Introducing Elements of Inquiry and Experimental Design in the First Year of an Undergraduate Laboratory Program

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ABSTRACT: This work further implements the “elements of inquiry” approach in a first-year undergraduate laboratory course, in a two-part inquiry sequence. In the first part of the sequence, students iteratively develop a “best-practice” TLC procedure. In the second part, students iteratively optimize a simple organic reaction using TLC, making experimental design choices within a controlled environment. The work has been well-received by students and instructors, as a reflection of real-world research challenges introduced at a first-year level.

KEYWORDS: Inquiry-Based/Discovery Learning, Esters, Thin-Layer Chromatography, First-Year Undergraduate/General, Organic Chemistry

INTRODUCTION

Inquiry learning is an established and widely used alternative to didactic (or expository) teaching and has been very successfully adopted in chemistry, and specifically in the chemistry laboratory laboratory over the past several decades. An inquiry lab program usually uses increasing levels of inquiry over multiple years, building from lower levels of structured and guided inquiry all the way to the research-like experiences of authentic inquiry. In particular, Seery’s 5-stage curriculum model of experimental design neatly encapsulates this escalation of skill and independence in a framework that can be applied beyond inquiry-based laboratories. Within the field, guided inquiry has emerged as a useful intermediate on this journey and has been well-utilized for addressing shortfalls in lab-specific concepts in UK higher education chemistry.

However, in an inquiry lab program, all or part of the first year is usually devoted to expository or confirmatory laboratories, designed to teach a foundation of techniques and skills before progressing onward. Although some exceptions do exist, they tend to span large, multiweek campaigns and operate at a guided-inquiry level. We are specifically interested in smaller, drop-in elements of inquiry that can be used within single-session laboratories. There is a growing body of work in this area, stemming from the influential work of Szalay and Toth in modifying single-session step-by-step school practicals. The practicals thus modified must have certain features, such as a quick core loop that is amenable to experimentation, and here we extend prior work applying these principles to modify university level lab courses to contain “elements of inquiry.”

OUR CONTEXT AND DESIGN RATIONALE

All first-year chemistry students at Strathclyde undertake a mandatory laboratory module in year 1, covering all areas of practical chemistry across both semesters of a two-semester year. Students arrive with one of at least six distinct qualifications, each with a different prior level of practical work, and so the core purpose of the lab is to provide students with a solid, consistent foundation of skills. Due to timetabling constraints, the lab is split into 3 h sessions. Each session is preceded by a prelab quiz (supported by videos and simulations) which carries a small assessment weighting. Prelab material is usually supportive in nature, with procedural information provided in the lab only when directly needed.

Practical sessions are delivered on a rota due to equipment restrictions, meaning that session content can depend only weakly on contemporary lecture content. A common scenario, this tends to weight investigation toward inductive rather than deductive reasoning, reducing the reliance on students having a comfortable grip of level-appropriate theory. Practical sessions are therefore framed in a way that steers students toward inductive reasoning.

The lab module contains some practicals which have previously been modified to include elements of inquiry-based learning. The modifications were introduced as part of a normal cycle of renewal and implemented and evaluated by...
undergraduate students working as research partners. This approach has proven to be very successful and is now used whenever a practical is replaced or updated.

Taking all of this into account, we sought to identify new areas where existing practicals could be modified to include elements of inquiry, or replaced entirely, with the added aim of introducing the concept of iterative experimental design.

Identifying Suitable Areas for Modification

Our aim was to introduce iterative experimentation to students, without exceeding limits on time, space, or consumables. Any new work must also not depend strongly on theory beyond the scope of corresponding lecture courses or introduce significant time-consuming dead-ends that could lead to frustration and cognitive overload.16,17 Two suitable experimental areas were identified: thin-layer chromatography (TLC) and reaction screening.

TLC is a ubiquitous research tool for synthetic organic chemists and also serves to teach many useful analytical chemistry concepts. The average organic synthesis researcher will run several TLCs with several different conditions to analyze a single reaction, and in doing so gain an innate understanding of the relationship between polarity and sample retention. The iterative loop for TLC is quick: from preparation to visualization in a few minutes.

Reaction screening is another common synthetic chemistry approach, where an array of small reactions are set up, with some variation of conditions leading to a reported table of results. Small-scale reactions may not be fully worked up but chromatographically or spectroscopically screened for success. The iterative loop here depends on reaction time and the analysis method, but again with scope for rapid turnaround depending on the reaction studied.

SEMESTER 1: INTRODUCING TLC

Using the “elements of inquiry” approach, an existing expository TLC practical was converted to include inquiry elements.13 This particular practical was the first time TLC is introduced, using a sequence where students first practice setting up a TLC using visible dyes and then analyze a mixture of UV-active unknowns. Two specific areas were modified with inquiry elements: plate setup and solvent choice.

Modification 1: TLC Plate Setup

In the existing expository sequence, students were given a full step-by-step procedure to set up and run a TLC plate, using a premade solvent mixture of a single fixed polarity. In the inquiry modification, students are asked to investigate the impact of four variables in a group. They are provided with a bare-bones expository procedure, but with four choices clearly highlighted:

- Solvent polarity: high or low polarity
- TLC chamber lid: present or absent
- Spotting intensity: single or multiple spotting
- Spot position: above or below the solvent line

Testing of each condition is distributed between a small team of students, and result pooling is used to collaboratively write a canonical TLC procedure to take forward (almost invariably identical to standard best-practices). By using result pooling, this sequence takes the same length of time as the previous expository sequence, but students proceed with an improved grasp of the basics of TLC.16

Students will still make traditional “mistakes”: for example, spotting on the reverse of a TLC plate, or pressing too hard and damaging the stationary phase. These errors would always happen, but the inquiry framework gives students permission to openly discuss failure and meaningfully learn from it. These errors do not occur often enough to be added to the list of variables to be explored, nor are they strongly cautioned against; those who do stumble into these errors can learn from them more easily here than in an expository framework.

Some TLC errors can be caused by factors outwith the immediate control of the students, and somewhat more difficult for a novice user to spontaneously recognize; to avoid these situations causing confusion, students were provided with an additional support sheet describing common TLC errors and how to avoid them (see Supporting Information for details).

Modification 2: Solvent Choice

Once a reliable TLC procedure is in hand, students use it to analyze a mixture of two colorless, UV-active unknowns of differing Rf values. The expository version of this procedure assigned each student a sample containing a pair of unknowns and provided a set of reference standards and a preprepared solvent mixture with the goal of identifying the contents of the mixture and calculating Rf values.

The inquiry modification used here is to introduce iterative optimization of solvent polarity. Students are given access to cyclohexane and ethyl acetate and advised to test their assigned mixture against 2-3 different compositions, with the aim of maximizing separation. Then, students run a final analysis against reference standards, using their own optimized solvent conditions. The goal of maximizing the separation of two spots invariably drives students away from very high or very low polarities, usually settling somewhere in the range of 10-50% v/v ethyl acetate/cyclohexane. Students are then asked to use their optimized solvent conditions to screen against reference standards and make an identification. Each student is assigned a separate mixture, but continued peer working is encouraged.

Compounds used as unknowns were selected on the basis of prior availability and were provided as premade standards of appropriate intensity (see Supporting Information for details). Almost any four compounds would be suitable as long as they are nontoxic, are UV-active, run well, and have a range of polarities with differentiable Rf values. In particular, two of the compounds run relatively close together (unknowns B and D, Figure 1), and these could be replaced with “easier” unknowns if desired.

Figure 1. TLC plates of an unknown (B + D) at four different possible polarities, collected by an experienced chemist during the process of COVID-19 virtualization. Students themselves spot across two plates, unknown + AB followed by unknown + CD.
Assessment is via a paper worksheet, which students complete and submit on the day of the lab itself (see Supporting Information for worksheet).

**Summary of TLC Sequence**

By the measure of existing inquiry learning definitions, this sequence sits at the Bretz and Towns inquiry level 1.5, since most of the procedure is given.\(^5\)\(^3\) However, since students converge on a known optimal procedure with minor variation, the “known outcome” does not fit well with any level of inquiry and could even be considered expository in nature (inquiry level 0). However, this sequence fits the “elements of inquiry” principle very well, as it gives students the sense of experimental control and prepares them for a subsequent inquiry lab.

During the COVID-19 pandemic, this practical ran remotely. The only modification needed was a bank of photographic images covering all possible permutations of conditions for the plate setup section, and a selection of polarities for solvent choice. Photographs were then available for students to download, alongside the standard lab manual procedure. Overall, less than 100 TLC plates were photographed, and the online conversion took less than a day (Figure 1 is a compilation of some permutations thus gathered).

**SEMESTER 2: ORGANIC REACTION OPTIMIZATION**

In the second-semester curriculum, an existing sequence was no longer fit for purpose and needed to be entirely replaced with another organic synthesis practical. With the aims of introducing elements of inquiry, experimental design, and reinforcement of the earlier TLC sequence, the following constraints were placed on the new practical.

The reaction to be studied should ideally
- Relate to accompanying theoretical content
- Have starting materials, products, or side products distinguishable by TLC
- Go from setup to outcome within 30 min, allowing iteration
- Use common, reasonably bench-safe reagents and conditions
- Proceed at room temperature, under a wide range of closely related conditions
- Yield a product that could be analyzed in detail in a subsequent practical

Some reactions met most of these criteria: For example, aldol condensations or some aromatic substitutions can be rapid and bench-safe but are not taught theoretically until the following year. After some initial literature searching, ester hydrolysis was identified as a promising area.

Ester hydrolysis fits well with the existing laboratory structure: Students handle carboxylic acids in five other practical sessions, including a titrimetric pK\(_a\) determination. To forge better links between organic and physical chemistry, the ester hydrolysis products were used in the subsequent pK\(_a\) determination practical itself.

Ester hydrolysis is traditionally considered to require elevated temperatures, aqueous base, and extended reaction times. However, a procedure was identified which gave good yields of carboxylic acids in as little as 10 min.\(^3\) After some initial proof of concept screening (Tables S1 and S2, Supporting Information), several substrates were selected.

### Identifying Suitable Substrates

Suitable substrates for this reaction have several requirements. They must
- Quickly and cleanly hydrolyze under most student-explored conditions
- Be distinguishable from their hydrolysis product by TLC
- Generate trivial-to-remove alcohols on hydrolysis
- Be commercially available for less than £1/gram

One class of compound that satisfies all of these requirements is methyl esters of electron-poor arylcarboxylic acids. Once converted into arylcarboxylic acids, there is a further requirement imposed by the pK\(_a\) determination section:
- Moderate aqueous solubility of free acid (>1 g/L at 20 °C)

In initial tests, liquid esters were found to be unreliable, potentially due to stirring effects. Given also the operational and safety challenges of students encountering weighable liquids for the first time, an additional parameter was screened:
- Ester must be a solid at room temperature

Many methyl esters were screened (Table S3, Supporting Information), and three were suitable. The ubiquitous presence of electron-withdrawing groups is not surprising, given that any plausible mechanism would be accelerated by an electron-deficient carbonyl. An additional advantage of the chosen compounds (Figure 2) is that students already have experience with isolating arylcarboxylic acids by precipitation, allowing a familiar bench-safe workup procedure.

![Figure 2. Chosen esters and the screening parameters (other than reaction completion) that were used to select them.](https://doi.org/10.1021/acs.jchemed.2c00311)

**Teaching Activity Design**

In designing the activity, reaction optimization was held as a central principle, so students are prebriefed with a flowchart (Figure 3) and given plenty of written and verbal encouragement to exercise reasoned choices.

Students are grouped into teams of 3–5, and each team is issued a separate substrate. Teams are given a partial procedure (Box 1), written in the condensed style of a journal experimental section, and a list of variables to modify.

**Box 1. Journal-Style Partial Procedure**

100 mg of ester was dissolved in 1 mL of an appropriate solvent and 0–5 equiv of an appropriate base were added (sometimes, no base was necessary). In some cases, 0–1 mL of water was also added. The reaction was stirred for 15 min at room temperature then halted by adding 5 mL of water. A sample was dissolved in methanol and analyzed by TLC.

- Choice of alcohol solvent: methanol, ethanol, or propan-2-ol
- Added water: Choose 0–1 mL of water

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indicating what total success looked like (Figure 4). With (50% v/v ethyl acetate in cyclohexane) and a diagram (sodium carbonate, isopropanol).

ment, but which students subsequently proved to be viable also included that had never been screened during develop-

which were less likely to be successful. Notably, options were

results in initial development, but some options were included

been to restrict options exclusively to those which yielded good

screening solvents). A more cautious approach would have

where a team exclusively tests variations on a condition known

structured such that teams were encouraged to diversify their

variables as appropriate. Written and verbal advice was

a small-scale test reaction but incorporating each of the

sequence

synthesis design loop rather than on revisiting the earlier

this information provided, attention is focused on the organic

synthesis design loop rather than on revisiting the earlier sequence’s reliance on student-developed TLC conditions.

Over time, it was established that students would test out pretty much any available liquid as a reaction solvent (TLC solvents, acetone, and dilute aqueous acids have all been screened). As the lab has a fairly restricted range of liquids available, there was no need to caution against this, but this may differ in other contexts if more hazardous reagents are at hand (see Hazards section).

The optimization is allocated between 1.5 and 2 h of a lab session, and this is usually sufficient time for each student to test two or three reaction conditions, giving the team as a whole somewhere between 6 and 15 data points. Of those, a majority usually indicate total conversion to product, with a 5-point Likert-scale survey and a selection of questions from a formal evaluation.

is not to systematically map out each condition, but simply to find one that works well.

The most successful conditions found by each team are scaled up by a factor of 10, and each member of the team separately follows the expository procedure they have created for themselves. The product is isolated by precipitation, and yield and melting point are determined. In a subsequent lab session, students self-sort into pairs and follow an expository pK_a determination procedure (this section can be shortened for time, to allow the synthesis to over-run). Finally, both lab sessions are prepared as a single joint writeup including a crude structure–activity relationship analysis on the pK_a data.

There are several places within this sequence where extraneous cognitive load is minimized by providing data or parts of procedures. For example, the test reactions are all run on 100 mg of ester substrate, and students are given the mass of base which corresponds to 1 mol equiv for each ester. This is an easy enough calculation, but not the objective of the work at hand. Including this calculation would not only lengthen the sequence, but also risk obscuring the core loop of the work behind a higher cognitive load barrier. Similarly, although the earlier sequence has students develop their own TLC solvent system, this is provided in this second sequence.

LESSONS FROM EXPERIENCE

The decision to allow scope for students to choose nonviable reaction conditions was validated by direct experience: initially, some test reactions always fail, but a student group as a whole invariably arrives at a viable set of conditions. This sequence has run for three academic years, and every single student (n = ~350) arrived at a viable set of conditions.

Individual students responsible for running failed reactions did not have a significantly negative emotional response but instead recognized the value of a negative data point and quickly moved on to a new set of conditions. The speed of iteration helps here; disappointment may only last for a few minutes. Students within a group who are struggling or work slower can still meaningfully contribute, due again to the speed of iteration. Such students are also invariably well-supported by their peer group.

Supportive and appropriately trained teaching assistants are critical for success, and they frequently report using this experience as a way of talking about the reality of research, bridging the perceptual gap for the students between themselves and a “real chemist”. Teaching assistants do need some specialist guidance to deliver inquiry-based education, and existing work in this area was an invaluable guide for updating training courses and support documents.

FORMAL EVALUATION

An ambitious attempt was made to formally evaluate the second sequence (organic reaction optimization) to measure any impact on experimental design ability. Students were given a 5-point Likert-scale survey and a selection of questions from

Figure 3. Flowchart given to students and used to structure their optimization work

Figure 4. TLC plate image used to indicate success or failure.
a previously published instrument used to measure experimental design ability, at three time points during the semester (early, mid, and end). Three time points were used as half of the class completed the sequence early and half completed the sequence late, allowing for control against general year-long improvement. However, engagement with evaluation was extremely poor, particularly toward the end of semester, and below the level that would yield useful data (Likert survey, n = 5, experimental design instrument, n = 2). This initial lack of engagement was due to a number of factors, including restrictions on recruitment due to local research ethics regulations. Evaluation in subsequent years was not possible owing to COVID-19 disruption.

Some impact could in principle be measured passively, by tracking changes to student attainment over time and comparing the relative performances of those who took the inquiry sequence early vs late in the semester. Grades were normalized for each practical and then normalized again for each week, a technique again inspired by the method of Lewis and Ralph. Controlling for these confounding factors, no difference in attainment was found, most likely because the grades analyzed were awarded for assessment criteria that did not judge experimental design ability.

Anecdotally, students consistently report this sequence as one of their favorites in standardised teaching evaluations. When polled, graduate teaching assistants felt that their groups learned much more about the process of doing research than they would have if they had not been involved. When asked if this was a useful onboarding experience, students consistently rated it as having provided a useful transition to their subsequent research experience.

■ HAZARDS

Safety glasses, lab coats, appropriate footwear, and gloves are all required throughout all practical sequences. Students should be prepared through an appropriate local risk awareness scheme; in this case, students are required to pass a mandatory safety assessment at the start of the year, and each prelab quiz contains a component of risk assessment specific to that lab.

TLC plates are precut, minimizing student exposure to silica dust. TLC solvents are measured, and TLC tanks are prepared in a fume hood and can be used on the open bench once covered over. Students choosing to investigate the effect of leaving a TLC tank uncovered are advised to conduct the investigation in a fume cupboard. Cyclohexane is substituted for hexane throughout owing to its much improved safety profile. Reference TLC samples are prepared in small quantities of solvent in a 2 mL narrow-necked vial and kept stoppered until used. TLC plates may subsequently be spotted on the open bench.

The esters and carboxylic acids used are irritants, and solid hydroxide bases are corrosive. Methanol, ethanol, and isopropanol are flammable and a health hazard and can be used at the open bench only in small amounts and at room temperature.

Student choice only varies the parameters of a reaction within a set window that does not have the scope to exceed typical hazards. Accidental addition of excess reagent does not lead to unexpected exotherms or toxic side products at any of the concentrations achievable. Short reaction times, low temperatures, and small scales minimize the possibility of unanticipated side products. Restrict readily available solvents and solutions to only those compatible with the reaction conditions.

■ CONCLUSION

Is this really an inquiry-based practical sequence? Although students converge on the expected procedures, they engage in an authentic procedure-generating process to do so. By Domín’s categorization rubric then, since procedures are at least partly student-generated and the scientific approach is deductive, the work is placed in the inquiry style. Using the more detailed inquiry level rubric of Bretz and Towns, each activity can be analyzed in greater detail.

The experimental aspects of the TLC sequence sit somewhere between guided and open inquiry, since the procedure/design is only partly provided. However, analysis and communication are tightly defined and would sit at a lower inquiry level, somewhere between confirmation and structured inquiry.

The organic reaction optimization sequence places fewer restrictions on results reporting and more confidently sits at a guided/open inquiry level in most areas. However, again, most inquiry characteristics are at least partially present, placing the work outwith any single neat category. This lack of definition is a consequence of focusing on experimental design specifically, without also raising the inquiry level of any postlab activities.

As a frequent critique of inquiry-based learning is the leap in complexity that comes with open-endedness, this work serves as a useful onboarding experience. The practical sequences can be incorporated early in a program of laboratory education in areas normally reserved for expository foundations, and the reported experience of students and staff is that the work provides a useful bridge between recipe-based laboratories and authentic research experiences.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00311.

- Screening development, technician procedures, and suitability of alternative esters (PDF, DOCX)
- Student lab manuals (PDF, DOCX)
- Teaching assistant documentation (PDF, DOCX)
- Student-completed worksheet for TLC practical (PDF, DOCX)
- Additional TLC interpretation supplement (PDF, DOCX)

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Notes

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