Experimenting with undergraduate practicals

Paper

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Abstract

This article provides an account of a practitioner's experiences and observations in a transition from the use of traditional (expository) style practicals to problem-based practicals in undergraduate chemistry laboratories. Specific examples are used to illustrate the principal features of the different styles of practical used and a representative selection of student and demonstrator comments on their initial experiences of problem based practical work is also included.

Introduction

Only a minority of chemists would challenge the view that laboratory work is an essential and desirable component of a chemistry degree course[1] and this is reflected in the criteria for the accreditation of chemistry degree courses by the Royal Society of Chemistry (RSC), which states a minimum requirement of 400 laboratory hours exclusive of the major research project.[2] However, whilst many espouse the importance of laboratory work in chemistry degree courses, it may be argued that, too often, insufficient consideration is given to the purpose these 400 hours fulfil and the reality on the ground is often a lack of clarity of purpose in much of what the students actually do. Given the very considerable resources (time, money, space, equipment etc.) devoted to support undergraduate laboratory work in chemistry it is unfortunate that the findings of Johnstone and Wham[3] in 1979 "*…that in the midst of an apparently active learning situation, it is possible for the student to be passive with his brain in neutral*" still strikes a resonance.

In 1995, Meester and Maskill[4] reported the results of a survey of first year practical classes from seventeen universities in England and Wales. They concluded:

"The aims of the course are stated in only half the manuals. The aims for the experiments are mostly contained in the experiment descriptions. Useful learning objectives are mentioned just once. The scientific level of the experiments does not exceed that of controlled, predictable experiments. Changes that have taken place in the style of practicals in secondary education are hardly reflected in tertiary education".

Meester and Maskill's study indicates that, at the time of their study, much undergraduate laboratory work in chemistry involved recipe style experiments with little opportunity for development of skills. Has the situation changed significantly since then? Alternative approaches to laboratory teaching have been published, such as problem-based learning,[5] but it is not clear whether such alternative approaches are significantly represented in undergraduate chemistry courses in the UK. In the US, recommendations made by the National Research Council for more inquiry-based learning in science education have clearly stimulated activity in the development of inquiry-based learning.[6, 7]

Johnstone and Al-Shuaili[8] recently reviewed the literature on the relationships between practices in undergraduate laboratory work and student learning. Their review includes an examination of types of laboratory work, which is based on Domin's[9] analysis. Domin[9] identifies four distinct styles of laboratory experiments (expository (or traditional/verification), inquiry, discovery and problem-based) that are distinguished in relation to 'outcome', 'approach' and 'procedure'.

The purpose of the present article is to relate the factors that stimulated a practitioner to alter the style of laboratory practical from traditional (expository) to a more problem-based style and to highlight some of the principal differences in terms of what the students do and experience with these different styles of practical work. A representative selection of student and postgraduate demonstrator comments on their experiences of problem-based practicals is included and serves to highlight some of the challenges associated with their use in undergraduate chemistry courses.

Discussion

When I commenced full time teaching of chemistry in HE in 1993, the laboratory manuals/courses within my remit were rather dated and entirely of the traditional expository style described by Domin.[9] Improvements to the laboratory manuals included attention to clarity in descriptions of procedures, rigour in equations, quantities and units and clarity in stating the purpose of the experiment. Post-lab activities, in the form of assessed questions designed to probe students' understanding of the experimental procedure and the background theory, were also included. Pre-lab work amounted to *"please ensure that you have read the laboratory script before coming to the laboratory"*.

In the process of marking some 2nd year physical chemistry laboratory reports about three years ago, I realised that the prescriptive way the practical work had been designed was generating 'good' reports with 'correct' data analysis and 'correct' results from most students. I found myself awarding first class marks to reports by students who I knew (from tutorials and other evidence) didn't really understand what they had done, why they had done it or what the results meant. Intuitively, I realised this was a poor learning experience for many students. Instead of the students doing the experiments, effectively I was trying to do the experiments (and much of the data analysis) through the students by providing them with increasingly precise instructions. I started to consider an alternative approach, recognising that the only way to learn certain aspects of experimental chemistry is for the student to design the experiment herself, reflect on any shortcomings of her design, make improvements and learn from her mistakes. This concurs with the *QAA Chemistry Benchmarking document,*[10] which states that graduate chemists should have developed "*Competence in the planning, design and execution of practical investigations, from the problem recognition stage through to the evaluation and appraisal of results and findings; this to include the ability to select appropriate techniques and procedures".*

I therefore developed a number of undergraduate chemistry practicals that are predominantly characterised by a problem-based approach in tandem with the use and development of transferable and subject-specific skills. I developed a number of practicals in physical chemistry (some from existing traditional style practicals[11, 12] and some adapted from or inspired by the literature[13, 12]), which feature clearly formulated and explicit objectives, but which omit detailed instructions to a greater or lesser extent. An additional, implicit feature in the design of some of these experiments is an attempt to encourage students to de-compartmentalise their subject knowledge (e.g. handling organic reaction mechanisms in a physical chemistry practical). The practicals have been used successfully across several modules at levels 1 and 2 during the past three years at Keele and some have been disseminated via the LTSN[12] and elsewhere.[14] Other practitioners at Keele have adopted similar approaches to the design of chemistry practicals and an account of one such experiment was published recently in this journal.[15]

To illustrate the principal features of the different practicals styles, aspects of two 2nd year physical chemistry practicals are discussed in more detail below:

The Influence of Ionic Strength on the Solubility of Barium Iodate Monohydrate

This experiment is used in a second year physical chemistry practical course and relates to lecture material on ion-ion interactions and Debye-Hückel theory. By the time the students commence this experiment they have covered much of the relevant material in the lectures.

This experiment is concerned with the influence of ionic strength on the solubility of barium iodate monohydrate and the use of experimental data to obtain the solubility product and mean activity coefficients of the barium and iodate ions. This practical ran in traditional prescriptive style at Keele up until 2000-2001 before being transformed into a problem-based practical. It should be noted that the time allocation for the traditional style experiment was around five hours but for the problem-based style it is closer to ten hours, which reflects increased time demands of problem-based practical activities.[9]

The traditional script describes a colorimetric method in which the concentration of iodate in solution is determined by quantitative conversion of the iodate to iodine using iodide, with the ionic strength being varied using KCl. The introduction to the practical covers the background theory and provides references to background reading. The experimental procedure and data analysis are detailed and prescriptive and implicitly address the student as a passive instrument. The main pitfalls in the experimental procedure and data analysis are spelled out explicitly so that most students negotiate a smooth path from experiment to report. To illustrate the style of the traditional script, the main parts of the experimental section of this practical are reproduced in Figure 1.

Figure 1. Extract from traditional prescriptive laboratory script.

Solubility of Barium Iodate Experimental Section (Traditional Practical)

The solubility of $Ba(IO_3)_2.H_2O$ *is measured by determining the concentration of* IO_3^- *in equilibrium with the solid for a range of solutions of differing ionic strengths. This is done by removal of an aliquot of the supernatant liquid followed by quantitative reduction (using iodide,* I^-*) of* IO_3^- *to* I_2*. As* I_2 *is coloured (*IO_3^- *is colourless), its concentration can be measured conveniently by visible absorption, and this can then be used to deduce* $[IO_3^-]$*. The apparatus used is a colorimeter, which is an instrument that measures absorbance at selected fixed wavelengths.*

(i) Apparatus

Digital colorimeter, 1 cm cuvettes, stoppered bottles, 25°C constant temperature water bath, volumetric glassware, solid $Ba(IO_3)_2.H_2O$*, solid KCl,* I_2 *solution (0.05 mol* dm^{-3} *in 0.1 mol* dm^{-3} *KI solution), KI solution (0.1 mol* dm^{-3}*), HCl solution (2 mol* dm^{-3}*), de-ionised water.*

(ii) Beer-Lambert Calibration Plot

Prepare a series of I_2 *solutions containing KI (iodide ions (*I^-*) enhance the solubility of* I_2 *by forming the* I_3^- *ion) using the solutions provided, such that the* I_2 *concentrations span the range,* $[I_2] = 10^{-4}$ *to* 5×10^{-3} *mol* dm^{-3} *(~8-10 solutions should be sufficient - use the stock KI solution for dilutions).* ***Prior to each absorbance measurement****, place a cuvette, containing KI solution only, in the sample chamber of the colorimeter and* ***set the reading to zero****. Using the most concentrated solution, select the optimum wavelength (~500 nm) for the absorbance measurements (consult a demonstrator for advice if you are unsure about this step). Next, measure the absorbance of each solution and construct a Beer-Lambert plot from the data. It is essential that you have a satisfactory calibration plot before proceeding further.*

(iii) Solubility Measurements

Using volumetric glassware, prepare a series of KCl solutions (100.0 cm^3 *in stoppered bottles) with similar concentrations to those shown in the table below, using* ***de-ionised*** *water (****the concentrations of the solutions you prepare must be known accurately****).*

$[KCl]/10^{-3}$ mol dm^{-3}	0	2	4	7	10	15	20	50

To each solution, add about 0.1 g of $Ba(IO_3)_2.H_2O$*. Warm each of the bottles to about 40°C, shake well and then allow them to equilibrate in a 25°C constant temperature bath for ~30 minutes, shaking periodically. Monitor the temperature of the bath to ensure it remains constant (~± 1°C).*

To estimate the IO_3^- content of each solution, take a 25.0 cm^3 aliquot using a pipette fitted with a short piece of PVC tubing containing a plug of cotton wool (to prevent extraction of un-dissolved solid) and place it in a 50.0 cm^3 volumetric flask. Add 1 cm^3 of HCl (2 mol dm^{-3}) and make the solution up to 50.0 cm^3 with 0.1 mol dm^{-3} KI. Mix well and measure the absorbance as before (remember to zero the colorimeter). Repeat each determination with a second 25.0 cm^3 aliquot.

In the problem-based script, detailed instructions are replaced with clearly stated objectives and some useful information (see Figure 2). The students have to decide how to do the experiment and draw up an experimental plan. In the problem-based practical, different groups of students used different methods in different ways and for different reasons; some used colorimetric methods, while others used titrimetric methods. Within the boundaries of available resources, safety and time limitations, students have control over the number, range and type of measurements they make. They have the opportunity to carry out preliminary trial experiments, encounter problems, 'go back to the drawing board' and review their preliminary efforts in the light of unforeseen practical difficulties. In short, they have the opportunity to be scientists.

Figure 2. Extract from problem-based laboratory script.

Solubility of Barium Iodate Experimental Section (Problem-based Practical)

Objectives

- *Determine the solubility product* (K_s^o) *for* $Ba(IO_3)_2.H_2O$.
- *Determine mean activity coefficients* ($\gamma_\pm$) *for* Ba^{2+} *and* IO_3^- *over a range of ionic strengths.*
- *Test the validity of the Debye-Hückel limiting law (DHLL).*

Useful Information and Equations

- *Iodate may be converted to iodine by iodide in acid solution.*
- *The solubility product,* K_s^o *of* $Ba(IO_3)_2.H_2O$ *is given by equation 2.*

$$K_s^o = a_{Ba^{2+}} a_{IO_3^-}^2 = [Ba^{2+}][IO_3^-]^2 \gamma_\pm^3$$

- *The solubility (s) is the number of moles of* $Ba(IO_3)_2$ *that dissolve per* dm^3 *of solution. Therefore, equation 2 may be re-written in terms of s (equation 3).*

$$K_s^o = 4s^3 \gamma_\pm^3$$

- *The Debye-Hückel limiting law and the expression for ionic strength are given below.*

$$\log_{10} \gamma_\pm = -A|z_+ z_-|\sqrt{I}$$

$$I = \frac{1}{2}\sum_i c_i z_i^2$$

Experimental

You will work in pairs. Prepare an experimental plan that outlines how you are going to perform the experiment and how you are going to analyse the data in order to extract the desired information. Bear in mind the availability of materials and equipment in the laboratory when planning your experimental approach. You must have your plan reviewed by a laboratory demonstrator before you start your experimental work.

- *Formulate plan.*
- *Discuss plan with demonstrator before proceeding*
- *Complete COSHH risk assessment*
- *Perform experiment*
- *Analyse results (individually).*

The Influence of Ionic Strength on the Rate Constant for the Reaction of Crystal Violet with Hydroxide ion:

This experiment is used in the second year physical chemistry practical course and also relates to the lecture material on ion-ion interactions and Debye-Hückel theory. The students commence the practical before they meet the material on ion-ion interactions in the lectures, but this is covered as they progress through the experiment. However, the first objective depends only on knowledge of first year kinetics. This experiment was introduced in problem-based style and had not been used previously at Keele.

Objectives

- Establish the rate law for the reaction
- Determine reaction rate constant over a range of ionic strengths
- Establish whether results support reaction mechanism by appropriate analysis
- Suggest a molecular mechanism for the reaction

The reaction between crystal violet and hydroxide ion is used widely in various guises as an undergraduate practical in many teaching laboratories. Under appropriate conditions, the reaction is accompanied by loss of the intense colour of the crystal violet and may be conveniently monitored by spectrophotometry. At

Keele the experiment is presented as a problem-based exercise in which the students, working in teams, have to establish the rate law for the reaction and study the influence of ionic strength on the reaction rate constant. They then have to propose a molecular mechanism for the reaction that is consistent with their experimental data.

A traditional prescriptive approach to this experiment would take decision making out of the hands of the students by, for example, detailing all concentrations to be used as well as the number and sequence of experimental runs. Assuming a colorimetric method, the traditional practical would likely prescribe a crystal violet concentration in the region of 10^{-5} M and specify a suitable wavelength to be used to monitor the reaction.

In the problem-based approach the students do not know how fast the reaction is; they need to try it out. It comes as quite a shock to students when enquiring about the whereabouts of the 'sodium hydroxide solution' to be told that they have to decide what concentration they require and then prepare it themselves. The students need to learn that crystal violet is intensely coloured and if the crystal violet concentration is too high it is possible for the reaction to be taking place without any apparent change in colour, as a number of Keele students have discovered. In the problem-based practical, students can find themselves in situations where, monitoring absorbance as a function of time, they obtain a sigmoidal curve because for the first ten minutes the absorbance reading is too high for the instrument to discriminate between the transmitted light levels. In such a situation the student can learn about instrumental limitations in absorbance readings and the consequences of the non-linear relationship between absorbance and transmittance at high absorbance values. It is unlikely that such situations and opportunities for learning will arise in a traditional expository style practical. The students also come to learn (rather than being told) that for practical reasons it is easier to determine the rate law by working under pseudo order conditions with hydroxide ion in excess. The students have to negotiate their way towards a suitable experimental approach in much the same way as researchers do.

Although all students end up using a colorimetric method in this practical, they adopt a variety of approaches in terms of, for example, the range of ionic strength used, the means of varying the ionic strength and temperature control. Indeed one particular group of students decided to work at a higher temperature (~40 °C) in order to increase the reaction rate. The change in temperature affects the dielectric properties of water and the value of the constant in the Debye-Hückel limiting law and with a little prompting these students were off calculating the value of 'A' at 40 °C, an outcome that is extremely unlikely in a traditional prescriptive practical.

It is interesting to note that during the first run of the crystal violet experiment some students immediately resorted to the Internet to find a procedure. I did not object to this because I made it clear that they would have to justify their method in any case and therefore they couldn't just follow it passively. These students became entrenched trying to reconcile various procedures from the Internet with the objectives of the experiment in front of them. They also learned quickly that they needed to be more critical of the material they were downloading rather than just accepting it as authoritative. So even in situations where students try to resort to a recipe and adopt a passive approach, the nature of the problem-based practical makes it difficult for them not to start thinking about what they are doing and why they are doing it. It is also interesting to note that the students who resorted to the Internet for a ready-made recipe made the slowest progress. Also, the demand on students to explain what they're doing and why they're doing it that way makes it perfectly possible to use the same experiment from year to year. Students may pick up some useful advice from the previous year's students, but the nature of the design of the practical class and the assessment methods makes this of only limited use. If it's not the student's 'own' approach, it will stand out in the laboratory discourse and in the assessment.

Assessment

In terms of reporting laboratory work for problem-based practical work at Keele, a variety of methods are used, including for example, team poster presentations with an element of peer assessment, individual laboratory reports and individual PowerPoint presentations. The principal difference in the student 'reports' based on problem-based work compared with 'reports' based on traditional practicals is that there is, by default, more variety in content and style, and because of this, instances of plagiarism are fewer than with reports on traditional practicals.

Assessment of the problem-based practicals is detailed and structured and is linked to achievement of objectives and explanation of the rationale for the experimental approach adopted in addition to other generic elements. In order to obtain a first class mark, the students need to demonstrate understanding of their specific experimental approach, data processing and the theoretical background to the experiment, rather

Figure 3. Assessment criteria for a problem-based practical

THE INFLUENCE OF IONIC STRENGTH ON THE SOLUBLITY OF BARIUM IODATE MONOHYDRATE
POWERPOINT® PRESENTATION ASSESSMENT CRITERIA

Criteria	**Mark**
Quality of slides (is there a title slide? are the slides clearly presented and structured? are they too busy or too thin? are results clearly presented and graphs and tables clearly labelled? is the experimental method concise and clear? are sources acknowledged and references cited?).	/15
Structure (is the material delivered in a logical and clear manner, including an introduction and conclusions?)	/15
Style (is the presenter audible and clear? is there eye contact with the audience? are key points emphasised?)	/15
Content (Results and Data Analysis) (what was the rationale for the experimental approach? were the objectives (see below) achieved? are statements, quantities and units accurate? is the quality of the data good or poor? are errors taken into account? are the conclusions drawn from the results justified? have errors been taken into consideration in arriving at the conclusions?) **Objectives**: ➢ Determine the solubility product (K_S^o) for $Ba(IO_3)_2.H_2O$. ➢ Determine mean activity coefficients ($\gamma_\pm$) for Ba^{2+} and IO_3^- over a range of ionic strengths. ➢ Test the validity of the Debye-Hückel limiting law (DHLL).	/40
Response to Questions (Does the presenter understand the specific details of his/her experimental approach, data acquisition and analysis? Does the presenter have an understanding of the meaning, significance and limitations of the results and of the background theory to the experiment? Can the presenter apply their general chemistry knowledge within the context of this experiment?)	/15
Total	/100

than, in the case of a traditional practical, successfully negotiating a prescribed algorithm in much the same way as every other student in the class. As an example, current assessment criteria for the barium iodate experiment are shown in Figure 3.

Student and Demonstrator Feedback

Written feedback was sought from students and demonstrators on their experiences of problem-based practical work and it is apparent from the feedback (written and oral) that students had never encountered this approach to practical work. A representative selection of comments from individual students and demonstrators is reproduced below. The first set of comments relates to the crystal violet experiment, which the students carried out first, and the second set relates to the barium iodate experiment.

Comments 1 (Crystal Violet).

- *I find this type of experiment much harder and more frustrating when things don't go to plan. However, it is a weaker part that could be improved if people did them more often.*
- *I found it quite hard to know what I was doing was actually right. Maybe you could have references to other similar experiments so that you could look at these and see how they did the experiment.*

- *The timing of the lecture material was out of step with the lab sessions, so the first week of the lab was spent being confused about what to do.*
- *Need more guidance – lots of contradictory advice.*
- *I do like the experiment but we need more background information as a lot of time is taken up from dry runs.*
- *More help needed with the design of experiment. A lot of different advice given which contradicted each other, therefore confusing. Designing an experiment did help understand what was going on a bit.*
- *This would be a good idea with more guidance given. A lot of help given was contradictory, which was very confusing.*
- *This technique of study is useful and more applicable to a work situation so it may be useful in the future even if it is not easy to get into this style of lab.*
- *Didn't really understand what the experiment was about until we finished it.*
- *The lecture material came too late – we don't really know what we're doing in our first few lab planning sessions. If we are on the wrong lines a hint should be given, as time is short. However, it is a refreshing approach. As it is group work, a group write up might have been a good idea.*
- *Demonstrators and lecturers need to be clearer in their explanations. There was too much 'see how it goes' and there was not enough time to take that approach. There should be more references, as recommending whole chapters of books can also be time wasting. There should be a starting point outlined, as when working in groups there is a lot of discussion and it takes time to start.*

Comments 2.

- *The second mini-project was much better than the first, as I had more idea as to what to do because we'd had more lectures so better understood the material, and had better guidance at the start of the project when it is most needed.*
- *This was the second mini-project, went better than the first, but I still find it tough, although it was stimulating.*
- *Try this out on the 1st years. More time was needed to work as a team to finalise plans.*
- *A little more time is needed to adjust to the new style of laboratory experiment, as we have never designed our own experiments before. Also, there seems to be a lot of deadlines in the last week or two of the module with assigned problems, oral presentations, lab reports and two workshops in for the last two weeks.*
- *Needed more time, and more guidance. More time is required to prepare.*
- *Please do not make us work in groups of four again – these groups are too large and it leads to too much faffing about and not enough work being done. More time was needed to complete these mini-projects as so much time was spent messing around making sure that the whole group understands what is going on.*

Postgraduate Demonstrator Comments

- *A good idea and good preparation for final year projects.*
- *Made students think and question why they were doing things.*
- *Harder for demonstrators because unpredictable and don't have a lab script to refer back to.*
- *Worried that I was misleading the students or giving the wrong advice.*
- *They need to know they should plan!*
- *Once they got into it they seemed to enjoy it and it makes them think.*

The feedback reveals that students find the problem-based style of practical work intellectually demanding, time-consuming and often frustrating. Also, it is clear that some students were confused, and therefore it could be argued that the problem-based approach is not an improvement on the traditional practical. However, this was the first time this style of practical had been used in chemistry at Keele, and it is evident from some of the comments that the students are not used to this style of practical. The problem-based style of practical has now been in use in several modules (principally level 2) for a number of years at Keele, alongside other more traditional practicals, and students, staff and demonstrators are now quite accustomed to them. It is apparent that the students became frustrated by contradictory advice and/or a perceived lack of advice and this is supported by a demonstrator's comment relating to anxiety about giving the 'wrong advice'. This highlights one of the main pre-requisites in managing this type of practical work: the tutors/demonstrators need to be very familiar with the material at a theoretical and practical level and they must proactively engage with the students to facilitate their learning. This style of practical work is certainly more demanding on student and staff demonstrators in the laboratory and consumes more laboratory time than that required for a traditional practical. Many of the comments above reflect the initial tendency of staff and demonstrators, in their first experience of this style of practical, to let the students find everything out for themselves, which coupled with instances of conflicting and contradictory advice did cause confusion and did not always support learning. However, with more experience in the supervision and management of problem-based practical work the teacher acquires a better understanding of what to expect from students according to their level and experience and learns above all to focus on student learning during laboratory discourse.

Conclusions

An account of a transition from expository style practical work to problem-based practicals has been described and discussed in the light of a practitioner's experiences and student feedback.

There are many challenges (for both students and teachers) associated with the use of problem-based practicals in laboratory teaching, but as well as being more demanding and frustrating they can also be more interesting, flexible and stimulating than the traditional style of laboratory practical, where inflexibility ensures that concept of the 'correct' answer and the 'correct' way of doing things prevails.

However, elimination of expository laboratory experiments from the undergraduate chemistry laboratory is not necessarily desirable, since such experiments fulfil different purposes. Indeed, there may be a synergistic effect in that students may learn more from individual types of experiments provided that they engage in a logically sequenced and balanced variety of laboratory work encompassing a range of experiment styles. For the student who has experienced other styles of laboratory work and has developed a capacity to think critically about experiment design, a traditional prescriptive script has the potential to become a different animal altogether; no longer a passive exercise but a further opportunity for critically evaluating how experiments are done. It is only a concern if the student adopts a passive approach; if a more critical approach is fostered then there is a place for this sort of practical as a learning tool.[6] As Carnduff and Reid[14] argue, '*to change the experience, you don't need to change the experiment, just what you do with it*'.

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