

# Practical Booklet 9

Transition elements

## Cambridge International AS & A Level Chemistry 9701

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## Introduction

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Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

## Guidance for teachers

### Aim

To investigate some complexes of the copper(II) ion,  $\text{Cu}^{2+}$ , and to prepare a complex of copper containing a bidentate ligand.

### Outcomes

Syllabus section 12.2(a), (b), (c), 12.3(c), 12.4(a), 12.5 and 5.3(a) (& 20.3(a)) as well as experimental skills 2 and 3

Further work: syllabus section 12.1(e)

Syllabus sections 12.1 (h) and 12.2 (m)

### Skills included in the practical

A Level skills	How learners develop the skills
Planning	draw up a simple risk assessment for experiments that have been carried out
Analysis	recognise and understand the significance of observations made from test tube reactions make predictions based on data collected
Conclusions	draw conclusions from observations make scientific explanations of the observations and conclusions that they have described

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	collect an appropriate quantity of observations, including subtle differences in colour or solubility of materials
PDO recording	record observations precisely
ACE analysis	describe and summarise the key points of a set of observations

## Method

- **Learners must wear eye protection for these investigations.** Gloves may be provided.
- Copper forms a range of observed complex ions which are easy to produce by test-tube reactions. The complexes absorb visible light at the red end of the spectrum, so most of them are varying shades of blue in colour.
- In the experiments described in Method A, learners will carry out some ligand substitution reactions and will observe a number of complex ions of copper(II). They will work with monodentate and multidentate ligands.
- Learners will consider the equilibria involved in these reactions. They will use Le Chatelier's principle to explain some of their observations and gain an understanding of the significance of stability constants.
- In Method B, learners will prepare a copper(II) complex containing aminoethanoic acid as a bidentate ligand, and use the technique of reduced pressure filtration to separate it from the reaction mixture. It may be necessary for learners (or pairs of learners) to share use of Buchner filtration apparatus.
- Learners should become confident in using small amounts of reagents to obtain all relevant observations. Learners should become confident in handling hazardous chemicals.

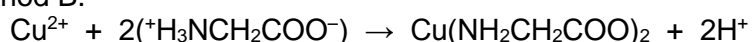
If time allows, **further work** may be carried out. Learners will carry out two redox reactions in which the copper(II) ion is reduced to copper(I). Reduction to copper(I) normally only occurs if the copper(I) compound to be produced is insoluble in water. Copper(I) iodide and copper(I) oxide (which is formed in both the Fehling's and Sandell's tests for aldehydes) are precipitated during the reactions described.

## Results

- Learners should record all their results using the correct terminology.

## Interpretation and evaluation

- The definition of ligand should be revised and the role of the lone pairs on nitrogen and oxygen atoms in the dative bonding of complex ions can be discussed.
- The stages in the formation of a new complex can be discussed ( $[\text{Cu}(\text{H}_2\text{O})_5\text{Cl}]^+$ , etc).
- Learners should be aware that there are many complex ions containing multidentate ligands. They should understand how the formulae of and charges on such complex ions can be worked out.
- Discussion can take place about the factors affecting colours of complexes (charge on central metal ion, ligand field strength, ligands approaching along (linear, square planar, octahedral) or between (tetrahedral) d-orbital axes).
- Learners should understand that, in the reaction of  $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$  with ammonia, the ammonia first acts as a base, precipitating insoluble copper(II) hydroxide. With excess ammonia, this is followed by a ligand substitution reaction involving ammonia molecules.
- The idea of zwitterion formation of amino acids can be discussed, after carrying out the preparation in Method B.



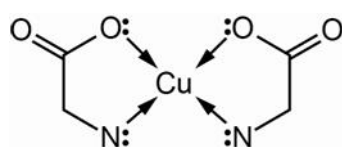
- The usefulness of increased speed of filtration under reduced pressure can be introduced.
- The effect of stability constants on ligand substitution reactions should be discussed.  
 $K_{\text{stab}}: [\text{Cu}(\text{EDTA})]^{2-} > [\text{Cu}(\text{NH}_3)_4]^{2+} > [\text{Cu}(\text{Cl})_4]^{2-}$

Reforming of the hexaaquo complex in step 2 of method A can be used to illustrate Le Chatelier's Principle.

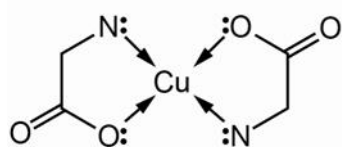
- The higher stability of the complexes involving chelating ligands can be discussed and linked to the increase in entropy (more particles on the right);  $\log K_{\text{stab}} [\text{Cu}(\text{en})_3]^{2+} \approx 18.1$   

$$[\text{Cu}(\text{H}_2\text{O})_6]^{2+} + \text{EDTA}^{4-} \rightleftharpoons [\text{Cu}(\text{EDTA})]^{2-} + 6\text{H}_2\text{O}$$

$$[\text{Cu}(\text{H}_2\text{O})_6]^{2+} + 3\text{en} \rightleftharpoons [\text{Cu}(\text{en})_3]^{2+} + 6\text{H}_2\text{O}$$

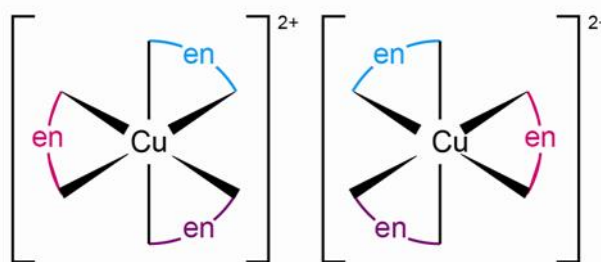


cis-



trans-

bis(glycinato)copper(II)



optical isomers: non-superimposable mirror image  
tris(1,2-diaminoethane)copper(II)

### Further work

- The effect of the  $d^{10}$  configuration on the colour of copper(I) iodide,  $\text{CuI}$ , can be discussed.
- If excess sodium thiosulfate is added, the  $\text{CuI}$  precipitate will dissolve.  
 (This may be compared to the 'fixing' stage of wet photography:  
 $\text{AgBr(s)} + 2\text{S}_2\text{O}_3^{2-}(\text{aq}) \rightarrow [\text{Ag}(\text{S}_2\text{O}_3)_2]^{3-}(\text{aq}) + \text{Br}^-(\text{aq})$ )
- The reduction of  $\text{Cu}^{2+}$  to  $\text{Cu}_2\text{O}$  by  $-\text{CHO}$  can be used to revise the reducing property of aldehydes.

## Information for technicians

Each learner will require:








- (a) Eye protection (gloves may be worn)
- (b) 8 x test-tube and 1 x boiling tube
- (c) 1 x test-tube rack
- (d) 4 x teat/dropping pipette
- (e) 1 x 50 cm<sup>3</sup> measuring cylinder
- (f) 1 x 250 cm<sup>3</sup> beaker
- (g) 2 x 100 cm<sup>3</sup> beaker
- (h) 1 x glass rod
- (i) 2 x spatula
- (j) 1 x Bunsen burner, heat proof mat, tripod and gauze
- (k) 1 x Buchner flask (side arm flask) \*
- (l) 1 x Buchner funnel and filter paper to fit the funnel \*
- (m) 1 x filter pump \*
- [N]** (n) 10 cm<sup>3</sup> 0.5 mol dm<sup>-3</sup> copper(II) sulfate
- [MH] [C]** (o) 5 cm<sup>3</sup> concentrated hydrochloric acid \*\*
- [MH] [C]** (p) 5 cm<sup>3</sup> '880' ammonia \*\*
- [N]** (q) 5 cm<sup>3</sup> 0.2 mol dm<sup>-3</sup> EDTA (disodium salt)
- [HH]** (r) 1 g aminoethanoic acid (glycine)
- [MH]**
- [MH] [N]** (s) 2 g copper(II) carbonate
- (t) 5 cm<sup>3</sup> 0.5 mol dm<sup>-3</sup> potassium iodide
- (u) 1 cm<sup>3</sup> starch indicator
- (v) 5 cm<sup>3</sup> 0.2 mol dm<sup>-3</sup> sodium thiosulfate
- [MH] [N]** (w) 0.5 g copper(II) sulfate pentahydrate
- (x) 1 g EDTA (disodium salt)
- [C]** (y) 5 cm<sup>3</sup> 2 mol dm<sup>-3</sup> sodium hydroxide
- (z) 0.5 g glucose
- (aa) 70 cm<sup>3</sup> distilled water
- (bb) paper towel
- (cc) pen or label for marking glassware

### Additional Instructions

\* If these items are not available then supply 1 x conical flask, 1 x filter funnel and filter paper.

\*\* These concentrated solutions should be situated in a fume cupboard if one is available.

## Hazard symbols

 <p>GHS02 (<i>flammable</i> <b>F</b>)</p>	 <p>GHS03 (<i>oxidising</i> <b>O</b>)</p>	 <p>GHS05 (<i>corrosive</i> <b>C</b>)</p>
 <p>GHS06 (<i>acutely toxic</i> <b>T</b>)</p>	 <p>GHS07 (<i>moderate hazard</i> <b>MH</b>)</p>	 <p>GHS08 (<i>health hazard</i> <b>HH</b>)</p>
	 <p>GHS09 (<i>hazardous to the aquatic environment</i> <b>N</b>)</p>	



# Worksheet

## Aim

To investigate some complexes of the copper(II) ion,  $\text{Cu}^{2+}$ , and to prepare a complex of copper containing a bidentate ligand.

## Method

### Safety:

- Wear eye protection. Gloves may be worn
- concentrated hydrochloric acid **[C]**
- '880' ammonia **[C] [N]** (concentrated aqueous ammonia)
- copper(II) carbonate **[H]**
- copper(II) sulfate pentahydrate **[H] [N]**
- $2 \text{ mol dm}^{-3}$  sodium hydroxide **[C]**

### Hazard symbols

**C** = corrosive substance

**N** = harmful to the environment

**H** = harmful or irritating substance

## Method A

1. To a 1 cm depth of  $0.5 \text{ mol dm}^{-3}$  copper(II) sulfate in a test-tube add concentrated hydrochloric acid **[C]** drop by drop with shaking until there is no further change. Keep this solution for tests 2 and 3.
2. To a 0.5 cm depth of solution from 1 in a test-tube add water in small portions until the test-tube is about half full. Shake the tube between additions of water.
3. To a 0.5 cm depth of solution from 1 in a test-tube add '880' ammonia **[C] [N]** drop by drop with shaking until there is no further change. Keep this solution for test 5.
4. To a 1 cm depth of  $0.5 \text{ mol dm}^{-3}$  copper(II) sulfate in a test-tube add  $0.2 \text{ mol dm}^{-3}$  EDTA until there is no further change.
5. Tip half the solution from test 3 into a test-tube and add  $0.2 \text{ mol dm}^{-3}$  EDTA until there is no further change.
6. Draw up a simple risk assessment for the experiments you have carried out

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## Worksheet, *continued*

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### Results

Record **all** your observations, including initial colours and states.

### Method B

1. Pour approximately 10 cm<sup>3</sup> of distilled water into a small beaker.
2. Weigh out approximately 1 g aminoethanoic acid (glycine) and dissolve it in the water.
3. Add copper(II) carbonate [**H**] to the solution with stirring until no more solid will dissolve.
4. Filter the mixture (using a Buchner flask and funnel and a filter pump if available). Transfer the filtrate (solution) into a boiling tube and leave it for crystals to form.

### Results

Record **all** your observations.

### Interpretation and evaluation (A and B)

In Method A you saw four different complexes of Cu<sup>2+</sup>.

These are [Cu(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup>, [Cu(Cl)<sub>4</sub>]<sup>2-</sup>, [Cu(NH<sub>3</sub>)<sub>4</sub>]<sup>2+</sup> and [Cu(EDTA)]<sup>2-</sup>. They are (distorted) octahedral, tetrahedral, square planar and octahedral complexes respectively.

1. Why are the four complexes different colours?
2. Why did the colour change gradually in step 1?
3. Explain your observations in step 3.
4. Which complex has the largest stability constant? Give a reason for your answer.
5. 
$$[\text{Cu}(\text{H}_2\text{O})_6]^{2+} + 4\text{Cl}^- \rightleftharpoons [\text{Cu}(\text{Cl})_4]^{2-} + 6\text{H}_2\text{O} \quad K_{\text{stab}} \approx 4 \times 10^5$$
Use this information to explain the change in colour in step 2.
6. What is the coordination number of copper in [Cu(EDTA)]<sup>2-</sup> and what is the charge on the EDTA ion?
7. The stability constant for EDTA displacing water from around the Cu<sup>2+</sup> ion is large (log *K* ≈ 18.8). There is little change in enthalpy for the reaction. Suggest a reason for this high stability.
8. In method A most of the ligands are monodentate but EDTA is hexadentate. Explain the meaning of the terms *monodentate* and *hexadentate*.

## Worksheet, *continued*

9. In method B the  $\text{Cu}^{2+}$  ion forms a complex with aminoethanoic acid, which is a bidentate ligand. Draw the structure of aminoethanoic acid. Explain how it can act as a bidentate ligand.

The complex formed has two aminoethanoate ions in a square planar arrangement around the  $\text{Cu}^{2+}$  ion. As the aminoethanoate ion is not symmetrical there can be two stereoisomers, cis- and trans-. The cis- form exists as pale blue needle-like crystals and the trans- form exists as blue-violet platelets. Which form of the complex did you make in method B?

Draw the two arrangements of  $[\text{Cu}(\text{NH}_2\text{CH}_2\text{COO})_2]$  to show the geometric isomers.

10. Copper(II) ions can also form a complex with another bidentate ion, 1,2-diaminoethane (often simplified in this context to 'en'). The formula of the complex ion is  $[\text{Cu}(\text{en})_3]^{2+}$  and illustrates a different form of stereoisomerism from the glycine complex.

Draw the two isomers of  $[\text{Cu}(\text{en})_3]^{2+}$  so that their 3-D structure is clear.

11. Would you expect the stability constant for  $[\text{Cu}(\text{en})_3]^{2+}$  to be closer to that of  $[\text{Cu}(\text{Cl})_4]^{2-}$  or  $[\text{Cu}(\text{EDTA})]^{2-}$ ? Give a reason for your answer.
12. What is the advantage of filtering using a Buchner flask, funnel and a filter pump, compared to normal filtration?

### Further work

1. To a 1 cm depth of  $0.5 \text{ mol dm}^{-3}$  copper(II) sulfate in a test-tube add about a 4 cm depth of aqueous potassium iodide. Divide the mixture into two portions in different test-tubes and carry out the following tests.
- Add a few drops of starch indicator.
  - Add aqueous sodium thiosulfate a few drops at a time with shaking until the thiosulfate is in large excess and no further change takes place.
2. Weigh out 0.40 g copper(II) sulfate pentahydrate **[H] [N]** in a small beaker. Weigh out 0.90 g EDTA (disodium salt) and add the solid to the beaker. Add  $40 \text{ cm}^3$  of distilled water and stir the contents of the beaker to dissolve the solids. Then add  $10 \text{ cm}^3$  of  $2 \text{ mol dm}^{-3}$  sodium hydroxide **[C]**. Label this mixture Sandell's solution **[H]**.

Half-fill a  $250 \text{ cm}^3$  beaker with tap water and heat it over a Bunsen burner. This will be your hot water bath.

Dissolve a small spatula measure of glucose in a 2 cm depth of distilled water in a test-tube. Add a 2 cm depth of Sandell's solution **[H]** then place the test-tube in the hot water bath and continue heating it until no further change takes place.

## Worksheet, *continued*

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### Results

Record **all** your observations.

### Interpretation and evaluation

1. Both experiments involve redox reactions.

Write an ionic equation for the reaction between  $\text{Cu}^{2+}(\text{aq})$  and  $\text{I}^{-}(\text{aq})$ .

Explain the colour of the precipitate formed when  $\text{Cu}^{2+}(\text{aq})$  reacted with  $\text{I}^{-}(\text{aq})$  in terms of the electron configuration of the metal ion.

2. In some text books glucose is referred to as an aldose (a reducing sugar).

What group present in glucose reacts with the  $\text{Cu}^{2+}$  complex in Sandell's solution?

Identify the copper compound formed in the reaction.

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