Teacher Resource Bank

GCE Chemistry
PSA10: A2 Inorganic Chemistry

• Carry out a redox titration
Technical Sheet

To analyse iron tablets by titration using potassium manganate(VII) in acidic solution.

Whenever possible, students should work individually.
If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

Requirements

- **Part 1**
  - weighing bottle or boat
  - five iron tablets
  - approximately 1 mol dm\(^{-3}\) sulfuric acid (50 cm\(^3\))
  - 100 cm\(^3\) conical flask with stopper

- **Part 2**
  - filter funnel and paper
  - deionised or distilled water in a wash bottle
  - 100 cm\(^3\) graduated (or volumetric) flask
  - burette
  - stand and clamp
  - 250 cm\(^3\) beaker
  - 25 cm\(^3\) pipette
  - pipette filler
  - 25 cm\(^3\) measuring cylinder
  - Two 250 cm\(^3\) conical flasks
  - 0.0200 mol dm\(^{-3}\) potassium manganate(VII) solution (150 cm\(^3\))
  - approximately 1 mol dm\(^{-3}\) sulfuric acid (100 cm\(^3\))

The iron tablets commercially available are usually labelled 200mg and contain 65mg of iron in the form of soluble Fe(II) ions. The quantities here are based on this assumption. Centres may choose to scale up the experiment to work with 250 cm\(^3\) graduated flasks.

The potassium manganate(VII) solution needs to be a standard solution and could be exactly 0.0200 mol dm\(^{-3}\)

**Centres are expected to carry out and be responsible for their own safety risk assessments.**
The analysis of iron tablets by titration using potassium manganate(VII) solution in acid.

Introduction
Iron tablets contain iron(II) sulfate which is a soluble inexpensive form of 'iron supplement'. The experiment is to determine the percentage by mass of iron(II) sulfate in each tablet.

Iron(II) ions can be oxidised to iron(III) ions by potassium manganate(VII) in acidic solution. In acidic conditions the deep purple solution of manganate(VII) ions is reduced to a very pale pink solution of manganese(II) ions. This solution is so pale as to appear colourless when dilute and, in practice, the marked difference in colour between these two oxidation states is useful as an end-point for this redox reaction.

The manganate(VII) ion accepts electrons and is reduced to colourless Mn$^{2+}$ ions according to the following half-equation.

\[
\text{MnO}_4^{-}(aq) + 8\text{H}^{+}(aq) + 5e^{-} \rightarrow \text{Mn}^{2+}(aq) + 4\text{H}_2\text{O}(l)
\]

The electrons are provided by the iron(II) ions which act as the reducing agent.

\[
\text{Fe}^{2+}(aq) \rightarrow \text{Fe}^{3+}(aq) + e^{-}
\]

The potassium manganate(VII) solution is added from the burette to the solution of the reducing agent and is immediately decolourised. As soon as the reducing agent is used up, the next drop of potassium manganate(VII) solution is not decolourised and colours the solution in the conical flask a pale purple colour (often described as a permanent pink colour). The end-point is the first appearance of this pale purple colour. Potassium manganate(VII) is therefore self-indicating and no other indicator is required. The acid used to provide H$^+(aq)$ is dilute sulfuric acid, which should always be in excess otherwise insoluble brown manganese(IV) oxide (MnO$_2$) will form.

The method described in this experiment ensures that there is a large excess of sulfuric acid in each titration.
It is the responsibility of the student to carry out and be responsible for their own safety risk assessment before carrying out this experiment. Wear safety glasses at all times. Assume that all of the reagents and liquids are toxic, corrosive and flammable.

Experiment

Part 1

a) Using a weighing bottle, weigh accurately five iron tablets.

b) Place the iron tablets into a 100 cm$^3$ conical flask and add approximately 50 cm$^3$ of the 1 mol dm$^{-3}$ sulfuric acid provided.

c) Stopper the conical flask, shake its contents well and then leave the tablets to dissolve. This is a slow process and should be carried out at least one day before the titration is to be attempted. The outer coating of each tablet is insoluble in water, but slowly breaks down in the acidic solution. The solution will need filtering before carrying out the titration.

Part 2

a) Without disturbing the residue, which will have settled to the bottom of the flask, carefully filter the solution directly into a 100 cm$^3$ graduated (volumetric) flask.

b) Rinse the residue in the filter paper into the graduated flask using a small volume of de-ionised or distilled water.

c) Add dilute sulfuric acid to make the solution in the graduated flask up to the mark.

d) Ensure that the contents of the graduated flask are fully mixed. You now have an acidified solution of iron(II) sulfate.

e) Fill a burette with the 0.0200 mol dm$^{-3}$ potassium manganate(VII) solution provided.

f) Pour some of the contents of the graduated flask into a clean 250 cm$^3$ beaker and, using a 25 cm$^3$ pipette and a pipette filler, measure out a 25.0 cm$^3$ sample of the iron(II) sulfate solution into a clean 250 cm$^3$ conical flask.

g) Using a 25 cm$^3$ measuring cylinder, measure out 25 cm$^3$ of the 1 mol dm$^{-3}$ sulfuric acid provided and add this to the contents of the conical flask.

h) Titrate this acidified sample of iron(II) sulfate solution by adding potassium manganate(VII) from the burette until the first permanent pink colour is seen.

i) You will only be able to carry out three titrations and if you are careful, you should be able to obtain at least two results that are concordant. Record the three results that you obtain.

j) Calculate and record the mean volume of potassium manganate(VII) solution used in the titration (the average titre). Show your working.
Analysing the data

The ability to calculate the percentage by mass of iron in the iron tablets is NOT part of the PSA but this is a useful task to complete. Your teacher can help you with this part of the work.

a) Combine the two half-equations (given in the introduction) to give the overall redox equation for the reaction that has taken place during the titration.
b) Use your overall equation to determine the ratio of moles of manganate(VII) ions that react with iron(II) ions.
c) Use the average titre to calculate the moles of manganate(VII) ions which have been used in the titration.
d) Calculate the amount, in moles, of iron(II) ions in the 25 cm$^3$ sample of iron(II) sulfate.
e) Calculate the amount, in moles, of iron(II) ions in the 100 cm$^3$ graduated flask at the start of the experiment.
f) Calculate the mass of Fe in the original five iron tablets and hence the mass of Fe in one iron tablet.
g) Compare your value for the mass of Fe with the information from the supplier about the composition of each iron tablet.
The specific marking guidance in the specification is as follows

2 marks: All areas of the task are carried out competently.
The burette is filled safely with the correct reagent (including below the tap)
The pipette and filler, burette and conical flask are all used correctly.
The titration results are concordant and the average titre is accurate.

1 mark: One of the areas of the task is performed poorly.
The burette is filled with the incorrect reagent or the funnel is left in or the burette is not filled below the tap OR
One of the pipette, pipette filler, burette or conical flask is used incorrectly OR
The titration results are not concordant or the average titre is inaccurate.

0 marks: At least two of the areas of the task are performed poorly.
The burette is filled with the incorrect reagent or the funnel is left in or the burette is not filled below the tap.
One of either the pipette, filler, burette or conical flask is used incorrectly.
The titration results are not concordant or the average titre is inaccurate.

Guidance for Teachers

Teachers are expected to exercise professional judgement in assessing the competence of their candidates in following the instructions.

Candidates should have been given guidance in the correct use of equipment and this guidance can continue during the practical session for which this PSA forms a part.

If, however, the guidance required is fundamental or frequent, then the student should not be awarded 2 marks.

Judgement of 2 marks, 1 mark or 0 marks will depend on whether the candidate can achieve concordance and whether the result is judged accurate.

Centres can judge accuracy either by comparing the student result with

- the known accurate value, assuming that this is certain.
- OR a teacher value for the titration taken using the same reagents and on the same day as the PSA.
- OR a class average which has excluded significant anomalies.

In each case the student value should be judged sufficiently accurate provided it is within 2% of the value chosen by the teacher, against which the class is being compared.
It is important to remember when marking these practical exercises that PSA is about student competence and that for a student to score full marks on this exercise perfection is neither expected nor required. In this exercise it would be harsh to withhold full marks if the student were to meet the specification criteria for 2 marks, but on one occasion were to leave the funnel in the top of the burette.