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PAPER - III

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Applied Analysis - I

UNIT - I

ANALYSIS OF ORES

GENERAL TECHNIQUES OF ANALYSIS APPLIED TO COMPLEX MATERIALS :-

The technique adopted differ from the Nature and the stable of the complex material.

Different stages in Analysis :-

- 1) Sampling of the material
- 2) Dissolution of the sample (or) complex material.
- 3) Separation of the constituents
- 4) Estimation of the contents.

1) Sampling of the material :-

The sampling techniques applied to the complex materials are different methods for ores, alloys, fertilisers and soil.

The sampling technique depends upon the stable of the complex material. For example, for solids Coring & quartering, for liquids Thieret-Sampling.

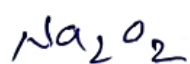
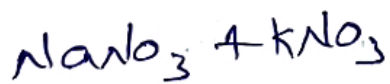
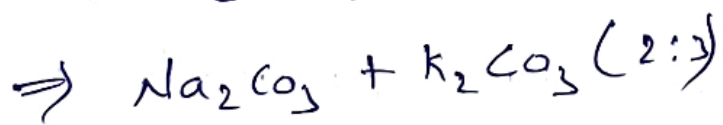
2) Dissolution of the Complex material :-

For the ores, the different techniques

are used to different types of materials.

Generally ores are not directly broad into solution by a single solvent.

- ⇒ oxide - ores are dissolved in Con. HCl and a mixture of Con. HCl and H_2SO_4
- ⇒ In case of sulphide ores, the ore is roasted with oxidising agent to convert into sulphate then it is dissolved in Con. HCl .
- ⇒ In case of metals and alloys Con. HNO_3 is used for dissolution of the sample by digestion.
- ⇒ In case of refractive materials, fluxes and skye fusion treatment methods are employed.
- ⇒ The sample is heated with fusion mixtures in Nickel (or) silver (or) iron crucible.



Potassium Pero Sulphate

Potassium Pyro sulphate

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3) Separation techniques:-

- ⇒ during the dissolution with acid treatment, silica will remain as insoluble residue.
- ⇒ It is filtered and separated and weighed as SiO_2
- ⇒ during the dissolution with acid treatment in case of certain ores some elements may be precipitated as insoluble residue along with silica. Separation is done by selective precipitation methods by the regulation of pH of the solution. For example, lead & barium are precipitated as sulphates treating the sample solution with H_2SO_4 .
- ⇒ The second group elements Cu, Pb are precipitated as sulphides.
- ⇒ The third group elements Fe, Al as hydroxide in alkali medium ($\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$)
- ⇒ Ca & Mg are precipitated as carbonates and Ca can also be precipitated as oxalate.
- ⇒ Phosphorus is precipitated as phosphates.
- ⇒ The sample solution is treated with certain organic reagents to convert the metal into a colored complex.

4) Estimation of the contents :-

Estimation can be achieved by mainly

3 types.

- i) volumetric method
- ii) Gravimetric method
- iii) Instrumental method.

SCOPE of metallurgical Analysis :-

Metallurgical analysis mainly based on the qualitative and quantitative analysis of the various raw materials, metals, alloys and other materials.

⇒ Qualitative analysis deals with the identification and separation of substances.

⇒ The qualitative analysis in the metallurgical field is particularly essential prior to quantitative analysis of unknown substances.

⇒ Quantitative analysis is concerned with determination of the amount of an element

(or) chemical substance present either alone

(or) in a mixture of the other substances.

⇒ Metallurgical analysis actually consists of four major steps.

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- i) Sampling, selection of a representative sample of the material to be analysed.
- ii) Conversion of the desired constituent into a form of suitable for measurements.
- iii) Measurements
- iv) Calculation and interpretation of the measurements.

b) ANALYSIS OF ORES :-

- 1) Iron ore :-
- 1) Moisture
 - 2) Loss of Ignition
 - 3) Total iron
 - 4) Ferrous iron (Fe^{+2})
 - 5) Ferric iron (Fe^{+3})
 - 6) Alumina / Al_2O_3
 - 7) Silica / SiO_2
 - 8) Titania / TiO_2
 - 9) Lime / CaO
 - 10) Magnesia / MgO
 - 11) Sulphur
 - 12) Phosphorus
 - 13) Manganese
 - 14) Alkalies [$Na_2O + K_2O$]
 - 15) Combined water
 - 16) Flue dust

1) Determination of Moisture :-

5gms of sample is heated in a vacuum oven at 105 to 110°C for 2 hours and until a constant weight is obtained. It is then cooled in a desiccator to room temperature and reweighed, the loss on the weight represents

The % of moisture, this can be calculated as follows.

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

2) Loss on Ignition :- (Calcination)

2gms of sample is taken in a Pt (or) Porcelain crucible and ignited gently in a muffle furnace is allowed about 500°C for 5 minutes and then at 950-1000°C for 30 minutes. The ignited sample is cooled in desiccator and weighed.

The loss on weight will represent the loss on ignition.

$$\% \text{ of Loss on Ignition} = \frac{\text{Loss in weight}}{\text{weight of the sample}} \times 100$$

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Total Iron:-

Estimation of Iron in an iron ore

Consisted of basically 3-steps.

⇒ Extraction of iron into solution

⇒ Free reduction (or) oxidation for volumetric analysis

(or) Precipitation for gravimetric analysis.

⇒ Titration in volumetric analysis (or) Ignition and weighing in gravimetric analysis.

Preparation of sample solution:-

The principle ores may be broadly classified

into categories. i) oxide ores

ii) Silicate ores

iii) Sulphide ores

⇒ Iron from the oxide ores can be efficiently brought into solution with Hot con. HCl but not with con. H_2SO_4 (or) HNO_3 because they are converted into sulphates and hydroxides.

The rate of the dissolution may be increased by addition of little $SnCl_2$, which will effect, the dissolution by reducing Fe^{+3} to Fe^{+2} .

⇒ Sulphide ores can be directly dissolved in mixture of HCl and HNO_3 sulphide ores can also be brought into solution, by roasting followed by dissolution in HCl.

iii) Silicate ores are not extractable with Con. HCl
Silicate ores will be decomposed by Na_2CO_3
This fused mass is dissolved in HCl .

Analysis by volumetric method:-

For the determination of total iron present in a ore sample is to be converted into ferrous state reduction of Fe^{+3} iron to Fe^{+2} iron is carried out by using a reducing agent such as SnCl_2 , Pb , Zn -Amalgam SnCl_2 is added to the acid solution,

SnCl_2 reduces Fe^{+3} iron to Fe^{+2} iron in presence of HCl . The excess of SnCl_2 is oxidising readily to SnCl_4 on the addition of HgCl_2 . The solution is boiled for few minutes, then the solution is filtered. The filtrate is titrated with oxidising agent like $\text{K}_2\text{Cr}_2\text{O}_7$ (or) KMnO_4 (or) ceric ammonium sulphate.

oxidation with $\text{K}_2\text{Cr}_2\text{O}_7$:-

The reduced Iron(II) solution is titrated with $\text{K}_2\text{Cr}_2\text{O}_7$ in presence of a mixture of H_2SO_4 and H_3PO_4 and also added few drops of diphenyl amine (or) Barium diphenyl amine sulphate indicator, the end point is reddish violet colour.

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$$\% \text{ of Total Iron} = \frac{\text{Vd. of } K_2Cr_2O_7 \times \text{Normality of } K_2Cr_2O_7}{\text{weight of sample}} \times 0.56 \times 100$$

Determination of Ferrous iron:-

The ore sample is moistured with distilled water and add $NaHCO_3$, HCl & HF . The digestion is carried out on gentle heat for 15 mins, After the digestion is completed the solution is diluted and then titration of the whole solution can be carried out against standard $K_2Cr_2O_7$, Fe^{+2} iron can be calculated from the volume required to oxidise Fe^{+2} to Fe^{+3} iron.

$$1 \text{ ml (or) } 0.1N \text{ } K_2Cr_2O_7 = 0.0056 \text{ gm of } Fe^{+2} \text{ iron.}$$

Determination of Fe^{+3} iron:-

Determination of ferric iron can't be made directly by either gravimetric (or) Titrimetric method. For estimating ferric iron Fe^{+2} iron and total iron contents of the ore are estimated from the above methods and the difference b/w two ^{will} give the content of Fe^{+3} iron in the ore.

$$\% \text{ of } Fe^{+3} \text{ iron} = \frac{\text{diff b/w total iron \& } Fe^{+2} \text{ iron}}{\text{weight of the sample}} \times 100$$

Determination of SiO_2 :-

The ore sample can be decomposed by acid treatment (or) by fusion with Alkali carbonates (or) bicarbonates. The fused mass is mixed with HCl and heated to dryness, this is filtered off ignited and weighed.

$$\% \text{ of } \text{SiO}_2 = \frac{\text{weight of the residue}}{\text{weight of the sample}} \times 100$$

Determination of Alumina (Al_2O_3) :-

Determination of Al_2O_3 is based on the selective formation of Aluminium phosphate from the acidic solution of above separation by the addition of sodium (or) Ammonium phosphates.



Ammonium phosphate

This AlPO_4 formed is stable and this can be ignited & weighed.

Procedure :-

The filtrate obtained after removal of SiO_2 is oxidised by addition of HNO_3 and boil to reduce the volume into 100ml then the

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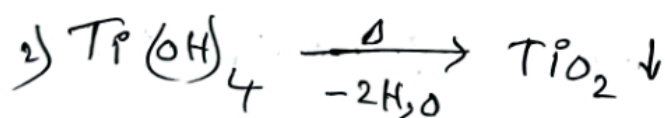
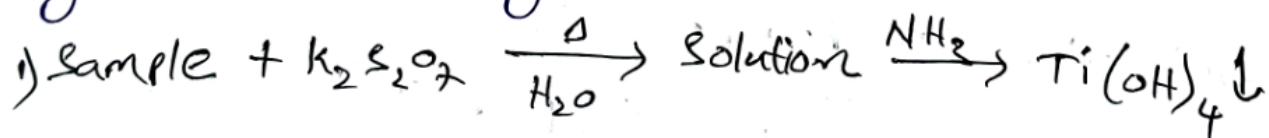
Solution is cooled and add 2ml of Br_2 from the oxidised solution hydroxides are precipitated by the addition of saturated NH_4Cl & NH_4OH .

After boiling the solution for 2 min to expel excess of ammonia. The precipitate is washed with hot water, filtered off and then dissolved in con. HCl. To this solution cold saturated solution of sodium phosphate (or) Ammonium phosphate is added. which is followed by carefully addition of NH_4OH forms permanent precipitate is obtained.

$$\% \text{ of } \text{Al}_2\text{O}_3 = \frac{\text{weight of residue} \times 0.417}{\text{weight of the sample}} \times 100$$

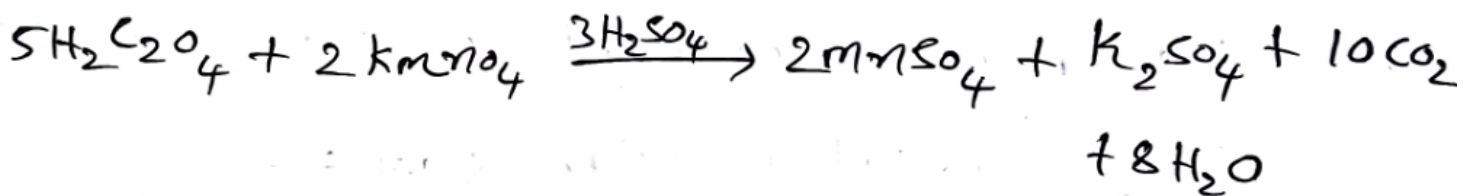
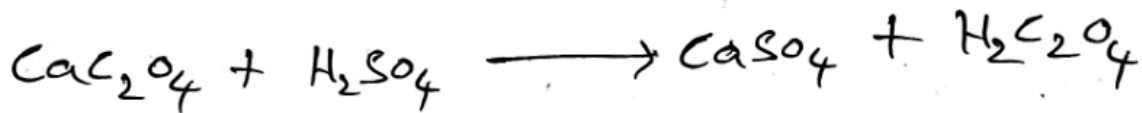
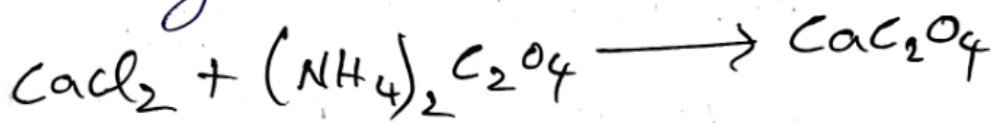
Determination of titania $\{\text{TiO}_2\}$:-

2gm of sample is decomposed by fusion with potassium pyro sulphate and is dissolved in water, HCl & H_2SO_4 . Then the solution is added with excess amount of ammonia, a precipitate of $\text{Ti}(\text{OH})_2$ is formed. which is then filtered and ignited then weighed as TiO_2 .



Determination of CaO (lime):

Volumetric method:- Determination of CaO is based on the formation of CaC_2O_4 by the addition of $(\text{NH}_4)_2\text{C}_2\text{O}_4$ to the solution and then CaC_2O_4 precipitation. It is dissolved in H_2SO_4 and titrated against standard KMnO_4 solution.



one ml of 0.1N $\text{KMnO}_4 = 0.0028$ grams of CaO.

Gravimetric method:-

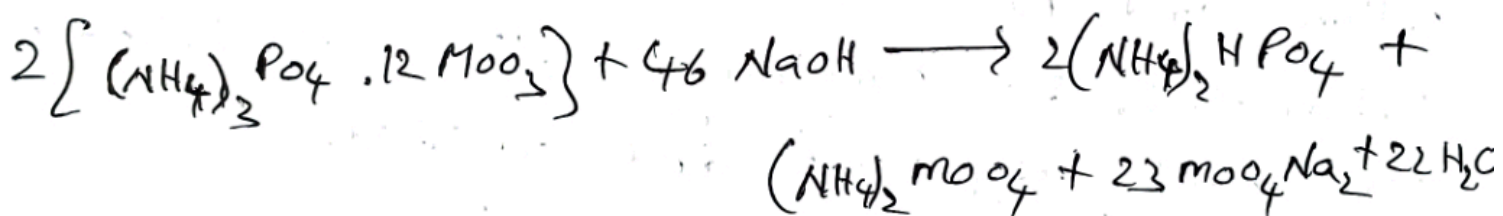
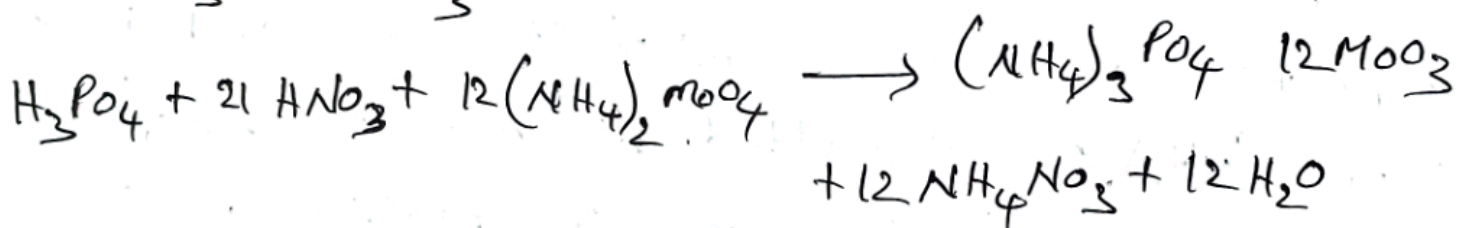
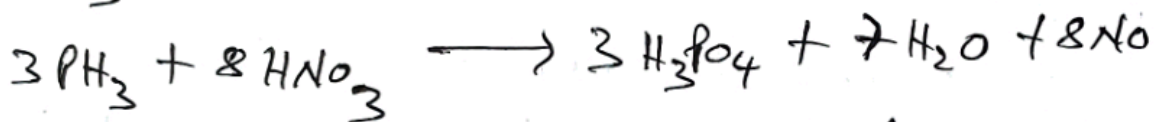
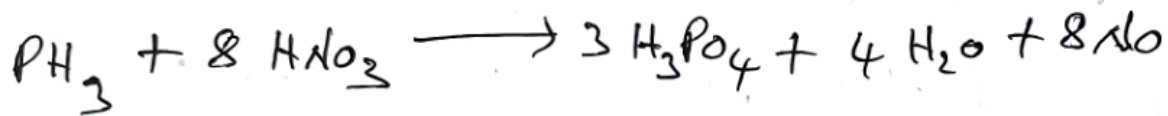
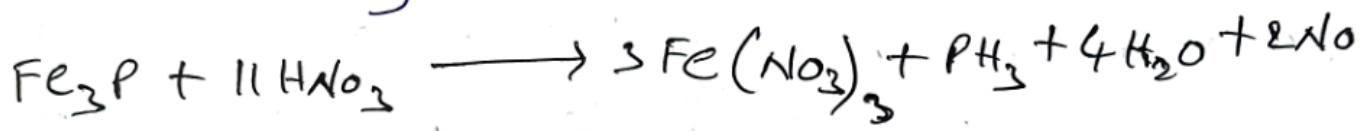
The ppt of CaC_2O_4 is obtained from the addition of ammonium oxalate to the ore sample solution. The ppt is filtered and washed with water containing $\text{H}_2\text{C}_2\text{O}_4$ & Ammonium oxalate. It may be ignited at $500-1200^\circ\text{C}$ and weighed as CaCO_3 (or) CaO.

Determination of Phosphorus:-

Phosphorus in steel exists as iron-phosphide $[\text{Fe}_3\text{P}]$. This is oxidised by HNO_3 to phosphoric acid which is partly in the meta and

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Partly in the ortho condition, the meta phosphoric acid is converted to the ortho condition precipitated as ammonium phosphomolybdate. From this ppt phosphorus can be determined by volumetric method.



$$1 \text{ lit } 46 \text{N NaOH} = 62 (2\text{P})$$

$$1 \text{ ml } 1 \text{N NaOH} = 0.001347 \text{ gm of P}$$

$$= 0.003084 \text{ gm of P}_2\text{O}_5$$

Estimation of MnO_2 :-

The procedure is based on the reduction of MnO_2 and oxalic acid resulting into formation of MnSO_4 in the presence of H_2SO_4 . The amount of $\text{H}_2\text{C}_2\text{O}_4$ required to dissolve available MnO_2 can be estimated by titrating the excess of oxalic

acid with $KMnO_4$.

126 gms of $C_2H_2O_4 = 87$ gms of MnO_2

Determination of Alkalies:-

The ore sample is dissolved in excess HCl , HNO_3 and heating the boiling then add again 1:1 HCl and heated for 15 min and filtered, and the filtrate is added with con. H_2SO_4 . All metals converted into respective metal sulphates and then con. HCl is added and ignited. Then $(NH_4)_2CO_3$ is added. All metals except sodium and potassium are precipitated as their carbonates. The remaining solution is boiled until get salt $[NaCl + KCl]$ and weighed.

$$\% \text{ of Alkali} = \frac{\text{wt of Ppt}}{\text{wt. of sample}} \times \text{factor} \times 100$$

$$\text{factor} = \frac{Na_2O + K_2O}{2[NaCl + KCl]}$$

Estimation of Combined water:-

2gms of sample is taken in a ignited porcelain (or) Alumina boat inserted in a glass tube having stoppers at both ends a suitable desiccant such as H_2SO_4 , $CaCl_2$ etc. is connected at in the

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the tube and the exist end is connected to weighed CaCl_2 tube provided with a gentle suction. The solution is passed along with a local gentle heat to temperature of above 300°C . After heating and suctioned for 15 min, the CaCl_2 tube is reweighed.

$$\% \text{ of Combined water} = \frac{\text{Increase in weight of } \text{CaO}}{\text{weight of Sample}} \times 100$$

Determination of Carbon in blast furnace flue dust and sintex :-

1 gm of sample is taken in a beaker and moistured with 10 ml of hot water and then 0.7 gms of NaF is added. The mixture is boiled to dissolved NaF completely and then add boiling HCl. The solution agitated gently along with gently heat and then diluted with hot water and filtered through a filter paper.

The carbon particles retained on the filter paper are washed thoroughly with 5% HCl and finally with hot water. The carbon is transferred from filter paper to silica ~~crucible~~ crucible, with the help of the fine jet of warm water. The water from carbon is expelled by heating the

crusible on a water bath, when the carbon is completely dried, it is cooled and weighed. The loss in the weight will represent the carbon content.

$$\% \text{ of Carbon} = \frac{\text{Loss in weight}}{\text{weight of sample}} \times 100$$

Determination of Sulphur:-

1 gm of sample digested with a strong HCl, evaporated to dryness. then the sample is oxidised with HNO_3 in presence of BaS_2 , now add a few drops of BaCl_2 then precipitated, filtered off and then washed with dilute HCl, finally ignited and weighed as BaSO_4 .

$$\% \text{ of Sulphur} = \frac{\text{weight of BaSO}_4}{\text{weight of sample}} \times 0.1374 \times 100$$

Determination of MgO:-

Determination of MgO is based on the precipitation of Mg-Ammonium-phosphate, from the slightly acid solution by the addition of diammonium phosphate and then making Alkaline.

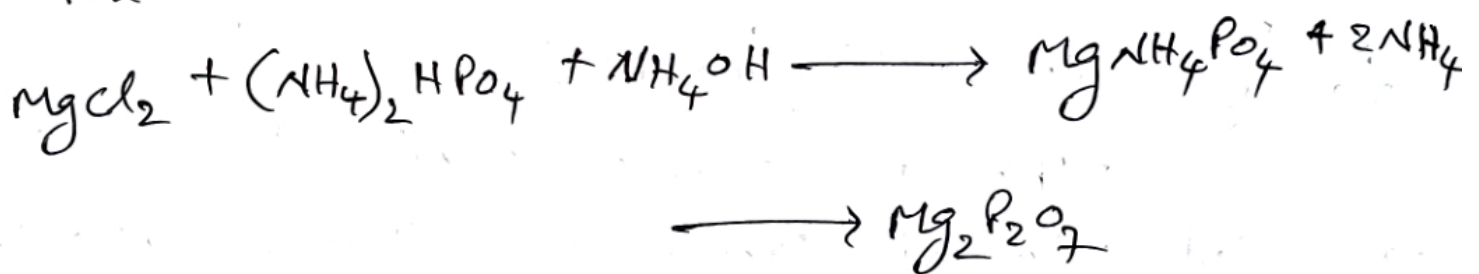
Magnesium-Ammonium-phosphate decomposes to magnesium phosphate on heating.

⑨ which is weighed in gravimetric method and the amount of MgO can be calculated as

$$MgO = 0.3622 \times \frac{\text{weight of } Mg_2P_2O_7}{\text{weight of the sample}}$$

Envolumetric method MgO can also be estimate as by dissolving Mg -ammonium-Phosphate is known quantity of standard H_2SO_4 and then titration bulk the excess of H_2SO_4 with standard $NaOH$ using methyl orange indicator. The amount MgO may calculated as ml of

$$1 \text{ ml of } 0.1N H_2SO_4 = 0.002016 \text{ gm of } MgO$$



Manganese ore :-

Pyrolusite is the main and cheap raw material for the production of pure manganese and manufacturing of ferro manganese alloy. The important constituents analysed in manganese

- ore are ---
- | | | |
|-------------|----------------|-------------|
| i) Total Mn | ii) SiO_2 | vii) CaO |
| ii) MnO_2 | iii) Fe_2O_3 | viii) P & S |
| iii) BaO | iv) Al_2O_3 | |

Sampling & dissolution technique is same as Iron ore.

The remaining constituents except BaO & manganese are also same as Iron ore analysis

Determination of total manganese:-

Total manganese can be estimated gravimetrically as well as volumetric method.

i) Gravimetric method [Pyro phosphate method]:-

In this method is precipitated as ammonium manganese phosphate $[NH_4MnPO_4]$. In slightly ammoniacal solution and then ignited to Pyro phosphate $[Mn_2P_2O_7]$.

The ore sample solution is treated with cold saturated solution of sodium phosphate. In slight excess then the solution is made strongly ammoniacal and boiled till the ppt becomes crystalline. The ppt may be dried at $100-105^\circ C$ and weighed as manganese ammonium phosphate (or) may be ignited at $800-900^\circ C$ and subsequently weighed as manganese phosphate.

$$\% \text{ of manganese} = \frac{0.3873 \times \text{wt. of the Mn phosphate}}{\text{weight of sample}} \times 100$$

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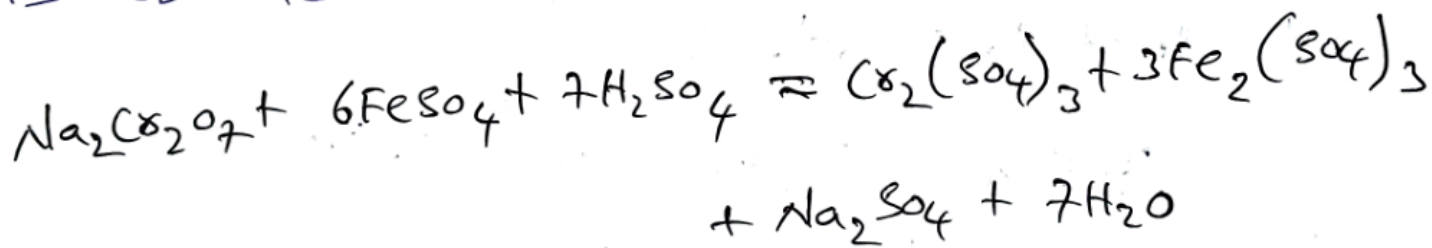
To the filtrate add H_2SO_4 to make the solution acidic.

Add $KMnO_4$ solution until a pink colour persists, Boil the solution until H_2O_2 is expelled before acidifying the chromate solution.

To the solution add a weighed amount of ferrous ammonium sulphate in excess and add the salt gradually by stirring until the yellow colour has disappeared and a drop of liquid gives blue colour with $K_2[Fe(Cr_2O_7)]$ titrate back the excess ferrous ammonium sulphate by

Standard $K_2Cr_2O_7$ (or) $KMnO_4$

The end point in the $KMnO_4$ titration is obtained when the solution becomes pink.



From this equation, it is clear that 335 parts of "Fe" are equal to 152 parts of Cr_2O_7 (or) 104 parts of "Cr".

$$Fe \times 0.3107 = Cr \quad \text{(or)} \quad Fe \times 0.4537 = Cr_2O_7.$$

Feo :-

The sample solution is added to the fused mixture (Na_2O_2) is heating given by the fused mass to dissolve in the distilled water

$\left\{ \begin{array}{l} \text{Cr convert to } \text{Na}_2\text{CrO}_4 \\ \text{Fe ppted to } \text{Fe}(\text{OH})_2 \end{array} \right\}$ is given filter

The residue is dissolved in dil. 20ml Alq. is added D.P.A indicator titrate with 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ then, observe ^{diphenylamine} green to deep violet

sample sol + Na_2O_2 [fused mixture] $\xrightarrow{\text{heat}}$ fused mass
 $\xrightarrow{\text{dissolve}}$ d.w. ^{dry} weight

$\left\{ \begin{array}{l} \text{Cr convert to } \text{Na}_2\text{CrO}_4 \\ \text{Fe ppted to } \text{Fe}(\text{OH})_2 \end{array} \right\} \rightarrow \text{Filter}$

residue dissolve in dil. HCl

20ml of aliquot $\xrightarrow[\text{D.P.A.}]{\text{Titrate}}$ 0.1N $\text{K}_2\text{Cr}_2\text{O}_7 \rightarrow$ green to deep violet

1ml of 0.1N $\text{K}_2\text{Cr}_2\text{O}_7 = 0.007185$ gms of Feo

$$\% \text{ of Feo} = \frac{\text{wt. of } \text{K}_2\text{Cr}_2\text{O}_7 \times 0.007185 \times 100}{\text{wt. of sample}}$$

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Phosphate rock ore :-

Raw material :-

Phosphate rock ore is mainly contains the Tricalcium phosphate, silica, organic metals, $CaCO_3$.

The constituents are

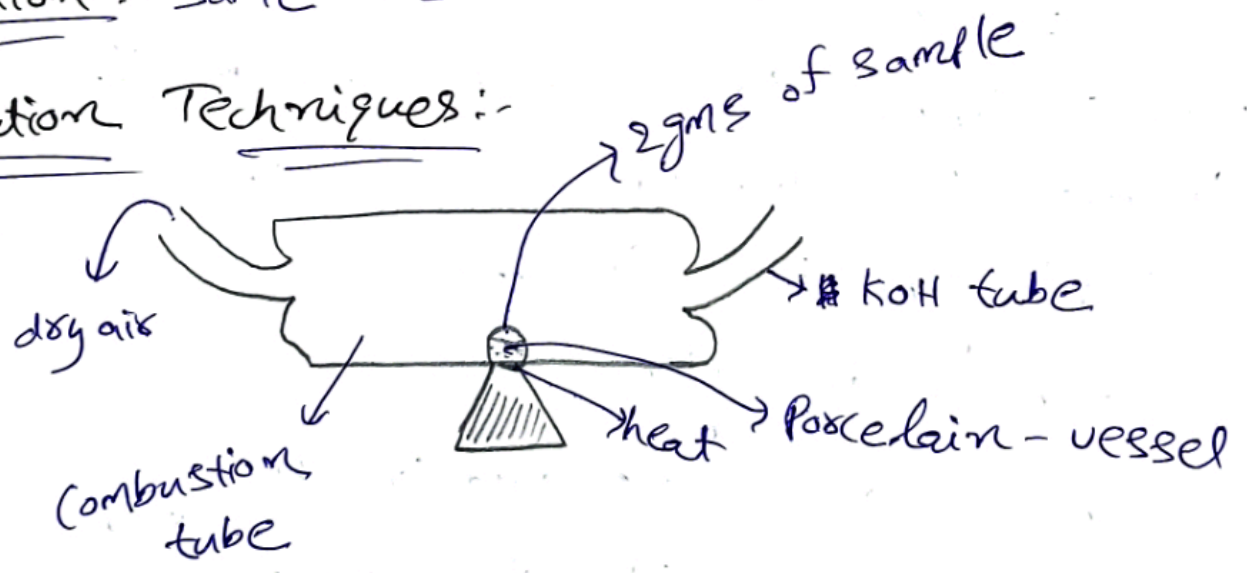
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|-------------|--------------|----------------------|
| 1) CaO | 6) S | 11) K_2O |
| 2) P_2O_5 | 7) Na_2O | 12) Cl |
| 3) F | 8) Al_2O_3 | 13) MnO |
| 4) SiO_2 | 9) Fe_2O_3 | 14) organic carbon |
| 5) CO_2 | 10) MgO | 15) loss of ignition |
| | | 16) moisture |

Sampling :- Same as iron ore

dissolution :- Same as iron ore

Separation Techniques :-

CO_2 :-



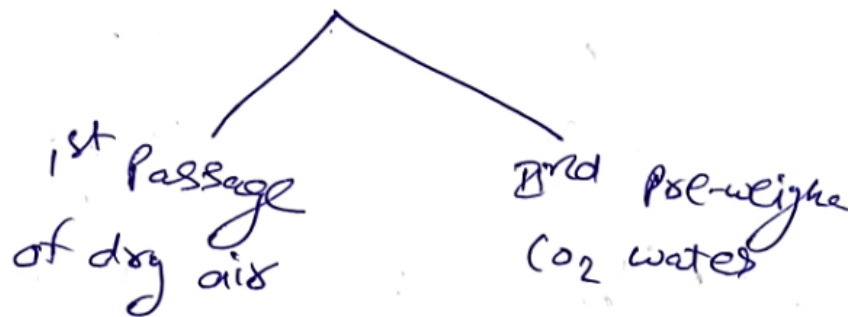
2gms of the sample solution taken in a porcelain vessel & kept in combustion tube having two ends. The passage of dry air

and the second pre weighed CO_2 water.

The sample is heated at $200^\circ\text{C} - 15\text{min}$ is given by the combined water present in the sample convert to water present in the sample convert to water vapour observed.

CO_2 tube is given by the reweight increase in weight is CO_2 .

2gms of sample (or) kept in combustion tube having two ends



Sample $\xrightarrow[200^\circ\text{C} - 15\text{min}]{\text{heat}}$ Combined water present in the sample convert to water present in the sample convert to water vapour observed.

reweight \leftarrow CO_2 tube
Increase in weight $\rightarrow \text{CO}_2$

$$\% \text{ of } \text{CO}_2 = \frac{\text{increased in wt}}{\text{wt. of sample}} \times 100$$

Na_2O \therefore Alkali \therefore Same as iron ore



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k_2O = Total alkali, same as iron ore

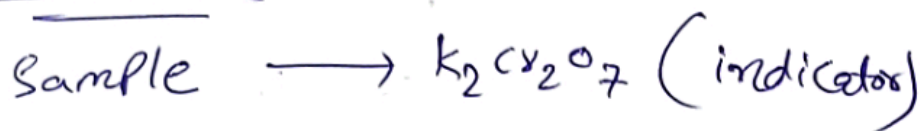
$$k_2O = KCl \times 0.6309$$

organic carbon:-

The loss on ignition substances of the moisture and the addition of carbon dioxide.

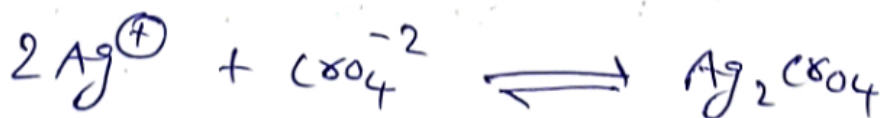
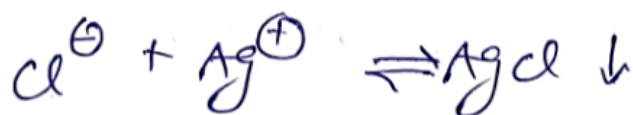
$$\text{loss on ignition} - \{ \text{moisture} + CO_2 \}$$

chloride (Cl):-



NaOH

Adjust pH of 100ml sample is 250ml of C.F to 7-10 drops of H_2SO_4 (or) NaOH, keep the pH at 9 and add 1ml of 5% $K_2Cr_2O_7$ solution stir well. Titrate with 0.0252N $AgNO_3$ solution to a permanent reddish tinge.



Excess

Red



Aluminium ore (Bauxite):-

Raw materials:-

Bauxite ore is the principle ore of Aluminium. To analyse the ore it is first ground to pass a 90 mesh sieve and then free from mechanically combined moisture by heating in an air oven at $105^{\circ}\text{C} - 110^{\circ}\text{C}$.

Constituents to be analysed:-

- | | |
|---------------------------------------|---------------------------|
| 1) Silica | 6) P_2O_5 |
| 2) Alumina | 7) CaO (lime) |
| 3) Fe_2O_3 Total iron | 8) MgO |
| 4) Titania | 9) Vanadium |
| 5) MnO | 10) Zirconium |
| | 11) Alkalies |

Sampling:-

This ore coming from mines and quarries,

The sample solution is reduced to cone and quartering method is decreasing particle size (90 mesh size) is kept in air tight bottle.

dissolution:-

Same as iron ore

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Separation Technique:Si, Al, Ti, P₂O₅, CaO, MgO, Alkalies

Some as iron ore traces.

MnO :- The filtrate obtained after removal of Iron, Aluminium, titanium etc. is taken in 500ml beaker bromine water and a small excess of ammonia are added, to the solution. The solution is boiled and filtered the precipitate is washed with dil. NH₄OH and ignited at a high temperature in a porcelain crucible. The residue is cooled and weighed as Mn₂O₄. From this weight the percentage of Mn and MnO can be calculated.

Vanadium :-

3-4 gms of sample is taken in a 500ml beaker to this 100ml of acid mixture. $\left\{ 350\text{ml d.w} + 250\text{ml } 1:1 \text{ H}_2\text{SO}_4 \right\}$ and $\left\{ 200\text{ml HCl} + 200\text{ml HNO}_3 \right\}$ is added. The beaker is covered and heated to white fumes of SO₂. The resulting mass is cooled and diluted the 100ml d.w and boil to the dissolved salts. The solution is filtered and the residue contains medium paper with pulp. The residue is wash with hot water then filter the solⁿ is adding of the 50ml HCl

is diluted to 300ml d.w to cool at 10°C and also added ice cold cupferron stir then precipitate is allow to stand 10 minutes given by the filter with pulp on a medium paper backed by dense paper.

The ppt is wash with ice cold cupferron solution.

The ppt is along with filter paper is dried ignite & cool & added of Na_2CO_3 (fusion mix) is extracting on hot water is filter.

The residue is wash with hot water.

The filtrate solⁿ is added 10ml of 1:1 H_2SO_4 is heated to boil and cool then add KMnO_4 solⁿ dropwise is strong pink colour then cool at 15°C and also added the excess of 0.1N ferrous ammonium sulphate. ~~was~~ unreacted 0.1N ferrous ammonium sulphate is titrate at temp {15-20°C} is 0.1N KMnO_4 then observe colourless to pink colour.

3-4 gms of sample + 1000ml acid mixture
 500 ml beaker $\left\{ \begin{array}{l} 350\text{ml d.w} + 250\text{ml 1:1 H}_2\text{SO}_4 \\ 200\text{ml HCl} + 200\text{ml HNO}_3 \end{array} \right\}$ $\xrightarrow{\text{converted \& heated}}$ to fumes of SO_3

Resulting mass $\xrightarrow[\text{dilute}]{\text{cool}}$ 100ml d.w \rightarrow dissolve

(15)

Salts \rightarrow Filter with medium paper with pulp

Residue $\xrightarrow[\text{with}]{\text{wash}}$ hot water.

Filter + 30ml HCl $\xrightarrow[\text{to}]{\text{dilute}}$ 300ml d.w \rightarrow Cool + Ice cold

Effusion $\xrightarrow{\text{stir}}$ ppt $\xrightarrow[\text{stand 10min}]{\text{allow to}}$ filter with pulp on a

medium paper backed by dense paper.

ppt along with filter paper $\xrightarrow{\text{direct}}$ Cool +
ignite (600°C)

Na_2CO_3 (fusion mixture) $\xrightarrow[\text{in}]{\text{extending}}$ hot water \rightarrow Filter

Residue $\xrightarrow[\text{with}]{\text{wash}}$ hot water.

Filterate + 10ml 1:1 H_2SO_4 $\xrightarrow[\text{to boil}]{\text{heat}}$ Cool + KMnO_4 soln

(dropwise) \rightarrow Strong Pink colour \rightarrow Cool at 15°C

+ excess 0.1N ferrous ammonium sulphate

unreacted 0.1N ~~KMnO_4~~ ~~colourless to pink~~

Ferrous ammonium sulphate $\xrightarrow{\text{Titrate}}$ 0.1N KMnO_4

\rightarrow colourless to pink.

$$\% \text{ of Vanadium} = \frac{\text{Vol. of } \text{KMnO}_4 \times 0.005095 \times \text{Abs. of } \text{KMnO}_4}{\text{wt of sample}} \times 100$$

Zirconium :-

3 to 4 gms of sample (or) ore is taken in a platinum crucible to this add 10ml of HNO_3 + 20ml of HF + 20ml H_2SO_4 is evaporated & cool

To this add some of water and 20ml of HCl. The mixture is heated to dissolve salts. If any insoluble residue is retained, it is fused with Sodium carbonate Borax mixture and extracted in dil. H_2SO_4 . The solution is added to the filtrate. The solution is adjusted to 200ml and cool at $10^\circ C$ to this ice cold cupferron solution is added until the ppt turns reddish brown ppt (partial ppt of Iron). The solⁿ is allowed to stand for 10min. The ppt is washed with ice water cold are transferred to Platinum crucible and burned off the carbon at $600^\circ C$. The residue is fuse with $KHSO_4$ and dissolve in 70-80ml of 10% H_2SO_4 . The filter of the residue is wash with hot water.

The filtrate of the solⁿ is added the 5ml of 5% H_2O_2 + 10ml 10% of $(NH_4)_2PO_4$ is allowed to stand for 10-12 hours at $60^\circ - 70^\circ C$ the ppt form is filtered and washed with cold ammonium nitrate solution. The paper and the ppt are ignited in a Porcelain crucible at $1100^\circ C$. The residue is cooled & weighed

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as $Zr_2P_2O_7$.

3-4 gms of sample + 10ml of HNO_3 + 20ml of H.F

(Pt (soluble))

+ 20 ml H_2SO_4 ~~evaporated~~ (cool + some water + 20ml

HCl heat) dissolve the salts → Adjusted to

20ml cool to $10^\circ C$ } if any residue is remains there is fused with Na_2CO_3 borax mixture & extracted in dil. H_2SO_4 & it is add to filtrate

+ ice cold Cupferron solⁿ → reddish brown ppt (partial pptn of iron)

→ allowed to stand for 10min + paper pulp is stirred in → The ppt along with paper pulp is filtered off with suction on a medium filter paper → The ppt is wash with ice cold Cupferron solⁿ → Filter paper.

→ Charred & burned of Carbon at $600^\circ C$.

→ The residue is fuse with $KHSO_4$ and is dissolve in 70-80ml of 10% H_2SO_4 → Filter

→ residue is wash with hot water.

Filtrate + 5ml of 5% of H_2O_2 + 10ml

of 10% $(NH_4)_2PO_4$ → allow to stand for 10-12 hrs

at $60-70^{\circ}\text{C}$ \rightarrow ppt formed \rightarrow Filter with medium
Filter Paper \rightarrow wash with cold ammonium
Nitrate solⁿ \rightarrow the paper & the ppt is
ignited in a Porcelain crucible at 1100°C
 \rightarrow lose due to cooled & weighed as $\text{Zr}_2\text{P}_2\text{O}_7$.

$$\% \text{ of } \text{Zr}_2\text{O}_2 = \frac{0.4647 \times \text{weight of } \text{Zr}_2\text{P}_2\text{O}_7}{\text{wt. of sample}}$$