D.N.R.COLLEGE (AUTONOMOUS): BHIMAVARAM
DEPARTMENT OF PG CHEMISTRY

APPLIED ANALYSIS – I

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Unit – I

Analysis of Ores

Ore:-

Minerals are natural materials in which the metals and their compounds are found in earth. Ores are those minerals from which metal are extracted conveniently and profitably. These ores contain good percentage of metal.

Sample:-

A representative sample could be chemical substance in scientific studies that can be taken to a laboratory and tested to analyze the result of any particular chemical reaction.

Sampling:-

Sampling is the process of collection of small portion from the bulk material. This represents the entire characteristics of the bulk material.

General techniques of analysis of applied to complex materials:-

Sampling of solids:-

Method – I: - Coning and Quartering Method:-

The bulk sample arranged in a cone shape. Now we can remove the top of the cone, we get a flattened shape. Then it is divided into four parts, the alternative parts are taken in to the top of cone add and then repeating the process 3 (or) 4 times. Finally we get a representative sample parts. This is representing the entire characteristics of bulk materials.

Method -2:-Longpile Alternative Shoved Method:-

In this method, the bulk sample arranged in a longpile (or) Path. Then its cuts in to a several parts, opposite parts are again converted in to longpile and also then it cuts into several parts are taken to this add remaining parts of the first longpile. Then it is again converted in to longpile. In this process repeating the 3 (or) 4 times. Finally we get a representative sample.

General methods of dissolution of complex materials:-

Dissolution:-

Oxides Ore – Dissolved in HCL, H_2SO_4.

Sulphide Ores – React with HNO_3 to Produce Sulphate later it's dissolved in HCL.

Refractory materials and Fluxes – React with fusing mixture then to produce a fused mass.

Ex: - KNO_3+$NaNO_3$ (two equal parts)

Separation methods:-

Silicon dissolved in HCL and then to form a precipitation and then filter and then taken in to a residue.

Analysis of ores:-

1) Iron ore:-

To write the – 1) Sampling a) Coning and quartering

b) Longpile alternative shoved method

2) Dissolution

3) Separation

Iron ore following constituents are:-

* Determination of Moisture

*Determination of Loss of ignition

* Determination of Total iron

* Determination of Ferrous iron

* Determination of Ferric iron

* Determination of Alumina

* Determination of Silica

* Determination of Titanium

* Determination of Lime

* Determination of Magnesia

* Determination of Sulphur

* Determination of Phosphorous

* Determination of Manganese

* Determination of Alkalis

* Determination of Combined water

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a Platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessiccator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of ore sample is taken in to a Platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a dessicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$\text{Fe (III)} + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, Fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and Sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \times \text{weight of } Fe_2O3}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl amine indicator.

$$Fe^{+3} + Sn^{+2} \rightarrow Fe^{+2} + Sn^{+4}$$

$$Fe^{+2} + Cr^{+6} \rightarrow Fe^{+3} + Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 (or) 4mL of NH_4OH and then to get Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4\ (OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\%\ \text{Alumina} = \frac{0.4178 \text{ X weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Silica:-**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c for 15min. after then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\%\ \text{Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Titanium:-**

Principle:-

Titanium present in the ore sample and then it is determined. Initially Titanium is +4 states and then it is converted in to +3 state by adding the sufficient amount of Zn-Hg (Zinc Amalgam) by the zones method. After then it is treated with Fe (III) by using Ferro Thio Cyanide indicator.

$$Ti^{+4}\rightarrow Ti^{+3}$$

$$Ti^{+3}+Fe^{+3}\rightarrow Ti^{+4}+Fe^{+2}$$

Chemical required: - Sodium Pyrophosphate (fusion mixture) Na_2PO_7, HCL, H_2O, H_2SO_4, Zn-Hg, Ferric Ammonium Sulphate, Ferro Thio cyanide indicator.

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

The filtrate is obtained after then removal of Iron, Silica, and Chromium. After then it is treated with fusing mixture and then to form a fused mass. After then to form a filter and residue, it is dissolved in HCL, H_2SO_4, and H_2O. After then it is treated with Zn-Hg by zones method. We get a reducing solution. Finally it is titrated with Ferric Ammonium Sulphate by using Ferro Thio Cyanide as an indicator.

$$\% \text{ Titanium} = \frac{0.00799 \times \text{Volume of Ferric Ammonium Sulphate}}{\text{Total weight of the sample}} \times 100$$

***Determination of Lime:-**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the ore sample in this determined by Calcium. Calcium Oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \times \text{Weight of CaO}}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesia:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of Magnesia. Magnesium Di Ammonium Phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium Phosphate

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Take 5g of ore sample taken in to a Platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for another analysis. After then it is heated at 350^c and we get an $Mg_2P_2O_7$. Finally cooled in a dessicator and then dried and weighed as $Mg_2P_2O_7$.

$$\%\ \text{Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of the ore sample is taken in to a Platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

*** Determination of Sulphur:-**

Principle:-

In this determination of Sulphur then it is converted in to Sulphates by the adding of fusing mixture to take the ore sample. After then it is treated with HCL and $BaCl_2$ solution and then we get a $BaSO_4$ precipitate.

$$SO_4^{-2} + HCL + BaCl \rightarrow BaSO_4$$

Chemicals required: - Fusing mixture, HCL, $BaCl_2$

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Accurately weight 5g of ore sample taken in to a Platinum crucible. After then it is treated with fusing mixture. Then we get a fused mass. After then it is dissolved in water and we get a Sulphate. After then it is treated with HCL and $BaCl_2$ and then we get a $BaSO_4$.

$$\% \text{ Sulphur} = \frac{0.1374 \text{ X Weight of } BaSO_4}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Phosphorous:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample it is oxidized by addition of 3 (or) 4mL of Nitric acid solution for the sample and then added to the sufficient amount of AmmoniumMolybdate and then it is heated. Finally we get an Ammonium PhosphoMolybdate.

$$(NH_3)_4PO_412MoO_4 + \text{Excess } NaOH \rightarrow Na_2MoO_3 + Na_2HPO_4 + NH_3 + H_2O$$

Chemicals required: - HNO_3, AmmoniumMolybdate.

Apparatus: - Platinum crucible, Desicator.

Procedure:-

Accurately weighed 5 g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3+ AmmoniumMolybdate and then it is heated and then we get an Ammonium PhosphoMolybdate. Finally collated in a desicator and then it is dried and then weighed as Ammonium Phospho Molybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \text{ X Weight of Ammonium PhosphoMolybdate.}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and AmmoniumMolybdate.

After then it is heated and then we get Ammonium PhosphoMolybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCL by using a Phenolphthalein indicator.

$$\text{Untreated } NaOH + HCL \rightarrow NaCl + H_2O$$

Factor: - 1mL of 0.1N NaOH = 0.003088g of P_2O_5

*** Determination of Manganese:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

The filtrate is taken after removal of Al, Fe, and Cr. To this added sufficient amount of Bromine water and NH_4OH and then we get a hydrated Manganese Oxide precipitate and then finally it is ignited and we get a Mn_3O_4.

$Filtrate+Br_2+NH_4OH \rightarrow Mn_2O_2H_2O \rightarrow Mn_3O_4$

Chemicals required: - Bromine water, Ammonium Hydroxide

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Filtrate obtained after removal of Al, Cr, and Fe. After then to this added sufficient amount of Br_2+NH_4OH.and then we get a Hydrated MnO_2H_2O precipitate. Filter the precipitate and then used for the analysis. Residue is taken in to a Platinum crucible. After then it is heated at 50-72^{0}c and then we get a Mn_3O_4. Finally it is cooled in a dessicator and then dried and then finally weighed as Mn_3O_4.

$$\% \text{ Manganese} = \frac{\text{Weight of } Mn_3O_4 \times 0.7203}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Filtrate obtained after removal of Al, Fe, and Cr. After then it is heated with H_2SO_4 and then we get a $MnSO_4$. After then to this added few ml of HNO_3 and then to firm a clear solution, the clear solution is titrated with $KMnO_4$.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.001648g of Manganese

*** Determination of Alkalis :-(NaCL & KCL)**

In this determination there is no direct method we are using the filtrate obtained after removal of Si, Fe, Al, Ti, Ca, Mn, Mg, P, S and etc. then it is evaporated up to certain conditions. Finally we get a residue which is represented as the total weight of alkalis. After then to this added sufficient amount Platinum Chloride and then we get a Potassium Platinum chloride precipitate. Filter the precipitate. Residue is taken in to a Platinum crucible and it is heated and then cooled in a desicator. Finally dried and weighed as a Potassium Platinum Chloride.

$\% KCL = 3/2 \times 0.30712 \times K_2P+Cl_6$

% NaCL = Total Alkalis - % KCL

*** Determination of combined water:-**

Accurately weighed 2g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of two ends. One end is passing through the dry air and another end is free weight $CaCl_2$ tube. After then sample is heated at 300^{0}c at 30min.then water vapors are collated at $CaCl_2$ tube then increasing the tube weight.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

Determination of carbon in the blast furnace, flue dust and sinter:

Take one gram of sample ore, moistened with 10ml hot water. Add 0.7gms of NaCl, boiled the contents so that NaCl completely dissolves, add 50ml of boiling HCL. Agitate the contents of the beaker and digest along with gentle heating. Then it is warm, dilute with 60ml water. Heat about for 10 mins and immediately filter. All the Carbon particles retain on the filter paper. They are thoroughly washed with 5% HCL followed by washings with hot water to free from the acid. These Carbon particles are transfer to Silica crucible with the help of fine jet water without sprinkling outside. Dry the Carbon particles, so that the water accompanying them is exhausted. It is completely dried in air oven at 105^O C without adhering them to the walls of the crucible. Then it is cooled and weighed.

In order to get accurate value, ignite the crucible for about 10-15 mns at 500^0C and at 900^0C for about 30mins till gases are evolved. Carbon is reacted with O_2 to give CO and CO_2. Then it is cooled and weighed. The difference of the two weights will give weight of Carbon particles.

$$\% \text{ of Carbon} = \frac{\text{Weight of Carbon}}{\text{Weight of Sample}} X100$$

2) Manganese ore:-

To write the – 1) Sampling. a) Coning and Quartering

b) Longpile alternative shoved method

2) Dissolution

3) Separation

Manganese ore following constituents are:

* Determination of Moisture

*Determination of Loss of ignition

* Determination of Combined water

* Determination of Total Manganese

* Determination of MnO_2

* Determination of SiO_2

* Determination of BaO

* Determination of Fe_2O_3

* Determination of Al_2O_3

* Determination of CaO

* Determination of Phosphorous

* Determination of Sulphur

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a Platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} X 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of ore sample is taken in to a Platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a dessicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of combined water:-**

Accurately weighed 2g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of two ends. One end is passing through the dry air and another end is free weight $CaCl_2$ tube. After then sample is heated at 300^0c at 30min.then water vapors are collated at $CaCl_2$ tube then increasing the tube weight.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

***Determination of Total Manganese :-(MnO_2) Manganese oxide:-**

Principle:-

It is present in the Manganese ore sample and then it is determined. Then it is dissolved in H_2SO_4 an also treated with excess amount of Oxalic acid (or) Sodium Oxalate. The untreated Oxalic acid is treated with $KMnO_4$.

$$\text{Sample}+H_2SO_4+\text{excess } H_2C_2O_4 \rightarrow MnSO_4+2CO_2+2H_2O$$

$$5H_2C_2O_4+2KMNO_4+3H_2SO_4 \rightarrow K_2SO_4+2MnSO_4+8H_2O+10CO_2$$

Chemicals required: - 20% H_2SO_4, Sodium Oxalate (or) Oxalic acid, $KMnO_4$

Apparatus: - Platinum crucible, Dessicator, Volumetric flask, Conical flask

Procedure:-

Accurately weight sufficient amount of ore sample taken in to a beaker. After then to this added sufficient amount of 20% H_2SO_4. Later then it is making up to 250mL volumetric flask from the above solution taken in to a 10mL filtrate solution and then it is treated with excess amount of Sodium Oxalate (or) Oxalic Acid. The untreated Oxalic acid is titrated with $KMnO_4$ solution.

Factor: - 126g of Oxalic acid = 8.7g of MnO_2

136g of Oxalic acid = 8.7 g of MnO_2

*** Determination of Silica :-(SiO_2)**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c fir 15min. after then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of BaO:-**

Principle:-

BaO present in the ore sample and then it is determinate. Barium Sulphate precipitate is formed by adding the sufficient amount of 20% H_2SO_4 to the sample solution.

$$BaO + 20\%\ H_2SO_4 \rightarrow BaSO_4$$

Chemicals required: - 20% H_2SO_4

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate is obtained, after then removal of Silica. This is used for the BaO. To take the 10mL of filtrate in a beaker. Then it is dissolved in sufficient amount of 20% H_2SO_4 and then we get a $BaSO_4$ precipitate. Filter the precipitate. Residue is taken in to a Platinum crucible. Then it is cooled in a dessicator. Finally weighed as $BaSO_4$ precipitate.

$$\% \text{ BaO} = \frac{\text{Weight of } BaSO_4 \times 0.6570}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of $Fe\ (OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe\ (III) + NH_4OH \rightarrow Fe\ (OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, Fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a $Fe\ (OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

% Iron = $\frac{0.6944 \text{ X weight of } Fe_2O_3}{\text{Total weight of the sample}}$ X 100

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. Then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl Amine indicator.

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4\ (OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

% Alumina = $\frac{0.4178 \text{ X weight of } Al_2O_3}{\text{Total weight of the sample}}$ X 100

***Determination of Lime :-(CaO)**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the ore sample in this determined by Calcium. Calcium Oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Phosphorous:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample it is oxidized by addition of 3 (or) 4mL of Nitric acid solution for the sample and then added to the sufficient amount of AmmoniumMolybdate and then it is heated. Finally we get an Ammonium PhosphoMolybdate.

$$(NH_3)_4PO_412MoO_4 + \text{Excess NaOH} \rightarrow Na_2MoO_3 + Na_2HPO_4 + NH_3 + H_2O$$

Chemicals required: - HNO_3, AmmoniumMolybdate.

Apparatus: - Platinum crucible, Desicator

Procedure:-

Accurately weighed 5 g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3+ AmmoniumMolybdate and then it is heated and then we get an Ammonium PhosphoMolybdate. Finally collated in a desicator and then it is dried and then weighed as Ammonium Phospho Molybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \text{ X Weight of Ammonium PhosphoMolybdate.}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and AmmoniumMolybdate.

After then it is heated and then we get Ammonium PhosphoMolybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCL by using a Phenolphthalein indicator.

$$\text{Untreated NaOH} + HCL \rightarrow NaCl + H_2O$$

Factor: - 1mL of 0.1N NaOH = 0.003088g of P_2O_5

*** Determination of Sulphur:-**

Principle:-

In this determination of Sulphur then it is converted in to Sulphates by the adding of fusing mixture to take the ore sample. After then it is treated with HCL and $BaCl_2$ solution and then we get a $BaSO_4$ precipitate.

$$SO_4^{-2} + HCl + BaCl \rightarrow BaSO_4$$

Chemicals required: - Fusing mixture, HCL, $BaCl_2$

Apparatus: - Platinum crucible, Desicator

Procedure:-

Accurately weight 5g of ore sample taken in to a Platinum crucible. After then it is treated with fusing mixture. Then we get a fused mass. After then it is dissolved in water and we get a Sulphate. After then it is treated with HCL and $BaCl_2$ and then we get a $BaSO_4$.

$$\%\ \text{Sulphur} = \frac{0.1374 \times \text{Weight of } BaSO_4}{\text{Total weight of the sample}} \times 100$$

3) Chromite ore:-

To write the – 1) Sampling. a) Coning and Quartering
b) Longpile alternative shoved method
2) Dissolution
3) Separation

Chromite ore following constituents are:-

* Determination of Moisture

*Determination of Loss of ignition

* Determination of Combined water

* Determination of Chromium

* Determination of SiO_2

* Determination of FeO

* Determination of Al_2O_3

* Determination of CaO

* Determination of MgO

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of ore sample is taken in to a platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min.It is called loss of ignition. Then it is cooled in a dessicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of combined water:-**

Accurately weighed 2g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of two ends. One end is passing through the dry air and another end is free weight $CaCl_2$ tube. After then sample is heated at 300^0c at 30min.Then water vapors are collated at $CaCl_2$ tube then increasing the tube weight.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

*** Determination of FeO and Chromium:-**

Principle:-

Chromium and FeO present in a Chromium sample is determined. Chromium existence Ferro Chromium and then it is treated with Sodium Peroxide and to this added water and Barium Chloride. Finally we get a Barium Chromate and Ferrous oxide.

$$FeOCr_2O_3+Na_2O_2 \rightarrow NaFeO_2+Na_2CrO_4$$

$$Na_2FeO_2+H_2O \rightarrow Fe\ (OH)_3$$

$$Fe\ (OH)_3 \rightarrow FeO$$

$$Na_2CrO_4+BaCL_2 \rightarrow BaCrO_4$$

Chemicals required: - Sodium Peroxide, Barium Chloride

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weight sufficient amount of ore sample taken in to a Platinum crucible and then Chromium existence Ferro Chromium and then it is heated with Sodium Peroxide and then we get a $NaFeO_2$ and Na_2CrO_4. After then it is dissolved in suitable solvent like water and then we get a precipitate filter the precipitate and then residue is taken Ferric Hydroxide is present in the residue and then it is heated ,we get a Ferrous Oxide. After the filtrate is taken, Sodium Chromate present in filtrate and then to this added sufficient amount of Barium Chloride by maintain the pH 5-7.Finally we get a Barium Chromate. And then cooled in a dessicator and dried and weighed as Chromium.

$$\% \text{ chromium} = \frac{\text{Weight of } BaCrO_4 \times 0.2074}{\text{Total weight of the sample}} \times 100$$

*** Determination of Silica :-(SiO_2)**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c fir 15min. after then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. We get loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total Iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe (III) + NH_4OH \rightarrow Fe (OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \text{ X weight of } Fe_2O3}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl Amine indicator

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3(or) 4mL of NH_4OH and then to get Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 (or) $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4\ (OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \text{ Alumina} = \frac{0.4178 \text{ X weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Lime :-(CaO)**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

***Determination of Magnesia:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of Magnesia. Magnesium Di Ammonium Phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium Phosphate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Take 5g of ore sample taken in to a Platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for another analysis. After then it is heated at 350^c and we get an $Mg_2P_2O_7$ finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

4) Phosphate rock Ore:-

To write the – 1) Sampling. a) Coning and Quartering

b) Longpile alternative shoved method

2) Dissolution

3) Separation

Phosphate rock ore following constituents are:-

* Determination of Moisture

*Determination of Loss of ignition

* Determination of Combined water and CO_2

* Determination of SiO_2

* Determination of CaO

* Determination of Al_2O_3

* Determination of Fe_2O_3

* Determination of MgO

* Determination of MnO

* Determination of Sulphur

* Determination of Phosphorous

* Determination of Organic Carbon

* Determination of Tri Calcium Phosphate

* Determination of K_2O and Na_2O

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a Platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of ore sample is taken in to a Platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a dessicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Combined Water and CO_2:-**

Accurately weighed 5g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^0c.Then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

$$\% \text{ CO2} = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Silica :-(SiO_2)**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c fir 15min. after then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Lime :-(CaO)**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the ore sample in this determined by Calcium. Calcium Oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 (or) 4mL of NH_4OH and then to get Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3} + HNO_3 + NH_4OH \rightarrow Al\ (OH)_3 \rightarrow AlPO_4 \rightarrow Al_2O_3$$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4\ (OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \text{ Alumina} = \frac{0.4178 \text{ X weight of } Al_2O_3}{\text{Total weight of the sample X 100}}$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe (OH) $_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe(III) + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe (OH) $_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \text{ X weight of } Fe_2O3}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe (OH) $_3$ precipitate. After then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl Amine indicator.

$$Fe^{+3}+Sn^{+2} \rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6} \rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

***Determination of Magnesia:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of Magnesia. Magnesium Di Ammonium Phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium Phosphate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Take 5g of ore sample taken in to a Platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for another analysis. After then it is heated at 350^c and we get an $Mg_2P_2O_7$ finally cooled in a dessicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

*** Determination of Manganese:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

The filtrate is taken after removal of Al, Fe, and Cr. To this added sufficient amount of Bromine water and NH_4OH and then we get a Hydrated Manganese Oxide precipitate and then finally it is ignited and we get a Mn_3O_4

$$\text{Filtrate}+Br_2+NH_4OH \rightarrow Mn_2O_2H_2O \rightarrow Mn_3O_4$$

Chemicals required: - Bromine water, Ammonium hydroxide

Apparatus: - Platinum crucible, Dessicstor

Procedure:-

Filtrate obtained after removal of Al, Cr, and Fe. After then to this added sufficient amount of Br_2+NH_4OH.and then we get a hydrated MnO_2H_2O precipitate. Filter the precipitate and then used for the analysis. Residue is taken in to a Platinum crucible. After then it is heated at $50\text{-}72^0$c and then we get a Mn_3O_4. Finally it is cooled in a dessicator and then dried and then finally weighed as Mn_3O_4.

$$\% \text{ Manganese} = \frac{\text{Weight of } Mn_3O_4 \text{ X } 0.7203}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Filtrate obtained after removal of Al, Fe, and Cr. After then it is heated with $H_2SO_4$and then we get a $MnSO_4$.after then to this added few ml of HNO_3 and then to firm a clear solution, the clear solution is titrated with $KMnO_4$.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.001648g of Manganese

*** Determination of Sulphur:-**

Principle:-

In this determination of Sulphur then it is converted in to Sulphates by the adding of fusing mixture to take the ore sample. After then it is treated with HCL and $BaCl_2$ solution and then we get a $BaSO_4$ precipitate.

$$SO_4^{-2}+HCl+BaCl\rightarrow BaSO_4$$

Chemicals required: - Fusing mixture, HCL, $BaCl_2$

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weight 5g of ore sample taken in to a Platinum crucible. After then it is treated with fusing mixture. Then we get a fused mass. After then it is dissolved in water and we get a Sulphate. After then it is treated with HCL and $BaCl_2$ and then we get a $BaSO_4$.

$$\% \text{ Sulphur} = \frac{0.1374 \text{ X Weight of } BaSO_4}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Phosphorous:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample it is oxidized by addition of 3 (or) 4mL of Nitric acid solution for the sample and then added to the sufficient amount of AmmoniumMolybdate and then it is heated. Finally we get Ammonium PhosphoMolybdate.

$$(NH_3)_4PO_412MoO_4+\text{Excess } NaOH\rightarrow Na_2MoO_3+Na_2HPO_4+NH_3+H_2O$$

Chemicals required: - HNO_3, AmmoniumMolybdate.

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5 g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3+ AmmoniumMolybdate and then it is heated and then we get an Ammonium PhosphoMolybdate. Finally collated in a dessicator and then it is dried and then weighed as Ammonium PhosphoMolybdate.

% Phosphorous = 0.378 X Weight of Ammonium PhosphoMolybdate.
Total weight of the sample X 100

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and AmmoniumMolybdate.

After then it is heated and then we get Ammonium PhosphoMolybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCL by using a Phenolphthalein indicator.

$$\text{Untreated NaOH} + \text{HCL} \rightarrow NaCl + H_2O$$

Factor: - 1mL of 0.1N NaOH = 0.003088g of P_2O_5

*** Determination of Organic Carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of ore sample is taken into a Platinum crucible. After then it is heated at 100^0c. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

% Moisture = Loss of weight of the sample
Total weight of the sample X 100

Loss of ignition:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is heated at 1100^0c. Then it is cooled in a dessicator. Finally loss of weight is obtained.

% Loss of ignition = Loss of weight of the sample
Total weight of the sample X 100

Combined water and CO_2:-

Accurately weighed 5g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end is passing through an O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^0c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

% Combined water = Increasing the $CaCl_2$ tube weight
Total weight of the sample X 100

% CO_2 = Increasing the KOH tube weight
Total weight of the sample X 100

*** Determination of Tri Calcium Phosphate:-**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added 20mL of HNO_3 and then to this added sufficient amount of Ammonium Molybdate and then we get an Ammonium Phospho Molybdate precipitate. Filter the precipitate. And then residue is taken and then sufficient amount of to this added $MgCl_2$ and NH_4OH. We get an

$Mg(NH_4)_2PO_4$ precipitate. Filter the precipitate. Filtrate is extracted and the residue is taken. Then it is heated and we get an $Mg_2P_2O_7$.finally we cooled in a dessicator and dried.

$$\% \text{ Tri Calcium Phosphate} = \frac{\text{weight of } Mg_2P_2O_7 \times 1.3932}{\text{Total weight of the sample}} \times 100$$

*** Determination of K_2O and Na_2O:-**

$$\% K_2O = 3/2 \times K_2PtCl_6 \times 0.19411$$

$$\% Na_2O = \text{Total Alkalis} - \% K_2O$$

In this determination there is no direct method we are using the filtrate obtained after removal of Si, Fe, Al, Ti, Ca, Mn, Mg, P, S and etc. then it is evaporated up to certain conditions. Finally we get a residue which is represented as the total weight of alkalis. After then to this added sufficient amount Platinum Chloride and then we get a Potassium Platinum Chloride precipitate. Filter the precipitate. Residue is taken in to a Platinum crucible and it is heated and then cooled in a dessicator. Finally dried and weighed as a Potassium Platinum Chloride.

$$\% KCL = 3/2 \times 0.30712 \times K_2PCl_6$$

$$\% NaCL = \text{Total Alkalis} - \% \text{ Aalkali}$$

5) Aluminium Ore (Bauxite):-

To write the – 1) Sampling. a) Coning and Quartering

b) Longpile alternative shoved method

2) Dissolution

3) Separation

Aluminium ore following constituents are:

* Determination of Moisture

*Determination of Loss of ignition

* Determination of Combined water

* Determination of Silica

* Determination of Al_2O_3

* Determination of Fe_2O_3

* Determination of Titania

* Determination of Lime

* Determination of Magnesia

* Determination of Sulphur

* Determination of Phosphorous

* Determination of Manganese

* Determination of Vanadium

* Determination of Zirconium

* Determination of Alkalis

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a Platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of ore sample is taken in to a Platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a dessicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of combined water:-**

Accurately weighed 2g of ore sample is taken in to a Procaline crucible. Then it is placed in a heating chamber consists of two ends. One end is passing through the dry air and another end is free weight $CaCl_2$ tube. After then sample is heated at 300^0c at 30min.then water vapors are collated at $CaCl_2$ tube then increasing the tube weight.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Silica :-(SiO_2)**

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c fir 15min. after then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get $Al(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$Al^{+3}+HNO_3+NH_4OH \rightarrow Al(OH)_3 \rightarrow AlPO_4 \rightarrow Al_2O_3$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4(OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \text{ Alumina} = \frac{0.4178 \text{ X weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Total Iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$Fe(III) + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$

Chemicals required: - HCL, NH_4OH, H_2S, fusing mixture ($K_2CO_3+Na_2CO_3$, KNO_3+NaNO_3)

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and Sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \text{ X Weight of } Fe_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl amine indicator.

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

***Determination of Titanium:-**

Principle:-

Titanium present in the ore sample and then it is determined. Initially Titanium is +4 states and then it is converted in to +3 state by adding the sufficient amount of Zn-Hg (Zinc Amalgam) by the zones method. After then it is treated with Fe (III) by using Ferro Thio Cyanide indicator.

$$Ti^{+4}\rightarrow Ti^{+3}$$

$$Ti^{+3}+Fe^{+3}\rightarrow Ti^{+4}+Fe^{+2}$$

Chemical required: - Sodium Pyrophosphate (fusion mixture) Na_2PO_7, HCL, H_2O, H_2SO_4, Zn-Hg, Ferric Ammonium Sulphate, Ferro thio Cyanide indicator

Apparatus: - Platinum crucible, Desicator

Procedure:-

The filtrate is obtained after then removal of Iron, Silica, and Chromium. After then it is treated with fusing mixture and then to form a fused mass. After then to form a filter and residue, it is dissolved in HCL, H_2SO_4, and H_2O. After then it is treated with Zn-Hg by zones method. We get a reducing solution. Finally it is titrated with Ferric Ammonium Sulphate by using Ferro thio Cyanide as an indicator.

$$\% \text{ Titanium} = \frac{0.00799 \text{ X Volume of Ferric Ammonium Sulphate.}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Lime:-**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the ore sample in this determined by Calcium. Calcium Oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4\rightarrow CaC_2O_4$$

$$CaC_2O_4\rightarrow CaCO_3$$

$$CaCO_3\rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic Acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

***Determination of Magnesia:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of Magnesia. Magnesium Di Ammonium Phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium Phosphate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Take 5g of ore sample taken in to a Platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for another analysis. After then it is heated at 350^c and we get an $Mg_2P_2O_7$ finally cooled in a dessicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample}} \text{ X 100}$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

*** Determination of Sulphur:-**

Principle:-

In this determination of Sulphur then it is converted in to Sulphates by the adding of fusing mixture to take the ore sample. After then it is treated with HCL and $BaCl_2$ solution and then we get a $BaSO_4$ precipitate.

$$SO_4^{-2}+HCl+BaCl \rightarrow BaSO_4$$

Chemicals required: - Fusing mixture, HCL, $BaCl_2$

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weight 5g of ore sample taken in to a Platinum crucible. After then it is treated with fusing mixture. Then we get a fused mass. After then it is dissolved in water and we get a Sulphate. After then it is treated with HCL and $Bacl_2$ and then we get a $BaSO_4$.

$$\% \text{ Sulphur} = \frac{0.1374 \times \text{Weight of } BaSO_4}{\text{Total weight of the sample}} \times 100$$

*** Determination of Phosphorous:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample it is oxidized by addition of 3 (or) 4mL of Nitric acid solution for the sample and then added to the sufficient amount of AmmoniumMolybdate and then it is heated. Finally we get Ammonium PhosphoMolybdate.

$$(NH_3)_4PO_412MoO_4+\text{Excess } NaOH \rightarrow Na_2MoO_3+Na_2HPO_4+NH_3+H_2O$$

Chemicals required: - HNO_3, AmmoniumMolybdate.

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Accurately weighed 5 g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3+ AmmoniumMolybdate and then it is heated and then we get an Ammonium PhosphoMolybdate. Finally collated in a dessicator and then it is dried and then weighed as Ammonium PhosphoMolybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \times \text{Weight of Ammonium PhosphoMolybdate.}}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and AmmoniumMolybdate.

After then it is heated and then we get Ammonium PhosphoMolybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCL by using a Phenolphthalein indicator.

$$\text{Untreated NaOH} + HCL \rightarrow NaCl + H_2O$$

Factor: - 1mL of 0.1N NaOH = 0.003088g of P_2O_5

*** Determination of Manganese:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

The filtrate is taken after removal of Al, Fe, and Cr. To this added sufficient amount of Bromine water and NH_4OH and then we get a Hydrated Manganese Oxide precipitate and then finally it is ignited and we get a Mn_3O_4

$$\text{Filtrate} + Br_2 + NH_4OH \rightarrow Mn_2O_2H_2O \rightarrow Mn_3O_4$$

Chemicals required: - Bromine water, Ammonium hydroxide

Apparatus: - Platinum crucible, Desicstor

Procedure:-

Filtrate obtained after removal of Al, Cr, and Fe. After then to this added sufficient amount of Br_2+NH_4OH.and then we get a Hydrated MnO_2H_2O precipitate. Filter the precipitate and then used for the analysis. Residue is taken in to a Platinum crucible. After then it is heated at 50-72^{0}c and then we get a Mn_3O_4. Finally it is cooled in a dessicator and then dried and then finally weighed as Mn_3O_4.

$$\% \text{ Manganese} = \frac{\text{Weight of } Mn_3O_4 \text{ X } 0.7203}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Filtrate obtained after removal of Al, Fe, and Cr. After then it is heated with $H_2SO_4$and then we get a $MnSO_4$.After then to this added few ml of HNO_3 and then to firm a clear solution, the clear solution is titrated with $KMnO_4$.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.001648g of Manganese

*** Determination of vanadium:-**

Principle:-

In this determination of vanadium and then to this added excess of Fe(II) and then vanadium +5 to +4 state the untreated iron (II) is titrated with $KMnO_4$ solution.

$$V^{+5} + \text{Excess of Fe (II)} \rightarrow V^{+4} + \text{Fe (III)}$$

Untreated Fe (II) $+MN^{+7} \rightarrow$ Fe (III) $+Mn^{+2}$

Chemicals required: - H_2SO_4, Fe (II), $KMnO_4$

Apparatus: - 250mL Beaker, Conical flask

Procedure: - Accurately weighed 5g of ore sample is taken in to a 250mL beaker then it is dissolved in sufficient amount of H_2SO_4 from the above solution we taken to the 25mL of sample taken in to a250ml beaker then to this added excess of Fe (II) and then we get Vanadium. The untreated Fe (II) is titrated with $KMnO_4$. We get a Fe (III).

$$\% \text{ Vanadium} = \frac{(A \times B)\, C \times 0.005095}{\text{Total weight of the sample}} \times 100$$

Where,

A = Volume of $KMnO_4$ required for sample solution

B = Volume of $KMnO_4$ required for back titration

C = Concentration of $KMnO_4$ solution

*** Determination of Zirconium:-**

Principle:-

It is present in the ore sample in this determined. Zn $(SeO_3)_2$ precipitate is formed by adding the selenious acid to the sample solution in the presence of HCL solution.

Chemicals required: - HCL, (SeO_3H_2)

Apparatus: - Platinum crucible, Dessicator

Procedure: - Accurately weighed 5g of ore sample is taken in to Platinum crucible and then to this added selenious acid in the presence of HCL. If the sample solution containing impurities like Ti, Ce, and Th. These are removed by adding the H_2O_2 to the sample solution the impurities are converted in to +3 to +1 state. In this state these are no reacted so it can be easily removed. After then to this added sufficient amount of selenious acid in the presence of HCL and then we get a Zr $(SeO_3)_2$ precipitate and then Per Titanic Acid in a side form. Filter the precipitate and then residue is taken and then it is heated and then cooled in a dessicator and dried and weighed.

$$\% \text{ Zirconium} = \frac{\text{Weight of Zr } (SeO_3)_2 \times 0.2643}{\text{Total weight of the sample}} \times 100$$

***Determination of Alkalis :-(NaCL & KCL)**

In this determination there is no direct method we are using the filtrate obtained after removal of Si, Fe, Al, Ti, Ca, Mn, Mg, P, S and etc. Then it is evaporated up to certain conditions. Finally we get a residue which is represented as the total weight of alkalis. After then to this added sufficient amount Platinum Chloride and then we get a Potassium Platinum chloride precipitate. Filter the precipitate. Residue is taken in to a Platinum crucible and it is heated and then cooled in a dessicator. Finally dried and weighed as a Potassium Platinum Chloride.

% KCL = $3/2 \times 0.30712 \times K_2P{+}Cl_6$

% NaCL = Total Alkalis - % KCL

UNIT-II

ANALYSIS OF FINIFHED PRODUCTS – I

ANALYSIS OF STEEL:-

It contains random in the form of roads, beams, bearings etc. This is contaminated by oil and grease.

Dissolution:-

Steel dissolved by HNO_3 solution. After then it is stored in an amber colour bottle.

Steel contains the following constituents are:-

*Determination of Carbon

*Determination of Silica

*Determination of Sulphur

*Determination of Phosphorous

*Determination of Manganese

*Determination of Nickel

*Determination of Chromium

*Determination of Magnesium

***Determination of Carbon:-**

Accurately weighed 5g ore sample is taken into a procaline crucible. Then it is placed in to a heating chamber and it contains free weighed KOH tube and another end passing through the dry air. Then sample is heated at 110^0c. After then CO_2 is observes in a KOH tube. Then increase the KOH tube weight. Finally KOH tube weighed.

$$\% \text{ Carbon} = \frac{\text{Increase KOH tube Weight X 0.2727}}{\text{Total weight of the sample}} \text{ X 100}$$

***Determination of Silica:-**

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to form a fused mass. After then it is heated with 1:1 HCL at 500^0c for 15min. After then cooled in a dessicator. Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X 100}$$

***Determination of Sulphur:-**

Accurately weighed 5g of ore sample is taken in to a procaline crucible. It is dissolved in HNO_3 then to form sulphates. Then it is treated with $BaCl_2$ and HCL. Sulphates are converted in to $BaSO_4$ precipitate.

$$\% \text{ Sulphur} = \frac{0.1374 \text{ X Weight of BaSO4}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Phosphorous:-**

It is determined by two methods.

1) Gravimetric method:-

Principle:-

It is present in the ore sample and it is oxidized by addition of 3 or 4mL of Nitric acid solution for the sample and then added to the sufficient amount of Ammonium Molybdate and then it is heated. Finally we get Ammonium Phospho Molybdate.

$$(NH_3)_4PO_4 12MoO_4 + \text{Excess } NaOH \rightarrow Na_2MoO_3 + Na_2HPO_4 + NH_3 + H_2O$$

Chemicals required: - HNO_3, Ammonium Molybdate.

Apparatus: - Platinum crucible, dessicator.

Procedure:-

Accurately weighed 5 g of ore sample is taken in to a platinum crucible. After then it is treated with HNO_3+ Ammonium molybdate and then it is heated and then we get an Ammonium Phospho Molybdate. Finally collated in a dessicator. Then it is dried and then weighed as Ammonium Phospho Molybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \text{ X Weight of Ammonium Phospho Molybdate}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and Ammonium Molybdate.

After then it is heated and then we get an Ammonium Phospho Molybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCL by using a phenolphthalein indicator.

$$\text{Untreated } NaOH + HCL \rightarrow NaCl + H_2O$$

Factor: - 1mL of 1N NaOH = 0.003088g of P_2O_5

*** Determination of Manganese:-**

It can be determined by two methods.

1) Gravimetric method:-

Principle:-

The filtrate is taken after removal of Al, Fe, and Cr. To this added sufficient amount of Bromine water and NH_4OH and then we get a hydrated Manganese oxide precipitate and then finally it is ignited and we get a Mn_3O_4.

$$\text{Filtrate} + Br_2 + NH_4OH \rightarrow Mn_2O_2H_2O \rightarrow Mn_3O_4$$

Chemicals required: - Bromine water, Ammonium hydroxide

Apparatus: - Platinum crucible, dessicator.

Procedure:-

Filtrate obtained after removal of Al, Cr, and Fe. After then to this added sufficient amount of Br_2+NH_4OH and then we get a hydrated MnO_2H_2O precipitate. Filter the precipitate and then used for the analysis. Residue is taken in to a platinum crucible. After then it is heated at $50\text{-}72^0$c and then we get a Mn_3O_4. Finally it is cooled in a dessicator, then dried and then finally weighed as Mn_3O_4.

$$\% \text{ Manganese} = \frac{\text{Weight of } Mn_3O_4 \text{ X } 0.7203}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Filtrate obtained after removal of Al, Fe, and Cr. After then it is heated with $H_2SO_4$and then we get a $MnSO_4$.After then to this added few ml of HNO_3 and then to firm a clear solution, the clear solution is titrated with $KMnO_4$.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.001648g of Manganese

***Determination of Nickel:-**

Accurately weighed sufficient amount of ore sample is taken in to a platinum crucible. Then it is dissolved in sufficient amount of HNO_3. After then to this added sufficient amount of H_2SO4 and Dimethyl Glyoxime. We get a Ni-DMG complex in precipitate form. Filter the precipitate and filtrate the residue. Residue is taken and then it is heated in a platinum crucible. Cooled in a desicator. Finally dried and weighed.

$$\% \text{ Nickel} = \frac{\text{Weight of Ni-DMG Complex X } 0.7858}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Chromium:-**

Accurately weighed 1g of ore sample is taken in to a platinum crucible after then to this added 20mL of HNO_3 and H_2SO_4 solution. After then it is heated and also dilute with hot water. Filter the solution. Filtrate is taken and then it is diluted in 100mL water. From the above solution 20mL of filtrate is taken and then to this added sufficient amount of H_3PO_4 and excess amount of Fe (II) solution. The untreated Fe (II) is titrated with $KMNO_4$ solution.

Factor: - Fe (II) = Volume of $KMNO_4$ required for titration. Cr = Fe (II) factor X 1.3105

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of Magnesia. Magnesium Di Ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium phosphate

Apparatus: - Platinum crucible, dessicator.

Procedure:-

Accurately weighed 5g of ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get an $Mg_2P_2O_7$ finally cooled in a desicator. Then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ x } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for the analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

ANALYSIS OF BLAST FURNACE SLAG:-

Waste material removed from the metal which is following smelting or refining process.

Dissolution:-

Sufficient amount of sample is dissolved in conc. HCL and then it is treated with sufficient amount of fusing mixture. We get a fused mass. This is used for the analysis.

The following constituents are:

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Calcium

*Determination of Magnesium oxide

*Determination of Manganese oxide

*Determination of Phosphorous

*Determination of Sulphur

***Determination of Silica:-**

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCl solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCl at 500^0c for 15min. After then cooled in a desicator. Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$\text{Fe (III)} + NH_4OH \rightarrow \text{Fe}(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCl, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, desicator

Procedure:-

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCl. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a desicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \times \text{Weight of } Fe_2O3}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Diphenyl Amine indicator

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - platinum crucible, desicator.

Chemical required: - HCl, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCl solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get $AlPO_4$ and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\%\ \text{Alumina} = \frac{0.4178 \times \text{Weight of } Al_2O_3}{\text{Total weight of the sample}} \times 100$$

*** Determination of Calcium:-**

It is determined by two methods

1) Gravimetric method:-

Principle: -

Calcium present in the Blast furnace slag ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4\rightarrow CaC_2O_4$$

$$CaC_2O_4\rightarrow CaCO_3$$

$$CaCO_3\rightarrow CaO$$

Chemicals required: - di ammonium oxalate

Apparatus: - platinum crucible, desicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium or lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount di ammonium oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the iron ore sample in this determination of magnesia. Magnesium di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCl, di ammonium phosphate

Apparatus: - platinum crucible, desicator

Procedure:-

Take 5g of Blast furnace slag ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCl and $(NH_4)_2PO_4$ and then to form a Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 350c and we get Mg2P2O7 finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample}} \text{ X 100}$$

2) Volumetric method:-

Accurately weighed 5g of the Blast furnace slag ore sample is taken in to a platinum crucible and then to this added sufficient amount 20mL of HCl and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

***Determination of Manganese oxide:-**

Accurately weighed sufficient amount of the blast furnace slag sample is taken in to a 250mL beaker. To this added 25mL of HCl + H_2SO_4+ HNO_3. To this heated up to NO_3^- vapors are evaporated and then to this added 1% $AgNO_3$ and 25% Ammonium ferric sulphate and 75mL of water. This solution is titrated with sodium arsenate solution.

$$\% \text{ MnO} = \frac{\text{Weight of } Mn_3O_4 \text{ X Volume of sodium arsenate}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Phosphorous:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the Blast furnace slag ore sample it is oxidized by addition of 3 or 4mL of nitric acid solution for the sample and then added to the sufficient amount of Ammonium molybdate and then it is heated. Finally we get ammonium phospho molybdate.

$$(NH_3)_4PO_412MoO_4\text{+ excess } NaOH \rightarrow Na_2MoO_3+Na_2HPO_4+NH_3+H_2O$$

Chemicals required: - HNO_3, ammonium molybdate.

Apparatus: - platinum crucible, desicator.

Procedure:-

Accurately weighed 5 g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is treated with HNO_3+ ammonium molybdate. Then it is heated and then we get Ammonium phospho molybdate. Finally collated in a desicator and then it is dried and then weighed as Ammonium phospho molybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \text{ X Weight of ammonium Phospho molybdate}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and ammonium molybdate.

$$\text{Untreated } NaOH\text{+ } Hcl \rightarrow NaCl+H_2O$$

After then it is heated and then we get ammonium phospho molybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCl by using a phenapthaleine indicator.

Factor: - 1mL of 1N NaOH = 0.003088g of P_2O_5

*** Determination of Sulphur:-**

Principle:-

In this determination of sulphur then it is converted in to sulphates by the adding of fusing mixture to take the Blast furnace slag ore sample. After then it is treated with HCl and $BaCl_2$ solution and then we get a $BaSO_4$ precipitate.

$$SO_4^{-2}+HCl+BaCl\rightarrow BaSO_4$$

Chemicals required: - fusing mixture, Hcl, BaCl2

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weight 5g of Blast furnace slag ore sample taken in to a platinum crucible. After then it is treated with fusing mixture. Then we get a fused mass. After then it is dissolved in water and we get a sulphate. After then it is treated with HCl and $Bacl_2$ and then we get a $BaSO_4$.

$$\% \text{ Sulphur} = \frac{0.1374 \text{ X Weight of } BaSO_4}{\text{Total weight of the sample}} \text{ X } 100$$

ANALYSIS OF REFRACTORY MATERIALS:-

a) Fire clay:-

It is a refractory material, which is the combination of water and other impurities like silica, Fe, Al, Mn, Ca, Mg, alkalis (K_2O, Na_2O).

Sampling:-

In Ferro analysis components iron is present contamination metal which is removed by magnets and remaining parts are arranged in lumps. We are following the sampling of solvent methods.

Dissolution:-

Take sufficient amount of sample and then it is treated with fusing mixture and then produced fused mass. Then it is dissolved in dil. HCl and H_2O. Filter the solution. Filtrate is used for another analysis.

Fire clay following constituent are:

*Determination of Moisture

*Determination of Loss of ignition

*Determination of Combined water and CO_2

*Determination of Organic matter

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Manganese

*Determination of Calcium

*Determination of Magnesium

*Determination of Alkalis (K_2O, Na_2O)

* **Determination of Moisture:-**

Accurately weighed 5g of fire clay ore sample is taken into a platinum crucible. After then it is heated at 105^{0}c at 2 hours. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of fire clay ore sample is taken in to a platinum crucible. After then it is heated at 500^{0}c for 50min. after then also apply the sudden heat at 1000^{0}c at 30min. it is called loss of ignition. Then it is cooled in a desicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

* **Determination of combined water and CO_2:-**

Accurately weighed 5g of fire clay ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^{0}c.then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

$$\% \; CO_2 = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

* **Determination of Organic Carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of fire clay ore sample is taken into a platinum crucible. After then it is heated at 100^{0}c. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

Loss of ignition:-

Accurately weighed 5g of fire clay ore sample is taken in to a platinum crucible. After then it is heated at 1100^0c. Then it is cooled in a desicator. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Combined water and CO_2:-

Accurately weighed 5g of fire clay ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-11000c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

***Determination of Silica**:-

Accurately weighed 5g of fire clay ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCl solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCl at 500^0c for 15min. After then cooled in a desicator. Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$\text{Fe (III)} + NH_4OH \rightarrow \text{Fe}(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCl, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weighed 5g of fire clay ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCl. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a desicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \times \text{Weight of } Fe_2O3}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Accurately weighed 5g of fire clay ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a diphenyl amine indicator

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al(OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - platinum crucible, desicator.

Chemical required: - HCl, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent

Procedure:-

Accurately weighed 5g of Blast furnace slag ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCl solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get AlPO4 and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \text{ Alumina} = \frac{0.4178 \times \text{Weight of } Al_2O_3}{\text{Total weight of the sample}} \times 100$$

***Determination of Manganese:-**

Accurately weighed sufficient amount of the fire clay sample is taken in to a 250mL beaker. To this added 25mL of HCl + H_2SO_4+ HNO_3. To this heated up to NO_3^- vapors are evaporated and then to this added 1% $AgNO_3$ and 25% Ammonium ferric sulphate and 75mL of water. This solution is titrated with sodium arsenate solution.

$$\% \text{ MnO} = \frac{\text{Weight of } Mn_3O_4 \times \text{Volume of sodium arsenate}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Calcium:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

Calcium present in the fire clay ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - di ammonium oxalate

Apparatus: - platinum crucible, desicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\text{\% Calcium or lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X } 100}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount di ammonium oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of magnesia. Magnesium di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCl, di ammonium phosphate

Apparatus: - platinum crucible, desicator

Procedure:-

Take 5g of Blast furnace slag ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCl and $(NH_4)_2PO_4$ and then to form a Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get Mg2P2O7 finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ x } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of the Blast furnace slag ore sample is taken in to a platinum crucible and then to this added sufficient amount 20mL of HCl and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

*** Determination of K_2O and Na_2O:-**

$$\% K_2O = 3/2 \text{ X } K_2PtCl_6 \text{ X } 0.19411$$

$$\% Na_2O = \text{total alkalis} - \% K_2O$$

In this determination there is no direct method we are using the filtrate obtained after removal of Si, Fe, Al, Ti, Ca, Mn, Mg, P, S and etc. then it is evaporated up to certain conditions. Finally we get a residue which is represented as the total weight of alkalis. After then to this added sufficient amount Platinum chloride and then we get a potassium platinum chloride precipitate. Filter the precipitate. Residue is taken in to a platinum crucible and it is heated and then cooled in a desicator. Finally dried and weighed as a potassium platinum chloride.

$$\% KCl = 3/2 \text{ X } 0.30712 \text{ X } K_2PCl_6$$

$$\% NaCl = \text{total alkalis} - \% \text{ alkali}$$

b) Flour spar:-

It is a refractory material and industrial flour spar mainly contains CaF_2 along with impurities such as silica, iron, Al, P, Ti, Mn etc.

Flour spar contains following constituents:

*Determination of Moisture

*Determination of Loss of ignition

*Determination of Combined water and CO_2

*Determination of Organic matter

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Manganese

*Determination of Phosphorous

*Determination of CaF_2

*Determination of MgF_2

*Determination of Lime

*Determination of Magnesium

*Determination of Titanium

*** Determination of Moisture:-**

Accurately weighed 5g of flour spar ore sample is taken into a platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g of flour spar ore sample is taken in to a platinum crucible. After then it is heated at 500^0c for 50min. after then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a desicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of combined water and CO_2:-**

Accurately weighed 5g of flour spar ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through a O_2 and the another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^0c.then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

$$\% \; CO_2 = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \times 100$$

*** Determination of organic carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of flour spar ore sample is taken into a platinum crucible. After then it is heated at 100^0c. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Loss of ignition:-

Accurately weighed 5g of flour spar ore sample is taken in to a platinum crucible. After then it is heated at 1100^0c. Then it is cooled in a desicator. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Combined water and CO_2:-

Accurately weighed 5g of flour spar ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-11000c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

***Determination of Silica:-**

Accurately weighed 5g of flour spar ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCl solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCl at 500^0c for 15min. After then cooled in a desicator. Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of $Fe(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe(III) + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCl, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weighed 5g of flour spar ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCl. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a desicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \text{ X Weight of } Fe_2O3}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of flour spar ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a diphenyl amine indicator

$$Fe^{+3}+Sn^{+2} \rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6} \rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH \rightarrow Al\ (OH)_3 \rightarrow AlPO_4 \rightarrow Al_2O_3$$

Apparatus: - platinum crucible, desicator.

Chemical required: - HCl, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent

Procedure:-

Accurately weighed 5g of flour spar ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCl solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get AlPO4 and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \quad \text{Alumina} = \frac{0.4178 \text{ X Weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Manganese:-**

Accurately weighed sufficient amount of the flour spar sample is taken in to a 250mL beaker. To this added 25mL of HCl + H_2SO_4+ HNO_3. To this heated up to NO_3^- vapors are evaporated and then to this added 1% $AgNO_3$ and 25% Ammonium ferric sulphate and 75mL of water. This solution is titrated with sodium arsenate solution.

$$\% \text{ MnO} = \frac{\text{Weight of } Mn_3O_4 \text{ X Volume of sodium Arsenate}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Phosphorous**:-

It is determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the flour spar ore sample it is oxidized by addition of 3 or 4mL of nitric acid solution for the sample and then added to the sufficient amount of ammonium molybdate and then it is heated. Finally we get a ammonium phospho molybdate.

$$(NH_3)_4PO_412MoO_4\text{+excess } NaOH \rightarrow Na_2MoO_3+Na_2HPO_4+NH_3+H_2O$$

Chemicals required: - HNO_3, ammonium molybdate.

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weighed 5 g of flour spar ore sample is taken in to a platinum crucible. After then it is treated with HNO_3+ ammonium molybdate and then it is heated and then we get a ammonium phospho molybdate. Finally collated in a desicator and then it is dried and then weighed as ammonium phospho molybdate.

$$\% \text{ Phosphorous} = \frac{0.378 \text{ X Weight of ammonium Phospho mpolybdate}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of iron ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and ammonium molybdate.

After then it is heated and then we get ammonium phospho molybdate. After then to this added excess amount of NaOH solution. The untreated NaOH is titrated with HCl by using a phenapthaleine indicator.

$$\text{Untreated NaOH+ Hcl} \rightarrow NaCl+H_2O$$

Factor: - 1mL of 1N NaOH = 0.003088g of P_2O_5

*** Determination of Calcium fluoride: - (CaF_2):-**

It is determined by two methods

1) Gravimetric method:-

Principle: -

Calcium present in the flour spar ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - di ammonium oxalate

Apparatus: - platinum crucible, desicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium or lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X } 100}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount di ammonium oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

$$\% \text{ CaF2} = \% \text{ CaO X } 1.3923$$

*** Determination of Magnesium fluoride :- (MgF_2)**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the flour spar ore sample in this determination of magnesia. Magnesium di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCl, di ammonium phosphate

Apparatus: - platinum crucible, desicator

Procedure:-

Take 5g of flour spar ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCl and $(NH_4)_2PO_4$ and then to form Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get Mg2P2O7 finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ x } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of the flour spar ore sample is taken in to a platinum crucible and then to this added sufficient amount 20mL of HCl and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

$$\% \ MgF_2 = \text{Weight of MgO X } 0.6623$$

C) Magnesite:-

It contains maximum amount of $MgCO_3$ and minimum of $CaCO_3$.

The following constituents are:

*Determination of Moisture

*Determination of Loss of ignition

*Determination of Combined water and CO_2

*Determination of Organic matter

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Lime

*Determination of Magnesium

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g ore sample is taken in to a platinum crucible. After then it is heated at 500^{0}c for 50min. after then also apply the sudden heat at 1000^{0}c at 30min. it is called loss of ignition. Then it is cooled in a desicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of combined water and CO_2:-**

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through a O_2 and the another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^{0}c.then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

$$\% \ CO_2 = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Organic Carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 100^{0}c. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

Loss of ignition:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is heated at 1100^{0}c. Then it is cooled in a desicator. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

Combined water and CO_2:-

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-11000c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Silica:-**

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCl solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCl at 500^0c for 15min. After then cooled in a desicator. Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe\ (III) + NH_4OH \rightarrow Fe\ (OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCl, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCl. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a desicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \times \text{Weight of } Fe_2O3}{\text{Total weight of the sample}} \times 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a diphenyl amine indicator

$$Fe^{+3} + Sn^{+2} \rightarrow Fe^{+2} + Sn^{+4}$$

$$Fe^{+2} + Cr^{+6} \rightarrow Fe^{+3} + Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH \rightarrow Al(OH)_3 \rightarrow AlPO_4 \rightarrow Al_2O_3$$

Apparatus: - platinum crucible, desicator.

Chemical required: - HCl, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCl solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get AlPO4 and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \text{ Alumina} = \frac{0.4178 \text{ X Weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Calcium:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

Calcium present in the ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - di ammonium oxalate

Apparatus: - platinum crucible, desicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium or lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount di ammonium oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of magnesia. Magnesium di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCl, di ammonium phosphate

Apparatus: - platinum crucible, desicator

Procedure:-

Take 5g of ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCl and $(NH_4)_2PO_4$ and then to form Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get Mg2P2O7 finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ x } 0.3622}{\text{Total weight of the sample}} \text{ X } 100$$

ANALYSIS OF FLUXES:-

a) Limestone:-

Lime stone contains the maximum amount of $CaCO_3$ and minimum amount of $MgCO_3$.

The following constituents are:

*Determination of Moisture

*Determination of Loss of ignition

*Determination of Combined water and CO_2

*Determination of Organic matter

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Lime

*Determination of Magnesium

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g ore sample is taken in to a platinum crucible. After then it is heated at 500^0c for 50min. after then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a desicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of combined water and CO_2:-**

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through a O_2 and the another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^0c.then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

$$\% \; CO_2 = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Organic Carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 100^0c. After then it is cooled in a desicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

Loss of ignition:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is heated at 1100^0c. Then it is cooled in a desicator. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Combined water and CO_2:-

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-11000c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

***Determination of Silica:-**

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCl solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCl at 500^0c for 15min. After then cooled in a desicator, Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of $Fe(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$Fe(III) + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCl, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - platinum crucible, desicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCl. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a $Fe(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a desicator and dried. Finally Fe_2O_3 weight is formed.

% Iron = $\frac{0.6944 \text{ X Weight of } Fe_2O_3}{\text{Total weight of the sample}}$ X 100

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a diphenyl amine indicator

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - platinum crucible, desicator.

Chemical required: - HCl, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCl solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get AlPO4 and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

% Alumina = $\frac{0.4178 \text{ X Weight of } Al_2O_3}{\text{Total weight of the sample}}$ X 100

*** Determination of Calcium:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

Calcium present in the ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4\rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - di ammonium oxalate

Apparatus: - platinum crucible, desicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium or lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount di ammonium oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of magnesia. Magnesium di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCl, di ammonium phosphate

Apparatus: - platinum crucible, desicator

Procedure:-

Take 5g of ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCl and $(NH_4)_2PO_4$ and then to form a Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get Mg2P2O7 finally cooled in a desicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ x } 0.3622}{\text{Total weight of the sample}} \text{ X 100}$$

b) Dolomite:-

The following constituents are:

*Determination of Moisture

*Determination of Loss of ignition

*Determination of Combined water and CO_2

*Determination of Organic matter

*Determination of Silica

*Determination of Iron

*Determination of Alumina

*Determination of Lime

*Determination of Magnesium

*** Determination of Moisture:-**

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 105^0c at 2 hours. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of Loss of ignition:-**

Accurately weighed 2g ore sample is taken in to a platinum crucible. After then it is heated at 500^0c for 50min. after then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a desicator and it is filter and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of combined water and CO_2:-**

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-1100^0c.then water vapors are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

$$\% \; CO_2 = \frac{\text{Increasing the KOH tube weight}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Organic Carbon:-**

% Organic matter = (% Loss of ignition - % Moisture + % CO_2

Moisture:-

Accurately weighed 5g of ore sample is taken into a platinum crucible. After then it is heated at 100^0c. After then it is cooled in a dessicator. It is dried and weighed. Finally loss of weight is obtained.

$$\% \text{ Moisture} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Loss of ignition:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is heated at 1100^0c. Then it is cooled in a dessicator. Finally loss of weight is obtained.

$$\% \text{ Loss of ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

Combined water and CO_2:-

Accurately weighed 5g of ore sample is taken in to a procaline crucible. Then it is placed in a heating chamber consists of free weighed $CaCl_2$ and KOH tube. One end s passing through O_2 and another end is $CaCl_2$ tube weight. After then sample is heated at 300^0-11000c.then water vapor are observed at $CaCl_2$ tube and CO_2 observed in to a KOH tube.

$$\% \text{ combined water} = \frac{\text{Increasing the } CaCl_2 \text{ tube weight}}{\text{Total weight of the sample}} \times 100$$

***Determination of Silica:-**

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced fused mass. After then it is heated with 1:1 HCL at 500^0c for 15min. After then cooled in a dessicator, Precipitate is formed and then filter the precipitate and then to form a residue. Finally loss of weight of the sample.

$$\% \text{ Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

*** Determination of Total iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the ore sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the ore sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$\text{Fe (III)} + NH_4OH \rightarrow Fe(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, dessicator

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

$$\% \text{ Iron} = \frac{0.6944 \text{ X Weight of } Fe_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

2) Volumetric method:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a diphenyl amine indicator

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina**:-

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 or 4mL of NH_4OH and then to get an Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get an $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\ (OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - platinum crucible, dessicator.

Chemical required: - HCL, HNO_3, NH4 $(OH)_3$, Na_2PO_4 or $(NH_4)_2PO_4$, Suitable solvent

Procedure:-

Accurately weighed 5g of ore sample is taken in to a platinum crucible. After then it is treated with HNO_3 and 3 or 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 or $(NH_4)_2PO_4$ and we get AlPO4 and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

$$\% \quad \text{Alumina} = \frac{0.4178 \text{ X Weight of } Al_2O_3}{\text{Total weight of the sample}} \text{ X } 100$$

*** Determination of Calcium:-**

It is determined by two methods

1) Gravimetric method:-

Principle:-

Calcium present in the ore sample in this determined by Calcium. Calcium oxalate precipitate is formed by adding the di Ammonium oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4 \rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di ammonium oxalate

Apparatus: - platinum crucible, dessicator

Procedure:-

The filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for the analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium or lime} = \frac{\underline{0.56 \text{ X Weight of CaO}}}{\text{Total weight of the sample X } 100}$$

2) Volumetric method:-

The filtrate is obtained after removal of Al, Fe; Cr. from the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesium:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the ore sample in this determination of magnesia. Magnesium Di ammonium phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Iron ore sample} + (NH_4)_2PO_4 \rightarrow Mg\ (NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di ammonium phosphate

Apparatus: - platinum crucible, dessicator

Procedure:-

Take 5g of ore sample taken in to a platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for the analysis. After then it is heated at 35^0c and we get Mg2P2O7 finally cooled in a dessicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \times 0.3622}{\text{Total weight of the sample}} \times 100$$

UNIT-III

ANALYSIS OF FINISHED PRODUCTS-II

A) Chemical Analysis of Cement:-

Cement is a mixture of Tri Calcium Silicate and Tri Calcium Aluminate and also contain small amount of Magnesium Oxide and Calcium Sulphate.

Sampling:-

Accurately weighed 3grams of Cement sample is passing through an over conveyor belt for time to time during the manufacturing process, collect the all sampling through 20mesh size. Finally then it is stored on an air tight bottle.

Dissolution:-

To take the one gram of sample is taken into a beaker. After then it is heated with water (or) concentration of HCL is added to the sample solution for dissolution purpose. Then heat the solution, until effervescences are formed.

Composition of Cement:-

Silica – 21.16%

Alumina – 5.65%

Iron – 2.23%

CaO – 63.23%

MgO – 4.16%

SO_3 – 1.16%

Loss of ignition – 0.99%

Insoluble residue – 0.09%

Cement containing the following constituents is:-

*Determination of Silica

*Determination of NH_4OH group

*Determination of Iron

*Determination of Alumina

*Determination of Lime (Calcium)

*Determination of Magnesia

*Determination of Sulphide Sulphur

*Determination of Total Alkalis (K_2O, Na_2O)

*Determination of Free CaO in Cement and Clinker

*Determination of Sulphur Tri Oxide

*Determination of Loss of Ignition

*Determination of Insoluble Residue

*** Determination of Silica:-**

Accurately weighed 5g of sample is taken in to a Platinum crucible. After then it is dissolved in sufficient amount of HCL solution. After then it is treated with fusing mixture and then to produced a fused mass. After then it is heated with 1:1 HCL at 500^0c for 15min. After then cooled in a dessicator, precipitate is formed and then filter the precipitate and then to form a residue. We get a loss of weight of the sample.

$$\% \quad \text{Silica} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \times 100$$

***Determination of NH_4OH group:-**

Filtrate is obtained, after then removal of Silica. Then it is washed with 1:1 NH_4OH. We get a Yellow (or) White colour precipitate is formed. Filter the precipitate, filtrate is removed, residue is taken. Then residues is washed with NH_4CL and also to this added 1:1 NH_4OH.we get a Yellow (or) White colour precipitate. Filter the precipitate, residue is taken. Then residue is washed with NH_4CL washing are added to the filtrate. Finally residue is taken into a Platinum crucible. Then it is heated at 1100^0C. Residue cooled in a dessicator. We get a weighed as a NH_4OH.

***Determination of Total Iron:-**

Iron is determined by two methods- - 1) Gravimetric method

2) Volumetric method

1) Gravimetric method:-

Principle:-

Iron present in the sample, in this determination of Fe $(OH)_3$ is formed by adding to the NH_4OH to the sample solution. After then it is heated. Finally we get a Fe_2O_3 precipitate.

$$\text{Fe (III)} + NH_4OH \rightarrow \text{Fe }(OH)_3 \rightarrow Fe_2O_3$$

Chemicals required: - HCL, NH_4OH, H_2S, Fusing mixture (K_2CO_3+Na_2CO_3, KNO_3+$NaNO_3$)

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Accurately weighed 5g of sample is taken in to a Platinum crucible. Then to this added sufficient amount of HCL. Now we can remove the Chloride, Mercury, and Silver. After then to this added H_2S. Now we can remove the Cu, Bi, Cd and Sulphides. After then it is treated with fused mixture. Then to form a Fe $(OH)_3$ precipitate. Filtrate is used for another analysis. Residue is taken and then it is washed with NH_3 and it is ignited and then cooled in a dessicator and dried. Finally Fe_2O_3 weight is formed.

% Iron = $\underline{0.6944 \times \text{Weight of } Fe_2O_3}$

Total weight of the sample X 100

2) Volumetric method:-

Accurately weighed 5g of sample is taken in to a Platinum crucible. After then it is treated with NH_4OH and then to form a Fe $(OH)_3$ precipitate. After then to this added to the sufficient amount of $FeCl_3$. After then it is treated with reducing agent $SnCl_2$ and then Fe^{+3} is converted in to Fe^{+2} state. After then it is added to the sufficient amount of H_2SO_4 and H_3PO_4. Finally it is titrated with $K_2Cr_2O_7$ by using a Di Phenyl Amine indicator.

$$Fe^{+3}+Sn^{+2}\rightarrow Fe^{+2}+Sn^{+4}$$

$$Fe^{+2}+Cr^{+6}\rightarrow Fe^{+3}+Cr^{+3}$$

Factor: - 1mL of 0.1N $K_2Cr_2O_7$ = 0.005g of Iron

*** Determination of Alumina:-**

Principle:-

In this determination of Alumina and then it is oxidized with HNO_3 and to this added 3 (or) 4mL of NH_4OH and then to get Al $(OH)_3$ precipitate. After then to this added sufficient amount of Na_2PO_4 or $(NH4)_2PO_4$ and we get $AlPO_4$ precipitate. After then it is washed with suitable solvent and it is ignited and dried and then finally weighed as Al_2O_3.

$$Al^{+3}+HNO_3+NH_4OH\rightarrow Al\,(OH)_3\rightarrow AlPO_4\rightarrow Al_2O_3$$

Apparatus: - Platinum crucible, Dessicator.

Chemical required: - HCL, HNO_3, $NH_4\,(OH)_3$, Na_2PO_4 (or) $(NH_4)_2PO_4$, Suitable solvent.

Procedure:-

Accurately weighed 5g of sample is taken in to a Platinum crucible. After then it is treated with HNO_3 and 3 (or) 4mL of NH_4OH and then to form Al $(OH)_3$ precipitate. After then filter the precipitate and then taken in to a residue and it is treated with HCL solution. After then to this added Na_2PO_4 (or) $(NH_4)_2PO_4$ and we get $AlPO_4$ Precipitate and then it is washed with suitable solvent. After then it is ignited and it is dried. Finally Al_2O_3 is formed.

% Alumina = $\underline{0.4178 \text{ X weight of } Al_2O_3}$
Total weight of the sample X 100

***Determination of Lime:-**

It is determined by two methods:

1) Gravimetric method:-

Principle: -

Calcium present in the sample. In this determined by Calcium. Calcium Oxalate precipitate is formed by adding the Di Ammonium Oxalate to the filtrate solution. After then it is heated at 500^0c, we get a Calcium Carbonate. After then again heated at 800^0-1000^0c.Finally we get a CaO.

$$\text{Filtrate} + (NH_4)_2C_2O_4\rightarrow CaC_2O_4$$

$$CaC_2O_4 \rightarrow CaCO_3$$

$$CaCO_3 \rightarrow CaO$$

Chemicals required: - Di Ammonium Oxalate

Apparatus: - Platinum crucible, Dessicator

Procedure:-

Filtrate obtained and after then removal of Al, Fe, Cr. From the above filtrate 10mL of filtrate solution is taken into a glass beaker. After then it is treated with $(NH_4)_2C_2O_4$ and then we get a CaC_2O_4 precipitate. After then it is filter the precipitate. Filtrate is used for another analysis. After then residue is taken and then it is heated at 500^0c and then we get a $CaCO_3$. After then again heated at 800^0-1000^0c and then we get a CaO.

$$\% \text{ Calcium (or) Lime} = \frac{0.56 \text{ X Weight of CaO}}{\text{Total weight of the sample X 100}}$$

2) Volumetric method:-

Filtrate is obtained after removal of Al, Fe; Cr. From the above filtrate solution is taken in to a glass beaker. After then to this added sufficient amount Di Ammonium Oxalate and then we get a CaC_2O_4 precipitate. After then it is dissolved in H_2SO_4 solution and then to form a $CaSO_4$ and Oxalic acid after then it is titrated with $KMnO_4$ solution.

Factor: - 1mL of 0.1N $KMnO_4$ = 0.0028g of CaO

*** Determination of Magnesia:-**

It can be determined by two methods

1) Gravimetric method:-

Principle:-

It is present in the sample in this determination of Magnesia. Magnesium Di Ammonium Phosphate precipitate is formed and then by adding to the sample finally it is ignited and then we get an $Mg_2P_2O_7$.

$$\text{Sample} + (NH_4)_2PO_4 \rightarrow Mg\,(NH_4)_2PO_4 \rightarrow Mg_2P_2O_7$$

Chemicals required: - HCL, Di Ammonium Phosphate

Apparatus: - Platinum crucible, Dessicator.

Procedure:-

Accurately weighed 5g of sample taken in to a Platinum crucible and then to this added sufficient amount of 20ml of HCL and $(NH_4)_2PO_4$ and then to form an Mg $(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then to take the residue and then filtrate is used for another analysis. After then it is heated at 350^c and we get an $Mg_2P_2O_7$. Finally cooled in a dessicator and then dried and weighed as $Mg_2P_2O_7$.

$$\% \text{ Magnesium} = \frac{\text{Weight of } Mg_2P_2O_7 \text{ X } 0.3622}{\text{Total weight of the sample} \quad \text{X 100}}$$

2) Volumetric method:-

Accurately weighed 5g of sample is taken in to a Platinum crucible and then to this added sufficient amount 20mL of HCL and $(NH_4)_2PO_4$ and then we get a $Mg(NH_4)_2PO_4$ precipitate. After then filter the precipitate and then taken in to a residue. The filtrate is used for another analysis. Titrate with excess of H_2SO_4. The untreated H_2SO_4 is titrated with NaOH solution.

Factor: - 1mL of 0.1N H_2SO_4 = 0.002016g of MgO.

*Determination of Sulphide Sulphur:-

Accurately weighed 15mL of Ammonical $ZnSO_4$ solution is taken into a500mL conical flask. Then it is dissolved in 100mL hot water. After then to this added 5g of Cement sample. Then the samples again dissolved in hot water and concentration of HCL solution and then boil the solution. After then solution is cooled and then to this added sufficient amount of $SnCl_2$ and starch as an indicator. Finally then this solution is titrated with Potassium Iodide solution.

$$\% \text{ Sulphide Sulphur} = \frac{EV}{\text{Total weight of the sample} \times 100}$$

Here,

E = Equivalent weight of Potassium Iodide solution
V = Volume of KIO_3^-

* Determination of Total Alkalis (K_2O and Na_2O):-

$$\% K_2O = 3/2 \times K_2PtCl_6 \times 0.19411$$

$$\% Na_2O = \text{Total Alkalis} - \% K_2O$$

In this determination there is no direct method we are using the filtrate obtained after removal of Si, Fe, Al, Ti, Ca, Mn, Mg, P, S and etc. Then it is evaporated up to certain conditions. Finally we get a residue which is represented as the total weight of alkalis. After then to this added sufficient amount Platinum Chloride and then we get a Potassium Platinum Chloride precipitate. Filter the precipitate. Residue is taken in to a Platinum crucible and it is heated and then cooled in a dessicator. Finally dried and weighed as a Potassium Platinum Chloride.

$$\% KCL = 3/2 \times 0.30712 \times K_2PCl_6$$

$$\% NaCL = \text{Total Alkalis} - \% \text{ Alkali}$$

*Determination of Free CaO in Cement and Clinker:-

Accurately weighed 1.2g of Cement sample is taken into a Round Bottom Flask (RBF) then to this added 60mL of Glycerol and Alcohol and also added pure glass beads. Its acts as prevent the anti bumping agent. After then attach the reflux condenser and boiling for few min. Then remove of reflux condenser and then cooled. Finally then it is titrated with an Ammonium Acetate solution.

$$\% \text{ Free CaO} = \frac{EV}{\text{Total weight of the sample} \times 100}$$

Here,

E = Equivalent weight of Ammonium Acetate solution

V = Volume of Ammonium Acetate solution

*Determination of Sulphur Tri Oxide:-

Accurately weighed 1g of Cement sample is taken into a 250mL beaker. Then it is dissolved in water and concentration of HCL and then boils the solution, until effervescences are evolved. After then to this added sufficient amount $BaCL_2$.We get a $BaSO_4$ precipitate. Filter the precipitate. Filtrate is removed and then residue is taken into a Procaline crucible. Finally it is dried and weighed as a $BaSO_4$ precipitate.

$$\% \text{ Sulphur Tri Oxide} = \frac{\text{Weight of } BaSO_4 \text{ X } 0.343}{\text{Total weight of the sample X } 100}$$

***Determination of Loss of Ignition:-**

Accurately weighed 2g of sample is taken in to a Platinum crucible. After then it is heated at 500^0c for 50 min. After then also apply the sudden heat at 1000^0c at 30min. it is called loss of ignition. Then it is cooled in a dessicator and filter the solution and then to form a residue. Finally loss of weight is obtained.

$$\% \text{ Loss of Ignition} = \frac{\text{Loss of weight of the sample}}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Insoluble Residue:-**

Accurately weighed 1g of Cement sample is taken into a 500mL beaker. Then to this added few ml of water (or) concentration of HCL and boil the solution, until effervescences are formed. After then it is dilute with 50mL of water and it is placed at a hot place. After then it is cooled, we get a precipitate, filter the precipitate, filtrate is removed, then residue is taken. After then to this added 30mL of Na_2CO_3. Then it is boiled below 15min. Then this solution is cooled. After then to this added dilute HCL and hot water (1:9) ratio. Then it is cooled, and then we get a precipitate. Finally residue is dried and weighed.

B) Analysis of Oils:-

I) Saponification number:-

The amount of KOH required neutralizing free fatty acids from the complete hydrolysis of one gram of the sample.

Preparation of Alcoholic KOH:-

Accurately weighed 35-45g of KOH is taken into a one liter volumetric flask. Then to this added 120mL of distilled water. After then it is diluted with 90% of Alcohol.

Procedure:-

Accurately weighed 2g of sample is taken into a conical flask. After then to this added 25mL of Alcoholic KOH and then reflux for 2hrs. After then to this added 2(or) 3 drops of phenolphthalein as an indicator. Finally then it is titrated with 0.5N HCL solution and also conduct the blank titration.

$$\text{Saponification number} = \frac{(A\text{-}B) \text{ X } 2805}{\text{Total weight of the sample}}$$

Here,

A = Volume of HCL by using Sample titration

B = Volume of HCL by using Blank titration

II) Iodine number:-

The Iodine number represent the number of centigrams of Iodine is observed by the one gram of the oil sample. I.e. the percentage of Iodine is observed.

The origin bands of the Fatty acids and Esters. These are called as Glyceroids. There are Mono, Di and Tri Glyceroids. Glyceriod contains number of double and triple bonds. So it can more number of iodine is consumed.

The amount of Iodine is observed is measure of unsaturation. The Iodine value is constant for particular acids.

Preparation of wij's solution:-

To take 8g of ICl_3 is dissolved in Glacial Acetic acid in 200mL and 9g of Iodine is dissolved in 300mL of CCl_4.There are two solutions make up with one liter volumetric flask with Glacial Acetic acid.

Procedure:-

Accurately weighed 0.1-0.15g Oil sample is taken into a glass stopped bottle (It is depends up on the Iodine number). To this added10mL CCl_4 stir the contents and then to this added 20mL of wij's solution and shake well and then to this added 10% KI. After then shake the solution. Then the solution is settled for few hours. Iodine is liberated. The untreated Iodine is titrated with Hypo (Sodium Thio Sulphate) and also conducts the blank titration.

$$\text{Iodine number} = \frac{(b-a) \times 1.269}{\text{Total weight of the sample}}$$

Here,
b = Volume of Hypo (Sodium Thio Sulphate) by using blank titration

a = Volume of Hypo (Sodium Thio Sulphate) by using sample titration

III) Acid number:-

The milligram of KOH required neutralizing free fatty acids from the complete hydrolysis of one gram of the sample.

Procedure:-

Accurately weighed 25mL of Alcohol and 25mL of Ether is taken into a conical flask. Then to this added sufficient amount of KOH and 1% 2 (or) 3 drops of phenolphthalein indicator. After then to this added 1-10g of Oil sample. Finally then it is titrated with 0.1N NaOH solution.

$$\text{Acid number} = \frac{\text{Titration Volume in ml} \times 5061}{\text{Total weight of the sample}}$$

C) Analysis of soaps:-

Soaps are generally Alkali solids of fatty acids and their contain water and small amount of impurities. Soaps are different types there are,

Commercial soaps – Na_2CO_3, Borax, Rosin

Hard soaps - NaOH, Mixture of Potash salts and Sodium salts

Soft soaps – Potash salts

Liquid soaps – Water, Coconut Oils which are also contain Potash Glycerol and Alcohol. It is heating and then removed Glycerol and Alcohol. Half heating then no separation. Cool – To form a fatty acid cake.

Soaps containing the following constituent are:-

*Determination of Moisture

*Determination of Volatile matter

*Determination of Total Alkali

*Determination of Total fatty matter

*Determination of Free caustic Alkali (or) Free fatty acids

*Determination of Sodium silicate

*Determination of Chloride

***Determination of Total weight of Moisture and Volatile matter:-**

Accurately weighed 1g of Soap sample is taken into a procaline crucible. Then it is placed at a heating chamber and it is heated at 110^0C. Finally loss of the weight is obtained. This is present the Moisture and Volatile matter.

Estimation of moisture:-

Accurately weighed 10g of Soap sample is taken in to a 500mL Round Bottom Flask (RBF).After then to this added 100mL of Xylene and Toluene reagents. These are acts as an antifort flotation. After then reflux at 25^0C and heated at few minutes. Finally water vapour is collected at the receiver.

$$\% \text{ Moisture} = \frac{\text{Volume of } H_2O \text{ at } 25^0C \text{ in ml X } 0.994}{\text{Total weight of the sample}} \text{ X } 100$$

% Volatile matter = Total weight of Moisture and Volatile matter - % Moisture

***Determination of Total Alkali:-**

Accurately weighed 5g of Soap sample is taken into a conical flask. After then to this added sufficient amount of water and then to this added excess amount of 0.1N HCL. After then it is heated and then cooled. Finally we get a fatty acid cake. Filter the solution; filtrate is removed. The untreated 0.1N HCL is titrated with NaOH by using Methyl Orange as an indicator.

$$\% \, Na_2O = \frac{\text{Volume of 0.1N HCL X Normality of HCL X } 0.31}{\text{Total weight of the sample}} \text{ X } 100$$

$$\% \, K_2O = \frac{\text{Volume of 0.1N HCL X Normality of HCL X } 0.47}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Total fatty matter:-**

To take 200mL of Soap sample is taken into a 500mL beaker. After then to this added 40mL of HNO_3 solution. After then it is heated and cooled in a water ice bath. Finally we get a fatty acid cake. Filter the solution, filtrate is removed. After then fatty acid cake is taken into a Platinum crucible. Finally dried and weighed as a total fatty acid cake.

***Determination of Free caustic Alkali (or) Free fatty acids:-**

Accurately weighed 20g of Soap sample is taken into a 250mL conical flask. After then it is dissolved in 100mL of neutral Alcohol. Then it is boiled and cooled. Filter the solution, filtrate is taken. Then it is titrated with H_2SO_4 by using phenolphthalein as an indicator. We can calculate the % of Na_2O and K_2O. The Alcoholic solution is acid. Then it is titrated with standard NaOH solution. Finally we can calculate the fatty acids in terms of oleic acid.

$$\% \text{ Alkali in terms of NaOH (or) KOH} = \frac{\text{Volume of } H_2SO_4 \text{ X Normality of } H_2SO_4 \text{ X } 0.04}{\text{Total weight of the sample}} \text{ X } 100$$

***Determination of Sodium silicate:-**

In this determination of Sodium Silicate (SiO_2) ash will be obtained. The fatty acid cake will be obtained. After hen determination of free Alkali and then to this added sufficient amount of water (or) HCL. Filter the solution, filtration by using ash less filter paper. Repeat the process 3 (or) 4 times. Finally SiO_2 ash weight is obtained.

$$\% \text{ Sodium Silicate } (Na_2Si_4O_9) = \frac{\text{Weight of } SiO_2 \text{ X } 1.258}{\text{Total weight of the sample X } 100}$$

***Determination of Chloride:-**

Accurately weighed 5g of Soap sample is taken into a500mL volumetric flask. After then to this added 250mL of hot water and excess amount of Magnesium Nitrate (or) Sodium Nitrate. Filter the solution; filtrate is taken into a 250mL conical flask. After then to this added 1 (or) 2 drops of Potash Chromate as an indicator. After then it is titrated with $AgNO_3$ solution.

Factor: - 1mL of 0.1N Silver Nitrate = 0.00585g of NaCl

D) Analysis of Paints:-

Paints containing the following constituents are:-

*Determination of Vehicle

*Determination of Pigment

*Determination of $BaSO_4$

*Determination of Total Lead

*Determination of Lead Chromate

***Determination of Vehicle:-**

It is a film forming materials. The combination of Binders and Thinners is called Vehicle. The Pigments and Binders up to make non volatile materials produce film forming material. The thinners are added to the pigments to produce film forming material is very broad.

Method-I:-

In the determination of Vehicle the concentration of binding power increase non volatile matter also increase and also thinners will be produce the very broad of the film. In this case binding power of the vehicle represents the non volatile vehicle.

$$100-B/B = 100-A/C$$

$$C = (100-A/100-B)\ B$$

Here,

A = % of Non Volatile matter

B = % of Non Volatile vehicle

C = Binding power of total paint

Method-II:-

Aluminium coil method:-

First of all initially free weighed coil is taken, and then binders and thinners passed through it. Then produce to the film forming material at the Aluminium coil. After then few min, finally the vehicle is representing the final Aluminium coil weight and initial Aluminium coil weight.

***Determination of Pigment:-**

Pigment represents the colour. These are 3 types.

I) coloured pigments

II) Inorganic pigments

III) Organic pigments

I) coloured pigments:-

These pigments are determined by IR and UV methods

II) Inorganic pigments:-

These Pigments are not based on Carbon chains and rings. Instead, they consist of dry ground minerals, usually metals and metallic salts. Because of their composition, Inorganic pigments are usually more opaque and more insoluble than organic pigments.

III) Organic pigments:-

Generally organic pigments are determined by 2 methods.

a) Extraction method

b) Ignition method

a) Extraction method:-

Sufficient amount of Pigment sample is taken into a separating funnel. Then it is dissolved in water and shakes the solution and the solution is stand for few min. settle the colour pigment in bottom of the separating funnel. After then the filtrate is removed. Finally pigment is dissolved in Ethanoic Sodium Hydroxide. After then shake the solution and stand for few min. Finally weighed as a coloured pigment.

b) Ignition method:-

From the above residue is taken and it is heated. Then weighed as a coloured pigment.

***Determination of $BaSO_4$:-**

Accurately weighed sufficient amount of Pigment sample is taken into a conical flask. After then it is dissolved in water, after then it is treated with Na_2CO_3 (fusing mixture). We get a melt solution. After then it is boiled and then we get a clear solution. Then it is dissolved in water. Finally we get a precipitate. Filter the precipitate. Residue is taken, after then it is treated with 1:1 HCL and neutralized with NH_4OH. Finally hen it is titrated with H_2SO_4. We get a $BaSO_4$ precipitate. Then it is taken into a platinum crucible. After then it is dried and cooled in a dessicator.Finally $BaSO_4$ precipitate weight is obtained.

$$BaSO_4 + Na_2CO_3 \rightarrow BaCO_3$$

$$BaCO_3 + H_2SO_4 \rightarrow BaSO_4$$

$$\% \, BaSO_4 = \frac{\text{Weight of } BaSO_4}{\text{Total weight of the sample}} \times 100$$

***Determination of Total Lead:-**

Accurately weighed 2g of Paint sample is taken into a conical flask. Then it is dissolved in water and it is acidified with Acetic acid and neutralized with NH_4OH. After then H_2S gas is passed through it. After then it is titrated with H_2SO_4.We get a Lead Sulphate precipitate. Residue is taken into a Platinum crucible. Then it is ignited and cooled in a dessicator. Finally dried and weighed as a $PbSO_4$.

$$\% \, \text{Total Lead} = \frac{\text{Weight of } PbSO_4 \times 0.0693}{\text{Total weight of the sample}} \times 100$$

***Determination of Lead Chromate:-**

Accurately weighed 2g of Paint sample is taken into a conical flask. Then it is dissolved in water. After then it is acidified with Acetic acid and neutralized with NH_4OH. Finally then it is titrated with $K_2Cr_2O_7$ and then we get a Lead Chromate precipitate. Then it is taken into a Platinum crucible. After then it is ignited and then cooled in a dessicator. Finally it is dried and weighed as a $PbCrO_4$ precipitate.

$$\% \, PbCrO_4 = \frac{\text{Weight of } PbCrO_4 \times 0.0799}{\text{Total weight of the sample}} \times 100$$

UNIT-IV

ASSESSMENT OF WATER QUALITY

*Sources of water:-

The following constituents are:-

I) Rain water

II) Surface water

III) Ground water

IV) Sea water

I) Rain water:-

It is primary source of water; a part of rain water is filtered into the ground water to form ground water.

A part of rain water evaporated in atmosphere some of the rain water runs to form streams and rivers. This flows ultimately into the sea.

The rain water is purest form in nature, in clear bright and sparking rain water becomes impure, when a passes through atmosphere.

The rain water picks up impurities from atmosphere, such as dust micro organisms smooth and gases like CO_2, N_2, SO_2, NH_3 etc…

II) Surface water:-

It contains:-

A) Reservoirs

B) Rivers

C) Streams

D) Ponds

E) Lakes

It originate mostly from rain water, most people of Indian depends up on surface of water sources. Surface water posse's inorganic, organic bacteria, viral combination. The quantity of surface water decreases due to evaporation, percolation and translocation.

The most important sources of surface water are lakes, ponds, rivers and storage reservoirs. The choice of quality and quantity of the water available.

A) Reservoirs:-

An artificial lake formed by the construction of dam across a valley is known as reservoirs.

Water in rivers and streams decreases during summer in order to ensure a continuous flow. In rivers a dam is constructed across the rivers. The surface water is stored in reservoirs.

The multipurpose of reservoirs are used to stored water and four power generator.

The water in the reservoirs maybe contaminated by the minerals and pollutants from the atmosphere. May likely to grow in reservoirs.

B) Rivers:-

They are the principles of sources of water, many cities and towns. The rivers may be classified as perennial and non perennial, the quantity of water in river depends on,

i) Topography

ii) Character and area of attachment

iii) Weather and season

The quality of water in river is not reliable because of it contains salt suspended matters and other impurities. The river water may be contaminated by industrial pollutants.

C) Streams:-

It can be formed by run of the flow of the in streams, changes from season to season.

D) Ponds:-

They are formed because of excessive digging of ground water for various purposes.

They are filter up with water during monsoon season. It can be filled with available atmospheric impurities. It can't be used as surface of water is supply.

E) Lakes:-

It is formed due to the collection of water in natural basis (or) depression in a mountainous area (or) plane areas.

Sometimes stream may lead its water into these bases. The qualities of water in lakes differ from season to season.

Lake can be used as source of water supply. Water is stored water a long time in a lake. So that the suspend impurities settle down.

The water in lake may be contaminated by the pollutants in atmosphere and water pollutants due to its.

III) Ground water:-

The water surface below the earth surface is known as ground water. The rain water (or) surface water enters in to ground by in filtration and percolation.

The water level changes during different seasons. The underground water (surface) source is classified as springs and wells. The wells are classified as shallow well and deeps.

Springs:-

The water inside the earth crush react the surface by internal pressures. This is known as springs. Springs are 2 types.

I) Hot water spring

II) Cold water spring

The water is contaminated by dissolved spring and surface spring and antis' ion spring.

The quality of the spring water depends mainly of geological and topographical conditions.

Wells:-

It is critical drilled into the ground for the purpose of surface water the bases for drilling wells are

i) Geological conditions to earth surface

ii) Porovisity of various layers

iii) Quantity of water which is stored.

In different layers wells are classified as shallower wells (or) case wells. The shallow wells are constructed in upper most layer of earth surface depth (8-10m). A shallower well should be at least 20min away from surface of contamination. Such as septic tanks and soap tanks. The quality of water depends on the atmospheric conditions and the depth.

These wells are dug in to previous layer the water is contaminated in minerals present in the earth crush. The depth of tube well ranges from 50-250feet.

IV) Sea water:-

Sea water is complex mixture of 96.5 percent water, 2.5 percent salts, and smaller amounts of other substances, including dissolved inorganic and organic materials, particulates, and a few atmospheric gases.

***Classification of water for different uses:**

These are classified in to 4 types.

i) Domestic water

ii) Industrial water

iii) Public water

iv) Agricultural water

i) Domestic water:-

This type of water is used for drinking and domestic uses such as cooking, washing, bathing.

ii) Industrial water:-

All industrial required water in large quantities water is used for boilers and during the process in manufacture of iron, steel, power industries.

iii) Public water:-

Water is required for public cleaning fire fighting maintained of garden, swimming pools and other purposes.

iv) Agricultural water:-

Water is mainly used for craft production, food production to grow vegetables, plants ext. Water is essential for social and economical development of community.

***Types of water pollutants and their effects:-**

The phenomenon by which the quantity of water in the hydrosphere is decrease is called as water pollution.

Water occupies a major portion of earth; very little water is available for drinking.

Drinking water is available in ponds, rivers and underground water. This is also used for cultivation.

In olden days the population was less. People used to perform their daily activities in the nearby lakes (or) ponds.

Thus there is a little chance for the water gets polluted. Now a day the number of industries and agricultural sector many unwanted substances were released in to the water. Then the water has got polluted highly.

Changes occurring in water due to pollution:-

If the pollutants are present in water the following changes are occur.

Change in the colour and increase the salinity of water. Uncontrolled growth of seeds in water, decrease in the growth of fish.

Effects of water pollutants:-

Polluted water is not used for drinking. Polluted water leads to contaminated decreases like cholera, jaundice, typhoid and diarrhea.

Aquatic life get destroyed, the number of tourists visiting beaches get decreased**.**

Types of water pollutants:-

i) Inorganic pollutants

ii) Organic pollutants

iii) Sediments and oils

iv) Domestic waste

v) Industrial and agricultural waste

vi) Fluorides.

A part of the waste matter polluting drinking water causes water borne decreases like jaundice, cholera etc...

The water pollutants get in to the water either from industries by agriculture operations (or) domestic sewages.

The detergents which are very widely used in our houses can pollute the water.

i) Inorganic pollutants:-

Salts, trace elements like Cu, Zn, As etc. metals coming out from chromium, platinum industries. Then it affects human held and aquatic animals.

Metals and complex compounds can present in water can effect metals disturb the water system. Algae can't grow properly such surroundings decrease photo synthesis and increase air pollution directly.

Cyanides, Hs, CO_2, NO_2 and sulphides can present in water. It can affect pH of water various and becomes toxic to aquatic animals.

Heavy metals like lead and Mercury can affect water become toxic. Fluorides present in water then the water can't be used for drinking purpose as bones and teeth of human beings get affected.

ii) Organic pollutants:-

The production of synthetic organic compounds increasing year by year. They include plastics, fiber, detergents, paints, pesticides, fungicides, insecticides, dyes and pharmaceuticals.

Their presence in water they imparts colour and offensive odor.

The domestic waste and industrial waste coming out from sugar canes, paper, leather and pulp industries degrade in presence of micro organisms. Water in 'DO' value is decreased.

Which affect the aquatic like both animals and plant? Insecticides and pesticides present in the water then the water get polluted.

Sewage from domestic commercial food processing and industrial effluents coming out from water then they consume 'DO'.

iii) Sediments and oils:-

Sediments:-

Sediment is soil materials that is moved and deposited in a new location.

Sediment can consist of rocks and minerals, as well as the remains of plants and animals.

It can be as small as a grain of sand (or) as large as a boulder.

Sediment move from one place to another through the process of erosion.

Oils:-

Oil pollution of seas due to spilling of the oil, during the transport and other means increased in recent years.

Because of increasing the use of oil by all the countries of the world.

Every year 25billions of crude oil transport across the seas and so sea water get polluted.

iv) Domestic waste:-

Domestic waste is waste that is generated as a result of the ordinary day to day use of domestic premise and is either collected by (or) on behalf of a local government as part of a waste collection and disposal system.

Example includes municipal solid waste (household trash/refuse), hazardous waste, and wastewater (such as sewage, which contains bodily wastes (faces and urine) and surface, radioactive waste, and others.

v) Industrial and agricultural waste:-

Industrial waste:-

Agro industrial wastes are used for manufacturing of bio fuels, enzymes, vitamins, antioxidants, animals feed, antibiotics, and other chemicals through solid state fermentation (SSF) a variety of microorganisms are used for the production of these valuable productions of SSF processes.

Agricultural waste:-

Agricultural waste is waste produced as a result of various agricultural operations. It includes manure and other waste from farms, poultry houses and slaughter houses, harvest waste, fertilizer run off from fields, pesticides that enter into water, air or soils and salt and silt drainers from fields.

vi) Fluorides:-

The concentration of fluorides up to 3ppm in drinking water is harmless. When it is exceeds 3ppm it can't be used for drinking.

In the district of Nalgonda, Guntur and prakasam in Andhra Pradesh. Water contains excess of fluorides. It reacts with calcium present in the body to form calcium fluoride (CaF_2).

$$Ca+F_2 \rightarrow CaF_2$$

By this reaction the colour of the teeth terns to yellow for the same reason the bones become weak causing the decrease Fluorides.

***Analytical methods for the determination of the following ions in water:-**

Anions:-

***Determination of CO_3^{2-} & HCO_3^-:-**

Principle:-

The main principle involved in water sample is reacted with Phenolphthalein as an indicator. It gives the half estimation of Sodium Carbonate.

Water sample is treated with Methyl Orange as an indicator. After then it is titrated with HCL solution. It gives the total value of Carbonate & Bicarbonate. We get the Bicarbonate from the total mixture & individual Bicarbonate.

$$Na_2CO_3 + HPh \rightarrow NaHCO_3 + Ph^-$$

$$Ph^- + HCL \rightarrow HPh$$

$$Na_2CO_3 + 2HCL \rightarrow NaCl + CO_2 + H_2O$$

$$NaHCO_3 + HCL \rightarrow NaCl + CO_2 + H_2O$$

Chemical Required: - Water sample, Phenolphthalein indicator, Methyl Orange indicator, HCL.

Procedure:-

To take 20 ml of water sample is taken in to a conical flask. Then to this added 1 (or) 2 drops of Phenolphthalein as an indicator. Then it is titrated with HCL solution. The end point is pink to colourless solution. In this case the burette value multiplied with 2. If give the value of Na_2CO_3. If is represented as (P).

To take 20ml of water sample is taken in to a conical flask. Then to this added 1 (or) 2 drops of Methyl Orange as an indicator. This solution is titrated with HCL solution. If gives the value of Carbonate and Bicarbonate. It is represented as (M).Finally we get the Bicarbonate (HCO_3^-) subtract from the (M.P).

$$Na_2CO_3 = P$$

$$Na_2CO_3 + NaHCO_3 = M$$

$$NaHCO_3 = M.\ P$$

***Determination of Fluoride (F^-):-**

Principle: -

The sample solution containing Fluoride ions react with Zr- Alizarin Slake to form colourless Zr- Fluoride (ZrF_6^{2-}) and coloured dye. The coluor of the dye becomes progressively weak with increasing amount of Fluoride ions.

Chemical required:-Water Sample, Sodium Arsenate (Na_3AsO_4), and Acid Zr-Alizarin reagent

Preparation of Zr- Alizarin red Slake:-

To take 300mg of $ZrOCl_2$ $8H_2O$ in 50mL,70mg of Alizarin red in 50mL, 800ml of 1.5N HCL(or) 1.2N H_2SO_4 taken into a 1000mL of volumetric flask and make up to the mark with distilled water.

Procedure:-

To take 100ml of sample solution is taken in to a breaker f then to this added 1 [or] 2- drops of Na_3AsO_4. To remove the Fluoride ions present in the solution then to this added 5ml of acid Zr- Alizarin reagent and mixed thoroughly and compare the sample and standard after one hour.

Calibration curve:-

From this graph we can calculate the concentration of unknown sample solution.

***Determination of Chloride (Cl^-):-**

Chlorine present in drinking water is harmless. It present in amount below 250ppm. In waste water the chloride contains is higher than in raw water.

Principle:-

The sample solution containing Chloride ions is initially reacting with Silver Nitrate [$AgNO_3$] solution. It forms an $AgCl^-$ precipitate. The untreated $AgNO_3$ is titrated with Potassium Chromate. Then we get an $Ag_2Cr_2O_4$.

$$Cl^- + Ag^+ \rightarrow AgCl$$

$$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4$$

Chemicals required: - 0.01N $AgNO_3$, Water sample, H_2SO_4 [or] NaOH, K_2CrO_4 indicator.

Procedure:-

To take 20 ml of a sample solution is taken in to a conical flask, then to this added H_2SO_4 (or) NaOH. It is used to maintain pH range 7-10. After then to this added 5% Silver Nitrate solution, it is used as an indicator. After then it is titrated with 0.01N Potassium Chromate solution, the end point is yellow to red colour.

Factor:-1mL of $AgNO_3$ = 1μg of Cl^-

*Determination of Sulphate [SO_4^{2-}]:-

Principle: -

Sulphate ions are present in the water sample by adding of HCL & $BaCl_2$ solution. It is weighed as a $BaSO_4$ precipitate.

$$\text{Water sample} + BaCl_2 \rightarrow BaSO_4$$

Chemical required: - Water sample, HCL, $BaCl_2$

Procedure:-

Accurately weight sufficient amount of water sample is taken in to a conical flask. This solution is dissolved in HCL and then to this added sufficient amount of $BaCl_2$ solution. After then precipitate will be obtained. Filter the precipitate; residue is taken in to a platinum crucible. It is heated and cooled. It is dried and weight as a $BaSO_4$ precipitate.

*Determination of Phosphate (PO_4^{3-}):-

Spectro Photometric method:-

Principle:-

The water sample is treated with HNO_3 and Ammonium Molybdate and Ammonium Vanadate. Then to form an Ammonium PhosphoMolybdate. It measures the absorbance at 460nm at spectrophotometric method.

Chemical required: - Water sample, 0.01N HNO_3, Ammonium Molybdate, and Ammonium Vanadate

Procedure:-

To prepare the known concentration of standard Phosphate solution. Now we can prepare the series of standard. For ex: - 100ppm, 200ppm, 300ppm, 400ppm from the solution.

Each series of standards known volume and equal weight of Ammonium Molybdate and Ammonium Vanadate and HNO_3 solution. Yellow colour precipitate will be obtained.

Each series of standards introduced of the spectrophotometer and measure the absorbance.

Now we can draw the calibration curve, finally the water sample is titrated with Ammonium Molybdate and Ammonium Vanadate is placed at spectrophotometer and measure the absorbance. Finally we calculate the Phosphate concentration in ppm.

Factor:-1mL = 50μg of Phosphate

Graph:-

On X- axis is taken in to a concentration.

On Y- axis is taken in to an absorbance.

We get a straight line and finally we get to measure the absorbance in graph. The absorbance is 460nm.

*Determination of Nitrate (NO_3^-):-

Principle: -

Nitrates are reduced to Ammonium by adding Devard's allay (50%Cu, 45% Al, and 5% Zn) in strongly alkaline. The Ammonia is distilled in to excess of standard acid and finally estimated spectrophotometrically (or) Idometrically.

$$3NO_3^- + 8Al + 5OH^- + 2H_2O \rightarrow 8AlO_2^- + 3NH_3$$

Chemicals required:-Water sample, Devard's alloy, NaOH, Nessler reagent, H_2SO_4 solution.

Procedure:-

To take 5000mL of sample in Ammonia (NH_4^+) distillation apparatuses. After then to this added 50ml 10% of NaOH and then to evaporate the 200mL solution, cool the solution. Then to this added 3g of Devard's alloy and 30 ml of 10% NaOH and immediately connect the flask with a vertical condenser. Who's out let dippers in to a receiver containing 200mL of $0.2NH_2SO_4$ distilled at 50^0C to 80^0C for one hour? Disconnect the receiver make up the volume of solution. In the receiver to 25ml to take 5 (or) 10ml of aliquot in a 50ml volumetric flask. Then it is neutralized to pH4-5. After then to this added 2ml of Nessler reagent and then to estimate the absorbance at 424nm. This spectro photometric method is valid for NO_3^- concentration greater than 0.5ppm. While for concentration exceeding 5ppm titrimetry method may be followed. In the later cases the distillate can be directly back titrated with standard alkaline by adding methyl red as an indicator.

Factor:-lml of H_2SO_4 = 0.06201g of NO_3^-

Graph:-

0n X- axis is taken in to a concentration in PPM.

On Y- axis is taken in to an absorbance.

We get an absorbance is measured.

*Determination of Nitrite (NO_2^-):-

Principle:-

In Nitrite sample is react with Sulphalinanide to form an Azodye. Now azodye is then to this added NEDA reagent and then to form a pink color complex.

Sample (NO_2^-) + Sulphalinanide → Azo dye

Azodye + NEDA → Pink color complex.

Chemicals required:-Sample (NO_2^-), Sulphalinanide, NEDA, (NEDA= 1-Napthol Ethylene Diamine Dihydro Chloride).

Preparation of Sulphalinanide:-

50g of Sulphalinanide dissolved in 500ml of 0.2N HCL solution

Preparation of NEDA:-

To take 0.83g of NEDA is dissolved in 200ml hot water. After then this solution is cooled and filtered. After then solution is dilute to 250ml of glacial acetic acid.

Preparation of series of standard solutions:-

By diluting of standard NO_2^- solution 100ppb, 200ppb, 300ppb, 1000ppb of series of standards.

Procedure:-

Each series of standard added sulphalinanide. Then we get an azodye. The azodye is acts as an intermediate. Azodye is treated with NEDA reagent. Then to form a pink colour complex.

Sample (NO_2^-) + Sulphalinanide → Azo dye

Azodye + NEDA → Pink color complex

Graph:-

On X – axis is taken in to a concentration in ppb.

On Y – axis is taken in to an absorbance.

The straight line we get a measured the absorbance.

***Determination of CN^-:-**

Spectro Photometric method:-

Principle:-

Cyanide in alkaline distillate obtained is converted in to Cyanide Chloride by reaction with Chloramines-T at pH<8. The cyanide chloride is then mixed with Pyridine Barbituric acid reagent and then to form a red blue dye, then measured at absorbance at 578nm.

Chemicals required: - Water sample, Chloramine – T, Pyridine Barbituric acid, diluted Acetic acid.

Procedure:-

To take 5-10mL of sample is taken in to a 25mL volumetric flask. Then to this added 0.2mL of 1% Chloramine – T solution and adjusted the pH-6 with diluted Acetic acid mixed thoroughly. After then to this added 2.5mL of Pyridine Barbituric acid reagent (15g of Barbituric acid + 7.5mL of Pyridine+15mL of concentrated HCL diluted to 50mL) and shake the mixture and allow standing for 10min and measured the absorbance red-blue dye at 578nm.

Graph:-

On X-axis is taken into a concentration.

On Y-axis is taken in to an absorbance. We measured the absorbance from the graph.

*Determination of Sulphide (S^{2-}):-

Principle:-

To take the sample solution and then to this added H_2SO_4 and Zn $(CH_3COO)_2$ solution. Then it is treated with excess of Iodine. Then untreated Iodine is titrated with Hypo by using back titration.

$$H_2S+I_2 \rightarrow 2HI+S$$

$$I_2+2Na_2S_2O_3 \rightarrow Na_2S_4O_6+2NaI$$

Chemicals required:-Water sample, Zn $(CH_3COO)_2$, Iodine solution, Hypo ($Na_2S_2O_3$), H_2SO_4, H_3PO_4, Starch.

Procedure:-

To take 500ml of sample is taken into a one liter distillated flask. Then to this added 2N of 1mL of $Zn(CH_3COO)_2$ and 2mL of 1N NaOH and also added 20ml of 18N H_2SO_4 quickly distilled the solution into a 200mL of 2.2% of $Zn(CH_3COO)_2$. Then we get a $ZnSO_4$ precipitate. Disconnect the receiver flask and then added excess of 0.05N I_2 solution and acidified with H_3PO_4, after 20min it is back titrated with 0.05N Hypo with excess of I_2 by using starch as indicator.

Factor:-1mL of 0.05N I_2 = 0.85mg of H_2S.

Cations:-

Determination of Fe^{2+}:-

Principle:-

Fe (II) present in the water sample, it is titrated with standard Sodium Vanadate by using DPA indicator.

$$Fe (II) +V (V) \rightarrow Fe (III) +V (IV)$$

Chemicals required:-Water sample, DPA indicator, H_2SO_4, H_3PO_4, and $NaVO_3$

Procedure:-

To take 10mL of water sample is taken into a250mLmof conical flask. Then to this added 5mL of H_2SO_4 and 3mL of H_3PO_4 and 2 (or) 3 drops of DPA indicator. Then it is titrated with $NaVO_3$ solution. The end point is colourless to Pale blue colour.

*Determination of Fe^{3+}:-

Spectro Photometric method:-

Principle:-

Fe (II) present in water sample. This Fe (II) is oxidized to Fe (III) by using oxidizing agent- $KMnO_4$, $K_2Cr_2O_7$. Finally Fe (III) is titrated with Potassium Thio Cyanide (KSCN).

$$Fe (II) \rightarrow Fe (III)$$

$$Fe (III) +KSCN \rightarrow [Fe (SCN)_6]^{3-}$$

Chemicals required:-Water sample, standard Fe (III) solution, $KMnO_4$, HCL.

Preparation of series of standard:-

By diluting the standard solution of Fe (III) solution depends on 2ppm, 4ppm, 6ppm, 8ppm, and 10ppm series of standard solutions.

Procedure:-

The series of standard and added 2mL of HCL and added small amount of KSCN and make up the solution. Then take respond for each series of standard procedure is obtained to unknown solution. We get the concentration of solution. The calibration curve for drawn between concentration and absorbance. We get the maximum absorbance of the solution.

Graph:-

On X – axis is taken into a concentration

On Y – axis is taken into absorbance.

We get an absorbance from the graph.

***Determination of Hardness of water (Ca^{2+} & Mg^{2+}):-**

Determination of (Ca^{2+} & Mg^{2+}):-

Principle:-

The estimation of water sample is treated with EBT and added buffer solution. Then it is titrated with standard EDTA solution. It gives the value of Ca_{2+} & Mg^{+2} mixture concentration. It is represented as "E". After then water sample is treated with 4N NaOH and wash the Magnesium. After then to this added Murexide as an indicator. Then it is titrated with EDTA solution. It gives the value of Ca^{+2}. It is represented as "M". Finally Mg^{+2}, Ca^{+2} are subtracted from total.

Finally value of Mg^{+2} = E-M

Here,

E= Volume of Mg^{+2} & Ca^{+2}

M = Volume of Ca^{+2}

$$Ca^{2+}+Mg^{2+}+EBT \rightarrow Ca^{2+}\text{-}EBT+Mg^{2+}\text{-}EBT$$

$$Ca^{2+}\text{-}EBT+EDTA \rightarrow Ca^{2+}\text{-}EDTA+EBT$$

$$Mg^{2+}\text{-}EBT+EDTA \rightarrow Mg^{2+}\text{-}EDTA$$

Chemicals required:-Water sample, EBT indictor, standard EDTA, pH-10 buffer, Murexide, 4N NaOH.

Preparation of pH-10 buffer:-

Weight 17.5g of NH_4Cl is dissolved in 142mL of Ammonia is taken in a 250mL volumetric flask; it is making up to the mark.

Preparation of EBT (Eriochrome Black T) indicator:-

Weight 0.2g of EBT dye stuff is dissolved in 15mL of Tri Ethyl amine and 5.0mL of absolute alcohol.

Preparation of Murexide indicator:-

1g of Murexide is dissolved in 100mL volumetric flask with distilled water.

Procedure:-

To take 10mL of water sample is taken in to a 250mL conical flask. After then to this added 10mLof distilled water and 2 (or) 3 drops of EBT indicator is added and 5mL of buffer solution is also added. After then solution is titrated with standard EDTA solution. Finally it gives a total value of mixture of Ca^{+2} & Mg^{+2}.

To take 10mL of water sample is taken into a 250mL conical flask. After then to this added 10mL distilled water and buffer solution and also added 4N NaOH and it s mask the Mg^{+2} as $Mg(OH)_2$ precipitate. After then to this added 2(or) 3 drops of murexide indicator. Then it is titrated with standard EDTA solution. Then it gives the concentration of Ca^{+2}.

Value of Mg^{+2} = Value of mixture (Ca^{+2} & Mg^{+2}) - Value of Ca^{+2}

Factor:-

1mL of 0.01M EDTA = 1.0mg of $CaCO_3$ (total)

1mL of 0.01M EDTA = 0.4008mg of Ca^{2+}

***Determination of Chromium (Cr^{+3}):-**

Principle:-

The sample is reacting with excess of per Sulphate and distilled water in presence of $AgNO_3$. After then boil the solution and then to form a Chromate ions (Cr^{+3}). After then Chromate ions are reacting with excess amount of per Sulphate in presence of $AgNO_3$ on heating, then to form a dichromate ions. The remaining amounts of per Sulphate react on hydrolysis, and then liberate HSO_4^- ions.

$$2Cr^{+3}+3S_2O_8^{2-}+7H_2O \rightarrow Cr_2O_7^{-2}+6HSO_4^-+8H^+$$

The Cr^{+6} ions react with Di Phenyl carbazide and 1:1 H_2SO_4 to form a violet coloured compound.

Chemicals required:-Sample, standard sample solution, H_2SO_4, diphenyl carbazide.

Preparation of standard series:-

By diluting of series of standard Cr^{+6} contains 2ppm, 4ppm, 6ppm, 8ppm, 10ppm.

Procedure:-

To take the sample and then added 1:1 H_2SO_4 and diphenyl carbazide make up to the mark. Before this convert Cr^{+3} ions to Cr^{+6} ions by adding of excess of per Sulphate in presence of $AgNO_3$. Then to form a violet colour complex and then to calibrate the curve between absorbance and concentration.

Graph:-

On X – axis is taken into a concentration

On Y – axis is taken into absorbance.

We get an absorbance from the graph

*Determination of As^{+5}:-

Silver diethyl dithio carbamate (As^{+5}):-

Principle:-

Arsenium is reduced AsH_3 by using zinc stannous chloride in acid solution in a gutsiest generate AsH_3.

Procedure:-

To take 25-35mL of sample is taken into a clear generated bottle. After then to this added 5mL of concentrated HCL and 2mL of Potassium Iodide and 0.5mL of 40% of $SnCl_2$. This solution is successively through mixing, after then allow 15min for reduction of As^{+5} to As^{+3}. After then to this added 3g of zinc into solution in the generator and connected the scrubber absorbance. It is filled with lead acetate solution. Allow for 13min warm to the solution. AsH(III) is releasing to scrubber containing glass wool integrated with 4 to 5 drops of 10% lead acetate and the absorber containing 4mL of silver diethyl dithio carbonate in pyridine for 45min pour the solution from the absorber directly into one cell and measure the absorbance of red solution at 535nm**.**

*Determination of lead (Pb^{2+}):-

Principle:-

Lead is reacting with dithiozone to form pink colour complex. It is extracted with dithiozone in CCl_4**.**

Chemicals required:-Water sample, CCl_4, Ammonical Sulphate-Cyanide solution, dithiozone.

Preparation of Ammonical Sulphate Cyanide solution:-

350mL of NaOH + 3g of Potassium Cyanide +10g of Sodium Sulphate. These three solutions are diluted to one liter water in a glass stopped bottle.

Procedure:-

To take 100ml of sample and then to this added 30mL of Ammonical Sulphate Cyanide solution and then to this added 10ml of dithiozone solution. After then it is extracted with CCl_4 for 30sec. and to measure the absorbance of lead to extract at 550nm.

Graph:-

On X – axis is taken into a concentration

On Y – axis is taken into absorbance.

We get an absorbance from the graph.

*Determination of Mercury (Hg^{+2}) :-(dithiozone):-

Principle:-

Mercury is reacting with dithiozone in presence of 1N H_2SO_4 to produce an orange chelating complex. This is extracted into CCl_4 and to measure the absorbance at 419nm.

Chemicals required:-Water sample, dithiozone, 1N H_2SO_4, CCl_4, H_2O_2, and $KMnO_4$.

Procedure:-

To take 100mL of water sample is sample is taken into a 500ml distillation flask. Then to this added 5% $KMnO_4$ and then this solution is mix thoroughly and then reflux for 4hrs in ice cold water. After then solution is cooled and then to this added few ml of H_2O_2 solution and then to removed $KMnO_4$. This solution is cooled and then solution is adjusted and then to this added 1N H_2SO_4 in presence of dithiozone is added. It is extracted with CCl_4, the extract make up to 25mL of volumetric flask. To measure the absorbance at 490nm.

Graph:-

On X – axis is taken into a concentration

On Y – axis is taken into absorbance.

We get an absorbance from the graph.

*Determination of copper (Cu^{+2}):-

Principle:-

Copper is react with diethyl dithio carbamate and then to form a blue colour complex. It is extracted with CCl_4 and to measure the absorbance by spectro photometric method.

Chemicals required:-Water sample, diethyl dithio carbamate, CCl_4, Buffer.

Procedure:-

To take 100mL of water sample and then to this added 20mL of distilled water and 30mL of diethyl dithio carbamate and then to form a blue colour complex. After then it is extracted with CCl_4 and to measure the absorbance at 450-460nm.

Graph:-
On X – axis is taken into a concentration
On Y – axis is taken into absorbance.
We get an absorbance from the graph.

*Determination of Zinc (Zn^{2+}):-

Principle:-

Zinc present in the water sample, then it is reacted with EBT indictor and then to form a Zn-EBT complex. After then solution is titrated with EDTA solution. EBT is replaced with EDTA and then to form a Zn-EDTA complex in the presence of buffer solution.

$$Zn^{+2} + EBT \rightarrow Zn\text{-}EBT$$

$$Zn\text{-}EBT + EDTA \rightarrow Zn\text{-}EDTA + EBT$$

Chemicals required:-Water sample, EBTA solution, EBT indicator, Buffer solution.

Procedure:-

To take 10ml of water sample is taken into a 250ml conical flask. Then to this added 5ml of 10% buffer solution. After then to this added 10ml of distilled water and 2 (or) 3 drops of EBT indicator. From the above solution is titrated with EDTA solution. The end point is wine red to blue color.

*Determination of Cd^{2+}:-

Spectro photometric method (Dithiozone):-

Principle:-

Cadmium is react with dithioze and then to form a pink to red color. It is extracted to $CHCl_3$. To measure the absorbance at 518 nm is spectro photo metrically.

Chemicals required: - Sodium-Potassium Tartarate solution, Sodium Hydroxide – Potassium Cyanide, Hydroxyl Amine.HCL solution, dithiozone, $CHCl_3$, Water sample.

Procedure:-

To take 250mL water sample is taken into a separating funnel. Then to this added sodium potassium tatrate solution and 5mL of NaOH-KCN solution and 1mL of hydroxyl amine hydrochloride solution and 15mL of dithiozone. This solution is mix well. To stop the funnel and shake for one min, to transfer the $CHCl_3$ layer to a second funnel containing to this added 25mL of cold tartaric acid and then added 10ml of $CHCl_3$ layer, then to this added 0.25ml of NH_2OH HCL and 15mL dithiozone and 5mL of NaOH-KCN solution, this solution is shake for one minute measure the absorbance at 518nm.

*Determination of Cobalt (Co^{+2}):-

Principle:-

Cobalt is oxidized to Co^{+3} by using any oxidizing agent like $KMnO_4$, $NaVO_3$, and $K_2Cr_2O_7$.

$$Co^{+2} \rightarrow Co^{+3}$$

When Co^{+3} is treated with Nitraso- R- Salt in the presence of acetate acidic medium and then to form a complex.

Chemicals required: - Sample, Nitraso- R- salt, acetate acetic acid.

Preparation of standard series:-

By diluting of series of standard Co^{+3} solution contains 2ppm,4ppm,6ppm,8ppm,10ppm series of standard solutions.

Procedure:-

From each series of standard to this added 2ml of Nitraso-R-salt and few ml of acetate acetic acid and make up to the mark. Then each series of standards introduced into colorimetric instrument then to take he respond from the absorbance.

Graph:-

From this graph we can calculate the unknown concentration of cobalt from the water sample solution.

*Determination of Dissolved Oxygen (DO):-

The amount of oxygen present in the water sample is called dissolved oxygen.

Procedure:-

To take 100ml of water sample is taken into a 250mL beaker then to this added 40% of KF of 20mL (to mask Fe^{+3}) and then to this added 2mL of 36% of MnSO4 and 2mL of alkaline iodine azide solution (it is a mixture of NaOH is 50g+ 13.5g of NaI and 1g of NaN_3 these solutions are diluted to one liter) shake well and allow the precipitate and settle the substance. After then to this added 6mL of H_2SO_4 solution. The mixture is shake well and then to form a precipitate. The precipitate is dissolved. Iodine is liberated; the liberated iodine is titrated with hypo by using starch as an indicator.

$$MnSO_4+2OH^- \rightarrow Mn\ (OH)_2$$

$$Mn\ (OH)_2+O_2 \rightarrow MnO_2+H_2O$$

$$MnO_2+2I^-+4H^+ \rightarrow I_2+Mn^{+2}+2H_2O$$

$$2S_2O_3^{2-}+I_2 \rightarrow 2I^-+S_4O_6^{2-}$$

Factor:-5mL of 0.025M $Na_2S_2O_3$ = 1mg in one liter “DO”

*Determination of Biochemical Oxygen Demand (BOD):-

The amount of oxygen used by the suitable micro organisms present in water during 5 days at 20^0C. It is called Biochemical Oxygen Demand.

For pure water BOD is about 1ppm. The municipal sewage as BOD values are 100-4000ppm.

BOD greater than 17ppm indicates high pollution and is harmful.

Procedure:-

To take two samples of known quantity of sewage water taken into a 300mL of BOD bottles.

The dissolved oxygen in the first bottle is determined immediately without incubation and it is taken as “D”. This gives the initial dissolve oxygen concentration of the sample.

Keep the second bottle incubated in the dark at 20^0C for 5days.

After 5days to take the second sample bottle and measure the dissolved oxygen of the sample taken as a “D2”. This gives the dissolved oxygen present after incubation.

During the course of incubation organic matter gets decompose.

The unused dissolved oxygen at different times of incubation is determined by the acidified with 1N H_2SO_4. The sample solution is titrated with sodium thiosulphate.

Each ml of thiosulphate used in the titrations is equivalent to one milligram for one liter of dissolved oxygen.

Thus a direct reading of the burette will give mg/lit of dissolved oxygen.

Sodium thiosulphate in acid converts to dithionic acid. This reacts with dissolved oxygen to form sodium dithionate.

$$Na_2S_2O_3+H_2SO_4 \rightarrow H_2S_2O_3+Na_2SO_4$$

$$H_2S_2O_3+O_2 \rightarrow H_2S_2O_6+H_2O$$

$$H_2S_2O_6+Na_2SO_4 \rightarrow Na_2S_2O_6+H_2SO_4$$

$$\text{BOD in mg/lit} = \frac{\text{Number of milligram of Oxygen required}}{\text{Number of liter of sample}}$$

$$\text{BOD in mg/lit} = \frac{(D_1-D_2)-(B_1-B_2)\,F}{P}$$

Here,

D_1 = DO of diluted sample before the incubated of sewage sample

D_2 =DO of diluted sample after the incubated of sewage sample

B1&B2 are DO levels of diluted water of the control sample containing seeds before and after incubation.

P = Decimal fraction of seeds added to the sample under investigation.

F = Ratio of seeds in the sample to seeds in the control.

*Determination of Chemical Oxygen Demand (COD):-

Principle:-

The amount of oxygen required to oxidize organic substance present in polluted water is called Chemical oxygen demand.

Oxygen by a strong oxidizing agent like $K_2Cr_2O_7$ in the presence of H_2SO_4 & Ag_2SO_4, $HgSO_4$

$$3CH_2O+2K_2Cr_2O_7^{2-} \rightarrow 4Cr^{+3}+3CO_2+7H_2O$$

Chemicals required:-Organic matter, $K_2Cr_2O_7^{2-}$, Ag_2SO_4, $HgSO_4$, Ferroine indicator.

Procedure:-

To take 20mL of the water sample and then to this added 30mL of diluted H_2SO_4 and added 10mL of standard $K_2Cr_2O_7$(0.25N) and then this solution is reflux for 2hrs. The solution is cooled and diluted with distilled water to make up 150mL, after then to this added few drops of ferroine as an indicator. The untreated dichromate solution is titrated with standard ferrous ammonium sulphate (Mohr's salt). The volume of ferrous ammonium sulphate consumed in the titration is recorded.

A blank titration is 20mL of distilled water in the place of sample is to be titrated in the same manner.

$$\text{COD in mg/lit} = \frac{(a-b) \times C \times 8 \times 1000}{\text{Volume of sample}}$$

Here,

a = Volume of Fe (II) used for blank titration

b = Volume of Fe (II) used for sample titration

c = Normality of Fe (II)

***Standards for drinking water:-**

Water quality parameters and standards:-

The parameters for water quality characterization are listed in the given table. The permissible limits, as laid down by the United States public health drinking water standards (USPH) and Indian standard insulation (ISI) are listed for comparison. There refer to domestic water supplies for drinking water.

S.NO	Parameters	USPH Standard	ISI Standard
1	Colour, odour, taste	Colourless, odourless, tasteless	-
2	Inorganic chemical pH	-	6-8.5-6-9
3	Specific conductance	300nmho cm^{-1}	-
4	Dissolved oxygen	4.0-6.0 (ppm)	3.0
5	Total dissolved solids	500	-
6	Suspended solids	50	-
7	Chloride	250	600
8	Sulphate	250	1000
9	Cyanide	0.05	0.01
10	Nitrate+ nitrite	<10	-
11	Fluoride	1.5	3.0
12	Phosphate	0.1	-
13	Sulphide	$0.1mg^{-1}$ (ppb)	-
14	Ammonia	0.5	-
15	Boron	1.0	-
16	Calcium	100	-
17	Magnesium	30	-
18	Arsenic	0.05	0.2
19	Barium	1.0	-
20	Cadmium	0.01	-
21	Chromium(VI)	0.05	0.05
22	Copper	1.0	-
23	Iron(filterable)	<0.3	-
24	Lead	<0.05	0.01
25	Manganese(filterable)	<0.05	-
26	Mercury	0.001	-
27	Selenium	0.01	0.05
28	Silver	0.05	-
29	Uranium	5.0	-
30	Zinc	5.5	-
Organics			
31	COD	4.0	-
32	Carbon CHCl3 extract	0.15	-
33	Ethylene blue active substance	0.5	-
34	Phenols	0.001	0.005
35	Pesticides	0.0050	-
36	Poly cyclic aromatic hydrocarbons(PAH)	0.2(ppb)/0.02(ppm)	-
37	Surfactants	200	-
Radio acticives			
38	Gross beta	1000 Pc/L	-
39	Radium-226	3Pc/L	-
40	Strontium-90	10Pc/L	<500

Bacteriological			
parameters			
41	Coli form cells/100ml	100	-
42	Total bacteria count/100mL	1 x 106	-

Hardness of water:-

Water hardness is the amount of dissolved calcium and magnesium in the water. Hard water is high in dissolved minerals, largely calcium and magnesium. You may have felt the effects of hard water, literally, the last time you washed your hands.

Definition:-

The property of water to form an insoluble curd with soap instead of lather. In other words it is the soap destroying property due to the presence of bicarbonates, sulphates and chlorides of calcium and magnesium.

Types of hardness:-

Temporary hardness:-

It is due to the presence of bicarbonates of calcium and magnesium and can be removed by boiling.

Permanent hardness:-

It is due to the presence of sulphates, nitrates and chlorides of calcium and magnesium.

Classification of hardness:-

Soft water→1 m.eq/lit

Moderately hard water→1.3m.eq/lit

Hard water→3-6m.eq/lit

Very hard water→ 6 and > 6m.eq/lit

Measurements of hardness:-

Hardness of water is expressed/measured in terms of milli equivalent/lit.

1 milli- equivalent=50mgs.of $CaCO_3$/lit.

Causes water hardness:-

Water becomes hard by being in content with soluble, divalent, metallic cations.

The two main cations that cause water hardness are calcium and magnesium.

Calcium is dissolved in water as it passes over and through limestone deposits.

How is water hardness determined?

Waters hardness is determined by the concentration of multivalent cations in the water.

Multivalent cations are positively charged metal complexes with a charge greater than it. Usually, the cations have the charge of 2^+; common cations found in hard include Ca^{2+} and Mg^{2+}.

Why is it called hard water?

Water can either be hard or soft, hard water does not describe the actual felling of the water. Instead, it is called hard water because it has mineral deposits like calcium and magnesium within the water molecules, water becomes hard when it seeps into the ground and absorbs calcium and magnesium.

Advantages of hardness:-

Reduced prevalence of,

1. Arteriosclerotic heart disease, 2. Degenerative heart disease, 3. Sudden death due to cardiac arrest, 4. Hyperetension and , 5. Tetny.

Removal of hardness:- A) For temporary hardness:-

Boling, addition of lime.

B) **For permanent hardness:-**

Addition of lime, Addition of sodium carbonate, Base Exchange method/permuted process.