Experiment 4: Estimation of Iron in haematite ore solution using standard $K_2Cr_2O_7$ solution by External Indicator method

Significance of the experiment*:* Iron and its alloys that demonstrate exceptional strength and hardness are used in making wide range of useful things that we come across in our daily life. Modern construction, transportation, and manufacturing industries are greatly dependent on Iron. Iron is not directly available as a pure metal from the earth's crust. However it is available as a mineral or ore that contains combination of elements, which is mined and refined to obtain pure metal. Before mining and refining of mineral/ore can start it is important to analyze the percentage of metal present in a particular mineral/ore of interest.[1]This information is important to choose the right mineral/ore (containing highest percentage of metal) among several available ores/minerals. Furthermore, this information is essential to estimate the commercially profitability/viability of extraction and refining of the mineral/ore. In this particular experiment you are only interested in the estimation of percentage of iron in one of the important ores of iron called Haematite that contains mainly Fe_2O_3 with little amount of silica (SiO_2).[2],[3] There are several ways of determination of percentage of iron in the given haematite ore. However we will use a simple and easy analytical method in this lab in which haematite is first treated with 1:1 hydrochloric acid followed by removal of insoluble silica through filtration. The resulting filtrate is then used for the analysis.

Aim : To determine percentage of iron in a given sample of haematite ore solution using standard $K_2Cr_2O_7$ solution.

Principle : The method uses potassium dichromate, an oxidizing agent that oxidizes Fe (II) to Fe(III) for the estimation of iron. First concentrated hydrochloric acid dissolves haematite ore to reacts with Fe_2O_3forming $FeCl_3$ (reaction 1). Note here the oxidation states of Fe in $Fe_2O_3$and $FeCl_3$are +3 and +3 respectively.

$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O$….. (1)

Next, $FeCl_3$ is reduced to $FeCl_2$ using reducing agent $SnCl_2$(reaction 2). Here the oxidation state of Fe in $FeCl_3$ and $FeCl_2$ are +3 and +2 respectively. $SnCl_2$ (Sn^{+2}) is oxidized to $SnCl_4$ (Sn^{+4}). Non-availability of stable reducing agents for direct titration has led us to follow a strategy that is being used here.

$2\ FeCl_3 + SnCl_2 \rightarrow 2\ FeCl_2 + SnCl_4$…..(2)

After the reduction, there should not be any unreacted $SnCl_2$(if it not removed it will lead to error in the estimation of Fe because potassium dichromate oxidizing agent will react with $SnCl_2$ along with $FeCl_2$). So the unreacted $SnCl_2$is reacted with $HgCl_2$ (Hg oxidation state is +2) resulting in the formation of insoluble Hg_2Cl_2(Hg oxidation state is +1) and $SnCl_4$(which can not be further oxidized) [reaction 3].

$SnCl_2 + 2\ HgCl_2 \rightarrow Hg_2Cl_2\downarrow + SnCl_4$ ….. (3)

Finally an oxidizing reagent potassium dichromate (containing $Cr_2O_7^{2-}$ions in solution) oxidizes $FeCl_2$ (Fe^{2+}) to $FeCl_3$ (Fe^{3+}). This reaction takes place in the presence of acid (HCl). The Cr gets reduced, $Cr_2O_7^{2-}$(Cr^{6+}) to $CrCl_3$ (Cr^{3+}) in this reaction (reaction 4).

$6\ FeCl_2 + K_2Cr_2O_7 + 14HCl \rightarrow 6FeCl_3 + 2CrCl_3 + 7H_2O + 2KCl$ ……(4)

Procedure:

Part A :Preparation of standard solution of $K_2Cr_2O_7$

Weigh potassium dichromate crystals accurately and transfer into a 250 cm^3 volumetric flask. Dissolve in ion exchange water and dilute up to the mark and mix well. Calculate the normality of potassium dichromate.

Part B: Estimation of Iron

Pipette out 25 cm^3 of the haematite ore solution into a clean conical flask. Add a 3-4 ml concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colourless. Add 1 more drops in excess to ensure complete reduction. Cool and add a quater test tube of mercuric chloride rapidly and mix well. A silky white precipitate of mercurous chloride is formed.

Place a number of drops of freshly prepared potassium ferricyanide indicator on a wax paper. Add a small quantity of potassium dichromate from the burette to the conical flask containing haematite solution and mix well. Remove a drop of the solution from the conical flask and bring it in contact with a drop of the indicator on the wax paper. The colour of the indicator turns blue. Repeat this operation after adding 1 cm^3 more of the potassium dichromate solution and again bring a drop of the mixture in contact with a fresh drop of the indicator. The indicator turns blue as long as the titration is incomplete. Continue the titration by adding increments of 1cm^3 of $K_2Cr_2O_7$ at a time and testing as above till a drop of the mixture fails to produce any colour with the indicator drop.

(Note: Clean the glass rod after every test). Repeat the titration by taking another 25 $cm^3$of the haematite solution. This time add most of the potassium dichromate solution required at a stretch and then titrate drop wise. Mix the content of the flask after every addition and test a drop of the titrated solution with a drop of the indicator as described above till the colour of the indicator does not change.

Result:

The percentage of iron in the given haematite ore solution is ……

Links to the external sources of information about the topic:

1.http://en.wikipedia.org/wiki/Ore

2.http://en.wikipedia.org/wiki/Hematite

3. http://www.oocities.org/capecanaveral/Hall/1443/iron.html

Experiment 4: Observation and Calculations

Part A: Preparation of standard solution of Potassium dichromate ($K_2Cr_2O_7$).

1. Weight of bottle + $K_2Cr_2O_7$ crystals= W_2 = ---------- g

2. Weight of empty bottle = W_1 = ---------- g

3. Weight of $K_2Cr_2O_7$ crystals = W_2-W_1 = ----------g

$$Normality\ of\ K2Cr2O7 = \frac{Weight\ of\ the\ salt\ (W2-W1)\ X\ 4}{Equivalent\ weight\ of\ K2Cr2O7\ (49)} = \quad \text{.....(a)}$$

Part B: Estimation of iron

Burette reading	Pilot reading	**Trial** I	**Trial** II
Final reading			
Initial reading			
Volume of $K_2Cr_2O_7$ run down (mL)			

Volume of $K_2Cr_2O_7$ consumed = ---------- mL (*b*)

Weight of Haematite ore in 250 mL = ---------- g (*W*) (‘*W*’ will be provided to you).

1000 mL of 1N$K_2Cr_2O_7$ = 55.85 g of iron (1 equivalent of Fe = 55.85 g).

`b'* mL of *`a' N $K_2Cr_2O_7$ = $\frac{55.85\ xaxb}{1000\ x\ 1}$ of Fe = ---------- g (*c*).

25 mL of haematite ore solution contains = ---------- g of Fe (*c*).

250 mL of haematite ore solution contains = 10 x c ---------- g of Fe (*`d '*).

Percentage of iron in given haematite ore sample = $\frac{d\ X\ 100}{Weightofhaematiteore\ (W)(g)}$

Result:

The percentage of iron in the given haematite ore solution is ……