

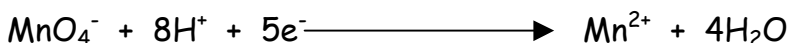
## ANALYTICAL CHEMISTRY

### 7.13.1 Volumetric Analysis

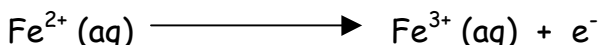
Volumetric analysis relies on methods involving accurate measurement of volumes of liquids (although one or more weighings may be necessary). Volumetric analysis can be carried out in a relatively short time and an experienced worker can be accurate to about 0.2 - 1%.

#### Titration of iron (II) with acidified manganate (VII)

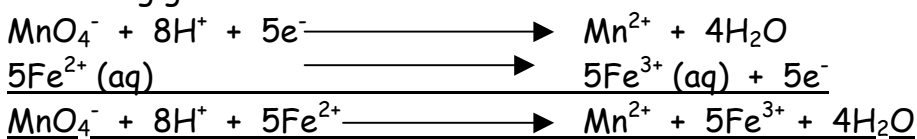
Potassium manganate (VII) (permanganate)  $\text{KMnO}_4$  is a powerful oxidising agent and can be used to estimate reducing agents such as iron (II) salts, ethanedioates (oxalates) and hydrogen peroxide solution in redox titrations. In acid conditions (produced by adding dilute sulphuric acid) potassium manganate (VII) is reduced.



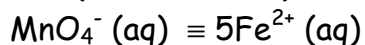
Iron (II) solutions can be oxidised



Combining gives



$\therefore$  1 mole of  $\text{MnO}_4^-$  ions (from  $\text{KMnO}_4$ ) will oxidise 5 moles  $\text{Fe}^{2+}$  ions



Generally about 0.02M  $\text{KMnO}_4$  is used, acidified with a few  $\text{cm}^3$  of 1M  $\text{H}_2\text{SO}_4$ .

Indicator and end-point

As the titration proceeds the purple colour of the permanganate is discharged giving a colourless solution. As soon as the permanganate is in excess the solution turns pink, so the potassium permanganate acts as its own indicator. The end point is the first permanent pink colouration.

### Estimation of iron in ammonium iron (II) sulphate $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$

- Weigh out accurately about 9.8 g of ammonium iron (II) sulphate crystals
- Dissolve in dilute sulphuric acid which has been boiled to remove air and then cooled (this prevents oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$ )
- Make up to  $250 \text{ cm}^3$  in a volumetric flask with the dilute sulphuric acid
- Transfer  $25 \text{ cm}^3$  of the iron (II) solution into a conical flask by pipette
- Add about  $15 \text{ cm}^3$  of  $1\text{M H}_2\text{SO}_4$
- Titrate with standard (0.02M) potassium permanganate solution from the burette until the first permanent pink colour is observed

9.85 g of ammonium iron (II) sulphate crystals were made up to  $250 \text{ cm}^3$  solution in cold, boiled-out, dilute sulphuric acid.  $25.0 \text{ cm}^3$  of the solution reacted completely with  $23.40 \text{ cm}^3$  of 0.02 potassium manganate (VII) solution.

Calculate the concentration of the alum solution and hence the percentage purity of the alum.

[Mr  $(\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) = 392$ ]