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Simple gravimetric chemical analysis – weighing molecules the microscale way

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Learn how to do quantitative chemistry using microscale techniques with bottle tops and inexpensive spirit burners that are relatively easy and quick to set up.

Quantitative chemistry using gravimetric analysis gives students the opportunity to experience chemical reactions, observe chemical changes, and use measurements of masses to determine the formula of a compound. This can be done using a combustion reaction, which results in a gain of mass (such as the reaction of magnesium with oxygen), or removing the water from a hydrated salt by heating, which results in a loss of mass[1]. These microscale practical activities are relatively simple and quick to do and can help students focus on the chemistry and reduce the load on working memory. Despite the small masses involved, the data generated from microscale experiments shows equivalent or better results than those obtained with traditional equipment, although a comparison of techniques is a useful exercise in error analysis. The advent of inexpensive, robust digital balances, measuring accurately to 0.01 g, has also allowed these methods to be more accessible and affordable than before.

Activity 1: Determining the formula of magnesium oxide

The determination of the formula of magnesium oxide by combustion of magnesium can yield variable results. Porcelain crucibles can be costly and can break during the experiment, and magnesium can escape when the lid is lifted. This product loss can reduce the accuracy of the result.

The microscale method uses an inexpensive alternative to expensive crucibles. The natural design of bottle tops allows a good flow of air with minimal loss of product.

This activity will take about 30 minutes and suitable for students aged 14–18.

Materials

- Magnesium ribbon, about 10–15 cm long (danger: flammable)
- Bunsen burner
- Two crown bottle tops (make sure the plastic coating has been removed from the bottle tops; this is easily done with a Bunsen burner and pair of tongs in a working fume cupboard)
- Mass balance
- Eye protection
- Nichrome wire, about 15 cm
- Small pipe-clay triangle
- Tripod

- Tongs
- Heating mat
- Activity 1 worksheet

Procedure

1. Find the total mass of two bottle tops and 15 cm of nichrome wire (M1) on a balance. Record the mass on the student worksheet.

Image courtesy of the author

2. Roll a 10–15 cm length of magnesium ribbon around a pencil and place the ribbon on one of the bottle tops.
3. Find the mass of the two bottle tops, nichrome wire, and magnesium ribbon (M2) and record on the worksheet.

Image courtesy of the author

4. Set up a Bunsen burner and tripod on a heatproof mat. On the tripod, place a pipe-clay triangle small enough to support the bottle top 'parcel'.
5. Sandwich the magnesium between the two bottle tops (serrated edges together). Wrap the wire round the bottle tops to keep them together.
6. Place the bottle tops securely on the pipe clay.

Image courtesy of the author

7. Heat the bottle tops with a strong blue flame for 10 minutes.

Image courtesy of the author

8. Switch off the Bunsen burner and allow the bottle tops to cool (for about 5 minutes).
9. Find the mass of the bottle tops plus nichrome wire and magnesium oxide. Record this mass as M3.
10. Use the masses of magnesium and magnesium oxide to calculate the number of moles of each substance. The molar ratio can be used to determine the formula of the compound.

Results and discussion

This activity can also be used with younger students who have not yet been taught mole calculations as way of introducing conservation of mass. Ask them to predict whether the mass of magnesium will get lighter, stay the same, or get heavier when heated, and test their prediction. Some will think the magnesium will get lighter, as they assume it will be 'burnt away' like carbon when it reacts with oxygen to form carbon dioxide. They are often surprised that oxygen atoms have mass, which can be measured on a balance after a combustion reaction with a metal.

A full explanation of the calculations can be found in the supporting material.

Sample result and calculation:

M1 = mass of bottle tops plus nichrome wire **= 3.87 g**

M2 = mass of magnesium plus nichrome wire and magnesium **= 4.11 g**

M3 = mass of the bottle top plus nichrome wire and magnesium oxide **= 4.26 g**

Mass of magnesium ribbon (M2 – M1 = 4.11 – 3.87) **= 0.24 g**

Moles of magnesium = mass of Mg/gram formula mass of Mg = 0.24/24.5 **= 0.0098**

Mass of oxygen used = M3 – M2 = 4.26 – 4.11 **= 0.15**

Moles of oxygen = mass of O/gram formula mass of O = 0.15/16 **= 0.0094**

Ratio of magnesium to oxygen = moles Mg/moles O = 0.0098/0.0094 **= 1.04**

The value should be close to one, giving a molar ratio of approximately one magnesium to one oxygen, which suggests the formula of magnesium oxide is indeed MgO.

Activity 2: Gravimetric determination of the formula of hydrated copper(II) sulfate

Gravimetric analysis to determine the moles of water present in a hydrated complex usually requires preweighing of a sample and heating to constant mass over a Bunsen burner using a crucible and a desiccator to prevent water from being reabsorbed from the air.

This method is quicker and uses a bottle top instead of a crucible, as described in Activity 1, along with spirit burners.

Spirit burners

Spirit burners burn cooler than Bunsen flames, which is advantageous for some experiments. A cheaper alternative to buying them from laboratory suppliers is to construct a homemade version made from small-scale jam jars.

A homemade spirit burner

Image courtesy of the author

A full guide on how to make a spirit burner is available in the supporting material. The assembly process can be observed in this video: https://www.youtube.com/watch?v=ndlycDnCM8c

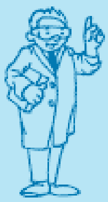
The spirit burners can be used for other microscale practical applications, such as flame tests and determining the melting points of covalent molecular and ionic substances,[2] as well as for the cracking of hydrocarbons.[3]

In this experiment, the use of a spirit burner limits the extent to which copper sulfate will decompose to release toxic sulfur dioxide:

$CuSO_4{\cdot}5H_2O(s)$ (pale-blue solid) $\rightleftharpoons$ $CuSO_4(s)$ (white solid) + $5H_2O(s)$

Copper(II) sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$) loses four of its water molecules at about 100 °C. The final water molecule is lost at 150 °C. With a Bunsen flame, a temperature of over 650 °C is reached, which causes the hydrated copper sulfate to decompose; the solid darkens and toxic sulfur dioxide and trioxide gases are released. As well as being hazardous, the decomposition affects the accuracy of the results. Using a cooler flame produced by a spirit burner prevents this decomposition.

This activity will take about 30 minutes and suitable for students aged 14–18.



Safety note:

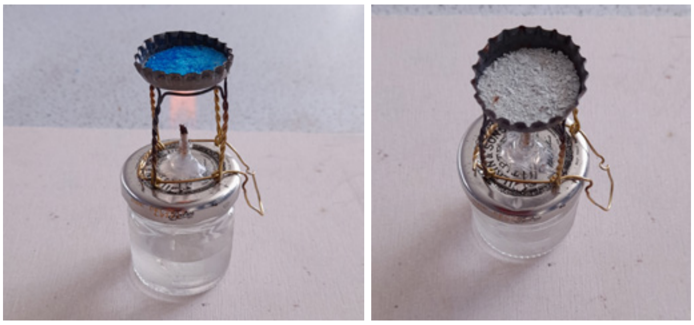
Wear eye protection.

Materials

- Bottle tops with plastic removed, as described in Activity 1
- Spirit burner (details of how to make one are given in the supporting material)
- Copper(II) sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$)
- Balance
- Spirit burner
- Spatula
- Tongs
- Optional: a muselet (this is a wire cage from a bottle of champagne or other sparkling wine) to act as a microscale tripod
- Activity 2 worksheet

Procedure

1. Put the bottle-top crucible on the balance and set it to 0.0 g using the tare button.
2. Add about 1.2 g of hydrated copper sulfate to the crucible. Record the mass on the student worksheet.
3. Optional: place the muselet on the spirit burner. This will act as a microscale tripod for the bottle top.
4. Place the bottle-top crucible on the muselet tripod and heat with an ignited spirit burner until the blue colour has been lost and white/colourless solid remains. Alternatively, hold the bottle top with tongs and heat as above.



Copper sulfate before (left) and after (right) heating

Images courtesy of the author

5. Remove the crucible from the spirit burner (and extinguish the flame).
6. Allow the crucible to cool.
7. Find the mass of the crucible plus anhydrous salt and record this.
8. Use the masses of hydrated and anhydrous copper sulfate to calculate the number of moles of copper sulfate and water present in the hydrated complex. The molar ratio can be used to determine the formula of hydrated copper(II) sulfate.

Results

A full explanation of the calculation can be found in the supporting material.

Sample result and calculation:

Mass of hydrated copper(II) sulfate used = **1.20 g**

Mass of anhydrous copper(II) sulfate after heating = **0.78 g**

Mass of water removed by heating = 1.20 − 0.78 = **0.42 g**

Number of moles of copper sulfate ($CuSO_4$) left after water was removed = 0.78 / 159.6 = **0.0049**

Number of moles of water removed by heating = 0.42 / 18 = **0.023**

Ratio of moles of water to copper sulfate = 0.023 / 0.0049 = **4.9**, which is 5 when rounded to nearest whole number.

The students can be shown the label of a bottle of hydrated copper sulfate and compare their result with the label to verify their result. The value should be close to five, giving a molar ratio of approximately five moles of water to one mole of copper(II) sulfate, which suggests the formula of the hydrated copper(II) sulfate is $CuSO_4{\cdot}5H_2O$.

Conclusion

Adrian and Bob have now reached the end of this series of articles on microscale chemistry. What started out as an antidote to the safety concerns of dealing with chemicals in schools, (storage, use, disposal) by education managers and the UK Health and Safety executive, in around 1993, has now attracted more enthusiasts because of the educational and economic benefits the techniques bring. Now we can add the promotion of sustainability, as directed by the United Nations, using the principles of green chemistry,[4] as formulated in 1998 by Paul Anastas and John C. Warner. At least 6 of the 12 principles of green chemistry can apply to school-taught chemistry.[5]

- **Prevention of waste:** droplets of solutions added to a plastic surface using transfer pipettes are just wiped away with a paper towel.
- **Less-hazardous chemical syntheses:** preparing copper sulfate crystals while avoiding scalds, burns, and the evolution of toxic gases; microelectrolysis of copper chloride solution.

- **Safer solvents and auxiliaries:** using water as the main solvent, as well as adopting salting-out procedures.
- **Design for energy efficiency:** spirit burners and hot water from a kettle can be used to avoid the use of fossil fuels (e.g., the Bunsen burner); using more energy efficient LEDs.
- **Catalysis:** using yeast to produce oxygen from hydrogen peroxide.
- **Inherently safer chemistry for accident prevention:** reducing concentrations, finding an alternative procedure to carry out the electrolysis of a molten lead bromide, and conducting small-scale catalytic cracking to avoid suck back.

This last principle is what CLEAPSS and SSERC in the UK have been doing since 1963.

We are often accused of removing the 'wow' moments that school chemistry brings. With the microchemistry approach, there are still explosions (dynamite soap bubbles), and there are more wow moments, such as the beauty of an array of colours in droplet art.[6] There are completely new demonstrations. Bob recently carried out a demonstration showing the electrical conductivity of molten sodium chloride, an observation that is quoted in many school texts as evidence of ionic bonding, but never easily demonstrated until now, by using microscale techniques[7] and the bottle-top crucible described in this article.

Acknowledgements

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References

[1] Worley B, Paterson D (2021) *Understanding Chemistry through Microscale Practical Work* pp 38-41. Association for Science Education. ISBN: 978-0863574788

[2] The Science on Stage webinar on microscale chemistry: https://youtu.be/LM97yXJlotQ?si=e_IGnqLuTiJdPV84

[3] The Royal Society of Chemistry resource to teach the cracking of long-chain hydrocarbons: https://edu.rsc.org/exhibition-chemistry/cracking/4010515.article

[4] The 12 principles of green chemistry: https://www.compoundchem.com/2015/09/24/green-chemistry/

[5] Green chemistry principles applicable in school chemistry: https://microchemuk.weebly.com/green-chemistry.html

[6] An article on chemical droplet art: https://uwaterloo.ca/chem13-news-magazine/september-2019/feature/indicator-droplet-art

[7] A video on the electrolysis of molten sodium chloride: https://www.youtube.com/watch?v=wKgDJYY6Vkk&t=60s

Resources

- Watch a webinar on microscale-chemistry techniques.
- Read about the 12 principles of green chemistry.
- Read an introduction to microscale chemistry in the classroom: Worley B (2021) Little wonder: microscale chemistry in the classroom. *Science in School* **53**.
- Discover simple adaptations of experiments to make chemistry accessible to students with vision impairment: Chataway-Green R, Schnepp Z (2023) Making chemistry accessible for students with vision impairment. *Science in School* **64**.
- Enhance your students' understanding of electrolysis using microscale chemistry techniques: Worley B, Allan A (2022) Elegant electrolysis – the microscale way. *Science in School* **60**.
- Use microscale techniques to do quantitative chemistry experiments: Worley B, Allan A (2023) Quick quantitative chemistry – the microscale way. *Science in School* **63**.
- Teach the chemistry of precipitation using microscale-chemistry methods: Worley B, Allan A (2022) Pleasing precipitation performances – the microscale way. *Science in School* **57**.
- Make chemistry practice fun with chemical card games: Johnson P (2024) Stealth learning – how chemical card games can improve student participation. *Science in School* **68**.
- Use geometry to estimate the CO2 absorbed by a tree in the schoolyard: Schwarz A et al. (2024) How much carbon is locked in that tree? *Science in School* **67**.
- Try some experiments with gases to illustrate stoichiometric reactions and combustion: Paternotte I, Wilock P (2022) Playing with fire: stoichiometric reactions and gas combustion. *Science in School* **59**.
- Promote critical thinking by adding some variables to the classic candle-mystery experiment: Ka Kit Yu S (2024) A twist on the candle mystery. *Science in School* **66**.

- Explore laboratory safety with creative horror stories about lab disasters: Havaste P, Hlaj J (2024) Lab disasters: creative learning through storytelling. *Science in School* **68**.
- Try a classroom activity to extract essential oils from fragrant plants: Allan A, Worley B, Owen M (2018) Perfumes with a pop: aroma chemistry with essential oils. *Science in School* **44**: 40–46.
- Read about the environmental costs of fireworks: Le Guillou I (2021) The dark side of fireworks. *Science in School* **55**.

AUTHOR BIOGRAPHY

Dr Adrian Allan is a teacher of chemistry at Dornoch Academy, UK. He was selected to represent the UK at the Science on Stage conferences in 2017 and 2019. He has presented Science on Stage webinars and workshops around Europe on microscale chemistry and using magic to teach science.

Bob Worley, FRSC, is the (semiretired) chemistry advisor for CLEAPSS in the UK. He taught chemistry for 20 years, and in 1991, he joined CLEAPSS, which provides safety and advisory support for classroom experiments. In carrying out these duties, he gained an interest in miniaturizing experiments to improve safety and convenience. He was awarded the 2021 Excellence in Secondary and Further Education Prize for significant and sustained contributions to the development and promotion of safe practical resources for teachers worldwide.