

ANALYSIS OF IRON ORES

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INTRODUCTION

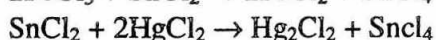
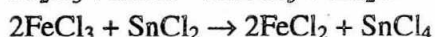
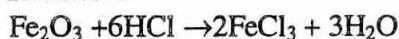
The important minerals that constitute iron ore hematite (Fe_2O_3) and magnetite (FeO , Fe_2O_3). other associated minerals are hydrates of iron like goethite. The radicals determined in iron ores are Fe(Total), FeO, Fe_2O_3 , Al_2O_3 , CaO, MgO, SiO_2 , P, S, LOI. Determination of these radicals are given below :

Determination of Total Iron

About 0.1g of ore sample was taken in a conical flask and 30 ml of conc. HCl and a few ml of water were added. Digestion was carried for 2 hr. The conical flask was boiled for 5 minutes and SnCl_2 was added in hot condition till yellow color disappears completely. The solution was cooled then about 10ml of HgCl_2 solution was added. A silky white precipitate was observed. About 20 ml of mixture $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$, a few drops of Barium diphenyl amine sulphonate (BDS) indicator were added. The solution was titrated against 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution. The presence of iron was known by its intense violet colour.

$$\begin{aligned} 1\text{ml of } 0.1\text{N } \text{K}_2\text{Cr}_2\text{O}_7 &= 0.05585\text{g of Fe} \\ \text{Total Fe \%} &= \frac{\text{Burette reading} \times 0.56}{\text{sample weight in gm}} \end{aligned}$$

Reactions

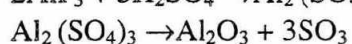
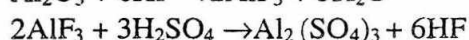
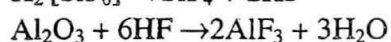
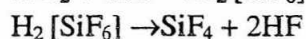
Determination of Silica (SiO_2)

About 0.5g of ore sample was taken in a 250 ml beaker and 30 ml of conc. HCl was added. The mixture was digested for 1 hr on a hot plate. A few drops of HNO_3 was added. The solution was evaporated to dryness completely. The mass was baked for 1 hr. The dried mass was cooled and again about 20 ml of conc. HCl and 30 ml of water were added. The solution was boiled and filtered through Whatman 40 paper with pulp in 250 ml in a volumetric flask. The residue was washed thoroughly with hot water till yellow colour disappears. Then the residue was ignited in a platinum crucible on a burner and then in a furnace at 1000°C for half an hour to a white ash. The crucible was cooled and weighed (W1). A few drops of water and 3 drops of H_2SO_4 and 15ml of HF were added. The solution was evaporated on a hot plate and then

ignited in the furnace at 950°C. The crucible was cooled and taken the weight (W₂). Add KHSO₄ in the crucible, melt it on a burner and extracted in 1:3 HCl. The solution was mixed in the original filtrate and the volume was made up to 250ml.

$$\% \text{ Silica} = (W_1 - W_2) \times 100 / \text{Sample wt.}$$

Reactions



Determination of alumina (Al₂O₃)

About 25 ml aliquot from the flask (after silica filtration), 5g of NH₄Cl in hot condition and NH₄OH were added. The solution was filtered through Whatman 41 paper and washed with hot water. The precipitate was dissolved in conc. HCl and hot water was added till yellow colour disappears. Filtrate was acidified with HCl and 25 ml of 0.01M EDTA was added. Few drops of xylenol orange indicator was added. The pH 5.5 was adjusted using NH₄OH and HCl in 1:3 ratio. About 10ml of ammonium acetate buffer of pH 5.0 was added, boiled and cooled. It was titrated against 0.01M zinc acetate trihydrate. Lemon yellow colour was turned into pink.(T1) 10 ml of 10% NH₄F solution was added. It was boiled cooled and titrated against 0.01M Zn²⁺ solution with pink colour end point.(T2).

$$\% \text{ of Alumina} = [2^{\text{nd}} \text{ titre value} \times 0.513] / \text{Sample wt}$$

Determination of FeO

About 0.5g of sample was taken in 500 ml conical flask and was mixed thoroughly with a few grams of Na₂CO₃. The funnel filled with Na₂CO₃ was placed on the top of flask to avoid the contact with air. 20 ml of HCl was added and digested slowly in hot plate till no black particle in the mixture. Distilled water free from dissolved O₂ (200ml) was added. About 20ml of acid mixture and few drops of BDS were added. The solution was titrated against 0.01N K₂Cr₂O₇. The presence of FeO content was indicated by its violet colour.

$$1\text{mL of } 0.01\text{N K}_2\text{Cr}_2\text{O}_7 = 0.07185\text{gm}$$

$$\% \text{ of FeO} = \text{Burette reading} \times 0.072 / \text{Sample wt.}$$

Determination of Metallic Iron (Fe⁰)

About 0.1g of sample was taken in 250mL volumetric flask, 3g of HgCl₂ was added and mixed thoroughly. 50mL of water was added and boiled for 5 minutes on hot plate. The solution was cooled in ice bath. The solution was made to 250 ml with boiled water and filtered through Whatman 40 paper in 100 ml volumetric flask. 20 ml of acid mixture and few

drops of BDS were added and titrated against 0.1N K₂Cr₂O₇. The presence of FeO was indicated by its violet colour.

$$\% \text{ of Metallic iron} = (0.56 \times BR \times 2.5) / \text{Sample wt.}$$

Determination of CaO and MgO

About 50ml of aliquots in two portions were taken in 25 ml each after silica filtration. The solutions were heated and NH₄Cl and NH₄OH were added. The solutions were filtered in Whatman 41 paper. The precipitate was washed by hot water. The filtrate was taken in 100 ml conical flask and 10 ml NH₄ buffer of pH 10 was added. Erichrome black T indicator was added and titrated it against 0.01M EDTA. The presence of Ca + Mg was known by ink colour as end point. About 10 pellets of NaOH, patton & reeders were added in to another flask (after R₂O₃ separation) The solution was titrated against 0.01M EDTA. The presence of Ca was indicated by ink colour.

EDTA used for CaO + MgO	=	Xml
EDTA used for CaO	=	Yml
EDTA used for MgO	=	(X-Y)ml
% of CaO	=	(Y × 0.056)/Sample wt.
% of MgO%	=	[(X-Y) × 0.040]/Sample wt.

Determination of Phosphorous

Take 1.0 gm sample, into a 400 ml beaker. Digest with 35 to 40 ml of concentrated hydrochloric acid on a hot plate or sand bath until the ore is attacked completely. Evaporate the solution just to dryness. If the sample contains arsenic, add five ml of concentrated hydrochloric acid and 0.5 to 1 ml of ammonium bromide and evaporate to dryness. Repeat the procedure to expel all arsenic. Add 20 ml of concentrated nitric acid and digest under cover and evaporate to syrupy consistency. Add 15 ml of nitric acid and again repeat the same. Take up the contents in 10ml of concentrated nitric acid and digest until the soluble matter has dissolved. Dilute to about 100 ml with water and filter through a No.41 filter paper. Wash the residue 5-6 times with hot dilute nitric acid solution. Preserve the filtrate.

Dry and ignite the residue in a platinum crucible until all the carbon is burnt off. Cool and moisten the residue with a few drops of water. Add two to three drops of dilute sulfuric acid and 7-8 ml of hydrofluoric acid. Evaporate to dryness. Cool and fuse the residue with one to two grams of sodium carbonate for 10 to 15 min. Cool and extract with 70 to 80ml of hot water containing 8-10 ml of concentrate nitric acid. Wash in the crucible and lid. If any insoluble remains, filter through a No. 40 filter paper and wash with hot water 5-6 time. Combine this solution with the filtrate preserved and concentrate to 150 ml.

Cool and neutralized with concentrated ammonium hydroxide until a slight persistent precipitate is formed. Redissolve the precipitate in few drops of concentrated nitric acid, add 1-2 ml in excess, and heat to 70°c-80°c. If the sample contains vanadium, add 2ml hydroxylamine hydrochloride solution. Add with stirring 30-40 ml of ammonium molybdate solution; 70 ml

for every 0.1 gm of phosphorus pentoxide. Continue stirring till the phosphorus is precipitated. Keep at 50 °c to 60 °c for 1 hour.

Filter the precipitate through a thick pad of filter paper pulp prepared on a perforated disk in a funnel. Wash the beaker and the precipitate 2-3 times with dilute nitric acid solution and then with potassium nitrate wash solution till 10 ml of filtrate collected in a test tube does not consume more than 1 drop of 0.1 N sodium hydroxide solution, using a drop of phenolphthalein indicator solution. Transfer the pad of paper pulp with the precipitate back to the beaker. Add 50 to 60 ml of water add with a jet of water, into the same beaker. Add 50-60 ml and add with stirring standard sodium hydroxide solution from a burette until all the five-ml of sodium hydroxide solution in excess and stir. Add 3-4 drops of phenolphthalein indicator solution and titrate with standard hydrochloric acid until the pink colour is completely discharged.

Carry out a blank on the same quantity of sodium hydroxide solution which was added in the test, and titrating with standard hydrochloric acid, using 3-4 drops of phenolphthalein indicator solution.

Calculation

$$\text{Phosphorus, percent} = \frac{(A-B) \times C \times 0.00135 \times 100}{D}$$

Where

- A = Volume in ml of standard
- B = Volume in ml standard hydrochloric acid required for the sample
- C = Normality of standard hydrochloric acid, and
- D = Weight in g of the sample taken.

Determination of Sulphur by Gravimetric Method

Weigh accurately about 5 g of the sample into a 400ml beaker. Add 1 g of Potassium bromide and 4-5 ml of water to dissolve the bromide. Add about 0.5 ml of liquid bromine and stir gently. Set the solution aside for about 15 minutes with occasional stirring. Now add 35-40 ml of concentrated hydrochloric acid and cover the beaker with a watch glass. Digest on hot plate until all the ore is attacked. Wash in the watch glass and evaporate to dryness. Bake the residue for about an hour and cool.

Take up the residue in 10 ml of concentrated hydrochloric acid by warming. Dilute to about 100ml boil, filter through a No. 41 filter paper and wash with hot dilute hydrochloric acid solution 5-6 times. Preserve the filtrate and concentrate to about 200ml. Neutralized the solution with concentrated ammonium hydroxide until a faint persistent precipitate is formed. Redissolve the precipitate in the minimum amount of concentrated hydrochloric acid and add 7-8 ml in excess.

Add 5 to 6 g of granulated Zinc to the solution obtained and warm on steam bath until all iron is reduced to the ferrous state and evolution of hydrogen ceases. Filter through a No.42 filter

paper and wash with hot dilute hydrochloric acid solution three to four times. Concentrate the solution and adjust the acidity so that it contains two percent by volume of hydrochloric acid. Warm it to 60-70 ° C and add 10ml of barium chloride solution dropwise with constant stirring. Let the solution stand for 2 hrs on hot plate and overnight at room temperature. Filter through no.42 filter paper and wash the precipitate with cold dilute HCl and then with hot water until free from chloride. Dry and ignite the precipitate in weighed platinum or silica crucible. cool in a desiccator and weigh as barium sulphate.

Calculation

$$\text{sulphur \%} = \frac{A \times 0.1374 \times 100}{B}$$

A = weight in gm of barium sulphate obtained
B = weight in gm of sample