Using Green Chemistry Principles as a Framework to Incorporate Research into the Organic Laboratory Curriculum

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Transformative Learning that links passion with lifelong purpose. - Simmons College
Educating women to become research-empowered, responsible global citizens. – Chemistry & Physics Dept.
Simmons College Chemistry and Physics

• all our majors complete year-long independent study research projects, write and defend a thesis.

• research is for students, by students and with students.

• more majors graduate in the sciences than intend to upon entering as first-years.

• faculty bar for tenure / promotion / post-tenure review is ~3 publications in 5 years.

• no graduate students / post doctoral associates

• currently 5.5 Tenure Stream Faculty + 3 FT Contract Faculty.

• 100 to 150 Majors in the pipeline

• resource constrained environment.

• ~50% of our majors are first in their family to attend college.

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Chemistry Related Majors & Minors 2011 – 2015 (187)

- Grad School Chemical Sciences
- Medical and Other Professional Schools
- Industry
- Other
13 Years of Green Chemistry Research in the Teaching Laboratory at Simmons College

• 2002 – 2006 Learning to be green: Involve students in the decision making process
  – Greening the Oxidation of Borneol to Camphor
    • A Guided-Inquiry Investigation into Green Metrics
• 2006 – 2008 Undergraduate Laboratory Renaissance Pilot
  – Cups to Cleaners Converting Trash to Treasure
• 2008 – 2009 ULR Phase 1 (200 Level Chemistry)
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Waste per student:
A halogenated organic waste mixture of:
- 15 mL of diethyl ether
- 10 - 20 mL of dichloromethane

An aqueous waste mixture of:
- 3 mL of glacial acetic acid
- 3 to 5 mL of aqueous sodium hypochlorite (5.25%, household bleach)
- 15 mL of ice brine
- 15 mL of 5% aqueous sodium carbonate solution
- 15 - 20 mL of 5% aqueous sodium hydroxide solution

Acetone waste to clean glassware

Solid Waste:
- starch iodine and litmus paper
- 1 - 2 g magnesium sulfate
- silica TLC plates

Energy used to produce camphor:
- house vacuum or aspirators, rotary evaporators, hotplate (1 h)

Time:
- set-up: 0.5 hour
- reaction: 1 hour
- work-up/purification: 1 hour
- characterization: 0.5 hour
Open-Inquiry Based Student-Lead Greening: Student Developed Questions:

Fall 2003:

Can we find a better TLC solvent system to replace CH$_2$Cl$_2$?
Can the Et$_2$O for TLC extract be replaced with a better solvent?
Can we eliminate the final extraction solvent (CH$_2$Cl$_2$ / Et$_2$O)?
Can a corked test-tube replace the glass vial for final sublimation?
Can a Petri-Dish replace the corked test-tube?
Can we heat the reaction for a longer period so we do not need to follow the reaction?
Can we eliminate the separate sublimation step and thereby eliminate the energy required for heating the sand?
How green is the characterization of the final product? Is TLC the best method of analysis? Can IR be successfully used?
Can we prevent the microwave from overheating by adding a beaker of water to the microwave?
Can we prevent the microwave from overheating by adding a dish of alumina?
Can we diminish the water signal in the IR of the camphor product, by first drying the silica gel and MnO$_2$ in an oven?
Can we increase the yield of camphor by placing an ice cold Erlenmeyer on top of the Petri-dish cover?
Can we maximize the yield of camphor by using a Petri-dish with a smaller thickness to decrease the distance between the reaction medium and the product collection vessel?
Can we increase the purity of the sublimed camphor by increasing the distance between the reaction medium and the product collection vessel?
Can we decrease the amount of MnO$_2$ that is “carried-up” with the sublimed camphor product, by adding a thin layer of pure silica gel over the reaction medium?
Can we minimize the amount of MnO$_2$ that is “carried-up” with the sublimed camphor product, by increasing the particle size?
Can we simply shake the reagents together instead of grinding them?
Can we create a semi-continuous reaction vessel by layering the MnO$_2$-silica over the borneol?
Can the same reaction be used for the conversion of other alcohols, such as norborneol?
Does freshly prepared MnO$_2$ “work better” than “store bought” MnO$_2$?
Can we increase the yield of camphor by spreading out the reaction medium on the bottom Petri-dish?
Can we increase the yield of camphor by piling the reaction medium in the center of the Petri-dish?
Can a beaker of dry ice atop the Petri Dishes, increase the condensation of product by cooling the surface without addition of water.

Can the catalyst and solid support be used a second time with fresh borneol?

Can the use of different reactor designs increase the product yield above 15%?

Does the particle size of the silica gel cause a statistically significant increase in yield or purity of camphor?

Can the reaction product ratio be followed by GC/MS using a standard GowMac GC using temperature settings typically suited for the separation of cyclohexane and toluene?

Can the presence of borneol in the camphor product down to a 5% be observed by 1H NMR (90 MHz, EFT Anasazi)?

Can the yield of camphor be increased from 12 - 15% by heating the reactor directly on a hot plate?

What is the best yield I can get by carefully controlling the temperature of the hot plate?

(85 - 92% while still maintaining a 90 - 95% purity level)

Can a better reactor can be designed for hotplate reaction?

Why can’t we cancel the rest of the semester and continue working on this experiment?”

“Why haven’t we been introduced to green chemistry earlier?”

“Why isn’t every lab a green chemistry lab?”

“Who in their right mind thinks that green chemistry is a bad idea?”

“Why is this (Green Chemistry) a separate concept from regular Chemistry?”
**Alternative Aparatus**  
By: Tayaba Naz

4” Watch Glass  
1” Watch Glass  
Wide-mouth Powder Glass Funnel  
Hotplate  

0.350g MnO$_2$  
0.70g Silica Gel  
0.40g Borneol

Step 1: Weigh the small watch glass and the funnel.  
Step 2: Mix the reagents in a small 50-mL beaker with a metal spatula for 5 minutes and pour the regents in a pile on the large watch glass.  
Step 3: Cover the reagents with the cut-off or wide-mouth powder funnel as shown in the figure above. Place the small watch glass on the mouth of the funnel.  
Step 4: Carefully put the setup on the hotplate and gradually raise the temperature of the hotplate to 165°C over ~5 minutes. Monitor the temperature with a thermocouple.  
Step 5: Hold the temperature of the hotplate at 165°C for 10 minutes.  
Step 6: Gradually raise the temperature of the hotplate to 200°C over ~5 minutes and hold the temperature of the hotplate at 200°C for 10 minutes.  
Step 7: Carefully remove the setup from the hotplate and allow it to cool for about 5 minutes. Ensure the temperature is below 45°C.  
Step 8: Weigh the funnel and the small watch glass with the sublimed camphor product and determine the isolated weight and percent yield of your product.  
Step 9: Run TLC/IR/NMR/GC on your product to check the purity.
EXPERIMENTAL PROCEDURE:

Weigh the small watch glass and the funnel. Mix the reagents (0.350g MnO$_2$, 0.70g Silica Gel, 0.40g Borneol) in a small 50-mL beaker with a metal spatula for 5 minutes and pour the regents in a pile on the large watch glass. Cover the reagents with a wide-mouth powder funnel as shown in the figure above. Place the small watch glass on the mouth of the funnel. Carefully put the setup on the hotplate and gradually raise the temperature of the hotplate to 165°C over ~5 minutes. Monitor the temperature with a thermocouple. Hold the temperature of the hotplate at 165°C for 10 minutes. Gradually raise the temperature of the hotplate to 200°C over ~5 minutes and hold the temperature of the hotplate at 200°C for 10 minutes.

Carefully remove the setup from the hotplate and allow it to cool for about 5 minutes. Ensure the temperature is below 45°C. Weigh the funnel and the small watch glass with the sublimed camphor product and determine the isolated weight and percent yield of your product.

Characterize product via:
- Sealed-capillary melting point determination
- TLC (10:1 Ethyl Acetate:hexanes)
- IR (KBr pellet)
- $^1$H NMR 90 MHz (CDCl$_3$)
- GC

85 - 92% yield (90 - 95% pure by GC).
Unanticipated outcomes: solid-phase oxidation

- high level of student engagement with content
- high level of student personal accountability
- engagement within research and scientific method
- increased enthusiasm for chemistry and chemical research
- true concern and appreciation for disposal and safety
- every student became a critical evaluator
- every student became a contributing member of my research group

Students did not want to just learn Green Chemistry they wanted to practice Green Chemistry.
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Cups to Cleaners Converting Trash to Treasure
Hydrolysis of PLA to Lactic Acid

Hypothesis
If PLA cups can be converted easily to lactic acid in the laboratory, the exercise would provide an excellent means of discussing many of the 12 Principles of Green Chemistry within the same activity. Furthermore, the experience has the potential to educate the general community.

Can PLA cups be converted to lactic acid via acid or base?

Which method is greenest?

Theoretical Reaction Equations

14. I believe I have learned something valuable that will help me in future laboratory courses.

15. my appreciation of a well-written, well-structured lab report has increased.

16. my understanding of all necessary components of a thoughtful, complete lab report (such as procedure and discussion) has increased.

17. I think I have learned something that will help me write a better lab report.

18. my appreciation of completing Pre-Lab work or (preparing before attending a lab) has increased.

19. I have gained insights into Scientific Research that I think will stick with me for the rest of my life.

20. I am more confident in my laboratory skills.

21. I am more confident in my ability to operate the IR and NMR instruments.

22. I am more confident in my ability to interpret IR and NMR spectra.

23. I better appreciate how to apply and use IR and NMR spectra to determine the outcome of a reaction.

Green Chemistry provided a scaffold and a process to involve students in research within the teaching laboratories.
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Undergraduate Laboratory Renaissance
Funded in part by a grant from the W. M. Keck Foundation 2008 - 2012

• a complete redesign of the laboratory program
• early, intensive student involvement in current research (approaching green)
• research throughout the laboratory curriculum
• special roles for upper-classwomen
• coherent communication among laboratory sections

Benefits for students -

  extensive, hands-on, genuine research experience
  pre-Independent Study experience
  experience in mentoring
  interdisciplinary approach to problem solving
  confidence and enthusiasm
  investment in the laboratory curriculum
  appreciation for all components of the laboratory
  dramatic improvement in skill application and mastery

for faculty -

  many student coworkers
  research-ready Seniors (Graduate Student Model)
  “teaching laboratory” products are research relevant molecules
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Green Enantioselective Chiral Reduction via Post-Consumer PLA Waste

Organic Chemistry II Lab
Fall 2008 & 2009:
Solving Unknowns
Reduction of Ketone
Chiral Reduction using
PLA environment

Organic Chemistry I Lab
Spring 2009 & 2010:
Ketone synthesis
Polymer hydrolysis / transesterification

Polylactic Acid (PLA)

Ethyl Lactate
Lactic Acid
Partially digested polylactic acid (n = small)

Reduction

Research Integration - Mapping a Research Project Onto a Course

What concepts, techniques, and methods are fundamental to this course objective?

a) begin with goals of “expository experiments,”

b) determine what is needed for research but is missing from this course,

c) Map PLA research onto Organic Chem II,

d) determine what is needed for research but is missing from previous courses.
What concepts, techniques, and methods are fundamental to this course objective?

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## Goals of “Expository Experiments”

<table>
<thead>
<tr>
<th>Old Labs</th>
<th>Goal for the old labs</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Solving unknowns (4 weeks)</strong></td>
<td><strong>Skills of determining physical properties,</strong> <strong>Carry out functional group tests,</strong> <strong>solubility tests and be able to use the results</strong></td>
</tr>
<tr>
<td><em>melting and boiling Points,</em> <em>functional group tests,</em> <em>solubility tests</em></td>
<td></td>
</tr>
<tr>
<td><strong>Spectral analysis: IR, NMR</strong></td>
<td><strong>Spectroscopy Methods: use, application, interpretation, logical reasoning, gather data, analyze data.</strong> <strong>Putting all of the data together.</strong> <strong>Logically deduce structure</strong></td>
</tr>
<tr>
<td><strong>A Green Microwave Oxidation and Reduction: Oxidation of Borneol to Camphor (2 weeks)</strong></td>
<td><strong>Synthesis, introduction to green chemistry,</strong> <strong>application of organic chemistry to materials science, drug discovery, current topics in organic chemistry.</strong></td>
</tr>
<tr>
<td><strong>The Aldol Condensation Reaction (2 weeks)</strong></td>
<td><strong>Synthesis, puzzle, unknown determination</strong></td>
</tr>
<tr>
<td><strong>Microwave Synthesis, Column Chromatography, Metallation and Visible Spectroscopy of 5,10,15,20-tetraphenylporphyrin</strong></td>
<td><strong>Alternative heating method, green chemistry, macromolecules, coordination chemistry.</strong></td>
</tr>
<tr>
<td><strong>Gold Monolayer Lab / Combinatorial Chemistry Lab</strong></td>
<td><strong>Application of organic chemistry to materials science, drug discovery, current topics in organic chemistry.</strong></td>
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Missing Components for Research

- Did not teach the process of unknown determination independent of scaffold.
- Could not apply the techniques from Org I that they learned.
- Disconnected from Org I and Gen Chem.
- Could not follow a literature procedure and perform a synthesis.
- Literature Searching.
- Collaborative setting: sharing of data and information.
What concepts, techniques, and methods are fundamental to this course objective?

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Map PLA research onto Organic Chem II

PART I
• Identification of an Unknown

PART II
• Solid-State Reduction of a Ketone
  • Literature Procedure
  • Some Repeated and Some New Ketones

PART III
• Asymmetric, Solid State Reduction of a Ketone
  • Based Upon Literature Procedure
  • Same Ketone as Part II
Organic Chemistry II
New Lab Design for Solving Unknowns

Did not teach the process of unknown determination independent of scaffold. 

*No lab manual!*

MSDS (material safety data sheets).
Proper waste disposal.

*List of chemicals in stockroom with Greeness ranking.*

Disconnected from Org I and Gen Chem.

*No prepared reagents! Plan what scale, Calculation of reagents, Prepare solutions/reagents*

No Collaborative setting

*Collaboration: sharing of data and information through Wiki among different lab sections*
# Solving Unknown Compounds

<table>
<thead>
<tr>
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<tbody>
<tr>
<td><strong>Learning Goal for Skills</strong></td>
</tr>
<tr>
<td><strong>Skills Activity</strong></td>
</tr>
<tr>
<td><strong>Assessment of Skills</strong></td>
</tr>
<tr>
<td><strong>Learning Goal for Concepts</strong></td>
</tr>
<tr>
<td><strong>Concepts Activity</strong></td>
</tr>
<tr>
<td><strong>Assessment of Concepts</strong></td>
</tr>
<tr>
<td><strong>Learning Goal for Capabilities</strong></td>
</tr>
<tr>
<td><strong>Capabilities Activity</strong></td>
</tr>
<tr>
<td><strong>Assessment of Capabilities</strong></td>
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</tbody>
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<tr>
<th>New Lab</th>
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<tbody>
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<td><strong>Learning Goal for Skills</strong></td>
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<td><strong>Skills Activity</strong></td>
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<td><strong>Capabilities Activity</strong></td>
</tr>
<tr>
<td><strong>Assessment of Capabilities</strong></td>
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Organic Chemistry II
New Lab Design for Research Integration

- Don’t know how to do literature searching.
  
  *SciFinder*

- Could not follow a literature procedure and perform a synthesis.
  
  *No procedure given*
  
  *Obtain procedure from published journal paper*

- Could not apply the techniques from Org I that they learned.
  
  *Figure out how to monitor the reaction,*
  
  *isolate/purify product,*
  
  *analyze the structure and purity*
# Reduction of Ketones

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<tbody>
<tr>
<td><strong>Learning Goal for Skills</strong></td>
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</tr>
<tr>
<td>Be able to perform a procedure from a lab manual</td>
<td>Be able to perform a literature procedure to reduce a known ketone</td>
</tr>
<tr>
<td><strong>Skills Activity</strong></td>
<td><strong>Skills Activity</strong></td>
</tr>
<tr>
<td>Perform reduction and measure yield.</td>
<td>Reproduce the reduction of a ketone from the literature.</td>
</tr>
<tr>
<td><strong>Assessment of Skills</strong></td>
<td><strong>Assessment of Skills</strong></td>
</tr>
<tr>
<td>Percent Yield</td>
<td>Lab Practicum</td>
</tr>
<tr>
<td><strong>Learning Goal for Concepts</strong></td>
<td><strong>Learning Goal for Concepts</strong></td>
</tr>
<tr>
<td>Hypothesize about what went wrong based on yield?</td>
<td>Be able to apply a literature procedure to unknown ketone.</td>
</tr>
<tr>
<td><strong>Concepts Activity</strong></td>
<td><strong>Concepts Activity</strong></td>
</tr>
<tr>
<td>Write a laboratory report with conclusions.</td>
<td>Modify a literature procedure and apply to unknown ketone.</td>
</tr>
<tr>
<td><strong>Assessment of Concepts</strong></td>
<td><strong>Assessment of Concepts</strong></td>
</tr>
<tr>
<td>Lab Report</td>
<td>Lab Report</td>
</tr>
<tr>
<td><strong>Learning Goal for Capabilities</strong></td>
<td><strong>Learning Goal for Capabilities</strong></td>
</tr>
<tr>
<td>None</td>
<td>Be able to evaluate data and redesign experiments to achieve a product.</td>
</tr>
<tr>
<td><strong>Capabilities Activity</strong></td>
<td><strong>Capabilities Activity</strong></td>
</tr>
<tr>
<td>None</td>
<td>Design the best method to carry out the reduction.</td>
</tr>
<tr>
<td><strong>Assessment of Capabilities</strong></td>
<td><strong>Assessment of Capabilities</strong></td>
</tr>
<tr>
<td>None</td>
<td>Final Lab Practicum</td>
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- Organic Chemistry I
What concepts, techniques, and methods are fundamental to this course objective?

Organic Chemistry I

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<td>Separation / Purification of Liquids</td>
</tr>
<tr>
<td>Extraction</td>
<td>Separation of Solids</td>
</tr>
<tr>
<td>TLC</td>
<td>Analysis - Purity Determination</td>
</tr>
<tr>
<td>Recrystallization</td>
<td>Purification of Solids</td>
</tr>
<tr>
<td>Melting Point</td>
<td>Analysis - Purity Determination</td>
</tr>
<tr>
<td>Column Chromatography</td>
<td>Separation</td>
</tr>
<tr>
<td>Gas Chromatography</td>
<td>Analysis - Purity Determination</td>
</tr>
<tr>
<td>Synthesis</td>
<td>Reactivity Exploration</td>
</tr>
<tr>
<td>Chiral Resolution</td>
<td>Separation - More Specific</td>
</tr>
<tr>
<td>Natural Product Isolation</td>
<td>Pharmaceutical Research, microscale</td>
</tr>
<tr>
<td>Functional Group Reactivity</td>
<td>Exploration of Functional Groups, Qualitative Observation</td>
</tr>
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Expository experiments lead to knowledge, comprehension and at best application, but are devoid of analysis, synthesis and evaluation.
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Organic Chemistry I

a) begin with goals of “expository experiments,”

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How do I locate information on safety, hazards, waste disposal, contamination issues, purchasing chemicals, scaling reactions?

What is the primary literature? How do I find published procedures? How do I adopt a procedure for my needs?

How do I scale a reaction? How much of a given reagent should I prepare?

When is it appropriate to choose a method of isolation and purification and how do I apply distillation, extraction, column chromatography, recrystallization to my synthesis?

How can I apply TLC to an unknown mixture? Choose a developing solvent? a mixed solvent system? Visualization techniques?

What steps are involved in applying recrystallization of an unknown solid mixture? Choice of solvent?

When can I separate compounds by column chromatography? How much can I separate?
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Map PLA research onto Organic Chem I

PART I - Preparation for Research

PART II - Synthesis, isolation, purification and characterization of an alkyl halide

PART III - Synthesis, isolation, purification and characterization of secondary alcohol
Part I – Preparation for Research – 3 weeks

How do I locate information on safety, hazards, waste disposal, contamination issues, purchasing chemicals?

Create a chemical inventory, MSDS, catalog info, ordering, quantity in stockroom, supplier info for inventory for all chemicals used this semester and rank chemicals according to a “Greeness scale.”

What is the primary literature? How do I find published procedures? How do I adopt a procedure for my needs?

Locate representative procedures to perform Synthesis 1 – “easy,” using SciFinder – must be a primary reference – not “googled.”

How do I scale a reaction? How much of a given reagent should I prepare?

Write procedure to synthesize 5 grams of crude alkyl halide. No prepared reagents! Plan what scale, calculate amount of reagent(s), prepare solutions/reagents.

Technique / Lab Skill Development

Dry and fractionally distill 25 mL of starting alcohol. Compare GC of impure and purified alcohol.
Part II – Synthesis I – 2 to 3 weeks

Perform synthesis of alkyl halide – “easy”
Liquid extraction to isolate product.
Characterize crude product by GC.
Purify by distillation.
Characterize pure product by GC.
Part III – Synthesis II – 5 weeks

*Perform Grignard reaction – more “skilled”*

How can I apply TLC to an unknown mixture? Solvent selection? Mixed solvent systems? Visualization techniques?
   *Perform mini workups*
   *Figure out how to monitor reaction by TLC*
   *Isolate product by extraction*
   *Characterize crude product by TLC, IR, $^1$H NMR, $^{13}$CNMR*

When is it appropriate to choose a method of purification and how do I apply distillation, extraction, recrystallization, column chromatography to my synthesis?
   *Purify product*
   *Characterize pure product by IR, $^1$H NMR, $^{13}$CNMR*
Student performance and critical thinking increased through the ULR Research Integrated Program even when concepts were not explicitly covered.
Student performance increased through the ULR Research Integrated Program.
Assessment #2: In-Lab and Written Practicum Assessment

- GC Operation
- Rotovap Operation
- IR Operation
- TLC Operation
- Distill Theory
- Distillation Set-up
- Extraction Theory
- Quant of sol to Prep Given a Lit Proc
- Percent Yield Calculations
- Theoretical Yield Calculations
- Limiting Reagent Calculations
- GC Calculations
- GC Theory
Critical thinking in the lecture course was observed to increase by research integrating the lab through the ULR Program.
<table>
<thead>
<tr>
<th>Statement</th>
<th>mean</th>
<th>mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>8. I believe I have learned something valuable that will help me in future laboratory courses.</td>
<td>4.68 +/- 0.7</td>
<td>5</td>
</tr>
<tr>
<td>9. my appreciation of a well-written, well-structured lab report has increased.</td>
<td>4.09 +/- 0.9</td>
<td>5</td>
</tr>
<tr>
<td>10. my understanding of all necessary components of a thoughtful, complete lab report (such as procedure and discussion) has increased.</td>
<td>4.23 +/- 0.9</td>
<td>5</td>
</tr>
<tr>
<td>11. I think I have learned something that will help me write a better lab report.</td>
<td>4.28 +/- 0.9</td>
<td>5</td>
</tr>
<tr>
<td>12. my appreciation of completing Pre-Lab work or (preparing before attending a lab) has increased.</td>
<td>4.57 +/- 0.8</td>
<td>5</td>
</tr>
<tr>
<td>13. I have gained insights into Scientific Research that I think will stick with me for the rest of my life.</td>
<td>4.43 +/- 0.8</td>
<td>5</td>
</tr>
<tr>
<td>14. I am more confident in my laboratory skills.</td>
<td>4.51 +/- 0.7</td>
<td>5</td>
</tr>
<tr>
<td>15. I am more confident in my ability to operate the IR instrument.</td>
<td>4.43 +/- 0.7</td>
<td>5</td>
</tr>
<tr>
<td>16. I am more confident in my ability to interpret IR spectra.</td>
<td>4.38 +/- 0.8</td>
<td>5</td>
</tr>
<tr>
<td>17. I am more confident in my ability to operate the GC instrument.</td>
<td>4.55 +/- 0.8</td>
<td>5</td>
</tr>
<tr>
<td>18. I am more confident in my ability to interpret GC spectra.</td>
<td>4.51 +/- 0.9</td>
<td>5</td>
</tr>
<tr>
<td>19. I am more confident in my ability to operate the rotovap.</td>
<td>4.64 +/- 0.6</td>
<td>5</td>
</tr>
<tr>
<td>20. I better appreciate how to apply and use IR spectra to determine the outcome of a reaction.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>21. I better appreciate how to apply and use TLC to determine the outcome of a reaction.</td>
<td>4.23 +/- 0.9</td>
<td>5</td>
</tr>
<tr>
<td>22. I now have a better appreciation for the value of research in my own learning and personal growth</td>
<td>4.47 +/- 0.7</td>
<td>5</td>
</tr>
</tbody>
</table>

Confidence dramatically increased by research integrating the lab through the ULR Program

QUALITATIVE ASSESSMENT OF ATTITUDES

1 – Strongly Disagree
3 – Agree
5 – Strongly Agree
It was hard and I know I panicked and didn’t do well on the practical. Still, it was the BEST lab experience I have ever had. I’ll probably just get by, by the skin of my teeth – but I have fallen in love with chemistry; because of not only this course but also by the way it was taught.

I feel that I have learned much more from this semester’s lab that I did in CHEM 113. The cook-book style of CHEM 113 allowed us to just follow through the motions without really understanding the material. My experience in CHEM 114 engaged the material in a much better way.

I felt that the prelab process and the absence of a generic procedure really helped me learn and understand a very significant amount more information about what and how exactly to do every procedure. It forces you to rely on your own preparation. I learned a lot in the process and hope this continues on next year.

I thought the research integrated lab was interesting and it really made me think outside the box. However, it was way too much work to handle alongside the class.

I really enjoyed lab. It was hands on and we were a part of it. It wasn’t handed to us and just told to reproduce it but we had to look, and think about it. It allowed me to be proud that I found the procedure and got my results based on how I thought it was best.

It was hard work. Lots of time, energy! Don’t change it, just give us more credit!
13 Years of Green Chemistry Research in the Teaching Laboratory at Simmons College

• 2002 – 2006 Learning to be green: Involve students in the decision making process
  – Greening the Oxidation of Borneol to Camphor
    • A Guided-Inquiry Investigation into Green Metrics
• 2006 – 2008 Undergraduate Laboratory Renaissance Pilot
  – Cups to Cleaners Converting Trash to Treasure
• 2008 – 2009 ULR Phase 1 (200 Level Chemistry)
  – Chiral Reduction using Post-Consumer Poly-lactic Acid Waste
    • Organic Chemistry II (Fall 2008) then Organic Chemistry I (Spring 2009)
• 2009 – 2010 ULR Phase 2 (Interdepartmental 200 & 300 Level)
  • Organic Chemistry II (Fall 2009) then Organic Chemistry I (Spring 2010)
• 2010 – 2012 ULR Phase 3 (100 Level Chemistry)
• 2010 – 2015 Continuation in Organic Chemistry I & II
Spring 2011, Fall 2011, Spring 2012: Switchable Polymeric Substrates Based Upon Thymine Photodimerization

Fluid → Reversible Linking → Rigid

![Thymine structure]

Vinylbenzyl Thymine

![Vinylbenzyl Thymine structure]
Synthesis and Characterization of Four Model Systems

VBT:VBA

VBT:4-VP

pVBT

VBT:VBC
Assessment #1: Pre- and Post-Exam

Pre-Exam scores improved – A research-integrated ULR General Chemistry Course better prepares students for Organic Chemistry.

Mole Calculations
Purification
Distillation / GC
Spectroscopy
TLC
Extraction
Measurements & Soln Calculations
Recrystallization
Safety & Waste Disposal
Assessment #2: In-Lab Practicum

Self-reported student confidence in the laboratory matches performance – A research-integrated ULR Course better prepares students to work in the lab.
<p>| | | | |</p>
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<tbody>
<tr>
<td>4.</td>
<td>The frequency of the quizzes forced me to keep abreast with the current material.</td>
<td>4.6</td>
<td>0.7</td>
</tr>
<tr>
<td>5.</td>
<td>The exams should have been more challenging.</td>
<td>1.7</td>
<td>1.0</td>
</tr>
<tr>
<td>6.</td>
<td>The level and difficulty of this course matched my expectations.</td>
<td>3.7</td>
<td>1.2</td>
</tr>
<tr>
<td>7.</td>
<td>I hoped the course would challenge me more.</td>
<td>1.7</td>
<td>1.0</td>
</tr>
<tr>
<td>8.</td>
<td>The course had a dramatic impact on my day to day life / sleep etc.</td>
<td>4.3</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Students are reading the assessments.
The research integration helped me:

• learn more by doing that just following a general procedure
• become so much more confident and comfortable in lab.
• become more confident in applying techniques to lab experiments
• apply concepts and understanding how to apply them in the future
• understand better the experimental chemistry, rather than the procedural
• to prepare better for the laboratory. Understand stuff before going to the lab
• to gain a better understanding of research in the real world, and that things don't always were as they should theoretically and how to work with that.
• learn how to work independently and with a partner without a manual telling me what to do. It helped me learn how to problem solve.
• interpret data. There isn't too much to compare to (other than other lab members data) so there isn't a definitive answer. You have to push harder to achieve the best results yourself.
• gain confidence in the lab and challenged me to think harder for myself
• understand concepts from class better because I had context
• think for myself instead of follow a procedure

Figure 1. A catalytic portrayal of the laboratory manual as a means of circumventing the utilization of higher-order cognitive skills.

Added Benefits / Challenges / Opportunities

• ‘research integration’ is student driven.
• teaching and instrumental assistants are our Senior Independent Study students.
• students are able to secure positions in REU / Internships during summers after their second or even first year at Simmons.
• Instructors read and analyze research results as opposed to 50 to 100 lab reports.
• students are able to enroll in multiple REU / Internships to investigate multiple programs or fields.
• graduating seniors often have contributed to or been involved with 3 to 5 different research projects.
• dramatic increase in students interested in research. 14 out of 54 students in Orgo I in Spring 2011, joined ongoing projects in the Gur-Lee research group for Fall 2011. 12 students joined in the Fall 2012.
• not all research projects are able to be paced with lecture equally well.
• quality vs quantity - can not always cover all techniques in a given semester / year.
• course preparation work is heavy before the semester but lighter throughout
• less chemical and stockroom preparation
• senior research / faculty research integrated into classroom – you can use teaching and instruction budget to pay for your research
• products synthesized are often used at least twice throughout the teaching lab and often in the research lab
• all lab sections lead by faculty – added challenges in our newly found resource constrained environment.
Current Evolution of “Best Practices”

• Consider Orgo I and Orgo II as a bundle with respect to Techniques and Instrumentation.

• Create Technique / Instrumentation “training modules” which are fully independent from a specific application or use.

• Each “training module” has a technique based reading assignment, a 20 minute pre-lab lecture, a 20 minute trouble shooting summative post-lab lecture, a 10 minute quiz, and a required “Technique and Protocol” summary that students collect on their own ePortfolio Wiki site.

• Lab reports are research based summaries – summative assessment

• Weekly Technique and Protocol summaries, serve to police student work throughout the semester - formative assessment.

• Research is mapped over the entire two semester course, and Techniques and Instrumentation is “brought in” when appropriate to the research.

• A one-hour collective pre-lab lecture (in addition to the 4 hour laboratory)

• Weekly 5 question – 5 minute “minimum competency quizzes” greatly aid in students coming prepared to do the work as compared to prepping a notebook with procedures that quickly change from one lab to the next.
Techniques and Protocols

- Chemical Information
- Infrared Spectroscopy – sample prep, instrument operation, spectral interpretation
- Thin-Layer Chromatography
- Recrystallization
- 13C NMR – sample prep, instrument operation, spectral interpretation
- Synthesis Preparation
- Solution Preparation - % solutions, molar solutions
- Synthesis Preparation
- Scaling a reaction
- Extraction and Product Isolation, Drying Agents
- Distillation and Boiling Points
- Rotary Evaporation
- Gas Chromatography - Sample prep, instrument operation, data interpretation
- 1H NMR – sample prep, instrument operation, spectral interpretation
- Column Chromatography
- Recrystallization and Melting Points
- HPLC - sample prep, instrument operation, data interpretation
Conclusions

• Start small, keep it simple
• Let student interest drive innovation
• Join the community – we are all here to help
• Embrace failure – students learn best when procedures fail and require fixing
• Spent time scaffolding research projects instead of prepping chemicals, and writing perfect laboratory manuals
Acknowledgements

Nancy Lee, Leonard Soltzberg, Michael Berger, Changqing Chen, Cheryl Lavoie and Jenna Canfield, Department of Chemistry
Velda Goldberg, Department of Physics
Mary Owen, Jane Lopilato, Liz Scott, Maria Abate Department of Biology
Diane Felicio, Advancement, Simmons College

Green Chemistry Institute of the American Chemical Society.
Simmons College Chemistry Physics Liaison, Alumni Office, Admissions Office, Jeff Stone, Aramark

Students enrolled in:

   CHEM 225 Organic Chemistry II, Fall 2008 - 2011
   CHEM 114 Organic Chemistry I, Spring 2009 - 2012
   CHEM 345 Biochemistry, Fall 2008 - 2011
   CHEM 347 Advanced Biochemistry, Spring 2009 - 2012
   BIOL 337 Molecular Biology, Spring 2010
   CHEM 226 Quantitative Analysis, Spring 2009 – 2012
   CHEM 113 Principles of Chemistry, Fall 2010 – 2011
   BIOL 341 Microbiology of Food, Water and Waste, Fall 2008 - 2010

W. M. Keck Foundation 2008 - 2011 ($244,866)
Simmons Presidential Fund for Faculty Excellence ($29,750) 2008 & 2011
Camille and Henry Dreyfus Foundation

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Merck/AAAS Undergraduate Science Research Program
Anne Coghlin Fund for Undergraduate Research
Thank you!

• Questions?
  – gurney@simmons.edu
  – (617) 521 - 2729
The research integration helped me:

- feel more comfortable being on my own in a laboratory
- understand more clearly what was happening in class and critically think
- gain independence in the lab and develop a greater appreciate for people that conduct research
- apply chemistry in a fun way
- become more interested in chemistry research.
- understand different techniques in the lab and become more comfortable, because we were actually applying the material on a "real-life" hands on project
- learn how to do work on my own
- take lab techniques and commit it to memory as something I can use if I want to do research
- gain confidence, gain experience, made me think more
- understand how to apply techniques to a wide variety of experiments
- be creative and feel more connected to the lab
- understand more about synthesis and how to properly operate in the lab setting. It also gave me more independence since my lab partner and I wouldn't always agree with a procedure, concept...etc
- to apply the material we were covering in class
Biomimetic Templated Growth of Calcium Oxalates using Microcontact Printed Self-Assembled Monolayers

Kidney Stone