The estimation of iron(II) and iron(III) in a mixture containing both

Iron is a transition element. A transition element is an element that forms at least one ion with a partially file d orbital [1]. Transition metals have several different oxidation states, so can be oxidized and reduced.

Oxidizing the Fe^{2+} will make it Fe^{3+}, and it will lose an electron.

$$Fe^{3+} \rightarrow e^- + Fe^{3+}$$

Reducing Fe^{3+} will bring it back to Fe^{2+}, as it will gain an electron.

$$Fe^{3+} + e^- \rightarrow Fe^{2+}$$

I have been given a solution containin g between 1.1g and 1.3g of iron ions, a mixture of Fe^{2+} and Fe^{3+}. To work out how much Iron(II) and Iron(III) is in the mixture, I will start with a titration. I will determine the amount of Fe^{2+} in the mixture originally by titrating with potassium permanganate, and therefore oxidising the Fe^{2+} to Fe^{3+}. Potassium permanganate, KMnO4, is self indicating, so when the oxidation point is reached, it changes brown. Then I will reduce the Fe^{3+} back to Fe^{2+} with zinc powder and work out the amount of Fe^{2+} in the remaining mixture by titrating the new mixture against approximately 0.1 molar potassium permanganate.

Then Fe^{2+} mass in 2nd solution – Fe^{2+} mass in 1st solution = Mass of Fe^{2+} originally in the solution.

This experiment will only give me the Mass of Fe^{2+} in the solution, so the Fe^{3+} will need to be worked out by subtracting the Mass off the Fe^{2+} from the $200cm^3$ solution used at the beginning.

Manganate to iron is a 1:5 ratio. The concentration of manganate at any time is 5 x less than Fe^{2+}. [2]

The equation for this reaction is

$$MnO4^- + 5Fe^{2+} + 8H^+ + 5e^- \rightarrow Mn^{2+} + 5Fe^{3+} + 5e^- + 4H2O$$

<u>Equipment</u>

$200cm^3$ of Fe^{2+}/Fe^{3+} solution
Burette
Funnel
Clamp
Clamp stand
Conical flask
Measuring cylinder
$KMnO_4$
Beakers

Safety

Formula	Fe
Physical properties	Form: grey crystalline powder, grey rods, chips or lumps Stability: Stable, but rusts readily in moist air Melting point: 1535 C Water solubility: negligible Specific gravity: 7.86
Principal hazards	Contact of powder with the eyes can cause irritation Powdered iron is flammable.
Safe handling	Wear safety glasses when working with powdered iron.
Emergency	Eye contact: Flush the eye with plenty of water. If irritation persists, call for medical help. Skin contact: Wash off with water. If swallowed: Call for medical help if the amount swallowed is substantial.
Disposal	Small amounts of iron can be disposed of in normal laboratory waste, unless local rules prohibit this.
Protective equipment	Safety glasses if handling powdered iron.

Formula	Zn
Physical properties	Form: silvery-white to blueish solid Stability: Stable Melting point: 420 C Water solubility: nil Specific gravity: 7.14
Principal hazards	** Contact with the eyes can cause irritation ** Powdered zinc is highly flammable (note that the "highly flammable" designation applies to the powdered material only)
Safe handling	Wear safety glasses.
Emergency	Eye contact: Flush the eye with plenty of water. If irritation persists, call for medical help. Skin contact: Wash off powdered zinc wit h soap and water. If swallowed: Drink plenty of water and call for medical aid if the amount swallowed is substantial.
Disposal	Consult local rules. Note that powdered zinc may present a fire risk, so should not be disposed of in laboratory waste bins.
Protective equipment	Safety glasses may be suggested if you will be handling zinc powder.

Formula	$KMnO_4$
Physical properties	Form: dark red to purple crystalline powder Stability: Stable, but decomposes if heated above 150 C. Melting point: ca 150 C (decomposes) Water solubility: moderate, produces solutions which are intensely coloured, even when quite dilute Specific gravity: 2.70
Principal hazards	This material is harmful if swallowed or inhaled. It is also harmful if absorbed through the ski n. Potassium permanganate is a strong oxidizing agent and may react very exothermically with organic materials.
Safe handling	Wear safety glasses and keep the solid or solution from contact with the skin.. Take care not to allow the solid to come into co ntact with flammable materials.
Emergency	Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for medical help. Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin appears damaged, call for medical aid. If swallowed: Call for immediate medical help.
Disposal	Small amounts of very dilute potassium permanganate solution can be flushed down a sink with a large quantity of water, unless local rules prohibit this. More concentrated solutions and waste solid should be retained for disposal by those in charge of the laboratory.
Protective equipment	Safety glasses. Protective gloves should not normally be necessary. If they are to be used, nitrile will provide some protec tion, but may degrade upon contact with solid or solution, so should be checked regularly and replaced if damage is apparent.

Method

The 200cm^3 of $Fe^{2+}/^{3+}$ solution, I will titrate against potassium permanganate, until I get 3 concordant results.

As the solution is at least 30% of each substance by mass, I will assume 50% is Fe^{2+}, and 50% is Fe^{3+}. This gives me a rough estimate that there are :

25cm^3/55.84 = 0.45 moles of Fe^{2+} and Fe^{3+}

Because the ratio of potassium permanganate to iron is 1:5, the concentration of manganate must be 5 times less than the concentration of iron ions.

0.45/5 = 0.1 = approximate concentration of manganate needed to b **e** used

$$MnO4^- + 5Fe^{2+} + 8H^+ + 5e^- \quad = \quad Mn^{2+} + 5Fe^{3+} + 5e^- + 4H2O$$

This tells me how much of Fe^{2+} there will be, as the purple potassium permanganate will turn the solution colourless when it is all oxidised into Fe3+ .

After 3 concordant results of the Fe2 + being oxidised to Fe3+ with a certain amount of potassium permanganate, and the iron solution is now Fe^{3+}, adding zinc powder will reduce it all back to Fe^{2+}. A little bit is added sparingly until a colour change appears to brown.

$$Zn + 2Fe^{3+} \quad = \quad Zn^{2+} + 2Fe^{3+}$$

To find the total moles now, of Fe^{3+}, I will titrate 25cm^3 of the new solution of all Fe^{2+} with the approximate 0.1 molar potassium permanganate, the same I used in the first experiment.

I will need to use more $KMnO_4$ in the second titration as there is more Fe^{2+} to oxidise. All of the solution should turn colourless and be oxidised again to Fe^{3+}. The amount of potassium permanganate used will be in proportion to the amount of Fe^{3+} in the 200cm^3 solution.

I will then calculate
Moles of Fe^{2+} in second solution – Moles of Fe^{2+} in first solution = Moles of Fe^{2+} in complete solution.
And the Fe^{3+} is worked out according to the results of the Fe^{2+}

References

[1] = Chemistry 2 glossary page 165 OCR CAMBRIDGE UNIVERSITY PRESS

[2] = Advanced chemistry page chapter 13.4 page 218

[3] = http://cartwright.chem.ox.ac.uk/hsci/chemicals/iron.html

[4] = http://cartwright.chem.ox.ac.uk/hsci/chemicals/zinc.html

[5] = http://cartwright.chem.ox.ac.uk/hsci/chemicals/potassium_permanganate.html