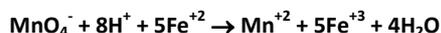


A potassium manganate(VII)/ammonium iron(II) sulfate titration

Theory

Potassium manganate(VII) (potassium permanganate, KMnO_4) solution can be standardised by titration against a standard solution of ammonium iron(II) sulfate solution. The iron(II) solution is measured by use of a pipette, whilst the potassium manganate(VII) solution is placed in a burette. The titration is carried out under acidic conditions, so the pipetted Fe^{2+} solution is acidified by addition of about 10 cm^3 of dilute sulfuric acid before commencement of the titration. The reaction is represented by the equation:



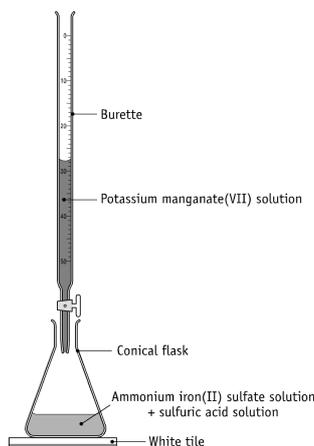
No indicator is needed, as the manganate(VII) ions are decolourised in the reaction until the end-point, when a pale pink colour persists.

Procedure

NB: Wear your safety glasses.

Wash the pipette, burette and conical flask with deionised water. Rinse the burette with the potassium manganate(VII) solution and the pipette with the iron(II) solution.

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.



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.

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.

The end-point of the titration is detected by the first persisting pale pink colour. Note the burette reading.

Repeat the procedure, adding the potassium manganate(VII) solution dropwise approaching the end-point until two titres agree to within 0.1 cm^3 .

Calculate the concentration of the potassium manganate(VII) solution.

Specimen Results

Rough titre	= 22.8 cm^3
Second titre	= 22.4 cm^3
Third titre	= 22.5 cm^3
Average of accurate titres	= 22.45 cm^3
Volume of iron(II) solution used in each titration	= 25.0 cm^3
Concentration of iron(II) solution	= 0.10 M

Specimen Calculations

$$V_A \times M_A \times n_B = V_B \times M_B \times n_A$$
$$22.45 \times M_A \times 5 = 25.0 \times 0.1 \times 1$$

$$M_A = 25.0 \times 0.1 \times 1 / (22.45 \times 5)$$
$$= 0.022 \text{ M}$$

$$\text{Concentration of potassium manganate(VII) solution} = 0.022 \text{ M.}$$

Solutions to student questions

Why is ammonium iron(II) sulfate suitable as a primary standard?

Because it is stable and available in a highly pure form.

Why is sulfuric acid added to the iron(II) solution prior to titration? Could hydrochloric acid or nitric acid be used instead of sulfuric acid? Explain.

Acidic conditions are necessary, because in neutral or alkaline conditions Mn^{7+} is reduced only as far as Mn^{4+} . Hydrochloric acid is not suitable as it would react with the KMnO_4 , and chlorine gas would be evolved. Nitric acid is not suitable as it is itself a very powerful oxidising agent - the NO_3^- ion is readily reduced.

In preparing for the titration, explain (a) why the pipette and burette were rinsed with deionised water followed by a little of the solutions they were to contain, (b) why the conical flask was rinsed with deionised water only.

(a) Deionised water washes out any residual solutions in the burette and pipette respectively. The second step was taken to remove any residual water, and so avoid dilution of the solutions when they are added to the burette and pipette respectively.

(b) Deionised water washes out any residual solution in the conical flask. If it were then washed out with the solution it was to contain, traces of it would remain, and there would not be a precisely known amount of the solution in the flask.

During the titration the sides of the conical flask were washed down with deionised water from a wash bottle. Explain why this procedure is necessary and why it can be carried out without affecting the result of the titration.

The washing process was carried out to ensure that all of the manganate(VII) solution added from the burette reacted with the iron(II) solution. It did not affect the result of the titration because only deionised water was added – no extra reactants were introduced into the flask.

One of the products of this reaction acts as a catalyst for the reaction. Which product is this? How could you demonstrate what substance is acting as the catalyst?

The reaction is catalysed by Mn^{2+} ions. This can be shown by taking a clean conical flask, pipetting the Fe^{2+} solution into it, acidifying it and then before starting to titrate adding some MnSO_4 solution (a convenient source of Mn^{2+}). Now the first droplet of MnO_4^- added decolourises immediately as there is Mn^{2+} in place to act as catalyst.

Why was sulfuric acid added in making up the ammonium iron(II) sulfate solution?

Iron(II) is very susceptible to air oxidation, forming iron(III), under neutral or alkaline conditions but this oxidation is inhibited in the presence of acids. The ammonium iron(II) sulfate solution is made up in dilute acid solution to make it stable towards air oxidation.