

Experiment No.4

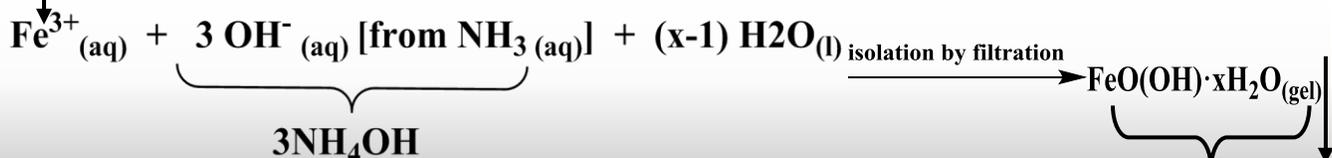
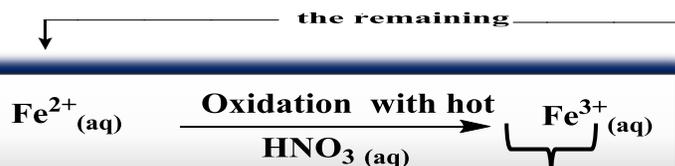
Determination of Iron as Ferric Oxide (Fe_2O_3)

Introduction

The gravimetric technique can be used to determine the percent weight (wt.%) of Fe in an iron sample.

A sample containing iron ions of ferric oxidation state, then a hydrated ferric oxide is precipitated from solution by adding ammonia used as precipitating reagent, and finally dehydrated by ignition to produce Fe_2O_3 .

Precipitation of ferric hydroxide is a gelatinous state in hot solution, so we heated solutions to avoid colloid solution. All ferrous ions must be oxidized to ferric form by using one of the strong oxidizing agents like (HNO_3 , H_2O_2 , and Br_2 precipitation equations as follows:



Materials

- 1- A sample containing iron salt as $[\text{Fe}(\text{NO}_3)_3, \text{Fe}_2(\text{SO}_4)_3, \text{FeCl}_3]$.
- 2- Diluted hydrochloric acid $\text{HCl}(1:1)$ and concentrated nitric acid (HNO_3).
- 3- Diluted ammonia solution.
- 4- NH_4NO_3 1% solution.
- 5- Watch glass, beaker, ashless filter paper and balance.

Procedure

- 1- Weight (0.4) g of ferric salt in (400mL) beaker clean and dry.
- 2- Dissolve sample with 30mL of distilled water, then add (5mL) HCl .
- 3- Heat the solution until boiling, add (1mL) of HNO_3 gradually then heat for a few minutes to ensure that all $\text{Fe}(\text{II})$ is oxidized to $\text{Fe}(\text{III})$ and the solution became clear yellow.
- 4- Dilute the sample to (100mL) by distilled water then heating the solution until boil after that (20-25mL) of ammonia solution adding slowly with stirring until the odor of excess ammonia will be smells.
- 5- Boil the solution for (1min), then leave it to room temperature for digestion, the precipitate will settle and the solution must be colorless.
- 6- Filtrate the solution slowly using ashless filter paper (Whatman 41, 110-mm diameter) then washing the precipitate with solution of hot NH_4NO_3 until Cl^- ions will be remove from the precipitate (Tested by AgNO_3).
- 7- Weight ceramic crucibles after heating for (15min) to redness degree and cooled to room temperature.
- 8- Transfer the precipitate and filter paper to the crucible and ignite firstly by benzene burner to charring filter paper, then transfer crucible to furnace at 500°C

Calculation

$$\text{Fe}\% = \frac{\text{Wt. Fe}}{\text{Wt. of sample}} \times 100$$

$$\text{Wt. Fe} = \text{G.F} \times \text{Wt ppt (Fe}_2\text{O}_3)$$

$$\text{G.F} = \frac{\text{A.Wt. (Fe)}}{\text{M.Wt. (Fe}_2\text{O}_3)} \times \frac{2}{1}$$

Discussion

- 1- What is the effect of ammonium hydroxide addition to the ferric salt solution? Explain with equations?
- 2- What is the chemical formula of precipitated and salt iron?
- 3- A sample of magnetite, not pure Fe_3O_4 weighed 0.5g converted chemically reaction to the ferric oxide weight 0.41g What is the percentage of Fe_3O_4 in the magnetite?
- 4- What is the purpose of concentrated HNO_3 addition the produced solution from the added of HCl to ferric salt?