Preparation of Salts

Concepts being developed

Practical work in chemistry is often accused of being a collection of recipes. These need to be learned by rote, which prevents the idea of student investigation and enquiry. When ideas of "heurism" and "learning by discovery" were practised by the pioneers of chemistry education in the UK, H E Armstrong, C E Browne leading to Gordon Van Praagh and the Nuffield initiative in post-war Britain, they all conceded that it could not be carried out all the time, even with the small classes at Christ's Hospital School. It has taken over 200 years from the essays of Lavoisier and Davy to get where we are today, so how can 11-18 year-olds discover the same from scratch? These pioneers wanted to move away from didactic teaching and encourage an understating of chemistry, not to train students to be chemists working in a lab. Much of the distrust of science during the pandemic of 2020 is down to the general public not even comprehending the particulate nature of matter.

In a report by Wellcome[1], they stated that *"22% of young people saying that when doing practical work, they 'just followed the instructions without understanding the purpose of the work' a lot of the time. Nearly half of young people (46%) reported following the instructions without understanding the purpose of the work a lot of the time or sometimes."* In the Gatsby report[2], Sir John Holman reported that "*In Finland we were struck by the sparse nature of many practical worksheets: students were given skeleton instructions and expected to work out the detail for themselves, but they knew why they were doing it*."

In forensic and analytical chemistry, it is important that scientists follow detailed instructions (recipes), as their actions may come under detailed scrutiny in a court of law.

The preparation of copper (II) sulfate crystals is carried out by every student in the UK. It involves many practical skills as well as a use of stoichiometry. This makes large demands on working memory. It is often assumed that if first carried out at the age of 14. a student retains that knowledge until examined when aged 16. An alternative microscale procedure may be carried out as a revision exercise. It may use alternative methods of heating and filtration, but the principles are still the same. The traditional approach involving Bunsen burners, which has many shortcomings and often results in toxic fumes affecting students, is addressed in the CLEAPSS document PP027[3].

Microscale activity: Preparation of Copper (II) Sulfate Crystals

Ensure that full planning and risk assessment is carried out before attempting this activity.

Outline requirements

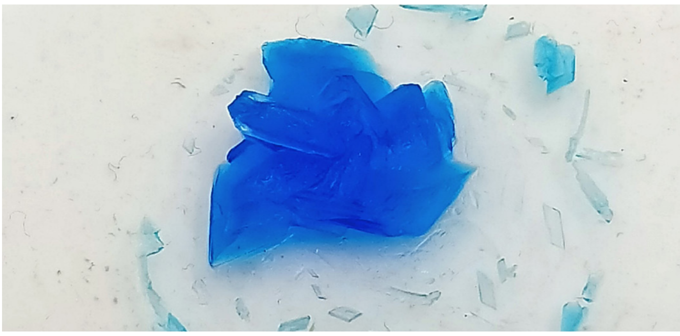
- 0.18–0.20 g of copper(II) oxide (WARNING: Harmful, environment). This is a slight excess of oxide to ensure that all the acid has reacted.
- 1.5 cm^3 of 1.4 M sulfuric acid (WARNING: Irritant). Concentrations at 1.5 M and above are deemed CORROSIVE and may require an increased level of PPE.
- eye protection
- Vial
- 2-cm^3 syringes
- Small watch glass
- Blunt end metal or plastic forceps
- Filtration equipment (see Technical Considerations)
- Heating equipment (see Technical Considerations)
- heatproof pad
- cotton wool

Outline method

1. Weigh 0.18-0.20 g of copper(II) oxide into a small glass vial.
2. Use a plastic dropping pipette to add 1.5 cm^3 of 1.4 M sulfuric(VI) acid to the vial.
3. Place cotton wool into the neck of the vial to prevent aerosol sprays.
4. Heat this mixture to almost boiling
5. Remove the vial (Care; it is hot) to a heatproof pad.
6. Filter the solution, collecting the filtrate in a small watch glass.
7. Let the solution evaporate naturally (for the best-shaped crystals) or heat until solid starts to appear around the edge of the 'puddle', then remove the watch glass, with forceps, onto the heatproof pad (good for lessons but difficult to judge).

Expected outcome

The reaction between copper(II) oxide and sulfuric acid can be achieved quickly at temperatures above 70°C and does not require the use of a (powerful) Bunsen burner. Beautiful blue crystals of copper sulfate-5-water form in the watch glass.



Teacher activities

Teachers can show that this is not a redox reaction but a neutralisation reaction with water being formed. This is different to metal/acid reactions discussed later, which are redox reactions.

$$CuO(s) + 2H^+(aq) + SO_4^{2-}(aq) \rightarrow H_2O(l) + Cu^{2+}(aq) + SO_4^{2-}(aq)$$

The aim is to produce pure hydrated copper sulfate crystals, but questions can be set. Examples are as follows:

a) Which of copper(II) oxide or sulfuric acid is the limiting reagent?

b) What should be the theoretical yield of hydrated copper sulfate crystals?

c) If you obtained a reduced mass of hydrated copper sulfate crystals, where has the solid been lost? What is the percentage yield?

d) Malachite is a natural form of basic copper carbonate ($CuCO_3.Cu(OH)_2$), found and mined all over the UK[4] as well as the world. Copper carbonate can be used in place of copper oxide to make copper sulfate and questions could be asked about environmental issues of using natural minerals in this way.

This method can also be applied to the use of copper carbonate ($CuCO_3.Cu(OH)_2$) (WARNING: Harmful, environment) in place of copper(II) oxide, but a new mass will need to be calculated. Now students can begin the process of discovery.

Teachers often wish they could find other acids to make other salts of copper. It can be disappointing, because the copper oxide/sulfuric acid reaction produces quick and beautiful results in a short space of time. Other acids are available such as ethanoic acid, but many have chemical issues, nitric acid acts as an oxidising agent, and some anions (e.g. chloride ions, nitrate ions) start acting as ligands.

Other metals can be considered, but the oxides producing other coloured salts are more toxic (as are the salts), leading to allergy issues in some people. Often the salts of other metals are colourless, even transparent. They lose or water of crystallization to the atmosphere and others gain water and dissolve. Some metal oxides (aluminium oxide) are really difficult to react with acids. Some metal salts (lead sulfate) are insoluble in water and form a coating on the metal oxide and so the reaction just stops. Practical chemistry can be extremely frustrating.

Microscale activity: Preparation of Zinc Sulfate Crystals

The reaction between zinc and sulfuric acid is quite slow, but the addition of copper(II) sulfate (which forms a coating of copper on the surface) speeds up the reaction, as well as warming the reaction.

Ensure that full planning and risk assessment is carried out before attempting this activity.

Outline requirements

- 1 zinc granule weighing more than 1.4 g
- 1.5 cm^3 of 1.4 M sulfuric acid (WARNING: Irritant). Concentrations at 1.5 M and above are deemed CORROSIVE and may require an increased level of PPE.
- eye protection
- Vial
- 2-cm^3 syringes
- Small watch glass
- Blunt end metal or plastic forceps
- Filtration equipment (see Technical Considerations)
- Heating equipment (see Technical Considerations)
- Heatproof pad
- Cotton wool

Outline method

1. Use a plastic dropping pipette to add 1.5 cm^3 of 1.4 M sulfuric(VI) acid to a vial.
2. Add 0.25 cm^3 of 1 M copper sulfate
3. Add 1 zinc granule to into a small glass vial.
4. Place cotton wool into the into the neck of the vial to prevent aerosol sprays.
5. Heat this mixture using hot water or a hot plate.
6. Remove the vial (WARNING: it will be hot) to a heatproof pad.
7. Filter the solution, collecting the filtrate in a small watch glass.
8. Let the solution evaporate naturally.

Expected outcome

A displacement reaction occurs forming copper on the zinc surface which acts as a catalyst (See Microscale activity 8.4 on page 80 of the book). At temperatures above 70°C the reaction subsides after about 10 minutes. There may still be some hydrogen produced, but a totally neutral solution will never form. The transparent crystals will form overnight naturally. Attempts to increase the rate of crystallization by forced evaporation, may only result in a white "mush".

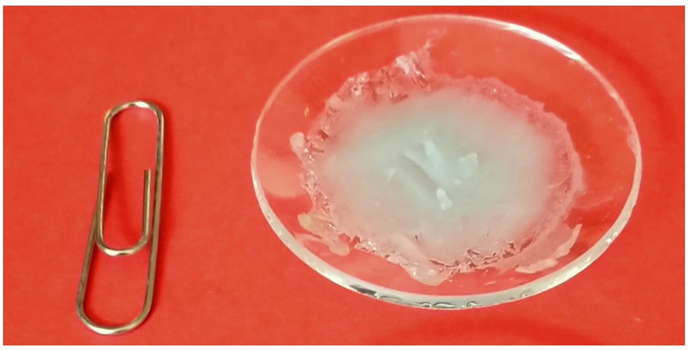
Teacher activities

This is a redox reaction with zinc being oxidised to zinc ions and the hydrogen ions reduced to hydrogen gas. The sulfate ions are spectator ions.

$$Zn(s) + 2H^{+}(aq) + SO_4^{2-}(aq) \rightarrow H_2(g) + Zn^{2+}(aq) + SO_4^{2-}(aq)$$

Other metals that can be tried are magnesium and iron. With iron, a green solid is formed. It is important not to make a neutral solution as this may result in a brown solid appearing.

For novelty value, pieces of metal paper clips can be cut up with wire snippers, rather than using the laboratory-supplied iron filings.



References

[1]Young people's views on science education Science Education Tracker Research Report February 2017 https://wellcome.org/reports/science-education-tracker-2016

[2]Good Practical Science http://www.gatsby.org.uk/uploads/education/reports/pdf/good-practical-science-report.pdf

[3]http://science.cleapss.org.uk/Resource-Info/PP027-Making-copper-sulfate-crystals.aspx [Membership required]. An open resource video of this method can be found on https://youtu.be/PTa8tkJ8rv0

[4]https://www.mindat.org/locentries.php?m=2550&p=14093

[5]http://science.cleapss.org.uk/Resource/GL254-Make-it-guide-microscale-distillation-apparatus.pdf [Membership required]. Details can also be found ere on https://www.scienceinschool.org/article/2018/perfumes-pop-aroma-chemistry-essential-oils/

