To determine the concentration of Potassium Permanganate($Kmn0_4$) using Ammonium Iron Sulphate.

- Standard solution = ammonium iron(II) sulfate(stable and available in a highly pure form)
- Why is $KMn0_4$ not a good primary standard –not available in a highly pure state and decomposes in sunlight.
- $KMn0_4$ is an oxidising agent. In this experiment it gets reduced by gaining 5 electrons.
- Burette = $KMn0_4$
- Conical flask = ***ammonium iron(II) sulfate***+ Sulphuric acid
- Indicator = No indicator is needed

To determine the concentration of Potassium Permanganate($KMnO_4$) using Ammonium Iron Sulphate.

- **Colour change = manganate(VII) ions are decolourised in the reaction until the end-point, when a pale pink colour persists.(autocatalysis**
- Ratio of Mn^{2+}:Fe^{3+}=**1:5**
- ammonium iron(II) sulfate solution is made up in dilute acid-stop oxidation of iron
- Sulphuric acid added to conical flask to allow the full reduction of Mn^{7+} to Mn^{2+}.
- You cannot use HCl because it would be oxidised or HNO_3 as it gets reduced.
- Exception! We read the top of the meniscus **instead of the bottom as $KMnO_4$ has a very** strong purple colour.

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$$

- What is the oxidising agent?
- What is the reducing agent?
- The most important piece of information in this equation is that the ratio is

Ratio of $Mn^{2+}:Fe^{3+}$ = **1:5**

List of equipment

- Safety glasses
- Pipette (25 cm^3)
- Pipette filler
- Burette (50 cm^3)
- Filter funnel
- Graduated cylinder (100 cm^3)
- Conical flask (250 cm^3)
- White tile
- White card
- Retort stand
- Boss-head
- Clamp
- Beakers (250 cm^3)
- Wash bottle

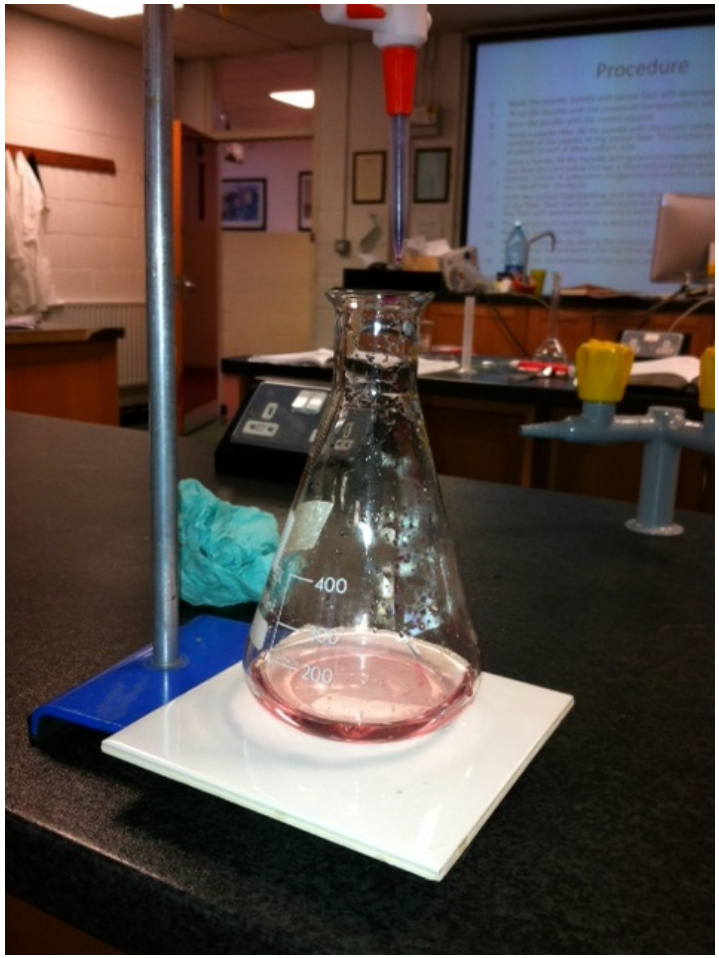
Procedure for making up the standard solution

1. Weigh out **9.8** g of **ammonium iron (II) sulphate crystals**
2. In a beaker add **20**ml of **sulphuric acid** to the 100ml of deionised water.
3. Transfer the crystals to the beaker and dissolve the crystals.
4. Add the solution to a 250ml volumetric flask.
5. Bring to the mark with deionised water and stopper and invert 20 times.

Procedure

6. Wash the pipette, burette and conical flask with deionised water
7. Rinse the burette with the potassium manganate(VII) solution
8. Rinse the pipette with the iron(II) solution.
9. Using a pipette filler, fill the pipette with the iron(II) solution and transfer the contents of the pipette to the conical flask. This solution is acidified by addition of about 10 cm^3 of dilute sulfuric acid.
10. Using a funnel, fill the burette with potassium manganate(VII) solution, making sure that the part below the tap is filled before adjusting to zero. Because of the intense colour of potassium manganate(VII) solutions, readings are taken from the top of the meniscus.
11. With the conical flask standing on a white tile, add the solution from the burette to the flask. Swirl the flask continuously and occasionally wash down the walls of the flask with deionised water using a wash bottle.
12. The end-point of the titration is detected by the first persisting pale pink colour. Note the burette reading.
13. Repeat the procedure, adding the potassium manganate(VII) solution dropwise approaching the end-point until two titres agree to within 0.1 cm^3.
14. Calculate the concentration of the potassium manganate(VII) solution.

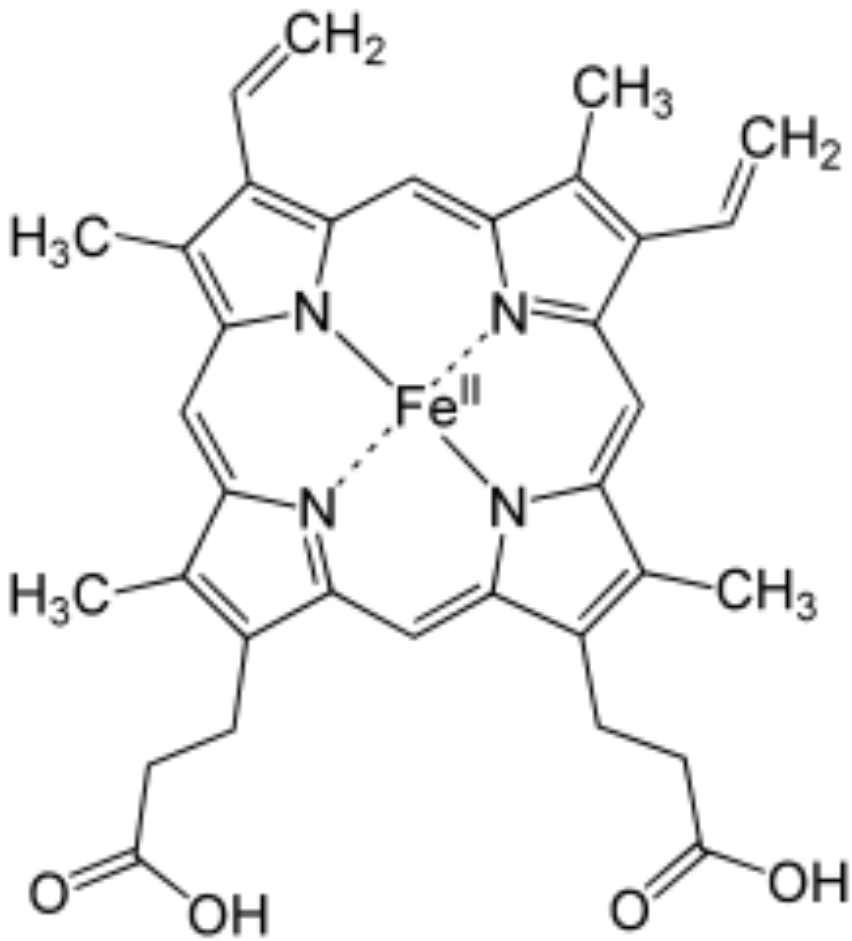
Diagram-note pale pink end point!



Calculation-using formula method at last!

- V_o=every group has different value
- M_o="x"
- n_o=1
- V_r=25
- M_r=
- n_r=5

Haemoglobin



Iron –essential mineral

- Haemoglobin is responsible for carrying oxygen around the body
- If a person has low iron they can be diagnosed with anaemia.
- Symptoms of anaemia are low energy, pale skin.
- More common in Women than men

To determine the amount of iron in an iron tablet

Making the iron tablet solution

- Weigh out 5 tablets and grind up using a pestle and mortar with ~5ml of H_2S0_4.
- Transfer the paste with ~100ml of H_2S0_4.
- Rinse all traces of iron from equipment into beaker.
- The outer coating of the tablets may not dissolve. This will not effect the experiment.
- Transfer to 250ml volumetric flask and bring to the mark with deionised water. Invert and stopper 20 times.

Titrate with an exact known conc. of potassium manganate(VII) solution

- Rinse pipette, burette and conical flask with deionised water.
- Wash out burette with $KMnO_4$ solution.
- Fill the burette to 0 with $KMnO_4$ and read the top of the meniscus.
- Pipette 25ml of iron tablet solution into conical flask.
- No indicator needed-why?
- Add 20ml of H_2SO_4 to the conical flask. Why?

Finding the concentration of Sodium thiosulfate solution using iodine

- It is important to use deionised water in this experiment as tap water contains small amounts of chlorine which may interfere with the result.
- Reaction in conical flask before titration begins is as follows:
- $2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + \mathbf{5I_2} + 8H_2O$
- Reaction in conical flask as titration proceeds is as follows:
- $\mathbf{2}S_2O_3^{2-} + I_2 \rightarrow 2I^- + S_4O\text{-}_6^{2-}$

Reagents

- **Starch solution:**
- Pour with stirring a paste containing 1 g starch and a little cold water into 100 cm^3 of boiling water. Boil for two minutes, and allow to cool. The solution should be stored in stoppered bottles.
- **0.5 M potassium iodide solution:** Dissolve 83 g potassium iodide in water and make up to a litre.
- **1 M sulfuric acid**:56 cm^3 of the concentrated acid is added slowly to about 700 cm^3 of deionised water. The mixture is stirred and made up to 1 litre in a volumetric flask with deionised water.

Procedure

- Steps 1 to 4 on page 202 already done as I have a solution of sodium thiosulphate made up.
- Rinse pipette, burette and conical flask with deionised water.
- Wash out burette with some sodium thiosulphate solution.
- Fill the burette to 0 with sodium thiosulphate and read the bottom of the meniscus.
- Pipette 25ml of $KMn0_4$solution into conical flask.
- Add 10ml of Potassium iodide solution to the conical flask.
- Add 20ml of H_2S0_4 to the conical flask. Why?
- No indicator needed until the conical flask contents turn pale yellow.
- Add few drops of **starch** indicator until the blue/black colour disappears to give a colourless solution. What will this tell us?

End-point pale yellow to blue black to…….

End-point-colourless(all iodine used up at this point)

Questions on titration

1. Why are iodine solutions made up using potassium iodide solution?

Iodine does not dissolve easily in water

2. Why does starch solution have to be freshly prepared?

It deteriorates quickly on standing.

3 Which of the three pieces of titration apparatus, the pipette, the burette or the conical flask, should *not* be rinsed with the solution it is to contain? Give a reason for your answer.

The conical flask should not be rinsed with the solution it is to contain. If it were washed out with the solution it was to contain, then traces of the solution would remain, and there would not be a precisely known amount of the solution in the flask.

4 Why is starch indicator added close to the end-point?

To give a sharp end point. What happens at the end-point?

The colour changes from blue-black to colourless.

Determination of the percentage of hypochlorite(Na0Cl) in bleach

- Bleach is a solution of**Na0Cl.**
- Hypochlorite ion reacts with excess iodide ion in the presence of acid to generate an iodine solution:
- $ClO^- + 2I^- + 2H^+ \rightarrow Cl^- + I_2 + H_2O$

- $I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$

Procedure

- The bleach solution must first be diluted to make a solution of suitable concentration for the titration.
- Using a pipette, add 25 cm^3 of bleach to a 250 cm^3 volumetric flask, and make the solution up to the mark with deionised water. The flask should be stoppered and inverted several times.
- Wash the pipette, burette and conical flask with deionised water. Rinse the pipette with the diluted bleach solution and the burette with the sodium thiosulfate solution.
- Using a pipette filler, fill the pipette with the diluted bleach solution and transfer the contents of the pipette to the conical flask.
- Add 10ml of potassium iodide and 20 cm^3 of dilute sulfuric acid to the conical flask – **this liberates iodine**.
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- Using a funnel, fill the burette with sodium thiosulfate solution, making sure that the part below the tap is filled before adjusting to zero.
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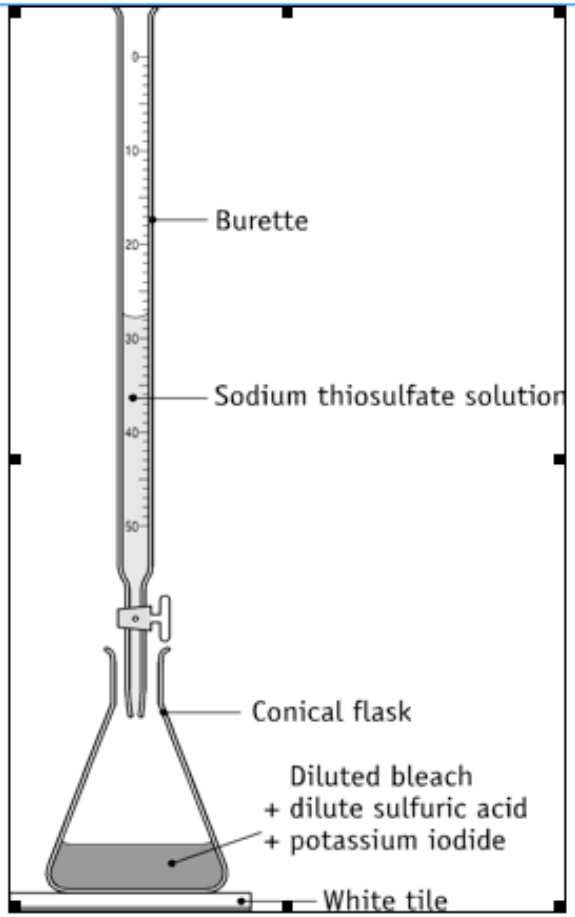
- With the conical flask standing on a white tile, add the solution from the burette to the flask. Swirl the flask continuously and occasionally wash down the walls of the flask with deionised water using a wash bottle.

Procedure

- Add a few drops of the starch indicator solution just prior to the end-point, when the colour of the solution fades to **pale** yellow. A blue-black colour appears. The thiosulfate solution should now be added drop wise, with thorough swirling.
- The end-point of the titration is detected when the blue-black colour changes to colourless. Note the burette reading.
- Repeat the procedure, adding the sodium thiosulfate solution drop wise approaching the end-point, until two titres agree to within 0.1 cm^3.
- Calculate the concentration of the iodine solution.
- Calculate the concentration of hypochlorite in the bleach solution.
- Calculate the percentage (w/v) of hypochlorite in the bleach solution.

Diagram


Burette
Sodium thiosulfate solution
Conical flask
Diluted bleach
+ dilute sulfuric acid
+ potassium iodide
White tile