DOI: 10.2436/20.2003.02.184 http://scq.iec.cat/scq/index.html

ISSN 2013-1755, SCQ-IEC **Educació Química EduQ** número 24 (2018), p. 58-62

«In a little you can see a lot»: the impact of practical microscale chemistry on chemical education

«Amb poc es pot veure molt»: l'impacte de la química pràctica a microescala en l'educació química

Robert Worley / Brunel University Science Park (United Kingdom)

abstract

Micro-chemical procedures in chemistry make practical work safer and less expensive. However, for these techniques to be acceptable to United Kingdom teachers, there has to be «added value». This comes in improved classroom management, reducing cognitive load on the short term-working memory, challenging long-held misconceptions at the molecular/ionic level, and even developing completely new experiments for students and demonstrations for teachers.

keywords

Practical chemistry, microchemistry, school chemistry, safety, technique.

resum

Els procediments a microescala en química fan que el treball pràctic sigui més segur i menys costós. Tanmateix, perquè aquestes tècniques siguin acceptables per als professors del Regne Unit, hi ha d'haver un «valor afegit». La microescala promou una millor gestió de l'aula, amb una reducció de la càrrega cognitiva a la memòria de treball a curt termini, un desafiament de les idees errònies a llarg termini a nivell molecular i iònic i, fins i tot, el desenvolupament d'experiments completament nous per als estudiants i de demostracions també noves per als professors.

paraules clau

Química pràctica, microquímica, química escolar, seguretat, tècnica.

In my role as safety adviser for United Kingdom schools at CLEAPSS[1] from 1991 to 2012 and now as a consultant, I was immediately drawn to microscale chemistry because miniaturizing allowed the teacher and students to undertake experiments which on a larger scale, either became unsafe or used expensive equipment. But as you will see later, I discovered more.

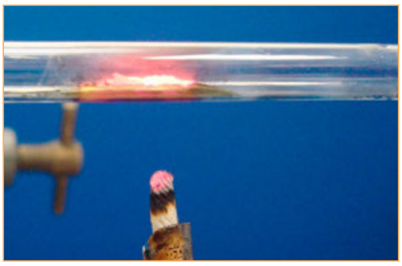


Figure 1. Copper(II) oxide being reduceced to copper with hydrogen in a Pasteur pipette with 60 ml of hydrogen stored in a syringe.

Improved safety

In 1984, after a violent explosion, a group of children were sprayed with concentrated sulfuric acid, used to dry hydrogen, prior to

---

[1] CLEAPSS consists of a small group experienced science teachers, financed by subscription of United Kingdom local authority employers and independent schools, both state and fee-paying. CLEAPSS works closely with the UK Department for Education and the UK Health & Safety Executive to ensure that teachers, school technicians and students work safely during science practical lessons. The aim is to minimize risk and reduce costs both in monetary and environmental terms, and ensure compliance to European Union and United Kingdom safety law. There are a number of schools in Europe who teach the United Kingdom and IB science curriculums that subscribe to our services as well. See *http://science.cleapss.org.uk/*.

burning it, in an experiment to reduce copper(II) oxide to copper. This teacher was the first to be fined under UK Health and Safety Law. I was teaching at the time and my employer told me not to do this experiment. I had never had an incident with it because I knew the dangers of trying to light an explosive mixture of air and hydrogen. On taking up my position at CLEAPSS, I found a safer, alternative procedure, on the *Microscale gas chemistry* website from Bruce Mattson at Creighton University (fig. 1) (*http://mattson.creighton.edu/Microscale_Gas_Chemistry.html*, *https://www.youtube.com/watch?v=b9UF6wycia8*).

There were other experiments causing safety issues in the United Kingdom, so much so that employers wanted them banned, in fear of prosecution by the Health & Safety Executive (HSE)[2] or civil action. The normal scale catalytic cracking reaction can lead to violent suck-back implosions caused by cold water coming in contact with a hot glass test tube (*https://www.youtube.com/watch?v=AajLtkJxPk0*). This just cannot happen with the closed glass Pasteur pipette method (fig. 2) (*https://www.youtube.com/watch?v=qQh2YXyFD7I*).

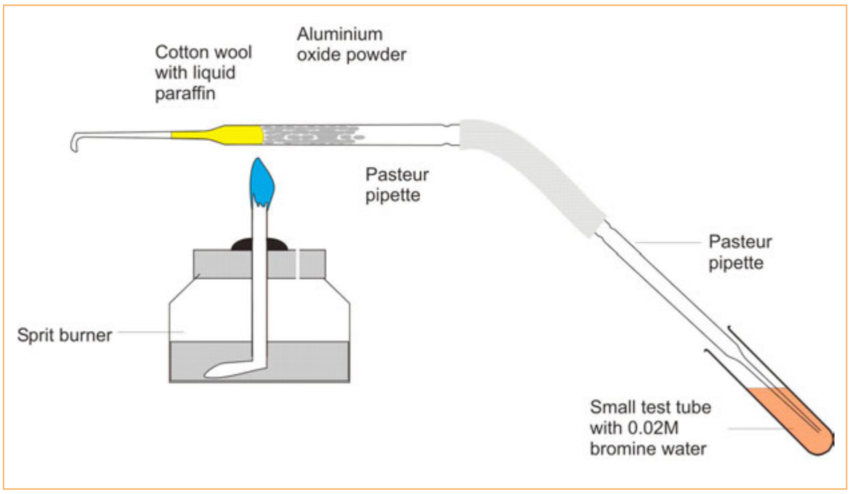


Figure 2. Catalytic cracking.

Reduced costs

A common response from teachers is that it saves on the cost of chemicals. However, there is a significant reduction on cost of equipment as well.

Schools in the United Kingdom buy a large-scale Hofmann voltameter (fig. 3*a*) for over £100 (but the taps can seize up through lack of use), and platinum electrodes for £70 (but the platinum electrode break due to pour soldering). The microscale version can be made for less than £30. With the three-way taps at the top of the syringes, the electrolyte can be raised and the gases removed for testing. The safety bonus is that saturated sodium sulfate solution can be used as the electrolyte in place of 1M sulfuric acid. The educational bonus is that bromothymol blue indicator can be added to show that the area around the anode is acidic and the area around the cathode is alkaline:

— At the cathode: $2H_2O(l) + 4e^- \rightarrow 2OH^-(aq) + 2H_2(g)$.

— At the anode: $2H_2O(l) \rightarrow 4H^+(aq) + O_2(g) + 4e^-$.

The ratio of the volumes of gases is 2:1, as predicted by the equations. The gases can be tested individually, mixed and exploded, and even used to fire a 3 mL plastic pipette bulb to the ceiling.

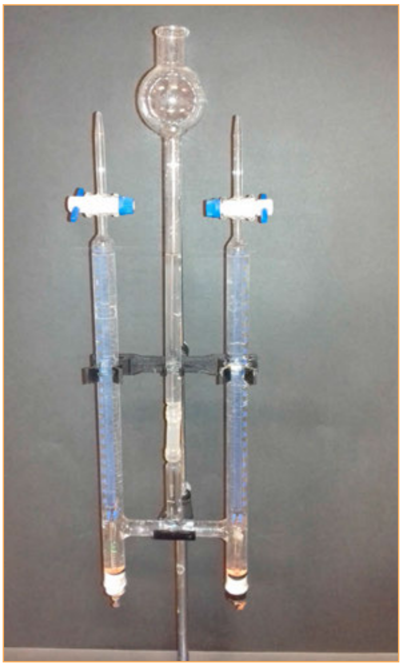


Figure 3a. Full-size Hofmann voltameter.

Figure 3b. Microscale Hofmann voltameter.

The colorimeter design has been developed from an idea from Norway (Kvittingen *et al.*, 2014). Normally, a machine would cost a minimum of £100.

In the version shown in fig. 4, a light-emitting diode (protected

---

[2] The HSE oversees Health & Safety Law in the United Kingdom. They have wide-ranging power to prohibit unsafe practices and prosecute those employees or employers at fault. See *http://www.hse.gov.uk*.

by a resister) supplies light of a particular wavelength (so no need for filters). The transmitted light is collected by an infra-red LED which provides a voltage on a multimeter.

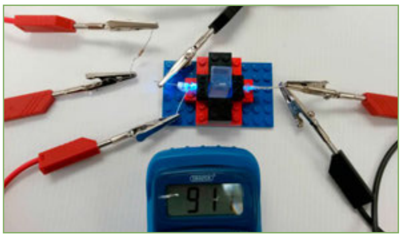


Figure 4. «Lego» colorimeter.

The equipment gives promising results. A series of solutions was made to determine the percentage of copper in brass and the graph in fig. 5 shows how the results follow Beers' Law. It is proposed that Arduino technology is now applied so that rapid changes in concentration can be followed as in reaction kinetics.

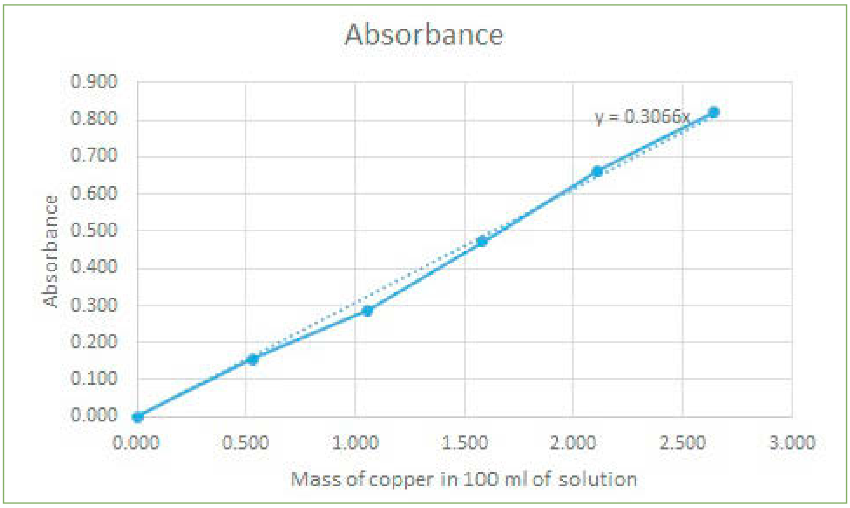


Figure 5. Absorbance of red light of copper(II) nitrate solutions.

Breakages are a constant worry to teachers in practical lessons. Porcelain crucibles crack when repeatedly heated to high temperature and holding magnesium ribbon to find the increase in mass. The results have a poor success rate as well. Crucibles (fig. 6*a*) were never designed for the repeated quantitative combustion of magnesium but for extracting precipitates from ashless filter paper in analytical chemistry and for taking samples of molten steel prior to analysis. Surprisingly to teachers, they are designed for single use only. This is why they often crack under intense heat. Students find it difficult to remove the cover to let air in. However bottle tops, with the plastic insert burnt away in a fume cupboard or outside, can be used and give extremely good results. For magnesium combustion, two bottle tops are sandwiched together and there is no need to move them with tongs during heating (fig. 6*b*). For analysis of hydrated salts, a nut and bolt arrangement is used (fig. 6*c*), and the bolt can be held over a spirit burner (avoids further decomposition of the anhydrous salt) with tongs, a clamp or even pliers from the hardware shop.

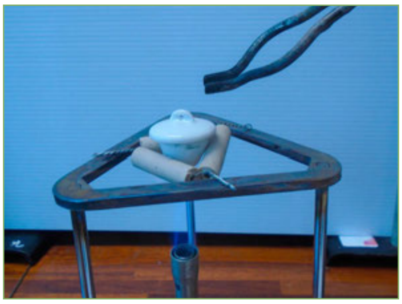


Figure 6a. The traditional method.

Figure 6b. Using bottle tops.

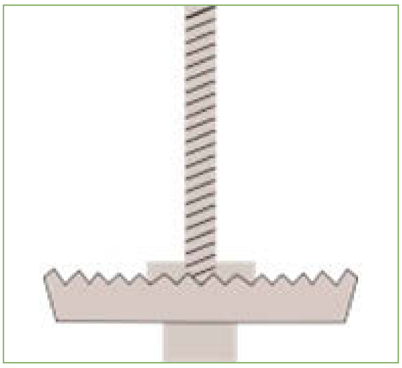


Figure 6c. The indestructible crucible.

The puddle: replacement of the test tube

The puddle is formed by adding one to four drops of an aqueous solution onto a hydrophobic surface such as a polypropylene sheet where the contact angle is over 90° (fig. 7). The addition of organic reagents can reduce the angle making the puddle spread out, but the presence of dissolved salts increases the angle. The angle is smaller with plastics such as polystyrene, polythene and PET (the covering on laminates), but the experiments still work.

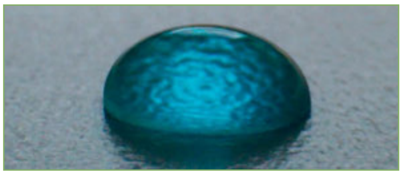


Figure 7. A puddle of 1M copper sulfate solution on polypropylene.

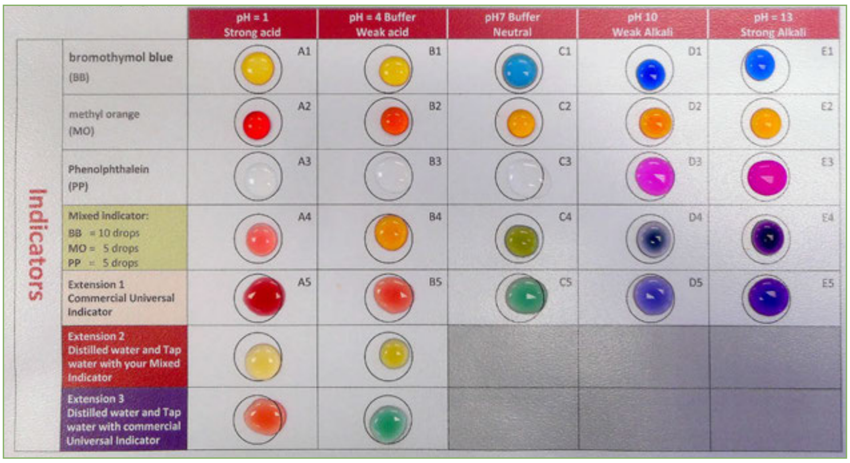
Working on procedural worksheets inserted into a polypropylene plastic folder, reactions can be carried out in the hemispherical droplet. In fig. 8, three to four drops (number depends on the diameter of the plastic pipette) of each buffer is placed in the columns of the first five rows. The indicators, bromothymol blue, methyl orange and phenolphthalein solutions, are then added across the rows. In the fourth row, the indicators are mixed to make a universal indicator which can be compared to a manufactured version in the fifth row. Finally, these mixed indicators can distinguish between distilled water, often acidic because of dissolved carbon dioxide, and tap water, often containing hydrogencarbonate ions.

Displacement and precipitation reactions can also be performed on the plastic sheet.

Diffusing gases and halos in puddles

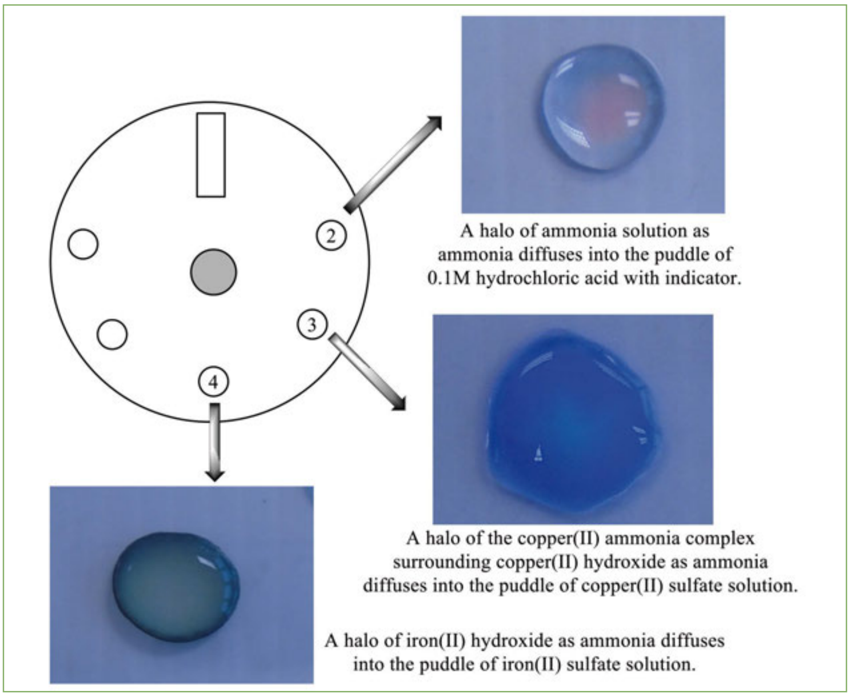
For studying the chemistry of ammonia (toxic) is used a Petri dish placed on top of an instruction sheet. Ammonia gas is generated from the addition of 2M ammonia solution to which anhydrous calcium chloride (warms the solution up a little). As ammonia gas diffuses within the Petri dish precipitates, complexes and colour changes with indicators are observed. There are beautiful halo effects as shown in fig. 9. Discussion of these halo effects allows classes to recap ideas about movement of gas molecules and observing diffusion in gases and liquids. Other gases, even toxic sulfur dioxide and hydrogen sulphide, can be investigated in a similar manner because levels of gas in the room are kept well below workplace exposure levels.

I had never seen these beautiful halo effect before. Here was evidence of molecules diffusing through air in the Petri dish, reacting with the outer layer of the chemicals in the puddle and gradually diffusing through the puddle. With copper sulfate solution, there were two halos as the precipitate formed followed by the dark blue tetraaminecopper(II) ion. It was at this point, carrying out chemical reactions in a puddle rather than a test tube, that produced observations required explanations which could enhanced the understanding of chemistry.



Indicators	pH = 1 Strong acid	pH = 4 Buffer Weak acid	pH7 Buffer Neutral	pH 10 Weak Alkali	pH = 13 Strong Alkali
bromothymol blue (BB)	A1	B1	C1	D1	E1
methyl orange (MO)	A2	B2	C2	D2	E2
Phenolphthalein (PP)	A3	B3	C3	D3	E3
Mixed indicator: BB = 10 drops MO = 5 drops PP = 5 drops	A4	B4	C4	D4	E4
Extension 1 Commercial Universal Indicator	A5	B5	C5	D5	E5
Extension 2 Distilled water and Tap water with your Mixed Indicator					
Extension 3 Distilled water and Tap water with commercial Universal Indicator					

Figure 8. Indicators changing colour with changes in pH.



A halo of ammonia solution as ammonia diffuses into the puddle of 0.1M hydrochloric acid with indicator.

A halo of the copper(II) ammonia complex surrounding copper(II) hydroxide as ammonia diffuses into the puddle of copper(II) sulfate solution.

A halo of iron(II) hydroxide as ammonia diffuses into the puddle of iron(II) sulfate solution.

Figure 9. Halo effects seen from diffusion of ammonia gas into droplets of solutions of: (2) hydrochloric acid with indicator; (3) copper(II) sulfate, and (4) iron(II) sulfate contained within a Petri dish.

When carrying out this experiment in the United States of America, a teacher suddenly said: «Gee, in a little you can see a lot!».

These methods have potential for use in public engagement and outreach activities for events such as science fairs. The small volumes of solutions used makes experiments carried out this way easily transported with minimal waste which can easily be contained.

Opposition and support from teachers

Despite giving superior results and observations, some teachers claimed the use of bottle tops, Lego colorimeters, plastic folders, etc., would disadvantage students should they come across questions in an exam. In part, this suspicion was why an initiative by the Royal Society of Chemistry in 1999 to provide schools in the United Kingdom with free samples of microscale chemistry kits and a book (Skynner, 1998) fell on stony ground. Schools in the United Kingdom were relatively well equipped with laboratories and there remains a strong tradition of the use of mainstream macroscale equipment such as glass test tubes, burettes and Bunsen burners, further reinforced by national examinations requiring familiarity with such techniques for their assessment. I am assured this is not true by exam boards, but the suspicion survives. Despite, inventing and selling plastic materials to the whole world as an alternative to glass, some school chemistry teachers still have a deep suspicion of the use of plastics in the laboratory.

However, some teachers informed us that these methods increased speed and efficiency in lessons as well time saved in clearing up, thus allowing time for teaching. The cracking procedure (fig. 2), as well as being safe, could be set up and completed in 20 minutes, allowing time to discuss the chemistry. The indicators on a plastic sheet (fig. 8) was over in 15 minutes with photographs taken. If there had been fifteen groups doing this experiment, four hundred and thirty-five test tubes would have had to be sourced and cleaned.

Plastic pipettes are used to transfer chemicals from vials. They come in different sizes and bulb volumes. It is very useful to know how many drops a pipette will deliver to 1 mL of liquid. There are plastic pipettes that deliver twenty, thirty and fifty drops respectively. Dropping bottles are very useful and obtained in bulk from medical and veterinary suppliers.

Conclusion

If a United Kingdom teacher of chemistry in 2017 were to look at a laboratory in 1917, that teacher would identify and use the same equipment. Perhaps, it is time to move on and give chemistry a more modern image alongside biology and physics.

When I started microscale, the emphasis was on safety. In order to have the procedures used by teachers, I had to look further than cite less expensive equipment and safer procedures as the main attributes. Instead I had to find «extra value» for these techniques over the traditional technique used for over one hundred years. Now I have reversed the priorities so the impact of microscale on practical chemistry in education describes:

— How it can be used to improve students' and teachers' understanding of how particles interact the sub-micro or nano-level.

— How it reduces the overload on the short-term working memory of students.

— How it improves the speed and efficiency of lessons and classroom management.

— How it uses new materials.

— How it incorporates green and sustainability credentials.

— How teachers can present chemistry in a non-laboratory environment.

— How it can reduce the cost of practical work.

— How it can improve safety.

Acknowledgements

Dr. Kay Stephenson is fantastic in discussions and presentations in how practical work assists student's understanding of chemistry. Mary Owen BSc improves immeasurably on my crude ideas and designs of equipment. Steve Jones, the director of CLEAPSS, has encouraged my further research in this area, when I should be retired.
I would also like to thank Prof. Fina Guitart and the organisers of ECRICE 2016 for inviting me, an ex-teacher, to display these new techniques to the academics of chemical education. I hope it encourages further research into how teachers in schools can apply the findings of academics in chemical education in using new practical techniques as a direct teaching tool in the understanding of chemistry, not just to train chemists.

References

Asheim, J.; Kvittingen, E. V.; Kvittingen, L.; Verley, R. (2014). «A simple, small-scale Lego colorimeter with a light-emitting diode (LED) used as detector». *J. Chem. Educ.*, vol. 91, No. 7, p. 1037-1039.

Skynner, J. (ed.) (1998). *Microscale chemistry: Experiments in miniature*. London: Royal Society of Chemistry.

Bob Worley
Is the (retired) chemistry advisor for CLEAPSS (*www.cleapss.org.uk*) in the United Kingdom. He acquired degrees in Chemistry at the University of Sheffield. He then taught students for twenty years, sixteen years as head of department at Norwich School in the United Kingdom, and finally twenty-one years at CLEAPSS. However, he cannot simply leave chemistry and education, so he now work part time for CLEAPSS solving all manner of technical and teaching problems.
E-mail: *bobworley4@gmail.com*.