①

M. MAHALAKSHMI
(Lec. in CHemistry)

PAPER - III

UNIT - III

ANALYSIS OF FINISHED PRODUCTS :-

* CHEMICAL ANALYSIS OF CEMENT :-

CEMENT :- Cement is a mixture of tricalcium silicate and Tri Calcium Aluminate and it also Contain Small amount of MgO & $CaSO_4$.

⇒ cement is the most widely used Construction material.

⇒ Quality depends on its Chemical Composition and Physical Properties.

⇒ Chemical Constituents of cement are Silica (SiO_2) alumina (Al_2O_3), Ferric oxide (Fe_2O_3), lime (CaO), Magnesia (MgO), Sulphur trioxide (SO_3), Alkali (Na_2O, K_2O). loss on ignition, Insoluble Residue.

⇒ cement analysis is essential for quality, Control, Standardization and Suitability.

⇒ Analysis ensures Compliance with Standards (IS, ASTM). → [American Society for Testing & materials]

40

→ Composition of cement are CaO - 60-67%, SiO_2 - 17, Al_2O_3 (3-8%), Fe_2O_3 (0.5-6%), MgO (0.5-4%), SO_3 (1-3%)

minor oxides are Na_2O, K_2O, TiO_2.

* **Contents to be analysed :-**

The contents to be analysed are

1) Silica (SiO_2) 2) Alumina (Al_2O_3) 3) Fe_2O_3 (Iron)

4) Lime (CaO) 5) MgO 6) SO_3 7) Loss on ignition

8) Insoluble residue 9) NH_4OH group 10) K_2O

11) Na_2O 12) S^{-2} sulphide sulphur.

* **Sampling :-** 3kgs of sample passing through Conveye belt from time to time during the manufacturing process collect all the particles and placed in sieve to ensure it is fine enough and finally it kept in a Air tight bottle.

* **Dissolutions :-**

1 gram of sample is heated with water & Con. HCl is added for dissolution. Heat the solution until efferescence is liberated.

* **Separation Techniques :-**

$$\underbrace{\text{Silica, Alumina, Iron, CaO, MgO}}_{\text{Same as iron ore (copy from that)}}$$

SO_3 :- The fiterate solution is added Con. Hcl, heat & cool and add Barium chloride ($BaCl_2$) is added drop wise and is given by the Barium sulphate precipitate is formed and is given by filter.

The ppt is washed with hotwater, ignited and cool & weigh as $BaSO_4$.

$$\text{Filterate} + \text{Con. Hcl} \xrightarrow{\text{heat}} \text{Cool} + BaCl_2 \text{(dropwise)} \longrightarrow$$

$$BaSO_4 \text{ ppt} \longrightarrow \text{Filter.}$$

$$\text{ppt} \xrightarrow[\text{with}]{\text{wash}} \text{hot water} \xrightarrow{\text{ignite}} \text{cool \& weigh as } BaSO_4.$$

$$\% \text{ of } SO_3 = \frac{\text{wt. of } BaSO_4 \times 0.3433 \times 100}{\text{wt. of sample}}$$

* **loss on ignition** :- Heat 1gm of the cement sample in a "Pt" crusible to full red heat 900-1000°c. Repeat the heating until a constant weight is obtained. difference between weight of the sample taken and after ignition weight is equal to loss on ignition.

* **Insoluble residue** :- Take one gram of sample is taken in a beaker and is added the Con. Hcl & heat it then until the effervescence is cool dilute with the distilled water is digest on

10) K_2O
11) P

steam bath is given the Filter.

The insoluble residue is dried cool weigh.

[1gm of sample (beaker) + con. Hcl $\xrightarrow{\Delta}$ until effervescen

$\longrightarrow$ Cool $\xrightarrow[\text{with}]{\text{dilute}}$ distilled water $\xrightarrow[\text{steam bath}]{\text{digest on}}$ filter,

insoluble residue $\xrightarrow{\text{dried}}$ cool & weigh.]

$$\% \text{ of insoluble residue} = \frac{\text{wt. of insoluble residue}}{\text{wt. of sample}} \times 100$$

Composition of cement :-

Silica	21.16%
Al_2O_3	5.65%
Fe_2O_3	2.23%
CaO	63.36%
MgO	4.2%
SO_3	1.61%
loss on ignition	0.97%
Insoluble Residue	0.09%

Contents to be determined :-

1) Silica
2) NH_4OH group
3) Al_2O_3
4) Fe_2O_3
5) CaO
6) MgO
7) SO_3
8) loss on ignition
9) Na_2O

10) K_2O

11) P_2O_5

12) S

Determination of silica [SiO_2]

Take 0.5g of sample in a platinum dish then the sample is moisten with 10ml of cold water to prevent bumping add 5-10ml HCl and digest under gentle heat then evaporate the solution to dryness on a steam bath. Dried mass is dissolved in [1:1] HCl and see that the material dissolved (or) not then cover with lid and digest for 10-12 min. Add 15ml of hot water and immediately filter and wash the separated SiO_2 thoroughly with hot water and reserve the residue.

Again evaporate the filtrate to dryness and break the residue in an air oven at 105-110°c for 1hour, cool and add 10-15ml [1:1] HCl then heat on the bath. It is dilute with water and filters immediately on a fresh filter paper, wash the ppt with hot water then reserve the filtrate and washings for the determination of NH_4OH.

Transfer total residue along with filter paper into a platinum crucible. It is ignited at 1100-1200°c until constant weight is obtain

Then SiO_2 obtained is treated with 2-3 d of water, 2 drops of H_2SO_4 followed by 10ml HF and evaporate to dryness. Dried mass is ignited at 1100°c for 3-4min it is cooled in a desiccator and weighed the substance. The loss in weight gives the weight of SiO_2.

$$\% \text{ of } SiO_2 = \frac{\text{Loss in wt} \times 100}{\text{wt of sample}}$$

Determination of NH_4OH group :-

The Filtrate obtained after the determination of silica is taken and add 10-15ml of Hcl if necessary. Add few drops of methylorange indicator and heat to boiling. Then treat with ammonium hydroxide [1:1] until the colour of the solution becomes distinctly yellow and add one drop in excess. Heat the solution containing the precipitate to boiling and boil for 50-60 seconds. Allow the precipitate to settle and filter. wash the precipitate with hot ammonium chloride. The Filtrate is used for the determination of remaining contents. The precipitate along with filterpaper is taken in a pre-weighed platinum crucible heat slowly until ter the Papers are charred and finally ignite at at 1050-1100°c until Constant weight is obtained. It is Coded and weighed as ammonium hydroxide group.

3g of Potassium pyro sulphate (or) Sodium Pyrophosphate is added to the crucible it is heated until the residue is dissolved in the melt. cool, dissolve the fused mass in water containing 2.5ml of H_2SO_4 and evaporate to dryness. ~~finally heat the crucible~~ ~~at~~ The residue is again extracted with water and digested for 30min. it is filtered and washed with hot water. Transfer the Paper contain the residue into a Platinum crucible. Dry and ignited the Paper first at low until the carbon of the Paper is completely Consumed without inflaming, and finally at 1100-1200°c until the Constant weight is obtained.

The residue is treated with a drop of water about 5ml of HF and a drop of H_2SO_4 and evaporates to dryness. Finally heat the crucible at 1050-1100°c for 1-2 min Cool and weigh. The difference between this weight and the weight previously obtained represents the amount of residual SiO_2. Substract this amount from the obtained amount of ammonium hydroxide group, the amount of SiO is added to the amount obtained in the determ of silica.

Determination of Fe_2O_3:-

Required Solutions:

1) **Stannous Chloride Solution:** 5g of $SnCl_2$ dissolved in 10ml Con. HCl and make up to 100ml with distilled water.

2) **Barium diphenyl amine sulfonate indicator:-** dissolve 0.3g of Barium Diphenyl amine Sulfonate in 100ml water.

3) **Standard Potassium dichromate:-** [1ml of $K_2Cr_2O_7$ = 0.004g of Fe_2O_3]: Dissolve 2.457g of Potassium dichromate in minimum amount of water and dilute to 1 liter.

Procedure:-

Take 1g of sample and add 40ml of cold water and the mixture is stirred vigorously by adding 10ml of Con. HCl. If necessary, heat the solution and grind the cement with glass rod until is evident that the cement is completely decomposed, then boil the solution and treat it with the $SnCl_2$ Solution, added drop by drop while stirring until the solution is decolorized. Add one drop in excess and cool the solution to room temperature. Then the excess of $SnCl_2$ is removed by adding 10ml of saturated $HgCl_2$ solution. Now add 10ml of [1:1] H_3PO_4 solution and two drops of diphenyl amine sulfonate indicator then dilute to 100ml ~~of~~ with distilled water. Then the solution is titrated with potassium di-chromate solution intense purple coloration is obtained it is the endpoint.

$$\% \text{ of } Fe_2O_3 = E \times V \times 100$$

where,

E = Fe_2O_3 equivalent of the potassium dichromate solution in grams per millimeter.

V = volume of potassium dichromate required in titration.

Determination of Al_2O_3 :-

The Fiterate obtained after determination of NH_4OH add ammonium chloride $\{NH_4Cl\}$ and ammonium hydroxide $[NH_4OH]$. Then Al and Fe are precipitated. Then Al_2O_3 is calculated by substracting the weight of Fe_2O_3 from total weight.

Determination of calcium oxide: [CaO]

The Filterate obtained from ammonium hydroxide is used. It is acidified with Con. Hcl till the solution is clear. Evaporate to 100ml then add 40ml of saturated bromine water then MnO_2 present in the sample is oxidized. To this Add ammonium hydroxide and boil the solution for 5min to become Alkaline.

Allow the precipitate to settle, filter and wash with hot water. Discard any manganese dioxide that may have been precipitated. Acidify the filterate with Hcl and boil until all the bromine is expelled. Add 5ml of Hcl diluted to 200ml with distilled water and add few drops of methyl red indicator and 30ml of warm ammonium oxalate solution. Heat the solution at 70-80°c and add ammonium hydroxide drop wise while stirring until the colour changes from red to yellow. Allow the solution to stand without further heating for 1hour with occasional stirring during the first 30mins. Filter and wash moderately with cold ammonium oxalate solution. Reserve the Filterate and washings.

Transfer the precipitate into a "Pt" crucible and ignite the mixture at 1100-1200°c until constant weight is obtained.

$$\% \text{ of CaO} = \frac{\text{wt of Precipitate} \times GF \times 100}{\text{wt. of sample}}$$

Determination of Magnesium oxide:

Reagents: Ammonium Nitrate wash solution: Dissolve 100g of Ammonium Nitrate in water add 200ml of ammonium hydroxide and diluted to 1 liter with distilled water.

Procedure:- The Filtrate obtained after determination of CaO is used for determination of MgO. then acidify the filtrate with HCl to this solution add 10ml of ammonium hydrogen phosphate $[(NH_4)_2HPO_4]$ and cool the solution by placing in a beaker of ice water; After cooling add ammonium hydroxide drop by drop while stirring constantly until the crystalline magnesium ammonium phosphate begins to form precipitate. Then keep the solution a side for atleast 8hours in a cool atmosphere, and then filter the precipitate. It is ignited in Pt crucible at 1100-1200°c until constant weight is obtained.

% of MgO = wt of $Mg_2P_2O_7$ × 72.4

72.4 = Molecular ratio of MgO to $Mg_2P_2O_7$ by wt
the sample used and multiplied by 100

$$\% \text{ of MgO} = \frac{\text{wt of Precipitate} \times GF \times 100}{\text{wt. of sample}}$$

Determination of Sulfur trioxide :-

Take 1g of sample and add 25 ml of cold water the mixture is stirred vigorously by adding 5ml of HCl. heat the solution if necessary then filter the solution and wash the residue thoroughly with hot water, dilute the solution to 200ml with distilled water and heat to boiling. Add slowly drop by drop from a pipette 10ml of hot $BaCl_2$ solution with continues stirring then precipitate is formed, It is filtered and washed with water and ignited in Pt crucible at 800-900°C and weighed as barium sulphate.

$$\% \text{ of } SO_3 = \frac{\text{wt of Precipitate} \times GF \times 100}{\text{wt of sample}}$$

GF = 0.3430

Determination of Sodium and Potassium oxide:

Sodium oxide and Potassium oxide in cement sample are determined by Flame Photometry.

Required Solutions:-

1) Stock solutions: 1.8858g of Nacl and 1.5830g of KCl Previously dried at 105-103°c for several hours are dissolved in water and diluted to 1liter in a volumetric flask and mixed thoroughly this solution Contains 1000 PPm each of sodium oxide and Potassium oxide.

2) Standard solutions: 10-100PPm standard solutions are Prepared by diluting the stock solution.

Calibration Curve:-

The Flame intensities of Standard solution are measured and then a calibration curve is constructed by Plotting flame intensity against the Concentration of Standard solutions.

Procedure:-

1g of sample is taken in a 250ml beaker to this add 10-25ml of water and 5ml of Hcl are added. It is diluted to 50ml and digested on a hot Plate for 15min it is filtered into a 100ml volumetric flask and make up to mark with distilled water.

The sample is introduced into the flame. wave length selector is set at the maximum response of Sodium oxide then the meter readi is measured. Then the concentration of Sodiu oxide is calculated from the calibration curve. similarly the wave length selector is set at the max response of Potassium oxide from the meter reading the concentration of Potassium oxide is calculated.

- sulphide → same as in steel analysis
- Loss on ignition → same as in Iron ore analysis.

Analysis of Soaps:-

The sodium (or) Potassium salts of higher fatty acids are called soaps.

⇒ Sodium salts are called hard soaps and Potassium salts are called soft soaps.

⇒ A hard soap is moderately soluble in water and does not give lather easily. soft soap dissolved fast and gives lather readily.

⇒ The soaps are soluble in water and the solution has excellent lathering and cleaning Properties. The basic Principle of making

* All kinds of soaps such as toilet, laundry, household, medicinal and industrial soaps is the same i..e the combination of higher fatty acids with sodium (or) Potassium hydroxide (or) Carbonate. Shaving creams and liquids soap are soft soaps.

The oil (or) fat is first hydrolyzed into glycerol and fattyacids then the fatty acids are Neutralizing by adding alkali to form soap. This Procedure is quicker, better controlled and makes the recovery of byproducts glycerol easier. The oils used for the manufacture of soap are usually coconut oil cotton seed oils, Palm oil and castrol oil.

SAmpling :-

Care should be taken in the sampling of soaps because the moisture content at the outer surface different from inner surface during the course of preparation.

If the soap is too soft cut the cake diagnally into 2 pieces, take the showing from fresh surface. If the sample is dry by running through meat cheaper from sample reduce into fine particles, sample should be mix kept it an air tight bottle.

CONTENTS TO BE DETERMINED:-

1) Determination of Moisture and volatile Matter
2) Determination of total alkali
3) Determination of Total Fatty Matter
4) Determination of Free Caustic Alkali (or) Free Fatty acid
5) Determination of Sodium Silicates.
6) Determination of Chloride
7) Determination of total unsaponified matter
8) Determination of saponified matter
9) Determination of unsaponified and saponified Matter

Determination of Moisture and volatile Matter:

weight out 20g of sample and dissolved in 100ml of hot water transfer the solution into a 250ml volumetric Flask and make up to mark. Pipette out 25ml of the solution into a weighed Platinum Crucible & evaporated to dryness in a steam bath then dries at 105°c until Constant weight is obtained. The weight of solid material remained in crucible is used to determine the amount of moisture and organic matter.

Same as in iron ore analysis.

Determination of Total Alkali:-

Dissolve 5g of the soap in hot water add 40ml of 0.5N HCl heated on a steam bath until the fattyacids have collected in a clear layer. cool the beaker in cold water. Break the cake of fatty acids and filter the Aqueous liquid leaving the Fatty acid in beaker. wash the beaker thoroughly with water along with fatty acid, and then combine the Filterate and washings. Then add methyl orange indicator and titrate the excess of acid with 0.1N NaOH calculate the amount of 0.5N acid consumed in terms of sodium oxide and Potassium oxide.

$$\% \text{ of } Na_2O = \frac{\text{Titrate value in ml} \times N \text{ of acid} \times 0.031 \times 100}{\text{weight of sample}}$$

$$\% \text{ of } K_2O = \frac{\text{Titrate value in ml} \times N \text{ of acid} \times 0.02855 \times 100}{\text{weight of sample}}$$

Determination of Total Fatty Matter : [TFM]

Take 200ml of the original hot soap solution into a beaker to this add dil. HNO_3 until the fatty acids are collected in a clean layer on a hot and the solution below is perfectly clear. Cooled in ice water remove the layer of fatty acid into another beaker shake the remaining liquid with $CHCl_3$, CCl_4 in a seperating funnel to remove the rest of the fatty matter. Transfer the $CHCl_3$ extract into the beaker containing the Fatty cake, evaporate the $CHCl_3$ dried at 108c and weighed as total Fatty matter.

$$\% \text{ of Total Fatty acid} = \frac{\text{wt of Fatty acid} \times 100}{\text{wt. of sample}}$$

Determination of Free caustic Alkali (or) Free Fatty acids;

Dissolve 5g of soap in hot 95% alcohol which is Neutralized with NaoH. Filer useing a hot water Funnels wash free from the soap with Neutral alcohol. Then titrate the Filterate with 0.1N acid useing Phenolpthalein indicator and calculate the excess of NaoH (or) KoH

$$\% \text{ of } Na_2O = \frac{\text{Titrate value in ml X N of acid X 0.031X100}}{\text{Weight of sample}}$$

$$\% \text{ of } K_2O = \frac{\text{Titrate value in ml X N of acid X 0.02855X100}}{\text{Weight of sample}}$$

3.2.2.3 *Determination of Total Fatty Matter: [TFM]*

Take 200ml of the original hot soap solution into a beaker to this add dilHNO_3 until the fatty acids are collected in a clean layer on a hot and the solution below is perfectly clear. Cooled in ice water remove the layer of fatty acid into another beaker shake the remaining liquid with $CHCl_3$, CCl_4 in a separating funnel to remove the rest of the fatty matter. Transfer the $CHCl_3$ extract into the beaker containing the fatty cake, evaporate the $CHCl_3$ dried at 100°C and weighed as total fatty matter.

$$\% \text{ of Total Fatty Acid} = \frac{\text{Wt of Fatty Acid X 100}}{\text{Wt of Sample}}$$

3.2.2.4 *Determination of Free Caustic Alkali or Free Fatty Acids:*

Dissolve 5g of soap in hot 95% alcohol which is neutralized with NaOH. Filer using a hot water funnels wash free from the soap with neutral alcohol. Then titrate the filtrate with 0.1N acid using phenolphthalein indicator and calculate the excess of NaOH or KOH

If the filtrate is acid then titrate with 0.1N NaOH and calculate the acid.

$$\% \text{ of Free Caustic Acid} = \frac{\text{Titrate Value in ml X N Of Acid X } 0.04 \text{ X } 100}{\text{Wt Of the Sample}}$$

$$\% \text{ Of Free Alkali or NaOH} = \frac{\text{Titrate Value in ml X N of Alkali X } 0.0282 \text{ X } 100}{\text{wt of the sample}}$$

$$\% \text{ of Free Acid} = \frac{\text{Titrate Value in ml X N of Alkali X } 0.0282 \text{ X } 100}{\text{Wt of the Sample}}$$

3.2.2.5 *Determination of Sodium Silicate:*

The precipitate obtained after the determination of volatile matter and moisture is used for determination of silica. The residue is dissolved in water and titrates with 0.1N HCl if the residue is not completely soluble in water the insoluble matter should be filtered off and the filtrate is evaporated to dryness ignited and weighed before titration.

Then the precipitate is fused with sodium carbonate or potassium carbonate the dissolved the precipitate in a hot water and HCl then filter and wash ignite very strongly and weigh as SiO_2, then calculate as $Na_2Si_4O_9$.

3.2.2.6 *Determination of Chloride:*

Dissolve 5g of the sample in 300ml of water boil to get all soluble matter into solution and an excess of neutral chlorine free magnesium nitrate solution about 25ml of 20% magnesium nitrite $[Mg(NO_3)_2]$ solution is added. Titrate the above solution with 0.1N $AgNO_3$ solution using potassium chromate as indicator.

3.3 Analysis of Oils

Oils and fats are esters of higher fatty acids with glycerol. The esters of glycerol are commonly known as "Glycerides". Fatty acids are both saturated and unsaturated.

Examples: Saturated Acids:

1. Myristic Acid	$C_{13}H_{27}COOH$
2. Palmitic Acid	$C_{15}H_{31}COOH$
3. Stearic Acid	$C_{17}H_{35}COOH$

Unsaturated Acids:

1. Oleic Acid	$C_{17}H_{33}COOH$
2. Linoleic Acid	$C_{17}H_{31}COOH$

Out of these, those which are solids at ordinary temperature are called fats and those are in liquids are known as oils.

The oils and fats are used in food, manufacture of soaps and medicines etc.,

Oils are classified into edible oils and inedible oils on the basis of carbon chain vegetable oils are also classified as non drying oils, semidrying oils and drying oils.

The important properties of oils are

1. Saponification Number
2. Iodine Number
3. Acid Number

3.3.1 Saponification Number:

It is the measure of the amount of saponifiable matter present in the sample. It indicates the average molecular weight of fat or oil.

It may be defined as the No. of milligrams of KOH required to neutralize the fatty acids obtained by compete hydrolysis of 1g of oil.

Or

Saponification value is milligrams of KOH required to hydrolysis 1g of the oil.

Reagents:

1. **0.5N Alcoholic KOH Solution:** 28g of KOH is dissolved in 500ml of alcohol and warmed the mixture is constantly stirred. The solution is allowed to stand overnight and then filtered. The filtrate is diluted to 1liter this will give approximately 0.5N KOH in alcoholic solution
2. **Phenapthalen Indicator:** 1% indicator solution is prepared by dissolving 1g of Phenapthalen in 100ml of 1:1 water alcohol mixture.
3. **0.5N HCl Solution:**

Procedure:

Take 1 or 2g of oil is taken in a round bottom flask. It is fitted with a reflux condenser and adds 50ml of 0.5N Alcoholic KOH is added. The flask is heated on a water bath for 1hour heating is continued until reaction is completed and liquid becomes quite clear. The solution is titrated with 0.5N HCl using phenolphthalein as indicator. The same procedure is repeated with blank i.e. with out taking oil or fat.

$$Saponification\ Value = \frac{[V_2 - V_1]\ 56\ X\ Normality\ of\ Acid}{Wt\ of\ the\ Sample}$$

V_1 = Volume of HCl required for sample in ml

V_2 = Volume of HCl required for Blank in ml

100ml of 1N HCl = 1000ml of 1N KOH = 56g of KOH

$$Average\ Molecular\ Weight\ of\ oil\ or\ fat\ = \frac{3\ X\ 56\ X\ 1000}{Saponification\ Value}$$

Oil	Saponification Value
Coconut Oil	245-270
Pam Oil	195-205
Cotton Seed Oil	190-197
Olive Oil	185-200

13

3.3.2 Iodine Value:

Iodine value indicates the degree of unsaturation of the constituent fatty acids in an oil or fat.

It may be defined as the no. of grams of iodine absorbed by 100g of oil or fat

It iodine value is determined by "Weiss Method" each oil have different iodine value.

Drying oils > 150

Semidrying oils = 150

Non drying oils < 150

Reagents

Weiss Reagent:

It is prepared by dissolving 8.5g of iodine and 7.5g of iodine mono chloride in warm glacial acetic acid and make up to 1liter by cold glacial acetic acid.

Procedure:

Weigh accurately 0.1g - 0.5g of the sample in a glass stopper flask. Dissolve the sample in 10ml of CCl_4 or $CHCl_3$. Warm slightly if necessary cool the solution take same volume of chloroform or CCl_4 in another flask as blank now add 25ml of the Weiss solution into the both flasks. Shake each flask very vigorously allow them to stand in dark for about half an hour. Now the solutions in each flask are diluted to 50-100ml with distilled water. Then add 15ml of 10% KI solution in each flask. Titrate the contents with standard 0.1N hypo using starch as indicator until the color changes from blue to color less.

$$Iodine\ Value = \frac{[V_2 - V_1]\ 12.692\ X\ Normality\ of\ Hypo}{wt\ of\ sample}$$

V_2 = Volume of Hypo Required from sample

V_1 = Volume of Hypo Required from Blank.

Oil	Iodine Number
Caster Oil	80-90
Olin Oil	75-95
Quenol Oil	175-185

17

3.3.3 Acid Numbor:

The acid value or acid number gives the amount of free fatty acid present in the sample it is determined by titration with standard KOH

It is defined as no. of milligrams of KOH required to neutralize the free fatty acid in one gram of oil sample.

Reagents

1. 0.1N Aq KOH
2. Phenapthalen

Procedure:

20g of the oil sample is taken into a flask to this add 50ml of 95% of alcoholic KOH and add Phenapthalen indicator it is heated to boil and cooled then the above solution is titrated with 0.1N KOH until pink color appears.

$$Acid\ Number = \frac{ml\ of\ 0.1N\ KOH\ X\ 5.61}{wt\ of\ the\ sample}$$

Oil	Acid Number
Coconut oil	2.25-10
Pam Oil	9-10
Caster Oil	0.1-0.3
Sunflower Oil	10-11

3.4 Analysis of Paints:

3.4.1 Introduction:

Paints are stable mechanical homogenous mixture of pigments. Pigments are insoluble inorganic and organic substances. Pigments are generally used to impact the color and protect the film from penetration.

The paints and pigments under analysis taken into the form by extender or vehicle the pain to be applies by brushing, dipping, spraying and roller casting.

The best suggestible oil used in the preparation of paints is lawns oil. Thinner are applied to maintain uniformly on the film coating. Thinners reduce the viscosity of blend, driers also be applied.

3.4.2 Classification of Paints:

1. Exterior House Paints
2. Interior Wall Paints
3. Fire Resistant Paints
4. Heat Resistant Paints
5. Chemical Resistant Paints
6. Luminous Paints
7. Emulsion Paints
8. Latex Paints
9. Cement Paints

3.4.2.1 Pigments:

Pigments are widely used in surface coating and also used in color impacting to ink, plastic, rubber glass vessels and ceramics.

Pigments are classified into different types basing on their applications.

1. White Pigments
2. Blue pigments: Ultramarine blue, Cobalt Blue and Iron Blue
3. Red Pigments: Red Lead, Red Cadmium, Iron Oxides.
4. Green Pigments: Chromium Oxide, Thio Cyanine green
5. Yellow Pigment: Zinc Chromate
6. Orange Pigment: Cadmium orange
7. Brown Pigment: Burnt Ember, Burnt Sienna
8. Metallic Pigment: Cu Powder, Al Powder, Zn Dust
9. Natural Pigment: Generally obtained in the form of clay, bride, mercury
10. Synthetic Pigment: Obtained in the form of oxides sulphide and sulphates etc.

Extenders:

Extenders applied to increases the consistency and setting of paint

Film forming materials:

Vehicle or film forming material generally obtained from unsaturation portion of the oils.

Thinners:

To apply the paint on the surface initially it is added

Examples: Turpentine.

16

Skinning Agent:

Poly Hydroxy alcohols to be used as skinning agents it preservative the chlorophenol.

3.4.3 Analysis of Paints:

Paints mainly containing two different stages under the analysis

1. Vehicle Analysis
2. Paint Pigment Analysis

Individual procedures are adopted depending up on the amount of pigment. Separation must be done by using centrifuging method and continues extraction method.

Centrifuging Method:

Fill the centrifuging tube with mixed paints add equal volumes of the other mix thoroughly place the tube in the centrifuging apparatus swell of the clear separation. Repeat the process again and again for effective separation. Then dry the pigment at 105°C until constant weight is obtained.

Continuous Extraction Method:

Mix the paint sample thoroughly so that pigments do not settle down take the paint in beaker and find out its weight. Pout the paint carefully a soxhlet extraction again weighs the beaker and determine the weight of paint transferred into the extraction. Fill the extractor with either and continue the extraction until the ether extract is color less. Remove the pigment form the extractor and again extract with ether. Finally evaporate the ether from the extract dried at 110°C and weigh the pigment.

3.4.3.1 Determination of Barium Sulphate:

The residue is taken in the crucible with a little water and adds a few drops of dil H_2SO_4. Then fill the crucible with HF and evaporate on a hot plate until all SiO_2 is removed as SiF_4. Fuse the residue obtained with excess of $KHSO_4$ digest the fused mass is completely soluble in water $BaSO_4$

3.4.3.2. Total Lead:

The filtrates obtained in above process add ammonium hydroxide until a precipitate begins to form then add just enough HCl to re dissolve it. Dilute the solution to about 500ml then pass hydrogen sulphide and then heat the solution to boil. Then filter of the black lead sulphide precipitate, Wash the precipitate with water then dissolve in con HNO_3 containing a little bromine water filter any sulphur that is present boil off the bromine gas.

Method: 1

To the filtrate add a slight excess of CH_3COOH. Then add on excess of potassium dichromate boil until the precipitate is settled filter through a gouache crucible. Washed the precipitate with hot water dried at 100°C place the crucible in Pt Crucible, ignited gently cooled in desiccator and weighed as lead chromate

Method: 2

To the filterate obtained in the above process add 5ml of con H_2SO_4 evaporate to remove SO_3 cooled and add 100ml of water. Let it stand for 1hour until the precipitate of lead sulphate is settled clearly filtered through a weighed gouache crucible. Washed with dil H_2SO_4 dried at 100°C ignited until a pure white residue of $PbSO_4$ is obtained cooled in desiccator and weighed as lead sulphate report the total lead as lead sulphate

3.4.3.3 *Lead chromate:*

Boil the filterate obtained in the determination of total lead until the H_2S totally boiled off to the filterate add ammonium hydroxide to precipitate Iron, Al, Cr as hydroxides. Add sodium peroxide filter and wash the precipitate with hot water repeat, with ammonium hydroxide again add sodium peroxide filter and wash the precipitate with hot water. The filtrate obtained after the removal of Iron and Al is boiled until sodium peroxide is decomposed.

Gravimetric Method

To the filterate add a slight excess of lead acetate heated on a steam bath until the precipitate of lead chromate is settled. Filter on a weighed gouache crucible washed with hard water dried at 105°C and ignited cooled and weighed as lead chromate report the percentage of chromium as lead chromate.

Volumetric Method:

To the filterate obtained after the removal of iron and aluminum add H_2SO_4 to make acidic add known excess of Mohr's slat solution titrate with 0.1N potassium dichromate solution using $K_3[Fe(CN)_6]$ as an indicator until blue color disappearance from the amount of ferrous ammonium sulphate we can calculated the amount of lead chromate.