To devise a volumetric procedure to determine the percentage of iron(ii) and iron...



Iron is a transition element, and all transition elements may be found in a variety of oxidation states, for example iron exists as both, Iron(II) and Iron(III). These kinds of elements can react with both oxidising and reducing agents due to the fact that they can be converted from one oxidation state to another.

Working out the percentage composition of both of the Iron ions will require two separate titrations. One of the titrations will react with only one of the ions however the other will react with the mixture as a whole. To be able to carry out a titration in which of all the mixture will react, a preliminary reaction will need to be done. Potassium Manganate (KMnO4) is an oxidising agent and will react with Iron(II) but will not react with Iron(III). This can be therefore used to work out the percentage of Iron(II) in the solution. To work out the percentage composition of Iron(III) a separate reaction will need to be carried out first. The Iron(III) will first need to be reduced to Iron(II) by reacting the solution with Zinc. The reaction can then be titrated with Potassium (KMnO4). The result can then be used to work out the new percentage of Iron(II) present. From the results of the two titrations, the percentage composition can be found.

I am told that the solution provided contains between 1. 1g and 1. 3g of iron ions as a mixture of Fe2+ (aq) or Fe3+ (aq). I have therefore decided to use the average mass of 1. 2g.

The approximate concentration of the iron solution is :-

No. of Moles of Iron = Mass/RMM of iron

= 1.2/55.8

= 0.0215

Therefore the concentration of Iron is :-

Concentration = $(No. of Moles \times 1000)/Volume$

 $= (0.0215 \times 1000)/25 \text{ cm}$

= 0.86 mol dm -3

The concentration of the second titration in which the Iron(III) is reduced to Iron(II)

could be calculated using the approximate concentration of iron solution and the mole ratio found in the equation. For the first titration in which the Iron(II) is reacting an approximate percentage composition will need to be found, which will allow the same concentration of Potassium Manganate to be used.

The approximate concentration of MnO 4- for second titration is: -

Concentration = 1/5 concentration of iron solution 0. 86

= 0.172 mol dm -3

Therefore I will be using 0. 2M of MnO 4- for both of my titrations.

TITRATION OF IRON(II) WITH POTASSIUM MANGANATE

Apparatus:

- * Burette
- * 25 cm3 pipette
- * Clamp stand
- * Conical flask
- * Beakers
- * Funnel
- * White tile
- * 30 cm3 measuring cylinder

Chemicals:

- * 200 cm3 of mixture containing both Iron(II) and Iron(III)
- * 100 cm3 of Potassium Manganate (0. 2M)
- * 80 cm3 of Sulphuric acid (1M)

Method:

Carefully wash out the burette and clamp the burette using. Make sure the tap is closed and fill with Potassium Manganate using a funnel. Open the tap and let some of the Potassium Manganate run out into a beaker to fill the jet of the burette to make sure that there are no air bubbles. Record the initial volume of the Potassium Manganate in the burette, reading from the bottom of the meniscus and record to two decimal places.

Carefully place the pipette into pipette filler and place it in the iron solution. Turn the thumb dial to suck up the solution. Suck up more solution then what is needed. Remove the pipette filler and cover the top of the pipette with your index finger and apply pressure. By decreasing the pressure the level of the solution will fall. Transfer the solution into a conical flask. Touch the end of the pipette against the inside of the conical flask to release the remaining solution. Add about 20 cm3 of dilute sulphric acid to the solution in the conical flask. Place the conical flask on the white tile underneath the jet of the burette and allow about 2 cm3 of the Potassium Manganate out into the conical flask and swirl the solution. As soon as the solution turns pink, close the tap and record the volume of Potassium Manganate used. Use this as the trial titration.

Refill the burette as before recording the initial volume and wash out the conical flask using distilled water and fill as you did before. Repeat the titration before, however when within 2 cm3 of the volume recorded for the trial titration, slow the flow of the Potassium Manganate so it is coming out drop by drop. This will ensure the highest level of accuracy. Repeat this process until you obtain results that are within 0. 1 cm3 of each other.

TITRATION OF IRON(III) WITH POTASSIUM MANGANATE

$$2Fe3+ (aq) + Zn (S) ---> 2Fe2+ (aq) + Zn2+ (aq)$$

Apparatus:

* Burette

* 25 cm3 pipette

* Clamp stand
* Conical flask
* Beakers
* Funnel
* White tile
* 30 cm3 measuring cylinder
* Glass Rod
* Filter paper
* Bunsen burner
* 10 cm3 measuring cylinder
Chemicals:
* 200 cm3 of mixture containing both Iron(II) and Iron(III)
* 100 cm3 of Potassium Manganate (0. 2M)
* Finely divided Zinc
* Potassium Thiocyanate solution
Method:

Pipette 25 cm3 of the iron solution into a conical flask. Add about 25 cm3 of sulphuric acid as well as adding 1 or 2 spatulas of the finely divided zinc. Heat over a gentle Bunsen flame until effervescence has almost ceased, this should take about 10-15 minutes. When a drop of the solution drawn out on a glass rod no longer gives a red precipitate with Potassium thiocyanate solution so Iron(II) is no longer present.

$$Fe3+ (aq) + KCN- (aq) ----> (FeKCN) 2+ (aq)$$

Filter the iron solution and rinse the remaining zinc, adding the rinsing to the filtrate. Titrate the iron solution with Potassium Manganate and record the result using the same techniques as in the previous titration.

Result of Titration of Iron(II):

Trial

TEST 1

TEST 2

TEST 3

Burette reading (cm3)

Final burette reading (cm3)

Initial burette reading (cm3)

Volume of solution used (cm3)

Average Titer:
Result of Titration of Iron(III):
Trial
TEST 1
TEST 2
Test 3
Burette reading (cm3)
Final burette reading (cm3)
Initial burette reading (cm3)
Average Titer:
Calculations:
* Titration 1. Iron(II)
Moles of MnO4- used = (volumes x concentration)/1000
= (volume x 0. 02)/1000
= A
Moles of Iron(II) in 25 cm3 = $A \times 5$ (using mole ratio)
= B

Moles of Iron(II) in 200 cm3 = $B \times 8$

= C

Mass of Iron(II) in solution = Moles x RMM of iron

 $= C \times 55.8$

= D

* Titration 2. {Iron(II) and Iron(III)}

Moles of MnO4- used = (Volume x concentration)/1000

 $= (Volume \times 0.02)/1000$

= E

Moles of iron(II) in 25 cm3 = $A \times 5$ (using mole ratio)

= F

Moles of iron(II) in 200 cm3 = $B \times 8$

= G

Mass of iron(II) in solution = Moles x RMM

 $= C \times 55.8$

= H

Mass of iron(III) present = H - D

- * The Percentage Composition by mass for each iron :
- 1. $Iron(II) = (D/H) \times 100$
- 2. $Iron(III) = (X/H) \times 100$

Health and Safety:

Potassium Manganate : Harmful by ingestion

Danger to the environment

Sulphuric Acid: Corrosive

Ingestion will be fatal

Potassium Thiocyanate: Harmful by ingestion

Skin contact may lead to ulceration

Zinc: Flammable

May act as an irritant

- * Safety glasses & lab coats must be worn.
- * Care must be taken when heating the solutions as chemicals may spit.
- * Tongs should be used at all times when handling the zinc to avoid skin contact.

- * If any chemical comes into contact with skin, wash effective area immediately with warm water, seek medical advice if necessary.
- * Care should be taken when moving around glass. Breakage should be reported immediately.
- * All practical work should be carried out in a well-ventilated area.