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Unfinished recipes: structuring upper division laboratory work to scaffold experimental design skills

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Abstract
Experimental design is a desirable outcome of laboratory education. Incorporating inquiry into the laboratory curriculum is attractive, but there are acknowledged concerns from practical, theoretical, and epistemological perspectives, and these are accentuated in upper-division courses. In this work, we draw on the extensive literature relating to experimental design and inquiry learning to conceive a pragmatic laboratory curriculum that invokes the development of experimental design skills in a structured way. The model also incorporates the core principles of formative assessment, so that students get a chance to improve their work based on feedback as they are doing it. We illustrate this model with two examples from our own practice of upper division physical chemistry, but the basis of the design is elaborated so that interested readers can adopt it for any aspect of practical chemistry where there is a desire to incorporate experimental design skills.

Graphical Abstract

Keywords
Audience: Upper-Division Undergraduate

Domain: Laboratory instruction, physical chemistry

Pedagogy: Inquiry-Based / Discovery Learning

Topic: Learning Theories
Introduction
Teaching students about the scientific method is among one of the variety of reasons for including practical work in the chemistry curriculum. Writing in this Journal in 1935, Carmody listed four objectives of practical work, including the opportunity to “develop training in the logical or "scientific" method of experimentation and thinking” and to “develop facility with and appreciation of or feeling for laboratory experimentation”. This interest in practical work as a means to teach the scientific method was reaffirmed in the post-Sputnik era. By the mid-1970s, a major US conference on laboratory education heard that there were generally two aims regarding laboratory work: those regarding the nature of the scientific process generally, as well as those relating to specific laboratory skills, and application of these skills to unknown processes. Curricular reform in the latter part of the twentieth century began to emphasize the role of incorporating what became known as inquiry into laboratory activities.

What is inquiry?
In his work on categorizing the types of laboratory instruction that can be used, Domin described four different approaches, depending on whether the outcome was predetermined or not, whether the approach taken by students was deductive or inductive, and whether the procedure was provided to the students, or devised by the students. The expository or recipe approach requires students to complete experimental work to arrive at a defined outcome, and to deduce some findings based on the general knowledge framework in which the experiment is set. The discovery approach is similar, but instead requires students to induce some meaning from their data by noticing trends or conflicts, and conceive an overarching statement that explains their results. The problem-based and inquiry approaches, both require students to develop their own procedure; Domin distinguishes between them by stating that problem-based approach requires deduction (students are working within a defined framework), whereas inquiry requires induction.

Inquiry approaches have become very popular, but there is often a conflict between what is published as inquiry approaches and this characterization. A helpful addition to this discussion is work by Bretz and co-workers, characterizing different levels of inquiry. Arguing that “there exist shades of inquiry with varying degrees of freedom in the student experience”, these authors propose a rubric to characterize four levels of inquiry, depending on whether students are provided with the problem or question to be addressed in the laboratory, whether the procedure is provided, and whether
the solution (or goal) is provided to the student. Level 0 in their rubric is equivalent to the characterization of expository labs, and the jump from Level 1 to Level 2 relates to requiring students to develop their own procedure.

Talanquer has used the above rubric to devise a useful descriptive rubric indicating how these levels of inquiry might work in practice. This outlines what is provided to students and what is expected of them. Central to our discussion here are two things. First is that the change in the level of background information provided to students reduces from being provided beforehand (Level 0), to being provided in the laboratory session (Level 1), to being provided in the laboratory session as needed (Level 2). Second is the change in how procedural information is provided to students, changing from being provided with detailed steps (Level 0), to being provided with an outline (Level 1), to requiring students to conceive of a procedure, with guidance (Level 2).

From this work on defining and characterizing laboratory approaches, several things emerge. Laboratory activities will differ depending on what information students are provided with, and what we subsequently require of students in their experimental work. Students may be provided with procedural details or not, or somewhere middle-point whereby general guidance as to the overall approach to pursue is given. Students may be given background information or not, or as is needed. Indeed, students may be given an overarching framework, or not, within which to base their work. Clearly these decisions will have major consequences for the nature of learning in the laboratory, and a significant challenge for those designing laboratory activities is making these decisions, and having good reason to make them.

Epistemological challenges

In addition to the challenges faced in considering the rubrics and laboratory instruction styles detailed above, there are overarching issues regarding the epistemology of laboratory education. In his essay, Kirschner defines two types of knowledge structure in science: the substantive structure of science – the body of knowledge making up science – and the syntactical structure of science – the habits and skills of those who practice science. In aiming to teach students about the syntactical structure of science, Kirschner argues that educators mistake teaching the nature of the scientific process (teaching how) with immersion of students in these processes (teaching by). The latter intends for students to assimilate an understanding of these inquiry processes by completing them as a scientist would. He argues that this is flawed, as students who have not been taught explicitly about how to conduct a particular process cannot learn about that process by simply acting it out. Citing Ausubel, he writes: “if a student is ever to discover [scientifically] then she must first learn. She ‘cannot learn adequately by pretending [to be] a junior scientist’.”
This argument has been well versed. Woolnough and Allsop wrote that in teaching practical science, the emphasis should be on teaching the way a problem-solving scientist works.\textsuperscript{10} Anderson distinguishes between “science” and “sciencing”, the latter being the processes scientists conduct, and states that students need to be taught these processes. Assuming they will assimilate them by inductive processes has little basis.\textsuperscript{11}

Cognitive demands in the laboratory
As well as epistemological issues, the design of laboratory work is also impacted by the literature on typical issues observed with laboratory education in practice. It is over 35 years since Johnstone and Wham wrote about the working memory demands of the laboratory, arguing that students in a typical laboratory session are overwhelmed by a variety of information that they meet in the laboratory, such as text and verbal instructions, details about instrumentation, underlying theory, etc.\textsuperscript{12} Surveys of students completing laboratory work in lower\textsuperscript{13} and upper\textsuperscript{14} division chemistry laboratories suggest that Johnstone’s observations have not changed much in the intervening time: one of students’ primary purposes is to finish the laboratory work in a timely fashion. There is often little intellectual engagement in the laboratory class itself, with intellectual effort focused on the report, post hoc. A typical approach to address intellectual engagement in the laboratory is to require students to complete pre-laboratory work, which aims to ameliorate the load in the laboratory itself. There are numerous examples of pre-laboratory work in various contexts\textsuperscript{15} with some notable examples of explicit consideration of cognitive load.\textsuperscript{16}

Cole and co-workers have written about the especial problems presented with upper division practical work, especially in physical and analytical chemistry courses, and demonstrated the value of pre-laboratory activities in addressing some of these difficulties.\textsuperscript{17} Such laboratory classes impose difficulties in terms of organization – as limited equipment means students “rotate” around different experiments as labs progress – and cognitive load – as students may not have received the lectures covering the theory associated with the practical work.

An alternative approach
As Cole highlights, upper division practical work generally relies on a substantial theoretical base, and uses experimental procedures often unfamiliar to students. Our initial work involved the preparation of pre-laboratory resources to support upper division experiments, guided by the literature on the use of pre-laboratory resources in reducing cognitive load.\textsuperscript{15} Cole and co-workers demonstrated that pre-practical activities have some impact in addressing this load.\textsuperscript{17} Before our redesign described below, our approach was to include pre-practical activities with our typical laboratory experiments that
students would conduct over four of their 6 weeks in the laboratory. The purpose of these were to introduce students to advanced techniques, data analysis, and reporting requirements of the upper division laboratory. For the final two weeks, students completed an “investigation” – an inquiry activity that required them to design and implement a mini-investigation. The overall curriculum design therefore intended to incorporate some expository laboratory work, guided by the principles of cognitive load, so that students could learn about practical approaches, and some inquiry work, to give students experience of experimental design.

Our experience with this approach was that while students found pre-laboratory information useful for the expository laboratories used in the first four weeks, these laboratories did not adequately prepare them for the experimental design aspects of the mini-investigation in the last two weeks. Feedback from students indicated that these latter activities were unmanageable, stressful, and very difficult for them to carry out. The jump from expository to inquiry was too great.

As well as the lack of preparation for experimental design, this approach was problematic because of the pre-laboratory information being provided to students. In physical chemistry particularly, laboratory work is often delivered before students have the associated lectures. Ameliorating this by using pre-laboratory activities means that the information provided shifts away from experimental considerations and is dominated by explanations of underlying theory. In the language of Kirschner, the focus is on the substantive knowledge rather than the syntactical knowledge. Because students only had one week per experiment, there was a pressure to assimilate whatever relevant theory that was necessary to write a report, at the expense of considering the experimental approach and experimental design. We felt that the pre-laboratory information needed to be more extensive if we wished students to more meaningfully engage in experimental design, and opted to move to a design where laboratory work would itself prepare students for inquiry, so that preparation extended into practical time.

Revised model design

In practice this approach manifested as follows: each “experiment” students complete incorporates an initial expository approach (Part 1) so as to introduce students to the concepts, methods, and analysis protocols. This activity built on pre-laboratory activities, but itself would be preparation an inquiry component (Part 2), where students build on their work in the first part, designing additional related experiments, grounded in the chemistry and the methods introduced in Part 1. This approach echoes the suggestion made by Kirschner at the end of his essay for a middle-ground between expository and inquiry, involving an initial standard approach for all students, but which can then go in a variety of possible
directions depending on what the students pursue in a more open-ended phase. This model was implemented with new experiments completed on rotation as before.

Complex Learning Environment

This design is also informed by our previous consideration of pre-laboratory activities, and in particular the consideration of laboratory learning environment as a complex learning environment. We advocate this description of laboratory learning because of the definition of complex learning environment is one where knowledge, skills, and attitudes need to be integrated, where a variety of knowledge and skills need to be coordinated, and that the learning scenarios requires the application of this in practice. The conception means that we can learn from the educational psychology literature how best to prepare learners for such an environment, and a particular consideration is of relevance here: that in supporting complex tasks, we should present learners with examples of the whole task, and sequence their approach in a simple to complex fashion. Therefore the primary objective behind our “Part 1” and “Part 2” approach is to allow learners introduce the many components to the complex task – the experimental details, the nature of data acquired, the related theory, and the approach to analysis – with guidance in the form of expository instructions. This means that students are led through the entire iteration of a task so as to become familiar with the various aspects of it.

Incorporating formative feedback

A further consideration of curriculum delivery is the incorporation of formative assessment. We are heavily influenced by the work of Sadler and have described previously how laboratory environments provide especial opportunity to meet his criterion of using feedback to give students an opportunity to improve their work as they are completing that work.

Previously in our curriculum, we assessed students on their performance in the laboratory as evidenced by their lab notebook, and their laboratory report. Students received extensive feedback on their report, but we found the commonly reported observation that students did not transfer this feedback well on to subsequent reports. In the revised model, we built in four feedback points during the course of laboratory work. The first was when students arrived into the laboratory. While it was not assessed, students were required to watch preparatory videos as pre-lab activities prior to coming to the laboratory. When students arrived in the laboratory, and prior to beginning experimental work, demonstrators (teaching assistants) asked students a series of questions relating to the experiment. The complex learning framework distinguishes between supportive information and procedural information, the former being information relating to the underpinning
theory and basis for experimental approach, the latter being related to stepwise instructions and guidance necessary to complete a task at hand. The framework advocates presenting supportive information in advance, and focusing on procedural information during the completion of the task. As the focus at this stage was on the supportive information – checking students’ understanding of the underlying principles and concepts, the rationale for experimental approach, etc – demonstrators asked students a few questions relating to these topics. This was a useful chance for students to check their understanding of the experiment and the experimental approach before getting on with the specific stepwise instructions.

Students were required to maintain a laboratory notebook, and this was signed off after the first two sessions. At this stage, the students would have piloted some experiments in beginning the second part of their experiment, and demonstrators used this time as a second feedback point to address any difficulties that arose.

A third feedback point was built into the start of the second week. Students were required to complete the analysis required for Part 1 of the experiment – and show their work to demonstrators. This was not as a report, but could be, for example, showing demonstrators the analysis conducted on graphing software. This provided a useful chance for demonstrators to feedback on the analysis completed to date, and help students identify any areas for improvement in their draft work. Finally, at the end of the second week, demonstrators would again sign off the laboratory book, and deal with any final queries from students. These four feedback points were aimed to ensure that students felt they could intellectually engage with the material as the laboratory work progressed, rather than leaving it until afterwards, an approach that is common in laboratory education. Once students completed the experiment, they submitted one report, covering both parts of the experiment. This report followed a journal article format, with students writing an abstract, introduction, procedure, results and discussion. The report was worth 85% of the grade, with in lab work (answering questions, lab book recording, Part 1 analysis completed for interim feedback) worth the remaining 15%. The overall process is shown in Figure 1.
Examples of this approach

The approach described here were incorporated into the third year of a five-year Master of Chemistry (MChem) course at a research intensive university in the UK, with a class size of 120 students. While five year MChem courses are the norm in Scotland, in England and Wales, MChem degrees are typically four years long. The third year of a Scottish course is approximately similar to the second year in an English university. This point in the curriculum is at the end of the formal laboratory education students complete, prior to independent research and project work carried out in Years 4 and 5. In third year physical chemistry, students attend for 6 hours a week with an assumption that they will spend 6 hours a week on processing and analysis in preparation for their report. (In contrast, students spend 12 hours per week in organic and inorganic chemistry labs as there is less outside work required for these experiments). Prior to our curriculum change, student completed 4 experiments; one per week over four weeks followed by a two-week “investigation”.

In order to incorporate the revised model into the existing timetable arrangement, students were assigned three experiments to complete over six weeks, so that one experiment took two weeks (four 3-hour laboratory sessions). The

### Figure 1: Components of a single laboratory assignment undertaken by students over a two week period (demonstrator is the term commonly used for graduate teaching assistants in the UK).

| Week 1 | Pre-Lab Video | Session 1 | Session 2 |
|--------|---------------|-----------|-----------|
| Video describing principal features of experimental approach | Demonstrator discusses experiment with student, with questions based on prelab. | Students devise a hypothesis for analogous chemical system to be investigated by similar technique |
| Video outlining underpinning core chemistry concepts for the experiment | Students follow procedures provided. Aim is to familiarise with experimental technique & underlying chemistry. | Students plan out experimental protocol based on information provided and learning from Session 1 |
| Laboratory notebook signed off by demonstrator after Week 1 | Students acquire data as directed | Students begin some initial test runs to test out their approach |

| Work between Week 1 & 2 | Sessions 3 & 4 |
|-------------------------|---------------|
| Students begin initial analysis of data to show at the start of week 2 | Demonstrator discusses progress and results from Week 1 |
| Students continue planned experiments reviewing approaches; gathering data | Laboratory notebook signed off by demonstrator after Week 2 |
| Students prepare a laboratory report | One report covering part 1 and part 2 submitted one week after lab. Students receive grade (including 15% lab mark) and feedback |
experiments were arranged so that Part 1 (expository) took about 3 hours, and Part 2 (inquiry) took up to 9 hours. For pragmatic reasons, we opted to build on our current suite of expository experiments and refashion those into Part 1, and building on an appropriate experiment that students completed as Part 2. This model meant that students completed their work over two weeks and this allowed time for students to begin some analysis and initial reporting work into their second week labs, where they could discuss with demonstrators. This offered a valuable opportunity for formative feedback, and ensured students began their analysis and processing work after the first week of the experiment. Students complete both parts in pairs, although their laboratory notebook and laboratory report is assessed individually.

We illustrate how we have incorporated this model into our curriculum with two examples from our laboratory rotation. The intention here is to show how existing “traditional” physical chemistry experiments can be modified to incorporate inquiry in a supported and structured way. After describing the experiments, we highlight some observations from implementation.

Our original curriculum included two well-known experiments: flash photolysis of azobenzenes and the kinetics of the hydrolysis of malachite green (analogous to the crystal violet experiment). In our original rotation, students would previously complete the necessary experimental work in pairs, in three hours. We decided to append these experiments (now labelled Part 1) with a Part 2, again completed in pairs, giving students the chance to study the chemistry of these systems in more detail, as well as be exposed to inquiry at an advanced stage in their laboratory programme.

Flash Photolysis

Our original flash photolysis experiment was based on the cis–trans isomerization of azobenzenes, similar to a version published in this journal, and this experiment became “Part 1” of the experiment in our new arrangement. This involved following a given procedure about completing the experiment. Briefly, students acquire an absorption spectrum of the azobenzene, and then monitor the absorbance at the wavelength of maximum absorption over time immediately after exposing a solution in cuvette to a camera flash. As described in the original article, the azobenzene can undergo photoisomerisation to the cis form, which thermally reverts to the trans form over 10s of seconds, and thus is easily monitored using this approach. Students explore the temperature dependence in one solvent (THF). They subsequently study the dependence of isomerization rate on solvent at one temperature, by studying isomerization rates in cyclohexane and acetone. The experiment is robust, works well as described in the original paper, and is easily completed by a pair of students working together within three hours.
At this point, students are required to start Part 2. We wished for students to have the opportunity to study azobenzenes further, building on their experience and familiarity with the experimental technique. A recent paper reported on the pH dependence on azobenzenes and this was used as a basis for the Part 2 experiments. Unlike Part 1, students were not given instructions. They were instructed to investigate the pH dependence of a provided azobenzene. Based on their analysis and interpretation in Part 1, they had suggest a hypothesis for what the effect of changing pH will be on the cis-trans restoration kinetics, and devise experiments to explore this. Students were given the link to the paper underpinning the experiment, which, with some work, would assist them in devising a protocol for their work. Students were also prompted to choose to acquire other data about this system – that could be temperature dependence of this new system or seeking a transient absorption (rather than relying on the bleaching peak). Part 2 usually took two to three lab sessions; one in the first week (typically planning pH solution concentrations, testing out approaches) and up to two in the second week (typically working through the pH ranges, and addressing the follow up prompt about temperature dependence or seeking transient absorption).

Hydrolysis kinetics
Malachite green undergoes hydrolysis in the presence of hydroxide, and our experiment mimicked those of published examples for crystal violet, whereby students would explore the rate dependence of malachite green as a function of hydroxide concentration to determine the pseudo-first and then the second order rate constant of reaction using UV/visible spectroscopy. Students completed this work in three hours, working in pairs.

In the revised arrangement, this experiment became Part 1. Students have previously completed an experiment to determine the critical micelle concentration of sodium dodecylsulfate using conductivity (similar to that described in this Journal) in their previous year. Micelles are known to influence reaction kinetics by either separating or containing together reactants, and indeed kinetics of reactions in the presence of micelles have been reported in this Journal. However in this case, students were directed to a recent research study exploring hydrolysis kinetics in the absence and presence of micelles, and were required to extract information from that paper to design an experiment exploring the rate of reaction in the presence and absence of micelles. Typically, this required students to determine the critical micelle concentration and study the reaction in the presence surfactant molecules at low and high concentrations, so that they were below and above the critical micelle concentration. Students who wished to further probe the system were encouraged to study the reaction at a range of surfactant concentrations or explore the system in the presence of different
concentrations of sodium chloride, or explore different temperatures. Part 2 usually took two to three lab sessions; one in the first week (determining the critical micelle concentration, planning out kinetic protocols, dilution calculations) and up to two in the second week (compiling high and low surfactant concentration data, and addressing the follow up prompt).

Discussion
The redesign of our approach to physical chemistry laboratories was driven by twin desires of wanting to improve students’ experimental design skills prior to independent work in their final years and in response to dissatisfaction of students about their time in physical chemistry laboratories. With respect to the latter, there were previous concerns about the perceived difficulty of laboratories, the lack of cohesion with the lecture syllabus, and the perceived lack of valuable feedback. In order to address these issues, and the desire to develop students’ experimental design skills, we sought to use a literature informed approach to build a new laboratory curriculum. This curriculum is grounded in the frameworks of cognitive load and complex learning, as well as considering the epistemological nature of laboratory learning.

The core features of the redesign was the incorporation of pre-laboratory activities that aligned with the principle of simple-to-complex sequencing described in the complex learning environment framework. This meant that students first became familiar with the concepts, approaches, and types of data their experiment generated, before embarking on a second part where they had to use their knowledge as an experiment design. These were coupled with extensive formative feedback involving demonstrators (teaching assistants) throughout the laboratory sessions.

The approach meant that our students completed fewer laboratory reports than the previous model, but these reports were more substantial in nature as they incorporated results from Part 1 and Part 2 of the experiment. Students received feedback on each report before submitting their next one.

The laboratory classes are in general supervised by academic staff who monitor the laboratory work for health and safety purposes, and manage the team of demonstrators (teaching assistants) present in the laboratory. These academic staff responded positively in feedback regarding these practical classes. In correspondence requesting feedback, staff members’ comments included: “students arrive well organized and are planning their work well – and they are enjoying it!” and that “students appear focused, know what they have to do and are able to work hard to achieve it”. The model used means that students write fewer reports, and a staff member was concerned that they were having to do less work and receive less feedback. This concern is acknowledged, but our emphasis was to improve the quality of feedback on a smaller number of reports. Technical staff also noted improvements, commenting that the new model was much more
manageable, as students previously requested a range of chemicals for investigations that could not be easily nor quickly sourced, whereas the new model allowed for "everything to be planned in advance".

Demonstrators – PhD students who spend some time in the teaching laboratory – are crucial to any teaching laboratory, but especially so in this model. A core aspect of their training was to encourage dialogue with students regarding discussing experimental work at the various feedback points, and elaborate on the kinds of support necessary to help students approach the second part of their experiment. To assist with the first part, a list of common questions for each experiment was devised, designed to build on pre-laboratory preparation and help student focus on the lab. These tended to focus on overall rationale for the approach being used, particular considerations for the experiment, expectations for outputs etc. The purpose was not to directly quiz students on their knowledge, but rather initiate a dialogue about the experiment to help students focus on the task at hand, encourage ongoing dialogue in the class as work proceeded, and ensure the importance of preparation was built into the laboratory class. Demonstrators reported that students tended to come to the practicals well-prepared, completed the interim work, and while experimental design was still challenging, the approach was seen as achievable. An example comment from a demonstrator with significant experience was "students know a lot more about what they are doing and why they are doing it". Because marks were allocated for the laboratory work (15%, encompassing laboratory performance, presenting of Part 1 data), students were generally prepared and ready for Part 1 on arrival in the laboratory. There was still some difficulties observed in beginning Part 2. This was somewhat anticipated, as it is the first time students did not have direct instructions. Therefore even though students were directed to articles providing overview information, in a significant number of cases, students had not sourced the supplementary information of the article giving some more explicit instructions, or found it difficult to translate the research article provided into a procedure they could use in the early week. Perhaps as a consequence of this, we saw little evidence of students pursuing investigative work beyond the remit of the problem assigned. This was due in part both to the guided structure of the Part 2 activity, but also because where students did consider some additional work sourced from additional reading, there was not appropriate equipment or materials to pursue that activity. In general terms then, where additional work was observed, it was typically in the data analysis completed by students, rather than experimental work itself. As the laboratory progressed over the second and third experiment, this issue dropped off substantially and students grew noticeably more confident in planning out their work.
Students was asked about their perception of physical laboratory classes in general at course liaison groupings and other similar student feedback fora. In general, where there had previously been vocal complaints about the difficulty of laboratory classes or the lack of feedback, after this iteration there was little complaint, and laboratory practical work was considered “enjoyable”. In an audit of assessment and feedback generally for students on the associated courses, students feeding back reported that the laboratory classes were much better structured and had much greater levels of feedback.

Interestingly, a discussion forum set up to help students with preparing for laboratory work, analyzing their data and preparing reports had substantially fewer posts in the year the new model was rolled out, compared to the previous year of iteration. One possible reason is that students did not need to query as much as the level of dialogue and feedback in the laboratory classes increased. However, we are examining the student perception of labs and the impact on their experimental design in much more detail in a separate study.

On the whole, we considered the revised model to be successful. The challenge in redesign and implementation of a new laboratory curriculum is the enormous up-front work necessary to devise new experimental protocols, and the consequent effect that has on laboratory support materials, technical requirements, and demonstrator training. Our approach of building onto existing experiments worked well, and reduced the workload associated with the new curriculum significantly.

**Conclusions**
We report a model for a revised laboratory curriculum which aims to structure students’ approach to experimental design and inquiry in advanced level laboratories. The approach is grounded in cognitive science and considerate of epistemological arguments regarding laboratory education. While our model described here aligns to a particular format, the core principles are highlighted so that it may be adopted by others in accordance with their own local conditions.

**Associated content**
Supporting Information
Laboratory manual protocols provided to students are provided.

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