
5.3 Candidate Individual Investigation Report 0407

Comparison of ways of finding the concentration of copper(ii) ions

INVESTIGATION INTO VARYING METHODS TO FIND THE CONCENTRATION OF Cu^{2+} IONS

I am investigating various methods for measuring the amount in moles of Cu^{2+} ions in a solution of CuSO_4 and evaluating which method gives the most accurate and reliable results. These methods are listed below:

1. A redox titration using iodide ions with sodium thiosulphate and starch indicator.
2. An acid base titration with a cation exchange resin in its hydrogen form and titrating with 0.1 mole sodium hydroxide solution.
3. Gravimetric or weighing method-using zinc powder to displace the copper ions. Any excess zinc powder could be removed with sulphuric acid.
4. Colourimetric to measure the deepness of the blue colour of the copper sulphate solution. This would work as the blue colour of the copper sulphate solution depends upon the concentration of Cu^{2+} ions in the solution
5. Compleximetric titration using a solution of EDTA and Fast sulphon black indicator.
6. Measuring electrode potential using electrochemical cells to find the concentration of Cu^{2+} ions possibly using the Nernst equation.

For all the above methods I will be using a solution of CuSO_4 with a concentration of 0.1 mol dm^{-3} and which therefore contain the same concentration of Cu^{2+} ions to see which is the most straight forward and gives the most accurate result

These methods are suitable for use as copper is a transition metal and exhibits various oxidation states and coloured compounds.

1. Redox Titration

The first method involves copper existing in different oxidation states. Copper has different oxidation states because it is a transition metal (d-block). All transition metals have multiple oxidation states due to the fact that their ionisation enthalpy increases at a slower rate as successive electrons are removed than is the case for the s-block metals.

	ΔH_1	ΔH_2	ΔH_3	ΔH_4
Ca [Ar]4s ²	+596	+1152	+4918	+6480
V [Ar]3d ³ 4s ²	+656	+1420	+2834	+4513

This means it is possible to carry out a redox reaction using iodine to reduce the Cu²⁺ ions to CuI and then titrating the iodine produced.

Equipment

- 10.0 cm³ pipette
- pipette filler
- 100cm³ conical flask
- burette
- ammonium ethanoate solid (10g)
- potassium iodide, solid (5g)
- sodium thiosulphate solution, 0.100mol dm⁻³ (50 cm³)

- freshly made starch solution (5cm^3)

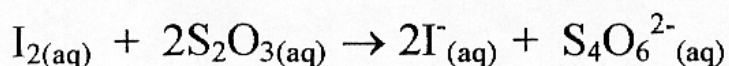
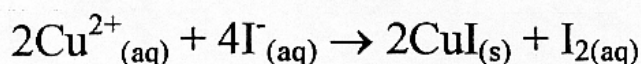
Method

1. Use a pipette and pipette filler to withdraw 10.0cm^3 of the copper sulphate solution and transfer it to a 100cm^3 conical flask.
2. Add a spatula measure of ammonium ethanoate to the conical flask to act as a buffer (to keep the pH of the solution constant). Using a glass rod test a drop of the mixture with Universal indicator paper: if acidic, add more ammonium ethanoate until the mixture is nearly neutral.
3. Add approximately 1g (a spatula measure) of potassium iodide to the conical flask. Swirl the contents. The solution will turn brown due to the formation of iodine and a beige coloured precipitate of copper (I) iodide will appear.
4. Fill a burette with 0.100mol dm^{-3} sodium thiosulphate solution. Make sure that the burette jet is also full of solution.
5. Record the volume reading in the burette before starting the titration. Add the sodium thiosulphate solution, in small volumes, to the contents of the conical flask until the iodine colour is nearly dispelled. Then add 1cm^3 of starch solution and continue the titration until the characteristic blue-black colour just goes. This is the end-point; the mixture will be cloudy light-beige colour due to the presence of precipitated copper (I) iodide.
6. Record the final burette reading and record the volume of sodium thiosulphate used. This is your rough titration.
7. Repeat the titration as often as necessary using the original solution of copper sulphate.

Results

	Start Volume cm ³	End Volume cm ³	Volume of thiosulphate used
Rough	3.00	9.60	6.60
1	9.60	19.80	10.20
2	19.80	28.60	9.00
3	0.00	9.90	9.90
4	10.10	25.30	15.20
5	0.00	10.30	10.30

$$\text{Average volume used} = \frac{9.90 + 10.20 + 10.30}{3} = 10.13$$



$$V = 0.01013$$

$$C = 0.1$$

$$0.01013 \times 0.1 = 0.001013 \text{ mols of sodium thiosulphate}$$

$$0.001013 \text{ mols of Cu}^{2+} \text{ in titration solution}$$

$$\text{Concentration of CuSO}_{4(\text{aq})} = \frac{0.001013}{0.010} = 0.1013 \text{ mols dm}^{-3}$$

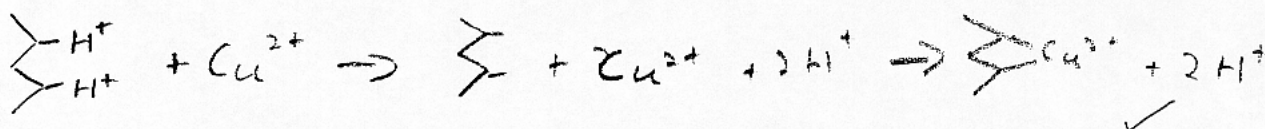
$$\therefore \text{Concentration of Cu}^{2+} \text{ ions} = 0.1013 \text{ mol dm}^{-3}$$

Evaluation

This experiment not too hard to actually do but it is hard to know when the titration has finished as it is hard to tell when the solution has stopped changing colour. Despite this the result was very accurate and any discrepancies can be put down to the errors in the measurements taken e.g. It would not be possible to get a solution of exactly 0.1 mol dm^{-3} .

2. Acid Base Titration

The next experiment is an acid-base titration. To do this experiment I had to use a cation exchange resin in its hydrogen form. When the CuSO_4 was filtered through the resin it displaced twice the amount of H^+ ions enabling me to do the titration.



Equipment

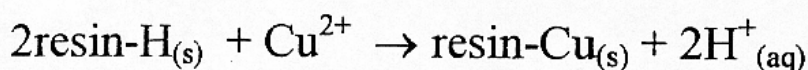
- Cation exchange resin
- Copper Sulphate
- 0.1 mol dm^{-3} Sodium hydroxide
- 2 × Burette
- Phenolphthalein
- Glass Wool (care use gloves)
- 250 cm^3 Conical flasks

Method

1. Put 10cm^3 of the resin into one of the burettes and pour some copper sulphate into a beaker. Make sure that the resin remains wet at all times.
2. Fill the other burette with the 0.1mol dm^3 sodium hydroxide
3. Place a conical flask underneath the burette with the resin then filter 10cm^3 copper sulphate through with 65cm^3 of distilled water until all the copper sulphate has been washed through.
4. Put a few drops of the phenolphthalein into the solution then titrate with the sodium hydroxide.
5. Repeat as necessary.

Results

	Start Volume	Finish volume	Total volume of sodium hydroxide used
Rough 25cm^3 (resin)	0	10.60	10.60
1) (10cm^3 resin)	0	19.90	19.90
2) (10cm^3 resin)	19.90	39.80	19.90



19.90 cm^3 of 0.1 sodium hydroxide used

$\therefore 10\text{cm}^3$ of CuSO_4 displaces 19.9 cm^3 H^+ ions

$$0.1 \times 0.0199 = 1.99 \times 10^{-3} \text{ mols of NaOH}$$

$$\text{mols of H}^+ \text{ displaced} = 1.99 \times 10^{-3}$$

$$1.99 \times 10^{-3} \div 2 = 9.95 \times 10^{-4} \text{ mols of Cu}^{+2} \text{ in } 10\text{cm}^3$$

$$\frac{9.95 \times 10^{-4}}{0.01} = 0.0995 \text{ mols dm}^{-3}$$

Evaluation

The final result is very accurate but there are some problems during the experiment. As can be seen from the table I was originally planning to filter the copper sulphate through 25cm^3 of resin, however the result was too low to be anywhere near correct. This was probably due to the fact that not all of the displaced H^+ ions were eluted so I reduced the amount of resin used.

3. Gravimetric

The next experiment is the gravimetric or weighing method. During this experiment a reaction occurs where the copper is displaced by zinc powder leaving me with Zinc Sulphate and copper dust. This reaction occurs because the zinc is more reactive than the copper. The reason that copper is more reactive than copper is that it is more electronegative and so will donate electrons to the copper atoms in the copper sulphate. This is also a redox reaction because the Copper changes its oxidation state from $2+$ to 0 and the zinc changes its oxidation state from 0 to $2+$.

Equipment

- Zinc dust
- Sulphuric acid
- Watch glass
- Access to balance
- Filter funnels

Method

1. Pour 100 cm^3 of copper sulphate into a beaker.
2. Add zinc dust to the solution of copper sulphate in small quantities with swirling until it is no longer blue and all the copper has been displaced from the solution
3. The solid obtained will contain unreacted zinc. Remove this by adding sulphuric acid (**CARE** corrosive), 1 cm^3 at a time with swirling until there is no further effervescence. (you may leave this overnight to ensure complete reaction)
4. Filter of the displaced copper using vacuum filtration
5. Wash the copper twice with 10 cm^3 portions of distilled water. Each time turn of the vacuum to give the liquid time to soak into the copper. Then spread the copper out on a weighed watch glass and allow the copper to dry.
6. Record the mass of the dry copper you have produced.

Results

Weight of watch glass and copper	= 16.3723g
Weight of watch glass	= 15.7164g
Weight of copper	= 0.6559g

$$\text{Number of mols of Copper} = \frac{\text{Weight of copper}}{\text{Mr}}$$

$$= \frac{0.6559}{63.55}$$

$$= 0.01032 \text{ mol in } 100\text{cm}^3 \text{ of } \text{CuSO}_4$$

$$\begin{aligned} \text{Number of mols in } 1\text{dm}^3 \text{ of } \text{CuSO}_4 &= \text{mol in } 100\text{cm}^3 \times 10 \\ &= 0.01032 \times 10 \\ &= 0.1032 \end{aligned}$$

\therefore There are 0.1032 mols of Cu^{2+} ions in the solution of CuSO_4

Evaluation

The result is very close to the actual concentration of the solution and is also close to the result of the first experiment. There were some problems with making sure that all the zinc was removed and I had to get rid of one lot of copper, as it had not fully dried out. Any errors can be put down to the accuracy of the equipment used in the experiment.

4. Colourimetric

This experiment is a colorimetric one. This experiment is possible because copper sulphate and indeed all transition metals have coloured salts and the deepness of the colour depends upon the concentration of the metal ions. This is because the electrons in the salts need a certain amount of energy to move up an energy level. This measure of energy corresponds to a certain wavelength of light. This means that the solution will absorb that wavelength leaving you with the opposite colour.

Equipment

- Colorimeter

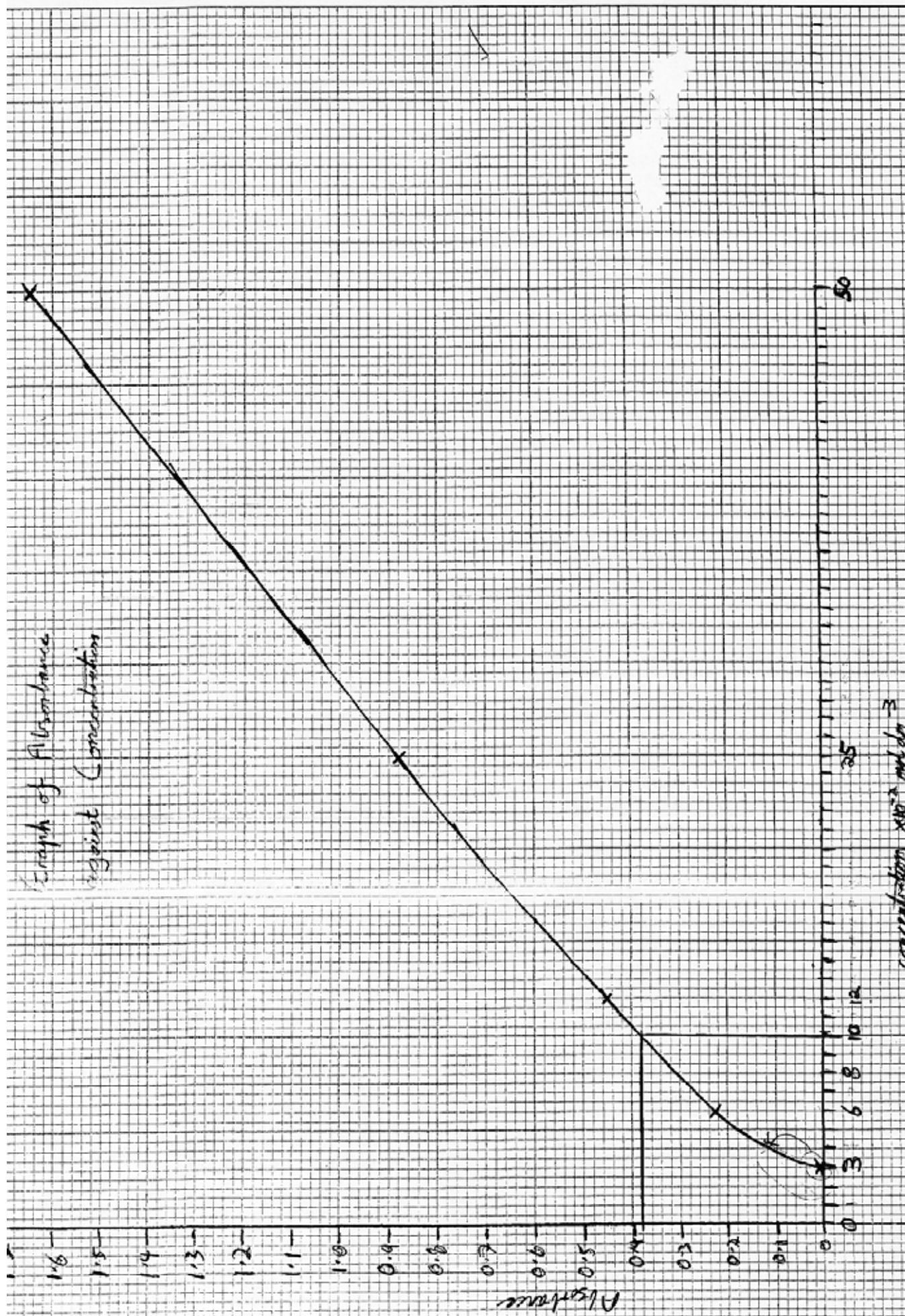
- Filters for the colorimeter
- Copper sulphate crystals
- $5 \times 200\text{cm}^3$ volumetric flask
- Cuvettes

Method

1. Weigh out 12.48g of copper sulphate crystals and then place them into one of the volumetric flasks. Add water up to the mark. Label this flask 0.5 mol dm^{-3} .
2. Take 100cm^3 of the 0.5 mol dm^{-3} and add it to another one of the flasks. Fill up to the mark with water. Label this flask 0.25 mol dm^{-3} .
3. Repeat the above process and label appropriately
4. Place filter number 680nm (this is because it corresponds to the complimentary colour to blue and will therefore be absorbed depending on the concentration of the solution) in the colorimeter and check the absorbency of each solution then find the absorbency of the test solution.

Results

Concentration mol dm^{-3}	Absorbency
0.5	1.65
0.25	0.88
0.125	0.45
0.0625	0.23
0.03125	0.11
Test	0.38



Evaluation

According to the calibration curve the concentration of the solution is 0.1 mol dm^{-3} . However the only way to be sure would be to increase the size of the scale but for the purpose of this experiment it is sufficient. If I were to do this experiment in industry I would gather far more data and use more accurate equipment and probably use a computer to check the results.

5. Complexiometric

The next experiment is a complexiometric one involving EDTA. A complex compound is one in which there is a central metal atom surrounded by one or more non-metal atoms called ligands or compounds depending on the number of bounds the metal atom and the non-metal atom can form

Equipment

- Burette
- 0.1 mol dm^{-3} EDTA
- Conical flasks
- Concentrated ammonia
- Fast Sulphon Black Indicator

Method

1. Fill the burette with the EDTA.

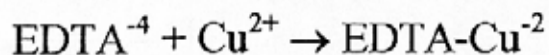
2. Put 20cm^3 of Copper sulphate solution into a conical flask then add 5cm^3 of concentrated ammonia (to act as a buffer) followed by a few drops of Fast Sulphon Black indicator.
3. Titrate the solution with the EDTA until the colour changes.
4. Repeat as necessary

Results

	Start Volume cm^3	Fin Volume cm^3	Amount of EDTA used cm^3
rough	0	19.90	19.90
1	19.90	40.40	20.50
2	0	20.10	20.10
3	20.10	40.10	20.00
4	0	20.05	20.05

$$\text{Average amount of EDTA used} = \frac{20.1 + 20 + 20.05}{3} = 20.05$$

$$0.1 \times 20.05 = 2.005 \text{ mol of EDTA}$$



$$\therefore 2.005 \text{ mol of Cu}^{2+} \text{ ions used}$$

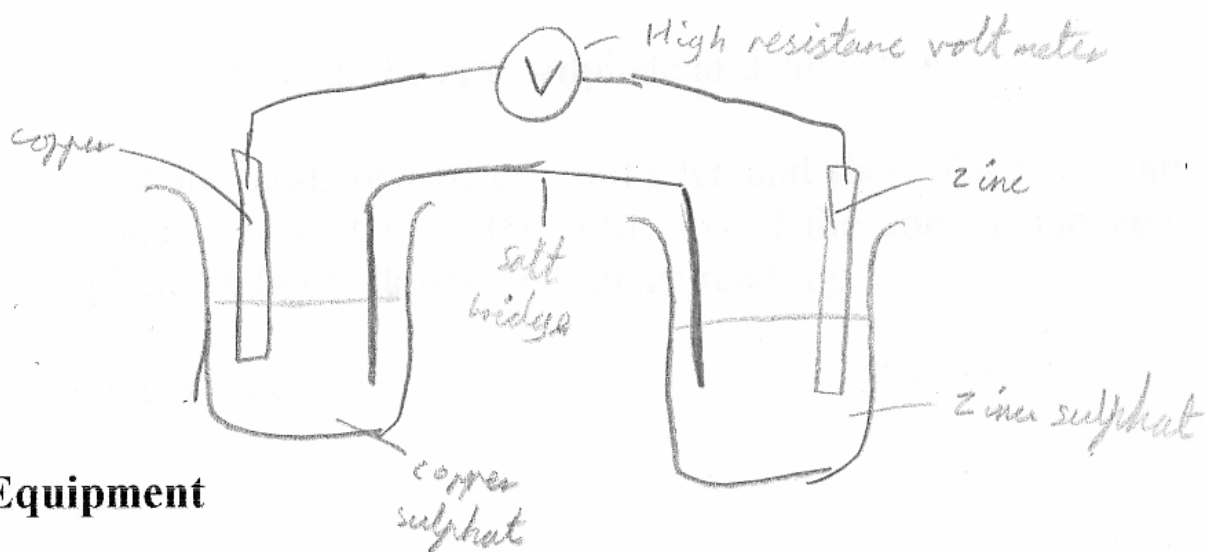
$$\therefore \frac{2.005}{20} = 0.10025 \text{ mol dm}^{-3} \text{ of Cu}^{2+} \text{ ions}$$

Evaluation

Again a very accurate result and close to the actual result. The differences can be put down to the accuracy of the instruments used and errors in the calculation.

6. Electrode Potential

This experiment involves the use of electrochemical cells to find the concentration to find the concentration of the Cu^{2+} ions. All half-cells are created by dipping a strip of metal in a solution of metal ions with a concentration of 1.00mol dm^{-3} . Joining two half-cells together makes an electrochemical cell. The first connection is made by joining the metal strips together with a wire but the solutions can not be joined in this way and nor can they be mixed. To get around this we soak a strip of filter paper in potassium nitrate (v) solution to create a salt bridge.



Equipment

- copper strips
- zinc strips
- high resistance digital voltmeter
- $5 \times 200\text{cm}^3$ volumetric flasks
- 100cm^3 of 1 mol Zinc Sulphate
- saturated potassium nitrate solution
- filter paper

Method

1. Create a set of 5 Different concentrated solutions as done in the colorimeter experiment.
2. Fill a beaker with saturated potassium nitrate solution and place some strips of filter paper into it to create the salt bridges.
3. Fill another beaker with 100cm^3 of 0.5mol dm^{-3} copper sulphate and place the copper strip in it.
4. Do the same with the zinc sulphate and zinc strips.
5. Attach the high resistance voltmeter and then place the salt bridges so that it is in the solutions of the copper and zinc sulphate and note down the highest reading.
6. Repeat as necessary.

Results

Concentration (mol dm^{-3})	Voltage (V)
0.5	1.072
0.25	1.050
0.125	1.036
0.0625	1.032
0.03125	1.023
Test	1.024

Evaluation

As can be seen from the above table there was very little point in doing a graph as the test voltage was too low to give the correct

concentration of the copper sulphate being tested. The reason for this may be because the voltmeter was broken or that there was a mistake made while setting up the experiment. Unfortunately I ran out of practical time before I could repeat the experiment.

Overall Evaluation

While the electrode potential equation did not work for reasons stated above it is still possible to choose which method for finding the concentration of Cu^{2+} ions is most effective and the most reliable. The most accurate experiment was the redox titration. But because it is difficult to know when the titration has finished it would not be my first choice.

While the accuracy of the colorimeter experiment depends upon the number of results taken and the size of the scale used it does have the potential to be more accurate than the redox reaction and it is easier to use and far more reliable. It would also be relatively simple to find the concentration of another solution of copper sulphate without having to redo the whole experiment as the results can be read from the graph. As such this is my first choice.

Risk Assessment

Sulphuric Acid - corrosive

zinc dust - flammable

copper sulphate - Harmful

Iodine - Harmful

Sodium Hydroxide - corrosive

Wear - goggles
- gloves

Bibliography

Chemical Ideas - general theory

Practical sheet M2:3 - method for redox titration
... " " gravimetric method

Chemistry review September 1999 pages 86-9 - experiment ideas

Understanding Chemistry p170

Chemistry in Context p 520

} Nernst equation and theory
behind electrode potential method

A Text Book of Quantitative

Inorganic Analysis Third Edition p441

- Method for complexometric
experiment

Synoptic Coverage

Concept applied in this investigation	Unit	Module	Year
Moles	EL	1	AS
Equations	EL, DF	1	AS
Concentration of Solutions	MI	2	
Electron Shells	EL	1	AS
Light + Electrons	EL, SS, CD	1, 3, 4	AS, A2
Colour	CD	4	A2
Chemistry of Colour	CD	4	A2
Acids and Bases	MI, O	1, 2	AS, A2
Buffers	O	4	A2
Oxidation + Reduction	MI, SS, AA	3, 2	AS, A2
Electrode Potentials	SS	3	AS, A2
Redox and Complexing	SS	3	A2
d block properties	SS	3	A2
d block complexes	SS	3	A2

5.4 Commentary on Individual Investigation Report 0407

Comparison of ways of finding the concentration of copper(ii) ions

Introduction

This investigation illustrates the type of project where a number of different analytical techniques are used to find out about the same or similar samples. They usually provide many opportunities for candidates to research relevant background theory and to plan experimental procedures. The difficulty in an investigation of this kind is the tendency of candidates to mix up and confuse drawing conclusions about the relative effectiveness of different procedures, which should be part of the analysing section, and identifying limitations within a particular procedure and assessing the effect of this on the accuracy and reliability of this procedure, which should be part of the evaluating section.

Planning

A clear aim at the beginning of this section provides a helpful framework for the rest of the report. Some attempt is made to link each of the experimental methods with relevant background theory but the coverage is brief and in outline only. Some expected equations are missing, others are incomplete and the effect of concentration on cell potential is not covered. This part of the plan does not quite meet the descriptor requirements at level P8b. The risk assessment is very basic and meets the descriptors at level P5b but not at level P8b. A reasonable number of references are listed but these lack detail and are not linked to specific parts of the text, this just meets the expectations at level P8b.

The experimental procedures are described for each of the methods used but expected detail such as the way in which the standard solution of copper sulphate was made up and the way the ion exchange resin procedure was modified is not included. The plan makes little attempt to justify or explain the choice of measurements that are made.

Overall, the plan does not quite meet requirements of the coursework descriptors at level P8 so a mark of 7 is appropriate for this section.

Implementing

Most data is recorded in an appropriate format and to an expected precision although some units are not included in some titration results tables.

The titres obtained from the redox titrations are in poor agreement and this should have prompted a repeat of the experiments involved. The determination of an additional gravimetric value would also have provided a more secure base from which to draw conclusions.

The recording aspect of implementing just meets the requirements of descriptors at level I8 but does not demonstrate additional achievement expected at level I11b. A mark of 8 is therefore appropriate.

Analysing

The candidate uses the raw data from a range of experiments to calculate a value for the concentration of the standard sample of copper sulphate. Some of these calculations are not clearly explained and contain errors and units are sometimes not included where expected. The colorimeter calibration curve is poorly presented. This aspect of this section just satisfies the descriptors at level A8a.

The conclusions drawn from the evidence are make no attempt to quantify the relative accuracy of different techniques. This aspect of this section just about meets the requirements of the descriptors at level A8b, but does not meet the descriptors at level A11b.

Overall, the analysis meets the coursework descriptors at level A8 but meets few of the additional requirements made by the descriptors at level A11. A mark of 8 is therefore appropriate.

Evaluating

Some of the main limitations of experimental procedures are identified, although the comments on the electrochemical cell experiments are very superficial. This aspect of this section meets the requirements of the descriptors at level E5a but do not fully meet the requirements of the descriptors at level E8a.

There are no comments on the precision of specific measurements. The descriptors at level E5b are therefore not met.

Overall, the evaluation does not fully meet the descriptors at level E5. A mark of 3 or 4 is appropriate, with a mark of 4 being suitable here.