Fostering Creativity in Students

A Short Synthesis Project for the Organic Chemistry Laboratory

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A short, two step synthesis project for students in organic chemistry provides practical experience in scale up, purification, isolation, and modification of synthetic procedures.

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Introduction

What would your reaction be if your organic lab instructor asked you to work with a classmate, execute a two-step synthesis of your own devised over the course of five weeks and hand in 0.100g of the final product, completely characterised, as proof of your success? How would you respond if you were also asked to write a short proposal describing the synthesis before you attempt it, and later to submit a final report, complete with appropriate spectral characterization of the product? This sounds like, and is, a challenging and perhaps daunting task at first. Yet, if the aim of your organic class is to teach you how to think as an organic chemist, then this exercise (although still a little overwhelming) is a reasonable test of your organic chemistry skills. After nearly a year of organic chemistry lecture and lab, this mini-project pulls together many skills your instructors have been trying to teach: how to think, write, and apply organic reactions. We have been doing this mini-synthesis project for two years at Grinnell College with success, and many students say this it is the best lab of their organic chemistry experience.

Many lab 'experiments' in chemistry courses are cut and dry exercises with known outcomes and focus on teaching techniques like distillation or crystallization rather than on exploring open-ended questions. As a result, many students leave the lab having

Students generally take organic chemistry in their second year of college in the US, after a year of general chemistry. Our course enrolls chemistry majors and non-majors alike.

learned little about how chemists really apply the organic reactions taught in class. Exercises with known outcomes reinforce the notion that organic chemistry is not a creative endeavour and that rote learning and memorization are the keys to success in the subject. It is not unusual for a student who has completed the organic chemistry course to be unable to decide how to separate a mixture of compounds or how to modify an experimental procedure (for example, to change scale or to handle a solid rather than a liquid).

In addition, most science students are challenged by writing in the specialized vocabulary of chemistry. Students write in lab notebooks and hand in lab reports, but because the outcome of the labs is known, it is difficult to write a report that poses and answers a novel, scientific question. Our two-step synthesis project teaches three important skills: thinking like a scientist, applying chemical knowledge in a practical manner in the lab, and writing like a scientist. With these as our primary goals, we have established guidelines to make the project workable within our constraints of time and money.

The Creative Work

The challenge is this: with a lab partner, propose a two step synthetic route, given a list of commonly available, inexpensive starting materials, a number of possible reaction types, and some suggested products. The list of possible targets limits the combinations of steps you could consider, but adventurous students may suggest products that are not on the list. You will have eight weeks altogether to work on the project: three weeks outside of lab, doing literature research to find procedures that could be used for your route, and then five weeks in the lab to actually do the synthesis and obtain 100 mg of product.

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This task may seem straightforward at first, but then you have to consider the constraints of the lab. Time is a significant factor. We require that experiments be done in a regularly scheduled lab

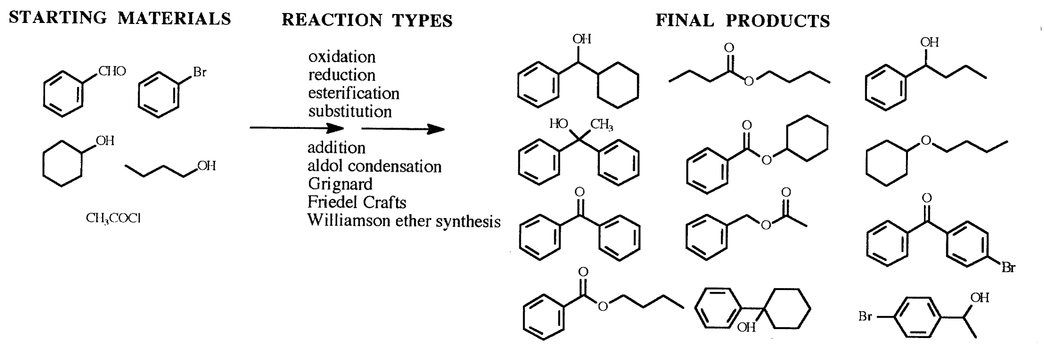

Figure 1 The two-step synthesis project.

period of 3 hours, for reasons of safety. The target molecule probably has not been reported in the literature, so you will have to modify procedures for similar compounds, including changing the scale. You will also have to decide the best way to purify the target so that it can be characterized.

A good way to tackle the problem is to look at the starting material list and to work out possible routes on paper. Then conduct your literature search. Are you interested in oxidizing cyclohexanol? Find a procedure for it. Can it be done in the 3-hour limit? What's the scale? How will you know that the reaction is complete? Maybe you need to find other procedures to see if they are more applicable to your starting material. Some good library resources include Chemical Abstracts Service and *cookbook* references such as the collective volumes of *Organic Syntheses*, and *Vogel's Textbook of Practical Organic Chemistry* that contain tested synthetic procedures, or Larock's *Comprehensive Organic Transformations* which is a leading reference to synthetic procedures. In addition, lab texts are great resources, providing more procedures that can easily be done in a typical lab period. It helps to work with a partner, because it is not unusual to have a collaborator in research, and because it always helps to have someone to bounce off ideas. All of the planning culminates in a 3-5 page proposal which is submitted about 10 days before the lab work is to begin. This timing gives the instructor an opportunity to comment on anticipated difficulties, as well as time to order

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any necessary materials.

You will continue to work with your partner to carry out the synthesis over the course of five weeks. Five weeks would seem to be plenty of time, but many problems occur that are familiar to experienced chemists. For example, the yield of your first step may be too low to carry material on to the next step; the procedure may not be appropriate for the particular compound; or the product's properties may be different from what was anticipated. The five-week period allows time to repeat reactions and to attempt alternative procedures. For example, one student can try new reaction conditions while the other makes more of the product of the first reaction. As you work, you will come to understand why the literature contains so many different methods to accomplish the same transformation!

Have You Been Successful?

[1]The use of the program PCNMR to process FIDs from high field instruments has been described in Swartz J, Vojta G M and Erickson L E. *J.Chem. Ed.* Vol.71. p.1069,1994.

The products of both the first and second steps must be characterized in some way to confirm that they are indeed new and different from the starting materials. For both compounds, you will obtain melting or boiling point data as well as the IR spectra. At Grinnell, for the final product only, students also submit a 50mg. sample for ^{1}H NMR which is run for them. They are then given a floppy disk with the FID (free induction decay) of their material, and they can process the FID outside of the lab.

The Written Work

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Another goal of the synthesis project is to familiarize you with the proper formats of a proposal, progress report, and final report. In many lab situations, you may have difficulty writing reports centered on a scientific question when the *experiment* is a technique rather than a real question. In this project, you are investigating a real question (how can you make this material efficiently and in good yield?) and you should find it easier to write a report with objectives, experimental procedure, results, discussion and conclusion.

Box 1.

Details of the Synthesis Project Proposal

1. *Project Summary or Abstract (150 words or less)*

Includes a statement of the research objectives, the scientific methods to be employed and the significance of the proposed research to the advancement of scientific knowledge.

2. *Project Description*

(a) Introduction

Establishes the background and context for your project proposal, slightly expanded from the Abstract.

(b) Experimental Design. (approx. 3- 5 pages)

Details your specific objectives and your plan for accomplishing the objectives. Describe your synthetic route and how you will determine if you were successful (spectroscopic characterization or comparison to known material?)

(c) Pilot Data.

Includes any pilot results or precedents from the literature that demonstrate that the project is feasible and that you have thought through potential difficulties, including the isolation and identification of your products.

(d) Research Schedule.

Set forth your precise time schedule for the research, in this case, five weeks. Make a timeline that indicates what parts of the project you will do in the various weeks.

(e) Summary.

Summarizes the proposal in one paragraph and makes the case for supporting the proposed research.

3. *References*

Includes any references that helped you to establish the feasibility of your project.

4. *Materials and Supplies.*

Indicates the types of required expendable materials and supplies. Budget limitations may restrict your choice of reagents if they are not on the instructor's list.

The first document you submit is a brief, 3-5 page proposal that describes the planned synthesis. The proposal must include these features: a project summary, a detailed project description, a list of references, and a list of materials and supplies needed. The contents of these sections are detailed in *Box 1*. The aim of a proposal is to convince your reader that you know what you want to do and how you will determine if you were successful, so it is important that you cite literature to assure your reader that your route (although novel) has a high probability of success. The research schedule should also convince your reader that you have allowed time for the unexpected events that make chemistry so interesting.

Once the lab work has begun, one-paragraph progress reports are handed in after each lab period. These brief reports summarize the day's work, and describe your plans for the next lab period. The progress reports are intended to encourage you to evaluate results and to plan for the lab period to come.

The final report contains a brief, one-paragraph summary of the aim of the project as well as the most significant observations and conclusions. The body of the report contains headings similar to those of papers in the literature.

One week after the project is completed, your team will submit a final, 3-5 page report to simulate the sort of report that supervisors and funding agencies require as assurance that their money was not squandered. The final report contains a brief, one-paragraph summary of the aim of the project as well as the most significant observations and conclusions. The body of the report contains headings similar to those of papers in the literature: introduction, experimental design and methods, results and discussion, and conclusions. The report must be fully referenced, using the format for references according to the Guidelines for Authors from *Journal of Organic Chemistry,* and abbreviations recommended by *The ACS (American Chemical Society) Style Guide*.

Lastly, appendices are attached of the spectra (IR, NMR) of the intermediate and final product.

Chemistry students who wish to go in for a research career or an industrial job must be creative, as they will often have to devise solutions to challenging problems. The typical laboratory practical experiments, however, provide little scope for the students to think and contribute to their laboratory training. They are invariably made to undergo old fashioned, routine exercises and provide a report, again in a *standard* manner. In this article, Mader and Liberko describe an approach they have taken for encouraging the creativity of the second semester organic chemistry students in Grinnell College, Iowa, USA. We feel that this general approach, with some minor modifications, can be adapted at the 3rd year BSc or 1st year MSc levels of our institutions. Even though many undergraduate colleges may not have adequate infrastructural facilities, creative thinking can actually help one get over such problems to do new and meaningful experiments.

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Our Experience With The Project

The synthesis project has been done by organic chemistry students for two years at Grinnell College with a good deal of success. All of the students must write a final report, regardless of whether or not their synthesis was completed. About half of the students make their target molecule and hand in the required amount of final product. Most often, the successful students do not have everything working as they had planned originally, but they realize that their division of labour is important for making progress, and they are willing to investigate other procedures. Even students who are unable to complete the synthesis learn a tremendous amount simply by coming to recognize that organic chemistry does require creativity, insight, and persistence. Only a small part of the project grade depends on actually making the target molecule because that is only a small measure of whether the students meet our real goal: learning to think as organic chemists.

Suggested Reading

- **Amenta D S and Mosbo J A. Attracting The New Generation of Chemistry Majors to Synthetic Chemistry without Using Pheromones. *J. Chem. Ed.* Vol. 71, pp. 661-664, 1994.**

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