Integrating Chemical Information Skills in a Problem-Based Second Semester Organic Chemistry Laboratory toward the Synthesis of Adipic Acid

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Abstract: There is a continued need for laboratory experiments that integrally involve chemical information skills with designing and solving a laboratory research problem. Herein, we describe a learning experience where second semester organic chemistry laboratory students carry out their own research activities toward the synthesis of adipic acid. This lab was developed out of a strategic partnership between the University Library and Department of Chemistry and Chemical Biology. The primary objectives of this lab include integrating chemical information skills into a problem-based laboratory experiment, increasing scientific research ability for chemistry students by involving a research cycle, and bolstering student perceptions of research and laboratory work. The relevance, safety, cost, scalability, growth potential, and success of this learning experience are also discussed.

Introduction

In an era of rapidly emerging scientific discoveries, the ability of chemists to efficiently find and utilize information is vital. The inclusion of basic online information searching tasks in student laboratories has been reported since the 1980's [1–4]. However, chemical information experiences that involve searching for a single fact or fragment of information do not reflect the rich needs of today's professional chemists. The ACS Committee on Professional Training has underlined the importance of chemical information searching on future professionals, stating "students should be able to use the peer-reviewed scientific literature effectively and evaluate technical articles critically. They should learn how to retrieve specific information from the chemical literature, including the use of *Chemical Abstracts* and other compilations, with online, interactive database-searching tools [5].” Additionally, the Special Libraries Association, Chemistry Division, suggests that “chemistry undergraduates should be able to understand the scope and nature of scientific literature, interpret and evaluate scientific literature, and follow a logical path of inquiry” [6].

Problem-[7] or project-based labs [8], which are mostly synonymous in literature, are the typical vehicle for involving chemical literature in undergraduate laboratories. These non-traditional approaches are defined by an outcome that is unknown to students and faculty, a procedure adapted by students, and a topic or problem that can be selected by either the student or faculty (to control the range of student research). These experiences can be found in the sophomore organic laboratory [8], while others are implemented in the final year or two of the undergraduate career [9–11]. Though problem-based experiments are often justly labeled [7, 8, 12] as time consuming, costly, potentially dangerous, and frustrating for both students and faculty, it is widely held that these experiences encourage higher-order cognitive skills [13, 14], ultimately preparing students for independent research.

Herein, we report a problem-based laboratory reliant on chemical information skills. The experiment was developed out of a strategic partnership between the University Library and Department of Chemistry and Chemical Biology at Indiana University Purdue University Indianapolis (IUPUI). Such a partnership has been successfully implemented in previous reports [15, 16]. Using chemical information skills, second semester organic chemistry laboratory students are empowered to design and carry out their own experiments toward the synthesis of adipic acid from cyclohexene. Recent literature [17–25] on adipic acid synthesis supports a diverse yet feasible assortment of research topics (Figure 1). Vital to the design of this experience are a number of reports that utilize aqueous biphasic, multi-catalyst reactions which provide a safe setting for significant diversity within a narrow synthetic pathway motivated by green chemistry. The primary objectives of this lab experience include integrating chemical information skills into a laboratory experiment, increasing scientific research ability for chemistry students at IUPUI by involving a research cycle, and bolstering student perceptions of research and laboratory work.

This particular research problem was selected because the synthesis of adipic acid, one of nylon’s fundamental building blocks, is being actively researched in the scientific community and has direct economic and environmental implications. Students are therefore actively participating in research where the answers are not immediately obvious yet are vital to the world around us.

The Setting

IUPUI is a large urban research university and is Indiana’s primary health sciences campus. The second semester organic chemistry laboratory course at IUPUI consists of two 2h50m

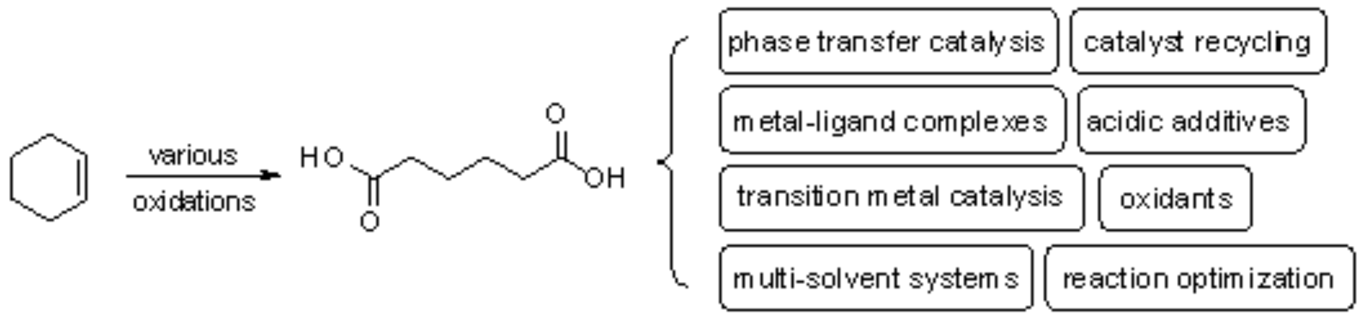

Figure 1. Potential research topics available in adipic acid synthesis.

meetings per week over a 15 week fall or spring semester. Each laboratory section's enrollment is capped at 20 students, with an annual enrollment of 250 students. Within the laboratory space itself, every pair of students share a fume hood, with individual lab drawers beneath. Each lab section is led by a teaching assistant (TA), most of which are junior or senior undergraduates.

Somerville and Cardinal describe the importance of a tiered approach [16] to teaching chemistry information skills, where students build on skills as they progress through their undergraduate program. Librarian involvement with chemistry classes at IUPUI follows a similar tiered approach. In introductory/freshman experience courses, the librarian introduces students to basic resources provided by the library and leads a discussion on differences between popular and scholarly information. Students locate books and reference resources (using an online catalog) for an introductory literature project, while also learning to identify the elements of a basic citation. Students therefore have a foundation of skills on which to build in the second semester organic chemistry course (the focus of this article). Finally, the librarian works with students near the end of their undergraduate degree program in a junior/senior chemistry capstone course, where the librarian teaches advanced search strategies in chemistry- (and other science) specific library databases as students pursue independent research.

The University provides students with access to SciFinder [26] and other scientific databases (Reaxys [27], Web of Science [28], etc.) as well as a number of chemistry-related journals. The University Library interlibrary loan staff provides support by obtaining articles and other sources of information not held by the University.

Experimental

Utilizing chemical information skills, students working in small teams of three or four develop a research question on the pathway from cyclohexene to adipic acid. Students engage in a research process that is critically reliant on scientific literature (Figure 2), which allows for student-faculty refinement cycles in the experimental planning, laboratory research, and presentation/writing stages.

This learning experience takes place over nine lab periods throughout the semester (see Table 1). The following is a detailed chronological outline of the laboratory.

Library Session 1. The chemical informationlearning experience is divided into two separate lab periods, both meeting in a computer classroom in the University Library. In

Library Session 1, the librarian gives a brief introduction to the Library web site as well as the online chemistry research guide [29]. The librarian introduces an assignment where the students are asked to perform two information-gathering exercises. In the first exercise the students use SciFinder (a chemistry-related database tool) to locate the CAS registry number and particular experimental properties for an assigned substance. Students then use the CAS registry number of their substance to find various spectral information from the Spectral Database for Organic Compounds (SDBS) [30]. In the second exercise students use SciFinder to identify and access two journal articles written by an assigned author. For many students this is likely the first time encountering the primary research literature in
the form of journal articles, so article format and structure are briefly discussed. In this exercise students gain basic information skills and are introduced to primary literature in chemistry, both of which provide a foundation for much richer information experiences in the weeks to follow.

Instructional Materials. A laboratory handout (see supplemental material) is provided to students the class period before Library Session 2. Students form research teams of three to four members. In the handout, students are provided a list and quantity of chemicals that will be available to them in the laboratory at the start of the experiment. The available chemical list is one of the most crucial elements of the successful implementation of this experiment and can be carefully chosen to balance the expertise of the faculty or TAs administering the lab and control the spectrum of possible research available to students.

The handout also contains a blank experimental plan form for students to complete and submit the class period before experimentation begins. This plan must be approved by the instructor and TA before proceeding. On this plan students are asked, above all, to identify a specific research question to drive their experimentation. Helping students understand the requirements of an interesting yet feasible, literature-supported research question is key to this experience. Also in the experimental plan, students are required to present a detailed overview of their proposed chemistry including: procedures, analytical plans, atom economy of each reaction, relevant safety information, total reaction costs, timeline, and all appropriate references. Student groups are required to perform 6 reactions over 5 laboratories, which could be varied to meet specific institutional needs.

Library Session 2. During the second library session, students use information skills to start developing an experimental plan. At the beginning of the session the librarian provides a brief review of the search features in SciFinder used in Library Session 1 as well as an overview of possible search strategies specific to their research project. The librarian then demonstrates how to use the library’s interlibrary loan services, as students often locate a key article only to realize the library does not have access to the journal. The librarian then leads a discussion on information pathways in science, comparing and contrasting searching for information on the open web with peer-reviewed scientific literature. This is followed by a discussion about the evaluation of sources in science.

The remainder of the lab period is devoted to students working in their groups to begin locating information needed for their experimental plan. A primary goal in this meeting is the development of a research question that implies proper use of the scientific method. The instructor provides feedback and coaching on the design of these questions. For example, some paraphrased student questions from the past year that were supported include: *What is the optimal pH of adipic acid synthesis? How many times can a phase-transfer catalyst be reused effectively in adipic acid synthesis? What properties of acidic ligands with the tungstate catalyst are most crucial? What is the ideal phase transfer catalyst to acidic additive ratio? What co-oxidant is more effective under a certain set of conditions? Which phase transfer agent is most effective in expediting the reaction?*

Table 1. Chronological laboratory outline and description

Lab (week of semester)	Class description
Library session 1 (1)	Learning chemical databases and exploring the literature
Library session 2 (8)	Developing a research question and experimental plan
Research meeting 1 (9)	5 min. group presentation and formal critique of plan
Lab session 1 (12)	Begin synthesis, focus on safety/planning/time management
Lab session 2 (12)	Problem-solving and refining research goals
Lab session 3 (13)	Continue synthesis thru Session 5, obtaining analytical results
Lab session 4 (13)	Outline scientific article and begin writing
Lab session 5 (14)	Finish lab work, instructor consultations of article draft
Research meeting 2 (15)	5 min. group presentation of research, refine article

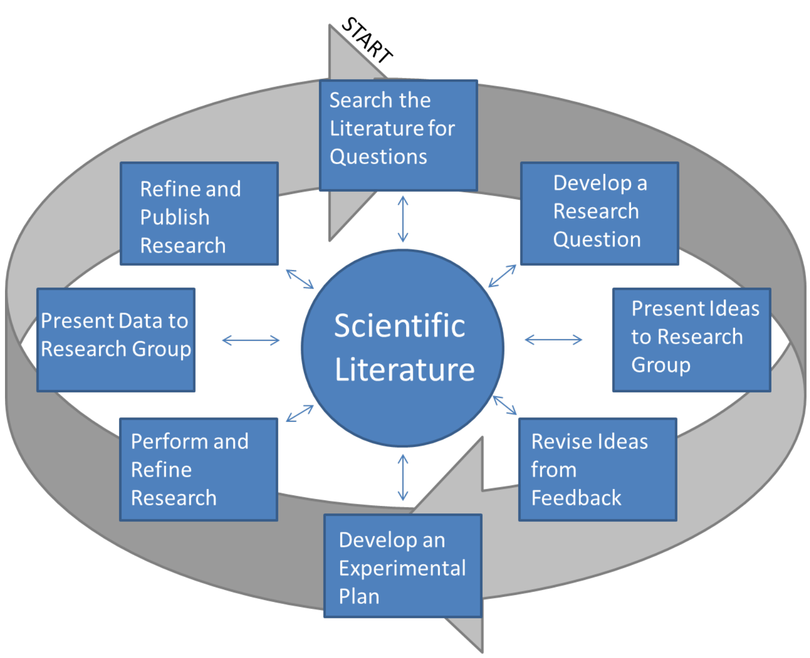

Figure 2. Research process guiding the student experience.

Other questions that required further student consideration include: Which oxidation procedure works best? Which of these reactions is the most efficient? What is the greenest reaction? Students are encouraged to identify a single independent variable, that can be changed without requiring alterations to other reaction conditions, and a dependent variable that clearly monitors the independent variable. The librarian acts primarily as a consultant to the student research groups, assisting them with specific point of need research assistance.

At this time, each research team receives an online data folder and a group email list, which allows them to store and share research and laboratory data, through the course management tool available at IUPUI. We have found this organizational and communication framework to be an essential piece of the experiment as it allows the instructor, librarian, and teaching assistants to observe the exchange of information between group members and provide timely feedback.

Research Meetings 1 and 2. In both of the class-wide research meetings, groups provide a 5 minute slide-based presentation related to their research. In the first meeting, they provide an overview of their research question, summaries of one or more relevant articles, a proposed synthetic pathway, and their motivation for pursuing the research. This meeting allows students a chance to improve their scientific communication skills, while also providing faculty, TAs, or other students a forum to offer helpful advice toward their experimental plan. Students and faculty use instructor-supplied forms to evaluate the research question and foundational literature. Formal summary critiques are returned to students by the instructor. Point values on these presentations were minimized to maintain a supportive and collaborative atmosphere. Students are allowed to request additional chemicals at this meeting. These requests may or may not be fulfilled depending on availability, safety, and sufficient literature evidence. This would only be recommended after developing a significant institutional knowledge of the involved chemistries. The finalized experimental plan document must be approved by the TA and instructor to begin research, and any future changes must also be approved.

In the second meeting, students present the findings of their research and scientific literature affected by the findings. Evaluator criteria include creating high-quality graphical/tabular representations, supporting a hypothesis using literature, and organizing results into coherent scientific arguments.

Laboratory Sessions. At the beginning of each lab day students are given important safety reminders on waste disposal, toxicity of reagents, danger/incompatibility of oxidizing agents, volatility and flammability, and proper equipment use (see Hazards section for more). Students are initially provided with the chemicals requested in their experimental plan. Expensive or limiting reagent materials are pre-massed (see suggested amounts in supplemental materials) into 20 mL labeled scintillation vials while solvents and low-cost chemicals are provided in the reagent fume hood.

Students are given guidance each day on planning, time management, effective analysis strategies, problem-solving, optimizing/refining research goals, and summarizing research for publication. In total, each group is required to carry out at least six reactions to obtain repeatable results from a small number of reaction variables. Students are instructed that the laboratory can be a multi-tasking environment where further literature searching, summarizing data, meeting with the group, or writing up results are perfectly acceptable tasks.

The instructor and TA take on very different "in-class" roles in this problem-based experiment than in a traditional lab. Therefore, unique training and preparation of TAs are needed (see supplemental material). Before the experiment begins, the instructor provides the TAs with a thorough collection of both online and peer-reviewed materials related to this research area. This information collection is then discussed with TAs and expanded based on the latest research cited in the students' experimental plans. TAs are instructed how to effectively read and critique experimental plans. Finally, weekly instructor and TA meetings are used to solve student issues, provide teaching and learning strategies, and maintain safety standards.

Student Journal-Style Article. Students are provided the Journal of Organic Chemistry (JOC) author guidelines [31] and an older word processor version of the document template for JOC Note submission. The instructor encourages students to use already published JOC Notes as guides for formatting, content breadth and depth, and use of scientific language. The instructor also provides an online lecture focused on translating research into an effective scientific article and offers consultations with groups regarding their article rough draft during Lab Session 5. The presentation of results is further optimized for "publication" during the Research Meeting 2. All group members utilize the entire group's combined research data to write their article. Students in each group are allowed to share the title, abstract, schemes/drawings and experimental section, but must write the introduction, discussion, and conclusion independently. Articles are submitted to the TA to be graded based on a detailed, subjective rubric (see supplemental materials) similar to what reviewers may informally follow. The papers are also submitted to the Turnitin online plagiarism detection service [32] to monitor plagiarism within the class or from other online sources. Although this plagiarism service does not search the entirety of the peer-reviewed literature, this provides a valuable tool to enforce ethical scientific writing practices.

Hazards. Problem-based labs are especially susceptible to safety hazards [12] as more freedom is given to students than traditional labs. Constant faculty oversight is needed in the planning and execution of these labs. In our experience, working within a narrowed research area, adipic acid synthesis, provides more manageable oversight than open research. We do provide constant warnings against several critical problems: 1) never heat closed systems, 2) never combine/heat reagents without a solvent, 3) observe care in the order of reagent addition, and 4) always closely monitor your reaction. General laboratory safety procedures were followed, which include wearing goggles and gloves, properly using a fume hood at all times, and wearing long pants and closed-toed shoes. All reagents provided to students were considered hazardous to some degree and contact with or inhalation of these chemicals should be avoided. Incompatibility of oxidizing agents with solvents or other chemicals was closely monitored by the faculty and teaching assistant using documents from the campus safety department. Metal waste was disposed of in a separate container from the organic waste for proper disposal. In any research setting it is critical for researchers to understand the safety risks of the materials they plan to use, so the student-derived experimental plan requires safety briefs on every chemical (prepared from the material safety data sheets, typically accessed through chemical manufacturer websites).

Results and Discussion

As mentioned above, most non-traditional labs require more time and input from faculty, and this experience was no different. However, students were asked to use much higher-level cognitive processes such as analysis, evaluation, problem-solving, creation, and teaching others. Therefore, we sought a research problem that could be scalable to any institution to minimize the negative aspects without sacrificing the benefits. This specific problem presented a relatively narrow and defined research area in which students developed expertise and shared ideas with one another. There were a few key factors identified in choosing this specific synthetic pathway that should be noted and potentially applied to other problem-based labs of this type.

Relevance. Students in the information age are very savvy at identifying when they are asked to solve a problem that is unimportant, "canned," or has been solved long ago. Therefore, we sought to involve a relevant problem that embodied the complexity of chemical problem solving and societal responsibility. The synthesis of adipic acid is one of the more important commercial processes in the modern world, yet it remains a significant source of pollution as discussed in the seminal green research in the area [17]. Research in this field has been expanding over the past 15 years and several new reports are published each year in related chemistries. Also, universities have successfully implemented the green synthesis of adipic acid in non-problem-based laboratories [33, 34].

Safety and Cost. A large portion of research is considered too expensive and unsafe to allow in a typical undergraduate laboratory. Therefore, the problem needed to center around somewhat "safe" chemistry (preferably done in aqueous, open-air environments) that ideally does not require extremely expensive materials [17–25]. The research surrounding adipic acid synthesis provides a reasonable example of these criteria. Though the cost of this experiment was scalable (see basic and expanded lists in Figure 3), it will no doubt be greater than traditional organic laboratories. However, the increased length of this experiment allowed up to three or four experiments to be replaced. For interested students, reagents to make 6,6-nylon were included in the basic reagent list.

Scalability and Growth Potential. If a problem-based lab does not have a potential for growth and change, then students will eventually develop an institutional knowledge of the experiment and may pass down their solutions to future students. However, the chemicals available to the students can routinely be adjusted to add or remove research possibilities. A wide variety of solvents, salts/buffers, oxidizing agents, metal catalysts, phase transfer catalysts, acids or bases, general reagents, and more could be provided to allow research questions on some of the topics listed in Figure 1. After several semesters of experience (2010-current, including summer, 1000+ students total), not all "available" chemicals were put on the student list to encourage students to think outside of the box and potentially request unlisted materials. In student hands, significant issues with the isolation of adipic acid occurred when performing reactions on a scale less than 2 grams, as initially suggested [34].

Evaluation. To assess the effectiveness of this laboratory, each of our initial objectives were considered with student output or feedback to determine if the experiment/students were meeting these objectives.

Objective 1. Integrating chemical information skills into a problem-based, research laboratory experiment.

Evaluation Measures and Outcomes. Student assignments from Library Session 1 and the experimental plan were considered for this objective. Students had very little difficulty successfully performing data retrieval exercises in Library Session 1. However, the most effective indicator of students' ability to integrate chemical information skills into a research laboratory was their ability to develop an acceptable literature-based research question to guide their experimental plan. Nearly every group required a significant faculty or TA intervention to their research question prior to submitting the final experimental plan version. Common interventions included completely redesigning the research question, selecting more appropriate reaction variables from the literature to answer the question, and identifying more effective information sources to guide research design. It was the authors' experience from informal interactions that students were not initially confident in using chemical information skills in a research laboratory. After two student-faculty feedback cycles in Research Meeting 1 and experimental plan corrections, all groups were able to successfully design an acceptable research experiment.

Objective 2. Increasing scientific research ability for chemistry students.

Evaluation Measures and Outcomes. Students were given feedback or evaluated at each step of the research process (assignment in parentheses): developing a well-designed and literature-supported plan (Research Meeting 1 presentation, experimental plan), carrying out the research in a detailed and well-organized manner (laboratory notebook), and summarizing and presenting their research (Research Meeting 2 presentation, mock Journal of Organic Chemistry Note). Grading criteria for these student-generated materials can be found in the supplemental material. When analyzing these materials, it was observed by the authors that most students adequately progressed through the research cycle (Figure 2). To investigate this further, a survey specific to this experience

Table 2. Student's self-reported preparedness for completing another research experiment (5 = very prepared and 1 = very unprepared)

Lab (week of semester)	Average	Standard Deviation
Handout received (7)	3.7	1.2
Library session 2 (8)	3.7	1.0
Research meeting 1 (9)	4.0	0.7
Lab session 2 (12)	4.1	0.9
Lab session 5 (14)	4.4	0.8
Research meeting 2 (15)	4.4	0.7

Basic reagent list (no solvents)	Expanded reagent list
sodium tungstate dihydrate	potassium permanganate
Aliquat 336	Celite
30% hydrogen peroxide	sodium periodate
potasium bisulfate	ruthenium chloride
sodium sulfate	Oxone
cyclohexene	pyridine
thionyl chloride	dil. sulfuric acid
60% w/v hexamethylenediamine	benzoic acid
sodium hydroxide	salicylic acid
dilute hydrochloric acid	dil. acetic acid
	sodium lauryl sulfate
	benzyltriphenylphosphonium chloride
	sodium acetate
	dichloromethane, hexanes, dimethylformamide

Figure 3. Potential reagent lists made available to students.

revealed student's self-reported "preparedness" (n = 35) to complete another research experiment at various points throughout the lab (Table 2). Students answered on a Likert scale where 5 is very prepared and 1 is very unprepared. Upon receiving the handout for the assignment, students reported a wide range of preparedness reflecting a diverse student population. Library Session 2, their first group interaction on the problem, yielded little increase in perceived preparedness. However, as student-faculty feedback cycles occurred, significant gains and reduced standard deviations were observed. This showed the importance of explicitly involving students in a mentored research process. The student-generated mock literature articles continually showed a strong grasp of the research process and an extensive use of scientific literature to inform their work. These mock articles were scored using a detailed set of criteria for each major section of the article (Title Information and Abstract, Introduction, Results/Discussion, Conclusions, Experimental, References and Scientific Writing). On a previous iteration of this lab's survey, students agreed (4.1 ± 0.6 out of 5 on a Likert scale, n = 73) to the statement, "You were adequately prepared to complete a research article." In terms of experimental success, students reported obtaining adipic acid on their 1st or 2nd attempt (average 1.6th attempt), even with a wide range of research questions.

Objective 3. Bolstering student perceptions of research and laboratory work.

Evaluation Measures and Outcomes. Two surveys were typically administered to students, one was specifically designed for this lab experience (multiple semesters cited above) and the other was an end-of-the-semester lab survey to capture general student perceptions of the entire course. Student response to this experiment was mostly positive, despite the significant amount of time and work required. Students agreed/strongly agreed (4.6 ± 0.6 out of 5 on a Likert scale, n = 35) with the statement, "This experiment better prepared you to understand and apply chemical research than a normal or traditional lab from our lab textbook." The end of the semester survey in an earlier semester showed that 49% of students (n = 85) found this to be their favorite lab experience of the semester, even amongst other non-traditional labs and among much less time-intensive labs.

Conclusion

In this lab experience students were given a relevant, real-world problem to solve with numerous potential research pathways available. By mirroring this experiment to actual synthetic research, students were provided with a more authentic experience of both the process of research and the role that information skills play in scientific discovery. Though the nature of the assignment required more involvement and less control for the instructor and librarian, the quality of student research output and student perceptions convinced the authors of the value of this laboratory.

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