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Laboratory Experiment

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Design Your Own Workup: A Guided-Inquiry Experiment for Introductory Organic Laboratory Courses

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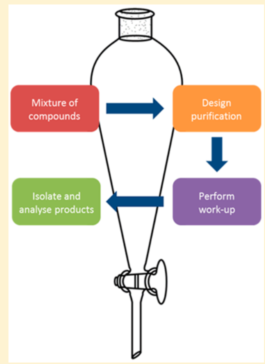
Ⓢ Supporting Information

ABSTRACT: A guided-inquiry experiment was designed and implemented in an introductory organic chemistry laboratory course. Students were given a mixture of compounds and had to isolate two of the components by designing a viable workup procedure using liquid−liquid separation methods. Students were given the opportunity to apply their knowledge of chemical and physical properties of organic molecules as well as develop their problem-solving skills by designing the workup themselves rather than following a pre-existing recipe. Students were able to show that this experiment improved their problem-solving skills and understanding of chemical concepts related to the workup process.



KEYWORDS: *First-Year Undergraduate/General, Laboratory Instruction, Organic Chemistry, Inquiry-Based/Discovery Learning, Problem Solving/Decision Making, Molecular Properties/Structure, Noncovalent Interactions, Separation Science*

INTRODUCTION

Laboratory teaching has played a central role in science education over the years[1−4] and has the potential to "enrich the formation of science concepts by fostering inquiry, intellectual development, problem-solving skills, and manipulative skills."[4] To develop these skills, attention must be paid to the type of experiments that students are asked to perform.[3,4]

Recipe-based "cookbook" experiments are highly prescriptive in their nature and have been shown to only develop lower-order practical skills.[3,4] In the context of organic chemistry experiments, this includes how to perform synthetic techniques and assign spectral data to compounds with a known chemical structure. Engaging students in active learning strategies is more likely to result in higher-order learning, including metacognition[5] which has been linked to problem-solving ability.[6,7]

To ensure that students have the opportunity to develop higher-order skills, research based mini-projects were introduced into the third-year laboratory course.[1,2,8] Experiments of this nature provide students with the opportunity to solve problems, apply concepts, formulate hypotheses and allow metacognition.[9−18] Students welcomed the introduction of these projects, recognizing higher-order skills were being developed through the projects; however, some students found a jump in difficulty from two prior years of recipe-based experiments. This experience has also been observed elsewhere.[14]

In our aim to improve our student's ability to perform inquiry-based experiments and develop their higher-order cognitive skills, guided-inquiry experiments have been introduced into the first two years of the synthetic chemistry laboratory courses. Herein a guided-inquiry experiment that requires students to apply their knowledge of chemical structure by designing their own workup procedures, and develops their problem-solving skills when performing the purification is described.

THE WORKUP

The workup by liquid−liquid extraction is a fundamental and frequently used method to isolate and purify mixtures at the end of an organic reaction.[18−20] A recent natural product synthesis was chosen to show how often a workup is used in the synthesis of an organic molecule (Figure 1).[21] In the 26 step synthesis of Paecilomycin B, 22 steps require a liquid−liquid extraction as part of the procedure.

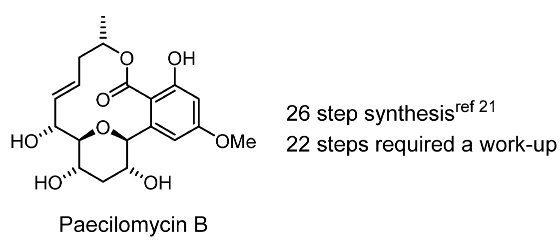


Figure 1. Importance of workups toward the total synthesis of Paecilomycin B.[21]

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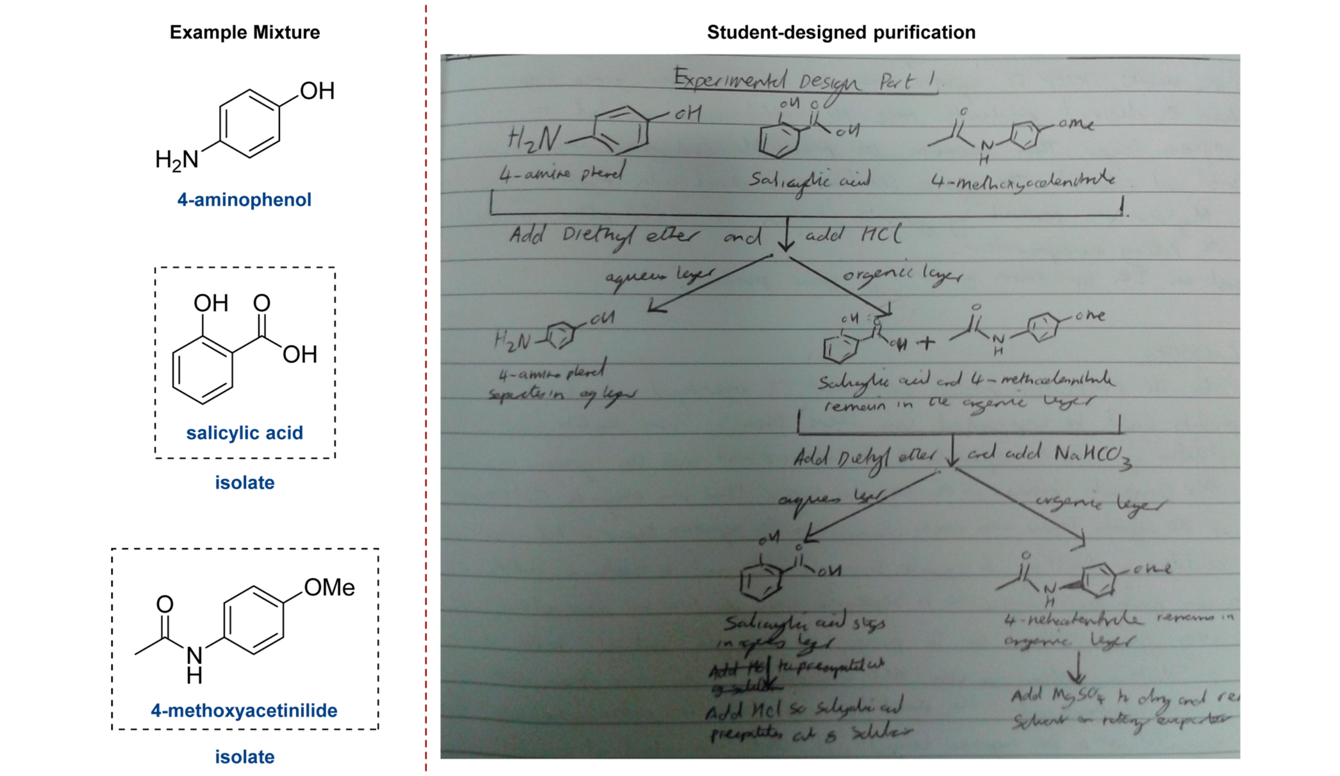

Figure 2. Example of a mixture with the two compounds that students had to purify and a possible workup of the same mixture designed by a student.

Due to its importance as a technique, students are taught how to perform a liquid–liquid extraction at the earliest opportunity in many practical courses and are given the opportunity to practice this skill in multiple experiments. The workup draws upon the application of fundamental physical and chemical principles such as states of matter, bonding, solubility, and acid/base theory taught in high school and early undergraduate chemistry courses. Because of the early introduction of the practical procedure and theoretical concepts, it was reasoned that asking students to design their own purifications of a mixture by liquid–liquid extraction would be a suitable guided-inquiry experiment in the first year of the laboratory course. It was expected that this experiment would also lead to an improvement in students' understanding of the concepts related to the workup process.

■ COURSE STRUCTURE AND EXPERIMENTAL DESIGN

The experiment was introduced to a first-year undergraduate laboratory course for Chemistry and Medicinal Chemistry majors in a research-intensive university in the U.K. Typically, 135 students are enrolled in a 30 credit module run over the course of 2 semesters with 9 h a week timetabled for this module. The students are split into three cohorts of approximately 45 students and rotate between organic, inorganic, and physical chemistry experiments every 3 weeks in each semester, while in the organic and inorganic rotations, students are split further into three subcohorts of approximately 15 students, and are timetabled to perform one experiment a week. This "design your own workup" experiment was part of a 9 h practical with a purification theme, where students were also asked to find their own solvent system to recrystallize a given compound. Students learn how to perform a liquid–liquid separation and are also taught the corresponding theory in semester 1, so the experiment was performed in semester 2. In theory at least, all students had the requisite knowledge and would be able to focus on the inquiry/problem-solving aspect of the experiment. Within the rotation system, there were nine different groups of 15 students performing this experiment each week over the course of semester 2.

Each group performing the experiment was given a different mixture to purify (see Supporting Information for full experimental details, including the list of different mixtures). Each mixture contained three compounds, from which the students had to isolate two of the components (see Figure 2 for an example). The compounds were chosen to contain acidic and basic functional groups with pK_a values and characteristic infrared absorption bands with which students were familiar. The combinations of these compounds were also designed so that students were required to isolate at least one of the components by an acidic or basic extraction. This meant students would have to apply their understanding of structure, solubility, bonding, acid/base theory, and pK_a to design a successful workup procedure. Each mixture was subjected to a trial purification to ensure it was feasible to purify the desired compounds. On rare occasions some mixtures proved problematic, so they were altered and tested again. However, the opened-ended nature of the experiment meant that it was possible for students to design a viable purification that had not been trialed by ourselves.

Students were informed of which mixture they were purifying in advance to starting the experiment (Figure 3). Prior to starting the experiment, students had to perform a prelaboratory multiple-choice quiz worth 10% of the overall grade for the experiment. The questions in the prelaboratory exercise tested the students' knowledge of pK_a values and how a liquid–

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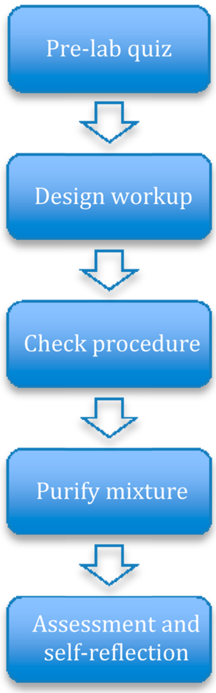

Figure 3. Structure of the experiment.

liquid extraction works (the quiz is available in the Supporting Information). By completing the quiz, an awareness of the conceptual understanding required to design a workup procedure was provided to the students. When students were scheduled to start their purification, they were expected to have designed a procedure in the form of a flowchart (Figure 2). These were presented to a demonstrator (teaching associate) who would look for any obvious errors in the procedure and highlight them for the student to amend. Once the procedure had been verified, a sample of the mixture was given to the students to implement their purification. The success of the experiment and the students understanding were measured by assessing the quality of the sample and a laboratory writeup. Purity was the most important criterion when assessing the quality of the samples. These were assessed by their sample's appearance and quality of the infrared spectrum. The laboratory writeup included the student's pre-experiment flowchart, a procedure of the workup their used written as a standard experimental procedure, analysis and interpretation of spectra, a discussion of their experiment, and a conclusion to enable self-reflection.

■ HAZARDS

This experiment should be undertaken in a fume hood, and students should wear all standard personal protective equipment in the laboratory (lab coat, safety glasses, and gloves). Dichloromethane is toxic, a suspected carcinogen, and a skin, eye, and lung irritant. Diethyl ether and ethyl acetate are highly flammable and should not be used near any naked flames. Sodium hydroxide (2 M aqueous solution) and hydrochloric acid (2 M aqueous solution) are caustic and corrosive. All solvents should be transferred to appropriate waste containers after use. Hazards for the chemicals used in the mixtures are given in the Supporting Information.

■ DISCUSSION

The experiment was performed in the 2014/15 academic year. Ninety-eight percent of students completed the prelaboratory exercise. A high average score and small standard deviation for this part of the experiment shows that students were able to understand the concepts relating to the workup process (Table 1). Approximately 90–95% of students were able design a flowchart that would have separated the compounds and show good understanding of how the mixture would be purified. For the 5–10% of students that struggled to design a purification procedure, assistance was given by the demonstrators.

Table 1. Student Results the Experiment (2014/15) (N = 135)

Section	Average Grade	Standard Deviation
prelab score (/10)	7.8	2.0
workup flowchart (/10)	8.4	1.9
overall grade (/100)[a]	73.8	13.4

[a]The overall grade shown has been adjusted to exclude marks awarded from another section of the experiment incorporating a recrystallization component.

During the experiment, students solved typical problems encountered with this part of an experiment. Many students were unsure how much solvent to use but were able to solve this by simply adding more solvent. Poor separation between layers was another commonly encountered problem which was often solved by the addition of brine. Students were allowed to problem-solve in a collaborative manner and many did so. It was pleasing that in their discussion, students were demonstrating their understanding of the purification process. This level of understanding was markedly superior to previous students who had only performed workups by following a recipe. As the students progressed through the separations, many gained confidence and were able to work more independently. All students completed this exercise within two 3 h laboratory sessions, although many only required one. Any traces of the other two components could be easily determined by infrared spectroscopy, so this was the method used to determine the purity of their samples. Some samples were liquids, so we chose not to include melting point determinations as part of the assessment criteria. The average final grade for the workup section of the experiment (i.e., excluding the recrystallization component) was 73.8%. Pleasingly, this was similar to the overall grades achieved from other experiments in the course.

Student feedback forms were used to evaluate students' understanding of the workup process and their problem-solving ability (N = 58/135). A 5-point Likert scale questionnaire was completed with space to provide comments (Figure 4).

Many students agreed that this experiment improved their understanding workup process and these concepts.

Whilst designing the workup was challenging, it did increase my knowledge of pK_a's...

My favourite experiment so far as it greatly increased my understanding

Students were in agreement that this experiment required them to solve problems and their problem-solving ability improved as a result of this experiment. The strong agreement with these statements helps to justify the value of skills development that a more open-ended experiment can provide.

As one of the aims of this experiment was to build student confidence with more open-ended experiments, students were

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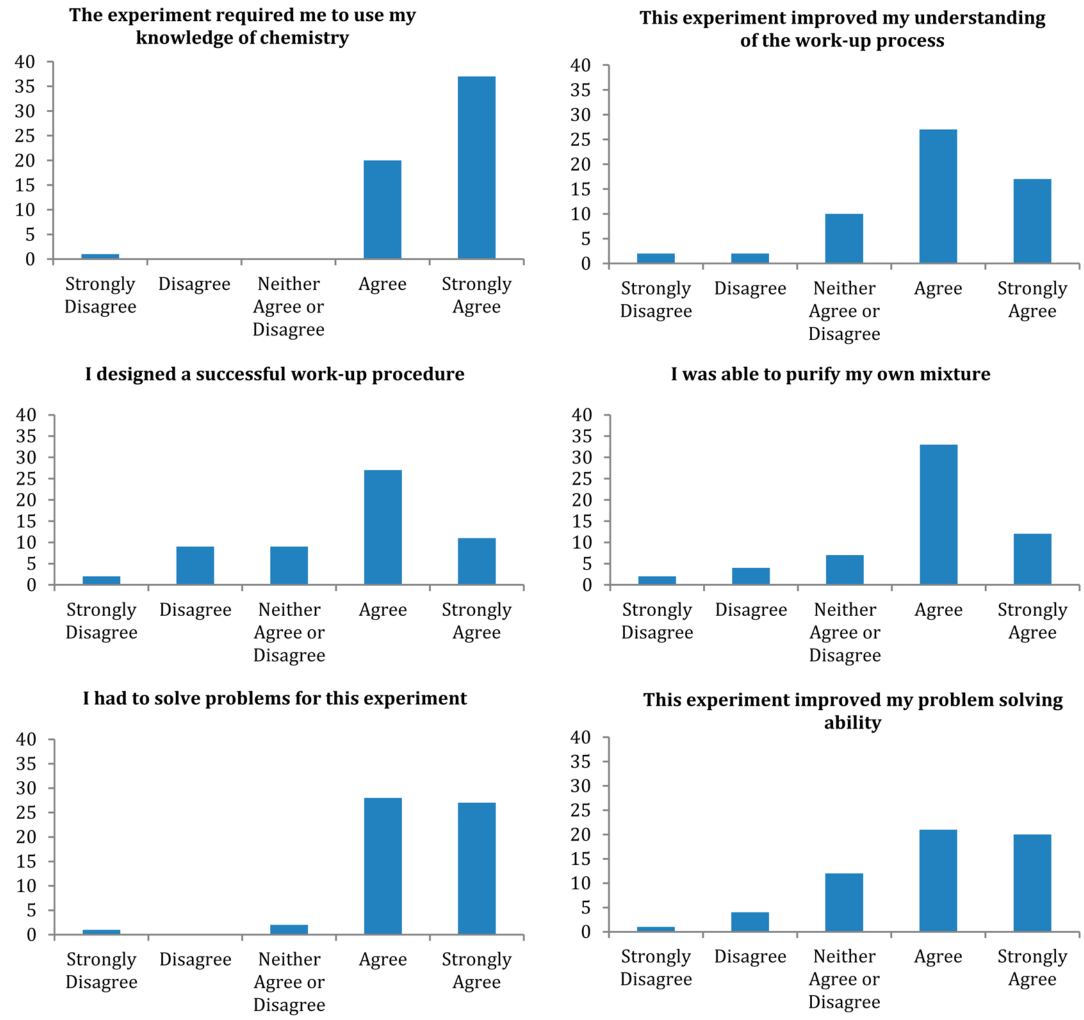

Figure 4. Student feedback on postexperimental learning gains (N = 58/135). Y-axis indicates number of respondents.

asked if they preferred this type of experiment to the recipe-based experiments to which they were more accustomed. Students responses were mixed, but its value was recognized even by those who preferred the recipe-based experiments.

> *Good experiment to think for ourselves. However, I prefer the usual experiments as I enjoy following instructions.*
>
> *I found this very useful*
>
> *I prefer the style of this experiment*

As students progress through the course, they will be required to perform more inquiry-based experiments. We shall continue to ask this group of students of their perception to inquiry-based experiments with the hope that increased exposure will also increase their confidence.

CONCLUSION

In summary, an introductory guided-inquiry experiment that requires students to apply their knowledge and understanding to design a purification procedure was developed. Students were able to design and perform their own workups. By doing so, students met the learning objectives of the experiment. The low cost and ease of creating different mixtures for students to perform make this experiment one that is easy to implement in teaching laboratories while having learning gains that could not be achieved in a recipe-based experiment.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00691.

Full experimental procedures, including details of the different mixtures used (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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