In a Little You Can See a Lot

The impact of practical microscale chemistry on chemical education

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The microscale approach to practical chemistry is not new. I remember semi-micro analysis in my A-level practical in the 1960s. However, those techniques are all but lost to teachers of today. Of course chemists carry out micro chemical activities naturally such as melting point determination and Thin-Layer Chromatography.

In the 1980s, many American Organic Chemists in Universities made a conscious effort to go microscale using novel equipment (Fig 1)[ii] and still do although it less popular in the UK. At school level, it was the pioneering effort by Professor John Bradley which was taken up by UNESCO (Fig 2), expanded to other sciences and sent many developing countries around the world[iii]. Success is not as overwhelming as UNESCO would like and John Bradley has recognised this[iv]. The issue is with government

Fig 1: Microscale organic reaction flasks

Fig 2 Microscale kit in use in South Sudan

The kit was sent to every school in the UK by the Royal Society of Chemistry in 1999 accompanied by an excellent book[v]. Many of our traditional chemists looked at the kit, made a rude a remark and threw it in the draw or worse. There is still a belief that it is cheap plastic, not real chemistry ("no Bunsen burner") but more importantly not recognised as *bone fide* methods by examination boards and text book authors. There are still a few schools that make use of the kit. It was sent to Ireland as well. The problem with kits is maintenance, training and credence plus it constrains further innovation.

I began Microscale and Reduced Scale Procedures to enable experiments to be carried out safely but the use of modern materials extended my interest into other areas of chemical education. I made one basic decision. It was not to be a kit but use readily available modern materials and equipment. I also cam e at the subject from a different direction; safety.

In 1984, after a violent explosion, a group of children were sprayed with concentrated sulfuric acid, used to dry hydrogen, prior to burning it, in an experiment to reduce copper(II) oxide copper. This teacher was the first to be fined under Health and Safety Law. Looking for a safer, alternative procedure, I found the methods of Bruce Mattson[vi] inspiring. See Fig 3. There were other experiments causing issues, so much so that some employers wanted them banned. The normal scale catalytic cracking reaction (Fig 5) can lead to violent suck-back implosions cause by cold water in contact with a hot glass test tube. This just cannot happen with a glass Pasteur pipette. You can read more about these in my website.[vii]

For example, the iron/sulfur reaction (Fig 5a) was causing distress to asthmatics when performed poorly. I was able to use small scale chemistry to perform the reaction and do a comparison of the mixture and compound (Fig 5b).

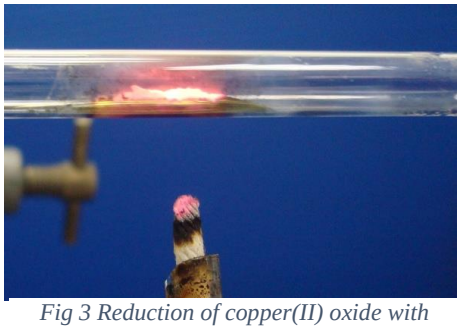


Fig 3 Reduction of copper(II) oxide with hydrogen

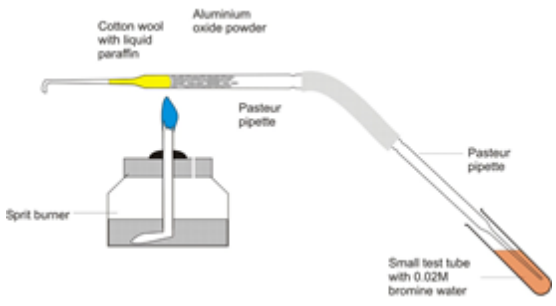


Fig 4 Catalytic cracking set up

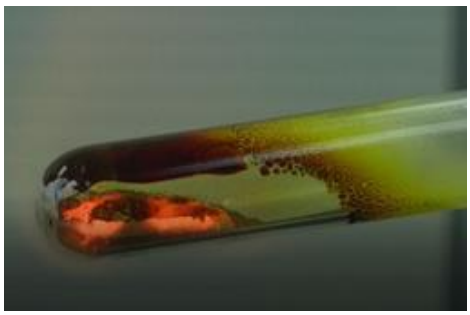


Fig 5a : 0.2 g of iron/sulfur mix ignites

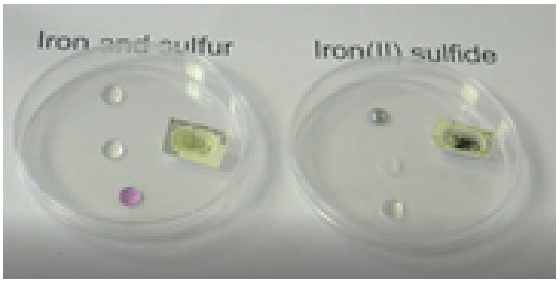


Fig 5b: Comparing the reactions of the compound and the mixture

Teachers often think that the microscale approach saves money on chemicals. This is true to some extant as 250g of x will last longer, but in fact it is often in equipment where the money is saved by going microscale Schools in the UK often report that the platinum electrodes on the Hofmann voltameter have come off. They are very expensive to replace but by using platinum wire, a microscale version has been devised (Fig 6). This equipment uses sodium sulfate solution as the electrolyte rather than sulfuric acid. This is not only safer but now, by using an indicator such as bromothymol blue, the acidic and alkaline environment around the anode and cathode respectively is easily demonstrated. And the ratio of gas by volume is 2:1, hydrogen oxygen. It also used a novel method of drawing off the gas, using plastic 3-way taps which can be attached to plastic syringes.

The Lego® support for the colorimeter may grab the headlines but is the remarkable property of that when light strikes a Light Emitting Diodes (LED), a voltage is produced[viii]. We found that light from a coloured LED has a narrow wavelength so no filters are required and a IR LED will respond to any wavelength of light of a shorter wavelength to produce a voltage read on an inexpensive multimeter (Fig 7).

Fig 6 Hofmann Voltammeter

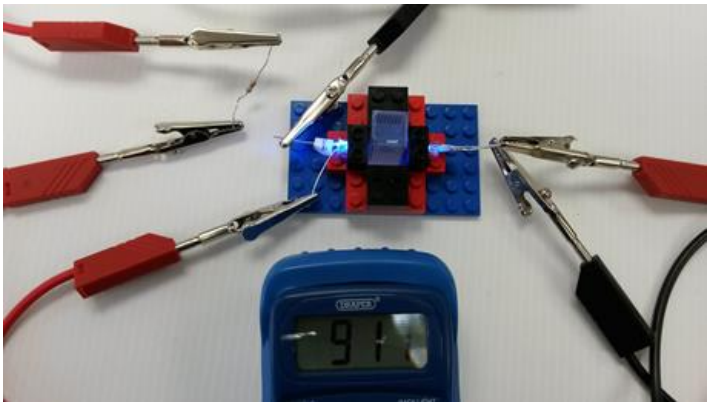


Fig 7: Lego colorimeter

Crucibles were never designed for the quantitative combustion of magnesium. They were designed for precipitates in ashless filter paper in analytical chemistry and more recently 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 oxygen in. The use of bottle tops with the plastic insert burnt away in a fume cupboard or outside. As a replacement for crucibles, the bottle tops gives extremely good results (Fig 7). For magnesium combustion, two bottle tops are sandwiched together and there is no need to touch them with tongs during heating as air can get inside the arrangement. For analysis of hydrated salts a nut and bolt arrangement is used (Fig 8) and the bolt can be held over a spirit burner (avoids further decomposition of the anhydrous salt) with tongs, a clamp or better still pliers from the hardware shop. To weigh this apparatus, the digital balances, often available for less than 10 Euros from online suppliers mean that a class set can be bought for the cost of a traditional balance. The one shown(Fig 10) has a capacity of 100g and reads to 2 decimal places; And it can be calibrated!

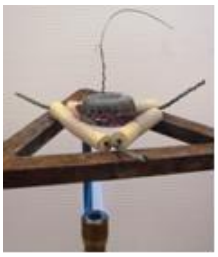


Fig7: Quantitative oxidation of magnesium

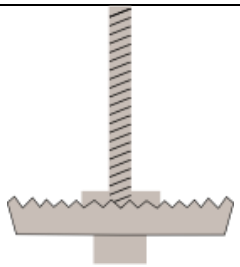


Fig 8: Adaption to a Crucible

Fig 9 Inexpensive digital balance

It is here I hit a problem. Despite giving superior results to crucibles, some teachers claimed the use of bottle tops, Lego colorimeters, etc, are established equipment and would disadvantage students should they come across questions in an exam. I am assured this is not true by exam boards but the suspicion survives.

I then began to receive anecdotal comments 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 4), as well as being safe, could be set up and completed in 20 minutes, allowing time to discuss the chemistry.

Working on procedural worksheets inserted into a polypropylene plastic folder, reactions could be carried out in the hemispherical droplet. When Professor Bruce Mattson at Creighton University saw this he christened it “Puddle Chemistry” after using it with his students. In Fig 9, 3 to 4 drops of each buffer is placed in the columns of the first 5 rows. The indicators are then added across the row. In the fourth row, the indicators are mixed which makes a universal indicator which can be compared to a manufactured version. Finally these mixed indicators can distinguish between distilled water, often acidic because of dissolved carbon dioxide and tap water, often containing hydrogencarbonate ions.

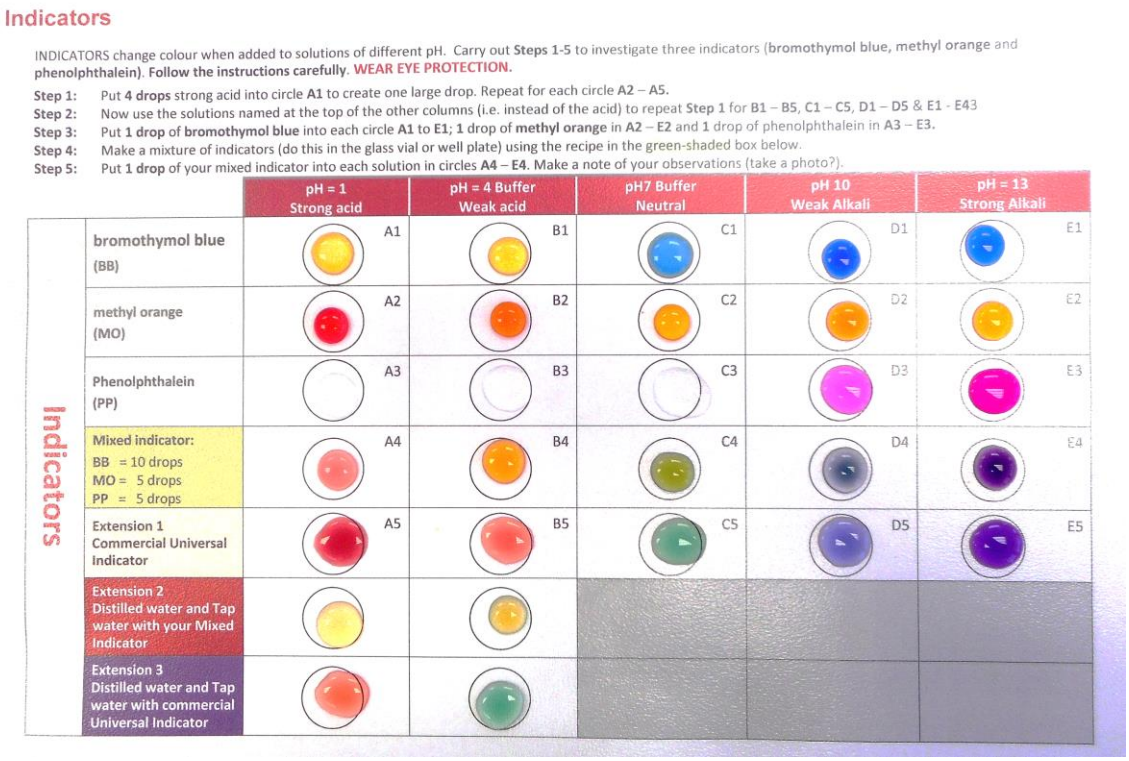
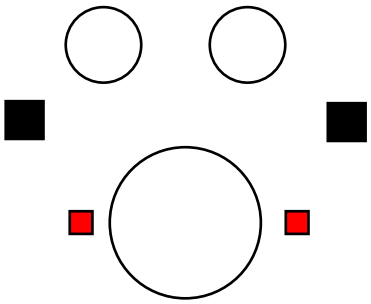


Fig 9:Indicators changing colour with pH

Displacement and precipitation reactions can also performed on plastic sheet. In doing these activities, it became apparent that teaching and learning was being enhanced. Precipitation reactions appear as magic to most children; they are presented to them as two clear and colourless liquids, which on mixing in a test tube, white or sometimes coloured (eg. yellow with silver or lead iodide) solid appears. Try this little procedure yourselves and think what is going on, how does this happen, can I produce a visual model of what is happening? You will need to photocopy/download/print the page and insert the page into a plastic folder or laminate it. You will need distilled water, a 3 ml plastic pipette, wooden splint, scissors, solid potassium iodide and solid silver nitrate (don’t worry about cost, only a little is used; lead nitrate can also be used but this might not be allowed! With the scissors, the end of the splint can be cut off to avoid contamination after use in transferring a solid or stirring.

A microscale investigation for you

- Place enough solid to cover one of the black squares and the other solid on the other black square
- Add water with the pipette to fill the two small circles.
- With the splint move half of the solids to the water in the small circles respectively and use the splint to stir. (Cut the end of the splint away to after each mixing to avoid contamination.
- Now mix the two clear liquids.
- Now move the rest of the solid to the nearest red squares.
- Add water to the large circle.
- Cut the splint in half and move the solids into the edge of the liquid from either side. (Do not stir but wait for about 30s to see the wonderful diffusing precipitate.) It is even better with lead nitrate.
- Other solids can be investigated as well



The spectacular and colourful macro observations in chemistry makes subject is very popular with students. It becomes less popular when teachers begin to introduce explanations at the symbolic and sub-micro or nano level as exemplified by the famous Johnstone Triangle (Fig 10), which summarises the difficulties of teaching chemistry. I hope the macro observation you made in your experiment is explained by Fig 11.

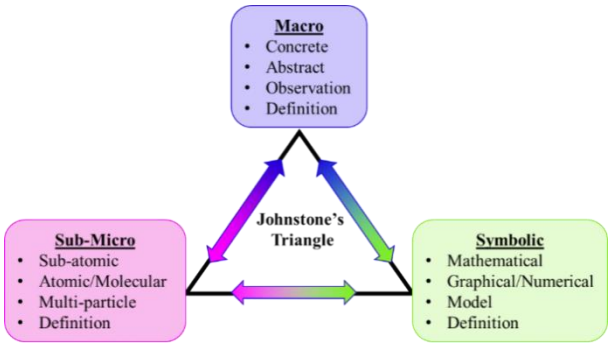


Fig 10: Johnstone trinagle

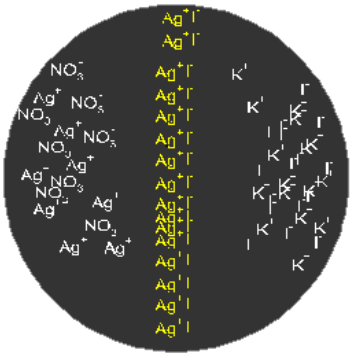


Fig 11 Ionic Model of the diffusing precipitate

But where is the evidence for ions? The evidence can be shown by the conduction of an electric current and CLEAPSS have made small conductivity indicator which can be made for less than £10

Fig 12a

Fig 12b

Fig 12c

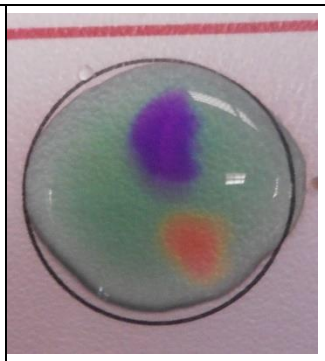


Fig 12d

Fig 12a shows the indicator with a red LED which is connected to a 330 to 500Ω resistor and 9 volt battery. The electrodes are carbon fibre available from online kite and hobby shops. In distilled water the LED does not light but in tap water (Fig 12a), it does. Fig 12b shows the addition of 1 grain of salt to a puddle of distilled water. With a little stirring the LED lights up showing the solution is now conducting and addition of Universal indicator show that chemical reactions are taking place (Fig 12d) at the electrodes (electrolysis). When doing these experiments in the USA, a teacher suddenly said "Gee, in a little you can see a lot!"

There is still an issue though. Is the solid made of ions or do the ions occur with the addition of water? The evidence here is that a molten salt conducts electricity (Fig 13a). The microscale approach now comes into its own although leap of faith has to be made from sodium chloride (Mpt 804°C) to silver bromide (Mpt 432°C). Not only does a LED light up but the bromine is seen at the anode (Fig 13b) and so little is produced that it can be carried out in the classroom. The glass tubing is 7mm medium borosilicate tubing which is bent slightly using the heat from a Bunsen burner.

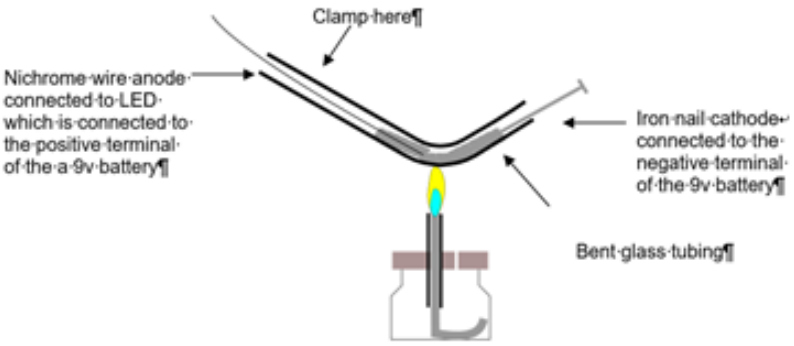


Fig 13a

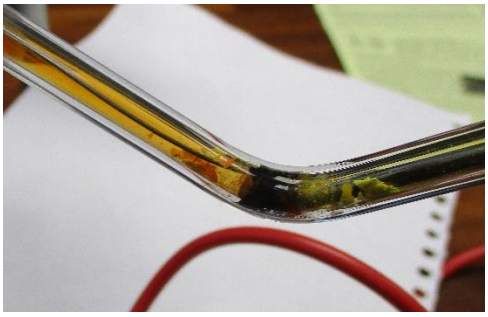


Fig 13b

Another aspect of the microscale approach is that it removes much of the paraphernalia which can confuse students in a practical lesson[ix] Practical work experiment has a "noisy start" with much going on which obscures the point the teacher is trying to make. Class teaching involves a simple idea which is elaborated on.

Titration is one such subject which combines all three aspects of the Johnston triangle along with skills in handling volumetric flasks, pipettes and filler, filling and using burettes. Added to that is to students a very difficult calculation involving balanced chemical equations. Microscale titration can deliver a simple introduction

It uses very fine pipettes which deliver 50 drops to the ml. These act as the burette (Fig 14). The clamp is turned so that drops are delivered one at a time until the indicator just changes colour. The measurements are made by weighing not by volume and the lap of faith is to assume that the density is $1gcm^{-3}$. By carrying out 3 weighings, the calculation can be carried out. The skill involved in using the professional equipment can be taught later. Having said that, the results are extremely good and the technique can be used for measuring yields in organic chemistry preparations and determining equilibrium constants.

Fig 14 Microscale titration

It has been observed that the speed and efficiency shown by students working on plastic sheets, microscale cocking and titration does overloading students with too much information (ie, short-term working memory) before a practical.

The photograph in Fig 15 shows students carrying practical chemistry, 100 years ago, in 1917. The same equipment is available still (in many cases the same experiments). We cannot say the same about biology equipment and experiments. I feel we have to use modern materials and efficient methods to make practical chemistry relevant to students and purposeful in delivering an understanding of subject.

At last these techniques such as microscale electrolysis of copper(II) chloride Fig 16 are being recognised by examination boards in the UK as exemplar experiments. To those who claim it is too small to be seen, modern projection equipment no has web cams, usb microscales and visualizer to aid the teacher. You may be fortunate in obtaining copper crystal growth from the cathode as shown in fig 17.

Fig 15: School Chemistry laboratory 1917

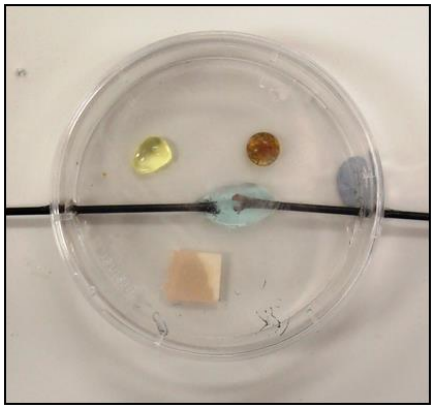


Fig 16: Electrolysis of copper(II) chloride solution. Chlorine diffuses from the anode to react wit 0.5M potassium bromide solution and 0.1M potassium iodide solution and bleaches dam blue litmus paper.

There is a website I have made which shows goes into more detail[x] and there are videos on the CLEAPSS You-tube channel[xi].

Fig 17Copper crysatls forming at the cathode during electrolysis. This was taken with an expensive Dinolite usb microeacle but crystal grown can be seen with a Veho usb microscale which retails at about £40.

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[i] www.science.cleapss.org.uk

[ii] Mayo, D W; R M Pike; S S Butcher (1986). *Microscale Organic Laboratory*. New York, NY: John Wiley & Sons. ISBN 0-471-82448-8. Williamson, K L (1989). *Macroscale and Microscale Organic Experiments*. Lexington, Mass: D C Heath. ISBN 0-669-19429-8.

[iii] http://www.iocd.org/WhatWeDo/microscience.shtml

[iv] http://www.ajol.info/index.php/ajce/article/viewFile/130621/120199

[v] *Microscale Chemistry: Experiments in Miniature*, John Skinner, RSC, 1998, ISBN-13: 978-1870343497

[vi] http://mattson.creighton.edu/Microscale_Gas_Chemistry.html

[vii] www.microchemuk.weebly.com

[viii] Based on work by L Kvittingen et al, J. Chem. Educ., 2014, 91 (7), pp 1037–1039

[ix] JOHNSTONE, A.H. and WHAM, J.B. (1982). The demands of practical work. Education in Chemistry,19 (7) p. 71-74.

[x] www.microchemuk.weebly.com

[xi] https://www.youtube.com/user/CLEAPSS