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A Level Chemistry B (Salters)

H433/03 Practical skills in chemistry

Sample Practical Insert

Date – Morning/Afternoon

Time allowed: 1 hour 30 minutes

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Iron and manganese in paper clips

A student describes below a project to find the amount of iron and manganese in some paper clips:

- To find the amount of iron in the paper clips, I decided to use a titration with potassium manganate(VII) solution.
- To find the amount of manganese in the paper clips, I found out that I could oxidise it in solution to potassium manganate(VII) and then use a colorimeter because the intensity of the purple colour I get depends on its concentration of MnO_4^- ions.

Part 1: Determination of iron in paper clips

I found the following method in an old book and decided to use it to find the amount of iron in the paper clips.

Determination of the percentage of iron in iron wire

Weigh out accurately about 1.4 g of iron wire and transfer it to a conical flask containing 25 cm^3 of dilute sulfuric acid and a few cm^3 of concentrated sulfuric acid to accelerate the reaction. Fit the flask with a rubber bung containing a short length of capillary tubing.

Warm the flask carefully to maintain a steady reaction and, when all the iron has reacted leaving only particles of carbon, cool the flask. Transfer the solution quantitatively to a 250 cm^3 volumetric flask and make up to the mark with dilute sulfuric acid and water. Pipette 25 cm^3 of this solution into a conical flask, add about 25 cm^3 of dilute sulfuric acid and titrate with standard potassium manganate(VII) solution (about 0.02 mol dm^{-3}).

[Reference: STARK, J G (1971): *Titrimetric analysis for A & S levels SI Edition* London, John Murray, 27]

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The reaction of iron with sulfuric acid is:

$Fe + H_2SO_4 \rightarrow FeSO_4 + H_2$

The half-equations for the titration reaction are:

$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$
$Fe^{2+} \rightarrow Fe^{3+} + e^-$

I had to ask what a 'capillary' tube was and was told that it is a glass tube with a small internal diameter.

These are my results:

Mass of paper clips added to the conical flask = 1.28 g
Concentration of manganate(VII) solution = 0.0200 mol dm^{-3}

	Titration 1	**Titration 2**	**Titration 3**	**Titration 4**	**Titration 5**
Final burette reading / cm^3	22.90	45.40	22.55	43.05	24.00
Initial burette reading / cm^3	0.00	22.90	0.00	20.95	1.55

Part 2: Determination of manganese in paper clips

I found a website that said that steel normally contains between 0.1 and 0.4% manganese. I was given the following worksheet that I could follow to find the amount of manganese in the paper clips. It involves reacting pieces of paper clip with nitric acid to produce a solution containing Mn^{2+} ions. The Mn^{2+} are then oxidised with potassium iodate(VII) to MnO_4^- ions.

Method for the determination of manganese in paper clips:

1 Weigh accurately about 0.25 g of cut-up paper clip.
2 Put it into approximately 70 cm^3 of 2.0 mol dm^{-3} nitric(V) acid in a beaker.
3 In a fume cupboard, warm but do not boil the acid to help the paper clip to dissolve. The nitric(V) acid oxidises the manganese to $Mn^{2+}(aq)$ ions.
4 Add about 10 cm^3 of phosphoric(V) acid to the beaker, followed by about 10 cm^3 of potassium iodate(VII) solution. Boil the solution carefully for 10 minutes. Allow the mixture to cool. [The phosphoric(V) acid prevents the precipitation of insoluble iron(III) salts.]
5 When the solution is cool pour it into a 100 cm^3 volumetric flask using a small funnel. It is important not to lose any of the solution. Rinse the remaining solution from the beaker and funnel into the flask with distilled water and add further distilled water to bring the solution in the flask exactly up to the mark.
6 Stopper the flask and shake it to ensure that the solution is uniform. All the manganese that was in the 0.25 g of paper clip is now in the purple solution as the manganate(VII) ion, $MnO_4^-(aq)$.

(Reference: DENBY, Derek, OTTER, Chris, STEPHENSON, Kay (eds) (2009), *Salters Advanced Chemistry Support Pack*, Heinemann, 71)

I was given a solution of MnO_4^- ions, labelled '2% Mn', which had the same concentration as one that would be produced in my experiment from 0.25 g of steel containing 2% manganese by mass. The solution was actually made up by dissolving potassium manganate(VII) crystals in distilled water in a 1 dm^3 volumetric flask. Starting with this solution, I produced the calibration curve (**Fig. 1**) using a colorimeter. I would have preferred to start with a '0.5% Mn' solution, but my teacher said that it wouldn't be as accurate as the '2% Mn' solution.

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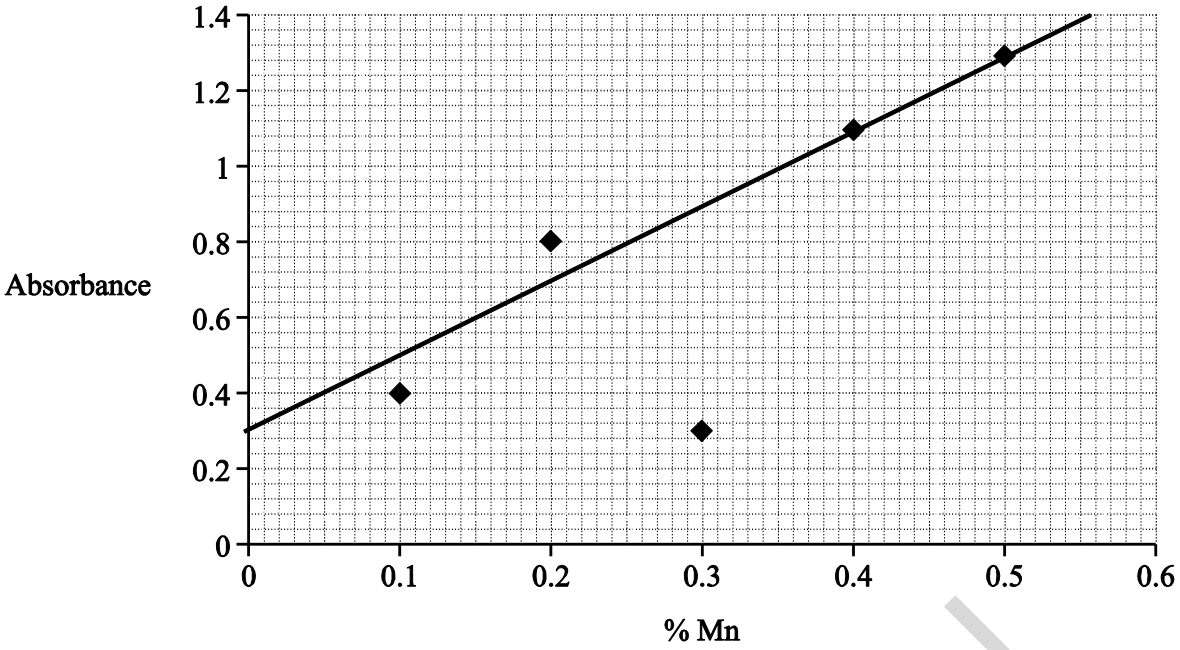

Fig. 1 % Mn against absorbance

The solution that I made from the paper clips gave an absorbance reading on the colorimeter of 0.64.

Comments on my experiments

I thought that my experiments went well. During the titration I had a bit of a problem with the pipette filler and I think I spilled some of the paper clip solution onto the bench as I was transferring it to the conical flask in one of my titrations.

One extra thing I could have done was to check the concentration of the MnO_4^- ions in the solution that I made from the paper clips by titrating it against a standard solution of Fe^{2+} ions.

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