

COPPER COIN ANALYSIS REFERENCE BOOKLET

SOURCES OF ERROR IN THE FORMATION OF THE COPPER(II) ANALYTE SOLUTION

1. The uncertainty in the volume readings when making up reagent solutions in volumetric flasks and measuring from pipettes and burettes. For darker solutions like the copper analyte solution, a bright LED torch was held stationary behind the volumetric scale when measuring quantities to improve accuracy as it resulted in easier judgement of a given volume. Burettes, pipettes and volumetric flasks were faced head on, level with the volumetric mark, when measuring out solutions and making them up to mark with distilled water. When making the copper analyte solution up to mark in a 500 cm³ volumetric flask, some extra ammonia solution was added to ensure that any precipitate that reformed on the addition of water was dissolved.
2. The loss of reagents during the transfer between apparatus. To minimise this loss, all apparatus and containers used to stir/transfer solutions were thoroughly washed into the final solution using distilled water. All washings were transferred when making a solution up to mark and care was taken to prevent the splashes of any fluid outside the confinement of containers. All magnetic stirrers and glass rods that were used to mix solutions were rinsed into the standard solution before it was made up to mark. These steps help minimise reagent loss.
3. Impurities in the reagents used may cause erroneous results and interfere in reactions. All chemicals used came from the supplied reagent containers and had minimal contact with apparatus. However, a sample of zinc sulfate powder appeared to be contaminated as it formed a green-coloured solution during the process of making a standard zinc sulfate solution. This had to be discarded and a new source of more pure zinc sulfate was utilised to make the standard 0.1M zinc sulfate solution. This does indicate however the uncertainty of 100% purity with school reagents. Achieving high purities is a very difficult and costly process in industry and so impurities are expected in a school laboratory.
4. Impurities present on the apparatus or in the laboratory. All solids were weighed out directly into clean glassware. All glassware and equipment were thoroughly washed using distilled water before use. Contact between the apparatus and reagents was kept to the minimal necessary amount. Separate dropper pipettes were used when required for different chemicals. All reagents were covered with cling film if they had to be left out (e.g. zinc sulfate solution during the dissolving process and the dissolving of the copper coin in nitric(V) acid). All solutions were kept in sealed containers and stored in a cool, dry place out of the sunlight when not in use.
5. Chemical compounds may settle out of their solution if they are allowed to rest for a long time between practicals. Before any solutions were transferred, reacted or measured out, they were inverted several times in their storage container to ensure they were homogeneous.

SOURCES OF ERROR SPECIFIC TO THE IODOMETRIC TITRATION

6. The iodometric titration was carried out in a mildly acidic solution. Acidic conditions result in oxidation of the excess iodide ions present if the titration was carried out slowly because the titration reactants are left exposed to atmospheric oxygen, decreasing the accuracy of the endpoint. The titration of the liberated iodine should have been conducted at a faster rate to prevent the undue exposure of iodide ions to oxidation.
7. Swirling of the conical flask containing the titration reaction mixture during the iodometric titration may cause loss of reactants. Swirling was done lightly and a clean dropper pipette of distilled water was used to ensure any solution that made its way up the sides of the flask was rinsed back down into the solution, and so not lost. The endpoint is based on the moles of iodine in the conical flask, and hence indirectly the moles of copper(II) present, which is unaffected by the addition of water.
8. The difficulty in judging the endpoint of the iodometric titration. The blue-black starch-iodine complex showed a slow transition through greyish-brown to white. It was difficult to determine the precise point when grey became white. Moreover, the iodometric titration can be subject to qualitative interpretation since humans perceive

colours and light differently. To avoid inaccuracy, I used titre 1 (the first accurate titre) as an endpoint reference for the sequential titrations as well as checking with colleagues' interpretations of my suggested endpoint colour.

9. The uncertainty in the measurements of the mass of the compounds used to make standard solutions such as the 0.1 M sodium thiosulfate solution. It was ensured that the top-pan balance was zeroed before use and that any reagents did not spill onto the top-pan, but rather into the contained beaker placed onto the balance. The final mass reading was recorded once the value had remained constant for 10 seconds. For a digital top-pan balance there is little that can be done to reduce error since its most significant uncertainty is due to the resolution.

SOURCES OF ERROR SPECIFIC TO THE ELECTROCHEMICAL ANALYSIS

10. The resistance of the voltmeter is not truly infinite and so a small current will flow through the voltmeter. This means that the electrochemical potential is not the true e.m.f. (the electrical potential energy transferred to other forms per coulomb of charge flowing through a source/cell). A high resistance voltmeter was used to try and ensure the electrochemical potential was accurate.
11. The crocodile clips/wires from the voltmeter may not have made complete electrical contact with the electrodes. The metal electrodes were thoroughly rubbed with emery cloth and rinsed in ethanol to remove any surface degradation and impurities. The crocodile clips on the leads of the voltmeter were securely attached to metal electrodes so they made a tight connection to reduce resistance between them.
12. Uncertainty in the concentrations of the standard copper(II) and zinc half-cells. This was mainly down to the resolution of the glassware used to dilute solutions and make up solutions to mark. All washings were transferred when making a solution up to mark and care was taken to prevent the splashes of any fluid outside the confinement of containers. All magnetic stirrers and glass rods that were used to mix solutions were rinsed into the standard solution before it was made up to mark. These steps help minimise reagent loss. Measuring apparatus was faced head and the readings recorded from the bottom of the fluid's meniscus to avoid parallax error.
13. The electrochemical cells were not measured under standard conditions of RTP. The true temperature of the laboratory was around 22°C and the pressure was unknown.
14. The copper(II) analyte solution was not a solution of copper(II) sulfate, like the standard concentrations of copper(II) half-cells. The analyte solution contained ammonia and trace amounts of ions which were not removed such as: Sn^{2+} , Zn^{2+} and NO_3^- . The presence of ions other than Cu^{2+} and SO_4^{2-} interfere with the electrochemical potential.
15. A lack of electrolytic substance in the salt bridge. The salt bridge should be abundant with positive and negative ions capable of carrying current between the two half-cells. The salt bridge was left to soak thoroughly for 10 minutes in a solution of concentrated potassium nitrate(V).
16. Impurities on the glassware and salt bridge. The glassware (100 cm³ beakers and glass rods) were washed thoroughly with distilled water and dried with paper towel before use as electrochemical cell containers. Clean filter paper strips were removed from a sealed packet and held using some clean tongs. At no point did the salt bridges come into contact with anything other than the tongs, concentrated potassium nitrate(V) and the glassware of the half-cells. This avoids the possibility of contamination of the salt bridge with foreign ions.
17. Insufficient contact between the electrodes and the solution. To avoid this, the electrodes were submerged as far as possible into the electrochemical cell solutions.
18. Varying internal resistance of the electrochemical cells. The distance between the electrodes and the salt bridge for a given half-cell were kept as constant as possible, placing them at the opposite sides of each beaker. The beaker size was kept the same and the volume of solution placed in each half-cell was maintained at 50 cm³ (measured using a clean burette rinsed with distilled water as well as the solution it held).
19. The salt bridge has a resistance and so results in a decrease in the overall electrochemical cell voltage. The salt bridge was always kept at the same length and was thoroughly soaked in potassium nitrate(V). the amount by which the salt bridge was submerged into each half-cell was kept roughly the same.

CALCULATIONS OF UNCERTAINTY FOR THE PREPARATION OF THE ANALYTE SOLUTION

Property	Calculation	Value	Calculation of percentage uncertainty	Percentage uncertainty	Reason
Mass of sodium thiosulfate dissolved to form 250cm ³ @ 0.1M	N/A	6.20g	$\frac{0.01}{6.20} * 100$	+/- 0.16% (2 s.f.)	The resolution of a digital top-pan balance is +/- 0.01g
Moles of sodium thiosulfate dissolved to form ~250cm ³ @ ~0.1M	$n = m / Mr$ = $\frac{6.20}{248.1841386}$	=0.024981... mol 0.0250 mol (3s.f.)	N/A	+/- 0.16% (2 s.f.)	% Uncertainty is the same as for the mass measured (6.20g)
Volume of standard 0.1M sodium thiosulfate solution	N/A	250cm ³	$\frac{0.2}{250} * 100$	+/- 0.08% (2 s.f.)	The absolute uncertainty of a class B volumetric flask is +/- 0.2cm ³
Experimentally determined concentration of sodium thiosulfate solution to form ~250cm ³ @ ~0.1M	$C=n/V$ = $\frac{0.024981 \dots}{0.250}$	0.099926... moldm ⁻³ = 0.100 moldm ⁻³ (3s.f.)	0.16...+0.08	+/- 0.24% (2 s.f.)	% uncertainty of moles adds with the % uncertainty of the volume
Original volume of copper(II) analyte (~500cm ³)	N/A	500cm ³	$\frac{0.2}{500} * 100$	+/- 0.04% (2 s.f.)	The absolute uncertainty of a class B volumetric flask is +/- 0.2cm ³
Theoretical mass of copper for the 2.49g 1982 coin	97% of the mass of coin = 0.97*3.49	3.3853g = 3.39g (3 s.f.)	$\frac{0.01}{3.49} * 100$	+/- 0.29%	The resolution of a digital top-pan balance is +/- 0.01g
Theoretically prediction of the concentration of the copper(II) analyte solution	$C=n/V$ $C = m/(Mr*V)$ $C = 3.3853/(63.546*0.500)$	0.106546...moldm ⁻³ = 0.106 moldm⁻³ (3s.f.)	0.29...+ 0.04	+/- 0.33%	% uncertainty in the mass of copper and % uncertainty in the volume of 500cm ³ of analyte solution add.

CALCULATIONS OF UNCERTAINTY FOR THE IODOMETRIC TITRATION

Property	Calculation	Value	Calculation of percentage uncertainty	Percentage uncertainty	Reason
Sodium thiosulfate titre volume	Mean of 26.45, 26.40, 26.45, 26.45	26.4375cm ³ = 26.44cm³ (2 d.p.)	$\frac{0.10/2}{26.4375} * 100$	+/- 0.19% (2 s.f.)	% uncertainty of a repeated measurement is equal to half the range over the mean.
Moles of sodium thiosulfate in mean titre	$n = C * V$ = 0.09992... * 26.4375	0.0026417... mol	0.189... + 0.241...	+/- 0.43% (2 s.f.)	% uncertainty of concentration adds with the % uncertainty of the titre volume
Pipette volume of 25cm ³ of copper(II) analyte solution	N/A	25cm ³	$\left(\frac{0.06}{25} * 100\right) + \frac{0.04}{0.04}$	+/- 0.28% (2 s.f.)	The absolute uncertainty of a class B pipette is +/- 0.06cm ³ . Moreover, the percentage uncertainty of the 500cm ³ analyte solution adds too.
Experimentally determined moles of copper (II) titrated	N/A	0.0026417... mol	N/A	+/- 0.43% (2 s.f.)	Equal to moles of thiosulfate in titre
Experimentally determined concentration of copper analyte solution based on the titration	$C = n/V$ = $\frac{0.002641 \dots}{0.025}$	0.10567...mol dm ⁻³ = 0.106 mol dm⁻³ (3 s.f.)	0.43... + 0.28...	+/- 0.71% (2 s.f.)	The % uncertainty of moles of copper titrated and the the % uncertainty volume of analyte solution must add
Experimentally determined moles of copper in the coin	$n = c * V$ = 0.10567... * 0.500	0.052835.... mol = 0.0528 mol (3 s.f.)	0.04... + 0.71...	+/- 0.75% (2 s.f.)	The percentage uncertainty of the standard 500cm ³ solution adds to the percentage uncertainty of concentration
Experimentally determined	$m = n * Mr$ = 0.052835.... * 63.546	3.35750...g = 3.36g (3 s.f.)	N/A	+/- 0.75% (2 s.f.)	Same as the % uncertainty in the number of moles

mass of copper in the coin					
Experimentally determined percentage of copper in the 2.49g 1982 coin	$\frac{3.35750}{(3.49 * 0.97)} * 100$	=99.17885635 99.2% (3 s.f.)	0.75... + 0.29...	+/- 1.04%	% uncertainty in the experimentally determined mass adds with the % uncertainty of the theoretical mass of copper in the coin.

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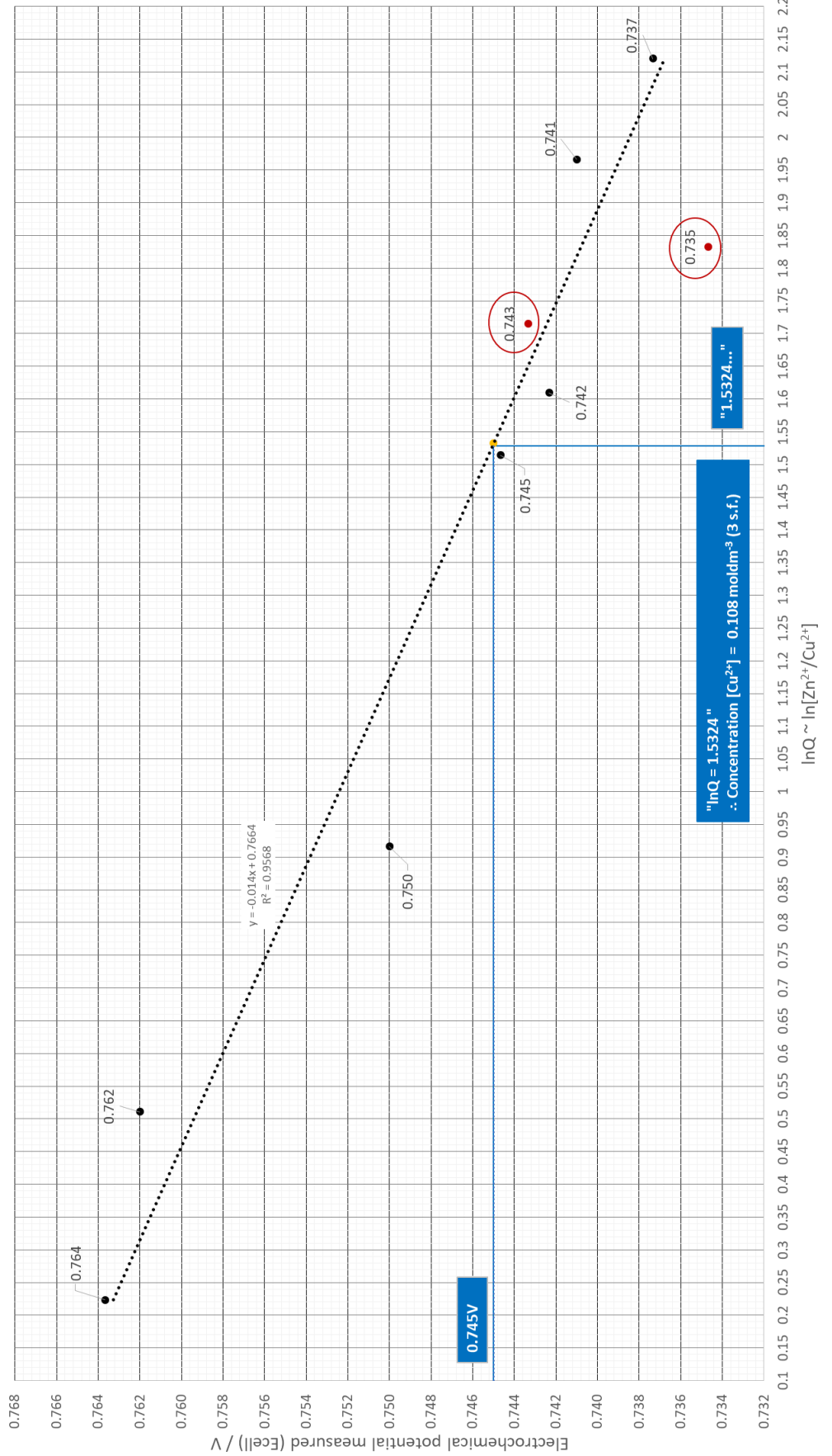
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CALCULATIONS FOR THE

ELECTROCHEMICAL ANALYSIS

Cell ID	Concentration of copper(II) / moldm ⁻³	Concentration of zinc(II) / moldm ⁻³	ln(Q) ~ ln([Zn ²⁺]/[Cu ²⁺])	Electrochemical potential, E _{cell} / V		
				Reading 1	Reading 2	Reading 3
1	0.4	0.5	0.223143551	0.762	0.763	0.766
2	0.3	0.5	0.510825624	0.763	0.759	0.764
3	0.2	0.5	0.916290732	0.748	0.750	0.752
4	0.11	0.5	1.514127733	0.741	0.746	0.747
5	0.1	0.5	1.609437912	0.740	0.742	0.745
6	0.09	0.5	1.714798428	0.740	0.741	0.749
7	0.08	0.5	1.832581464	0.738	0.731	0.735
8	0.07	0.5	1.966112856	0.741	0.738	0.744
9	0.06	0.5	2.120263536	0.735	0.739	0.738
A	Unknown	0.5 ln(0.5/[Cu ²⁺])		0.746	0.743	0.745

Graph to show the calibration between the electrochemical cell voltage and $\ln Q \sim \ln[Zn^{2+}/Cu^{2+}]$ for different concentrations of copper(II) half-cell and a constant 0.5M Zinc(II) negative half-cell in a 'Daniel cell' at ~RTP



Property	Calculation	Value	Calculation of percentage uncertainty	Percentage uncertainty	Reason
Mass of zinc sulfate dissolved to form 500cm ³ @ 0.5M	N/A	71.89 g	$\frac{0.01}{71.89} * 100$	+/- 0.014% (2 s.f.)	The resolution of a digital top-pan balance is +/- 0.01g
Experimentally determined moles of zinc sulfate dissolved to form 500cm ³ @ 0.5M	$n = m / M_r$ = $\frac{71.89}{287.541}$	=0.25001... mol 0.250 mol (3s.f.)	N/A	+/- 0.014% (2 s.f.)	% Uncertainty is the same as for the mass measured (71.89)
Volume of standard zinc sulfate solution	N/A	500cm ³	$\frac{0.2}{500} * 100$	+/- 0.04% (2 s.f.)	The absolute uncertainty of a class B volumetric flask is +/- 0.2cm ³
Experimentally determined concentration of zinc sulfate solution to form 500cm ³ @ 0.5M	$C=n/V$ = $\frac{0.25001 \dots}{0.500}$	0.50003... moldm ⁻³ = 0.500 moldm ⁻³ (3s.f.)	0.04+0.014...	+/- 0.0054% (2 s.f.)	% uncertainty of moles adds with the % uncertainty of the volume
Pipette volume of 50cm ³ of copper(II) analyte solution	N/A	50cm ³	$(\frac{0.06}{50} * 100) + 0.04$	+/- 0.16% (2 s.f.)	The absolute uncertainty of a class B pipette is +/- 0.06cm ³ . Moreover, the percentage uncertainty of the 500cm ³ analyte solution adds too.
Measured value of E _{cell} for the 50cm ³ copper(II) analyte solution	N/A	0.745V	$(\frac{0.002/2}{0.745} * 100)$	+/- 0.13% (2 s.f.)	% uncertainty of a repeated measurement is equal to half the range over the mean.
Corresponding value for lnQ	N/A	1.53247...	Uncertainty calculated by excel LINEST function: +/- 0.001327353 %unc = $(\frac{0.0013273\dots}{-0.013962\dots} * 100)$	+/- 9.5% (2 s.f.)	% uncertainty of the line of best fit is found by [slope uncertainty / slope gradient]*100

Experimentally determined concentration of copper(II) analyte solution	$\ln Q \sim \ln[\text{Zn}^{2+}]/[\text{Cu}^{2+}]$ so $[\text{Cu}^{2+}] = (e^{\ln(0.500)\dots - 1.532476})$	= 0.108 mol dm⁻³ (3s.f.)	0.0054+0.16+0.13+9.5	+/- 9.80% (2 s.f.)	% uncertainty of the concentration of zinc sulfate, the 50cm ³ volume of copper(II) analyte, measured value of E _{cell} , and the line of best fit all add up.
Experimentally determined moles of copper in the coin	$n = (c \cdot V)$ $= 0.1080000941 \cdot 0.500$	0.054000...mol = 0.0540 mol (3 s.f.)	N/A	+/- 9.80% (2 s.f.)	The % uncertainty is the same for the concentration of 500cm ³ analyte solution since the uncertainty in the 500cm ³ volume has already been included.
Experimentally determined mass of copper in the coin	$m = n \cdot M_r$ $= 0.054000 \dots \cdot 63.546$	3.43148...g = 3.43g (3 s.f.)	N/A	+/- 9.80% (2 s.f.)	Same as the % uncertainty in the number of moles
Theoretical mass of copper for the 2.49g 1982 coin	97% of the mass of coin $= 0.97 \cdot 2.49$	3.3853g = 3.39g (3 s.f.)	$\frac{0.01}{3.49} \cdot 100$	+/- 0.29%	The resolution of a digital top-pan balance is +/- 0.01g
Experimentally determined percentage of copper in the 2.49g 1982 coin	$\frac{3.43148 \dots}{(2.49 \cdot 0.97)} \cdot 100$	=101.36433...% 101% (3 s.f.)	0.29...+0.80	+/- 10% (2s.f.)	% uncertainty in the experimentally determined mass adds with the % uncertainty of the coin's mass measurement