from the oven and allow it to cool, in air to a room temperature. Determine the colour of the heat treated sample by the method as prescribed for determination of colour. Report the result in Hazen colour units.

DETERMINATION OF SPECIFIC GRAVITY

1.4.1 **Apparatus/Equipments:**

- Hydrometer of desired range.
- Measuring Cylinder 100 ml.
- Constant Temperature Water Bath
- Thermometer

1.4.2 **Procedure:**

Pour the liquid into a clean and dry 100 ml measuring cylinder.

Allow the entrapped air bubbles to escape out of the viscous liquid, In case of excessive air bubbles, the material should be preheated to remove all air bubbles. Keep the measuring cylinder containing liquid in the water bath maintained at specified temperature till it attains the temperature of the bath. Lower the hydrometer gently into the liquid & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material and note down the reading.

In case of critical supplies the specific gravity should be determined by specific gravity bottle. The test procedure employed would be as per IS 3672-1966.

DETERMINATION OF REFRACTIVE INDEX

1.5.1 **Outline of the Method:** The refractive index is determined by Abbes Refractometer at a temperature mentioned in specification using diffused daylight or any convenient light as an illuminate.

1.5.2 **Apparatus/Equipments:**

Refractometer: An Abbes Refractometer with the scale graduated directly in terms of refractive index of the D-line of sodium at a temperature mentioned in specification. Water supply is used for maintaining this temperature.

1.5.3 **Procedure:**

Place the Refractometer in front of a suitable source of light (either day light or electric light). Adjust the temperature of the Refractometer to a temperature mentioned in specification by the circulation of water. Clean the prisms with alcohol and clamp it dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eyepiece on the cross wire and the reading lens of the scale by moving the prism-arm and find a position where the lower part of the field is dark and the upper part is bright. In general, the border line, which is coloured, shall be corrected by turning the mill head on the right of the telescope until a sharp black and white edge is obtained.

Move the prism-arm until this black edge just crosses the intersection of the cross hair. Read the refractive index on the scale to the fourth decimal place.

DETERMINATION OF WATER CONTENT

- Moisture is determined by the Karl Fischer method.
- Take about 5 g of the material, weigh accurately and determine the water content as per **IS-2362-1973**.

OR

- By Oven method

1.6.1 **Apparatus/Equipments:**

- Oven maintained at a temperature of 105⁰C
- Electronic Balance
- Petri dish

1.6.2 **Procedure:**

Place a beaker in the oven for half an hour and allow it to cool in a desiccator, when it acquire the room temperature, weigh it accurately. Put sample in the beaker and weigh it again. Place the beaker, alongwith the sample in the oven (maintained the temperature at 105⁰C) for one hour. Remove the sample from the oven and put it in the desiccator for cooling. Now find out the weight of the beaker containing the sample.

1.6.3 **Calculation:**

$$\% \text{ age of Moisture} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

w₁ = Weight of the Beaker

w₂ = Weight of the sample + Beaker

w₃ = Weight of the sample (After heating) + Beaker

DETERMINATION OF ASH CONTENT

- 1.7.1 **Apparatus/Equipments:**
- Silica/ Platinum crucible
 - Hot plate
 - Furnace

1.7.2 **Procedure:**

Slowly burn approximately 50 g, of the material in several portions in a weighed platinum or silica crucible and ignite finally in a furnace at $600 \pm 30^{\circ} \text{C}$ until all the carbonaceous matter has disappeared. Cool in a desiccator and weigh.

1.7.3 **Calculation** : Ash content, percent by weight $= \frac{W_1}{W_2} \times 100$

where, W_1 = weight in g of the residue, and
 W_2 = weight in g of the material taken for the test.

DETERMINATION OF ACIDITY BEFORE HEATING

- 1.8.1 **Outline of the Method:** Acidity is determined by titration with standard solution of sodium hydroxide and phenolphthalein as an indicator.

- 1.8.2 **Reagents:**
- Rectified Spirit
 - Standard Sodium Hydroxide Solution - 0.1 N.
 - **Phenolphthalein Indicator:** - Dissolve 0.1 g of Phenolphthalein Indicator in 100 ml of 60 percent rectified spirit and make faintly pink by the addition of dilute sodium hydroxide solution.

1.8.3 **Procedure:**

Take 50 ml of rectified spirit, add 0.5 ml of phenolphthalein indicator and neutralise with standard sodium hydroxide solution then add to it 25 g of the sample and titrate the mixture against standard sodium hydroxide solution till the pink colour persists for at least 5 seconds.

1.8.4 **Calculation** : Acidity, percent by weight $= \frac{V \times N \times E}{W \times 10}$

V = volume in ml of sodium hydroxide solution used, and

W = weight in g of the material taken for the test.

N = Normality of N/10 NaOH

E = Equivalent weight.

| | | |
|---------------------------|---------------------|------|
| Use equivalent weight for | - Phthalic acid | = 83 |
| | - Adipic acid | = 73 |
| | - Tri Mellitic acid | = 70 |

DETERMINATION OF ACIDITY AFTER HEAT TREATMENT

1.9.1 **Outline of the Method:** The increase acidity is measured after specified heat treatment by titration against standard solution of sodium hydroxide and phenolphthalein as an indicator.

1.9.2 **Procedure:**

Take out the sample after heat treatment (No. 1.3) and determine its increased acidity by the method prescribed in No. 1.8.

DETERMINATION OF ESTER CONTENT:

1.10.1 **Outline of the Method:** The ester content is saponified by potassium hydroxide in ethanol solution and titrated against standard hydrochloric acid.

1.10.2 **Reagents:**

Potassium Hydroxide Solution - approximately 1 N solution in rectified spirit

Standard Hydrochloric Acid Solution - 1 N.

Phenolphthalein Indicator: Dissolve 0.1 g of phenolphthalein in 60 percent rectified spirit and make faintly pink by addition of dilute sodium hydroxide solution.

1.10.3 **Procedure:**

Weigh accurately about 5.0 g of the material in a 500-ml round bottom flask. Add accurately with a pipette 50 ml alcoholic of potassium hydroxide solution and 5 ml of DM water. Attach the flask to water cooled reflex condenser and heat for one hour. Carry out a blank test simultaneously. After cooling, wash down the inside of each condenser twice with 20 ml of DM water. Disconnect the flask and wash each joint with further 20 ml of water. Add 0.5 ml of phenolphthalein indicator and titrate the mixture immediately against standard hydrochloric acid solution until the pink colour is discharge.

1.10.4 **Calculation** : $\text{Ester content, percent by weight} = \frac{N \times (V_2 - V_1) \times E}{W \times 10}$

V_2 = Volume in ml of standard hydrochloric acid required for blank,

V_1 = Volume in ml of standard hydrochloric acid required for test sample,

W = Weight in g of the material taken for test and

N = Normality of HCl

E = Equivalent weight.

| | | |
|---------------------------|----------|----------|
| Use equivalent weight for | DOP/DIOP | = 195.3. |
| | DIDP | = 223 |
| | DBP | = 139.2 |
| | DOA | = 186 |
| | TOTM | = 182 |
| | DINP | = 209 |

DETERMINATION OF SPECIFIC CONDUCTANCE OF

PHTHALATE PLASTICIZERS

1.11.1 Principle:

The conductivity of 40 % (by wt) solution of DOP in low conducting AR or redistilled iso propanol is determination at 30⁰C and the ratio of conductivity of solution to the solvent is expressed as specific conductivity/ Conductance.

- 1.11.2 Apparatus/Equipments:
- Conductivity bridge/ Conductivity Meter
 - Conductivity cell (cell constant about 1)
 - 100 ml beaker
 - Stiring rod
 - Electronic balance
 - Thermometer.

- 1.11.3 Reagents: - Isopropoanol AR Grade.

1.11.4 Procedure:

Weight 10 gm of DOP in a dry and clean 100 ml beaker and add gradually 25 gm of isopropyl alcohol. Stir with glass rod to obtain homogeneous solution. Read the temperature of the solution, bring the solution to 30⁰C by keeping it in a constant temperature bath. Now read the conductivity/ resistivity of the solution and pure solvent simultaneously by conductivity bridge using same conductivity cell.

1.11.5 Calculations:

Specific conductance of DOP at 30⁰C =

$$\frac{\text{Conductivity of solution at } 30^0\text{C}}{\text{Conductivity of Solvent at } 30^0\text{C}} = \frac{\text{Resistivity of Solvent at } 30^0\text{C}}{\text{Resistivity of Solution at } 30^0\text{C}}$$

SECONDARY PLASTICISERS

2.1 **APPEARANCE:** Clear viscous liquid free from contamination on visual inspection.

89H9FA-B5HCB'C: '7C@CI F.'

Compare the colour of fresh supplies with the colour of the standard/approved sample.

DETERMINATION OF SPECIFIC GRAVITY AT 27⁰C:

2.3.1 **Apparatus/Equipments:**

- Hydrometer of desired range
- Measuring Cylinder - 100 ml
- Constant Temperature Water Bath

2.3.2 **Procedure:**

- Thermometer (0 - 50⁰C/100⁰C).

Pour the chlorinated paraffin into a clean and dry 100 ml measuring cylinder.

Allow the enterpreted air bubble to escape out of the viscous liquid, in case of excessive air bubbles the materiel should be preheated to remove all air bubbles. Keep the Measuring Cylinder containing Chlorinated paraffin wax in a constant temperature bath maintained at 27⁰C, till it attains the temperature of the bath. Lower the hydrometer gently in to the chlorinated paraffin & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material & note down the reading.

In case of critical supplies the specific gravity should be determined by specific gravity bottle. The test procedure employed would be as per IS 3672.

DETERMINATION OF REFRACTIVE INDEX:

2.4.1 Outline of the Method: The refractive index is determined by Abbe's Refractometer at 27⁰C using diffused daylight or any convenient light as an illuminate.

2.4.2 Apparatus/Equipments:

Refractometer: An Abbe's Refractometer with the scale graduated directly in terms of refractive index of the D-line of sodium at a temperature of 27⁰ C. Water supply is used for maintaining this temperature.

2.4.3 Procedure:

Place the refractometer in front of a suitable source of light (either day light or electric light). Adjust the temperature of the refractometer to $27 \pm 0.1^{\circ}$ C by the circulation of water. Clean the prisms with alcohol and clamp it dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eyepiece on the cross wire and the reading lens of the scale by moving the prism-arm and find a position where the lower part of the field is dark and the upper part is bright. In general, the border line, which is coloured, shall be corrected by turning the mill head on the right of the telescope until a sharp black and white edge is obtained.

Move the prism-arm until this black edge just crosses the intersection of the cross hair. Read the refractive index on the scale to the fourth decimal place.

DETERMINATION OF VISCOSITY:

2.5.1 Apparatus/Equipments:

- Brook Field Viscometer
- 500 ml. beaker
- Water bath

2.5.2 Procedure:

Pour the sample in 500 ml beaker and condition it 27⁰C temperature till the temp. of the sample maintained 27⁰C . Now put the spindle of pre-calibrated viscometer into a sample. Set the instrument at particular r.p.m. and cross check the value at different r.p.m. Note the reading directly in centipoise (CP) at 27⁰C.

DETERMINATION RATE OF VOLATILE LOSS:

- 2.6.1 Apparatus/Equipments:**
- Oven maintained at $100 \pm 1^{\circ}\text{C}$
 - Petri dish of 8.0 ± 0.5 cm internal dia
 - Venire Calliper
 - Electronic Balance
 - Desiccator

2.6.2 Procedure:

With the help of vernier Calliper the internal diameter of petri dish should be determined. It should be 8.0 ± 0.5 cm. Fill the petri dish with 25 g. of chlorinated paraffin and put it in the oven for 15 minutes, in order to expel out bubble enclosed under the liquid. Cool the petri dish to room temperature in a desiccator. Weigh the petri dish and contents accurately. Place the petri dish with the contents in the oven at $100 \pm 1^{\circ}\text{C}$ for one hour., After one hour, remove the sample from the oven and cool it in the desiccator, weigh again and find the difference in weight.

2.6.3 Calculation:

$$\text{Rate of volatile loss gm/cm}^2/\text{sec.} = \frac{m_1 - m_2}{\pi r^2 t}$$

where

| | | |
|-------|---|--|
| m_1 | = | Mass of the petri dish + CPW |
| m_2 | = | Mass of the petri dish and CPW after t second exposes to oven temperature. |
| t | = | Time in second |
| r | = | Radius of petri dish |

DETERMINATION OF COLOUR STABILITY:

- 2.7.1 Apparatus/Equipments:**
- Oil bath maintained at $180^{\circ}\text{C} \pm 1^{\circ}\text{C}$ or air circulating oven kept at the same temperature.
 - Test tube 50 ml. capacity or 100 ml. beaker
 - Stop watch

2.7.2 Procedure:

Pour about 30 ml of chlorinated Paraffin wax in the test tube or beaker as the case may be, and place the test tube/beaker with contents in the oil bath or oven maintained at $180^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Remove the test tube/ beaker at the end of 20 minutes.

- 2.7.3 Conformity:** Material stands approved, if the colour change is within agreed/ approved range.

DETERMINATION OF CHLORINE CONTENTS:

2.8.1 Principle: The chlorine associated with chlorinated Paraffin wax reacts quantitatively with sodium metal to form Sodium-chloride. The chloride so formed should be estimated by volhard's method for estimation of chlorides.

2.8.2 Apparatus/Equipments:

- Weighing bottle 10 ml capacity
- Measuring cylinder 100 ml capacity
- Round bottom flask 500 ml
- Simple condenser (effective length) with standard joint so as to fit on the round bottom flask.
- Heating mantle
- S.S. Knife/Scissor.
- Conical 250 ml.

2.8.3 Reagents:

- Benzene AR Grade
- Isopropanol AR Grade
- Silver Nitrate solution (N/10)
- Sodium Metal AR Grade
- Nitro benzene AR Grade
- Nitric Acid AR Grade
- Phenol phthalien indicator.

2.8.4 Procedure:

Weigh .1 to .15 g of CPW sample into clean & dry 500 ml round bottom flask. Dissolve the material in 10 ml benzene, shake well to ensure complete dissolutions, add to it 50 ml Isopropanol and few boiling chips and 3-4 g of freshly cut sodium metal. Now fit the condenser on the round bottom flask vertically, circulate tap water slowly through the condenser. Allow the contents of the round bottom flask to reflux for 2 hrs on heating mantle.

After two hrs heating, allow the RB flask along with contents to cool. Now add 50 ml. Isopropanol solution in distilled water gradually and carefully through the top of the condenser to react any free sodium metal. Allow the contents to cool. Add few drops of phenolphthalein solution to the contents of RB flask & neutralised with conc. HNO_3 . Now acidify the contents with 1 : 1 HNO_3 gradually by frequent cooling.

Add 30 ml of standardised AgNO_3 solution (N/10) should be added. The excess of AgNO_3 is to be estimated by titrating against (N/10) ammonium thiocyanate solution using few drops of nitrobenzene and ferric alum as an indicator to sharp brick red end point and run a blank simultaneously. For blank determination 30 ml silver nitrate solution (N/10) duly acidified with nitric acid and titrated with ammonium thiocyanate using nitro benzene and ferric alum as an indicator.

2.8.5 Calculation: $\% \text{ of Chlorine contents in CPW} = \frac{3.55 \times (V_1 - V_2) \times N}{M}$

V_1 = Volume of ammonium thiocyanate solution used for blank.

V_2 = Volume of ammonium thiocyanate solution used for sample solution.

N = Normality of N/10 ammonium thiocyanate solution.

M = Weight of sample.

EPOXY PLASTICIZERS

- 3.1 **APPEARANCE:** Clear, light yellow, oily liquid free from contamination on visual inspection.

DETERMINATION OF SPECIFIC GRAVITY AT 30⁰C:

- 3.2.1 **Apparatus/Equipments:**
- Hydrometer of desired range.
 - Measuring Cylinder 100 ml.
 - Constant temperature water bath
 - Thermometer (0 - 50⁰C/100⁰C)

3.2.2 **Procedure:**

Pour the Epoxy Plasticizers into a clean and dry 100 ml measuring cylinder.

Allow the enclosed air bubble to escape out of the viscous liquid, in case of excessive air bubbles, the material should be preheated to remove all air bubbles. Keep the measuring cylinder containing Epoxy Plasticizers in the water bath till it attains the temperature of the bath. Lower the hydrometer gently in to the Epoxy Plasticizers & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material and note down the reading.

In case of critical supplies, the specific gravity should be determined by specific gravity bottle. The test procedure employed would be as per IS 3672.

DETERMINATION OF REFRACTIVE INDEX:

- 3.3.1 **Outline of the Method:** The refractive index is determined by Abbe's Refractometer at 30⁰C using diffused daylight or any convenient light as an illuminate.

- 3.3.2 **Refractometer:** - An Abbe's Refractometer with the scale graduated directly in terms of refractive index of the D-line of sodium at a temperature of 30⁰C. Water supply is used for maintaining this temperature.

3.3.3 **Procedure:**

Place the Refractometer in front of a suitable source of light (either day light or electric light). Adjust the temperature of the Refractometer to 30⁰C \pm 0.1⁰ C by the circulation of water. Clean the prisms with alcohol and clamp it dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eyepiece on the cross hair and the reading lens of the scale by moving the prism-arm and find a position where the lower part of the field is dark and the upper part is bright. In general, the border line, which is coloured, shall be corrected by turning the mill head on the right of the telescope until a sharp black and white edge is obtained.

Move the prism-arm until this black edge just crosses the intersection of the cross hair. Read the refractive index on the scale to the fourth decimal place. The accuracy of the instrument should be checked by a standard of known refractive index.

DETERMINATION OF ACID VALUE:

3.4.1 Apparatus/Equipments:

- Burette
- Conical Flask 250 ml.
- Measuring cylinder. 100 ml.
- Electronic Balance.

3.4.2 Reagents:

- KOH AR grade
- Alcohol
- Phenolphthalein indicator

3.4.3 Procedure:

Transfer about 5 ml. of Epoxy Plasticizer to be tested for acid value into dry and clean weighing tube, and weigh the weighing tube alongwith contents. Transfer max. material into a clean and dry conical flask carefully so that the material should not stick at the side of the conical flask or the weighing tube. Weigh the weighing tube alongwith remaining material accurately; the difference in weight would give the quantity of material taken for test.

Now add to the conical flask 50 ml. of alcohol & shake to dissolve the material to get uniform solution. Now titrate the resulting solution with standardised alcoholic KOH solution using phenolphthalein as an indicator. Run the blank i.e. titrate 50 ml. alcohol with the same KOH solution to obtain the blank reading. The volume of KOH consumed for sample solution should be corrected by subtracting the value for blank.

$$3.4.4 \quad \text{Acidity (mg KOH/ g)} = \frac{V \times N \times 56.11}{W}$$

V = Corrected volume of KOH consumed

N = Normality of alcoholic KOH

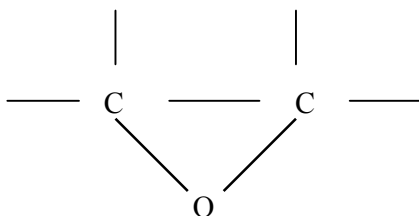
W = Weight of sample.

DETERMINATION OF OXIRANE NUMBER IN EPOXY

PLASTICIZERS:

3.5.1 Scope: - Application to epoxidized Fatty materials and epoxy compound in general.

3.5.2 Method: This method determines to oxirane oxygen contained in the following grouping



Under the prescribed conditions of this method, the oxygen is titrated directly with hydrogen bromide in acetic acid.

3.5.3 Reagents:

1. Glacial acetic acid, A.R. grade, acetic anhydride free.
2. Hydrogen bromide gas, anhydrous, available in cylinders or 30-32 % hydrogen bromide in acetic acid A.R. grade.
3. Crystal violet (Gentian violet), Eastman Kodak Co.
4. Potassium hydrogen phthalate A.R. grade, Standard for acidimetry. Dry for 2 hours at 120°C and allow to cool in an efficient desiccator prior to use.
5. Benzene, A.R. grade or chlorobenzene, A.R. grade.

3.5.4 Solutions:

1. Crystal violet indicator solⁿ.; Dissolve 0.1 g of crystal violet in 100 ml. Of glacial acetic acid.
2. Hydrogen bromide 0.1 N in acetic acid.;
 - a. Prepare by bubbling hydrogen bromide gas through glacial acetic acid to approximately 0.1 N a torsion type balance may be used to estimate the amount of hydrogen bromide added.
 - b. Or prepare by diluting 30 to 32 % (about 4 N) hydrogen bromide in acetic acid with glacial acetic acid to approximately 0.1 N.

3.5.5 Standardization of the hydrogen bromide (0.1 N in acetic acid) solution :

Weigh accurately about 0.4 g of dry potassium hydrogen phthalate and dissolve in 10 ml. Glacial acetic acid. Heat carefully to dissolve, using a hot plate. Titrate solution at room temperature with hydrogen bromide using no more than 0.1 ml. (5 drops from a fine dropper) of crystal violet indicator. Standardization should be in duplicate with a difference not to exceed 0.0004 N. Re Standardize each day sample are analyzed.

$$\text{Normality} = \frac{\text{Weight of phthalate}}{0.2042 \times \text{Volume consumed of Hydrogen Bromide}}$$

3.5.5 Procedure:

1. Weigh about 0.3 to 0.5 g of the sample accurately transfer into a 50-ml Erlenmeyer flask. Dissolve the sample in 10 ml. Of benzene or chlorobenzene / Glacial acetic acid (in case of epoxy resins use chlorobenzene) add stirring bar and crystal violet indicator (Maximum 0.1 ml or 5 drops with a fine dropper)
2. Place the rubber stopper in position and lower the tip of the burette until the discharges just above the solution. This is important to avoid loss of hydrogen bromide.
3. Stir and titrate the sample (rapidly at first.) with the 0.1 N hydrogen bromide solution to a bluish-green end point that persists for 30 seconds. Control the rate of magnetic stirrer so as avoid splashing.

3.5.6 Calculation: Oxirane oxygen content (%) = $\frac{\text{Volume consumed of H Br} \times \text{N} \times 1.60}{\text{Weight of sample}}$

Ref.: A.O.C.S.Official Method cd.3a-63, Reapproved 1973.

METHOD FOR TESTING COLOUR STABILITY:

- 3.6.1 Apparatus/Equipments:
- Oil bath maintained at $180^{\circ}\text{C} \pm 1^{\circ}\text{C}$ or air circulating oven kept at the same temperature.
 - Test tube 50 ml. capacity or 100 ml. beaker
 - Stop watch

3.6.2 Procedure:

Pour about 30 ml of sample in the test tube or beaker as the case may be, and place the test tube/beaker with contents in the oil bath or oven maintained at $180^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Remove the test tube/ beaker at the end of one hour.

- 3.6.3 Conformity: Material stands approved, if the colour change is within agreed/ approved material.

TEST PROCEDURE FOR STABILIZERS

4.1 COLOUR ESTIMATION:

- 4.1.1 Minimum Acceptable Standard:** Sample of each supplier shall be sealed and kept separate. Sample and standard approx 5 gm each shall be spread by spatula on white paper and compared visually.

Any rejection shall be reported by visual inspection only.

DETERMINATION OF SPECIFIC GRAVITY:

4.2.1 SPECIFIC GRAVITY FOR SOLID:

- 4.2.1.1 APPARATUS/EQUIPMENTS:**
- Specific gravity bottle 25 ml.
 - Electronic Balance

- 4.2.1.2 Reagents:**
- Toluene AR Grade

4.2.1.3 Procedure:

Take a dry and clean R.D. bottle and find out its weight. Now fill with toluene and close it with the stopper, find out its weight. Dry the R.D. bottle again and put some dry sample, find out its weight. Now fill this R.D. bottle with toluene and shake it so that no air bubble remains in the bottle. Now allow the R.D. bottle to stand for some time. When the solid particles settle down, put some more toluene and close it with stopper and find out its weight.

- 4.2.1.4 Calculation:** All the determination should be carried out at 27°C

$$\text{Specific gravity} = \frac{(W_3 - W_1)}{(W_3 - W_1) - (W_4 - W_2)} \times 0.855$$

Where,

| | |
|-------|---|
| W_1 | = Weight of empty R.D. bottle |
| W_2 | = Weight of R.D. bottle + toluene |
| W_3 | = Wt. of R.D. bottle + sample |
| W_4 | = Wt. of R.D. bottle + toluene + sample |
| 0.855 | = Sp. Gravity of Toluene. At 27°C |

SPECIFIC GRAVITY FOR LIQUID:

- 4.2.2.1 Apparatus/Equipments:**
- Hydrometer of desired range.
 - Measuring Cylinder 100 ml.
 - Constant Temperature Water Bath
 - Thermometer (0-100°C)

4.2.2.2 Procedure:

Pour the liquid into a clean and dry 100 ml measuring cylinder.

Allow the enclosed air bubbles to escape out of the viscous liquid, in case of excessive air bubbles, the material should be preheated to remove all air bubbles. Keep the measuring cylinder containing liquid in the water bath maintained at specified temperature till it attains the temperature of the bath. Lower the hydrometer gently into the liquid & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material and note down the reading.

In case of critical supplies the specific gravity should be determined by specific gravity bottle. The test procedure employed would be as per IS 3672-1966.

DETERMINATION OF MOISTURE:

- 4.3.1 Apparatus/Equipments:**
- Oven maintained at a temperature of 80°C/105°C
 - Electronic Balance
 - Petri dish

4.3.2 Procedure:

Place a petri dish in the oven for half an hour and allow it to cool in a desiccator, when it acquire the room temperature, weigh it accurately. Put sample in the petri dish and weigh it again. Place the petri dish, alongwith the sample in the oven (maintained the temperature at 80°C/105°C) for one hour. Remove the sample from the oven and put it in the desiccator for cooling. Now find out the weight of the petri dish containing the dry sample.

4.3.3 Calculation:

$$\text{\%age of Moisture} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

- w_1 = Weight of the petri dish
 w_2 = Weight of the sample + petri dish
 w_3 = Weight of the dry sample + petri dish.

METAL OXIDE CONTENTS:

DETERMINATION OF LEAD OXIDE IN TBLS, DBLP, STABILIZER-A, DBL-PHOSPHITE:

4.4.1.1 Apparatus/Equipments:

- Volumetric flask 500 ml.
- Conical Flask 250 ml.
- Burette 50 ml
- Pipette 10 ml
- Measuring Cylinder 100 ml
- Hot Plate with energy regulator
- Electronic Balance
- Oven

4.4.1.2 Reagents:

- Nitric acid AR grade
- Toluene AR grade
- EDTA AR grade
- Xynol orange AR grade
- Hexamine AR grade

4.4.1.3 Procedure:

Weigh accurately about 2.5 g of pre dried stabiliser and transfer quantitatively in a clean and dry conical flask, add to it 50 ml 1:1 HNO₃ solution and boil to dissolve. Cool the contents and transfer to 500 ml volumetric flask carefully, shake the contents and pipette out 10 ml for titration. Add 25 ml distilled water & two drops of xynol orange 0.1% indicator. Add Hexamine till the colour change from yellow to pink or red. Titrate with standardised EDTA solution to sharp lemon yellow end point and note the volume consumed.

4.4.1.4 Calculation: $\text{Lead oxide \% age} = \frac{\text{Volume consumed} \times \text{Mor. of M/50 EDTA} \times 223}{\text{Weight of sample} \times 10}$

DETERMINATION OF LEAD OXIDE IN LS, DBLS, AND OTHER CONTAINING FATTY ACID COMPONENTS:

4.4.2.1 Apparatus/Equipments:

- Conical flask 250 ml.
- Burette 50 ml
- Pipette 10 ml
- Measuring Cylinder 100 ml
- Heating Mantle with energy regulator
- Electronic Balance
- Round bottom flask 500 ml.
- Condenser

4.4.2.2 Reagents:

- Nitric acid AR grade
- Toluene AR grade
- EDTA AR grade
- Xynol orange AR grade
- Hexamine AR grade

4.4.2.3 Procedure:

- Weigh accurately 2.5 gm of sample in 500 ml round bottom flask.
- Add 25 ml of concentrated HNO_3 , 50 ml of distilled water and 30 ml of toluene.
- Reflux for 1 hour (add 1-2 boiling chips) using water condenser.
- Cool to room temperature.
- Transfer the contents to a 500 ml separating funnel. Collect lower aqueous layer in a 500 ml volumetric flask. Wash the organic layer twice with water, combine with initial aqueous layer and make up to the mark with water. Preserve the organic layer part, pipette out 10 ml aliquot in a 250 ml conical flask, add 50 ml distilled water.
- Add two drops of xynol orange 0.1% indicator. Add Hexamine till the colour change from yellow to pink or red.
- Titrate against M/50 EDTA solution till the colour changes from pink to lemon yellow.

4.4.2.4 Calculation:

$$\text{Lead oxide \%age} = \frac{\text{Volume consumed} \times \text{Mor. of M/50 EDTA} \times 223}{\text{Weight of sample} \times 10}$$

ESTIMATION OF BARIUM AS BARIUM OXIDE IN

BARIUM STEARATE:

4.4.3.1 Principle: Barium Stearate is decomposed with 1:1 hydrochloric acid to produce barium chloride and stearic acid, Stearic acid being insoluble and the same would be separated out on cooling whereas the barium chloride remains in solution. Barium could be estimated gravimetrically as sulphate.

4.4.3.2 Apparatus/Equipments:

- Desiccator
- Beaker 250 ml. and 500 ml.
- Funnel
- Silica crucible
- Wash bottle
- Muffle furnace
- Hot Plate
- Electronic Balance
- What man filter paper no.41 & no.42

4.4.3.3 Reagents:

- Hydrochloric acid AR Grade
- Sulphuric acid AR Grade

4.4.3.4 Procedure:

Weigh accurately pre dried Barium Stearate powder (1-1.5gm) & transfer quantitatively into 250 ml. beaker. Add to it 50 ml. of 1:1 hydrochloric acid solution, warm on the hot plate to dissolve all the barium Stearate and when dissolution is completed, it is indicated by appearance of white powder. Allow the contents to cool. Filter the contents through what man filter paper no. 41 to separate out stearic acid. Wash the residue on filter paper with warm distilled water till the last washing is free from chloride ion. Combine the filtrate and washing in a 500 ml. beaker. Reduce the volume to 200 ml. Add dilute sulphuric acid to precipitate the sample. Allow the precipitate to settle. Check the supernatant liquid for complete precipitation, by adding further quantity of dilute sulphuric acid solution & when precipitation is completed, allow it to settle for two hours.

Filter the supernatant liquid through what man filter paper no. 42. Wash the precipitate with hot water continue washing of ppt on filter paper with warm water till washing is free from chloride ion. Transfer filter paper along with ppt in the dry empty silica crucible. Contents of the crucible heated first at low heat and then strongly in the muffle furnace. Cool the silica crucible alongwith contents in a desiccator. Weigh accurately.

4.4.3.5 Calculation:

$$\text{Barium Oxide \%} = \frac{65.7 \times X}{m}$$

where X = mass of the barium sulphate residue
m = mass of the sample initially taken for test.

DETERMINATION OF CADMIUM AS CADMIUM OXIDE:

4.4.4.1 Apparatus/Equipments:

- Sintered crucible G-4
- Filter pump
- Air oven maintained at 105°C
- Kipp's apparatus.

4.4.4.2 Procedure:

Weigh out accurately about 1 gm of the sample and transfer it to a beaker, add 50ml of 1:1 hydrochloric acid .Digest the content for one hour on a hot plate at 60-65°C. During this treatment cadmium goes to solution as cadmium chloride .Filter after cooling and wash the precipitate with warm distilled water thoroughly. Collect the washing and filtrate together. Now neutralise the solution with ammonium hydroxide. Boil the neutral solution and pass steady stream of hydrogen sulphide slowly under pressure through a jet. A yellow precipitate is obtained; pass the gas for half an hour to ensure complete precipitation. Boil the content for half an hour.

Take a sintered crucible G-4 and put in the oven at 105°C for half an hour and allow it to cool in a desiccator, weigh it and filter through it & wash the precipitate with warm water and dry the same at 105°C for one hour. Cool the crucible in the desiccator weigh it and find out the weight of precipitate and calculate the percentage as follows:

4.4.4.3 Calculation: Cadmium Oxide % age = $\frac{88.8 \times m}{M}$

m = Weight of the cadmium sulphide

M = Weight of sample taken for the test.

DETERMINATION OF METAL OXIDE CONTENTS IN

CALCIUM STEARATE:

- 4.4.5.1 Apparatus/Equipments:**
- Auto burette 25 ml capacity
 - Beaker 250 ml capacity
 - Glass Funnel
 - Volumetric flask 500 ml.
 - Pipette 10 ml.
 - Hot plate

- 4.4.5.2 Reagents:**
- Hydrochloric acid AR grade
 - Murexide indicator
 - Normal NaOH
 - M/50 EDTA

4.4.5.3 Procedure:

- (a) Weigh out accurately 1-2 gm pre dried material and transfer quantitatively into the beaker. Add to it 25 ml. of 1:1 Hydrochloric acid and digest the content on the hot plate to dissolve all the powdered material. When the dissolution is completed, cool the contents to solidify stearic acid formed. Filter the content through what man filter paper No.41, wash the residue with warm DM water to remove chloride ions. Collect the filtrate and washing in to a 500 ml. volumetric flask & make up the volume up to the mark.
- (b) Pipet out 10 ml. from stock 500 ml. solution and add 15-20 ml. DM. water. Add 0.5 g of murexide indicator and N - NaOH solution to increase pH. Now titrate with M/50 EDTA solution and note the volume consumed when the colour change from pink to purple.

4.4.5.4 Calculation:

$$\begin{array}{lcl} \text{Metal Oxide Contents (\%)} & = & \frac{V \times M \times 56}{W \times 10} \\ \text{(As Calcium oxide)} & & \end{array}$$

$$\begin{array}{lcl} V & = & \text{Volume consumed of M / 50 EDTA} \\ M & = & \text{Molarity of M / 50 EDTA} \\ W & = & \text{Weight of sample.} \end{array}$$

- 4.4.5.5 Alternate Method:** Dissolve ash contents (No.4.7) of calcium stearate in 1:1 HCl and transfer to 500 ml volumetric flask. Make up the solution up to the mark and follow the procedure of (No.4.4.5.3 (b)).

DETERMINATION OF WATER SOLUBLE SALT:

- 4.5.1 Apparatus/Equipments:**
- Volumetric Flask 500 ml
 - 100 ml Beaker
 - 100 ml Measuring Cylinder
 - Glass Funnel
 - What man Filter Paper No. 41
 - Hot Plate

4.5.2 Procedure:

Take 10 gm of accurately weighed dry sample and mix it with 500 ml of pure distilled water thoroughly. Now filter it through Whatman filter paper No.41 and take 100 ml of the filtrate and transfer to 100 ml clean, dry and pre weighed beaker. Boil it on hot plate up to dryness. Cool it in desiccator and weigh it.

- 4.5.3 Calculation:** Water soluble salt (%) = $\frac{W_2 - W_1}{W} \times 100$
- W_1 = weight of empty beaker.
 W_2 = weight of beaker after drying the sample
 W = Weight of sample.

4.6 SIEVE ANALYSIS:

- 4.6.1 Apparatus/Equipments:**
- Standard Sieve of 200 mesh
 - Beaker 250 ml
 - One Palette Knife
 - Electronic Balance
 - Oven
 - Brush

- 4.6.2 Reagents:**
- DOP
 - Toluene Commercial

4.6.3 Procedure:

Accurately weigh 10 gm of the sample dried at 105°C for one hour and transfer to a 250 ml beaker. Thoroughly incorporate a sufficient quantity of DOP with a palette knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of toluene solvent and transfer with further quantity of toluene solvent Wash the residue on the sieve with Toluene solvent gently brushing with a soft camel hair brush, until washing is clear. Dry the residue to constant weight at 105 ± 2°C.

Express the result as the percentage of retention.

- 4.6.4 Calculations:** Percentage of retention over 200 mesh = $\frac{W_1}{W_2} \times 100$
- W_1 = weight of the sample retained
 W_2 = Weight of sample.

DETERMINATION OF ASH CONTENT:

4.7.1 Apparatus/Equipments: - Silica Crucible 25 ml

- Muffle furnace maintained at $850 \pm 50^{\circ}\text{C}$.
- Desiccator
- Electronic Balance

4.7.2 Procedure:

Weigh accurately 2 to 5 g of oven dried material into a silica crucible. Keep the silica crucible alongwith contents in the muffle furnace at room temperature. Now increase the temperature of muffle furnace gradually to $850 \pm 50^{\circ}\text{C}$, ignite the contents to constant weight. Remove the crucible from the furnace, cool in the desiccator to room temperature, weigh the crucible accurately.

4.7.3 Calculation: Ash Content % by weight = $\frac{W_1}{W_2} \times 100$

W_1 = weight in g of the Ash

W_2 = weight in g of material taken for test.

4.8 DETERMINATION OF FREE STEARIC ACID:

4.8.1 Principle: The Free stearic acid is dissolved in a benzene or toluene where the metal stearic would not be affected; the quantity can be estimated by evaporating the solvent.

4.8.2 Apparatus/Equipments:

- Wide mouth bottle 200 ml. with stopper
- Funnel $2\frac{1}{2}$ " dia
- Pipette 20 ml.
- Evaporating dish
- Conical flask
- Oven
- What man filter paper No. - 41

4.8.3 Chemicals: - Pure benzene/ Toluene

4.8.4 Procedure:

Weigh approx 10 g of the material and transfer quantitatively in to the wide mouth bottle containing 100 ml. of benzene/toluene. Put stopper and shake properly to dissolve all free fatty acid. Continue the shaking for 10 minutes. Filter, collect the filtrate in a conical flask. Pipette out 20 ml. of filtrate in to a clean, dry and pre weighed evaporating dish.

Evaporate the solvent in an oven till all the solvent vaporise. Do not bake the contents. Clean the outer surface of the evaporated dish with acetone to remove moisture. Weigh accurately the evaporating dish along with contents.

4.8.5 Calculation: Percentage of free stearic acid = $\frac{500 \times X}{m}$

X = the residue left after evaporation of solvent in the dish and

m = Quantity of sample initially taken for test.

DETERMINATION OF MELTING POINT:

- 4.9.1 Apparatus/Equipments:**
- Melting point apparatus
 - Thermometer of suitable range
 - Capillary tube

4.9.2 Procedure:

Seal the lower end of melting point tube. Fill the powdered sample in the melting point tube so that the sample forms a thread of 25 mm. length. Place the melting point tube containing the sample and thermometer side by side in the melting point apparatus. Start heating & select the suitable range by selecting the knob on the instrument and read thermometer reading when the sample just melts. This will be indicated by the disappearance of the thread.

DETERMINATION OF BULK DENSITY:

- 4.10.1 Apparatus/Equipments:**
- Measuring Cylinder 25 ml capacity.
 - Electronic Balance.

4.10.2 Procedure:

Weigh about 10 g of sample and transfer to 25 ml measuring cylinder. The initial volume of the sample should be less than 25 ml. Now tap it 14 times at the height of 3-4 cm. Note the reduced volume.

- 4.10.3 Calculation:**
- $$\text{Bulk Density (g/ml)} = \frac{M}{V}$$
- V = Volume of sample in cylinder after tapping
M = Weight of sample taken for test.

DETERMINATION OF REFRACTIVE INDEX:

- 4.11.1 Outline of the Method:** The refractive index is determined by Abbes Refractometer at a temperature mentioned in specification using diffused daylight or any convenient light as an illuminate.

- 4.11.2 Refractometer:** An Abbes Refractometer with the scale graduated directly in terms of refractive index of the D-line of sodium at a temperature mentioned in specification Water supply is used for maintaining this temperature.

4.11.3 Procedure:

Place the Refractometer in front of a suitable source of light (either day light or an electric light). Adjust the temperature of the Refractometer to a temperature mentioned in specification by the circulation of water. Clean the prisms with alcohol and clamp it dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eyepiece on the cross wire and the reading lens of the scale by moving the prism-arm and find a position where the lower part of the field is dark and the upper part is bright. In general, the border line, which is coloured, shall be corrected by turning the mill head on the right of the telescope until a sharp black and white edge is obtained.

Move the prism-arm until this black edge just crosses the intersection of the cross hair. Read the refractive index on the scale to the fourth decimal place.

LUBRICANTS

5.1 COLOUR:

5.1.1 Minimum Acceptable Standard:

SOLID: Sample of each supplier shall be sealed and kept separate. Sample and standard approx. of 5 gm each shall be spread by spatula on white paper and compared visually.

LIQID: Take 50 ml each of standard sample and supply in seprate 100ml beaker and then compare colour of supplies with the colour of the standard/ approved sample.

APPEARANCE:

Powder should be free from contamination as per standard sample.

5.2 DETERMINATION OF MELTING POINT:

- 5.2.1 Apparatus/Equipments:
- Melting point apparatus
 - Thermometer of suitable range
 - Melting point capillary tube

5.2.2 Procedure:

Seal the lower end of melting point tube fill the powdered sample in the MP tube so that the sample forms a thread of 25 mm length. Suspend the melting point tube containing the sample and thermometer side by side in the MP apparatus. Start heating (select the suitable range by the selecting knob on the instrument) read the thermometer when the sample just melts. This will be indicated by disappearing of “thread”.

5.3 DETERMINATION OF ACID VALUE IN LUBRICANTS:

- 5.3.1 Apparatus/Equipments:
- Hot Plate
 - Heating Mantle
 - Conical flask 250 ml
 - Burette 50 ml
 - Measuring Cylinder 100 ml

- 5.3.2 Reagents:
- KOH AR Grade
 - Alcohol
 - N/10 HCl
 - Phenolphthalein Indicator

5.3.3 Procedure:

Weigh accurately 3-4 g of sample in 250 ml clean and dry conical flask and dissolve in 50 ml of neutral alcohol & warm on hot plate for complete dissolution (if necessary) Cool and titrate against standardised alcoholic KOH using phenolphthalein as an indicator.

- 5.3.4 Calculation: Acidity (mg KOH/g) =
$$\frac{N \times V \times 56.11}{W}$$

N = Normality of alcoholic KOH
V = Volume consumed during titration
W = Weight. Of sample

5.4 DETERMINATION OF SAPONIFICATION VALUE:

- 5.4.1 Apparatus/Equipments:
- Electronic Balance
 - Round Bottom Flask capacity 500 ml.
 - Conical Flask 250 ml
 - Condenser
 - Heating Mantle
 - Burette 50 ml

- 5.4.2 Reagents:
- Alcohol
 - KOH AR Grade
 - Standard HCl
 - Phenolphthalein Indicator

5.4.3 Procedure:

Weigh accurately about 3 g of the sample in a round bottom flask. Add 50 ml of freshly prepared Alcoholic KOH solution. Take another 50 ml of the same for determination of blank and titrate with standard HCl. Put some small stone pieces in a round bottom flask and then reflux it for 1 hour. After the reaction is over, cool it and add about 15 ml of neutral alcohol and titrate with standardised HCl using phenolphthalein as an indicator.

5.4.4 Calculation:

$$\text{Saponification value (mg / g)} = \frac{(V_1 - V_2) \times N \times 56.11}{W}$$

| | | |
|-------|---|--|
| V_1 | = | Volume in ml. consumed for determination of blank |
| V_2 | = | Volume in ml. consumed for determination of sample |
| N | = | Normality of HCl. |
| W | = | Weight of sample |

5.5 DETERMINATION OF IODINE VALUE:

- 5.5.1 Apparatus/Equipments:
- Iodine flask 500 ml capacity
 - Burette
 - Pipette 25 ml and 10 ml
 - Electronic Balance

- 5.5.2 Reagents:
- Iodine mono chloride AR Grade
 - Carbon tetra chloride AR Grade

5.5.3 Procedure:

Weigh accurately about 1.0 g of sample in iodine flask; add 15 ml of carbon tetra chloride (CCl_4) and heat on the water bath/heating mantle to dissolve completely. Then add 10 ml of iodine mono chloride solⁿ. (0.2 N ICL in acetic acid. Mix 16.24 Gms ICL in 1000 ml glacial acetic acid.) Cork the flask and keep it in the dark for one hour. Similarly take the blank reading by keeping another flask but without the sample. 15 ml of CCl_4 and 10 ml of iodine mono chloride solⁿ.

When the reaction is over remove both the flask. Add around 15 ml of 10% KI solution. Then titrate against 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution till the colour change to Pale Yellow. Then add 1% starch indicator and titrate till the blue colour turn to white.

5.5.4 Calculation: Iodine Value (g/100 g) = $\frac{(\text{B.R.} - \text{S.R.}) \times 0.1 \times 12.69}{W}$

| | | |
|-------------|---|------------------|
| Where, B.R. | = | Blank reading |
| S.R. | = | Sample reading |
| W | = | Weight of sample |

5.6 DETERMINATION OF SPECIFIC GRAVITY:

- 5.6.1 Apparatus/Equipments:**
- Hydrometer of the desired range.
 - 100 ml. measuring cylinder
 - Constant temperature bath
 - Thermometer (0 – 110 °C)

5.6.2 Procedure:

Pour the liquid into a clean and dry 100 ml measuring cylinder.

Allow the enclosed interpreted air bubble to escape out of the viscous liquid; in case of excessive air bubbles the material should be preheated to remove all air bubbles. Keep the measuring cylinder containing liquid in a constant temperature water bath maintained as per specification, till the material in the measuring cylinder attains the temperature of the bath. Lower the hydrometer gently in to the liquid & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material and note down the reading.

In case of critical supplies the specific gravity should be determined by specific gravity bottle. The test procedure employed would be as per IS 3672-1966.

FILLERS

6.1 **APPEARANCE:** Powder free from contamination as per standard sample.

6.2 **COLOUR:**

6.2.1 **Minimum Acceptable Standard:** Sample of each supplier shall be sealed and kept separate. Sample and standard approx. of 5 g each shall be spread by spatula on white paper and compared visually.

- Any rejection shall be reported only by visual inspection.

6.3 **DETERMINATION OF BULK DENSITY:**

6.3.1 **Apparatus/ Equipments:** - Cylinder 25 ml capacity.
- Electronic Balance.

6.3.2 **Procedure:**

Weigh about 10 g of sample and transfer to 25 ml cylinder. The initial volume of the sample should be less than 25 ml. Now tap it 14 times at the height of 3-4 cm. Note the reduced volume.

6.3.3 **Calculation:**

$$\text{Bulk Density (g/ml)} = \frac{M}{V}$$

V = Volume of sample in cylinder after tapping
M = Weight of sample taken for test.

6.4 **SPECIFIC GRAVITY:**

6.4.1 **Apparatus/ Equipments:** - Specific gravity bottle 25 ml. capacity
- Electronic Balance

6.4.2 **Reagents:** - Toluene AR Grade

6.4.3 **Procedure:**

Take a dry and clean R.D. bottle and find out its weight. Now fill with toluene and close it with the stopper, find out the weight. Dry the R.D. bottle again and put some dry sample, find out its weight. Now fill this R.D. bottle with toluene and shake it so that no air bubble remains in the bottle. Now allow the R.D. bottle to stand for some time. When the solid particles settle down, put some more toluene and close it with stopper and find out its weight.

6.4.4 **Calculation:** All the determination should be carried out at 27°C.

$$\text{Specific gravity} = \frac{(W_3 - W_1)}{(W_3 - W_1) - (W_4 - W_2)} \times 0.855$$

Where,

W₁ = Weight of empty R.D. bottle
W₂ = Weight of R.D. bottle + toluene
W₃ = Weight. Of R.D. bottle + sample
W₄ = Weight. Of R.D. bottle + toluene + sample
0.855 is Specific Gravity of Toluene at 27°C.

6.5 DETERMINATION OF MOISTURE:

- 6.5.1 Apparatus/Equipments:
- Wide mouth weighing bottle 50 ml. capacity with Suitable glass lid/ Petri dish
 - Desiccator
 - Oven maintained at $105 \pm 1^{\circ}\text{C}$

6.5.2 Procedure:

Place clean weighing bottle (lid open)/ Petri dish in the oven at $105 \pm 1^{\circ}\text{C}$ for 30 minutes. Cool the weighing bottle/ Petri dish in the desiccator to attain room temperature & weigh accurately. Now transfer about 3/4 g powder sample in the pre weighed weighing bottle/ Petri dish. Weigh the weighing bottle/ Petri dish along with content accurately. Keep the weighing bottle (lid open)/ Petri dish in the oven at $105 \pm 1^{\circ}\text{C}$ for a period of one hour. Remove the weighing bottle/ Petri dish from an oven and cool it in desiccator. Now record the final weight.

6.5.3 Calculation: Percentage of moisture $= \frac{W_1 - W_2}{W_1 - W} \times 100$

W = Weight of the empty and dry weighing bottle/ Petri dish
W₁ = Weight of the weighing bottle/ Petri dish + sample
W₂ = Weight of the weighing bottle/ Petri dish + Sample duly dried.

6.6 SIEVE ANALYSIS:

- 6.6.1 Apparatus/Equipments:
- Standard Sieve of 300 mesh
 - Beaker 250 ml
 - One Palette Knife
 - Balance
 - Oven
 - Brush

- 6.6.2 Reagents:
- DOP
 - Toluene Commercial

6.6.3 Procedure:

Accurately weigh 10 g of the sample dried at 105°C for one hour and transfer to a 250 ml beaker. Thoroughly incorporate a sufficient quantity of DOP with a palette knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of toluene solvent and transfer with further quantity of toluene solvent Wash the residue on the sieve with Toluene solvent gently brushing with a soft camel hair brush, until washing is clear. Dry the residue to a constant weight at $105 \pm 2^{\circ}\text{C}$.

Express the result as the percentage of retention.

6.6.4 Calculations: Percentage of retention over 300 mesh $= \frac{W_1}{W_2} \times 100$

Where W₁ = weight of the sample retained
W₂ = Weight of sample.

6.7 DETERMINATION OF COATING:

6.7.1 Apparatus/Equipments:

- Electronic Balance
- 250 ml beaker
- Watch Glass
- 500 ml Volumetric Flask
- Conical Flask 250 ml
- 100 ml Measuring Cylinder
- Hot Plate
- Burette 25 ml/50 ml.
- Pipette 10 ml

6.7.2 Reagents:

- Conc. HCl
- M/50 EDTA
- Murioxide
- Erichrom Black T
- Ammonia Buffer
- Normal NaOH
- Phenolphthalein Indicator
- N/10 NaOH
- Methanol AR grade

6.7.3 Procedure:

Weigh accurately about 2.5 g of pre dried sample into 250 ml beaker, add 50 ml 1:1 HCl and boil it to dissolve, cool it at room temperature and keep it in a freeze for 2 hours to separate coating. Now filter through Whatman Filter paper No.41 and remove chloride by washing with hot (55-60°C) DM water. Make up the filtrate into 500 ml volumetric flask. This is a stock solution.

Take 50 ml of ethanol /methanol in a dry and clean conical flask & add 1-2 ml Phenolphthalein indicator, remove acidity present in ethanol /methanol by titrating with N/10 NaOH. Now dissolve the coating in ethanol /methanol by slight warming. Cool and titrate against N/10 NaOH, note the volume consumed.

6.7.4 Calculation: Coating as Calcium Stearate(%) =
$$\frac{M \times V \times 303}{W \times 10}$$

where M = Molarity of N/10 NaOH
V = Volume consumed during titration
W = Weight of sample

6.8 DETERMINATION OF CaCO₃:

6.8.1 Procedure:

Pipette out 10 ml. from stock solution (No.-6.7.3) in a clean conical flask & add 15 to 20 ml. D.M. water. Now add 5 to 10 ml. NaOH solution to increase the pH of the solution and murexide indicator. Titrate with standardised M/50 EDTA solution and note the volume consumed during titration when colour change from pink to purple.

6.8.2 Calculation:

$$\text{CaCO}_3 \text{ \%age} = \frac{V \times M \times 100}{W \times 10}$$

M = Molarity of M/50 EDTA

V = Volume in ml. of M/50 EDTA solution

W = Weight of sample in gm taken for test

6.5 DETERMINATION OF MOISTURE:

6.5.1 Apparatus/Equipments:

- Wide mouth weighing bottle 50 ml. capacity with Suitable glass lid/ Petri dish
- Desiccator
- Oven maintained at $105 \pm 1^\circ\text{C}$

6.5.2 Procedure:

Place clean weighing bottle (lid open)/ Petri dish in the oven at $105 \pm 1^\circ\text{C}$ for 30 minutes. Cool the weighing bottle/ Petri dish in the desiccator to attain room temperature & weigh accurately. Now transfer about 3/4 g powder sample in the pre weighed weighing bottle/ Petri dish. Weigh the weighing bottle/ Petri dish along with content accurately. Keep the weighing bottle (lid open)/ Petri dish in the oven at $105 \pm 1^\circ\text{C}$ for a period of one hour. Remove the weighing bottle/ Petri dish from an oven and cool it in desiccator. Now record the final weight.

6.5.3 Calculation:

$$\text{Percentage of moisture} = \frac{W_1 - W_2}{W_1 - W} \times 100$$

W = Weight of the empty and dry weighing bottle/ Petri dish

W₁ = Weight of the weighing bottle/ Petri dish + sample

W₂ = Weight of the weighing bottle/ Petri dish + Sample duly dried.

6.9 DETERMINATION OF MgO:

6.9.1 Procedure:

6.9.1.1 Pipette out 10 ml from stock solution (No.-6.7.3) in a clean conical, flask & add 15 to 20 ml. D.M. water and 15 to 20 ml. of Ammonia buffer, 1 ml. of Eriochrom Black T indicator. Titrate against standardised M/50 EDTA solution, note the volume consumed during titration. When the colour change from wine red to blue.

6.9.1.2 Now Pipette out another 10 ml from stock solution (No.-6.7.3) in a clean conical, flask & add 15 to 20 ml. D.M. water, add 10 ml. N-NaOH solution and murexide indicator. Titrate with M/50 EDTA solution and note the volume consumed during titration when colour change from pink to purple.

6.9.2 Calculation: $\text{MgO \% age} = \frac{(V_1 - V_2) \times N \times 40}{W \times 10}$

V_1 = Volume in ml of M/50 EDTA solution consumed in A,

V_2 = Volume in ml of M/50 EDTA solution consumed in B,

W = Weight of sample in gm taken for test.

N = Normality of M/50 EDTA.

6.10 DETERMINATION OF pH OF 1% SUSPENSION:

6.10.1 Apparatus/Equipments:

- Electronic Balance
- Wide mouth bottle 200 ml. capacity with stopper
- Measuring cylinder 100 ml. capacity
- pH meter with glass and calomel electrodes
- Beaker 100 ml. capacity

6.10.2 Reagents:

- Buffer solution of 4 & 10 pH

6.10.3 Procedure:

Weigh accurately about 1.0 g of sample, transfer quantitatively into the wide mouth bottle & close with stopper. Pour 100 ml distilled water in the wide mouth bottle containing 1 g of sample. Shake vigorously for three times of 5 minutes each. Dip the electrodes of pH Meter (calibrated) into water extract of sample & read the pH of the solution.

7.0 PIGMENTS

7.1 DETERMINATION OF MOISTURE:

- 7.1.1 Apparatus/Equipments:**
- Petri dish
 - Desiccator
 - Electronic Balance
 - Oven maintained at $105 \pm 1^\circ\text{C}$

7.1.2 Procedure:

Place clean petri dish in the oven at $105 \pm 1^\circ\text{C}$ for 30 minutes. Cool the petri dish in the desiccator to attain room temperature & weigh accurately. Now transfer about 3/4 g powder sample in the pre weighed petri dish. Weigh the petri dish along with content accurately. Keep the petri dish in the oven at $105 \pm 1^\circ\text{C}$ for a period of one hour. Remove the petri dish from an oven and cool in desiccator. Now record the final weight.

7.1.3 Calculation: Percentage of moisture =
$$\frac{W_1 - W_2}{W_1 - W} \times 100$$

W = Weight of the empty and dry petri dish

W_1 = Weight of the petri dish + sample

W_2 = Weight of the petri dish + Sample duly dried.

7.2 SIEVE ANALYSIS:

- 7.2.1 Apparatus/Equipments:**
- Standard Sieve of 200 mesh
 - Beaker 250 ml
 - One Palette Knife
 - Electronic Balance
 - Oven
 - Brush

- 7.2.2 Reagents:**
- DOP
 - Toluene Commercial

7.2.3 Procedure:

Accurately weigh about 20 g of the sample dried at 105°C for one hour and transfer to a 250 ml beaker. Thoroughly incorporate a sufficient quantity of DOP with a palette knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of toluene solvent and transfer with further quantity of toluene solvent Wash the residue on the sieve with Toluene solvent gently brushing with a soft camel hair brush, until washing is clear. Dry the residue to constant weight at $105 \pm 2^\circ\text{C}$. Express the result as the percentage of retention.

7.2.4 Calculations: Percentage of retention over 200 mesh =
$$\frac{W_1}{W_2} \times 100$$

W_1 = weight of the sample retained

W_2 = weight of sample.

7.3 DETERMINATION OF pH:

- 7.3.1 Apparatus/Equipments:
- pH meter with glass electrode
 - Funnel
 - Electronic Balance
 - Measuring Cylinder 100 ml.
 - Whatman Filter No. 41.

7.3.2 Procedure:

Take approx 2 g. of sample and mix it thoroughly with 100 ml. of distilled water. Allow it to settle, filter with Whatman filter No.41 and Read the pH of filtrate on the pH meter which is earlier calibrated with standard buffer solution.

7.4 COLOUR STABILITY:

- 7.4.1 Apparatus/Equipments:
- Petri dish
 - Air circulatory oven at $180 \pm 1^\circ\text{C}$

7.4.2 Procedure:

Take about 10 g of sample in the petri dish, and place it in the air circulatory oven maintained at $180 \pm 1^\circ\text{C}$. After one hour, cool the sample in the desiccator. Now compare its colour fastness with that of original sample.

7.5 DETERMINATION OF SPECIFIC GRAVITY:

- 7.5.1 Apparatus/Equipments:
- Specific gravity bottle 25 ml.
 - Electronic Balance

- 7.5.2 Reagents:
- Toluene AR Grade

7.5.3 Procedure:

Take a dry and clean R.D. bottle and find out its weight. Now fill with toluene and close it with the stopper, find out its weight. Dry the R.D. bottle again and put some dry sample, find out its weight. Now fill this R.D. bottle with toluene and shake it so that no air bubble remains in the bottle. Now allow the R.D. bottle to stand for some time. When the solid particles settle down, put some more toluene and close it with stopper and find out its weight.

- 7.5.4 Calculation: - All the determination should be carried out at 27°C .

$$\text{Specific gravity} = \frac{(W_3 - W_1)}{(W_3 - W_1) - (W_4 - W_2)} \times 0.855$$

- W_1 = Weight of empty R.D. bottle
 W_2 = Weight of R.D. bottle + toluene
 W_3 = Wt. of R.D. bottle + sample
 W_4 = Wt. of R.D. bottle + toluene + sample
0.855 = Sp. Gravity of Toluene at 27°C .

7.6 DETERMINATION OF TiO₂ CONTENTS:

- 7.6.1 Apparatus/Equipments:**
- Beaker 500 ml.
 - Crucible silica
 - Funnel
 - Conical flask 250 ml.
 - Hot plate
 - Electronic balance
- 7.6.2 Reagents:**
- Ammonium sulphate AR grade
 - Sulphuric acid AR grade
 - Ammonia solution AR grade

7.6.3 Procedure:

Weigh accurately about 0.1 g of sample and add 2-3 g of Ammonium sulphate and 30 ml. of Sulphuric acid. Digest on hot plate till the solution is clear i.e. dissolution of sample is completed. Now cool it and add 50 ml. of D.M. water to dilute the solution. Heat will be generated during the course of reaction. Cool the solution up to room temperature and add Ammonia solution till the precipitation is completed. Now stand for 3-4 hour for the settling of the precipitate. Filter from whatman filter paper No.41 and wash the filter paper 4 to 5 times with warm water. Now ignite the residue at 850°C for one hour in pre weighed silica crucible. Cool it in a desiccator and weigh again.

7.6.4 Calculation: $\text{TiO}_2 \text{ Contents (\%)} = \frac{W_2 - W_1}{M} \times 100$

W_1 = Weight of empty silica crucible

W_2 = Weight of silica crucible after ignition of sample precipitate

M = Weight of sample.

7.7 DETERMINATION OF ASH CONTENT:

- 7.7.1 Apparatus/Equipments:**
- Silica Crucible 25 ml
 - Muffle furnace maintained at $850 \pm 50^\circ\text{C}$.
 - Desiccator (containing an efficient desiccant)

7.7.2 Procedure:

Weigh accurately 2 to 5 g of an oven dried material into a silica crucible along with contents in the muffle furnace at room temperature. Now increase the temperature of muffle furnace gradually to $850 \pm 50^\circ\text{C}$, ignite the contents to constant weight. Remove the crucible from the furnace, cool in the desiccator to room temperature, weigh the crucible accurately.

7.7.3 Calculation: $\text{Ash Content \% by weight} = \frac{W_1}{W_2} \times 100$

W_1 = weight in g of the Ash

W_2 = weight in g of material taken for test.

7.8 COLOUR APPEARANCE:

7.8.1 Minimum Acceptable Standard:

Sample of each supplier shall be sealed and kept separate. Sample and standard approx. of 5 g each shall be spread by spatula on white paper and compared visually.

Any rejection shall be reported only by visual inspection.

8.0 OTHERS:

8.1 COLOUR:

8.1.1 Minimum Acceptable Standard: Sample of each supplier shall be sealed and kept separate. Sample and standard of approx. 5 g each shall be spread by spatula on a white paper and compared visually.

Any rejection shall be reported only by visual inspection.

8.1.2 APPEARANCE: Powder should be free from contamination as per standard sample.

8.2 DETERMINATION OF BULK DENSITY:

8.2.1 Apparatus/Equipments:

- Measuring Cylinder 25 ml capacity.
- Electronic Balance.

8.2.2 Procedure:

Weigh about 10 g of sample and transfer to 25 ml cylinder. The initial volume of the sample should be less than 25 ml. Now tap it 14 times at the height of 3-4 cm. Note the reduced volume.

8.2.3 Calculation:

$$\text{Bulk Density (g/ml)} = \frac{M}{V}$$

V = Volume of sample in cylinder after tapping

M = Weight of sample taken for test.

8.3 **DETERMINATION OF MOISTURE:**

- 8.3.1 **Apparatus/Equipments:**
- Petri dish.
 - Desiccator
 - Oven maintained at $105 \pm 1^{\circ}\text{C}$
 - Electronic Balance

8.3.2 **Procedure:**

Place clean Petri dish in the oven at $105 \pm 1^{\circ}\text{C}$ for 30 minutes. Cool the Petri dish in the desiccator to attain room temperature & weigh accurately. Now transfer about 3/4 g powder sample in the pre weighed Petri dish. Weigh the Petri dish along with content accurately. Keep the Petri dish in the oven at $105 \pm 1^{\circ}\text{C}$ for a period of one hour. Remove the petri dish from an oven and cool in desiccator. Now record the final weight.

8.3.3 **Calculation:** Percentage of moisture = $\frac{W_1 - W_2}{W_1 - W} \times 100$

W = Weight of the empty and dry Petri dish

W_1 = Weight of the Petri dish + sample

W_2 = Weight of the Petri dish + Sample duly dried.

8.4 **DETERMINATION OF pH:**

- 8.4.1 **Apparatus/Equipments:**
- Beaker 250 ml.
 - pH meter with glass electrode

8.4.2 **Procedure:**

Take about 5 g of sample and mix it thoroughly with 100 ml. of distilled water. Allow it to settle and place the beaker in the water bath so that it acquired the required temperature 27°C . Read the pH on the pH meter which is earlier calibrated with standard buffer solutions.

8.5 SIEVE ANALYSIS:

- 8.5.1 Apparatus/Equipments:
- Standard Sieve of 200 mesh
 - Beaker 250 ml
 - One Palette Knife
 - Electronic Balance
 - Oven
 - Brush

- 8.5.2 Reagents:
- DOP
 - Toluene Commercial

8.5.3 Procedure:

Accurately weigh about 10 g of the sample dried at 105°C for one hour and transfer to a 250 ml beaker. Thoroughly incorporate a sufficient quantity of DOP with a palette knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of toluene solvent and transfer with further quantity of toluene solvent. Wash the residue on the sieve with Toluene solvent, gently brushing with a soft camel hair brush, until washing is clear. Dry the residue to a constant weight at 105 ± 2°C.

Express the result as the percentage of retention.

- 8.5.4 Calculations: Percentage of retention over 200 mesh = $\frac{W_1}{W_2} \times 100$
- W_1 = weight of the sample retained
 W_2 = Weight of sample.

8.6 DETERMINATION OF SPECIFIC GRAVITY:

8.6.1 SPECIFIC GRAVITY FOR SOLID:

- 8.6.1.1 APPARATUS/EQUIPMENTS:
- Specific gravity bottle 25 ml.
 - Electronic Balance
 - Toluene AR Grade

8.6.1.2 Reagents:

8.6.1.3 Procedure:

Take a dry and clean R.D. bottle and find out its weight. Now fill with toluene and close it with the stopper, find out its weight. Dry the R.D. bottle again and put some dry sample, find out its weight. Now fill this R.D. bottle with toluene and shake it so that no air bubble remains in the bottle. Now allow the R.D. bottle to stand for some time. When the solid particles settle down, put some more toluene and close it with stopper and find out its weight.

- 8.6.1.4 Calculation: All the determination should be carried out at 27°C.

$$\text{Specific gravity} = \frac{(W_3 - W_1)}{(W_3 - W_1) - (W_4 - W_2)} \times 0.855$$

Where,

- W_1 = Weigh of empty R.D. bottle
- W_2 = Weigh of R.D. bottle + toluene
- W_3 = Wt. of R.D. bottle + sample
- W_4 = Wt. of R.D. bottle + toluene + sample

0.855 is Specific Gravity of Toluene.

8.6.2 SPECIFIC GRAVITY FOR LIQUID:

- 8.6.2.1 Apparatus/Equipments:**
- Hydrometer of the desired range.
 - 100 ml. measuring cylinder
 - Constant temperature bath
 - Thermometer (0 - 110°C)

8.6.2.2 Procedure:

Pour the liquid into a clean and dry 100 ml measuring cylinder.

Allow the enclosed air bubble to escape out of the viscous liquid, in case of excessive air bubbles the material should be preheated to remove all air bubbles. Keep the measuring cylinder containing liquid in a constant temperature bath maintained as per specification, till the material in the measuring cylinder attains the temperature of the bath. Lower the hydrometer gently in to the liquid & when it has settled, depress it about two scale division on the hydrometer to which the liquid rises with the eye placed at the level of principle surface of the material and note down the reading.

In case of critical supplies the specific gravity should be determined by specific gravity bottle. The test procedure employed shall be as per IS 3672-1966.

8.7 DETERMINATION OF REFRACTIVE INDEX:

- 8.7.1 Apparatus/Equipments:** **Refractrometer:** An Abb's Refractometer B-L Model with scale graduated directly in terms of refractive index of the white light at a temperature of 25°C.

- 8.7.2 Reagent:** - Toluene AR Grade

8.7.3 Procedure:

Place the Refractometer in front of a tungsten lamp and adjust the circulation of water so as to bring the prism to 25°C. Clean with benzene and wipe dry. Spread drop of sample solution prepared in toluene as a solvent upon the lower prism and clamp the prism together. Adjust the mirror so that the light enters the telescope. Focus the eye piece on the cross wire and reading lens of the scale. Upon moving the prism arm a position can now be found where the lower part of the field lens is dark and the upper part is bright. In general the border line will be coloured. Correct by turning the milled head on the right of the telescope so that a sharp black and white edge is obtained. Move the prism arm until this black edge just crosses the intersection of the cross hairs. Read the Refractive Index from the scale estimating the fourth decimal place. Calibration of Refractometer should be done with a standard of known refractive index.

8.8 DETERMINATION OF MELTING POINT:

- 8.8.1 Apparatus/Equipments:**
- Melting point apparatus
 - Thermometer of suitable range
 - Capillary tube

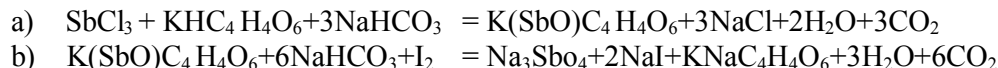
8.8.2 Procedure:

Seal the lower end of melting point tube. Fill the powdered sample in the melting point tube so that sample forms a thread of 25 mm length. Place the melting point tube containing the sample and thermometer side by side in the melting point apparatus. Start heating & select the suitable range by selecting the knob on the instrument and read thermometer reading when the sample just melts. This will be indicated by the disappearance of the thread.

DETERMINATION OF ANTIMONYTRIOXIDE:

8.9.1 Outline of the method: A known quantity of the material is dissolved in hydrochloric acid, treated with potassium hydrogen tartrate and sodium bicarbonate or carbonate and titrated iodometrically.

8.9.2 Reaction: Following reactions take place during estimation:



8.9.3 Apparatus/Equipments:

- Electronic Balance
- Burette 25 ml.
- Conical Flask

8.9.4 Reagents:

- Concentrated hydrochloric acid AR Grade
- Iodine solution - 0.1 N.
- Potassium hydrogen tartrate AR Grade
- Sodium Bicarbonate or sodium carbonate AR Grade
- Starch indicator 0.05% solution

8.9.5 Procedure:

Weigh about 0.1 g of the material and dissolve in about 5 ml of concentrate hydrochloric acid without boiling as antimony tri chloride is volatile with steam then add about 3-4 g of potassium hydrogen tartrate and 20 ml. of water (more water may be added). Then add about 5-6 g of solid sodium carbonate or Bicarbonate. (Titration is possible only when pH is more; hence excess of sodium carbonate or bicarbonate is added). Add 15-20 ml. of water, then add 1 ml. of starch indicator and titrate against 0.1 iodine solution. At the end point colour change from colour less to blue.

8.9.6 Calculation: Antimony oxide, percent by mass $= \frac{7.288 \times N \times V}{M}$

N = Normality of standard iodine solution,
V = volume in ml of standard iodine solution used, and
M = mass in g of the sample taken for the test.
1 ml. N/10 iodine = 0.007288 g. Sb_2O_3

DETERMINATION OF MOLYBDENUM TRIOXIDE:

8.10.1 Apparatus/Equipments:

- Beaker 500 ml. and 250 ml
- Conical Flask 250 ml.
- Measuring cylinder 100 ml.
- Silica crucible
- Electronic Balance
- Furnace

8.10.2 Reagents:

- Barium chloride AR grade
- Ammonia solution AR grade
- Lead acetate tri hydrate AR grade
- Hydrochloric acid AR grade
- Acetic acid AR grade
- Ammonium nitrate AR grade
- pH Indicator.

8.10.3 Qualitative test for sulphate:

Dissolve 0.5 - 1 g. of the sample in 5 ml of A.R. grade Conc. hydrochloric acid by warming in a test tube. Add a little water, if necessary. Dilute the solution with sulphate free water to 15 ml. The solution should remain clear.

Add a few drops of 6% barium chloride solution (6 g of barium chloride in 100 ml. of solution in water) into the above solution of the sample. A white precipitate or turbidity indicates the presence of sulphate.

8.10.4 Determination of Mo O₃ content:

- The following procedure should be applicable only if the test in (No.8.10.3) is negative.
- Dilute ammonia solution 1:2 V/V.
- Acetic acid solution 1:1 V/V.

LEAD ACETATE SOLUTION 4%:

- 4 g of lead acetate tri hydrate is dissolved in 1 ml. of glacial acetic acid and a small amount of water, warming, if necessary. The solution is then dilute to 100 ml.
- Weigh accurately about 0.15 g of the sample in a 500 ml. beaker and dissolve it in about 50 ml of dilute ammonia solution. Add acetic acid solution gradually till the solution is distinctly acidic to the litmus paper. Add 12 g of AR grade ammonium acetate (as a concentrate solution) and dilute the whole solution to 200 ml.
- Heat the solution almost to the boiling point (do not boil) and add lead acetate solution drop wise (either from burette or a graduated pipette) with stirring, occasionally allowing the hot solution to settle to observe the supernatant liquid. The temperature should be maintained near the boiling point throughout the addition of lead acetate & when adequate quantity of lead acetate solution has been added then the milky white supernatant becomes appreciable clear. At this point gently boil for 2 minutes, and then allow it to settle. Add a few drops of the lead acetate solution to the supernatant to confirm for complete precipitation. Avoid excess of the precipitant. Now digest on a hot plate (or water bath) without boiling, and then allow to settle for 1 hour.
- Filter by decantation through whatman filter paper No.42. Wash 3 times by decantation with 75 ml hot solution of 2% ammonium nitrate solution. twice with cold water to remove ammonium nitrate.
- Burn the filter paper in a previously weighed silica crucible, first on a low temperature then to the full heat. Cool the crucible & weigh it.

8.10.6 Calculation:

$$\begin{aligned} \text{MoO}_3 \text{ Content} &= \frac{(W_2 - W_1)}{W} \times 39.23 \\ (\% \text{age by weight}) & \end{aligned}$$

W = Weight of sample taken
W₁ = Weight of crucible
W₂ = Weight of crucible + Pb MoO₄