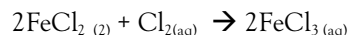
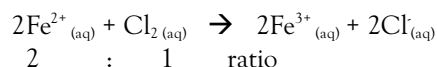


**Aims:** Deduce the concentration of a saturated solution of chlorine

In order to determine the concentration of aqueous chlorine one must firstly reduce the chlorine to its ions, using a reducing agent, in this case Fe(II). I will be using FeCl<sub>2</sub> powder for a source of Fe<sup>2+</sup>.

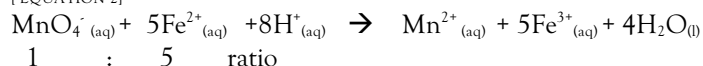


[ EQUATION 1 ]



Now if one can calculate the amount of reducing agent left over, it will be possible to calculate the initial Cl<sub>2</sub> solution concentration. It is possible to do this by titration of the Fe<sup>2+</sup> with Potassium Manganate.

[ EQUATION 2 ]



Acidic conditions are necessary, because in neutral or alkaline conditions Potassium Manganate is reduced no farther than Mn<sup>+4</sup> and a source of H<sup>+</sup> is also needed. Sulphuric acid is a good source of H<sup>+</sup>, and the SO<sub>4</sub><sup>-2</sup> ions are not reactive. Hydrochloric acid would react with the KMnO<sub>4</sub>, and chlorine gas would be evolved. Nitric acid is not suitable as it can become involved in redox processes where the NO<sub>3</sub><sup>-</sup> ion is reduced.

The end-point of the titration is detected by the first persisting pale pink colour change from purple. Even at the end-point the pink colour will fade after a short period as the MnO<sub>4</sub><sup>-</sup> ions react with Mn<sup>2+</sup> ions.

As the chlorine solution is of concentration approximately 7g dm<sup>-3</sup> it is possible to calculate quantities and concentration of other reactants so the experiment will give realistic outcomes. For instance if the concentration of MnO<sub>4</sub><sup>-</sup> is too low in the titration with the Fe<sup>2+</sup> then the volume used will be large, possibly requiring more than one burette volume. This introduces unnecessary inaccuracies, of having to repeat the set-up of the burette and also increases the time for the experiment. By working through an example calculation using a reasonable volume titre of approx 20.00 cm<sup>3</sup>, it is possible to determine the relevant quantities.

#### Reduction

- conc. chlorine solution = 7g dm<sup>-3</sup>
- 7g of chlorine in moles = No of grams/Molar Mass of Cl<sub>2</sub> = 7/70.9 = 0.0987 mol
- conc. chlorine solution = 0.0987 mol dm<sup>-3</sup>
- volume of Cl<sub>2</sub> solution is 50cm<sup>3</sup> (50cm<sup>3</sup> pipette)
- no moles Cl<sub>2</sub> = conc. x volume(dm<sup>3</sup>) = 0.0987 mol dm<sup>-3</sup> x 0.05 dm<sup>3</sup> = 0.00494 mol
- no moles of Fe<sup>2+</sup> will be twice that of Cl<sub>2</sub> in accordance with equation 1
- no moles of Fe<sup>2+</sup> = 2 x 0.00494 = 0.00988 mol
- amount of FeCl<sub>2</sub> reacting = Molar Mass of FeCl<sub>2</sub> x No moles

- amount of  $\text{FeCl}_2$  reacting =  $126.75 \times 0.00988 = 1.25\text{g}$

### Titration

- We must add exactly 1.25g of  $\text{FeCl}_2$  in order to react all of the chlorine, but it is much safer to add excess, and then work out how much  $\text{Fe}^{2+}$  is unreacted by titration.
- As 1.25 g of  $\text{FeCl}_2$  is required a safe excess is to add 2.0 g of  $\text{FeCl}_2$ .
- $\text{FeCl}_2$  expected remaining =  $2.0 \text{ g} - 1.25\text{g} = 0.750\text{g}$
- 0.75g  $\text{FeCl}_2$  in moles = 0.00591 mol
- No moles of  $\text{MnO}_4^-$  =  $0.2 \times$  no moles of  $\text{Fe}^{2+}$  in accordance with equation 2
- No moles of  $\text{MnO}_4^-$  =  $0.2 \times 0.00591 \text{ mol} = 0.00118 \text{ mol}$
- Conc = No moles / volume ( $\text{dm}^3$ ) [burette reading]
- Conc of  $\text{MnO}_4^-$  =  $0.00118 \text{ mol} / 0.020\text{dm}^3 = 0.059 \text{ mol dm}^{-3}$

### Acid required

- Using equation 2 we can see that the reaction requires 8x the number of moles of  $\text{MnO}_4^-$
- Moles of  $\text{MnO}_4^-$  = 0.00118 mol
- Moles of Acid required (at least) = 0.00944 mol
- $0.025 \text{ dm}^3$  of  $0.5 \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$  = 0.0125 mol

This gives a safe excess of acid to use in the titration reaction

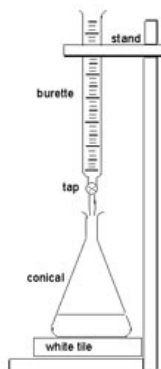
From these calculations you can see that one should use at least 0.125g of  $\text{FeCl}_2$  reducing agent and  $\text{MnO}_4^-$  of concentration approximately  $0.06 \text{ mol dm}^{-3}$  for the titration.

### Method

#### Equipment:

50  $\text{cm}^3$  Burette  
 50  $\text{cm}^3$  Volumetric Pipette  
 Pipette Filler  
 250  $\text{cm}^3$  Conical Flask  
 Clamp Stand  
 White filter paper  
 Glass rod  
 Balancing scales  
 Weighing boat

Figure 1



1. Use a volumetric pipette and pipette filler to deliver  $50.0 \text{ cm}^3$  of the chlorine solution into a  $250 \text{ cm}^3$  conical flask. Using the balancing scales and a weighing boat measure out approximately 2.0 g of  $\text{FeCl}_2$  solid. Record the mass of the weighing boat then add  $\text{FeCl}_2$  until the mass reads equal to the initial weight of the weighing boat + 2.0 g. Record the weight of  $\text{FeCl}_2$  - total weight of boat and  $\text{FeCl}_2$  - initial boat weight. Add to the conical flask containing the  $\text{Cl}_2$  and mix gently using the glass rod.

2. Wash a burette with a little of the  $\text{KMnO}_4$  solution. Then fill the burette, allowing the solution to run into the tip of the burette, ensuring no air bubbles are present. Record the start reading on the burette, making sure you read from the actual level of the solution ignoring the raised meniscus.
3. Add using a volumetric pipette,  $25\text{cm}^3$  of  $0.5\text{ mol dm}^{-3}$   $\text{HCl}$  to the conical flask containing the  $\text{Cl}_2$  solution.
4. Arrange the apparatus as shown in fig.1. Run the  $\text{KMnO}_4$  drop wise out of the burette into the conical flask containing the chlorine solution. Use your left hand to open the tap and your right hand to swirl the flask. The first run should be used as a trial, to determine the approximate end point. This will allow you to anticipate the end point and determine a more accurate result.
5. The end point occurs when a persistent pink tint remains for at least 5 seconds. When this is noticed the end volume of the burette should be recorded. Place the white filter paper under the conical flask, to give a better background on which to spot the colour change.
6. The titre is the change in volume of solution in the burette from the start to the end and is the amount of  $\text{MnO}_4^-$  needed to react with the  $\text{Fe}^{2+}$ .
7. Repeat the procedure at least 3 times until you get 2 values within  $0.1\text{cm}^3$ . Obtain an average titre

Example Table of Results:

	Trial Titration	Titration 1	Titration 2	Titration 3
End vol.				
Start vol.				
End-Start vol				

## Example Calculation

- Final average titre found to be  $23.65\text{ cm}^3$
- $23.65\text{ cm}^3$  of  $0.05\text{ mol dm}^{-3}$   $\text{MnO}_4^- = 0.02365 \times 0.05 = 0.00118\text{ mol MnO}_4^-$
- In accordance with equation 2  $\text{no. moles Fe}^{2+} = 5 \times \text{no. moles MnO}_4^-$
- $\text{no. moles Fe}^{2+} = 0.00118 \times 5 = 0.00591\text{ mol}$
- $\text{no. moles FeCl}_2 = \text{no. moles Fe}^{2+} = 0.00591\text{ mol}$
- $0.00591\text{ moles of FeCl}_2 \text{ in grams} = 0.00591 \times 126.75 = 0.749\text{ g Fe}^{2+}$
- If there is  $0.749\text{g FeCl}_2$  left over from the  $2\text{g}$ , then  $1.251\text{ g Fe}^{2+}$  must be reduced
- $\text{No Moles of Fe}^{2+} \text{ reduced} = \text{No Moles of FeCl}_2 \text{ reduced}$
- $\text{No Moles of Fe}^{2+} \text{ reduced} = 1.251/126.75 = 0.00988$
- $\text{No Moles of Cl}_2 = 0.5 \times \text{No Moles Fe}^{2+} \text{ in accordance with equation 1}$
- $\text{No Moles of Cl}_2 = 0.00494$
- $\text{Concentration of Cl}_2 \text{ solution} = \text{mol/volume(dm}^3) = 0.0988\text{ mol dm}^{-3}$
- $70.90 \times 0.0988 = 7.00\text{g}$
- $\text{Concentration of Cl}_2 \text{ solution} = 7.00\text{ g dm}^{-3}$

## Risk Assessment

When dealing with any chemicals a lab coat and goggles should be worn at all times.

Potassium Permanganate in its solid form is a very strong oxidizer and if it comes into contact with other material may cause a fire. However as we will only be using a dilute aqueous solution

this hazard will be minimal. Even in aqueous state it may cause severe digestive tract irritation or eye irritation which is why it is essential to wear goggles. Sulphuric acid is corrosive and an irritant, even though only weak concentrations are being used eye protection and lab coats should be used throughout.

When dealing with glassware extra precautions should be taken, do not use glassware above shoulder level; always fill the burette from below shoulder height.