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| Microscale Chemistry |
| The Chemistry of Iron compounds |

**A simple method for investigating the reactions of iron II and III compounds. Using a technique which can be applied to many other reactions.**

**CfE Level 3**

Through experimentation, I can identify indicators of chemical reactions having occurred. I can describe ways of controlling the rate of reactions and can relate my findings to the world around me.

SCN 3-19a

*(possible links elsewhere for some of the reactions)*

**CfE Advanced Higher** - Inorganic and Physical Chemistry

Transition metals

**Introduction**

This is a series of simple reactions that demonstrate many of the properties of iron compounds: oxidation, reduction complex formation etc.

Microscale chemistry is a familiar concept but the method employed here takes it a step further. The reactions are all carried out using drops of reagents on a laminated sheet of paper (or on paper inside a plastic pocket). This has a few advantages:

* The instructions are built into the workspace (the sheet) so are more easily followed.
* At the end of the experiment, clearing up simply involves wiping the sheets with paper towel. No fiddly, small tubes or dimple trays to wash up.
* Because such small amounts of reagents are being used, a kit for the experiment will take up a very small amount of space.

If the explanations of the reactions are printed on the reverse of the sheet, pupils will not be able to check their answers until they have finished.

**You will need**

|  |  |
| --- | --- |
| Laminated sheets | Small pieces of magnesium ribbon and a magnet |
| Iron II sulphate solution (1.4g in 3cm3 of water) | 1M Hydrochloric acid |
| 20 vol Hydrogen peroxide [irritant] | 0.4M Sodium hydroxide solution [irritant] |
| 0.1M Potassium hexacyanoferrate II | 0.5M sodium thiosulphate |
| 0.1M ammonium (or potassium) thiocyanate | Droppers / Pasteur pipettes |

The reactions are carried out by placing drops of the solutions in the appropriate place on the sheet.

In an ideal world, a clean Pasteur pipette would be used for each solution. It is quite acceptable, however, to re-use the same dropper/pipette - as long as it is rinsed between solutions. (This is particularly important before using the thiocyanate).

The Chemistry of Iron Compounds

You are given a vial with 0.2g of iron (II) sulphate. Add 1.5cm3 of distilled water to the solid, stir/shake to dissolve and place it in the circle.

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| 2. Add 2 pieces of magnesium turnings.  Move a bar magnet slowly towards the circle | 2. |  |  |
| 3. Add 5 drops of 0.4M sodium hydroxide | 3. |  |  |
| 4. Add 1 drop of 1M hydrochloric acid and 5 drops of 20vol hydrogen peroxide | 4 | 5. Take 1 drop of the solution from **circle 4** and add 5 drops of 0.4M sodium hydroxide solution. | 5. |
| 6. Take 1 drop of the solution from **circle 4** and add 5 drops of 0.5M sodium thiosulphate solution. Stir the solution with your pipette | 6. | 7. Take 1 drop of the solution from **circle 6** and add 5 drops of 0.4M sodium hydroxide solution. | 7. |
| 8. Add 1 drop of 0.1M potassium hexacyanoferrate (II) | 8. | 9. Take 1 drop of the liquid from **circle 4** and add 1 drop of 0.1M potassium hexacyanoferrate (II) | 9. |
| 10. Add 1 drop of 0.1M ammonium (or potassium) thiocyanate | 10. | 11. Take 1 drop of the liquid from **circle 4** and add 1 drop of 0.1M ammonium (or potassium) thiocyanate | 11. |

**Place 2 drops of this solution in each of the circles in boxes 2, 3, 4, 8 & 10**

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| 1. |  | 2. | 3. | 4. | 5. |
| ***Solution A***  Take 0.4g of iron (II) sulphate . Add 3.0 cm3 of distilled water to the solid, stir/shake to dissolve | *Place* ***3*** *drops of* ***solution A*** *in circle* ***1****. Observe at intervals while carrying out the rest of this activity* | Put **2-3** magnesium pieces in circle **2**. Add **3 drops** of **solution A**. Slide a magnet *slowly* towards the mixture. | **1 drop** of **solution A**.  Add **5 drops** of sodium hydroxide . | **1 drop** of **solution A**.  Add **1 drop** of potassium hexacyanoferrate(II) . | **1 drop** of **solution A**.  Add **1 drop** of potassium thiocyanate.  **Now go to Step 6.** |
| 6. |  |  | 7. | 8. | 9. |
| **Solution B.**  **To 1 drop** of **solution** **A**.  Add **2 drops** of hydrochloric acid and **3 drops of** hydrogen peroxide. Mix. |  |  | **1 drop** of **solution B**.  Add **5 drops** of sodium hydroxide. | **1 drop** of **solution B**.  Add **1 drop** of potassium hexacyanoferrate(II). | **1 drop** of **solution B**.  Add **1 drop** of potassium thiocyanate.  **Now go to Step 10.** |
| **Note**: the reactions in Steps 10 and 11 are difficult, as they need speedy action!  The aim is to reduce iron(III) ions back to iron(II) ions with the thiosulfate ions.  If you’ve been successful – you should get a green precipitate in Step 11. | 10. | 11. | **Solutions used:**   * sodium hydroxide, NaOH(aq), 0.4 M * hydrochloric acid, HCl(aq), 1 M * hydrogen peroxide, H2O2(aq), 20 ‘vol’ * sodium thiosulfate, Na2S2O3(aq), 0.5 M * potassium hexacyanoferrate(II), K4Fe(CN)6(aq), 0.1 M * potassium thiocyanate, KSCN(aq), 0.1 M | | **Wear eye protection**  Use a wooden splint for stirring. After use, break off the end & place in the waste bin. |
| **1 drop** of **solution B**.  Observe carefully as you add **5 drops** of sodium thiosulfate solution. Mix\*. | Take **1 drop** from the mixture in **circle 10**.  Add **5 drops** of sodium hydroxide solution. |

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**The Chemistry of Iron Compounds**

1. A displacement reaction producing metallic iron, which is magnetic

Mg + FeSO4 = MgSO4 + Fe

1. A green precipitate of iron (II) hydroxide is formed

FeSO4 + 2NaOH = Na2SO4 + Fe(OH)2

1. The solution goes brown as green Iron II is oxidised to brown iron III and bubbles appear as the excess hydrogen peroxide is broken down.

2 Fe2+ + H2O2 + 2 H+ → 2 Fe3+ + 2 H2O

1. A brown precipitate of iron (III) hydroxide is formed

Fe2(SO4)3 + 6NaOH = 3Na2SO4 + 2Fe(OH)3

1. A dark purple colour appears and then fades.

The purple colour is a complex - Fe(S2O3)2

It then decomposes

Fe(S2O3)2 + Fe3+ → 2Fe2+ + S4O6 2–

The iron III has been reduced to iron II

1. A green precipitate of iron (II) hydroxide is formed

Fe2+ + 2NaOH = 2Na+  + Fe(OH)2

1. There should be a white precipitate but it is oxidised very rapidly to a blue, known as Turnbull’s Blue. This has been shown to be identical to Prussian Blue obtained with iron III but with different particle size (See 9 below)
2. A dark blue complex, Prussian Blue, is formed

K+ + Fe3+ + [FeII(CN)6]4- → KFeIII[FeII(CN)6]

With iron II, the same complex is formed as the iron II is oxidised to iron III first.

1. No reaction
2. A dark red complex, of an iron III thiocyanate ion, is formed

Fe3+ + CNS1- = 2Na+  + [Fe(CNS)]2+

This test is specific to iron III, but it is very sensitive so may give a red colour with iron II solutions due to contamination.