

Academic Research Inspired Design of an Expository Organic Chemistry Lab Course

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(Received December 22, 2017; Accepted February 6, 2018)

ABSTRACT. In this paper, we present fortified instructional methods that contributed in improving students' interest toward the expository organic chemistry laboratory course. Reformed TA (Teaching assistant) training and allocation method, a thorough course orientation session, text-light/graphics-heavy results PPT reports, and journal article templated-term papers have improved students' satisfaction in the organic chemistry laboratory course. These methods could be implemented while maintaining the traditional organic chemistry laboratory instruction styles and hence could be broadly applicable.

Key words: Chemistry, Organic chemistry laboratory course instruction methods, Expository instruction style

INTRODUCTION

The organic chemistry laboratory course is an essential component of undergraduate organic chemistry education. While most research-oriented institutions like Korea Advanced Institute of Science and Technology (KAIST) provide experimental research opportunities for undergraduate students via programs such as URP¹ (Undergraduate Research Participation Program), the research opportunity quota still cannot match the students' needs.² Hence, well-designed organic chemistry laboratory courses behave as important channels to expose students to experimental aspects of organic chemistry as a means to reinforce the theoretical principles and spark the interest toward the subject. Besides the traditional expository instruction method in which students follow directions from a manual to obtain predetermined outcomes, chemistry instructors have developed various lab teaching styles that are based on inquiry-, discovery-, or problem-based instruction methods.^{3,4} However, expository instruction style with regard to the minimization of resources such as time, space, equipment, and personnel renders it the most prevalent instruction method.³

Past years' students' surveys on organic chemistry laboratory course at KAIST have revealed their frustration with the workload of the course. They had submitted a pre-lab note and a multi-page post-lab report on a weekly basis. The main problem associated with students' dissatisfaction was the notion that newly presented course materials and course credits (2 credits)⁵ were not reflecting the efforts and time they had invested for the course. It seemed

that students couldn't bridge the course materials and newly acquired skillsets to their future needs as a researcher.⁶ We recognized that in order to enhance student's learning experience with expository lab courses, the course environment should mimic closely to a research environment. We were cognizant that knowledge cannot be transferred intact to the students and should be constructed in the mind of the learner.⁷ Therefore, we restructured the entire course and the evaluation criteria in a way that can emulate the actual organic chemistry research practices. The orientation session led by faculty members provided students with compulsory information applicable in organic chemical research. The formats of pre- and post-lab reports and term papers have been changed accordingly. We redesigned the organic chemistry laboratory course to be a preparative session to introduce the entirety of an actual organic chemical research and not merely an exhibition of learning sessions of limited experimental skills. Herein, we describe the detailed instructional methods that have greatly improved students' satisfaction in an expository lab course setting.

COURSE PREPARATION

Design of the Contents

2017 Spring semester CH352 (Chemistry Major Lab II or Organic Chemistry Laboratory) was designed as a nine-week course. An Orientation session was held on the first week. Eight experiments were expected to be completed within a six-hour session per week. Six experiments were conducted based on contents published in the *Journal of*

Chemical Education each with modifications from the original paper. One experiment was designed based on the research by Larrow and Jacobsen.⁸ Below is the list of experiments.

- Menthol Purification Thin-Layer Chromatography and Column Chromatography⁹
- Menthol Esterification: Setting Up a Reaction and Conducting Work-up⁹
- Diels–Alder Reaction¹⁰
- Reductive Amination via a Solid-Solid Reaction and Acetylation¹¹
- Optical Resolution of 1,2-Diaminocyclohexane Biginelli Reaction¹²
- Nucleophilic Addition to Carbonyl: Grignard Reaction with a Ketone
- Converting Aniline to Brominated Acetanilide: A Multistep Synthesis¹³

The course contents development was aided by two undergraduate researchers who performed senior projects in our laboratory. Experiments to be included in the organic chemistry laboratory course were then chosen after a discussion among the course instructors and the undergraduate researchers. They ran the experiments following the literature procedures and made proper modifications accordingly. As a part of their undergraduate thesis project, the lab manual was generated under the supervision of the instructor.

Restructured Teaching Assistant (TA) System

Six TAs were assigned to forty-six students enrolled in the course. TAs were selected among graduate students conducting research in organic chemistry laboratories. Previously at KAIST, each TA supervised two experiments. They were responsible for one experiment as a primary TA and one as a supportive TA. Hence, students rotated and were assisted by different TAs for each experiment.

As a means to maximize the TAs-students intellectual bonding, new TA-student mentorship has been implemented. A team of two TAs supervised 14–16 students for all eight experiments during the 2017 CH352 course. A two-day TA bootcamp was held prior to the 2017 spring semester. Every TA conducted all eight experiments using the undergraduate laboratory glassware and equipment. Modifications of experimental procedures and orders of additional necessary chemicals, glassware, and equipment were made accordingly based on the feedback of the TA bootcamp. Lab manuals were then updated accordingly.

ORIENTATION SESSION

The orientation session was held on the first week of the course. Unlike previous years in which TAs were giving introductory comments, a faculty member in the chemistry department who designed the lab course led the orientation discussion. The following items were discussed during the orientation session.

Specific Aims

The clear objective of the course was introduced to the students.

“This organic chemistry laboratory course is intended to provide students majoring in chemistry with fundamental and essential *experimental skills* and *concepts* in organic chemistry.”

The term “experimental skills” has been delineated as follows.

- Recognizing potential safety hazards
- Conducting experiments safely and efficiently

The term “concepts” has been delineated as follows.

- Understanding the mechanism of the reactions
- Drawing organic structures using a computer
- Searching the organic chemistry literature
- Writing a scientific report

After introducing the goals of the course, the discussion was transitioned to the safety in the laboratory.

Safety

Eighteen safety-related rules were introduced to students.¹⁴ Below are some of the outlined rules.

- Wear approved eye protection at all times while in the laboratory.
- Wear shoes at all times. No open toe shoes are allowed in the lab.
- Eating, drinking, and smoking are strictly prohibited in the laboratory at all times.
- Know where to find and how to use all safety and first-aid equipment.
- Consider all chemicals to be hazardous unless you are instructed otherwise. *Dispose of chemicals as directed by your TA.*

The instructor used personal lab experience stories as a means to draw students’ attention to the safety issues. For example, one faculty member instructed students from his

own experience and observations that accidental chemical splashes have occurred frequently to a researcher working in a lab. He emphasized that, therefore, laboratory researcher should wear eye protection gear at all times while working in a lab. The engagement of instructor's personal stories was not only effective in drawing students' attention but providing them incentives to strictly abide with laboratory safety rules.

A Material Safety Data Sheet (MSDS) was introduced to students. An MSDS provides information about proper storage of a substance, first aid, spill response, safe disposal, toxicity, flammability, and additional useful materials. The MSDS searching method was explained to students. The instructor informed the students that thorough reading of the MSDS of chemicals prior to the experiment is mandatory and was enforced through the hand-written pre-lab note.

TA Introductions

During the orientation session, the name, the affiliated research group, and the research interests (including a recently published paper) of each TA was provided by the instructor. Following a general introduction of the TAs, each TA was also asked to come to the stage to introduce themselves. This process helped the students familiarize themselves with the TAs. Furthermore, the introduction of each TA's research interests and recent research achievements led the students to acknowledge them as organic chemistry experts and helped to generate a sense of reverence towards the TA team.

Grading Policy

The grading policy of the course was delineated to the students. Below is the grading policy of 2017 CH352 course. Detailed explanation of each item will be discussed in the latter sections.

Pre-lab notes	25%
Experiments and results PPT files	25%
Mid-term paper	25%
Final paper	25%

Introduction to Chemistry Journals

Publishing research achievements in scientific journals is an indispensable process in scientific research. We designed the CH352 course to be the introductory course of scientific publication process. Various chemistry journals were introduced to students using the "web of knowledge" platform. Various numerical matrices associated with the journals

were discussed. Representative journals specialized for organic chemistry were introduced. Methods on how to search chemistry articles were also taught. Students were reminded that a thorough referencing is expected for the midterm and final papers.

Introduction to Scifinder

Scifinder is a representative search engine that provides access to sources of references, substances, and reactions in chemistry and related sciences. It became an indispensable tool for those practicing synthetic chemical research. During the CH352 orientation session, the role and functions of Scifinder as well as the way on how to install the program was introduced to the students. We encouraged students to use Scifinder for the preparations of pre-lab report, results PPT slides, and term papers.¹⁵

Introduction to Chemdraw

The development of various chemical structure editors has facilitated the organic chemistry writing process. Chemdraw, first developed in 1985 by David Evans, Sally Evans, and Stewart Rubenstein,¹⁶ is inarguably the most widely used molecular structure editor. Chemdraw software was introduced to students during the orientation and demos on how to draw chemical structures using this program were conducted. A protocol on how to install the software was also presented. Students were encouraged to use Chemdraw in generating chemical structures for the course reports.

Introduction to Ethical Guidelines of the Course

The most common misconducts were introduced and the zero-tolerance policy against these misbehaviors was explained.

- Plagiarism of literature or reports from previous years
- Data fabrications
- Inappropriate attitudes toward the TAs

The importance of proper paraphrasing and referencing was stressed.¹⁷ We used turnitin (turnitin.com) as a platform to collect students' reports and crosscheck plagiarism. This web-based service helped us to reduce the administrative workloads related to collecting and grading term papers and providing feedback to students. We noticed that the use of turnitin system had a preventive effect of keeping students away from plagiarism.

We explained to the students that experimental data fabrications as well as adoptions of peer's data in writing their

own reports are grave scientific misconducts. In the case of low yields or unexpected side products, we advised students to thoroughly analyze the experimental procedure and rationalize the results.

The role of TAs was pivotal for a successful laboratory course. Building a constructive relationship between TAs and students based on mutual trust was imperative. A thorough formal introduction of each TA helped to form a sense of respect among students. The instructor made clear that expression of inappropriate attitudes towards TAs such as the use of vehement language or violent behavior will not be allowed in any case.

PRE-LAB NOTES

Students were obligated to handwrite a pre-lab note before each laboratory session. Students prepared their pre-lab notes which contained the following contents.

- MSDS of new reagents and solvents
- A step-by-step experimental procedure
- Answers to the pre-lab questions in the lab manual posted on the course website

Details of each experiment were posted on the course website (<http://www.chemlabinkaist.net/>). Purpose of the experiment, background information, experimental procedures, post-laboratory questions, and pre-laboratory questions were included in the experiment lab manuals. Students were guided to read the lab manual carefully before the lab session. They were asked to pay attention to the potential hazards of reagents and solvents used in the experiment. We required the students to handwrite the MSDS of each chemical in the pre-lab note.

A step-by-step experimental procedure of the experiment was also included in the pre-lab note. We made students use their pre-lab note and not the lab manuals during the lab session. This required students to be well acquainted with the experimental procedure and prepare a detailed pre-lab note. Lab manuals contained pre-lab questions regarding safety issues and essential concepts of the experiment. Students were reinforced of them by handwriting their answers instead of copying it on a word document.

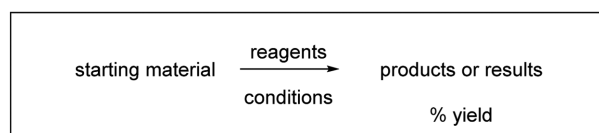
EXPERIMENTS AND RESULTS POWERPOINT SLIDES

A major component of our redesigned instructional methods was changing the format of the results report. Previously,

students submitted a multi-page text-heavy experiment results report after each experiment. After years of students' surveys and analysis of the course evaluations, we noticed severe student dissatisfaction related to excessive workloads associated with the preparation of the weekly experiment results report. Based on the feedback, we designed a new experiment results report which emulates "the lab notebook" of an ordinary organic chemist in a research group.

We asked students to generate and submit text-light, scheme- and photo-heavy PPT slides (maximum 5 slides) after each experiment. A general guideline for the PPT slides provided to students is presented in *Fig. 1*. A reaction scheme generated with Chemdraw and a starting materials and reagents table were expected to be included in the results PPT slides. Detailed and chronological experimental procedure was incorporated to the PPT slides. The order of reagents' additions and notable changes in colors, phases, or temperatures of the reaction were encouraged to be noted. We made students take pictures of key apparatus, set-ups, TLC plates used for reaction monitoring, and other notable observations. We then encouraged students to insert the relevant pictures into the results PPT slides (*Fig. 2*). Detailed purification methods of the crude reaction mixture and product yields were included in the results PPT slides. Spectroscopic data and their analysis were added to the PPT report. We also asked students to provide answers

A. Chemical reaction



B. Starting materials and reagents table

reagents	source	mmol	equiv.	MW	M (mg)	V (μ L)
X	X	X	X	X	X	X
⋮	⋮	⋮	⋮	⋮	⋮	⋮
⋮	⋮	⋮	⋮	⋮	⋮	⋮

C. Detailed experimental procedure and observations

13:30 Reagent A added to a CH_2Cl_2 solution of B at 23 °C.
14:00 Color change from X to X observed.
15:00 TLC reaction monitoring.
⋮
⋮

D. Purification and isolation of products

Flash column chromatography conditions
Recrystallization conditions
Spectroscopic data
Structures and yield of products

Figure 1. Template for the experiments and results PPT slides.

Exp4. Solid-Solid reaction of reductive amination

1:26pm reaction preparation
1:32pm reaction start
 - color : bright orange
1:40pm add 10ml ethanol
 - slightly dissolved
1:47pm add sodium borohydride
 - bubble(H₂)
 - color : clear orange
 - all dissolved
2:00pm add 2ml acetic acid
2:04pm add 2ml acetic anhydride
2:06pm heating
2:24pm add 75ml water
 - white solid precipitation
2:35pm ice bath
2:40pm vacuum filter
 - white solid
3:00pm NMR sampling

1:26pm reaction preparation
 White
Cc1ccc(N)cc1 41-46°C
p-Toluidine
 Pale yellow
Cc1ccc(O)cc1OC 40-42°C
o-Vanillin

1:32pm reaction start
 Bright orange
Cc1ccc(O)cc1OC
Imine

1:40pm add 10ml ethanol
 Slightly dissolved

Low melting point => Impurity => Lowering of melting point => Melting, direct reaction
 - Ecofriendly, no solvent
 - Fast reaction

1:47pm add sodium borohydride (reductive amination)

H₂ bubble clear orange solution All reactants are dissolved

Cc1ccc(O)cc1OC

Exp4. Solid-Solid reaction of reductive amination

2:00pm add 2ml acetic acid + add 2ml acetic anhydride

2:06pm heating (15min)
 Amide formation
Cc1ccc(O)cc1OC + CC(=O)OC(=O)C → Cc1ccc(O)cc1OC(=O)C
 80°C
 Clear solution

2:24pm add 75ml water
 Strongly stirring

2:35pm ice bath
 Crystallization

2:40pm vacuum filter
 Washing by water
 Air dry
 White solid
 1.36g (Yield : 95%)

More clear solution

Solubility
 Ethanol >> Water

Cc1ccc(O)cc1OC(=O)C

Figure 2. Sampling of slides from experiments and results PPT slides report.

to the post-laboratory questions in the report.¹⁴

MIDTERMS AND FINAL PAPERS

Students were required to submit two term papers using the ACS *JACS* template available online.¹⁸ Each student

was randomly assigned with two experiments two weeks prior to the midterm and final due dates and was asked to write up a midterm report in Korean and a final report in English. The following items had to be included in the midterm and final reports: Title, abstract, introduction, figures and schemes generated by Chemdraw, results and dis-

cussion, detailed experimental procedures, conclusion, and references. We encouraged students to read many relevant papers and emulate the presentation styles with proper paraphrasing and referencing.

STUDENTS' FEEDBACK

Satisfactory features of the course and factors that can be improved were surveyed at the end of the semester. Thirty-one students (67%) provided feedback. In addition to the survey, instructors had an end of semester dinner with TAs and students to obtain feedback. Below is the summary of the feedbacks.

Satisfactory Features (Number of Responses in Parentheses)

- New format of the results PPT slides report (21)
- Helpful and friendly TAs (12)
- New format of the midterm and final reports (10)
- Learning new experimental techniques (6)
- Introduction to chemdraw (2)
- Helpful orientation (1)

Students were most satisfied with the newly implemented weekly results report style. 68% of students commented that they were satisfied with the new format of presenting their experimental results. They mentioned that time required to prepare the results report could be saved. While time-saving, the schemes and pictures-based PPT report style led students to pay attention to the actual experimental procedures and take pictures of noteworthy observations. Students have developed a habit of taking chronological notes during the lab session which is an essential part of organic chemistry research. Below are samples of responses from the students' survey:

The course was not about getting good grades for the result report. It was about learning how to carry on an experiment from start to finish and learning how to write proper lab-notes.

Less pressure on writing the post lab report allowed me to focus more on the experiments and learn what research is all about.

I could save time on writing the result report and spend more time preparing for the experiment the night before and have a clear mind during the experiment.

Notably, students showed great satisfaction with the TAs. 39% of respondents showed appreciation for the mentorship and guidance that TAs had shown. The head TA who previously had taught this course a few times informed us that this year's TAs were more dedicated in teaching as compared to those in previous years'. We attribute these positive changes to the restructured TA allocation method. Allocating the same TAs for all experiments conducted during the semester to the same group of students enabled an interpersonal bond formation. Furthermore, the concise PPT-based experimental results report reduced the grading workload of the TA. Students expressed satisfaction with the new format of the midterm and final reports. They especially liked that they could expose themselves to actual research papers.

I never had the chance to read chemistry journal articles. Writing the term papers in a JACS style template allowed me to familiarize myself with journal articles.

There was also positive feedback regarding the use of Chemdraw and the orientation session.

Factors to be Improved (Number of Responses in Parentheses)

- Insufficient guidelines on midterm and final reports (12)
- Insufficient equipment instruction such as balances, drying oven, and rotovaps (10)
- Dirty glassware from previous users (7)
- Insufficient feedback of results PPT slides (6)
- Explanation on how to use basic equipment such as rotovap (5)
- Insufficient feedback of the midterm paper (5)
- Lack of contents to write the term paper (3)
- Long experiment time, better protocol for the flash column chromatography (3)
- Discrepancy between the actual experimental procedure and the procedure on the manual (3)
- Benchtop NMR machine (2)
- Experiment conducted individually (1)
- Hand-written pre-lab note (1)
- Expository instruction style (1)
- Excessive advantages for those who have lab experiences (1)

While students showed satisfaction with the JACS style term papers, they also answered that guidelines on how to write them were insufficient. We plan to hold a session on how to write a scientific report in the next laboratory course.

23% of students complained about the cleanness of shared glassware. 16% of students also replied that not enough explanations were given on how to use basic equipment such as rotovap. We will update the manual and hold an introductory session which will describe basic experimental operations such as washing glassware and using basic equipment in the laboratory. Prompt feedback on reports will have to be provided. A few students also informed us that a single experiment did not contain enough material to write up a *JACS* style term paper. One student pointed out the lack of intellectual excitement in the expository laboratory teaching style. We plan to apply problem-based instruction method to some experiments for the upcoming laboratory courses.

CONCLUSION

We restructured the organic chemistry laboratory course by mimicking the actual organic chemistry research convention. Experiments and results PPT slides were designed after a daily routine of lab-note taking for synthetic chemistry researchers. Journal article-templated term papers were implemented to provide students with an experience of scientific manuscript preparation. Molecular structure drawing software and journal searching methods were introduced during the orientation session. These exertions greatly improved students' interest evidenced by survey results and feedback from personal interactions. We also noticed that grouping the same TAs and students for all experiments has contributed to higher students' satisfaction. We plan to further improve the laboratory course by an in depth discussion about the scientific manuscript preparation, timely feedback of reports, detailed introduction of basic experimental skills such as glassware washing and rotovap usage protocol, and problem-based experiments where students have to answer questions based on their experimental data.

Acknowledgments. The authors are grateful for the financial support provided by the National Research Foundation of Korea (2015R1C1A1A020363), the Institute for Basic Science (IBS-R010-D1), and the POSCO Science Fellowship of POSCO TJ Park Foundation.

Supporting Information. Additional supporting information is available in the online version of this article.

REFERENCES

1. Undergraduate Research Participation Program at KAIST. <http://kchannel.kaist.ac.kr/CH751-000> (accessed Sep. 9, 2017).
2. Brown, C. F. *J. Chem. Educ.* **1951**, *28*, 382.
3. Domin, D. S. *J. Chem. Educ.* **1999**, *76*, 543.
4. Weaver, M. G.; Samoshin, A. V.; Lewis, R. B.; Gainer, M. *J. J. Chem. Educ.* **2016**, *93*, 847 and references therein.
5. 130 credits are required for a B.S. degree at KAIST.
6. Holbrook, J. *Making Chemistry Teaching Relevant*, In Proceedings of the 18th International Conference on Chemical Education, **2005**, *6*.
7. Bodner, G. M. *J. Chem. Educ.* **1986**, *63*, 873.
8. Larrow, J. F.; Jacobsen, E. N. *J. Org. Chem.* **1994**, *59*, 1939.
9. Egan, M.; Margaret Connors, E.; Anwar, Z.; Walsh, J. J. *J. Chem. Educ.* **2015**, *92*, 1736.
10. McDaniel, K. F.; Weekly, R. M. *J. Chem. Educ.* **1997**, *74*, 1465.
11. Touchette, K. M. *J. Chem. Educ.* **2006**, *83*, 929.
12. Holden, M. S.; Crouch, R. D. **2001**, *78*, 1104.
13. Cardinal, P.; Greer, B.; Luong, H.; Tyagunova, Y. *J. Chem. Educ.* **2012**, *89*, 1061.
14. See supporting information for details.
15. Swoger, B. J. M.; Helms, E. *J. Chem. Educ.* **2015**, *92*, 668.
16. Evans, D. A. *Angew. Chem. Int. Ed.* **2014**, *53*, 11140.
17. Garner, H.; Pulverer, B.; Marusic, A.; Petroveck, M.; Loadsman, J.; Zhang, Y.; McIntosh, I.; Titus, S.; Roig, M.; Anderson, M. *Nature* **2012**, *481*, 21.
18. Tilstra, L. *J. Chem. Educ.* **2001**, *78*, 762.