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Developing Students' Critical Thinking, Problem Solving, and Analysis Skills in an Inquiry-Based Synthetic Organic Laboratory Course

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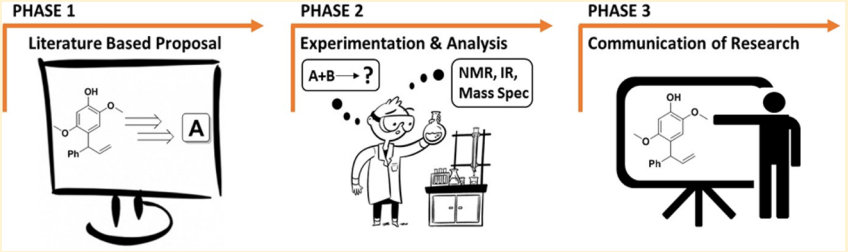
Supporting Information



ABSTRACT: A course is described where students are engaged in an inquiry-based quarter-long research project to synthesize a known pharmaceutical target. Students use literature search engines, such as Reaxys and SciFinder, and the primary chemical literature as resources to plan and perform the synthesis of their pharmaceutical target. Through this process, students develop critical thinking, problem solving, and data analysis skills in the laboratory setting. Teaching assistants guide students through three phases of the research process: (1) literature-based proposal, (2) experimentation and analysis, and (3) communication of research findings by oral presentation and written report. This course is designed for upper-division chemistry majors, taken as the third-quarter organic laboratory class after two quarters of introductory laboratory courses and two quarters of the associated organic chemistry lecture courses. Nine different teaching assistants have taught the course described to over 90 students in four different quarters.

KEYWORDS: *Upper-Division Undergraduate, Laboratory Instruction, Organic Chemistry, Inquiry-Based/Discovery Learning, Synthesis*

Many students majoring in chemistry develop a curiosity about research early on in their undergraduate career. Limited research opportunities for undergraduates leave many students unable to explore this curiosity and forces them to look toward undergraduate teaching labs to satisfy this interest. However, there is a consensus in the chemical education literature that traditional organic laboratory experiments do not provide students with an authentic research experience and deprive them of the necessary skills to be successful scientists.[1a−e,2] These traditional labs place little emphasis on critical thinking, planning, interpreting results, or discussing conclusions.[1] This laboratory approach can lead to little more than factual verification. To address the laboratory curriculum concerns mentioned previously, many authors advocate for a project-based approach.[1a,3] This project-based approach provides many degrees of freedom with flexibility in the targets, procedures and chemicals, which provides a more authentic research-like lab experience.

To provide students an opportunity to participate in a research-like setting and to develop their data interpretation and analysis skills, we have implemented an inquiry-based laboratory course for our chemistry majors. This course is ideally taken at the end of their second year or beginning of their third year, after two quarters of organic laboratory. In this project-based course, students have the opportunity to learn to navigate the primary chemical literature and to develop critical-thinking skills and advanced research and laboratory techniques.[4] Students are assigned to synthesize a pharmaceutical target through a process modeled after academic research with three phases: (1) literature-based proposal, (2) experimentation and analysis, and (3) communication of research findings via an oral presentation and written report. This course was designed to minimize the demand placed on the undergraduate stockroom while providing students a simulated research experience. Herein, we report our approach to implementing this course at UCSB.

COURSE GOALS

To foster a research environment, our goal is to encourage the mentoring relationship between each student and their teaching assistant and to encourage the investment of each student in their project. The teaching assistant's role in the course is to be a mentor, encouraging students to use the scientific method,

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ask questions about their results, overcome problems when necessary, and make valid conclusions about their work. This helps the students become engaged in their project. The teaching assistant is also a resource for students when they do not understand an idea or a new technique. When needed, the teaching assistant can intervene to ensure that each student manages their time efficiently and stays motivated throughout their synthesis project. The element of the "right or wrong answer" is attenuated in the inquiry-based approach. Rather, the teaching assistant helps students realize that there are generally many methods to reach their synthesis goals, but one might produce better results, be safer, cheaper, or more efficient.

The successful student realizes that there are a variety of aspects to consider when approaching their project and any problems that arise. For example, the student must use their previous knowledge of organic chemistry combined with their new search tools (Reaxys and SciFinder) as a means to build their knowledge and formulate successful laboratory protocols. The students must also reassess their synthetic procedures and adapt their methods when unexpected results are encountered. As a result, students are directly engaged in developing their own learning process and in gaining a sense of responsibility for their project and their education. Additionally, students often become interested in other students' projects and engage in discussion between groups about the larger goal of their synthesis and the differing synthetic procedures and concepts. We have found that when students have an invested interest in their project, they often come to the instructor with proposals, asking for guidance rather than instruction. This learning transformation is important as they begin their transition to becoming scientifically minded individuals.

PHARMACEUTICAL RESEARCH PROJECT

Project Targets

Seven distinct targets (Figure 1) have been implemented thus far.[5] The targets chosen provide a tangible, real-world application of organic synthesis as opposed to the abstract syntheses performed in traditional labs. However, the potential syntheses are not limited to those in this manuscript. This enables the projects to be continually rotated. Each quarter, our aim is to have distinct targets for each group in each section. Complete synthesis guides for the pharmaceutical targets, including the necessary laboratory equipment, reagents, and procedures for the routes we have determined to provide the best student results, are available in the Supporting Information.

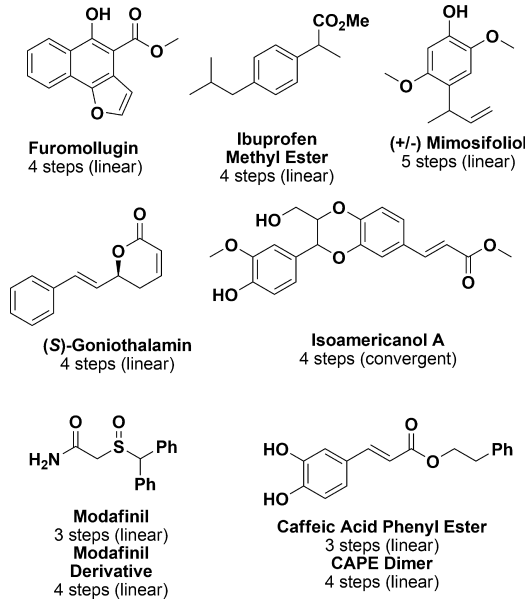

Figure 1. Pharmaceutical targets.

COURSE IMPLEMENTATION

The course design and curriculum described was taught to 90 students in ten unique laboratory sections, spanning four quarters, and instructed by seven different teaching assistants. Each section contained up to twelve students and met for two sessions that lasted 4 hours each per week over a ten week quarter. Requirements pertaining to the laboratory space and project materials are detailed in the Supporting Information for both the instructor and the person(s) responsible for setting up the laboratory.

Students work in pairs; each pair is assigned to synthesize one of the target structures. Students are then guided through the three phases outlined below.

Phase 1: Literature-Based Proposal

The first phase of the course is dedicated to developing the project using Web-based resources. Students begin with a retrosynthesis assignment, guided by the teaching assistant, and learn the importance of their assigned target molecule.[6] Next, they delve into the primary literature with the aid of Reaxys and SciFinder to find a viable synthetic pathway.[7] Most of the target molecules have more than one known synthetic route; a worksheet is provided to guide their planning toward a feasible, safe, and cost-effective known route that has been identified for each pharmaceutical target so the stockroom can plan ahead of time what reagents each group will use. However, the students are given the freedom to try other routes/reagents if they can find sufficient literature precedent. Students are required to discuss their synthesis plan with the instructor and make necessary adjustments before proceeding to the laboratory. The background research, retrosynthetic analysis, and synthesis plan are then used to write a "grant-like proposal" that ultimately serves as a starting point for writing their written report (Phase 3).[8]

Phase 2: Experimentation and Analysis

During the second phase, students complete the synthesis of their pharmaceutical target; each synthetic route is 3−5 steps in length and uses readily available starting material. Each group is given 4 g of starting material and is encouraged to begin on a smaller scale (∼100 mg), then scale-up when the reaction conditions are validated. Due to the diverse nature of the projects, students are required to complete reaction sheets that detail the reaction scheme, intended experimental setup and procedure, workup, and characterization predictions. Students are also asked to include any relevant physical and safety data about the reagents they will use. As students work through their procedures, they use characterization techniques such as TLC and NMR spectroscopy to analyze their results.[9] Although most of the procedures are reported in the literature, students are required to adapt protocols for their project within the constraints of their laboratory setting. Instructors continually assess student progress and provide recommendations. An instructor guide for each synthesis is included in the Supporting Information.

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Phase 3: Communication of Research

In the third and final phase of this course, each group presents their research findings and each student writes a manuscript, using the *Organic Letters* template, to communicate their progress. Approximately 2 weeks before the conclusion of the course, students submit a draft of their initial manuscript and slides that will be used in their oral presentation. The instructor and student peers (if desired) provide feedback to the students before they submit the final manuscript and present their work orally. Emphasis is placed upon writing and effective presentation styles. The final oral presentation is delivered to their lab mates, teaching assistant, and the class instructor on the last day of instruction. Classmates are required to ask questions and provide written peer review commentary and scores to the instructor. After a peer-led discussion, the instructors ask additional questions and provide constructive criticisms on presentation content and style. We have found that for many students this is their first opportunity to present scientific findings.

HAZARDS AND SAFETY

Some of the reagents used in this course are hazardous to health or dangerous when used improperly. These hazards are outlined in the Supporting Information in the instructor guides. To be certain students used these reagents properly, Standard Operating Procedures (SOPs) were developed and kept in the laboratory for reference. Students signed the SOPs pertaining to their project after reading them in order to ensure understanding. Instructors made certain to be mindful of students using these reagents and closely supervised their work. Proper disposal containers were provided for organic, aqueous, and solid waste. Students also completed a safety video and a brief online quiz through the on-campus Environmental Health and Safety Web site. Proper attire was required at all times. This included lab coats, goggles, and gloves.

STUDENT FEEDBACK

Student feedback was positive and indicated that they had valuable pedagogical relationships with both their teaching assistant and their project. They commented on learning about synthesis and the problem-solving aspects that are necessary to complete a research project. Students realized that it was necessary to think about the *what*, *why*, and *how* questions through analysis of results. Students often mentioned that the independent nature of the research project was unexpected, but the ability to see a complex project from beginning to end made it more interesting than previous laboratory courses. With this ownership of the learning process, teaching assistants noticed that the students had a greater desire to learn and a greater appreciation for what is learned. Since this course has many different projects running concurrently, it encouraged students to think more independently and help each other understand conceptual points and knowledge rather than just check with their lab mates to be sure they were doing the experiments "correctly". Although most students still found organic chemistry difficult, they realized that synthesis requires more than just following a recipe. Select student quotes are included below. All student feedback collected is found in the Supporting Information.

> "I actually feel like I did real chemistry for the first time."
> "It taught you to be organized and effective in your planning."
> "Nothing is necessarily set in stone, use common sense and apply it to the basic principles given. [This course] allowed for critical thinking besides just following instructions."
> "I liked how it was one big experiment as opposed to several small ones."

CONCLUSIONS AND POSSIBLE ADAPTATIONS

The Supporting Information includes reported literature yields and we have found that while many students were able to achieve these yields, or even higher, others struggled. However, most students that were diligent were able to synthesize their final product in the time allotted. This course was implemented into a 10-week quarter system, but can easily be adapted to the semester system in a variety of ways. In a semester system, we suggest implementing this course as an advanced organic laboratory course or as part of the second semester organic chemistry laboratory. If the semester is divided into two parts, the first part can be dedicated to other wet-lab experiments and Phase I of the pharmaceutical research project to allow for more investigative time and instructor feedback in the planning stages. The second half can then be used for Phase II and III of the project. An alternate way to implement this course in the semester system is to expand the curriculum to go in depth on related topics including the following:

(1) A presentation on grant proposal writing with more opportunities for feedback.
(2) A more in-depth assignment or group-work to introduce retrosynthesis of more complex molecules.
(3) Hands-on demonstrations of standard laboratory techniques (this could include time to implement reagent titration or purification for the projects).
(4) An iterative review process for writing the final report manuscript.
(5) A discussion on how to formulate figures, schemes, and tables for publication.
(6) Demo presentation given by the teaching assistant, professor, or guest speaker to address presentation skills and style.
(7) A discussion on green chemistry and analyses of how the students can make their synthetic routes "greener".

If laboratory constraints exist such as the lack of nitrogen lines or dry solvents,[10] the authors suggest alternative adaptations in the Supporting Information within the instructor guides and stockroom guidelines. If Internet resources are limited and students do not have access to Reaxys, SciFinder, or the primary literature articles, we suggest providing the necessary articles for the corresponding project to the students in a literature packet or having the articles available to check out upon request. A list of the necessary journal articles for instructors to request is included in the Supporting Information within the synthesis guides. This approach does not allow students to explore the chemical literature in the most authentic way, but nonetheless, it provides students with the opportunity to read the primary literature, to adapt the experimental procedures, and to learn about the process of chemical research in a pedagogical laboratory setting.

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■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00678.

Student packet, which includes relevant student handouts, a sample schedule, and useful appendix items (PDF, DOCX)

Instructor packet, which contains student handouts specific to each project, a sample schedule, instructor guides with experimental details for all targets, laboratory and chemical requirements, and sample quizzes (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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