

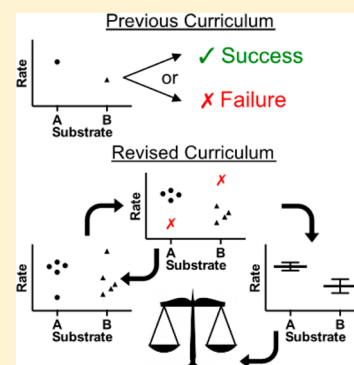
Aligning the Undergraduate Organic Laboratory Experience with Professional Work: The Centrality of Reliable and Meaningful Data

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S Supporting Information

ABSTRACT: Many traditional organic chemistry lab courses do not adequately help students to develop the professional skills required for creative, independent work. The overarching goal of the new organic chemistry lab series at Seattle University is to teach undergraduates to think, perform, and behave more like professional scientists. The conversion of experiments that inhibited professional development into simple but authentic studies that generate reliable and meaningful data is described. This strategy seems to help students better assimilate into research groups and could easily be applied to a broad range of experiment types and undergraduate teaching environments.



KEYWORDS: Second-Year Undergraduate, Laboratory Instruction, Collaborative/Cooperative Learning, Constructivism, Organic Chemistry

Individual chemistry departments organize their curriculum around different outcomes based on the student populations they serve and the level of professional development they aspire to achieve.^{1,2} Like many primarily undergraduate institutions, our students conduct undergraduate research as a capstone. For this reason, the goal of the organic chemistry lab series at Seattle University is to teach undergraduate students to think, perform, and behave more like professional scientists^{3–5} so that they are better prepared for their research experiences and their eventual careers. To achieve this goal, our prior organic chemistry lab curriculum⁶ was scrutinized in two ways. First, skill sets were identified that are required for high quality independent research, but that were missing from the curriculum. Second, we identified the ways in which the curriculum seemed to inhibit creative thought, independent work, and meaningful collaboration.

Examination of the experiments in our prior organic chemistry lab courses revealed two opportunities for improvement. First, most experiments lacked an authentic context and scientific question because students produced outcomes that were known in advance by both students and faculty. This problem has been discussed at length in the literature in the context of inquiry-, discovery-, problem-, and argument-based labs; such experiments provide a more natural context for scientific thinking.^{7–15} A second issue has received some attention in the literature in the context of POGIL laboratories and is arguably more fundamental:¹⁵ asking students to reflect on the meaning of a single *datum* is something a practicing chemist would be reluctant to do without repeating an experiment or without the context of other closely related results. Rather, scientific thinking requires experiments that generate *data* that are worthy of analysis, critical thinking, and discussion.^{15–20}

This article, one component of a much larger project to revise our organic chemistry laboratory courses,^{21,22} describes an approach to redesigning several organic chemistry lab experiments that previously failed to produce a plurality of data of sufficient quality about which students could think and write authentically as scientists in training. Our new three-quarter organic chemistry lab series contains approximately 50% redesigned, data-generating experiments, and 50% experiments that are more traditional. In the first quarter course, which focuses on learning the commonly used lab techniques of organic chemistry, two of five experiments have been redesigned. In the second quarter course, which focuses on using the basic lab techniques of organic chemistry in procedures that have been published in the scientific literature, two of four experiments have been redesigned. In the third quarter course, which focuses on applications of organic chemistry toward solving a novel (unpublished) problem, the previous experiments have been converted into a quarter-long research project. Redesigning experiments to generate reliable and meaningful data has enabled our organic chemistry students to engage in authentic data analysis, in data interpretation, and in data-driven decision-making.

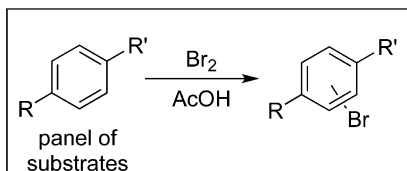
REDESIGNING EXPERIMENTS TO PROMOTE SCIENTIFIC THOUGHT

Four features were incorporated into each redesigned experiment, when possible: (1) generation of reliable and meaningful data; (2) authentic data analysis and processing, both in real-time and also post-lab; (3) progressive instruction in data

interpretation; and (4) data-driven decision-making. Each feature is discussed below in more detail.

Using Collaborative Work To Generate Reliable and Meaningful Data

Here, the phrase “reliable and meaningful data” refers to data sets that are derived from multiple replicates that could be treated with the tools of basic statistical analysis to identify outliers before calculating the average and standard error. The importance of reliable and meaningful data for helping students develop their scientific thinking skills can best be illustrated by considering what prior experiments tended to accomplish in the absence of these traits (Figure 1). For example, in one



Prior lab

In the laboratory:

- each student acquires one datum for each of the 8 substrates

Outside the laboratory:

- each student attempts to interpret differences between the datum she/he acquired for each substrate
- students write a lab report

Redesigned lab

In the laboratory:

- each student acquires 5–8 data points for one of the 6 substrates
- data are projected in real time during lab and discussed
- based on data, students refine procedures or obtain additional replicates to mitigate variability

Outside the laboratory:

- using reliable raw data, students process the data and interpret its meaning, weighing differences in light of data scatter
- students write a scientific article

Figure 1. Comparison of the prior lab design to the redesigned experiments for the electrophilic aromatic bromination of substituted benzenes.

traditional experiment students conducted an investigation of the relative rates of electrophilic aromatic bromination reactions.²³ Each student team ran the bromination reaction one time on each member of a panel of 5–8 substrates, recording reaction completion times. Because each team performed only one replicate per substrate, data interpretation often proved impossible because of erroneous data that resulted from inexperience. As a result, students often concluded that their experiment was “a success”, or that it “failed” due to “experimental error”. Because students, who thought they had “good” data, knew (or thought they knew) the outcome in advance, they were reluctant to think critically about their findings. The experiment seemed to reinforce students’ view of themselves as inauthentic participants in science, rather than as scientific discoverers.

Thus, the primary objective of the redesign process was to develop experiments to generate data that would be sufficient for meaningful analysis and reliable enough to draw sound and valid conclusions. The constraint of lab time was the greatest

obstacle for students to generate these types of data sets. To streamline many experiments, a “divide-and-conquer” strategy was introduced where each team was assigned a slightly different experimental condition (e.g., different substrate structure, reaction time, or reaction solvent) with the aim to explore a particular variable as a class of collaborators. Each student team was responsible for conducting multiple replicates (typically 5–8) of their reaction; student data were pooled in a spreadsheet and projected in real-time (during lab). Naturally, replication resolved many of the reliability problems associated with a single observation generated by individual student teams. The inevitable variability in data focused students on technique rather than reliance on some known or speculated “experimental error” as a reflexive justification of unexpected outcomes. The generation and analysis of replicates is especially important early in students’ laboratory experience because novices are just beginning to develop the technical skills required to reproduce experimental procedures with accuracy and precision. This also allows students to self-assess the quality of their lab work.

In redesigned experiments, all data were shared between all members of the lab period. Students drew conclusions based on the compiled set of data as a whole, rather than on their individual experimental results.¹⁵ In this way, experiments done by each isolated student team were converted into a meaningful and collaborative investigation¹⁷ of an authentic scientific question. This relatively simple adjustment more accurately reflects the research laboratory environment because it demonstrates to students the obvious advantages of collaboration and replication. This approach is particularly effective for faculty who aim to conduct experiments where the outcome is designed to oppose students’ expectations because this strategy makes it difficult for students to disregard unexpected results or to ascribe some vague notion of “experimental error” for a “failed” experiment. Instead, students are forced, as scientists are, to generate a cogent explanation for unexpected results.

Incorporating Data Analysis and Processing Both in Real-Time and Post-Lab

Focusing on the generation of reliable and meaningful data has enabled authentic data analysis and processing both during and after the lab period.¹⁵ At the most basic level, periodic breaks are scheduled during the lab period for preliminary discussions about pooled data, which are displayed in real-time using a computer and digital projector.¹⁵ A list of leading questions are used in these discussions (Box 1).¹⁵ These faculty-facilitated conversations are motivated by the need for a scientifically acceptable level of variability before students begin to interpret

Box 1. Example Leading Questions Used in Discussions That Focus on the Level of Confidence in the Data Being Generated, and the Relationship of the Experimental Design to the Quality of the Data

- Is the experimental design capable of generating the required reproducibility?
- If not, how can the experimental design be modified?
- Is the data quality (both accuracy and precision) sufficient to address the aim of the experiment?
- What are the likely origins of variability?
- How could the procedure be improved to mitigate the observed variability?
- Are there experimental treatment groups that require additional replicates?

a final data set. Although this level of analysis is usually qualitative, it helps build a valuable conceptual foundation for understanding data scatter. For example, when the results of two student groups were similar, but not identical, the need for replication and data analysis became obvious. The outcome of these conversations is often a refinement of procedures or a clear need for more replicates. A more rigorous treatment of data is incorporated into our scientific writing program²¹ in which students conduct comprehensive data analysis after the lab period.¹⁵ After completing a hands-on workshop on basic statistical analysis, students identify statistical outliers, and calculate averages and standard for any pooled data generated in the course.

Teaching Data Interpretation

Elements of data interpretation are taught in two ways, both of which are designed to help move students along a continuum toward how practicing chemists actually think about data and communicate their interpretations. At the end of each lab period, the instructor leads a discussion about the compiled data set, emphasizing how scientists analyze large data sets, interpret their meaning, and make claims. For example, leading questions are asked such as What are the general trends in the data? What are reasonable interpretations of the data? What are the implications regarding substrates or reaction conditions not investigated? With what certainty can you make your claims? These discussions are geared toward preparing students for the comprehensive interpretation they will conduct independently as homework.

As part of our scientific writing program,²¹ students are taught the types of rhetorical arguments that professional chemists make in scientific articles. As homework, students make their own written claims through direct reference to their processed data. This design was informed, in part, by the Science Writing Heuristic (SWH);^{18–20} however, our program is distinct from the SWH in that our process of writing scientific claims is embedded within an authentic professional genre, the scientific article.^{24–26}

Making Data-Driven Decisions

Whenever possible, some aspect of data-driven decision-making is incorporated into experiments. Ideally, students use compiled data to generate a new hypothesis that will be tested in later experiments (either later that day or in a subsequent lab period).¹⁵ However, it has also been useful to engage students in more limited applications of data-based decision-making. For example, several experiments in the first quarter lab course use a divide-and-conquer approach to optimize the specific details of a particular purification. In that context, students' analysis of preliminary data from their small-scale experiments leads to a decision about how a larger-scale purification will be conducted, mirroring the process of a professional chemist. In a similar way, several investigative experiments in the second quarter lab course involve analyzing data in real-time (during lab) so that additional replicates can be conducted, as is usually necessary. By emphasizing data-driven decision-making, the continuity of scientific work is stressed; specifically, data sets are not generated as an end unto themselves, but rather are applied toward solving a problem or generating new lines of inquiry, thus closing the loop on the scientific method. This active interplay between bench work, qualitative analysis, and experimental design is, perhaps, one of the best models we provide to students for how practicing professionals approach scientific inquiry.

SUMMARY OF REDESIGNED EXPERIMENTS

A summary of each redesigned experiment is given below; complete details of all the experiments are available in the Supporting Information.

Many published experiments used in organic chemistry teaching laboratories have students learn separation and purification techniques (e.g., recrystallization, extraction, distillation, thin layer chromatography, column chromatography) after running a reaction, as a synthetic chemist would do. In ideal circumstances, such experiments provide a natural context for when each purification technique is used. However, for students, the learning objectives for such experiments can often appear muddled. Are students meant to focus on reaction setup? To explore the connection between reaction conditions and mechanism? To master the specific details of a particular purification? Or all of these? Because our prior experiments on thin layer chromatography and recrystallization were inadequately focused, students often failed to master the most important concepts underlying these common lab techniques. Thus, in redesigned experiments, the chemical reaction was removed to allow for deeper learning that is focused only on our desired learning outcomes.

Thin Layer Chromatography

For thin layer chromatography (TLC), the learning outcomes are to be able to run TLC on any sample and to develop an understanding, based on data, of the factors that affect R_f value. In line with these outcomes, the redesigned TLC experiment asks students to investigate how R_f values are affected by eluent and analyte structure. Each student pair chooses three of six different eluents and three of 12 different solid analytes, and determines the R_f value for each combination. Thus, a class of 8 student pairs can generate R_f data for 72 conditions. The instructor then uses the projected data to lead a discussion on the factors that affect R_f value. Each team is then provided an unknown binary mixture containing two of the 12 previously analyzed compounds, and students learn how to prepare an appropriately diluted solution of the sample for TLC analysis. Students then use the combined R_f value data to select conditions that will provide adequate separation of their two compounds, and conduct new TLC analysis using standards to identify the two components of their mixture.

In this experiment, the divide-and-conquer approach allows the class to quickly gather reliable R_f values, which are then interpreted, discussed, and used to inform students' decisions of how to conduct a subsequent TLC analysis. Discussion of the initial R_f value data engages students in thinking about data during the lab when the professor can model the process and is available to correct illogical thinking or erroneous conclusions. At the end of our redesigned experiment, students know how to conduct TLC, that is, how to prepare samples, use standards, and choose eluents. Perhaps more importantly, students leave the lab with a much more intuitive understanding of the dependence of R_f values on analyte functional group and eluent choice. This improved understanding has been particularly apparent in our research laboratories where students have become much more adept at predicting changes in R_f value during a reaction (e.g., monitoring an acylation reaction by TLC) or at determining eluent conditions for column chromatography (e.g., transitioning from mixtures of EtOAc:hexanes to mixtures of CH₃OH:CH₂Cl₂). Although this basic experiment design is not entirely novel,²⁷ our particular approach has led to a reliable, meaningful and voluminous data set that is obtainable in a single

3 h lab period from which conclusions can easily and reliably be drawn.

Recrystallization

Similarly, a recrystallization experiment was redesigned to focus student learning on the desired outcome: to demonstrate the ability to carry out a recrystallization in a real world setting (where the ideal solvent, compound solubility and optimized temperatures are unknown) and to understand the factors that affect recrystallization yield and purity. The scientific objective of this experiment is to purify a crude 9-fluorenone mixture contaminated with 9-fluorenol. To begin, each student team determines the solubility of 100 mg of a mixture of 9-fluorenone/9-fluorenol in one of six different solvents and at two different temperatures. Solubility data are shared across the entire class, and the instructor leads a discussion of the meaning of the data in the context of the recrystallization goal. Each team then selects a recrystallization solvent and conducts the recrystallization on 1 g scale. Again, acquired data (solvent volume, percent recovery and purity) are shared across the entire lab section, and the instructor leads a discussion aimed at data interpretation (e.g., how the volume of solvent used affects percent recovery and purity). At the end of this redesigned experiment, students understand how a recrystallization solvent is selected based on experimental data, and they are equipped to use this skill to conduct recrystallizations in their research labs.

Reduction of 9-Fluorenone

The first chemical reaction that students perform in the revised curriculum is a model study similar to those often conducted in synthetic research labs: to develop a method for the reduction of a 9-fluorenone moiety in a complex enzyme inhibitor, students are asked to optimize reaction conditions using 9-fluorenone as a model system and NaBH_4 as a reducing agent. The two variables selected for optimization are reaction solvent and reaction time. Student teams investigate reaction completion times in methanol, ethanol, isopropyl alcohol, and THF. Reaction completion times are estimated by TLC. As in the experiments above, student data are collected in real time and additional replicates are conducted until the trends observed are unambiguous. On the basis of the data, students conduct a synthesis of 9-fluorenol using the optimized conditions on a scale sufficient for isolation and characterization by ^1H NMR, ^{13}C NMR, and IR spectroscopy.

Two Synthetic and Two Kinetic Experiments: A Quarter-Long Experimental Study

A traditional experiment on the relative rates of electrophilic aromatic bromination reactions was redesigned into a four-period inquiry-based experimental study (Figure 1). The project asks students to investigate the bromination of 1,4-disubstituted benzenes in aqueous acetic acid as solvent, rather than using the conditions they learn in the associated lecture course (i.e., aprotic organic solvent and Lewis acid catalyst). This context provides students with an authentic problem that is not directly discussed in most organic chemistry textbooks.

In this study, the first lab period is dedicated to the synthesis of 4'-methylacetanilide. In the second lab period, students measure the relative rates of bromination of the synthesized 4'-methylacetanilide and two other purchased substrates. Each student team investigates the reaction time of one substrate; thus, in a class of 16 students, each substrate is investigated by at least two teams. Students conduct sufficient replicates to provide a reliable data set, discarding outliers using the Q test.

Usually, this is accomplished with about eight runs per substrate, resulting in six runs that all pass the Q test; a complete data set is easily obtained within 2 h. At this point, the class pauses to discuss the data with the goal of identifying preliminary trends. On the basis of the discussion, the class imagines additional aromatic substrates that might serve to elucidate further the preliminary trends identified. After class the instructor determines which substrates are suitable (with respect to cost, toxicity, commercial availability, etc.) to generate a list of three new substrates for students to test in a future lab period.

The third lab period is dedicated to the synthesis of one of the three new substrates. The fourth lab period is spent as the second lab day was, measuring the relative rates of the three new aromatic substrates (the student-synthesized substrate plus two purchased substrates). As before, there is enough redundancy built in to obtain reliable and meaningful data from many replicates.

This four-period experimental study represents an excellent example of how students can use data in the way that scientists do. Armed with data that are reliable enough to make reasonable predictions, students then test their predictions using new substrate arenes, and they plainly see how and why scientific studies are conducted, and they seem to enjoy this process. In our experience, prior synthetic experiments often suffered from severe inauthenticity that was easily articulated by students as they discarded their isolated product into chemical waste at the end of the lab period, or never used their isolated product subsequently. By using synthesis to make substrates that are part of a larger study, students understand that synthesis is often not an end of its own, but may be used as a tool for numerous kinds of scientific investigations. Collaborations like this one have largely eliminated students' "race to finish" that we routinely observed in our prior experiments.

■ EFFECTS OF THE REDESIGNED EXPERIMENTS

Although no pre-/post-test data were collected to demonstrate the effectiveness of the course changes aimed at teaching undergraduate students to think, perform, and behave more like professional scientists, anecdotal support for our redesigned organic chemistry laboratory courses is nonetheless valuable.

As an instructor in the organic chemistry lab courses, these changes seem to have reduced the sense of "futility" or "busy work" that many of the best students often reported in the prior curriculum. The redesigned experiments, and in particular the quarter-long experimental study, successfully model to students what the process of doing science is like. The reduced scope of many experiments has provided instructors ample time to discuss data and model scientific thinking during lab, but it also provides students ample time to think and ask pertinent questions during lab. Finally, faculty find it easier to pilot new experiment ideas because students are less troubled by what they previously would have thought of as a "failed" experiment.

Instructors in subsequent courses (quantitative analysis, physical chemistry and biochemistry) have reported numerous advantages of the redesigned organic chemistry lab experiments. These instructors have noted that students start their courses acculturated to the idea of shared data sets and the importance of their individual data in the context of a large collaboration. Students also more readily anticipate the need for deeper statistical analysis and error analysis that they learn in these later courses. Also noteworthy is the fact that students arrive in subsequent courses expecting to be answering unknown questions in the laboratory. Although this can be

challenging in courses like physical chemistry, it is a natural fit for courses in quantitative analysis and biochemistry.

Faculty research mentors also report benefits of the revised organic laboratory curriculum. Because many experiments in the organic chemistry lab courses resemble bona fide research, it has become easier to identify students who are likely to transition successfully into undergraduate researchers. Also, students appear to find it easier to solve problems in purification, to modify procedures, to use different kinds of graphing and statistics software, to find characterization data in the published literature, and to interpret data independently. Finally, faculty report that collaborative efforts within their research labs proceed more smoothly, as students are already acculturated to the needs for sharing data, refining procedures as a team, and presenting their results in a cogent and more professional manner.

■ LIMITATIONS OF USING THIS APPROACH

As with any curriculum change, the one described here involves several trade-offs when compared to more traditional experiments. One significant trade-off is depth versus breadth. To allow sufficient time for replication, data pooling, analysis and discussion, the complexity of some experiments has been reduced. In addition to the reduction of experiment breadth, the skills required to teach data analysis and professional writing take considerable time to develop. In our curriculum, this is accomplished using workshops conducted during the lab period, placing a constraint on experimentation time. To minimize the impact on experimentation time, many of the workshops were divided into smaller installments that are conducted during gaps in lab activity; for example, some writing workshops are completed during a reaction reflux or other long incubation times.

■ THE ROLE OF TRADITIONAL EXPERIMENTS IN THE CURRICULUM

As mentioned above, about half of the experiments have been redesigned in the year-long series of organic chemistry laboratory at Seattle University, and about half are more traditional experiments. This is intentional because there is an important role for some experiments that would not benefit from a divide-and-conquer approach. In addition, even purely synthetic experiments sometimes generate copious data (e.g., ^1H NMR, ^{13}C NMR, IR, MS, mp, bp, % yield, % ee) that students report and discuss without replication, as a professional would when conducting a synthetic study. For synthetic experiments that generate primarily spectroscopic and physical data, the interpretation done as homework includes comparisons to data from the primary literature, which students learn in class during workshops that provide the tools to find and interpret reliable chemical information of this type. Skills related to finding and using literature data are reinforced in the context of a writing program, where students must provide citations to the primary literature for the spectral data they discuss.

■ CONCLUSIONS

This article presented an approach to redesigning many organic chemistry teaching lab experiments to produce data sets that were reliable and meaningful enough to enable authentic scientific thinking and writing. These experiments aimed to train students in the “habits and ways of thinking” of professional scientists by engaging them in the iterative process of a profes-

sional who is conducting a scientific study. A laboratory culture characterized by careful experimental technique, responsible and engaged collaboration within groups, and clear communication between groups who are working independently was created. By having students work collaboratively to address an authentic problem with the tools of experimental design, data replication and data analysis, the intent was to model for them what it is like to be a scientist, and to prepare them to assume that role in their undergraduate research projects and beyond.

■ ASSOCIATED CONTENT

■ Supporting Information

Course syllabi and lab manuals, instructor teaching notes, PowerPoint presentations, writing instruction packet, and sample writing assignments. This material is available via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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