Implementing the Science Writing Heuristic laboratory report format in the undergraduate organic chemistry laboratory

Jacob Daniel Schroeder
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Implementing the Science Writing Heuristic laboratory report format in the undergraduate organic chemistry laboratory

by

Jacob Daniel Schroeder

A dissertation submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Chemistry

Program of Study Committee:
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Iowa State University

Ames, Iowa

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Table of Contents

ACKNOWLEDGEMENTS .............................................................................................................................. vii

CHAPTER 1: GENERAL INTRODUCTION ................................................................................................. 1
Chemistry Education Literature Concerning Laboratory Learning .......................................................... 1
The Science Writing Heuristic .................................................................................................................. 7
Organization of the Dissertation .............................................................................................................. 8
References .................................................................................................................................................. 11

CHAPTER 2: THE LABORATORY-LECTURE CORRELATION: FROM THE
SCIENCE WRITING HEURISTIC TO THE TRADITIONAL ORGANIC
CHEMISTRY LABORATORY ...................................................................................................................... 15
Abstract .................................................................................................................................................... 15
Introduction ............................................................................................................................................... 16
Background ............................................................................................................................................... 18
Methodology ............................................................................................................................................ 20
Results ..................................................................................................................................................... 22
Discussion ............................................................................................................................................... 30
References ............................................................................................................................................... 32

CHAPTER 3: RECRAFTING THE ORGANIC CHEMISTRY LABORATORY
REPORT – USING THE SCIENCE WRITING HEURISTIC TO FOSTER GUIDED
INQUIRY .................................................................................................................................................... 38
Abstract .................................................................................................................................................... 38
Introduction ............................................................................................................................................... 39
History ....................................................................................................................................................... 40
General Modifications ............................................................................................................................. 41
Rationale ................................................................................................................................................... 45
Methods and participants ....................................................................................................................... 45
Results ....................................................................................................................................................... 46
   Dehydration ........................................................................................................................................ 46
   Nucleophilic Substitution ..................................................................................................................... 47
Abstract.............................................................................................................................. 92
Why Facebook?.................................................................................................................. 94
Methods and Participants .............................................................................................. 96
The WebCT Group Page................................................................................................. 97
The Facebook Group ...................................................................................................... 98
Discussion......................................................................................................................... 99
Conclusions...................................................................................................................... 100
References........................................................................................................................ 101
List of Tables and Figures.............................................................................................. 105

CHAPTER 7: GENERAL CONCLUSIONS.................................................................... 114
Summary of Conclusions............................................................................................... 114
Implications ....................................................................................................................... 117
Limitations....................................................................................................................... 119

APPENDIX A: CONVERTED LABORATORY MANUAL ......................................... 121
Table of Contents............................................................................................................ 122
Introduction..................................................................................................................... 123
Chem231L Course Policies and Procedures................................................................. 124
General Safety Guidelines & Rules in the Chemical Laboratory .............................. 129
Glassware and Equipment ............................................................................................. 131
The Science Writing Heuristic—A Brief Description.................................................. 133
The Science Writing Heuristic—Detailed Explanation............................................... 134
Lab 1 – Techniques: A Glimpse into the Chemist’s Toolbox...................................... 136
Lab 2 – Which Beverage Contains the Most Caffeine?............................................... 141
Lab 3 – Margarine in the Chemistry Lab..................................................................... 147
Lab 4 – Alkenes: Gathering a Wealth of Evidence to Support Claims...................... 152
Lab 5—Electrophilic Aromatic Substitution: The Effect of Directing Groups on Benzene ....................................................................................................................... 157
Lab 6—“Clean” Chemistry: From Margarine to Soap?................................................ 164
Lab 7—Molecular Models: The World of 3-Dimensional Chemistry......................... 168
Lab 8—Substitution vs. Elimination: The Chemical Competition ............................ 180
Lab 9—Artificial Scents: The Chemistry of Fragrances ............................................. 186
Lab 10—Sodium Borohydride: From a One-Time Accident to H₂ Fuel Cells .......... 189
Lab 11—Chemical Inventory: Preparing for the EPA Inspection ............................... 192
Lab 12—The Aldol Condensation: The Carbon Construction Company .................. 196
Lab 13—Radical Chemistry: Bromine vs. the Hydrocarbons ................................ 201

APPENDIX B: PREPARATORY NOTES FOR TEACHING ASSISTANTS ..................... 206
TA Notes – Checking In ................................................................................................ 206
TA Notes – Prep of Acetanilide .................................................................................. 207
TA Notes – Caffeine Extraction .................................................................................. 208
TA Notes – Hydrogenation .......................................................................................... 209
TA Notes – Dehydration ............................................................................................... 211
TA Notes – Aromatic Substitution ............................................................................... 212
TA Notes – “Clean Chemistry” (Soap) ..................................................................... 213
TA Notes – Models ....................................................................................................... 214
TA Notes – The Chemical Competition ...................................................................... 215
TA Notes – Artificial Scents ....................................................................................... 217
TA Notes – Reductions with NaBH₄ .......................................................................... 218
TA Notes – Chemical Inventory .................................................................................. 219
TA Notes – Aldol Reaction ........................................................................................... 221
TA Notes – Radical Chemistry .................................................................................... 222
TA Notes – Lab Practical Exam ................................................................................... 223

APPENDIX C: SAMPLE LABORATORY REPORTS ...................................................... 225
Preparation of 4-methylcyclohexene (traditional) .................................................... 226
Alkenes: Gathering a wealth of evidence to support claims (SWH) ......................... 229
Preparation of 2-chloro-2-methylpropane (traditional) ........................................... 234
Substitution vs. Elimination: The Chemical Competition (SWH) ............................ 238
Electrophilic aromatic substitution: The nitration of methyl benzoate (traditional) .... 240
Electrophilic aromatic substitution: The effect of directing groups on benzene (SWH) ........................................................................................................................................ 245
APPENDIX D: ASSESSMENT AND EVALUATION ................................................... 249
  Introductory Survey ....................................................................................................... 249
  Mid-Term Evaluation ..................................................................................................... 250
  End of Semester Evaluation ........................................................................................... 251
  End of Semester Evaluation for Lecture ...................................................................... 251

APPENDIX E: LABORATORY PRACTICAL EXAMINATION ................................ 253
  Exam ................................................................................................................................. 253
  Materials List .................................................................................................................. 255
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CHAPTER 1: GENERAL INTRODUCTION

Chemistry Education Literature Concerning Laboratory Learning

Chemistry has been considered a “hands-on” science, so it is not surprising that the chemistry laboratory has long been understood to be a critical and invaluable component to any chemistry course (1). Bowers argued, “The information brought by real contact with things is remembered much easier,” (1a, p. 207). Schlesinger noted that it was important to understand what the laboratory was to accomplish that could not be accomplished in the lecture (1b). He identified the general aims of the laboratory were to:

1. illustrate and clarify principles discussed in the classroom by providing actual contact with materials
2. give the student a feeling of the reality of science by an encounter with phenomena which otherwise might be to him no more than words,
3. make the facts of science easy enough to learn and impressive enough to remember
4. give the student some insight into basic scientific laboratory methods, to let him use his hands, and to train him in their use.

With three of the four objectives discussing physical work and observation, he argued, “The student’s powers of observation should no more be expected to develop spontaneously through unguided experience than his knowledge of facts and his ability to think should be allowed to grow by haphazard reading and chance encounters with thought-provoking problems,” (p. 525)

Despite the perceived notion that “learning by doing” was acceptable as well as effective, a debate began escalating over whether chemistry laboratories should even be offered at all. In 1936, Hunt reviewed the literature and argued for the replacement of the laboratory with faculty-led demonstrations because they were both popular among faculty and economical in terms of time requirements and cost (2):

“The biggest advantage of the demonstration method is the saving of student time. The second important feature is the saving of money by the student and university. To date there are no reliable statistics proving that the student learns a great deal more by one system than the
other; however, those teachers who have used both systems do not contend that the only way for a student to learn chemistry is for him to perform the experiment.

Knox addressed the issue by describing the notion of replacing laboratory work with demonstrations was only considered because it was cost effective, not because it benefited the student academically (3). In his view, the demonstration method of teaching laboratory skills had developed primarily because of financial difficulties brought about by World War I and the “unprecedented increase in school enrolments”. The idea of abandoning the laboratory in favor of demonstrations he argued “would mean a degeneration of science teaching in the secondary schools,” (p. 171).

This was further elaborated in 1942 by Adams, who observed that the literature up to that point had reviewed some fifty references (covering a period of over 30 years) on experimental studies relating to the individual versus the demonstration laboratory (4). Based on his review, he concluded that,

“At the conclusion of a laboratory course in beginning chemistry those pupils who have had individual instruction do consistently better than those who have seen the experiments performed for them in class or those who have heard the instructor explain the experiments in lecture, when these pupils are measured by a laboratory performance examination…Contrary to the conclusions of many of the previous investigators the individual method of laboratory instruction is superior, particularly for the superior student, while the lecture-demonstration method may be somewhat better for students at the lower intelligence levels,” (p. 267).

Many educators perceived the “students at the lower intelligence levels” to be those not pursuing a degree in chemistry but who were enrolled in the introductory or elementary chemistry courses. The debate over what should be taught in the laboratory, or even how it should be taught, continued on. Thomas addressed these elementary courses specifically, arguing that educators should be teaching non-science majors how to use the scientific method (5). This argument was based on the premise that a debate existed between those
advocating the lecture-demonstration approach and those favoring the hands-on laboratory simply because a general dissatisfaction with the results of laboratory work in elementary courses was beginning to become widespread. Thomas argued, “it is probably fair to state that the orthodox laboratory course in general chemistry largely fails to teach students the scientific method,” (p. 379). In order to make the laboratory meaningful, the following objectives were suggested: (1) wherever possible, plan the experiment to include an unknown; (2) avoid detailed directions; (3) segregate questions dealing with background material from those based upon experimental results; (4) the results of the experiment should not be given in advance; and (5) experiments should be designed so that the student uses the results to plan future work. In developing experiments in this manner, Thomas noted that, “errors in judgment, procedure, and technique are numerous; but is that not actually an advantage? Does not the student learn through such a process?” (p. 380).

After the end of World War II, it was becoming increasingly clear that the science laboratory was not only beneficial but also necessary. Yet, the debate over what to teach and how to teach it continued on. In 1954, Xan repeated the calls to train students on how to use the scientific method, arguing, “what we really need to do is to develop trained [individuals] with the true scientific attitude to lead us through the Atomic Age and the freshman lab is the place to catch them,” (6, p. 520).

While many educators agreed with that statement, it was becoming increasingly more evident that in order to “catch them”, the freshman laboratory would need to be a place where students could experience a sense of excitement and develop critical thinking skills. But in a review of the available laboratory manuals at the time, Mallinson and Buck argued, “the majority of laboratory experiments require mere imitation rather than critical thinking,”
For the next 15 years, many educators were calling for a change to the laboratory, arguing that it needed to be more problem-based, more realistic, and more inductive (8). Where the prior arguments for lecture-demonstration laboratories primarily revolved around cost, these new calls for change included statistical data that showed new methods were superior in promoting student achievement (9).

In addition to statistics, a theoretical framework was emerging based upon Jean Piaget’s stages of intellectual development (10). Of interest to chemical educators were the concrete operational and the formal operational stages (10b). According to Herron,

“The concrete operational student does not think in terms of possibilities and is not able to understand abstract concepts which depart from concrete reality. The formal operational student, however, begins thinking in terms of what might happen and to envision all the changes that are possible…But the starting point for the concrete operational student is always the real rather than the potential. His reasoning is always based on real observations and is limited to extrapolations from these sensory experiences. He does not delineate all possibilities and think of the observed as simply a special case of the possible,” (10c, p. 147).

This would imply that a student with a concrete operational intellectual level might have a difficult time learning and applying abstract chemical concepts. It was starting to become apparent that the “cookbook” nature of the laboratory fostered concrete operational rather than formal operational thinking, leading to memorization rather than understanding (10b).

In response to this theoretical framework, a number of new laboratory experiments were developed, emphasizing real-world applications (11). According to these authors, these real-world experiments could help students better contextualize these abstract concepts in terms of practical applications. The idea was that if students entered the laboratory with a concrete operational level of understanding, they would more likely be able to progress to the
formal operational level by starting out with a topic with which they were either familiar, or
had some prior experience.

Despite the influx of “real-world” laboratory experiments, the laboratory was still not
significantly affecting student learning. In their review of the role of the laboratory in
science teaching, Hofstein and Lunetta note:

“The research has failed to show simplistic relationships between experiences in the
laboratory and student learning…Appropriate laboratory activities can be effective in
promoting logical development and the development of manipulative and observational skills
and in understanding scientific concepts. They also can promote positive attitudes, and they
provide opportunities for student success and foster the development of skills in cooperation
and communication,” (12, p. 212).

They argue that because many of the studies in their review had not reported significant
results, the laboratory was at least as effective in promoting student growth on the variables
measured, as were more conventional modes of instruction. But they did point out that if any
differences did exist between the two modes of instruction, they were “probably masked by
confounding variables, by insensitive instrumentation, or by poor experimental design”.

Although Hofstein and Lunetta’s review did highlight some positive aspects of the
laboratory in affecting student learning, many educators remained skeptical. In his review of
the many studies undertaken comparing laboratory instruction with other modes of
instruction, Toothacker points out that none of these studies had reported any significant
differences with respect to student learning outcomes (13). These studies included a
comparison between conventional laboratories and demonstrations (14a), conventional
laboratories, demonstrations, or no laboratory contact at all (14b), lecture sections, lecture
sections with a recitation, and lecture sections with a laboratory session (15), and a lecture
with a laboratory session or a discussion session (16). These results led Toothacker to argue,
"Introductory student laboratory work is not achieving its objectives. The situation will not be improved by merely replacing verification experiments with investigative experiments. A major restructuring of the laboratory curriculum is needed," (13, p. 519).

And the major restructuring he was referring to was to eliminate the laboratory as a requirement for all freshmen and sophomore physics classes because the majority of these students would never work in a physics laboratory. A few years later, Pickering made similar comments after pointing out that the current laboratory model was nothing more than an illustration, with an objective “to verify the already-known, and woe to those whose data do not agree,” (17). Although he agreed with Toothacker that “training in technique is hardly important to the vast majority of our clientele who will not be chemists,” he also made the point that “the art of logical deduction from data is”. Rather than eliminating the laboratory altogether, he advocated that if the laboratory was to illustrate anything, it should be the scientific method.

In 1986, Bodner (18) argued the problem was a lack of a learning theory:

“Until recently, the accepted model for instruction was based on the hidden assumption that knowledge can be transferred intact from the mind of the teacher to the mind of the learner. Educators therefore focused on getting knowledge into the heads of their students, and educational researchers tried to find better ways of doing this. Unfortunately, all too many of us who teach for a living have uncovered evidence for the following hypothesis. Teaching and learning are not synonymous; we can teach, and teach well, without having the students learn” (p. 873)

Bodner summarized a constructivist learning philosophy with a single statement: “knowledge is constructed in the mind of the learner”. Since that time, many educators began to develop experiments and classroom activities based on the constructivist learning theory. There have
been several literature reviews concerning this philosophy in the chemistry classroom, many of which are discussed in the literature reviews of the individual chapters contained herein.

**The Science Writing Heuristic**

The Science Writing Heuristic (SWH) was developed to promote learning from laboratory work by incorporating a writing to learn strategy and by challenging students to make claims about the data they obtained during experimental procedures and to substantiate those claims with appropriate evidence gathered through experimentation (19). Having its roots in constructivist learning theory (10c, 18), it serves as a tool to help students negotiate and develop an understanding of new knowledge that may run counter to their preexisting knowledge. In a broad sense, the theory of constructivism recognizes that students enter a classroom with prior beliefs, ideas, and a preexisting mental framework. When introduced to new concepts, students can apply this preexisting mental framework to interpret the new data (assimilation) or they can modify the preexisting mental framework in a way to make it fit with the new data (accommodation) (20). The other option is for students to fail to accomplish either of these two processes, instead having to resort to either memorizing the new material in order to “just get by”, or to ignore it all together.

The SWH was also designed to incorporate a learning cycle in the collaborative-guided inquiry activity. The cycle consists of three separate but reversible stages: exploration, term introduction, and concept application (21, 22). During the exploration phase, students are given a number of possible scenarios that could take place in the laboratory. They work together, trying to observe and manipulate data in order to discover a pattern among the experimental results (varying the mass, the concentration, etc.). Once students recognize a pattern, the instructor can facilitate a discussion by asking leading
questions to help students understand why the pattern is evident, i.e., what factors in the experimental work led to the results. Once the reasons for the pattern are articulated, new terms and concepts can be introduced to reflect the experimental results obtained. For the final phase of the cycle, the SWH incorporates a Reading and Reflection component, in an effort to guide students by applying the newly discovered concepts and terms to other contexts that may be more pertinent to them. Several relevant reports are cited within this dissertation highlighting the benefits of implementing the SWH in the laboratory and the impact this laboratory learning approach has had on student performance in lecture. These include significant increases in student achievement across all educational grade levels (23), specifically in general chemistry (24), organic chemistry (25), 7th grade biology (26), and high-school cell biology (27). The SWH template has also been used to train new graduate teaching assistants (28). Other researchers nationally have also begun implementing the Science Writing Heuristic, reporting similar results (29). A detailed discussion about the individual components of the Science Writing Heuristic can be found in Appendix A.

Organization of the Dissertation

This dissertation is organized into seven chapters and five appendices. The numbering of all figures, tables, and references are self-contained within each chapter. Chapter 1 provides an historical survey of the literature concerning laboratory work, as well as the history and development of the Science Writing Heuristic (SWH) laboratory report format. Chapter 2 presents the results of the initial pilot study undertaken to analyze the effectiveness of the introductory organic chemistry laboratory and its relationship to the lecture. This analysis constituted a longitudinal study, in which students from a prior study in general chemistry were followed into the subsequent organic chemistry course. The
previous study showed that these students performed significantly higher than a group of students in a traditional control group on lecture exams, quizzes, and on an ACS Diagnostic Exam while using the SWH laboratory format. This pilot study was also an investigation into whether or not having prior experience with the SWH laboratory format would have any impact on how well students would do on lecture exams in organic chemistry. At the time, the organic chemistry laboratory followed a traditional, verification format. Student lecture exams were analyzed with a specific focus on questions directly related to the laboratory experiments preceding the exam. A group of students with a traditional laboratory background from the previous semester was selected to serve as a control group. The two groups of students were matched based on their performance on the first organic chemistry exam.

The results obtained from this pilot study led to drafting a new introductory organic chemistry laboratory manual, incorporating collaborative guided-inquiry techniques using the Science Writing Heuristic laboratory format. Chapter 3 describes the discrepancy between student scores on laboratory reports and on lecture exams. Analyzing the traditional laboratory manual revealed that students can simply copy the answers out of the laboratory manual, thereby scoring well on laboratory reports, but fail to understand how the ideas and concepts in the laboratory can be applied to the lecture. Because of this, the laboratory manual was rewritten to incorporate the Science Writing Heuristic. This chapter discusses how the former laboratory manual was converted into a guided inquiry laboratory manual and how the laboratory was restructured to more closely align with the lecture. Student performance on lecture exams and laboratory reports using the traditional laboratory format during the spring of 2006 and the SWH during the spring of 2007 were compared.
Chapter 3 is further expanded with Appendix A, the most recent version of the converted laboratory manual, which is presented in its entirety to help illustrate much of the rationale provided in the chapter. Appendix B provides instructors with a guide of how to implement each experiment the way that it was intended. Teaching assistant notes for each experiment are provided describing the learning goals, how to effectively direct the class, as well as some things to look out for, such as common student misconceptions, safety hazards, and some areas known to be present problems in the laboratory. Appendix C is a sampling of written student laboratory reports for the three topics discussed in Chapter 3.

Chapter 4 describes an analysis of the relationship between student abilities to predict the products of nucleophilic substitution reactions and to write a mechanism for a similar, if not the same reaction. A comparison is made between students from two different terms (i.e., traditional vs. SWH approaches) who received perfect scores on the nucleophilic substitution laboratory report and their performance on a nucleophilic substitution exam question in the lecture. Chapter 4 also describes the introduction of Process Oriented Guided Inquiry Learning (POGIL) in the lecture in conjunction with the SWH approach in the laboratory. Quantitative results are shown for student performance on laboratory reports and lecture exams, while qualitative results are compiled to show student perceptions of organic chemistry both prior to and after taking the course.

Chapter 5 discusses the development of a laboratory practical examination to replace the standard written multiple-choice final exam conducted during the traditional laboratory. This research investigated the relationship between the total points and percentages students obtained on laboratory reports and pre-laboratory quizzes compared with performance on the final laboratory examination. The results indicate that students in a traditional laboratory did
very well on laboratory reports (91.8%), yet on average scored nearly 27 percentage points lower on the laboratory final (64.1%). After introducing the laboratory practical final exam, scores were more consistent with performance on laboratory reports.

Chapter 6 explores the role of technology in learning and discussing chemical concepts. An emerging area of research is emphasizing the educational benefits of harnessing social networks as communication tools. With the National Research Council issuing a call for educators to create discourse in the science classroom, social networks have become a focal point due to their popularity. Since their origin, the growth of these networks is staggering, creating a multitude of students who are already familiar with many of the communication tools that these sites offer. A recent survey of 677 college professors conducted by Thomson Learning found that nearly 50% of those surveyed feel social networking sites have or will change the way students learn. And according to Cisco CEO John Chamber’s prediction at Comdex 1999, “Education over the Internet is going to be so big it is going to make e-mail look like a rounding error”. Considering the overall growth of the Iowa State University Facebook community (60% in two years), this research focused on whether or not students would utilize this format for discussing chemistry concepts. A comparison is made with a private Facebook group and WebCT to determine which forum students were utilizing more in discussing course-related content.

Chapter 7 discusses the limitations and implications with regard to the research presented in Chapters 2 – 6, and explores future directions for further research.

References


CHAPTER 2: THE LABORATORY-LECTURE CORRELATION: FROM THE SCIENCE WRITING HEURISTIC TO THE TRADITIONAL ORGANIC CHEMISTRY LABORATORY


Jacob D. Schroeder

Abstract

The sequence of courses taken by non-science majors at a mid-western university consists of a semester of general chemistry followed by introductory organic chemistry. We have adopted the use of the Science Writing Heuristic (SWH) for the laboratory portion of one of our general chemistry courses during the past two years and have found that student performance on exams as well as critical thinking and reasoning ability when writing laboratory reports has improved. In contrast, the subsequent organic chemistry course that these students enroll in uses the traditional laboratory format, where students are asked to make a known product following a cookbook style “recipe”. After observing the organic chemistry course for the past three semesters, we have found that students coming from an SWH background are outperforming their peers who come from a more traditional background. These SWH students achieve higher exam scores, with one of the biggest differences coming from their success in answering questions on exams that are laboratory-specific. These questions include writing mechanisms, completing reactions, and identifying products. We believe that students trained in using the SWH laboratory format are better
able to utilize the laboratory experience to help them learn concepts that are covered in the lecture portion of the course.

**Introduction**

The chemical education research group at Iowa State University has implemented the Science Writing Heuristic (SWH) in some first year general chemistry laboratories. Throughout the eight semesters of implementation, the group has found a number of promising results in terms of student achievement (Burke, Greenbowe, & Hand, 2006). Students enrolled in a chemistry course that implemented the SWH format were better able to design an experiment to address a hypothesis compared to students who participated in a traditional laboratory activity, as measured on laboratory practical examinations (Greenbowe & Hand, 2005). More importantly, it has been shown that student performance on chemistry lecture examinations improves with the effective implementation of the SWH approach (Burke, Hand, Poock, & Greenbowe, 2005; Poock, Burke, Greenbowe, & Hand, accepted; Rudd, Greenbowe, Hand, & Legg, 2001). But no longitudinal studies have been completed to investigate whether this increase in student performance is lasting or carries over, especially to the second year of organic chemistry.

A substantial amount of criticism has been aimed at organic chemistry laboratory work (Baru & Mohan, 2005; Cooley, 1991; Haas, 2000; König, 2002; Mohrig, 2004; Moody & Foster, 1997; Pickering, 1985, 1988, 1991; Ruttledge, 1998; Stoub, 2004); primarily, students follow a recipe to produce a known result thus verifying a concept that was addressed during the lecture. Many of the above researchers argue that students are not challenged to reconcile results or truly understand what they are doing in these verification style laboratories. This has also shown up in the lecture portion of the course, where
Pungente and Badger (2003, p. 779) argued, “organic chemistry is viewed by some students as little more than a rite of passage, or the academic equivalent of hazing.” Taagepera and Noori (2000) found that misconceptions in organic chemistry persisted even after two years of college chemistry. With this general criticism of the organic chemistry laboratory mounting and the previous success our group uncovered in the general chemistry course, we were led to consider if these same strategies from an SWH general chemistry laboratory could be implemented in the organic chemistry laboratory. Several instructional methods have been shown to be effective in improving the value of what students actually learn in organic chemistry, including peer-led team learning (Tien, Roth, & Kampmeier, 2002; Wamser, 2006), the POGIL project (Creegan, 2006; Straumanis, 2004), active and cooperative learning (Carpenter & McMillan, 2003; Jones-Wilson, 2005; Paulson, 1999), and the incorporation of technology (Grossman et al., 2006). Many researchers have found that a research-based laboratory allows students to see practical applications of the work completed, which stimulates their interest (Amenta & Mosbo, 1994; Davis, Hargrove, & Hugdahl, 1999; Gilbert, De Jong, Justi, Treagust, & Van Driel, 2002; Newton, Tracy, & Prudenté, 2006).

A good starting point would be to simply ask the purpose of the chemistry laboratory (Hart, Mulhall, Berry, Loughran, & Gunstone, 2000). A number of reviews have been published addressing that very question (Hofstein & Lunetta, 1982, 2004; Lazarowitz & Tamir, 1994; Lunetta, 1998; Tobin, 1990). The organic chemistry laboratory manual used at Iowa State University answers this question in the following manner:

This lab course, while it presumes knowledge of organic chemistry lecture material, is intended as its own course. The purpose is to introduce various
techniques and thought processes [italics added] that practicing chemists apply to problems on a regular basis in labs around the world. Laboratory courses, unlike lecture courses, require learning manual skills and observation [sic] skills that are honed by repetition and enhanced by knowledge from science lecture courses. Attention to detail is important for success. Experiments have been designed to be relatively easy; your challenge is to get significant amounts of pure product and a thorough understanding of each manual process [italics added]. (Clague, 2005a, p. 6)

Given this emphasis on skill development rather than knowledge construction, an interesting question began to emerge as students progressed from general chemistry to organic chemistry, which led to a pilot study to address the question: Is there a carry-over effect for students who have had a prior chemistry course in which the SWH format was implemented in the laboratory? It had already been shown that these students outperformed their traditional classmates on lecture examinations (Greenbowe & Burke, 2008). Would these students continue to use SWH strategies even though they were now in a traditional laboratory? Would they perform at a higher level on the in-class examinations than students from a traditional laboratory background? If they did, then perhaps an argument could be made to extend the SWH into the organic chemistry laboratory.

**Background**

The course under study is a first year, introductory, organic chemistry course entitled Elementary Organic Chemistry (Chem 231), which is taken for three credits along with a corresponding laboratory component. The course catalog description is:
A survey of modern organic chemistry including nomenclature, structure and bonding, and reactions of hydrocarbons and important classes of natural and synthetic organic compounds. *For students desiring only an elementary course in organic chemistry.* [italics added] Students in physical or biological sciences and premedical or pre-veterinary curricula should take the full year sequence (Chem) 331 and 332. (Iowa State University, 2004)

Students who enroll in this course are not going to be doing any detailed chemistry when they graduate. As such, many students see this course as nothing more than a burden or a requirement that their own department forces upon them. In talking with several students, they appeared to approach this course with the “D for done” mentality, wanting nothing more than to just survive and pass the course.

Historically, the average examination scores have often displayed this mindset. Prior course data on examination scores throughout the semester do indicate general trends (Table 1). First, students typically perform better when they take the course in the summer. This performance difference could be attributed to the smaller class size during the summer or the pace at which the course progresses. The summer classes meet for five one-hour periods per week for eight weeks, compared to three 50-minute sessions per week for 16 weeks during the semester. Second, students generally performed better on the first examination. This is not surprising considering that a majority of the material on the first examination may be familiar to students from their general chemistry course. Finally, it seems the natural trend is in the negative direction. Some discrepancies in this trend do exist and appear to have a direct correlation to the drop deadline in the course (last day to withdraw from a course without penalty). During this time, professors generally have applied
a normal curve across the student distribution despite the actual scores. It is not surprising to see a 40% result on an examination turn into a grade of C or better, especially if the average for the class was less than 40%, as shown by the Fall 2003 students’ performance on examinations 2 and 5.

Table 1. Historical overview of average examination scores for Chem 231

<table>
<thead>
<tr>
<th>Class</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fall 2005</td>
<td>61.1</td>
<td>55.4</td>
<td>61.9</td>
<td>54.7</td>
<td>53.6</td>
</tr>
<tr>
<td>Summer 2005</td>
<td>73.0</td>
<td>59.6</td>
<td>56.5</td>
<td>70.5</td>
<td>--</td>
</tr>
<tr>
<td>Spring 2005</td>
<td>64.5</td>
<td>47.7</td>
<td>58.8</td>
<td>49.3</td>
<td>45.3</td>
</tr>
<tr>
<td>Fall 2004</td>
<td>70.9</td>
<td>55.0</td>
<td>65.1</td>
<td>62.4</td>
<td>56.6</td>
</tr>
<tr>
<td>Summer 2004</td>
<td>63.9</td>
<td>63.3</td>
<td>69.2</td>
<td>66.2</td>
<td>--</td>
</tr>
<tr>
<td>Fall 2003</td>
<td>58.8</td>
<td>38.9</td>
<td>47.9</td>
<td>54.8</td>
<td>36.7</td>
</tr>
</tbody>
</table>

Note. Data were not collected from the spring 2004 course; summer courses did not have a fifth examination.

Methodology

One of the goals of the pilot study was to examine if there were advantages in being able to translate what was learned from laboratory activities to lecture examinations for the students who had previous SWH experiences. The organic chemistry laboratory focused primarily on introducing students to new techniques, in addition to striving for obtaining high percentage yields. Topically, the laboratory syllabus at times did coincide with the lecture syllabus, although this was not intentional as the laboratory manual stated that the laboratory was to be considered as its own course. Many of the concepts covered in the laboratory were also covered in the lecture course at the same time, inevitably ending up on the lecture
course’s hour examinations. This study focused on those questions that were laboratory specific, since many of the reactions the students were asked to complete on the hour examinations in lecture were nearly identical to the reactions that they had done in the laboratory.

The students in Chem 231 consisted of a collection of 118 students who had each taken a prior chemistry course – the three most common being one of the large, general chemistry courses: Chem 163, Chem 164, or Chem 178. In addition, several students were taking this course for a second or more time, while others had transferred from either a community college or another university. Students from the Chem 163 course had prior training with the SWH laboratory format. This group of 18 students is hereafter referred to as the SWH group. Another group of 18 students was selected based on their equal performance to the SWH group on the first in-class examination. Each student in this group, labeled as the TRAD group, had taken a chemistry course the preceding spring semester in which the traditional laboratory format was used. These prior courses included Chem 164, the second semester sequence of Chem 163 specifically designed for non-science majors, and Chem 178, considered to be the general chemistry course for science and engineering majors. Students were not selected if they had taken the organic chemistry course (Chem 231) or if they had not taken a chemistry course the prior semester. With the different levels and requirements of each prior chemistry course, it was believed that prior grades for different courses were not comparable. In other words, an A earned in a lower level course (Chem 163, Chem 164) could not be translated into a B in the upper level course (Chem 178). In addition, no standardized testing took place in Chem 231, which made setting a baseline at the beginning of the term nearly impossible.
Data were collected by analyzing student responses on in-class examinations that were given throughout the semester. Typically, these examinations were given every two weeks; and some subject matter related to the two laboratory experiments performed during that time. Examination questions directly relating to the laboratory experiments were pinpointed as a method of comparison between the two groups of students. Scores on these questions were averaged based on the total score possible. Statistical analysis was performed using the Data Analysis toolkit in Microsoft Excel®. A two-tailed $t$-test was performed assuming unequal variances; a $p$-value less than 0.05 was deemed as significant.

**Results**

The average scores for all five examinations are shown in Table 2, with $N$ representing the number of students in each group and $SD$ the standard deviation. The statistical analysis is shown in the lower half of the table, with values for degrees of freedom (DF), the $t$-statistic ($t$-stat), and the probability of obtaining a larger $t$-value on a two-tailed $t$-test ($p > t$).

Having started with equal scores on examination 1, the two groups start on a divergent pathway with examination 2 that continues throughout all five examinations. On the last four examinations, the major difference between the two groups can be attributed to one page on each examination that asked students to either complete a reaction by writing the correct structure of the product or by circling the most stable product if multiple products can form. Starting with examination 2, the SWH group of students outperformed the TRAD group 60.5 to 52.6% ($t = 1.07; p > t = 0.29$) for the entire examination. In terms of laboratory-specific questions, two reactions stand out. The first reaction is a dehydration reaction involving the use of sulfuric acid to form an alkene.
Table 2. Average examination scores for the two groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Examination Number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
</tr>
<tr>
<td>SWH</td>
<td>60.1</td>
</tr>
<tr>
<td>N</td>
<td>18</td>
</tr>
<tr>
<td>SD</td>
<td>15.8</td>
</tr>
<tr>
<td>TRAD</td>
<td>60.1</td>
</tr>
<tr>
<td>N</td>
<td>18</td>
</tr>
<tr>
<td>SD</td>
<td>15.7</td>
</tr>
</tbody>
</table>

**Statistical Analysis**

<table>
<thead>
<tr>
<th>Examination Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
</tr>
<tr>
<td>DF</td>
</tr>
<tr>
<td>t-stat</td>
</tr>
<tr>
<td>p &gt; t</td>
</tr>
</tbody>
</table>

**Notes**

<sup>a</sup> Prior to examination 3, one student from this group withdrew from the course.

<sup>b</sup> Two students did not take the examination.

<sup>c</sup> One student did not take the examination.

* Statistically significant

As shown in Figure 1, the *cis* isomer has the two carbon-bearing groups on the same side of the double bond. The *trans* isomer has these two groups on opposite sides of the double bond. Although the possibility of forming both products exists, students received full credit for their answer only if they circled the *trans* isomer. Partial credit was given to students who circled the *cis* isomer. Full credit for this question was worth three points. Of the 18 SWH students completing the reaction, 15 (83%) did so correctly, compared to 11 correct responses from the 18 TRAD students (61%). The SWH group averaged a score of 2.5 for this problem, compared to 1.8 for the TRAD group (*t* = 1.49, *p > t* = 0.145).
The second reaction is an oxidation of an alkene using potassium permanganate. This reagent is used to convert alkenes into diols (two OH groups). This question was worth four points, with full credit awarded if students were able to draw the diol (stereochemistry omitted). On this question, 7 SWH students gave correct responses (39%), compared to only 1 from the TRAD students (5%). For this question, the SWH group obtained an average score of 1.6, compared to 0.2 for the TRAD group ($t = 2.56, p = 0.018$).

During the two weeks preceding examination 2, students performed two laboratory experiments; the first involved the dehydration of an alcohol to form an alkene. This experiment, entitled *The Preparation of 4-methylcyclohexene: Dehydration of an Alcohol*, consisted of two parts (Clague, 2005b). In the first part, students are given the reaction scheme representing what they will be doing. In the second part, students are told about the qualitative unsaturation test using potassium permanganate. Both processes are illustrated schematically in Figure 2.

![Figure 1. Examination 2 laboratory-specific questions](image)
Two weeks after examination 2, students took examination 3. The SWH group outperformed the TRAD group by almost 20 points ($t = 2.83, p = 0.008$). Once again, the differences between the two groups can be attributed to student performance on the reaction page of the examination. The reaction page consisted of six problems, each worth four points. Again, students were asked to write the expected product when given a set of reaction conditions. To focus on laboratory-specific questions, three questions from the examination are shown with the correct answers in Figure 3.

Both the Jones reagent and PCC (pyridinium chlorochromate) are chromium compounds capable of oxidizing an alcohol functional group to a carbonyl group. The Jones
reagent is used to convert a primary alcohol into a carboxylic acid (first reaction) and a secondary alcohol into a ketone (third reaction). PCC will convert the primary alcohol into an aldehyde (second reaction).

For the first reaction, 12 of the SWH students (71%) gave correct responses, compared to 3 of the TRAD students (18%). On this question, SWH students obtained an average of 2.8 compared to 0.8 for the TRAD students ($t = 3.35, p = 0.002$). For the second reaction, 11 of the SWH students gave correct responses (65%), compared to 1 correct response for the TRAD students (6%). On this question, SWH students obtained an average of 2.6 compared to 0.4 for the TRAD students ($t = 4.12, p < 0.001$). On the third reaction, 10 of the SWH students were correct (59%), compared to 1 correct response for the TRAD students (6%). The average point totals for this question were 2.4 for the SWH group and 0.5 for the TRAD group ($t = 3.35, p = 0.003$).

In the laboratory preceding examination 3, students performed an experiment entitled *Analysis and Identification of Aldehydes, Ketones, and Alcohols* (Clague, 2005c). This experiment generally follows the protocol of identifying an unknown mystery solution as one of the three types of functional groups mentioned in the title. Students are expected to complete a series of chemical tests to help them narrow down the choices. In one step of the procedure, they are told:

Primary and secondary alcohols can be oxidized to form carbonyl (C=O) compounds, using [the] Jones reagent (Cr$^{+6}$). This is due to the relative ease with which the carbon-hydrogen bond is broken. The rate at which the alcohol reacts is the basis for determining whether the alcohol is primary or secondary. In contrast, carbon-carbon bonds are not easily broken; thus, tertiary alcohols are not oxidized. (Clague, 2005c, p. 214)
Figure 4. Information given in the laboratory preceding exam 3

Figure 4 illustrates what happens to three different types of alcohols upon reacting with the Jones reagent (CrO₃). An R group attached to a carbon signifies a general representation of any other carbon-containing group. With one R group attached, the first reaction involves a primary alcohol and, upon reacting with the Jones reagent, becomes converted to a carboxylic acid. The addition of a second R group to the alcohol in the second reaction produces a secondary alcohol that, upon reacting with the Jones reagent, is converted to a ketone. With three R groups attached in the third reaction, a tertiary alcohol is formed that will not react with the Jones reagent. Tests using PCC were not performed in the laboratory; however, in the textbook for the course (Ouellette, 1998), the two reagents are used together to discuss the concept of alcohol oxidation.

Two weeks after examination 3, students took the fourth examination. The SWH group outperformed the traditional group by 12.6 percentage points, but this difference did not show statistical significance ($t = 1.70; p = 0.099$). Just as in the prior examinations, the main difference in performance is attributed to the reaction page. There were ten reactions; each was worth four points. Again, students were asked to predict the product of a reaction when given a set of reaction conditions.
The laboratory-specific questions are shown in Figure 5. These three reactions test the concept of the reduction of a carbonyl compound to form an alcohol. In the first reaction, hydrogen and nickel are used to reduce both the double bond at the end of the chain as well as the aldehyde on the other end. For this reaction, 13 of the SWH students answered correctly (76%), compared to 4 of the TRAD students (24%). In terms of average score, the SWH group obtained 3.3, compared to 2.0 for the TRAD group ($t = 2.68, p = 0.012$). Lithium aluminum hydride ($\text{LiAlH}_4$) is used in the second reaction. This reagent will reduce all carbonyl groups. For this reaction, 14 of the SWH students answered correctly (82%), compared to 9 TRAD students (53%). In terms of average score, the SWH group obtained 3.4 compared to 2.5 for the TRAD group ($t = 1.71, p = 0.097$). Sodium borohydride ($\text{NaBH}_4$) is used in the third reaction and is a reagent that can only reduce ketones and aldehydes. For this reaction, 13 of the SWH students answered correctly (76%), compared to 8 of the TRAD students (47%). The SWH group obtained an average of 3.1 compared to 2.5 for the TRAD group ($t = 1.01, p = 0.322$).

In the laboratory preceding examination 4, students completed an experiment entitled *Sodium Borohydride Reduction of Benzophenone & para-Tolualdehyde* (Clague, 2005d). The
experiment focused on the use of sodium borohydride as a popular reagent for reducing the carbonyl group of aldehydes and ketones to form primary and secondary alcohols. After a brief paragraph describing how the reducing properties of sodium borohydride were discovered by accident, two reactions were drawn to illustrate the reaction of sodium borohydride with a ketone and an aldehyde (Figure 6).

![Figure 6. Information given in laboratory preceding examination 4](image)

The manual also highlights the difference between sodium borohydride and lithium aluminum hydride: “It (LiAlH₄) reacts violently with water releasing hydrogen gas. It is a more powerful but less selective reducing agent than sodium borohydride in that it can reduce aldehydes, ketones, carboxylic acids, and esters into alcohols [italics added]” (Clague, 2005d, p. 200). Two more reactions are drawn to illustrate this point. Interestingly, one of the questions at the end of the laboratory asks what compound will be formed when each of the following compounds is reduced with sodium borohydride:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-butanone</td>
<td><img src="image" alt="2-butanone" /></td>
</tr>
<tr>
<td>Benzenaldehyde</td>
<td><img src="image" alt="Benzenaldehyde" /></td>
</tr>
<tr>
<td>Butyraldehyde</td>
<td><img src="image" alt="Butyraldehyde" /></td>
</tr>
<tr>
<td>Benzophenone</td>
<td><img src="image" alt="Benzophenone" /></td>
</tr>
</tbody>
</table>

Structurally, butyraldehyde resembles the actual question that was asked on the examination. Despite this similarity, less than half of the 17 TRAD students were able to answer that question correctly.
The final examination, consisting of half comprehensive and half new material, was given at the end of the semester, three weeks after the last laboratory was completed. Unfortunately, the new material covered in the course did not have corresponding laboratory experiments. For this reason, a detailed analysis for the two groups was not completed. However, the difference in performance between the two groups should be noted. The SWH group obtained an average of 59.7% compared to 40.8% obtained by the TRAD group ($t = 2.39$, $p = 0.024$).

**Discussion**

Of the three examinations analyzed, the SWH group of students performed reasonably well on the reaction pages and overall on the final examination. However, the TRAD group of students did not match this performance. The main factor in choosing the two groups was performance on examination 1. Even though the two groups started out statistically equal, they never again were on the same footing. Despite the sometimes very wide margin between the two groups, significant differences were only observed for examinations 3 and 5. This is due in part to the large variances and small populations of each group.

Of the eight questions analyzed that corresponded to laboratory work, the SWH group consistently outperformed the TRAD group. On five of the eight reactions, these differences were shown to be significant. These data would indicate that the SWH group outperformed their traditional counterparts, even though the corresponding laboratory was taught in a traditional manner. Considering that this same SWH group showed statistical gains in their general chemistry classes the preceding semester, perhaps it is not surprising to see this group learning more from the laboratory in a subsequent course. It is possible that this shows a
“carryover” effect, but this cannot be said without having any written student laboratory reports. An argument could be made that, once trained to use the SWH, students can see their improvement when taking examinations. Since organic chemistry is perceived to be “harder”, students may very well have used the SWH structure when it came to writing laboratory reports.

The laboratory was divided into seven sections, all taught by graduate teaching assistants and following a traditional format. With different teaching assistants responsible for different sections, there could be a teacher effect. However, both groups of students were scattered among all seven sections. No one laboratory section was found to perform statistically higher than any other section on the in-class examinations. Similarly, these students’ prior courses were all taught by different instructors, each no doubt emphasizing different topics more than others. What is certain is that, for the organic chemistry course studied, students with an SWH background outperformed students with a traditional background. This pilot study provides ample evidence to suggest that the SWH students are applying what they encountered in the laboratory to the in-class examinations.

To further address some of these questions, it would be better to track all students who took a general chemistry course during the preceding semester as opposed to the small groups focused on in this pilot study. In addition, laboratory notebooks should be analyzed to determine how well students are writing conclusions and analyses based on the data they obtain. It may then be possible to state that there is a direct correlation between the laboratory and the lecture.

With the success of the students with a SWH background, perhaps it is time to consider implementing the SWH or one of the other methods reported in the literature in
organic chemistry laboratories. With time, the criticism against the traditional laboratory format will only continue to grow. With the proper assessment strategies, we would be able to say for certain that use of the SWH was the main reason these students performed so well on the laboratory-specific questions.

References


the Royal Society of Chemistry Web site:


Abstract

Students in a traditional laboratory have often received good marks for their written laboratory reports, but there appears to be no correlation between these scores in the laboratory and scores in the lecture. An analysis of the traditional laboratory manual that had been in use for an introductory organic chemistry laboratory revealed that all answers and solutions were provided to the student in advance, which led the authors to believe, led to the lack of a correlation. In response the authors have converted the traditional laboratory manual for organic chemistry into a collaborative guided inquiry laboratory manual that implements the Science Writing Heuristic (SWH) approach and laboratory report format. This article describes the conversion of a traditional organic chemistry laboratory into a guided-inquiry laboratory that implements the SWH approach and compares student performance on lecture exams after using the SWH to a traditional group of students enrolled in the same course during a different term. The experiments described are meant to be more practical, relating to issues of health, medicine, and industry. Rather than verifying known outcomes, students instead are asking testable questions, performing experiments, and
solving problems based on the data they obtain. Essentially, they are using the scientific method.

**Introduction**

Several years ago, we reported the process of converting a traditional general chemistry laboratory into a collaborative guided-inquiry laboratory that utilized the Science Writing Heuristic (SWH) approach and laboratory report format (Rudd, Greenbowe & Hand, 2001). This resulted in a corresponding improvement in student attitudes toward chemistry and an increase in the level of student performance in the lecture (Burke, Greenbowe & Hand, 2006). However, these gains quickly flattened once these same students enrolled in the subsequent introductory organic chemistry course. Many students not majoring in chemistry do not anticipate taking introductory organic chemistry because it has developed a reputation of being the make-or-break course in many interdisciplinary programs. The responses to a survey given at the beginning of the semester reveal roughly 60 – 70% of students say they expect organic chemistry to be one of the most difficult courses they will have to take during college.

Part of the problem could be in how the introductory organic chemistry course has been taught in the past, with a corresponding laboratory that follows the traditional format. Many educators have argued that this format does little to actively engage students (Horowitz, 2007; Domin, 1999; Bodner, Hunter & Lamba, 1998), having devolved into nothing more than a place where students spend three hours trying to replicate known results. If students do not obtain the expected results, they quickly become frustrated, noting in their laboratory reports that some sort of human error had skewed their results (Pickering, 1985).
Another reason why students dislike organic chemistry could be the lack of “real-world” applications to which they can relate it.

**History**

Since our initial article (Rudd, et al., 2001), subsequent studies have shown that implementing the SWH laboratory format effectively has resulted in a significant increase in student understanding in the lecture, as measured by in-class exams, quizzes, and on an American Chemical Society standardized examination (Burke, Greenbowe & Hand, 2006; Poock, Burke, Greenbowe & Hand, 2007). To further these efforts, the students who participated in those studies were tracked to see whether the gains they made in general chemistry would be maintained during the introductory organic chemistry lecture and laboratory (Schroeder, 2007). A pilot study revealed that performance on introductory organic chemistry exams for students who had used the SWH laboratory approach in general chemistry was significantly better than that of students who had used the traditional laboratory format in general chemistry. Yet overall student performance for both groups of students in organic chemistry was consistent with previous semesters. Analysis of lecture examinations showed that many students performed poorly on questions directly related to material that was introduced in the laboratory.

Much has been reported in the literature about making laboratory work more practical (Stanger-Hall, K., Merriam, J. & Greuling, R.A., 2007; Pelter, 2006), specifically by designing experiments in the context of a real-world scenario, such as cases, puzzles, or mysteries. These experiments put students in the role of scientists, investigating chemistry concepts and applying experimental results within the context of the scenario presented to them.
This process began with three primary issues to address: the timing of the experiments, the duration of the pre-laboratory period, and the context of the experiments. In the traditional laboratory format, students were introduced to new topics in the lecture well before these topics were introduced in the laboratory. DiBiase and Wagner (2002) reported significant increases in student performance on lecture exams after aligning the laboratory with the lecture and incorporating inquiry-based experiments. Aligning the lecture with the laboratory allows for a discussion of concepts in both laboratory and lecture, affording students the opportunity to see the connection between the two, providing context and relevance to both.

Jalil (2006) reported that experimenting first and using the experimental data as a backdrop for a discussion better matched the majority of students’ preferences regarding their understanding, enjoyment, and positive feeling of certain achievement while performing the experiments when compared to a more traditional approach. A long pre-laboratory session can take time away from actual experimental work, leaving many students unable to finish the experiment in the allotted time. Without a post-laboratory discussion, students are left to find meaning for the experiment on their own. To this end, Amato has argued that introductory chemistry courses have lost touch with the excitement of the field and become a trial for students and often an embarrassment for professors (Amato, 1992).

**General Modifications**

The experiment schedule for both the traditional and SWH laboratory is shown in Table 1. Because University holidays fell on a Monday during one week of classes in both the fall and spring term, every section scheduled during that holiday week was cancelled. As
a result, the next two experiments (TLC and extraction) introduced concepts such as polarity and density nearly three weeks after they were initially discussed in the lecture.

Table 1: Experiment comparison between traditional and SWH

<table>
<thead>
<tr>
<th>Week</th>
<th>Traditional Experiment</th>
<th>SWH Experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Check in to assigned locker</td>
<td>Check in to assigned locker</td>
</tr>
<tr>
<td>2</td>
<td>Recrystallization and melting points</td>
<td>Techniques: A glimpse into the chemists toolbox</td>
</tr>
<tr>
<td>3</td>
<td>No experiment (Labor Day Holiday)</td>
<td>Which beverage contains the most caffeine?</td>
</tr>
<tr>
<td>4</td>
<td>Thin layer chromatography (TLC)</td>
<td>Making margarine in the chemistry laboratory</td>
</tr>
<tr>
<td>5</td>
<td>Isolation of caffeine from tea and coffee</td>
<td>Alkenes: Gathering a wealth of evidence to support claims</td>
</tr>
<tr>
<td>6</td>
<td>Preparation of 4-methylcyclohexene</td>
<td>Electrophilic aromatic substitution: The effect of directing groups on benzene</td>
</tr>
<tr>
<td>7</td>
<td>Friedel-Crafts alkylation of 1,4-dimethoxybenzene</td>
<td>Molecular models: The world of 3-dimensional chemistry</td>
</tr>
<tr>
<td>8</td>
<td>Structural formulas and molecular modeling</td>
<td>Substitution vs. elimination: The chemical competition</td>
</tr>
<tr>
<td>9</td>
<td>Nucleophilic substitution: Preparation of 2-chloro-2-methylpropane</td>
<td>Clean chemistry: From margarine to soap</td>
</tr>
<tr>
<td>10</td>
<td>Sodium borohydride reduction of benzophenone</td>
<td>Artificial scents: The chemistry of fragrances</td>
</tr>
<tr>
<td>11</td>
<td>Analysis and identification of aldehydes, ketones, and alcohols</td>
<td>Sodium borohydride: From an accident to H₂ fuel cells</td>
</tr>
<tr>
<td>12</td>
<td>Preparation of esters</td>
<td>The Aldol condensation: The carbon construction company</td>
</tr>
<tr>
<td>13</td>
<td>Preparation of a soap</td>
<td>Chemical inventory: Preparing for an EPA inspection</td>
</tr>
<tr>
<td>14</td>
<td>No experiment (Thanksgiving break)</td>
<td>No experiment (Thanksgiving break)</td>
</tr>
<tr>
<td>15</td>
<td>Preparation and application of azo dyes</td>
<td>Lab practical examination</td>
</tr>
<tr>
<td>16</td>
<td>Written lab final examination; check out of lockers</td>
<td>Check out of lockers</td>
</tr>
</tbody>
</table>

To avoid this delay, the schedule was reorganized so that students who were scheduled for the Monday laboratory section were asked to perform the experiment during
another session that met during the week. Students were given an excused absence if they
could not reschedule, but almost all students found another section that had adequate space
for them. This one change in timing kept every other experiment synchronized with the
lecture. In many cases, the Monday and Tuesday sections were introduced to new concepts
in the laboratory before the ideas were introduced in the lecture on Wednesday.

To address the issue of content delivery and the duration of the pre-laboratory session,
all experiments were written in a narrative style suggesting several possible outcomes. Since
the correct products or answers were not given, students would ask testable questions about
the scenario during the pre-laboratory session. In the traditional laboratory, the title of the
experiment usually provided the purpose and often the outcome of the experiment (i.e., The
prepartion of…). Renaming the experiment allowed the laboratory to take on an element of
discovery, as students quickly realized they would be performing an experiment to help them
answer the questions they posed during the pre-laboratory session.

The majority of students who enroll in the course are health science, nutrition,
agricultural, or pre-pharmacy majors. The experiments were written in a manner to show
applications of the concepts to these fields. In general, all twelve experiments were written
in the following format:

• **Introduction**—one or two brief paragraphs outlining the scenario, introducing the
  problem, how it relates to prior experiments, and how a scientific experiment could
  be performed that would enable students to generate conclusions based on evidence
  collected and analyzed during an experiment.
• *The Big Idea*—why the issue needs to be addressed, where the concepts become relevant to not only people in the field, but to society at large. This section serves to show the larger picture, i.e., why the issue is worthy of scientific exploration.

• *Analysis*—how chemistry can be used to provide evidence to support any claims made with regard to the question(s) under study; what tools chemistry can offer to help students generate conclusions (IR, melting points, $^1$H NMR, etc.)

• *Before the Lab*—questions for students to consider before they come to the laboratory. This section helps students formulate beginning questions that they can answer by performing the experiment.

• *After the Lab*—questions or ideas for students to consider after they have completed the experiment. This section serves to provide context, allowing students to explore the applicability of the experimental results to larger issues with which they may already be familiar (*trans* fats, artificial flavorings, fuel cells, antioxidants, etc.). Students are encouraged to consult the Internet or any other sources of information that could help to provide some meaning to what took place during the experiment.

Two of the traditional experiments (*Thin Layer Chromatography* and *Isolation of Caffeine*) were combined into one (*Which beverage contains the most caffeine?*). This allowed even further flexibility, featuring two important concepts in the context of one laboratory experiment. This allowed for the incorporation of *Making Margarine in the Chemistry Lab*, an experiment not offered during the traditional laboratory. Alkenes are usually introduced in the lecture during this time, with an emphasis on *cis* and *trans* double bonds. Therefore, the experiment highlights topics with which students are already familiar—partially hydrogenated vegetable oils and *trans* fats. The other major difference in the schedule was
the elimination of *The Preparation and Application of Azo Dyes* experiment, since the topic was never discussed in the lecture. This provided the opportunity to replace the written multiple-choice laboratory final with a laboratory practical examination.

**Rationale**

The primary reason for reforming the laboratory was the large discrepancy in student scores in the laboratory versus their scores in the lecture. The last semester the traditional laboratory was offered, student scores on written laboratory reports were exceptional. Combining the laboratory report scores for all 11 experiments, we found the overall average percentage score on the laboratory reports was 91.8% (202 points out of 220 maximum). Despite this performance in the laboratory, student scores in the lecture were often much lower, even when laboratory concepts were included on the lecture exams shortly after an experiment was completed.

**Methods and participants**

This study compared the performance of two different groups of students taking the same introductory organic chemistry during two different spring terms. The control group consisted of students who took the course during the Spring 2006 term, the last time the traditional laboratory format was implemented. Total enrollment for this group was 104. The experimental group consisted of students enrolled during the Spring 2007 term when the SWH laboratory format was implemented. Total enrollment in this group was 115. Student performance data on specific lecture exam questions will be presented including three topics: dehydration, nucleophilic substitution, and electrophilic aromatic substitution. Student performances on these three subjects will be compared to their performance on the corresponding laboratory report.
Results

Dehydration

During the Spring 2006 term, students completed the laboratory experiment Preparation of 4-methylcyclohexene, treating 4-methylcyclohexanol with a combination of sulfuric and phosphoric acids to produce 4-methylcyclohexene. The average score on the laboratory report was 18.6 out of 20 (93%), with 33 of the 104 students obtaining a perfect score (31.7%). Two weeks after completing the experiment in the laboratory, students were asked to predict the product of a dehydration reaction on a lecture exam (Figure 1). Of the 33 students who received a perfect score on the laboratory report, 11 drew the correct product for the reaction on the exam (33%).

\[
\text{CH}_3\text{CH}_2\text{CH(OH)CH}_3 \xrightarrow{\Delta, \text{H}_2\text{SO}_4} \]

Figure 1: Dehydration reaction on Spring 2006 exam

During the Spring 2007 term, students completed the laboratory experiment Alkenes: Gathering a Wealth of Evidence to Support Claims, following the same experimental procedure as used for the dehydration experiment during the Spring 2006 term, but from the new laboratory manual. The average score on the laboratory report was 16.5 out of 20 (82.5%), with 14 of the 115 students obtaining a perfect score (12.2%). Three weeks after completing the experiment, students were asked to predict the product of a dehydration reaction on a lecture exam (Figure 2). Of the 14 students who received a perfect score on the laboratory report, 12 (85.7%) drew the correct structure of the product for the dehydration reaction on the exam.
During the Spring 2006 term, students completed the laboratory experiment *Nucleophilic Substitution: $S_N1$ and $S_N2$ Reactions*. Students were responsible for two reactions: treating $t$-butyl alcohol with hydrochloric acid to produce $t$-butyl chloride, and treating 1-butanol with a combination of hydrobromic and sulfuric acids to produce 1-bromobutane. However, due to a shortage of hydrobromic acid, the second reaction was omitted. The average score on the laboratory report was 18.1 out of 20 (90.5%), with 31 of the 104 students obtaining a perfect score (29.8%). One week after completing the experiment in the laboratory, students were asked on an hour exam to predict the product for two substitution reactions (Figure 3). Of the 31 students who received a perfect score on the laboratory report, 9 drew the correct product for the first reaction, and 9 drew the correct product for the second reaction on the exam (29.0%).

(1) Draw the configuration(s) of the $S_N2$ reaction product(s) of (R)-CH$_3$CHBrCH$_2$CH$_3$ + OH

(2) Draw the configuration(s) of the $S_N1$ reaction product(s) of (R)-CH$_3$CHBrCH$_2$CH$_3$ with H$_2$O

During the Spring 2007 term, students completed the laboratory experiment *Substitution vs. Elimination: The Chemical Competition*, following the same experimental procedure as used for the substitution experiment during the Spring 2006 term, but from the new laboratory manual. Because of the inconsistency of the substitution of 1-butanol, that
experiment was omitted from the new manual. The average score on the laboratory report was 17.2 out of 20 (86%), with 14 of the 115 students obtaining a perfect score (12.2%). One week after completing the experiment, students were asked to predict the product of two nucleophilic substitution reactions on the lecture exam (Figure 4). Of the 14 students who received a perfect score on the laboratory report, 11 (78.6%) drew the correct structure of the product for the first reaction and 10 (71.4%) drew the correct product for the second reaction on the exam.

---

**Figure 4: Nucleophilic substitution reactions on Spring 2007 exam**

**Electrophilic Aromatic Substitution**

During the Spring 2006 term, students completed the laboratory experiment *Aromatic Substitution: Friedel-Crafts Alkylation and Nitration*. But due to time constraints, only the nitration experiment was performed. Students were responsible for treating methyl benzoate with a mixture of sulfuric and nitric acids to produce m-nitromethylbenzoate. The average score on the laboratory report was 17.5 out of 20 (87.5%), with 21 of the 104 students obtaining a perfect score (20.1%). Two weeks after completing the experiment in the laboratory, students were asked to predict the product for four electrophilic aromatic substitution reactions on the lecture exam. For the purposes of comparison, two are shown (Figure 5).
Of the 21 students who received a perfect score on the laboratory report, 3 (14.3%) drew the correct products for the halogenation reaction (1) and 11 (52.3%) drew the correct product for the alkylation reaction (2).

During the Spring 2007 term, students completed the laboratory experiment *Electrophilic Aromatic Substitution: The Effect of Directing Groups on Benzene*, following the same experimental procedure as used for the nitration of methyl benzoate during the Spring 2006 term without the alkylation reaction, but with a separate experiment for the bromination of acetanilide. The average score on the laboratory report was 17.3 out of 20 (86.5%), with 25 of the 114 students obtaining a perfect score (21.9%). One week after completing the experiment, students were asked to predict the product(s) of several electrophilic aromatic substitution reactions on the lecture exam (Figure 6). Two were chosen for a comparison to the Spring 2006 term. Of the 25 students who received a perfect score on the laboratory report, 5 (20%) drew the correct structures of the products for the halogenation reaction (1) and 16 (64%) drew the correct product for the alkylation reaction.
**Discussion**

The overall average percentages for student laboratory reports are shown for both groups in Table 2, along with the percentage of students within each group who obtained a perfect score on the laboratory report. The data show that for all three experiments the Spring 2006 students, using a traditional laboratory format, scored higher on the laboratory reports and with the exception of the aromatic substitution experiment, produced a larger percentage of perfect scores.

**Table 2: Spring 2006 and 2007 laboratory report comparison**

<table>
<thead>
<tr>
<th></th>
<th>Dehydration</th>
<th>Nucleophilic Substitution</th>
<th>Electrophilic Aromatic Substitution</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Spring 2006</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Overall class average (%)</td>
<td>93.0</td>
<td>90.5</td>
<td>87.5</td>
</tr>
<tr>
<td>Percentage of perfect scores</td>
<td>31.7</td>
<td>29.8</td>
<td>20.1</td>
</tr>
<tr>
<td><strong>Spring 2007</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Overall class average (%)</td>
<td>82.5</td>
<td>86.0</td>
<td>86.5</td>
</tr>
<tr>
<td>Percentage of perfect scores</td>
<td>12.2</td>
<td>12.2</td>
<td>21.9</td>
</tr>
</tbody>
</table>

Those students who received perfect scores on their laboratory report are further evaluated by how well they did on class lecture exams, with regard to questions specifically related to the laboratory (Table 3). The data from Table 3 show the opposite trend, with a
much higher percentage of students in the Spring 2007 group successfully answering related questions on the class lecture exam.

*Table 3: Percentage of correct answers on related questions on lecture exams by students who obtained a perfect score on their laboratory report.*

<table>
<thead>
<tr>
<th></th>
<th>Dehydration</th>
<th>Nucleophilic Substitution</th>
<th>Electrophilic Aromatic Substitution</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Spring 2006</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Number of students</td>
<td>33</td>
<td>31</td>
<td>21</td>
</tr>
<tr>
<td>with a perfect</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>laboratory report</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>score</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Percentage of correct</td>
<td></td>
<td>(1) 29.0</td>
<td>(1) 14.3</td>
</tr>
<tr>
<td>answers on</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>the lecture exam</td>
<td>33</td>
<td>(2) 29.0</td>
<td>(2) 52.3</td>
</tr>
<tr>
<td>Number of correct</td>
<td>[11 students]</td>
<td>[9 students each]</td>
<td>[3 for (1); 11 for (2)]</td>
</tr>
<tr>
<td>exam answers</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Spring 2007</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Number of students</td>
<td>14</td>
<td>14</td>
<td>25</td>
</tr>
<tr>
<td>with a perfect</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>laboratory report</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>score</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Percentage of correct</td>
<td></td>
<td>(1) 78.6</td>
<td>(1) 20.0</td>
</tr>
<tr>
<td>answers on</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>the lecture exam</td>
<td>85.7</td>
<td>(2) 71.4</td>
<td>(2) 64.0</td>
</tr>
<tr>
<td>Number of correct</td>
<td>[12 students]</td>
<td>[11 for (1); 10 for (2)]</td>
<td>[5 for (1); 16 for (2)]</td>
</tr>
<tr>
<td>exam answers</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

These conflicting data can be put in context by analyzing the students’ written laboratory reports (Appendix C). Sample laboratory reports (one written in the traditional format, the other written in the SWH format) are shown for each of the three experiments. These may highlight why some students are consistently scoring higher on the reports, but lower on the lecture exam. Every laboratory experiment undertaken during the Spring of 2006 followed a specific grading rubric (Appendix C, p.225). But by comparing this rubric to the laboratory manual and to student laboratory reports, it becomes evident why the students in the Spring 2006 group are obtaining higher scores than their SWH counterparts in the Spring 2007 group. Looking through the traditional laboratory manual, one can easily
find the *answers* to the fill-in-the-blank grading rubric. When looking at the dehydration experiment in the laboratory manual, the reaction scheme is given immediately after the first paragraph, showing the student what reaction is going to take place, what product is going to form, how it is going to form (reaction mechanism), and how to calculate the theoretical and percent yield. When the laboratory manual is compared to one of the laboratory reports that received a perfect score, this fill-in-the-blank procedure becomes evident. In the sample student laboratory report provided on p. 227, the *Purpose* replicates the information provided by the first paragraph in the laboratory manual. The reaction scheme, reaction mechanism, reagent table, and the calculation for theoretical yield and percent yield all follow this. The laboratory manual provides a detailed description for analyzing the product using IR spectroscopy, including peaks to use to verify that the correct product was obtained. This same conclusion offered by the laboratory manual can be found in the student laboratory report as well. Based on the grading rubric and the information supplied in the laboratory manual, the only information supplied solely by the student appears to be the masses of the starting material and the product.

When compared to a laboratory report written with the SWH format, the differences become clear. No *answers* are given anywhere in the procedure—instead students needed to compile all of the available evidence gathered during the course of the experiment to deduce the answers. In addition, no formula shows students how to calculate the percent yield, the reaction mechanism is not supplied, and there are no detailed conclusions offered by the laboratory manual. This required the student to supply the evidence, piece by piece, in order to support the claim of what the structure of the product is. In the SWH laboratory report (Appendix C, pp. 229-234), it should also be noted that for the dehydration experiment, the
student obtained an IR reference spectra for comparison. After putting all of the evidence together, the student then reported in the Reading and Reflection, “I felt like a detective”. For the reaction in the Reading and Reflection concerning which product would most likely form, the student not only chose the correct product, but also was able to articulate why by saying, “The double bond on product B has more substituents than A, therefore it is a more stable product”.

These same patterns can be found on the laboratory reports for nucleophilic substitution. The student who wrote the traditional laboratory report never mentioned the terms SN1 or SN2 outside of the purpose, and no comparison was made between the two. No factors were listed to show that the student understood the difference between the two possible pathways. Instead, the laboratory report again looked very similar to the answers provided by the laboratory manual. By comparison, the level of detail provided by the student in the SWH laboratory showed again how all of the available evidence could be gathered from the experiment and used to conclude what the structure of the product was. It was also evident that a significant amount of reading took place for the Reading and Reflection; SN1 and SN2 reactions were compared to each other and to elimination reactions, with a detailed discussion about the factors favoring each pathway.

In the case of electrophilic aromatic substitution, the same patterns emerged for the traditional laboratory report. Although the student did a good job analyzing the results, nowhere in the report was there any mention of directing groups. By comparison, the student using the SWH laboratory format made it clear from the Beginning Questions what the class was trying to find out from the experiment. By using two starting materials with different directing groups, the class could make claims based on the results obtained when everyone
finished their experiment. This was shown in the SWH laboratory report when the student claimed, “As a result of our experiment, I was able to answer the beginning questions”. The student explained the difference between activating and deactivating groups providing a mechanism for both of the reactions performed.

**Conclusions**

Students in a traditional laboratory course often received very good scores on their written laboratory reports. But there appeared to be no correlation between the scores on these laboratory reports with those in the lecture, as measured by in-class exams. This lack of correlation led the authors to convert the traditional laboratory into a collaborative guided inquiry laboratory implementing the Science Writing Heuristic (SWH). The result has been a slight decrease in average scores on the laboratory reports, but a noticeable increase in student performance on lecture exam questions specifically related to the concepts introduced in the laboratory. In each of the three experiments mentioned in this article, the traditional laboratory report supplied all information that students would need in order to properly carry out the experiment, but in so doing, failed to challenge students to think about what they were doing and how it could be applied to broader contexts. Without all of the answers given in detail in the SWH laboratory manual, students were forced to use experimental data and observations to make claims about what happened in the laboratory and to provide evidence from a variety of laboratory procedures to justify them. This allowed students to work collectively, fostering a community within the classroom. In addition, by having students articulate for themselves the results of laboratory work rather than rely on the laboratory manual, they took on a sense of ownership of the information gained from the laboratory. By making claims and supporting them with evidence, students were faced with the
responsibility of explaining to the instructor what they had learned. By doing so, the
information can be retained and applied to new contexts such as lecture exams.

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CHAPTER 4: IMPLEMENTING POGIL IN THE LECTURE AND THE SCIENCE WRITING HEURISTIC IN THE LABORATORY—STUDENT PERCEPTIONS AND PERFORMANCE IN UNDERGRADUATE ORGANIC CHEMISTRY

Adapted from a paper published in Chemistry Education Research and Practice, 2008, 9, 149-156.

Jacob D. Schroeder and Thomas J. Greenbowe

Abstract
This study investigated the possible connection between effective laboratory activities and student performance on lecture exams. In a traditional undergraduate organic chemistry course for non-science majors, students could predict the products of organic reactions, but struggled to provide reaction mechanisms for those same reactions, despite obtaining perfect scores on their laboratory reports where reaction mechanisms were required. In addition, student attitudes toward chemistry in general were sharply negative after taking organic chemistry. To address these two issues, we implemented POGIL activities in the course and the Science Writing Heuristic in the laboratory to replace the standard lecture format and verification laboratory experiments. This paper will focus on student performance on nucleophilic substitution reaction mechanisms on a class exam. Performance on these questions improved compared to students in past traditional classes. In addition, students were given a pre-class and post-class survey regarding their perceptions of the course. At the conclusion of the term, many students thought the class was easier than what they initially expected. This illustrates the view that non-science majors have the ability to learn organic
chemistry from a mechanistic point of view, and integrate concepts learned in the laboratory with concepts presented in the lecture.

**Introduction**

The science laboratory has been viewed as a critical component of the learning process (Lloyd, 1992). Over the years, a number of reviews have been published on the effectiveness of the general chemistry laboratory (Hofstein and Lunetta, 1982, 2004; Lazarowitz and Tamir, 1994; Lunetta, 1998; Tobin, 1990). These reviews indicate that a lack of evidence exists to support the idea that traditional laboratories are effective in promoting meaningful learning. Hawkes (2004) suggested, “Duplicating what we chemists do in our laboratories (or what chemists of earlier generations used to do) does not enhance students’ understanding of chemistry, but makes chemistry an irrelevance”. This leads the traditional laboratory format to be summarized as a cookbook or verification approach that does little to help students learn concepts (Bodner et al., 1998).

The organic chemistry laboratory has been criticized for the same general reasons that the general chemistry laboratory has been. Baru and Mohan (2005) have argued for the incorporation of an element of discovery in laboratory activities to ensure that student interest and enthusiasm are retained. Cooley (1991) emphasized that laboratory work should focus more on getting students to obtain and interpret data rather than making representative compounds and learning techniques. He argued, “When they (the students) are given an explanation of what the data mean, they accept such interpretations without question and complete the laboratory with minimal effort or ability to interpret data” (p. 503). Mohrig (2004, p. 1083) argues from a practical standpoint that since the traditional laboratory lacks evidence of producing meaningful learning, “wouldn’t it be just as effective to tell the
students what they would see, without mounting expensive and time-consuming labs?” A growing number of researchers have been calling for reform in the science education laboratory (Venkatachelam and Rudolph, 1974; McComas, 2005; Truax, 2007). The cookbook nature of the traditional laboratory fosters what Cutler describes as a “creeping passivity” (2007), a reference to students who are not engaged in the laboratory or in the classroom. Disengaged students begin to look at laboratory work as something they just have to show up to complete, and they can quickly lose any prior interest they may have had. This leads to students oftentimes failing to see how the experiment is relevant to them (Reid and Shaw, 2007). This is especially true in a laboratory course offered to those students who are not majoring in chemistry (Singh, 1999; Kelley and Gaither, 2001; Weidenhamer, 2007). Many students fail to see why they have to take the course, often looking at it as nothing more than a requirement. As Pungente and Badger (2003, p. 779) have noted, “organic chemistry is viewed by some students as little more than a rite of passage, or the academic equivalent of hazing”.

Domin (1998) and, more recently, Horowitz (2007) have reviewed several efforts to reform the laboratory, including discovery-based experiments, inquiry-driven experiments, project-based learning, and collaborative learning activities. While each of these has been shown to improve student performance on exams about content as well as student attitudes, the complexities involved in organic procedures and the knowledge students bring into the course certainly plays a role in the ultimate success of these efforts. The incorporation of research-based laboratory experiments has also shown success (Gilbert et al., 2002; Newton et al., 2006). While these experiments would certainly arouse student interest, they can be
time-consuming when students do not have the knowledge or the experience to be ready to complete them.

An alternative to the traditional approach in the laboratory is the Science Writing Heuristic (SWH) laboratory report format. This format is based on the theoretical framework of a learning cycle whereby students explore concepts to look for trends or patterns rather than verify an expected outcome (Lawson et al., 1989; Keys et al. 1999). The learning cycle consists of three phases: exploration, term introduction, and concept application (Lawson, 2001). The exploration phase should raise questions, complexities, or contradictions. The SWH format incorporates this phase by providing students with an experiment with no direct answers, but rather many possibilities based on previous concepts covered. As the experiment is being completed, students record their data on the blackboard. These data serve as the class data, and allow students to look for trends or patterns. This allows for the introduction of new terms and concepts based on the data generated. Once the trend or pattern is found, the instructor can progress into the concept application phase. By using the data obtained during the experiment, students can use the trends or patterns found to make conclusions about examples in different contexts.

In the traditional laboratory format, the opposite is true; students follow a given set of procedures to verify a fact or synthesize a compound. In this setting, if students obtain what they are supposed to obtain, writing up the laboratory report requires little difficulty, as the explanations and answers are provided. With the possibility of only verifying one correct answer, students are not forced to reconcile their results if these results do not agree with what they were supposed to obtain (Pickering, 1985). When this happens, students can
become frustrated and oftentimes write in their laboratory reports that they had faulty equipment or some forms of human error were present (Rudd et al., 2002).

Table 1 shows how the SWH laboratory report format differs from the traditional format. In a traditional laboratory report, students start with the title of the experiment (The Preparation of…) and the intended purpose (to make a certain compound; to ‘do’ a specific reaction), both of which are supplied in advance. To follow the learning cycle approach, the SWH format replaces the purpose with beginning questions (exploration). These questions are student-generated and can only be answered by completing an experiment. The intention is that as a class, students decide what they are trying to investigate rather than having the purpose overtly given.

*Table 1: Laboratory report format comparison*

<table>
<thead>
<tr>
<th>The Traditional Laboratory Report</th>
<th>The Science Writing Heuristic Report</th>
</tr>
</thead>
<tbody>
<tr>
<td>Title and Purpose</td>
<td>Beginning questions</td>
</tr>
<tr>
<td>Procedure</td>
<td>Safety considerations</td>
</tr>
<tr>
<td>Data and observations</td>
<td>Tests and procedures</td>
</tr>
<tr>
<td>Balanced equations, calculations, graphs</td>
<td>Data and observations</td>
</tr>
<tr>
<td>Discussion</td>
<td>Claims</td>
</tr>
<tr>
<td>Conclusion</td>
<td>Evidence</td>
</tr>
<tr>
<td></td>
<td>Reading and reflection</td>
</tr>
</tbody>
</table>

Other major differences between the two formats are the claims, evidence, and reading and reflection components of the SWH format. After completing an experiment and collecting the appropriate data, students can answer their beginning question as a claim (term
introduction). The support for this claim is evidence and can consist of spectral data, data generated by other groups, or any trends or patterns found in the class data table. The reading and reflection component allows students to ask themselves whether their results made sense. By asking this question, they can compare what happened during the experiment to what is or was covered in the lecture. Students are also required to do a small amount of research online to see whether they can apply what they did in the laboratory to some topic more relevant to them (concept application).

We have reported that in the general chemistry laboratory students spend less time writing laboratory reports and teaching assistants spend less time grading them when using the Science Writing Heuristic laboratory report format (Rudd et al., 2001). In addition, students who used the SWH format performed significantly higher on an ACS standardized examination, as well as on in-class lecture exams and quizzes (Burke et al., 2006; Poock et al., 2007). It has also been reported that middle- and high-school students who used the SWH format in biology have scored higher on multiple-choice and conceptual questions (Hand et al., 2004; Hohenshell and Hand, 2006). Other researchers have reported similar findings after implementing this method in a variety of courses across all grade levels (Gravelle, 2006; Hand, 2007; Sarquis, 2007).

**Background**

In our initial pilot study, we tracked students who had previously used the SWH laboratory format in general chemistry to see whether the success they had would continue in organic chemistry. For that study, the SWH students were matched with another group of students who had just completed a general chemistry course in which the traditional laboratory report format was implemented. Analysis of the in-class exams revealed that the
group of students who had prior experience with the SWH report format outperformed the traditional students, even though the traditional format was implemented in the organic chemistry laboratory (Schroeder, 2007). In a subsequent study, we focused on student performance on more challenging exam questions, such as sketching reaction mechanisms and completing synthetic sequences or retrosynthetic analyses. In the traditional organic chemistry course we found that all students performed well when predicting reaction products, but when asked to provide a mechanism to show how that same reaction worked, the number of correct responses dropped by over 90%.

The focus of this work is on the concept of nucleophilic substitution. In the traditional organic chemistry course, students completed a laboratory experiment in which they reacted a tertiary alcohol (tert-butyl alcohol) with concentrated hydrochloric acid (Clague, 2006). The goals of the experiment were for students to get exposed to an SN1 reaction, develop the ability to tell the difference between an SN1 and an SN2 reaction, and learn the trend for carbocation stability. This experiment took place two weeks prior to an exam that asked questions specifically dealing with this concept. Two types of questions on the exam should be noted; first, predicting the product of a reaction given reaction conditions, and second, writing out the mechanism for a similar reaction (Figure 1).

During a spring semester (in sequence), 90 out of the 111 people (81%) attempting the first problem received full credit. However, when the same students were asked to sketch the mechanism in the second question, a total of twenty-eight people attempted it, of whom seven did so correctly. What this shows is that even though students could recognize reaction conditions and write the correct product, they did not understand how the product is formed.
Draw the structure of the product formed in the following reaction:

\[ \text{CH}_3\text{CH}_2\text{C(CH}_3)_2\text{OH} \xrightarrow{\text{HCl}} \]

Draw the mechanism for the following reaction:

\[ \text{H}_3\text{C}^-\text{C}^-\text{OH} \xrightarrow{\text{HCl}} \]

Interestingly, on the laboratory report for nucleophilic substitution, thirty-one students received a perfect score, which according to the grading rubric includes a complete detailed mechanism showing \textit{tert}-butyl alcohol reacting with HCl to form \textit{tert}-butyl chloride. But only five people from this group receiving perfect laboratory report scores attempted this mechanism on the exam, three of whom were correct. Perhaps these students could not remember writing the mechanism in their laboratory report or never learned it in the first place. Since the mechanism was given in the laboratory manual, it could be reasonable to assume the latter.

This work focused on the same course offered during the subsequent summer term, at which point the authors were assigned as co-instructors. In contrast to the traditional format this course had used before, we implemented the Science Writing Heuristic in the laboratory and also updated the lecture portion of the course to allow for the implementation of some POGIL (Process-Oriented Guided Inquiry Learning) activities based on the model presented by Minderhout and Loertsher (2007). The effectiveness of POGIL in general and organic chemistry has previously been described, having been shown to not only increase student performance, but also decrease the number of students who withdraw from the course (Farrell \textit{et al.}, 1999; Spencer, 1999; Straumanis, 2004). Implementation of both POGIL in
the lecture and the SWH in the laboratory, we believed, would help our students understand more of the difficult concepts mentioned above.

**Methods and Materials**

The introductory organic chemistry course met every day for one hour during 8 weeks of the summer session. The corresponding laboratory met for two 3-hour sections per week. The enrollment was twenty-four. The required textbook for the lecture was *Essential Organic Chemistry* (Bruice, 2006) but other books (Hart et al, 1998; McMurry, 2000; Smith, 2006) were used in conjunction with Straumanis’ *Organic Chemistry: A Guided Inquiry* to supplement the POGIL activities. For the laboratory, we converted the previous traditional laboratory manual into an inquiry-based laboratory manual implementing the SWH laboratory format (Schroeder *et al.*, 2006). Data were collected for quantitative analysis by photocopying student exams and recording performance on targeted questions. The performance for the summer group of students was compared to the performance of the traditional group mentioned above who took the course the spring semester that same year. For a qualitative analysis, a survey was given at the beginning of the course to gauge student perceptions of the format, and what their expectations were for the course. An evaluation was given at the end of the course to see whether student perceptions had been changed as a result of the course.

The lecture was divided into three 20-minute blocks (Table 2). During the first block, students gathered into groups and began working on the activities collectively. Oftentimes, students completed an experiment dealing with material they had not yet seen in the lecture, which allowed the instructors to frame the new activity as an extension of previously completed laboratory work. This served the dual purpose of giving the instructors an idea of
what the students were able to learn from the experiment, as well as giving the students a sense of continuity between the laboratory and the lecture.

Table 2: Block style arrangement for the lecture

<table>
<thead>
<tr>
<th>Block</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1&lt;sup&gt;st&lt;/sup&gt; block</td>
<td>Hand out activity covering new material. Give a brief introduction over what was done before and how this new activity was going to try to build on previous knowledge.</td>
</tr>
<tr>
<td>2&lt;sup&gt;nd&lt;/sup&gt; block</td>
<td>Allow the students to work collectively in groups. Walk around and visit each group to see what kind of progress is being made, and if any major difficulties are being found. Help students by acting as a facilitator instead of the source of all answers.</td>
</tr>
<tr>
<td>3&lt;sup&gt;rd&lt;/sup&gt; block</td>
<td>Gather the class as a whole and discuss some of the concepts being addressed in the activity. Go over common misconceptions or difficulties experienced by all groups. All groups take part in the discussion.</td>
</tr>
</tbody>
</table>

During the second block, the instructors would check with each individual group to see what progress was being made. Students were encouraged to ask other groups questions before they would come to the instructors for help. When problems were encountered on specific concepts, other groups would offer insight into how they approached these problems. This transformed the traditional classroom into the students’ own scientific community. During the final block, the instructors facilitated a discussion in which the groups of students had the opportunity to discuss how they had arrived at their solutions to the problems presented in the activity. It is important to note that the instructors needed to guide the discussion in this format; otherwise it might have been difficult to keep progressing. A new activity was given to the students every other lecture on average.
The laboratory operated in a similar manner to the lecture. All answers and explicit details were removed, instead being replaced with suggested variations. To help create a sense of community, students were encouraged to work together, put a class data table on the board, and draw conclusions based on the data that were collected and compiled. Prior to an experiment, everyone would meet in the pre-laboratory room. Students were asked whether they had beginning questions that could be answered by doing the experiment. With no products given in the manual, many questions focused on what the product would be, but in all cases a second, more productive question would surface (i.e. will the product be different if the acid is used as a catalyst or in equal amounts?). After students agreed upon at least two questions to study, the teaching assistants would go over the experiment, guiding students how they could proceed in the laboratory to be able to answer their beginning questions. A quiz was given after this introduction to ensure that students would come to the laboratory prepared; this allowed the pre-laboratory to be more effective and more discussion-based.

Once in the laboratory, the teaching assistants would visit each group at different stages of the experiment to see whether any difficulties or questions were arising. Students were encouraged to ask questions from other groups before asking the teaching assistants. At the conclusion of the laboratory period, students gathered around the blackboard to analyze collectively the class data obtained. The teaching assistants would serve as facilitators, asking questions of different groups. This allowed the groups of students, and not the teaching assistants, to bear the responsibility of explaining their data to the rest of the class. When a consensus emerged, students were able to make claims that would answer their beginning questions. With all groups reporting their findings, students would use the class data as evidence to support their claim. Putting all of this information together during the
laboratory period ensured that everybody had most of the laboratory report written before they left. The only task students needed to complete on their own outside of the laboratory was often the reading and reflection component.

**Experimental**

Rather than lecturing over nucleophilic substitution, the instructors supplied students with two activities (Straumanis, pp. 97-106; pp. 107-116). The first activity focused on one-step nucleophilic substitution (SN2) while the second focused on two-step nucleophilic substitution (SN1).

---

**Figure 2: One-step nucleophilic substitution model**

Both activities first supply a model for students to use as a reference. The model for the first activity is shown in Figure 2. Following this model are a series of critical thinking questions that start by building on previously covered concepts with an increasing level of difficulty for each subsequent question. For the first question, students were asked to identify both the incoming group and the leaving group for each reaction. This is followed by a question asking students to place $\delta^+$ and $\delta^-$ above portions of each of the carbon reactants. For the final question, students need to use curved arrows to draw a reaction mechanism. An extended model follows, exploring such concepts as inversion of stereochemistry and reaction rates. In this manner, students can start out with a basic model...
that extends concepts covered previously (electronegativity and bond polarity) and applies them to new concepts (S<sub>N</sub>2 reactions, leaving group ability, and reaction rates).

The second activity contained two models concerning S<sub>N</sub>1 reactions, followed by a series of questions. For the first model, students needed to label carbocations as primary, secondary, or tertiary. When provided with energy diagrams students could compare the differences between these three to determine a stability trend. The second model focused on the rate-determining step, comparing the S<sub>N</sub>1 reaction to the S<sub>N</sub>2 reaction in activity 1 to uncover the differences between the two. The two activities conclude with a summary table (Table 3) that students fill in with the following points:

- Very polar solvent better, but weakly polar OK
- Very polar protic solvent required to stabilize ion intermediates
- Must be 2°, 3°, allyl or benzyl
- Methyl or 1° preferred, 2° OK too
- Dependent on the identity and concentration of both nucleophile and electrophile
- Dependent only on the identity and concentration of electrophile
- Inverted (switch from R to S or vice versa)
- Racemic mixture produced

Table 3: Summary of factors leading to S<sub>N</sub>2 vs. S<sub>N</sub>1 reactions

<table>
<thead>
<tr>
<th>Reaction Type</th>
<th>Solvent</th>
<th>Stereochemistry</th>
<th>Electrophile</th>
<th>Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>S&lt;sub&gt;N&lt;/sub&gt;2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S&lt;sub&gt;N&lt;/sub&gt;1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

These two activities required two lecture periods to complete followed by a third period used for finalization, double-checking, and review.
For the laboratory, a clear distinction between the traditional experiment and the guided-inquiry SWH experiment needs to be made. The title of the traditional experiment, *The preparation of 2-chloro-2-methyl propane*, was renamed as *Substitution vs. elimination: the chemical competition* (Schroeder et al., pp. 43-48). This removed the expected answer from the title and allowed students to question what would happen during the experiment. The procedure was the same as that followed for the traditional experiment, but without the explicit details (*i.e.* which layer to remove during the extraction, what to look for in the infrared spectrum). One of the more distinctive aspects is how the new experiment builds upon knowledge gained from prior experiments. Earlier in the term, students performed a dehydration reaction, treating an alcohol with a strong acid to produce an alkene. In this new substitution experiment an alcohol is again reacted with a strong acid, but students find out that the amount of acid has changed. This leads students to wonder whether they will form what they did before or if something new will happen.

**Results & Discussion**

**Part 1: Exam performance**

Our first objective was to compare the exam performance of the summer term students to that of the students who took the traditional course the semester before. To compare the two groups, we wrote two questions on the exam similar to questions on the prior exam (Figure 3). The first question was identical to the question given during the traditional course; students needed to draw the correct structure of the product. During the summer, 22 out of 23 students answered correctly. This is comparable to the performance of the students in the traditional course.
Draw the structure of the product formed in the following reaction:

\[ \text{CH}_3\text{CH}_2\text{C(\text{CH}_3)}_2\text{OH} \xrightarrow{\text{HCl}} \]

In the reaction below, the conditions favor an E1 reaction in which two possible products could form.

(a) 2 points – draw the structure of the two products
(b) 4 points – Draw a reasonable reaction mechanism showing the formation of both products
(c) 2 points – explain why the major product is formed.

\[ \text{CH}_3\text{CHCH}_2\text{Cl} \quad + \quad \text{Na} \quad \text{O} \quad \text{CH}_2\text{CH}_3 \]

---

Figure 3: Nucleophilic substitution and elimination exam questions

The second problem was a multi-step question combining three different types of questions. Performance on this question was mixed; 11 out of 23 students drew the major and the minor elimination products correctly. More importantly, 8 out of the 23 sketched a complete mechanism that showed the formation of both products. Nine of the students correctly explained why the more substituted alkene was produced.

It should be noted that during the previous semester, 111 students in the traditional course had had the opportunity to provide a mechanism for their reaction, but only twenty-eight actually tried and of these, only seven were correct. In terms of percentages, we were able to see an increase in the number of correct responses to the mechanism portion of this question (25% for the traditional group compared to 34% for the summer group). More importantly, every student who took the exam over the summer term attempted to solve the problem, whereas in the traditional course, 75% of the students did not attempt it. Partial credit was awarded for this mechanism with eleven out of the twenty-three students receiving
2 of the 4 points and three out of the twenty-three receiving 3 of the 4 points. Only one student during the summer term did not receive any points. What this shows is that while using the SWH format, students in the summer term were more confident in attempting the problem, and with one exception, the students were at least able to get it started.

In terms of performance on the corresponding nucleophilic substitution laboratory report during the summer term, the average score out of 24 points was 17.7, with only four students receiving a perfect score. Two of these students received full credit on both of the exam questions while the other two missed only one point on both of the questions. Thus, when compared to the students in the traditional setting, the students during the summer term received lower average scores on the corresponding laboratory report but performed much better on the corresponding exam. We continued using POGIL activities on other topics such as Grignard reactions, aldol reactions, and retrosynthetic analyses. Unfortunately, these types of questions were not asked of previous students in the traditional course so no comparison of problems can be made.

**Part 2: Student perceptions**

When we look at student perceptions of this course, it is clear that they shared many of the concerns that students before them did. When asked on the beginning survey what their expectations for this course were, nearly 40% of those who answered (8/19) believed that it was going to be more difficult than any other chemistry course they had taken. When asked whether their previous laboratory experience had been practical, almost half of those who answered said no (11/23). In addition, when asked what their primary reason for taking this course was all but three of them (21/24) said that it was a requirement. Not surprisingly, these results support the idea that organic chemistry is perceived as a difficult course. Other
questions on the survey are shown in Table 4. These questions were based on a 1 – 5 Likert scale (1 – strongly disagree; 5 – strongly agree).

**Table 4: Student perceptions concerning laboratory work (beginning survey)**

<table>
<thead>
<tr>
<th>Question</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>I would rather learn concepts in the lab before going to the lecture</td>
<td>2.9</td>
</tr>
<tr>
<td>The lab should help me understand concepts that are covered in the lecture</td>
<td>4.6</td>
</tr>
<tr>
<td>My past experience in the general chemistry lab should help me with this lab</td>
<td>3.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>On average, how much time do you expect to spend writing your laboratory report each time?</td>
</tr>
</tbody>
</table>

From the responses to the questions in Table 4, it would appear that students have a slight preference for addressing a concept in a lecture before going into the laboratory. The high level of agreement to the second question in which the laboratory should follow a lecture further corroborates this. Student comments indicated they wanted to "learn in lecture before doing in the laboratory". Despite the number of negatives mentioned previously, students appeared to be much more confident when asked about their prior laboratory experience, and whether or not it would help them (surprising, in that many of the students thought their prior laboratory experiences in general chemistry were not practical). And when asked about the time commitment that would be required, they gave an average range between 1.1 – 2.1 hours to write a laboratory report.

At the conclusion of the summer class, students completed the end-of-term evaluation, with questions similar to those given on the beginning survey. These results are shown in Table 5. An overwhelming number of students believed the laboratory helped them
understand topics discussed in lecture, even though many of the laboratory experiments addressed the topics prior to them being covered in the lecture. What this could mean is that exploring new concepts in the laboratory better prepares students for the activity-based lecture.

Table 5: End of term evaluation of the laboratory

<table>
<thead>
<tr>
<th>Did the laboratory help you understand topics discussed in lecture?</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Yes</td>
<td>16</td>
</tr>
<tr>
<td>Sometimes</td>
<td>3</td>
</tr>
<tr>
<td>No</td>
<td>0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Did the format of the lab report help you organize and put together your report?</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Yes</td>
<td>18</td>
</tr>
<tr>
<td>No</td>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>How has this lab compared with the expectations you had for it at the beginning of the semester?</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Easier</td>
<td>8</td>
</tr>
<tr>
<td>As expected</td>
<td>5</td>
</tr>
<tr>
<td>Harder</td>
<td>2</td>
</tr>
<tr>
<td>More interesting</td>
<td>2</td>
</tr>
<tr>
<td>More challenging</td>
<td>2</td>
</tr>
<tr>
<td>Other</td>
<td>2</td>
</tr>
</tbody>
</table>

| On average, how much time did you spend writing each lab report? | 1.3 – 1.9 hours |

Concerning the SWH laboratory report format, all but one student thought it helped them organize their thoughts into a workable report. Four of these who agreed did express some reservation. Two of the students thought that some aspects of the report seemed repetitive, while another two were concerned that they never knew what the grader was looking for. The most compelling series of responses were to the question of how the laboratory was perceived at the end of the term compared to the expectations the students had
for it at the beginning. At the end of the semester, eight students thought the laboratory was
easier than what they had expected it to be. Still, five students said it had met their
expectations, although none of these five wrote negative comments concerning the
laboratory. Only two students thought the laboratory was as hard or harder than what they
had expected going in. Finally, in terms of the time requirement, many students felt they
spent nearly as much time writing each report as they expected to, although the higher end of
the range is slightly lower than it had been in the beginning of the semester.

A separate evaluation was also given for the lecture. The major differences between
our approach to this course compared to the previous traditional approach were the reliance
on group work, activities, the laboratory—lecture correlation, and the removal of most of the
lecturing. According to Table 6, a majority of students found the activities helpful. Some of
the students gave mixed responses, saying that some of the activities were very helpful
whereas others were not. In terms of being in groups, again a majority of the students liked
this approach better than the traditional lecture, although five students did see a need for a
more balanced approach. When asked about the level of difficulty of the activities, students
gave an overall average of 3.3 on a 5-point Likert scale (5 – much too difficult; 1 – much too
easy). This seems surprising since many of the activities were geared toward undergraduate
chemistry majors rather than non-majors. In terms of the length of the activities, again
students were in the middle, commenting that the length of each activity was about right.
The last point to mention is the difference between the amount of time students expected to
spend studying for class compared to the amount of time they actually spent studying for
class. As can be seen from the table, this amount was less by roughly two hours.
Table 6: End of term evaluation of the lecture

Did you find the activities helpful in understanding the material?

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Yes</td>
<td>12</td>
</tr>
<tr>
<td>Sometimes</td>
<td>5</td>
</tr>
<tr>
<td>No</td>
<td>2</td>
</tr>
</tbody>
</table>

Did group work seem more beneficial for understanding concepts, or would more lecturing been beneficial?

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Group work</td>
<td>9</td>
</tr>
<tr>
<td>Some combination</td>
<td>5</td>
</tr>
<tr>
<td>Lecture</td>
<td>5</td>
</tr>
</tbody>
</table>

In terms of the activity assignments, can you rate the level of:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Difficulty</td>
<td>3.3 (5 point scale)</td>
</tr>
<tr>
<td>Lengthiness</td>
<td>3.4</td>
</tr>
</tbody>
</table>

On average, how much time do you expect to spend studying for the lecture?

5.5 – 9.1 hours

On average, how much time did you spend studying for the lecture?

3.6 – 7.1 hours

Taken together with the value given for the duration of the activities, these data suggest that most of the work done for this course took place during the class period.

Conclusions

The primary goal of implementing POGIL in the lectures along with the SWH format in the laboratory was to encourage students to think more critically while allowing them to construct their own knowledge. This is believed to have directly resulted in students achieving a higher success rate on what they initially perceived to be difficult questions (mechanisms). Students appeared more confident when attempting these types of questions than in the previous semester when a large majority of students skipped them. For the substitution example shown here, all but one student received at least half the points.
Mechanisms are difficult for students to learn, even for chemistry majors. The fact that these students appeared to show this confidence was encouraging.

Another goal was to integrate the laboratory component of the course fully with the lecture component. We were deeply concerned that past students had received very high marks in the laboratory, but no correlation existed with the lecture marks. This was evident from student performance on laboratory reports during the traditional spring semester. For the nucleophilic substitution experiment, 31 students out of 104 received a perfect score on their laboratory report, which included a full mechanism showing the conversion of tert-butyl alcohol into tert-butyl chloride. Yet when asked to complete this same mechanism two weeks later on an exam, only five of these thirty-two students tried and only two of them were successful. During the summer, all students attempted this problem and the success rate increased.

We also wanted to see whether implementing these two methods would change student perceptions of the level of difficulty of organic chemistry. At the beginning of the summer term, the majority of the students reported that they were taking this class simply because it was required. Most were also expecting it to be a very hard class; some even remarked that it would be the hardest chemistry class they would ever have to take. But based on the end-of-term evaluations, it would appear that many of these students were surprised by what they initially thought. Despite these activities being geared toward chemistry majors, this group of students did not think the activities were too hard, and they did not think they were too long. Only two of the students did not think the activities to be helpful in guiding them to an understanding of the material. Their overall preference for
group work, in addition to the decrease in the amount of studying outside of class, leads us to believe that most of the work was completed during the period.

Research has shown that each of these teaching methods on its own increases student performance and improves student attitudes. Combining these two together showed us that our students could be challenged with difficult questions and still perform well. Although this study only focused on one topic from one exam, the clear distinction between the performance of the two groups on this concept, and their perceptions of the course as a whole, is evident. To better compare the traditional approach to the guided inquiry approach it would be beneficial to implement these same strategies during a spring semester with the same instructor for both courses. During the spring, the time gap between the substitution laboratory experiment and the corresponding class exam was roughly two weeks. During the summer, this gap decreased to just a few days. It may be possible that information was stored in short-term memory, allowing students in the summer to be more apt to recollect it. Nevertheless, with the change in student perceptions regarding the level of difficulty, the amount of work, and the connection between the laboratory and the lecture, we feel confident that these results would be replicated in a future study.

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CHAPTER 5: A PRACTICAL EXAMINATION FOR THE INTRODUCTORY ORGANIC CHEMISTRY LABORATORY

Adapted from a paper submitted for publication to The Journal of Chemical Education

Jacob D. Schroeder and Thomas J. Greenbowe

Abstract

This article introduces a new laboratory practical examination for the introductory organic chemistry laboratory. In the traditional laboratory, students were given a written multiple-choice laboratory final exam, consisting of questions they had seen previously throughout the semester on the pre-laboratory quizzes. Despite having seen these questions, the average score was nearly 27 percentage points lower than the total average percent achieved on laboratory reports, and just over 10 percentage points lower than the total average percent achieved on quiz. As part of our efforts to convert the traditional organic chemistry laboratory into a collaborative guided inquiry laboratory, we have replaced the written multiple-choice final with a laboratory practical examination, in order to more effectively evaluate students in the laboratory setting.

Introduction

The effectiveness of laboratory practical exams for assessing student laboratory work has been periodically reported in this Journal (1). Neeland has reported that by combining an organic laboratory practical exam with a Problem Based Learning (PBL) format, “students took the lab more seriously, and end[ed] up actually learning the lab skills” (2). In a separate study involving the use of a practical exam in a traditional organic laboratory,
Casanova and Tunstad reported that overall student performance on laboratory reports was significantly higher than their performance on the lab practical exam (3). They argue, “The factors on which students have been traditionally evaluated, particularly the quality of their notebook write-up, may inadequately measure their intrinsic laboratory ability,” echoing prior criticisms by Dumon and Pickering that “little work has been done to develop questions that will test laboratory understanding at a deeper level than conceptual recall” (4). After reviewing laboratory-based instruction, Hilosky et al. questioned whether the laboratory was worth the time and the expense (5).

We observed similar results for our traditional organic chemistry laboratory for non-science majors. During the spring of 2006, 104 students were graded on eleven 20-point laboratory reports. The average total laboratory report score was 202 (220 points maximum; 91.8%), yet the average score on the written laboratory final exam was only 64.1% (100 point maximum). This discrepancy can be attributed to the nature of the written exam, composed of a collection of multiple-choice questions students had seen previously on each of the eleven 15-point pre-laboratory quizzes. With an average total quiz score of 122.5 (165 points maximum; 74%), it would appear that performance on the written final exam was dictated by how well students could memorize their prior quizzes.

These results led to replacing the traditional “cookbook” format of the introductory organic chemistry laboratory with a collaborative guided-inquiry format that uses the Science Writing Heuristic (SWH) approach (6). The multiple-choice final exam was replaced with a 3-hour practical examination that students would complete during their normal laboratory section. This has since resulted in an improvement in both student attitudes toward organic chemistry and overall performance on lecture exams (7).
Exam Design

Rather than setting up experimental stations, a scenario was created that would require the collection and analysis of experimental data to answer questions posed during a job interview (Box 1). This approach seemed better suited for the non-chemistry major students enrolled in the course, adding a sense of relevance to an actual scenario. It also fit into the theme created by many of the experiments completed during the term that brought more context and applicability into the laboratory. The practical exam was highlighted on the syllabus, giving students ample time to know it was a part of the course. One week prior to the exam, students were given the introduction to the scenario, which outlined what their task would be but not explicitly how they were going to accomplish it.

Box 1: Lab practical exam introduction

Introduction

After graduating from college with a Bachelor’s Degree in your chosen field of study, you get your first big job interview with a reputable company. Drake, the interviewer, has had a chance to look over your résumé and your transcripts, and is intrigued that you have taken an organic chemistry course. He understands that you aren’t “a chemist” but wants you to see what you learned from the course.

You are competing against many others for this job, and everyone seems to have this organic chemistry course in common. To narrow down the applicant pool, Drake has designed a way for you to show how much organic chemistry you know. One of Drake’s old college buddies, Blake, just happens to be an organic chemist who is trying to develop a cure for cancer. Blake is in need of some alcohols for some crucial experiments he needs to finish his reaction sequence. Due to budget cuts, however, he is unable to buy the necessary compounds. Drake tells Blake that he has 130 applicants who claim that they can do organic chemistry, especially a reaction to produce alcohols, since they had done it in the course before. As such, a deal is struck where Blake agrees to allow these 130 applicants to make the necessary compounds for him. The person who will get the job will not necessarily be
the person who makes the most amount of compound, but rather the person who is able to explain to Drake what exactly happened in preparing these compounds. Drake isn’t a chemist either, so he is more concerned that you explain to him what you did and what it means.

To prepare alcohols in this course, you reduced a carbonyl group using sodium borohydride, NaBH₄. Based on the experiment, you were able to test the reaction and observe some of its limitations. An assortment of different unknown compounds will be made available to you in the reagent hood (ketones and aldehydes). Your job will be to select one of these, determine what it is, set up a reaction to reduce it, and then identify what your resulting product is. You can work with your lab partner, but since you are competing against others, you don’t want to talk to other groups about it because doing so could give them an unfair advantage. Your “supervisor” will only answer questions dealing with safety, so you will be entirely on your own. You will have access to the IR, ¹H NMR printouts, TLC plates, the melting point device, and all solvents and chemicals needed to complete the task. You can use only your laboratory notebook as a reference. Past lab reports or lab manuals are not allowed. You will also be provided with a reagent table.

Students were allowed to use their laboratory notebooks, which contained carbon copies of all laboratory reports they had written during the term. Because students worked with a partner during all prior experiments, they were allowed to work with the same partner for the exam, with the stipulation that they could not talk with other pairs. They were also required to select a different unknown from the one chosen by the pairs working next to them. Teaching assistants responsible for each section would only answer questions relating to safety. Because students previously experimented with sodium borohydride, they could adapt the prior procedure for the practical examination.

The practical exam required only one experiment, but emphasized concepts from several experiments completed during the term. As shown in the supplemental material,
students first needed to identify their unknown sample. To minimize time requirements, students were given a data sheet providing the chemical formula, some physical properties, and the infrared and $^1$H NMR spectra for each of their individual unknowns. This portion of the exam reinforced concepts introduced during a prior qualitative analysis experiment (7), including a variety of qualitative tests to distinguish alcohols, aldehydes, and ketones from one another and $^1$H NMR spectroscopy for structure identification.

After determining the structure of the unknown sample, students could begin the sodium borohydride reduction. Since the reaction time was only 15 – 20 min, students were not waiting for an extended period of time for the reaction to finish. After completion, the product could be worked up and collected by filtration or extraction. Students could then perform a TLC to obtain information about polarity, followed by a series of qualitative tests to support the claim that the product was indeed an alcohol. In many cases this could be determined solely with IR spectroscopy, but many students subjected their product to a Jones oxidation to add more evidence to their claim of synthesizing an alcohol. Students generally finished the exam in two hours, spending the majority of time determining the structure of the starting material.

**Point Distribution**

The laboratory practical exam was worth 50 points, the majority allocated to the correct starting material and product identification and for the theoretical and percent yield calculations. Each of the teaching assistants grading the exam was assigned to only one question to ensure consistency. Overall average scores for laboratory reports, quizzes, and the laboratory final exam are shown for the past five terms (Table 1).
Table 1: Overall average student scores on reports, quizzes, and final exam.

<table>
<thead>
<tr>
<th></th>
<th>Spring 2006</th>
<th>Fall 2006</th>
<th>Spring 2007</th>
<th>Fall 2007</th>
<th>Spring 2008</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total report average %</td>
<td>91.8</td>
<td>84.8</td>
<td>85.0</td>
<td>82.9</td>
<td>82.8</td>
</tr>
<tr>
<td>(standard deviation)</td>
<td>(11.30)</td>
<td>(25.75)</td>
<td>(23.86)</td>
<td>(23.41)</td>
<td>(29.62)</td>
</tr>
<tr>
<td>Total quiz average %</td>
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<td>61.8</td>
<td>67.7</td>
<td>67.6</td>
<td>67.5</td>
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<tr>
<td>(standard deviation)</td>
<td>(17.37)</td>
<td>(7.30)</td>
<td>(7.66)</td>
<td>(6.59)</td>
<td>(6.45)</td>
</tr>
<tr>
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<td>79.4</td>
<td>64.0&lt;sup&gt;b&lt;/sup&gt;</td>
<td>83.8</td>
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<tr>
<td>(standard deviation)</td>
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<td>(4.45)</td>
<td>(5.32)</td>
<td>(9.72)</td>
<td>(5.73)</td>
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<td>114</td>
<td>116</td>
<td>118</td>
<td>114</td>
</tr>
</tbody>
</table>

Notes:
<sup>a</sup> Performance on multiple-choice laboratory final
<sup>b</sup> Students performed lab practical individually

Discussion

During the Spring 2006 term, the overall average laboratory report score was 91.8%, over 27 points higher than the average score on the written laboratory exam. The relatively high average report score can be attributed to the fill-in-the-blank grading rubric used in the traditional laboratory format. Students simply needed to copy any explanations or product identities from the introduction for each experiment in the laboratory manual, making sure that they obtained the results they were supposed to. With the final laboratory exam composed of the pre-laboratory quiz questions given during the term, it is not surprising to see the average score on the exam more closely reflect the overall average on the quizzes.

After introducing both the SWH format and a laboratory practical examination for the Fall 2006 term, we observed a decrease in overall average scores for both laboratory reports and quizzes. Without the overt explanations and exact product identities that had been provided in the laboratory manual, students had to use the experimental data they obtained to provide explanations in their laboratory reports. Percentages on the pre-laboratory quizzes most likely decreased because many of the multiple-choice questions were replaced with fill-
in-the-blank or short answer questions. The sharp decrease in the standard deviation reflects the point total decreasing from 15-points to 5-points. A much closer alignment was found between the average total laboratory report score and the average score on the laboratory practical exam. This did not hold true for the Fall 2007 term because the course instructor required students complete the laboratory practical exam individually. Because students were allowed to work with a partner for every prior experiment, this came as a surprise to many of them, and led to an increase in anxiety as well as the time commitment required to complete the exam. We have since allowed students to work in pairs, as long as their partner during the exam has been the same partner throughout the semester. The pair would hand in one copy of the exam, and both students would receive the same score. Points would be deducted from both students if the teaching assistants noticed one person doing all of the work.

**Conclusions**

The introduction of a laboratory practical exam has shown that students were capable of designing and completing an experiment with no guidance from the teaching assistants. They were also able utilize skills and apply concepts from previous experiments involving some chemicals they have not seen before (the unknowns). Although the experiment was similar to one completed previously, it was framed in a new context, requiring more than the combination of two reagents to produce a product under the guidance of an instructor. By working independently of the teaching assistants, students have shown that they are capable of producing a product in modest yields (45 – 70%) and providing experimental evidence to support the identity of the product. From the data in Table 1, we can say that student performance on the laboratory practical exam is more closely aligned with their performance.
on laboratory reports. The collaborative nature introduced by the Science Writing Heuristic minimized the anxiety that typically comes with a final exam and has shown how well students work with each other in order to complete the task.

References


CHAPTER 6: THE CHEMISTRY OF FACEBOOK: USING SOCIAL NETWORKING TO CREATE AN ONLINE SCIENTIFIC COMMUNITY FOR THE ORGANIC CHEMISTRY LABORATORY

Adapted from a paper accepted for publication in Innovate

Jacob D. Schroeder, Thomas J. Greenbowe, and Gerry McKiernan

Abstract
Since their establishment, online social networks such as Facebook have attracted millions of members and users throughout the world. As of June 1, 2008, the Iowa State University (ISU) Facebook network included over 35,000 registered members, an increase of nearly 60 percent since 2006. While participation within the Facebook community had grown, participation in the WebCT discussion forum associated with the organic chemistry course was extremely rare. In response to this decline, the authors created a Facebook group for students enrolled in a one-semester undergraduate organic chemistry laboratory at ISU as an alternative environment to facilitate communication among students and the course instructor.

Considering the number of ISU students with active Facebook accounts, the significant time they spend viewing and updating profiles, and the fact that many students were already familiar with the various Facebook communication features, it is possible that they would be more likely to discuss course-related concepts with Facebook than with WebCT. This article focuses on the relative effectiveness of WebCT and Facebook for facilitating virtual communication among students enrolled in the course. A comparison
between the two platforms is presented along with the benefits and consequences associated with using Facebook for engaged social learning.

According to the National Science Education Standards (1996), teachers should strive to guide and facilitate learning by orchestrating discourse among students about scientific ideas. One recommendation is that students be able to articulate how they know what they know and how their knowledge connects to larger ideas, other domains, and the world beyond the classroom. Iowa State University has tried to facilitate this by incorporating WebCT with all courses. This platform includes two essential components – a bulletin board and a chat function – providing students a forum to discuss topics of interest, submit questions to other students in the course, and to engage in real-time discussions. Yet these forums were rarely used, and the level of interaction among students was minimal; instead students primarily logged on to WebCT to check their grades. This lack of communication could be due to the “content first nature” of WebCT that structures interactions around the course, the textbook, or the instructor (Maloney, 2007, ¶3; Downes, 2007, ¶20).

Thompson (2007, ¶6) notes that Web 2.0 technologies, and specifically social networking sites such as MySpace and Facebook, have a very strong influence on the lives of millions of students. This leads many educators to wonder what role, if any, social networking has in education (Joly, 2007). The 2008 Horizon Report, released by the New Media Consortium and the EDUCAUSE Learning Initiative, suggests that educators give serious consideration to emerging information technologies such as social networking services for possible educational applications and purposes. The National School Boards Association also issued a report noting that students access their profiles as well as those of
their “friends” on social networking sites nearly to the extent that they watch television (NSBA, 2007). The report consists of results from two separate online surveys of 1,277 nine- to 17-year-old students, 1,039 parents, and telephone interviews with 250 school district leaders responsible for institutional Internet use policies. The findings include:

- Almost 60 percent of students who use social networking discuss educationally-related topics online; more than 50 percent specifically discuss schoolwork
- 21 percent of students report posting comments on message boards every day; 41 percent say they do so at least once a week
- 76 percent of parents expect social networking to help their children improve their reading and writing skills or facilitate clearer expression
- 87 percent of school district leaders say “strong educational value and purpose” will be a requirement for them to permit student access to any social networking site

One of the conclusions of the report is that educators develop strategies for utilizing the educational value of social networking primarily because many of these services offer tools that inherently appeal to students, including students who are reluctant to participate in the face-to-face classroom. This has also been highlighted in a video produced by students at Kansas State University (KSU) in response to a survey that found that the typical KSU student read eight books per year yet viewed more than 2,300 web pages and 1,281 Facebook profiles (Wesch, 2007).

**Why Facebook?**

McKiernan (2007) has explored efforts using Facebook for various library outreach initiatives, reporting many of his findings on his blog and at several conferences. With Facebook being the second most trafficked social networking site, a comparison is necessary with MySpace, the most heavily trafficked social networking site. The typical MySpace users tend to be those who are ostracized in school, not fitting in with the popular high school
paradigm, while the typical Facebook users tend to come from families who emphasize education (Boyd, 2007, ¶3-4). It has been argued that the social networking site of choice for most college students is Facebook (Thompson, 2007, ¶6), having described itself as “a social utility that connects you with the people around you” (Facebook, 2008). This is certainly true at ISU: 35,454 members of the Iowa State community, including former and current students, staff, and faculty, were registered Facebook members as of June 1, 2008. By the same comparison, MySpace reported 25,714 members. And according to TechRadar, Facebook is currently the fastest growing social networking site and is predicted to equal the number of registered users on MySpace by late 2008 (TechRadar, 2008). Taken together, these findings provided the primary reasons to explore the use of Facebook for educational outreach.

While research on the use of Facebook for educational purposes is somewhat limited, a recent survey of 677 college professors by Thomson Learning shows that nearly 50 percent of the respondents familiar with social networking sites “feel such sites have or will change the way students learn” (Thomson Learning, 2007). Yet, several news reports suggest a schism exists between students who actively use Facebook and educators or administrators looking to tap into a new audience (The Guardian, 2007; Roper, 2007; Hass, 2006; Woo, 2005). This is most likely due to issues concerning privacy and security as well as what some students view as an encroachment of their own space (Sickler, 2007). This was highlighted recently when the creator of a Facebook group was threatened with expulsion for allegedly providing a forum for students to cheat (Pagan, 2008). Currently, law students at the University of Ottawa are suing Facebook, alleging 22 violations of Canada’s Personal Information Protection and Electronic Documents Act (Lawson, 2008; CBC, 2008). In
addition to privacy concerns, Bugeja (2007) argues that the use of computers during class
time can amount to a “digital distraction”. He cites examples of faculty banning laptops in
large lecture courses, which reportedly resulted in increased student attention and better
performance on exams. These are valid concerns, but the fact remains that this is the
student’s space and a place where a significant amount of time is spent in their own familiar
environment. This research is exploratory, with two questions in mind: (1) would students
discuss chemical concepts in a Facebook group more than in WebCT, and (2) would the
group become a distraction or lower student attention or exam scores?

Methods and Participants

This study took place during the Fall 2007 term, involving 128 undergraduate
students enrolled in an introductory organic chemistry laboratory for non-chemistry majors.
During the first laboratory meeting for each section, the course instructor (Schroeder)
distributed an invitation for all students to join the Facebook group (Chemistry 231 L)
(Figure 1). Any Facebook member could view the group description, but joining the group
required instructor approval. Once approved, students would be able to view the discussion
board, the wall, videos, photos, and posted items. The instructor promoted the Facebook
group as a community where students could discuss questions with one another, the course
instructor, teaching assistants, or the project librarian (McKiernan). The group was not
meant to replace WebCT but to supplement it. Grades were still posted on WebCT along
with various Web links. Students were encouraged to use WebCT to ask questions or discuss
class issues in WebCT first and then in the Facebook group if no responses came through
WebCT. The instructor would serve only as a moderator, helping to guide discussions and
not to be “friends” with any members of the group during the term. Membership in the Facebook group was optional, but 52 students eventually joined (~41%).

**The WebCT Group Page**

A general summary of student activity within WebCT for the entire term was generated to show the login frequency and the length of time the average user stays logged in (Figure 2). When generated for each month of the term, an initial spike is shown for September, but the level of activity steadily decreases throughout the remainder of the term (Table 1). Eight discussion topics took place – a total of 17 posts – none of which occurred after September 30 (Figure 3). Posts in general followed a direct question-direct answer approach. The detailed discussion thread for September (Figure 4) shows the most complex question in the second entry from the top (Sep 23, 6:40PM). This student asked the community if anyone could offer any tips in regard to naming a particular compound that was produced during an experiment. The instructor replied back the next day, but no one else in the class offered any suggestions, even though similar questions were posted and discussed on the Facebook group discussion board. Compared with the percentage of total time associated with the use of other WebCT tools, students made minimal use of the discussion board feature (Figure 5). The most frequently used feature in WebCT was the “Web Links” tool, a collection of instructor-supplied links to potentially useful external Web resources. Following is the “My Grades” feature, which allows students to see their scores for each assignment that was graded. The “Discussion” feature comes in 4th, with an average user time of only 58 seconds. This could indicate that students preferred not to use these functions on WebCT for course-related communication, or that they would check on the discussion board but quickly go elsewhere if they did not see anything new or useful.
**The Facebook Group**

The Facebook group was designed to be informal, in much the same way as the WebCT group, serving as a forum for students to ask questions relating to their laboratory experiences and try to find relevance and context (Figure 6). Similarly, in both groups contact information for the instructor was posted directly on the group home page, allowing students to have a direct link to the instructor. Within Facebook users can upload photos to complement associated text (Figure 7). This function has demonstrated to be one of the more beneficial Facebook features, as it allows anyone in the group the opportunity to respond to a comment, explanation, or observation with relevant diagrams, figures, or other graphics. In addition, the “Post Item” feature allows any group member to post Internet links to any potentially interesting or relevant Web site (Figure 8). Although students did not use this feature, the instructor used this function to post links to the American Chemical Society’s “Molecule of the Week”, a visual representation of a molecule along with a brief description about its use.

Throughout the term, twenty topics were presented on the discussion board with a total of 67 posts (Figure 9). On the WebCT discussion board, only 17 posts were made during the same time period. This disparity not only highlights the amount of time students spend on Facebook, but also how more willing they were to take part in discussions. While the WebCT discussions only generated one or two responses, in Facebook students posted ten messages dedicated to one topic on two separate occasions, with other topics generating four to six replies. A portion of one of the more popular discussion topics (“Question about lab #7”) shows the level of detail provided in the question and the give-and-take nature in the replies (Figure 10). A student started the discussion late in the afternoon, and pointed out the
fact that it was a bit late to ask a question (the report was due the next day). Despite this, the question does not specifically ask for an answer, but instead gives other students the chance to see where she is having difficulty. A classmate responded to the question in 38 minutes and recommended what the first student should do to figure the problem out. Sixteen minutes later, the first student responded agreeing with the suggestion. A third student posted a question relating to the topic seven minutes later. The instructor then replied seven minutes later with a suggestion. This exchange involved four separate individuals, contained five separate posts, and occurred in a little more than one hour.

Discussion

The first question to consider was usage, i.e., would students participate more on Facebook than they do on WebCT? Although only 41% of students eventually joined the Facebook group, the number of posts about chemistry on Facebook was nearly 400% greater than for WebCT. Many students cited these discussions in their laboratory reports.

Measuring the academic impact of these discussions, however, is problematic. With the discussion on the WebCT discussion board ceasing at the end of September, there was no longer a control group to use for comparisons. Students did not indicate why they stopped using the WebCT discussion board and switched over to the Facebook group. We are left to speculate they did so, because they were already accessing Facebook for their personal use, and from time to time checked in to see if any new topics were being discussed, just as the usage statistics for WebCT show. Unfortunately, Facebook does not have a tool for tracking usage statistics, so this is only speculation. Whether or not these discussions improved students test scores is beyond the scope of this study, because individual student exams were not collected. Overall, the averages obtained on the exams taken are similar to what they
have been in the past, which could indicate that students were no more distracted by using Facebook for the chemistry course than have ever been while using WebCT. We can report, that when an assignment is due in short order students will use Facebook to communicate and they will do so often.

Conclusions

One of the key issues regarding the effectiveness of the introductory chemistry instruction has been the low level of student participation in and outside of the class. Every student enrolled at Iowa State University has access to WebCT for every course they are registered in. In light of the high level of participation and activity by members of the Iowa State University community, a course-related Facebook group seemed a strong alternative virtual environment by which students communicate and interact. Although only 41% of students enrolled in the organic chemistry course ultimately joined the Facebook group, the level of discussion about chemistry increased by almost 400% compared to WebCT, and postings focused on more complex topics. Students never used the Facebook discussion board to appeal for answers; they used it to ask for assistance from either the instructor or other students. When the other students replied they did not give direct answers, but rather mentioned how they were approaching the problem and offered suggestions. If class data needed to be sent out or posted anywhere, the Facebook group was the first place that many of them mentioned.

Although a large percentage of Iowa State University students are Facebook members, a sizable number are not. This is evident by the 59% who did not join the group. Whether they were not registered Facebook members or if they did not feel comfortable using Facebook for classwork is an open question. If more students were to join the
Facebook group in the future, then an increase in the number of topics being discuss should also likely increase. The degree and level of complexity should also likely increase, as topics become increasingly more specific and refined as more students engage in the discussion.

Based on this significant increase in virtual student participation and engagement, we would encourage others to create Facebook groups to supplement face-to-face classroom instruction. These results have left the door open for further research into how Facebook and other social networking tools may be used as a reliable discussion board, or even a bulletin board to post announcements. To gauge academic impact, exam questions could be written reflecting some of the topics being discussed on the Facebook group. However this would require all students to join the group, and at this stage, it would appear that many students are reluctant to join. With proper promotion and management, other educators might also observe broader student engagement. In creating an online community within a platform familiar to and used by students, organic chemistry concepts were being discussed outside of class—an outcome seldom observed. Yet the more students speak the language of Organic Chemistry, the more familiar they will become with it.

References

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(accessed June 14, 2008)


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(accessed June 14, 2008)


List of Tables and Figures

Figure 1: Facebook group invitation
Figure 2: General WebCT activity summary

Table 1: Monthly usage activity

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* There were only 11 days of the semester during the month of August
Figure 3: Overview of WebCT discussion board
Figure 4: Detailed WebCT discussion thread
Figure 5: Percent usage of all WebCT tools
**Figure 6: Facebook group home page**

**Figure 7: Facebook group photos**
Figure 8: Facebook group posted items
**Figure 9: Facebook group discussion board**
Figure 10: Facebook group detailed discussion board
CHAPTER 7: GENERAL CONCLUSIONS

Summary of Conclusions

The research presented in this dissertation began after the author, as a teaching assistant for general chemistry, began using the Science Writing Heuristic approach. Following a year of teaching general chemistry he was assigned to be a teaching assistant for Organic Chemistry (Chem 331, for science majors), and many of his former general chemistry students were now in the same class. After being exposed to the SWH format, students were used to it and were annoyed at having to go from this format to the traditional format. At the time, all research on the implementation and effectiveness of the SWH was conducted in general chemistry. The possibility of introducing it to an upper level course existed, and would provide more data as to whether the methodology was effective in areas beyond general chemistry.

A research plan began to take shape during the Spring 2005 semester, as a study was underway in the general chemistry course for “soft science” majors (Chem163) investigating the use of laboratory practical examinations in concert with the SWH laboratory format (1). The results of that study indicated that these students, who entered the course with a low level of beginning chemistry knowledge and who were taught with the SWH approach demonstrated a higher level of academic success in the course as measured by the ACS CAL diagnostic exam compared to students in previous years, with similar beginning chemistry knowledge and who were not taught with the SWH approach.

Although these results were positive, the question remained whether there would be any lasting impact beyond the one term of general chemistry. If these students made significant gains on the ACS CAL diagnostic exam, then what should be expected of them
for the subsequent course many of them were required to take, Chem 231 (organic chem for non-science majors)?

These questions led to the pilot study described in Chapter 2. By following the general chemistry students who had used the SWH format into organic chemistry we were able to compare their performance on lecture exams with others who had various backgrounds of their own. After the first lecture exam, we matched the “SWH” students with “traditional” students who had identical exam scores, as long as they also had completed a chemistry course with a laboratory component the preceding semester. We found statistically significant differences between the mean scores for the two groups of students, due primarily to their performance on questions on the reaction page of the exam and specifically, on those questions that were related to concepts studied in the laboratory. Although not entirely conclusive, this led us to believe that students with the SWH background found the laboratory more beneficial than those with a traditional background.

The organic laboratory was taught using the traditional format, requiring students to adapt to a new type of laboratory experience. Despite this, they were able to apply laboratory concepts introduced in a traditional laboratory toward lecture exams better than students who were already familiar with the traditional laboratory.

A follow-up to that pilot study was undertaken during the Spring 2006 semester. Although we found similar results, performance on the exams for all students, regardless of background was poor, ranging from an average low of 37.7 to an average high score of 55.1. After the course was over, our research shifted gears to try to determine where the problems arose. This is discussed in Chapter 3, which describes some key elements which may be inherently built in to the nature of the relationship between the traditional laboratory and the
lecture. According to the laboratory manual (2) “This lab course is intended as its own course…your challenge is to get significant amounts of pure product by a thorough understanding of each manual process” (p. 6). An analysis of laboratory report scores revealed a 91.8% overall average. But many of the experiments included every possible detail of what the students were going to synthesize, how it happened, and how to analyze the data to “verify” that indeed it worked the way it was supposed to. With this in mind we sought to recraft the Chem 231 laboratory course, rewrite the manual, optimize reaction conditions, and implement the Science Writing Heuristic approach. In its first full semester of implementation, we found that overall student laboratory report scores on average decreased slightly, while at the same time their scores on laboratory-related questions on the lecture exam increased considerably.

In Chapter 4 we identified another area that may have contributed to the low scores during the Spring 2006 semester. We found that given the appropriate starting materials, students in many cases could predict the products of many reactions but could not write a reaction mechanism for them (even though an experiment with the exact same reagents took place only one week prior to the exam). We highlighted the number of students who obtained a perfect laboratory report score, which according to the grading rubric included a detailed step-by-step mechanism. This was compared to student performance during the summer term, when we used POGIL activities in the lecture along with the SWH approach in the laboratory. We were able to show that student performance on mechanism problems increased compared to the traditional group, and student attitudes toward organic chemistry in general were positive after the course concluded.
In Chapter 5, we reported our findings after developing and implementing a laboratory practical examination for the organic chemistry laboratory (non-majors). Following the theme of Chapters 3 and 4, we again identified key weaknesses in the traditional laboratory format. We found no correlation between laboratory report scores and the written multiple-choice final exam. The written final was composed of numerous quiz questions the students had seen previously throughout the semester, yet they still scored on average ten percentage points lower than the overall quiz average. Student results on the laboratory practical have generally been in the 75% - 85% range, which is closer to how well they do on their weekly laboratory reports.

In Chapter 6, we explored the use of Facebook as a discussion board to supplement WebCT. We compared the number of student postings in WebCT and Facebook to determine which forum students were more likely to use to communicate. Past usage statistics indicate that many students only used WebCT to check their grades. In this study, we found that all discussion activity in the WebCT forum ceased at the end of September. At the same time, several discussion topics opened up in the Facebook group. We noticed that the level of communication was much more detailed on the Facebook group than it ever was in WebCT. Rather than asking for direct answers, or supplying them to others who asked questions, students who asked questions would indicate where they are having difficulties in solving problems. Similarly, students who replied to comments never just gave away answers; instead they offered suggestions to help the questioner come to their own solutions.

**Implications**

One of the major implications as a result of this research is that the standard traditional “cookbook” laboratory needs an overhaul, especially in the courses for the non-
science major. In three separate instances we have pointed out how students spent three hours in a laboratory following directions to make a product, only to forget what they were doing a week later, when that same concept was tested by an exam question. If we want to turn people away from chemistry, this seems to be an approach to take to do it.

The results obtained through this research have shown that students in the “soft sciences” are indeed capable of learning abstract chemistry concepts in the laboratory and applying them toward exams given in the lecture. More importantly, written student laboratory reports indicated that several students could apply many of the concepts learned in the laboratory to areas within their own fields. The most important lesson we have tried to teach non-science majors has been how to use the scientific method. But in order to get there, students need to do science; they need to be able to experience it, whether in a laboratory or through the use of computer simulations. In both cases, students have to be able to carefully obtain data, thoroughly analyze it, and apply it to a specific question or scenario. It is important to develop new experiments that are practical or case-based, in order for the non-science major to develop any interest. This has been argued for over 50 years in the literature because part of a quality education requires at least some fundamental ability to understand basic chemical concepts, think critically and logically, and construct a form of argument or analysis using supporting evidence. For non-science majors, this seems to indicate that we should strive to equip them with the tools they will need to think like scientists, rather than to just “do what scientists do”.

The original proposal for this work was to introduce the SWH format into the chemistry majors program and based on this research, it remains an avenue worthy of consideration. The students who enroll in the introductory organic chemistry course (for
non-majors) have many of the same misconceptions as the chemistry majors do. These include writing reaction mechanisms, identifying correct products, and interpreting data. More emphasis would certainly be required on developing and practicing techniques, but put in the proper contexts, experiments could be developed in which students come to understand how and why a specific technique works and when to use it.

**Limitations**

The major limitation of this research is that it only includes those students who have taken this one course, Chem 231 for non-chemistry majors. Typically from one semester to the next, a different instructor teaches this course along with several different teaching assistants. This makes any statistical comparison between two different semesters difficult. In addition, a big difference between the fall and spring semester exists with regard to the students who enroll in one semester or another. The spring semester is considered to be the “on” sequence for students required to take only one semester of general chemistry, while the fall semester serves to catch people who had taken a second semester of general chemistry. This research also ends when these students finish taking the course. It would be of great interest to follow some of these students into even more advanced classes with laboratory components, such as biochemistry, anatomy, or animal ecology to see whether the SWH format (or some version thereof) is something a student continues to use, just from being used to it.

It would also be helpful to do more qualitative research while implementing the SWH approach. This could be done in the form of individual interviews and a more thorough coded analysis of laboratory notebooks. Although much of the research focused on quantitative aspects (exam scores), it would be beneficial to understand student reasoning
when they write down an answer. Are students spending more time trying to figure out what the question is asking rather than understanding a concept? If so, then an analysis of exam performance would not necessarily be testing whether or not a student understands the material, but rather knows how to read the question.

Another area to address would be to examine how different teaching assistants implement the SWH format. This has been analyzed in general chemistry (4), but not in the organic chemistry course. The SWH requires two templates, one for the student and one for the instructor. Simply adopting a laboratory manual that has experiments written for use with the SWH approach is not going to automatically convert a traditional laboratory into a guided-inquiry laboratory. Both teacher and student have to be fully committed to the learning process.
APPENDIX A: CONVERTED LABORATORY MANUAL

Adapted from the 4th edition, published Summer 2008 by Hayden McNeil: Plymouth, MI

Chemistry 231L
Laboratory Experiments

2008 – 2009

Jacob D. Schroeder • Thomas J. Greenbowe
Iowa State University
# Table of Contents

Introduction ........................................................................................................................................... 123  
Chem 231L Course Policies and Procedures ......................................................................................... 124  

- I. Required Equipment .................................................................................................................. 124  
- II. General Information .................................................................................................................. 124  
- III. General Techniques .................................................................................................................. 125  
- IV. General Safety Rules .................................................................................................................. 126  

General Safety Guidelines and Rules in the Chemical Laboratory .................................................... 129  
Glassware and Equipment .................................................................................................................... 131  
The Science Writing Heuristic—Brief Description .............................................................................. 133  
The Science Writing Heuristic—Detailed Explanation ........................................................................ 134  

Lab 1 Techniques—A Glimpse into the Chemist’s Toolbox ............................................................ 136  
Lab 2 Which Beverage Contains the Most Caffeine? ........................................................................ 141  
Lab 3 Margarine in the Chemistry Lab ............................................................................................... 147  
Lab 4 Alkenes: Gathering a Wealth of Evidence to Support Claims .............................................. 152  
Lab 5 Electrophilic Aromatic Substitution:  
    The Effect of Directing Groups on Benzene .............................................................................. 157  
Lab 6 “Clean” Chemistry: From Margarine to Soap? ....................................................................... 164  
Lab 7 Molecular Models: The World of 3-Dimensional Chemistry .................................................. 168  
Lab 8 Substitution vs. Elimination: The Chemical Competition ....................................................... 180  
Lab 9 Artificial Scents: The Chemistry of Fragrances ......................................................................... 186  
Lab 10 Sodium Borohydride: From a One-Time Accident to H₂ Fuel Cells ............................. 189  
Lab 11 Chemical Inventory: Preparing for the EPA Inspection ....................................................... 192  
Lab 12 The Aldol Condensation: The Carbon Construction Company ........................................... 196  
Lab 13 Radical Chemistry: Bromine vs. the Hydrocarbons ............................................................. 201
Introduction

Welcome to organic chemistry, Chem231L. To many people entering this course, organic chemistry can be described as a pretty challenging subject. To some extent this is true. In an effort to make this subject more practical, the experiments you will complete have been designed to coincide with many of the topics you will be discussing in the class, Chem231. The order in which these experiments have been arranged is not necessarily the order in which you will complete them. You will be given a copy of a syllabus outlining when the experiments will be completed.

Educational research shows that you will not learn organic chemistry just by merely following a set of instructions and copying what is already written down in the manual. For this reason, these experiments have been designed to follow an inquiry-based approach. What this means is that in most of the experiments, the “answers” are not just given to you so you can copy them down. Instead, you will discover what the answers are only after completing the experiment. This way, it is believed that in the process you will be able to develop good laboratory techniques to minimize errors, all while helping you to make conclusions based on your data.

At the end of each lab you will see headings titled Before the Lab and After the Lab. The Before the Lab bullets are not meant to be your beginning questions. Instead, they are meant to guide you to think about specific aspects of the experiment. The After the Lab bullets are meant to challenge you to think about some practical aspects of the work you just completed. This way, you may be able to develop some context for what you did in the laboratory.

Do your best to stay caught up in this laboratory as well as in the course. Do not wait until the night before your lab section meets to begin working on your laboratory report. Past experience shows this to be very unhelpful. Also, it is a good idea to take part in the end-of-class discussions with your instructor and fellow students. Each laboratory section is scheduled for three hours, but most of them will only require two hours. Use this extra hour of time to ask your instructor questions if you are unsure of any of the concepts that are introduced in this course.

Take a glance at the table of contents and see what kind of experiments you will have the opportunity to do. Organic chemistry can be difficult, but sometimes it can actually be pretty interesting. Do the best you know how to do, and be sure to ask as many questions as you need to.
Chem231L Course Policies and Procedures

1. Required Equipment

1. Safety Glasses

Safety glasses are to be worn at all times in the laboratory. This is a state law. Students without safety glasses will not be allowed to work in the laboratory. Points will be deducted if you fail to have your glasses for an experiment.

2. Lock

You are expected to provide your own lock (combination or key). The University lock on your locker will be unlocked for you during the first two weeks of the course. Points will be deducted if you fail to provide your own lock after that time.

3. Laboratory Notebooks

The laboratory notebook is what you will be expected to write in. Never come to the laboratory without it. Students will not be allowed to experiment without a laboratory notebook. We recommend the purchase of a laboratory notebook that creates carbon copies of what you write down.

4. Ink Pen

All laboratory reports are to be written in ink (blue or black). All observations and data are to be recorded in the laboratory notebook during the experiment. Any scratch paper may be taken or thrown away by your teaching assistant or instructor.

II. General Information

1. Grading

Each laboratory report will be worth 20 points. A 5-point quiz will be given for each experiment. In addition, a practical examination will be given at the end of the semester. The total points possible will be determined by how many experiments you complete. The practical exam is generally worth 20% of your overall grade.

2. Attendance

You will be expected to attend every laboratory session this semester. Conflicts known prior to the experiment (e.g., scheduled university activities) must be made known by email to the instructor in charge of the course or the TAs responsible for your section. If you miss an experiment, it is your responsibility to notify your TA as soon as possible to see if you can make the missed laboratory up in another section. You will be able to make up an experiment only during the week that it is scheduled. Contact a TA from another section to see if there is room for you to make up the experiment in his/her section. Failure to make up a missed experiment will result in a score of 0 for that laboratory report. You will only be able to make up a missed laboratory session twice during the semester. Any missed experiments beyond the two will result in no grade.

3. Laboratory Reports

You are expected to write a report covering what you did in the laboratory for each experiment you perform. These reports will be due one week after the completion of the experiment. Failure to hand in a laboratory report on the due date will result in a reduction of points.
Reports will be deducted 2 points per school day beyond the due date, but will not be accepted beyond one week from the original due date.

4. Check-in Procedure
Go to the locker assigned to you by the instructor. Fill in your name, locker number, date, university ID number, and local address in the spaces provided on page xii.

Carefully check the equipment in your locker against the equipment list given on page xiii. Make sure all glassware is clean and has no cracks or chips. Replacement items may be obtained from the laboratory storeroom in the center of the laboratory. After all of the required equipment is in the lockers and in usable condition, return the completed check-in sheet to your instructor. Do not leave the laboratory until the locker assigned to you has been properly secured.

5. Clean-up Procedure
It is important that the laboratory be kept neat. The bench top, shelves, and fume hoods assigned to you must be sponge wiped at the end of each laboratory period. Of course, spills and splashes will always occur. When these accidents happen, please be considerate of others and clean them promptly. This is especially true of the balances and areas around them. Also, be sure to keep the common areas clean. This includes the area where the common chemicals are. Be sure to put lids on bottles after you use them. If problems persist in cleanliness, your TA has the authority to deduct points from each person in the class. Everyone is responsible for keeping the laboratory clean.

6. Check-out Procedure
If you wish to drop the course after checking into the laboratory, remember that you will need to drop the lecture portion of the course as well. You must talk with the instructor about this. If you do drop the course, make immediate arrangements to check out of the laboratory promptly. **Do not wait until the end of the term to check out.**

The day of checkout, the instructor will return your checkout sheet. You and your instructor will make sure that all equipment in your lockers is present and in good condition. If it is not, you are responsible for cleaning or replacing it. Remember that it is more efficient to clean and replace equipment during the term than it is to wait until the end of the semester during the rush of checkout. **Anyone failing to check out by the last laboratory period will be charged a check-out fee of $50.00.**

7. Notes
- No work may be done in the laboratory outside of the regularly scheduled periods.
- No work may be done in the laboratory unless a TA is present.
- Safety gloves and aprons are optional but highly recommended. They will be provided in the laboratory.

III. General Techniques
1. Glassware Assemblies
Your microscale glassware kit contains threaded glass joints, which can be joined with threaded nylon connectors. When assembling an apparatus, the connectors should be tightened to a joint until it is snug. Overtightening a connector to a
threaded glass joint will cause the joint to break away from the glassware.

The vials and flasks in your microscale glassware kit are equipped with threaded black caps and septa. One side of each septum is soft silicone, while the other side is Teflon—a chemically inert white material. For best results, the white Teflon side of the septum should be on the inside of the cap and the soft silicone side should be on the outside of the cap.

2. Massing and Transferring Reagents
It is helpful to transfer reagents directly to and from a tared (pre-massed) container. This way, differences in masses can be easily determined. Solid chemicals can be placed on a watch glass or directly into a reaction vessel. When transferring small amounts of reagents, it is important to minimize losses. Therefore, liquid reagents should be transferred with a pipette or syringe. Small amounts of liquids are seldom poured.

3. Heating and Cooling Methods
When heating your glassware, the hot plate assembly can be used. Alternatively, an electric heating mantle and a regulator can be used. Your glassware is made of Pyrex and will withstand a great range of temperatures. Thick-walled conical vials, however, should not be exposed to sudden changes in temperature. Therefore, you should not heat the thick-walled conical vials directly on a hot plate. Sudden temperature changes in conical vials will cause the thick glass walls to expand or contract unevenly, which will result in a cracked vial.

4. Using and Storing Reagents
Many reagents are volatile and give off a strong odor. Moreover, some reagents react with oxygen and water. For this reason, stock reagents should be capped whenever they are not in use. Experiments should be done in a fume hood whenever possible. Other reagents are corrosive. Aprons, safety goggles, and protective gloves should be worn whenever working in the laboratory.

5. Waste Disposal
Chemical waste should be disposed of safely. *Never pour chemical waste down a sink drain.* Always use an appropriate waste container. Used paper towels should be discarded in wastebaskets. When placed in the sink, paper towels will plug the drains. Broken glass is always placed in the boxes reserved for broken glass. Used syringe needles are disposed of in appropriately labeled metal cans. Avoid inadvertently discarding stir bars, small o-rings, and IR salt plates.

IV. General Safety Rules

1. Eye Protection
Always wear safety glasses. These are required by law. You will not be able to work in the laboratory without your safety glasses. If any chemicals get in your eyes, flush your eyes immediately by using the eye wash station.
2. Handling Chemicals

Minimize direct physical contact with all chemicals. Protect your hands from toxic and/or caustic materials by using a pair of gloves in the laboratory. Laboratory aprons have been provided in an attempt to protect students from spills and splashes. Wash your hands before leaving the laboratory.

3. Fume Hoods

Chemists use fume hoods to protect themselves and others from exposure to materials that produce vapors, fumes, and dust. When the fume hoods are working properly, they will draw a significant volume of air from the laboratory. Like any tool, the fume hood should be used properly if it is going to remove odors from the laboratory.

The following guidelines are suggested:

- Sash openings should be kept at a height of 12” or less
- To minimize odors, all transfers of solvents should be made inside the hood whenever possible
- Users should keep their heads outside of the plane of the sash
- Use the lights in the hood and maintain a clean sash window
- Exhaust ports and supply vents should be free of debris
- Remain alert for any changes in airflow
- Report persistent problems to your instructor

4. Fire Hazards

Many organic chemicals and their vapors are highly flammable. Be aware of and avoid potential risks. Chemicals, equipment, books, etc. are easily replaced. You are not!

To minimize fire hazards: use a heating mantle, a hot plate, or a steam bath in the hood for heating liquids. Use a Bunsen burner only when directed to do so. Check your assembly carefully before heating it. (Make sure you are not heating a closed system, and all glass joints are fitted together properly.)

In the event of a fire, immediately alert your instructor and others around you. Move away from the fire, but do not panic. Close any open gas valves. For burning clothing, smother the flames with a fire blanket (in a horizontal position). If small quantities of chemicals catch on fire, most fires will simply burn out quickly. Use a fire extinguisher if necessary.

Experiments are carefully designed to minimize hazards. Read the directions for each experiment and make sure you understand them before going into the laboratory. If you have questions, ask your instructor.

5. Safety Equipment

Fire extinguishers, eye wash stations, fire blankets, safety showers, and first aid kits have been provided in the laboratory. Know their locations and be knowledgeable about their use.
6. Use of Chemicals
Read chemical labels carefully! When you are finished with a chemical, replace the bottle cap and return the bottle to its proper location. Clean all spills immediately and carefully. Report all serious spills to your instructor. Please take only what you need for an experiment. Do not, however, return excess materials to a reagent bottle. Pass them on to someone else or put them into a waste bottle. Do not pour solvents into the sinks or drain troughs.

7. Accidents
In the event of an injury, your instructor should be notified immediately. For anything beyond a very minor injury, a student accident form should be completed and signed. These forms are helpful in preventing future accidents and are available in Rm. 0732, the Chemistry Laboratory Storeroom.
Students who require the treatment by a nurse or physician can be examined at Student Health Service. Any costs of an initial treatment at the Student Health Service not covered by a student’s own health insurance will be paid by the Chemistry Department. If needed, transportation will be furnished.

8. Before Leaving the Laboratory
Make sure you have put all of your equipment away and have returned checked-out items to the storeroom. Sinks should be free of debris and bench tops should be wiped clean. Make sure all water, gas and steam valves have been closed. Clamps and hoses should be returned to their proper location. Wash your hands before leaving the laboratory.
General Safety Guidelines & Rules in the Chemical Laboratory

Iowa State University, Department of Chemistry

The following safety guidelines and rules must be followed at all times while you are in the laboratory. Failure to follow these procedures will result in your immediate dismissal from the laboratory session. Willful repeated noncompliance provides adequate reason for expulsion from the course.

1. Since your eyesight can be permanently damaged by either spilled chemicals or flying glassware you must, according to Iowa State Law, wear approved eye protection over your eyes in the laboratory at all times.

2. Know the location of all safety equipment in the laboratory. Each laboratory has a fire extinguisher, eye wash fountain, safety shower, main gas shutoff valve, and emergency exits. In the event of a fire alarm, know the proper route for exiting the building.

3. Appropriate clothing must be worn at all times in the laboratory. A laboratory apron will be provided. Shoes or sneakers must be worn to protect your feet from accidents: possible chemical spills or possible broken glassware. Sandals are permitted but are not encouraged.

4. Long hair must be tied back so that it does not accidentally come in contact with a burner flame, equipment, or any chemicals.

5. Neither food nor drink is allowed in the laboratory at any time since the danger of contamination with noxious or toxic substances is present. In no circumstance is anyone allowed to smoke in the chemical laboratory.

The health of others, your need to concentrate, and the possible presence of flammable substances precludes smoking.

6. All chemical spills or drips of reagents must be immediately cleaned up. Use wet paper towels or a brush and pan. Dispose of waste material properly. Do not place paper in the sinks or troughs.

7. Always read any label carefully before removing a chemical from a reagent bottle. Always make sure that you have obtained the correct reagent and the proper concentration. Serious accidents may occur when the wrong substances are mixed.

8. Never contaminate a reagent bottle. Do not insert anything (spatula, etc.) into a reagent bottle. Reagents should be obtained from a reagent bottle by pouring the chemical into a clean container. Do not pour chemicals back into a reagent bottle, unless specifically told to do so. Either share the chemical with someone who needs to use it or consult your instructor.

9. Come to the laboratory prepared to do the experiment. Lab work is permitted only during the schedule lab periods under the direct supervision of a qualified instructor. Do not perform any unauthorized experiments. Students who do not conduct themselves in a mature manner or who endanger themselves or others by their actions will be told to leave. Do not remove any chemicals from the laboratory.

10. Report all accidents and injuries to your instructor. Dial 911 to report emergencies.

I did read and do understand the above guidelines: ________________________________

Section Number____________________ Date____________________________________
**Complete at Check-in:** All of the equipment listed is in good condition and is in my locker.

<table>
<thead>
<tr>
<th>Name ___________________________</th>
<th>University ID ___________________________</th>
</tr>
</thead>
<tbody>
<tr>
<td>Local Address ___________________</td>
<td>Locker Number __________________________</td>
</tr>
<tr>
<td>Signature of Teaching Assistant</td>
<td></td>
</tr>
</tbody>
</table>

**Complete at Check-out:** All of the equipment listed is in good condition and has been returned to the locker.

<table>
<thead>
<tr>
<th>Signature of Teaching Assistant</th>
<th>Date __________________________</th>
</tr>
</thead>
</table>
# Glassware and Equipment

<table>
<thead>
<tr>
<th>Qty.</th>
<th>Glassware and Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Air condenser, 100-mm</td>
</tr>
<tr>
<td>1</td>
<td>Claisen adapter</td>
</tr>
<tr>
<td>1</td>
<td>Three-way adapter</td>
</tr>
<tr>
<td>1</td>
<td>Straight drying tube, 50-mm</td>
</tr>
<tr>
<td>2</td>
<td>Round-bottom flask, 10-mL</td>
</tr>
<tr>
<td>1</td>
<td>Hickman distillation head</td>
</tr>
<tr>
<td>2</td>
<td>Conical vial, 3-mL</td>
</tr>
<tr>
<td>2</td>
<td>Conical vial, 5-mL</td>
</tr>
<tr>
<td>1</td>
<td>Water-jacketed condenser</td>
</tr>
<tr>
<td>1</td>
<td>Vacuum adapter</td>
</tr>
<tr>
<td>1</td>
<td>Thermometer connector, blue</td>
</tr>
<tr>
<td>3</td>
<td>Small connector, blue</td>
</tr>
<tr>
<td>2</td>
<td>Intermediate connector, blue</td>
</tr>
<tr>
<td>2</td>
<td>Large connector, blue</td>
</tr>
<tr>
<td>3</td>
<td>Hose connector, blue</td>
</tr>
<tr>
<td>2</td>
<td>O-ring, Viton</td>
</tr>
<tr>
<td>2</td>
<td>PTFE stir bar, micro, (1/8&quot; × 1/2&quot;)</td>
</tr>
<tr>
<td>2</td>
<td>PTFE spin vane</td>
</tr>
<tr>
<td>1</td>
<td>Filter flask, 25-mL</td>
</tr>
<tr>
<td>1</td>
<td>Round-bottom flask, 25-mL, threaded</td>
</tr>
<tr>
<td>1</td>
<td>Hirsch funnel</td>
</tr>
<tr>
<td>1</td>
<td>Vacuum gasket (rubber)</td>
</tr>
<tr>
<td>1</td>
<td>Bent drying tube</td>
</tr>
<tr>
<td>1</td>
<td>Glass syringe, 2cc, plunger and barrel</td>
</tr>
<tr>
<td>1</td>
<td>NMR tube and cap</td>
</tr>
<tr>
<td>1</td>
<td>Beaker, 100-mL</td>
</tr>
<tr>
<td>1</td>
<td>Beaker, 150-mL</td>
</tr>
<tr>
<td>2</td>
<td>Beaker, 200-mL</td>
</tr>
<tr>
<td>1</td>
<td>Beaker, 400-mL</td>
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<tr>
<td>1</td>
<td>Beaker, 600-mL</td>
</tr>
<tr>
<td>1</td>
<td>Cylinder, graduated, 10-mL</td>
</tr>
<tr>
<td>1</td>
<td>Cylinder, graduated, 100-mL</td>
</tr>
<tr>
<td>2</td>
<td>Flask, Erlenmeyer, 25-mL</td>
</tr>
<tr>
<td>2</td>
<td>Flask, Erlenmeyer, 50-mL</td>
</tr>
<tr>
<td>2</td>
<td>Flask, Erlenmeyer, 125-mL</td>
</tr>
<tr>
<td>1</td>
<td>Flask, Erlenmeyer, 250-mL</td>
</tr>
<tr>
<td>1</td>
<td>Water-jacketed condenser</td>
</tr>
<tr>
<td>1</td>
<td>Funnel, glass, 35 × 50 mm</td>
</tr>
<tr>
<td>2</td>
<td>Watch glass, 3–5”</td>
</tr>
<tr>
<td>1</td>
<td>Round-bottom flask, 50-mL, 19/22</td>
</tr>
<tr>
<td>1</td>
<td>Round-bottom flask, 100-mL, 19/22</td>
</tr>
<tr>
<td>1</td>
<td>Separatory funnel, 125-mL, 19/22</td>
</tr>
<tr>
<td>1</td>
<td>Stopper, glass, 19/22</td>
</tr>
<tr>
<td>1</td>
<td>Stirring bar, magnetic, 1”</td>
</tr>
<tr>
<td>1</td>
<td>Brush, 1/2”</td>
</tr>
<tr>
<td>1</td>
<td>Brush, 3/4”</td>
</tr>
<tr>
<td>1</td>
<td>Burner, semi-micro</td>
</tr>
<tr>
<td>2</td>
<td>Centrifuge tube, with cap, 15-mL</td>
</tr>
<tr>
<td>1</td>
<td>Centrifuge tube, with cap, 50-mL</td>
</tr>
<tr>
<td>1</td>
<td>Clamp, test tube holder</td>
</tr>
<tr>
<td>2</td>
<td>Spatula (1 double-ended)</td>
</tr>
<tr>
<td>1</td>
<td>Stirring rod, glass, 10”</td>
</tr>
<tr>
<td>1</td>
<td>Tongs, crucible</td>
</tr>
<tr>
<td>12</td>
<td>Test tube, 10 × 75 mm</td>
</tr>
<tr>
<td>6</td>
<td>Test tube, 22 × 175 mm</td>
</tr>
<tr>
<td>1</td>
<td>Test tube rack</td>
</tr>
</tbody>
</table>
The Science Writing Heuristic—A Brief Description

**Beginning Questions (2 points)**
What are you trying to figure out by doing an experiment?

**Safety (1 point)**
What specific cautions need to be taken? What kinds of chemicals will you be using?

**Procedure (1 point)**
How are you planning on performing your experiment? How much material are you using? How are you going to collect and identify what your product is?

**Data (4 points)**
What were the masses of the starting material(s) and the product(s)? Did anything happen that could help explain a poor yield? All calculations need to go here in addition to any observations that you deem relevant to the experiment.

**Claims (2 points)**
You asked a question to start the experiment off. That was the reason why you came to the laboratory in the first place. After completing the experiment, what did you find out? Can you answer your beginning questions?

**Evidence (5 points)**
A claim is only as good as the evidence you can provide to support it. Imagine getting into an argument with someone who won’t believe what you are saying until they can see the proof. You need to highlight anything in the class data that helps support what you are claiming.

**Reading and Reflection (5 points)**
These experiments are designed to coincide with the topics you will be covering in lecture. What is the correlation between the laboratory and the lecture for the experiment you did? Use your textbook in addition to the Internet to find practical meaning behind what you did for the experiment. Also, account for any errors that happened. This can help you explain the purity of your compound as well as your percent yield.
The Science Writing Heuristic— Detailed Explanation

**Beginning Questions**

After reading through the experimental details, you should be able to think of some questions that can be answered by coming into the laboratory. Most questions have a cause and effect nature. Prior students have asked some of the following questions: What is the relationship between the melting point of a substance and the purity of that substance? What effect do different functional groups attached to a benzene ring have on the substitution pattern during electrophilic aromatic substitution? Is there a relationship between the structure of the starting material and the type of reaction it will undergo? These questions all require experimental work in order to answer. Generally, these questions should serve as a basis for why you are doing the experiment, i.e. what you are trying to figure out. There are some questions that are not productive. Some of these include: What is the color of my product? What is a percent yield? What is a drying agent? These questions usually can be answered without doing any laboratory work at all and are therefore not useful. When coming up with questions, try to think of something the whole class can do and by pooling data, a trend can be developed.

**Safety**

In the organic laboratory, safety is the number one priority. Many of the chemicals you will be using are much more dangerous than those that you worked with in general chemistry. Make this section specific to the experiment that you will be doing. Every experiment requires safety glasses and gloves, that is a given. What chemicals are you specifically dealing with, though? There are safety hazards associated with each experiment, but you need to be aware of how you will protect yourself as well as others from chemicals that are dangerous. Will you be prepared should something go wrong? The point here is that you know what you are working with, as well as what you will do in the event of an accident.

**Procedure**

Rather than just copying a procedure down that is given to you, all you need to do is summarize what you plan on doing in the laboratory. If someone were to use just the procedure in your laboratory notebook, would they be able to successfully complete the experiment? For the practical exam given at the end of the semester, you will be able to use your laboratory notebook, but not the laboratory manual. You will want to be able to reproduce your results, since you don’t know what will show up on the practical exam.

**Data**

From your procedure, you may need to start with 3 g of starting material. But how much did you actually use? The balances in the laboratory go to four decimal places. In keeping true to significant figures, you need to copy all of those numbers down. This will ensure you have a more accurate value for your theoretical yield and percent yield when you calculate those values. It is also important for you to draw the reaction scheme for the experiment you are doing. This allows someone to
know what you are doing just by glancing at your notebook. It is important to keep good observations as well. If some of your reaction mixture spills on the bench, your percent yield will decrease. When it comes time for you to explain why your percent yield is lower than another group’s, this observation can be used to explain why. Remember, someone else may try to replicate your work to make sure you are honest. If you see a color change, the formation of a gas, or the formation of two layers, you will want to write that down to point it out. This way, someone else will know what to look for when doing the experiment.

**Claims**

After completing your experiment and comparing with other members of the class, you should be able to answer your beginning questions. If you asked if there was a specific relationship between the structure of the starting material and what reaction pathway it would take, you should be able to answer that question after you see more data from other groups. If you have difficulties coming up with any claims, chances are you will need to revisit your beginning question.

**Evidence**

After making your claim, you need to support it with evidence. You will be able to gather a wealth of evidence by obtaining spectral data and melting points in addition to a host of other techniques. You should also draw out the reaction mechanism to show how your product was produced. You can use a class data table if it shows a trend, but you are responsible for explaining the trend. A class data table with no explanation is about as good as no class data table at all.

**Reading and Reflection**

One of the most important aspects of doing experimental work is learning something practical. After completing the experiment you need to ask yourself if what you did makes sense. Does it tie into any topics that you are covering in the lecture? If so, how? You will also want to address any issues regarding your percent yield or the purity of your product. Reflection also asks you to do a little bit of research over the topic that you are experimenting with. With information so readily accessible online, use a search engine to find some information relating to what you did. Many of these experiments tie in with many important aspects of daily life. These include trans fats, artificial flavors, biodiesel production and hydrogen fuel cells, to name a few. These experiments are not meant to just keep you busy for three hours as part of a requirement. It is hoped that you will gain an understanding of the chemistry involved in many of these important issues. If you know the science behind something, you will be more able to make an educated decision. You can use such search engines as Google® or Dogpile®, or some free online encyclopedias such as Wikipedia®. Either way, there is a substantial amount of information available that may help put your experiment into some context. If WebCT is being used, take advantage of the chat rooms, forums, or anything else that your instructor builds into the page.
Lab 1 – Techniques: A Glimpse into the Chemist’s Toolbox

Introduction
Before you can really get your hands dirty in the laboratory, you need to be introduced to some techniques that you will be using throughout the semester. It would be pretty easy for the storeroom manager to give you a slew of compounds so that you could run various tests on them, but realistically, how practical is that? The idea in this experiment will be for you to actually make a pretty important type of compound. You may be using this compound in a later experiment, but before you can use it, you need to make sure it is what you claim it is.

The Big Idea
Imagine for a moment that you are working at a big-time pharmaceutical company, producing various drugs that millions of people will be ingesting. Should the consumer worry about what is all packed into that little pill? The average consumer doesn’t have the ability to crush up the little pill and analyze it to confirm that what the box says about it is true. Therefore, a lot of trust is put into the company, that they are hiring competent scientists. If something gets out to the public that isn’t pure, or perhaps is mislabeled, the company will most likely end up in court facing millions of dollars under a class action lawsuit. The result is an increase in prices for everybody else to help alleviate the financial burden experienced by the company. This is just a medical example, but similar comparisons could be made for quality control scientists in various other fields. Imagine an agronomist spraying a fertilizer on your lawn that ends up killing the grass or a horticulturalist who grows a plant promoting a disease rather than fighting it.

The Techniques
Techniques used in quality control are numerous. In this experiment you are asked to focus on two main methods. They are melting point comparison and recrystallization. In the example of the pharmaceutical chemist, the product that was obtained was a solid. When solids are made in the laboratory, one of the most common methods used to determine the purity of it is to simply take the melting point of it. If it is a known compound, all you need to do is compare the experimental melting point you found to the known melting point given in the literature. Although on the surface this may sound simple, there are many things that go into obtaining an accurate melting point. First of all, the compound needs to be heated slowly. If the compound is heated too rapidly the temperature will increase too quickly, leading to a melting point with a broad range. Second, the melting point range starts when you first see visible signs of melting. Once the entire sample is melted, the temperature is once again recorded. The two temperatures define the range in which the compound melts. To determine the melting point, you will use a Mel-Temp apparatus. The optimal setting is one in which the temperature increases about 1 degree per 3 or 4 seconds (should be somewhere around 3 or 4 on the Mel-Temp dial). After obtaining the melting point, a comparison to the known melting
point will tell you what the next step in the process is. In most all cases, the first time the melting point is taken it will be a few degrees off, or the range will be too broad. In this case, you will need to purify your compound; this will be done by a process known as recrystallization.

In its broadest sense, all recrystallization involves is dissolving your solid in a suitable solvent and then waiting for the solid to form again (crystallize). If impurities are present in the sample, they usually stay dissolved in the solvent. The crystal can be collected by filtration using the Hirsch funnel. Now of course, there is more to it then that. You need to find a suitable solvent; you can’t just use anything that happens to be on the bench top. Solvent selection will comprise the second part of this experiment.

**Part 1: Preparation of Acetanilide**

Let’s go back to the pharmaceutical chemist mentioned above. He’s been given the task to make a compound called acetanilide, the structure of which is shown to the right. This compound is important for a variety of reasons. It is used as an inhibitor in hydrogen peroxide and is also used to stabilize cellulose ester varnishes. In the pharmaceutical industry, acetanilide was found to have analgesic and fever-reducing properties. Not surprisingly, it is found in the same class of drugs as acetaminophen. It was used as a precursor in the synthesis of penicillin as well as a host of other pharmaceuticals. Unfortunately, it was discovered in 1948 that acetanilide could lead to a condition called methemoglobinemia ("brown blood") ultimately leading to liver and kidney damage. Acetaminophen showed none of these toxicity issues, so it quickly replaced acetanilide.

Knowing this information, the chemist went to the literature and found a procedure for making acetanilide using a fairly straightforward approach that doesn’t take much time at all. This procedure is shown below.

**Procedure**

In a 125-mL Erlenmeyer flask, mix 1 mL (0.012 mol) concentrated hydrochloric acid with 30 mL of distilled water. To this solution, add 1.1 mL (1.12 g, 0.012 mol) of aniline and stir the solution for a few minutes. In a separate flask, prepare a solution of 1.8 g (0.013 mol) of sodium acetate trihydrate (CH$_3$CO$_2$Na • 3H$_2$O) in 4 mL of water.

To the first solution, add 1.6 mL (1.66 g, 0.03 mol) of acetic anhydride and swirl the contents to mix evenly. Immediately add the sodium acetate solution and mix the reactants thoroughly by swirling.

Cool the reaction mixture by placing the reaction flask in an ice water bath and stir vigorously while the product crystallizes. Collect the crystalline product using a Hirsch funnel and a filter flask. Wash the crystals with cold water and allow them to dry. A typical yield is 1.0–1.4 g. If necessary the product may be recrystallized from water. One gram of acetanilide dissolves in 185 mL of cold water or 20 mL of boiling water. Ethanol may also be used for the recrystallization of acetanilide. One gram of acetanilide dissolves in 3.4 mL of ethanol or 0.6 mL of boiling ethanol.
With a procedure in hand, you should be ready to get things moving. Being this early in the course, however, your knowledge of many of the experimental details may be somewhat limited. But there are a few things that should be pointed out. Take a look at the mole ratios being used for the starting materials. What do you notice? This is most likely what the chemist will be looking at first, since he has to mass-produce this substance on a much larger scale. It was mentioned earlier that you might be using the acetanilide for a later experiment. Chances are you may want to modify this procedure if you want a little more product to work with. You do have the luxury of working with a partner, so here is a suggestion. Each of you can go ahead and prepare some of this compound. At the end of the experiment, you and your partner can combine what you have made. The procedure says a typical yield is around 1–1.4 grams. If you work together, you should expect somewhere in the neighborhood of 2–3 grams of product.

Safety Concerns

Concentrated hydrochloric acid is a very strong acid with nasty fumes. If you get any on you, rinse it off immediately under the sink for several minutes. If necessary, have someone get the instructor. Be sure to wear safety glasses as well as gloves. Aprons are also available if you choose to wear one.

Part 2: Analysis

After preparing the acetanilide, both you and your partner should have some solid. What does it look like? Does your sample look similar to your partner’s? To obtain a melting point, you will need to get a melting point tube. It has an opening on one end. To get some sample into it all you need to do is press the open end into a small amount of your solid sample. You’ll need to get that sample down to the bottom end of the tube; once you do that you’re ready to go with the Mel-Temp. Your instructor can assist you with operating the temperature device.

Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aniline</td>
<td>93.13</td>
<td>–6.3</td>
<td>184.1</td>
<td>1.02</td>
</tr>
<tr>
<td>Hydrochloric acid (12 M)</td>
<td>36.46</td>
<td>—</td>
<td>—</td>
<td>1.18</td>
</tr>
<tr>
<td>Sodium acetate trihydrate</td>
<td>136.08</td>
<td>324</td>
<td>—</td>
<td>1.45</td>
</tr>
<tr>
<td>Acetic anhydride</td>
<td>102.10</td>
<td>–73.1</td>
<td>139.9</td>
<td>1.08</td>
</tr>
<tr>
<td>Ethanol</td>
<td>46.07</td>
<td>–114.3</td>
<td>78.4</td>
<td>0.79</td>
</tr>
<tr>
<td>Acetanilide</td>
<td>135.17</td>
<td>113–115</td>
<td>304</td>
<td>1.22</td>
</tr>
</tbody>
</table>
Once you’ve obtained the melting point, it’s time to recrystallize (purify) the compound that you made. As mentioned earlier, you’ll first need to select the appropriate solvent. The procedure that our chemist found mentioned both water and ethanol as a choice for recrystallizing. You are given ratios of how much solvent is required to dissolve acetanilide. There’s a pretty big difference between the two based on the information given. What can you say about it?

The ideal solvent for recrystallizing is one in which the solid does not dissolve at room temperature. If it does, how would you get any solid out? Knowing this information, you will be able to do the first test to determine whether you want to use water or ethanol. Place a small spatula tip-full of your solid product in each of two small test tubes. Add about 3 mL of cold water to one of the test tubes and about 3 mL cold ethanol to the other. What do you see? Can you eliminate one of the solvents based on this first test? If you cannot, the next step is to heat the tube in a hot water bath (temperature of ~65°C). You won’t want to have the hot water bath boiling; remember, ethanol boils at 78°C and water boils at 100°C. If placed into boiling water, ethanol will quickly boil away, and you won’t have any solvent left. Heat the test tube for about 5 minutes or until the solid inside the test tube completely dissolves.

If it doesn’t all dissolve in 10 minutes, then chances are that it never will. If this happens, then you can eliminate that solvent.

Once the heating process is complete, allow the test tube to cool slowly. After awhile, you may see some crystals begin to form as the solution cools further. If you see this happening, then you have found a suitable solvent to recrystallize with. A suitable solvent for recrystallization is one in which the solid does not dissolve in the cold solvent, but does dissolve once the solvent is heated. Once you determine your solvent, you can use the ratios that were provided in the procedure to carry out the recrystallization on your entire sample.

Clean Up

Nothing from the experiment goes down the drain. All chemical waste should be discarded into the appropriate waste container found in the chemical hood. Glassware can be cleaned by rinsing it with a small amount of acetone. After you have recrystallized and collected your sample, take the final mass of it. Combine your sample with your partner’s in a vial and leave it in your locker. As always, make sure the area that you worked in is clean when you finish up.

Before the Lab

- What information determines whether a solvent is suitable for recrystallization?
- What is the best way to determine a melting point (heating rates)?
- Do you remember how to calculate theoretical yield and percent yield? If not, it would be a good idea to look up how.
- How does the range in the melting point relate to the purity of the product?
After the Lab

• Look up recrystallization using a search engine such as Google® or an online encyclopedia such as Wikipedia®. What information can you find that relates to what you did for this experiment?

• What effects can impurities have on the melting point of a substance?

• Calculate the theoretical yield and the percent yield.
Lab 2 – Which Beverage Contains the Most Caffeine?

Introduction
What comes to mind when you hear the term “psychoactive?” According to www.dictionary.com, psychoactive is defined as “affecting the mind or mental processes.” Such terminology is often used to describe the effects of legal prescription drugs, as well as a host of illegal drugs. The “drug” that you will be looking at is quite possibly the most popular psychoactive drug on the planet—caffeine. Caffeine belongs to a class of compounds known as xanthine alkaloids. Other compounds found in this class include theobromine (chocolate) and theophylline (black or green tea). The alkaloids as a whole contain an even greater number of compounds such as the opium alkaloids and the compounds responsible for the amphetamines.

Caffeine is found in coffee, tea, and cocoa and is an ingredient in over 70% of the soft drinks consumed in the United States. It has been estimated that the per-capita intake of caffeine in the U.S. ranges from 170 to 200 mg/day.

To put context to this range, you will be determining how much caffeine is in a sample of a variety of beverages. The samples you will analyze are coffee, tea, and an assortment of commercial sodas.

As a class, you will be responsible for finding out which of these beverages contains the most caffeine per volume.

The Big Idea
In order for you to isolate caffeine, you will need to decaffeinate a beverage that contains it. Experimentally this can be done using a technique called extraction. The process of extraction depends on the polarity of two different substances. You may already be familiar with the concept of “like dissolves like.” Consider the following example: What happens when you shake up a bottle of Italian salad dressing? You’ll see some bubbles forming and a solution that seems to be mixed. After letting it stand awhile, though, you’ll see two separate layers. This happens because one substance is polar (aqueous vinegar), while the other substance is nonpolar (oil).

Since the two substances are different in terms of polarity, they will not stay mixed. In addition to polarity, extraction takes advantage of the density of solutions. Consider what happens when you drop a few ice cubes in a glass of water. The ice cubes will float because they are less dense than water. Similarly, in our salad dressing example, the oil layer is on top of the aqueous vinegar layer because the oil is less dense than the vinegar.
You have an item of glassware in your lockers called a separatory funnel that will allow you to separate the layers.

Since you are building an arsenal of techniques to employ throughout this course, another one you will learn about will be Thin Layer Chromatography (TLC). This process is useful for showing the purity of compounds, monitoring a reaction to determine if it is complete, and in some cases, for product identification. Since extraction makes use of the differences in solvent polarity, the two techniques work hand in hand. To do a TLC, you’ll need to have a TLC plate. It looks just like a plastic plate, but on one side has a rough coating of a polar material. You can spot a sample on this coating and then develop it by putting it in a developing jar that contains a developing solvent. This process is illustrated below.

In step 1, you can use a capillary tube to draw a small amount of the solution containing your product. You can spot the plate just by lightly pressing the capillary tube onto the rough surface of the plate. Once your spot has dried, you can put the plate into a jar with a small amount of developing solvent (step 2). Depending on the polarity of your product, it will travel up the plate with the solvent. In order to see how far up the plate the product traveled, you will need to shine a UV lamp on the plate (step 3).

Step 4 shows a plate in which two substances were spotted. The spot on the left represents a polar compound; the spot on the right represents a non-polar compound. The non-polar compound will travel further up the plate, because it doesn’t “mix” well with the polar backing on the plate. In order to quantify this, you can calculate an Rf value (see figure on following page). This value is obtained by dividing the distance the spot traveled by the distance the solvent traveled.

**Safety**

- 10% NaOH solution is a very strong base. Caution should be taken when using this solution, as it can cause burns.
- CH$_2$Cl$_2$ is a carcinogen. Try not to get this on you, and be careful of the vapors. Be sure to wear gloves. Remove them immediately if at any time you spill any CH$_2$Cl$_2$ on yourself.
- You will be using a UV lamp to develop your TLC plates. Ultraviolet light is harmful so do not look directly into the lamp, as it can damage your eyes.
Developed TLC plate
A: Standard containing two components x and y
B: Sample 1 containing x
C: Sample 2 containing y
D: Sample 3 containing y and another, unknown compound

Solvent front at 8 cm:
$R_f$ for x = $\frac{2 \text{ cm}}{8 \text{ cm}} = 0.25$

$R_f$ for y = $\frac{5 \text{ cm}}{8 \text{ cm}} = 0.625$

---

**Procedure**

**Coffee and Tea**

You’re going to have to brew both of these. You can work in partners, but each person will need to do their own extraction. Add 100 mL of distilled water to a 250-mL beaker and heat on the heating mantle. There’s no need to boil the water, but crank the heat up to get it warm. Just before the water reaches boiling, carefully remove the beaker from the heating mantle and drop in one tea bag or one coffee bag. Allow the bag to brew for about 10 minutes, squeeze all of the liquid out, and then remove the bag. At this point the bag can be thrown away. Cool the beaker in an ice water bath for about 5 minutes. Once the solution is close to room temperature, remove the beaker from the ice water bath.

---

**Reagent Data**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methylene chloride (dichloromethane)</td>
<td>84.93</td>
<td>−96.7</td>
<td>39</td>
<td>1.33</td>
</tr>
<tr>
<td>Water</td>
<td>18.02</td>
<td>0</td>
<td>100</td>
<td>1.00</td>
</tr>
<tr>
<td>10% aqueous NaOH</td>
<td>40.00</td>
<td>~0</td>
<td>~100</td>
<td>~1.00</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>88.11</td>
<td>−83.6</td>
<td>77.1</td>
<td>0.89</td>
</tr>
<tr>
<td>Hexanes</td>
<td>86.18</td>
<td>−95.0</td>
<td>69.0</td>
<td>0.65</td>
</tr>
<tr>
<td>Caffeine</td>
<td>194.19</td>
<td>237</td>
<td>−</td>
<td>1.2</td>
</tr>
</tbody>
</table>

**Soda**

Add 100 mL of the soda of your choice to a 250-mL beaker (make sure you and your partner are doing the same soda). If you started with the coffee or the tea, you should now have a beaker containing roughly 100 mL brewed coffee or tea. (From here out the directions pertain to all beverages.) Use a strip of pH paper to test
the pH of your solution. In order to extract the caffeine, you will need to change the pH to around 8–9. This can be achieved by adding ~1 mL of 10% aqueous NaOH. Add this dropwise using a pipette and after stirring the solution, test the pH again. Compare the color of your pH strip to the guide posted on your fume hood. If necessary, add additional NaOH to change the pH of your solution.

Once you have the correct pH, clamp your 125-mL separatory funnel to the rack in your fume hood. Make sure the stopcock is closed, and carefully pour in about 1/2 of your beverage. To the beverage, add about 15 mL dichloromethane (CH$_2$Cl$_2$—may also be called methylene chloride). Put the stopper into the separatory funnel, making sure that it is snug but not too snug to where you can’t remove it. Carefully remove the separatory funnel from the rack and slowly tip it upside down. Open the stopcock to relieve any built up pressure and then close it. Gently swirl the contents of the separatory funnel, stopping to relieve pressure until no more pressure remains in the funnel. (You’ll probably need to do this at least 3 times; swirl, vent, close…swirl, vent, close…swirl, vent, close.) Don’t shake the funnel as you would a bottle of salad dressing; doing so will result in a mixed solution that will take a very long time to separate (see figure to the right).

Clamp the separatory funnel to the rack again and take note of what you see inside. The density of water is 1.0 g/mL and of CH$_2$Cl$_2$ is 1.3 g/mL. You want to keep the organic layer. (Which layer will it be?) You can drain the organic layer into a 250-mL beaker. Remember, you want to separate the layers, so you’ll need to judge when to close the stopcock. After removing the organic layer, extract the aqueous layer again with about 15 mL fresh CH$_2$Cl$_2$. Follow the same procedure you did before, gently swirling and venting your separatory funnel. Again, drain off the organic layer and add that to the 250-mL beaker you used the first time. Drain the aqueous layer into a 400-mL beaker and set aside.

Add the other half of your beverage to the separatory funnel, and to that add 15 mL fresh CH$_2$Cl$_2$. Run your extraction the same way you did the first time, slowly swirling the mixture. Again, you’ll need to extract twice, each time using 15 mL CH$_2$Cl$_2$. Add your organic layers to the 250-mL beaker you’ve been using, and the aqueous layer to the 400-mL beaker with the other aqueous layer.

Now would be a good time to take note of what you have. You should have a 400-mL beaker with ~100 mL of aqueous solution, and a 250-mL beaker that has ~60 mL of CH$_2$Cl$_2$ (4–15 mL extracts). The organic layer you have saved probably has a few bubbles in it, and depending on how accurate you were in separating the layers, a little bit of the aqueous solution. Carefully add your entire organic layer to the separatory funnel. Use a pipette to add a small amount of fresh CH$_2$Cl$_2$ to rinse out the
250-mL beaker. Add the rinse to the separatory funnel. To this, add ~20 mL of saturated aqueous NaCl solution. Again, you will need to follow the steps for extracting the organic layer (stopper, tip upside down, vent, swirl, vent…). Drain the organic layer into a clean, dry 250-mL beaker and drain the aqueous layer into your 400-mL beaker (the one with all the other aqueous layers in it).

OK…so now you have a 250-mL beaker that has roughly 60–65 mL CH₂Cl₂ in it, and a 400-mL beaker with roughly 140 mL of aqueous soda in it. You may have some water in your organic layer. This can be removed by adding ~1 – 1.5g of magnesium sulfate (MgSO₄). This compound is a drying agent that will absorb the remainder of the water out of your organic solution. Swirl your solution, making sure to minimize clumping of the drying agent. At this stage, you and your partner should check to make sure you both have the same compound. This can be achieved by using Thin Layer Chromatography (TLC). You and your partner can each put a spot on the same TLC plate. For this experiment you can use a mixture of ethyl acetate and hexanes as the developing solvent. After developing the plate, calculate the Rₚ value for any spots you see. If you and your partner have the same compound, what do you expect to see on the plate?

If you both agree you have the same compound, then you can combine your solutions into one of the 250-mL beakers. Try not to dump too much of the drying agent in with it, as you’ll want to get rid of that later. Rinse out the empty 250-mL beaker with a few milliliters of fresh CH₂Cl₂ and transfer the rinse into the combined extract. To remove the solvent, you will use an instrument called a rotary evaporator (picture below). Ask your instructor for help in setting this instrument up. To use it, you’ll need to carefully pour your solution into a clean 100-mL round-bottom flask (measure the mass of this flask before you pour anything into it). After the solvent is gone, what do you see in the flask? Measure the new mass of the flask with the compound in it and see how much you have. If you have enough, you should take a melting point of your sample to see how close it is to the melting point for caffeine.

Before the Lab

- Polarity and density—how does extraction take advantage of these properties?
- Polarity—How does TLC take advantage of this property?
- What else can TLC be used for?
- Make a prediction of which substance you think will contain the most caffeine.
After the Lab

• Compare the melting point of your caffeine sample to that found in the reagent table. What can you conclude based on this?

• Based on class data, which substance contained the most caffeine? Are the class results accurate? There are numerous sources online that list the amount of caffeine in beverages. Compare the class data with one of these tables.

• Using TLC, can you say anything about the polarity of caffeine?

• Why was it necessary to adjust the pH of the aqueous solution in the beginning? (Think acid–base chemistry.)
Lab 3 – Margarine in the Chemistry Lab

Introduction

Margarine is a generic term used to refer to any kind of butter substitute. In the 1800s, manufacturers produced margarine by taking clarified (purified) beef fat, extracting the liquid portion under pressure, and then allowing it to solidify. At the time, America’s dairy farmers produced vast quantities of butter and, feeling the impact of the growing margarine industry, lobbied the government to pass a host of laws regulating the production of margarine. These included taxes, licenses to make or produce it, and forbidding the addition of yellow food colorings. Despite an extensive battle with the dairy lobby, margarine still exists today, and is sold under a wide variety of brand names. According to the National Association of Margarine Manufacturers, at the end of the 20th century, the average American consumed just under 4.2 lbs of butter compared to nearly 8.3 lbs of margarine annually. Currently, margarine can be made in the laboratory from vegetable oils by a process called hydrogenation. You have probably seen partially hydrogenated vegetable oil listed as an ingredient in many of the foods that you consume. For today’s experiment, you will carry out the hydrogenation process, turning liquid vegetable oil into semi-solid margarine.

The Big Idea

Hydrogenation can be classified as either an oxidation-reduction reaction or an addition reaction. In a broad sense, this reaction is used to convert alkenes to alkanes by adding a hydrogen atom to each carbon of the double bond. Since carbon can only have four bonds, the double bond gets converted into a single bond. The carbons in a double bond are said to be unsaturated. When reacted with hydrogen (H₂), the carbons become saturated since four atoms are now attached—the most allowed for carbon. This is illustrated in the following general scheme. The Pd/C underneath the reaction arrow is the catalyst used to keep the reaction going.

Consider this when thinking about saturated vs. unsaturated fats. Saturated fats are long chain esters that contain no double bonds. Every carbon in the compound is connected by way of a single bond. Unsaturated fats exhibit a similar long chain, but with one or more double bonds on each of the carbon chains. Since saturated fats tend to have higher melting points than their unsaturated relatives, they are more commonly used as a spread.
Unsaturated fats are typically found in oils. That being said, much study has been dedicated to both types, since saturated fats lead to plaque buildup in the arteries, and unsaturated fats contain trans fats, which have been linked to coronary heart disease.

As an example, the scheme above illustrates what happens to glyceryl trioleate (the fatty acid in olive oil) when it reacts with 2 moles of hydrogen (4 H atoms). Notice how two of the chains are converted into single bond chains, leaving the third as an unsaturated chain. This is what is meant by a partially hydrogenated vegetable oil.

Since hydrogen gas is explosive, you will be generating hydrogen gas from cyclohexene rather than using H₂ gas directly. This reaction will form hydrogen as well as benzene as shown below.

Because you will be generating hydrogen gas indirectly it is pretty hard to measure just how much you are using. Given this, it is hard to determine how many double bonds will be present in your product. You could make a completely saturated compound, or you may even reduce just one of the double bonds.

In order for you to determine the extent of the hydrogenation, you can use the unsaturation test. This is explained on page 150.

**Safety**
- Safety glasses and gloves are required.
- Dichloromethane vaporizes easily. Handle this reagent in a fume hood.
- Cyclohexene really stinks; only use under a fume hood.
- Benzene is formed in small quantities. It is a carcinogen.
- There are two separate waste jugs; one for halogenated waste (dichloromethane, bromine) and one for non-halogenated waste (everything else).

### Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Olive oil/vegetable oil</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>10% Palladium on carbon</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Cyclohexene</td>
<td>82.15</td>
<td>83</td>
<td>0.811</td>
</tr>
<tr>
<td>Benzene</td>
<td>78.11</td>
<td>80</td>
<td>0.874</td>
</tr>
<tr>
<td>Petroleum ether</td>
<td>—</td>
<td>35–60</td>
<td>0.640</td>
</tr>
<tr>
<td>Hexanes</td>
<td>86.18</td>
<td>68–70</td>
<td>0.672</td>
</tr>
<tr>
<td>Dichloromethane</td>
<td>84.93</td>
<td>40</td>
<td>1.325</td>
</tr>
<tr>
<td>Bromine</td>
<td>79.91</td>
<td>59.5</td>
<td>3.119</td>
</tr>
</tbody>
</table>
Procedure

Starting with a 25-mL round-bottom flask containing a magnetic stir bar, add 2.0 g of one of the oils (olive oil if no others are present). Then add 3.0 mL of cyclohexene, followed by 60–70 mg of 10% palladium on charcoal. Attach a water condenser to the flask, making sure to attach the water hoses.

Incoming water goes in the bottom; outgoing water goes out the top (see diagram on next page). Once your apparatus is assembled, clamp your equipment in the hood, and place the flask on the heating mantle. Heat this to reflux and stir for 50 minutes. After the 50 minutes passes, shut down the heat, but keep the solution mixing. Allow it to cool to room temperature.

Reflux?

The diagram below shows an example of a reflux apparatus. What you are doing is heating your solution up to a boil. Rather than let the vapors escape into the atmosphere and risk losing all of the liquid in your solution, the condenser allows the vapors to drain back into the flask once they condense from the vapor phase. Once your solution begins to boil, you should see a vapor ring begin climbing up the condenser. Optimal heating will keep the vapor ring somewhere in the middle of the condenser: too much heating and the vapors escape; too little heating and the reaction will take longer. You may need to adjust your heating setting, depending on where you see the vapor ring.
After cooling to room temperature, add 2.5 mL of petroleum ether through the top of the condenser. Continue stirring until the ether drains down into the flask and mixes with the reaction mixture. Disconnect the condenser and, with a pipette, transfer the reaction mixture into a 15-mL centrifuge tube. Rinse the flask with an additional 2 mL of petroleum ether, swirl that around, and add the rinse to the centrifuge tube.

Place the centrifuge tube into the centrifuge (make sure that another tube is opposite to keep the centrifuge balanced). Centrifuge the mixture for a couple minutes. After removing the centrifuge tube, use a pipette to transfer the liquid portion to a pre-massed 50-mL round-bottom flask. Attach the flask to the rotary evaporator and remove the petroleum ether solvent. What is left in the flask?

**Unsaturation Test**

In order to quantify the amount of hydrogenation you did, you’ll need to do an unsaturation test using a bromine solution. When bromine is added to compounds containing double or triple bonds, a noticeable reaction takes place. Bromine (Br₂) is red-orange, but when it reacts the resulting solution turns colorless. It reacts in a similar fashion as the H₂ does. Knowing this, what do you suppose happens when Br₂ is added across a double bond?

First, you’ll need to test your starting material. In one of your large test tubes, add 10 drops of 1.0 M bromine in dichloromethane solution. Using a pipette, add dropwise a sample of liquid oil until the red color of the bromine disappears. Add this slowly, and take care to make sure the drops go directly to the bottom of the test tube. Be sure to count the number of drops you added. Repeat this process for your product using another test tube with 10 drops of 1.0 M bromine in dichloromethane solution. You may need to warm it a bit to liquefy it. Again, record how many drops you add to make the color disappear. Knowing this information will tell you if you completely saturated your compound, or if you prepared a partially hydrogenated vegetable oil. How does this work? What do you expect to see? Would you expect more drops of your product to turn the bromine colorless or more drops of your starting material? Is there a relationship between the amounts required for both? These are just some of the questions you will need to think about in order to determine what you have for a product.

When finished, be sure to discard the waste in the appropriate waste container. Save any remaining product that you had formed for a later experiment. You and your partner can combine products at this point, placing them in a labeled vial with the mass.

**Before the Lab**

- Why would an unsaturated fat be used primarily as an oil and not as a spread?
- What reaction type does hydrogenation fit into?
- What do the terms saturated and unsaturated mean when referring to carbon compounds?
- Given the general structure of an alkene, predict the product after a reaction with H₂ or Br₂.
- If other oils are present: do you think that different oil types (vegetable, canola, olive, peanut, etc.) would produce different results? Explain.
After the Lab

- What were the biggest sources of error in this experiment? Think about the bromine test. The oil contains double bonds, but so does something else that you used in the reaction. Can you identify what this reagent is?

- It was mentioned that saturated fats could lead to plaque buildup in the arteries. Using melting points, can you propose how this could happen?

- This experiment only touches on the issue of saturated vs. unsaturated fats. What type of fats are trans fats? What’s all the “media buzz” about? (Take advantage of the Internet.)
Lab 4 – Alkenes: Gathering a Wealth of Evidence to Support Claims

Introduction

Alkenes are a vast and important class of compounds in organic chemistry. You saw alkenes during the hydrogenation of vegetable oils and were introduced to them briefly with a couple of their reactions (hydrogenation, bromination). Alkenes serve as important intermediates in organic synthesis, leading to compounds that eventually become drugs used to alleviate ailments that many suffer from. As they are so numerous, there are many ways of preparing them. Outside of the pharmaceutical industry, the most common industrial way of preparing them is by refining petroleum. An alternative method you will explore will be dehydration. What comes to mind when you think of dehydration? Keeping in mind that carbon needs to have four bonds, you will be looking at what happens when one of the groups “falls off,” momentarily leaving the carbon with only three bonds.

The Big Idea

It’s one thing to be able to memorize a specific reaction by using flash cards; it’s quite another to be able to tell a pharmaceutical representative that the product you made is what you say it is. Sure, on paper the reaction makes sense, perhaps a mechanism can be drawn for it; but how can you prove without a shadow of a doubt that the substance in the vial is what you say it is? This experiment takes you back to the first one, where you are put in the shoes of a chemist trying to make a compound for a very important company. We’ve already seen what the implications could be if the product was contaminated or wasn’t what you claimed it would be.

\[
\begin{align*}
\text{OH} & \quad \text{H}_2\text{SO}_4 / \text{H}_3\text{PO}_4 \\
\text{CH}_3 & \quad \text{Product}
\end{align*}
\]

You will complete one reaction for this experiment. However, this one reaction can fall into many categories. Since the starting material is losing water, this is a dehydration reaction. It is also called an elimination reaction for the same reason (elimination = loss of). This one reaction will introduce you to three more techniques—boiling point analysis, infrared spectroscopy, and distillation.

If the boiling point for a compound is already known, then all that is required is a simple comparison between the experimental boiling point and the known boiling point. Infrared spectroscopy uses infrared light energy as a means of analyzing compounds. IR data allows us to tell what types of functional groups are present in a specific compound. Being able to interpret a spectrum will take some time, but for now you can rely on your instructor to help you, since this topic may not have been addressed fully in class.

This experiment will also introduce you to a technique called distillation. A diagram of a typical distillation apparatus is given in the procedure section. Distillation will allow you to separate compounds based on their boiling points. In the context of this experiment, it will also reinforce Le Châtelier’s Principle. Basically, what this
principle says is that if you remove a product from an equilibrium reaction, more product will be formed to reestablish the equilibrium.

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The last thing to focus in on will be with writing a mechanism. Perhaps this is something you may already be familiar with. A mechanism is simply a series of steps involving the path the electrons take to convert reactants to products.

This may not look like much in words, so below is an example of one using the same reaction conditions that you will be using.

This is a three-step reaction scheme as depicted in the mechanism. In the first step, a lone pair of electrons from oxygen attacks a proton from the acid (H₃O⁺). The result is the formation of a protonated alcohol and water. In step two, the protonated alcohol “falls off” as water, leaving behind a carbon with only three bonds, and as a result a positive formal charge.

![Chemical Reaction Scheme]

Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-methylcyclohexanol</td>
<td>114.19</td>
<td>173</td>
<td>0.91</td>
</tr>
<tr>
<td>Product</td>
<td>96.17</td>
<td>101</td>
<td>0.81</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>98.0</td>
<td>213</td>
<td>1.90</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td>98.08</td>
<td>337</td>
<td>1.84</td>
</tr>
<tr>
<td>10% Sodium bicarbonate</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Saturated sodium chloride</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>
Since the reaction conditions are acidic, the acid will be regenerated when the water molecule produced in step one attacks a proton on a carbon next to the positive charge. The electrons that formed the C–H bond fold down to make a double bond, which neutralizes the charge. This results in the formation of the alkene, as well as the regeneration of the acid catalyst.

**Safety**

- Sulfuric acid and phosphoric acid are to be handled with care, as they can cause burns.
- Do not boil the reaction mixture to dryness. Doing so could result in cracked or shattered glassware.

**Procedure**

Add 6 g of 4-methylcyclohexanol to a 25-mL round-bottom flask. Mass the flask before and after the addition to be sure of the exact mass of starting material you are using. To this, add 1.8 mL of 85% phosphoric acid, and 1 mL of concentrated sulfuric acid. Swirl this mixture to mix the reagents and then add a magnetic stir bar.

In the fume hood, assemble the distillation apparatus as shown below, using a 15-mL centrifuge tube as a receiving flask. Make sure that the thermometer tip is just below the side arm of the condenser. This will allow for an accurate reading of the boiling point.

When the distillation apparatus is assembled, turn the water on to the condenser. Begin heating the reaction mixture, keeping an eye out for the vapor ring that will begin to climb up the flask. You may need to adjust the heat, depending on where you see the vapor ring. You may notice the reaction mixture beginning to boil, but in order to judge the reaction, you’ll need to keep an eye on the temperature. Based on the boiling points of the materials in the reagent table, what do you suppose will be the first substance to boil off?
Collect everything that comes over up to a temperature of 105°C, being careful not to distill to dryness. Once no more material comes over, you can turn off the heat and allow the mixture to cool. Remove the centrifuge tube and cap it immediately. The leftover material in the reaction flask can be dumped in the waste container. This flask contains a mixture of very strong acid, so do take caution with it. Now you are ready to get the product.

You’ve already carried out the process of extraction using a separatory funnel during the caffeine extraction. Instead of using the separatory funnel, you can use your centrifuge tube to do the same thing. Add 1 mL of distilled water to the centrifuge tube. Close the cap on the tube and gently shake it. Now take a look at the tube and see if you can see two distinct layers. You’ll want to remove the aqueous layer with a pipette (see the reagent table).

To the remaining organic layer, add 2 mL of 10% aqueous sodium bicarbonate, close the cap, lightly shake, and then slowly open the cap to remove any built up pressure. Repeat this process until no more pressure builds up and then discard the aqueous layer. Aqueous sodium bicarbonate is a base. Given the reaction conditions, what do you suppose this step is necessary for?

To the remaining organic layer, add 2 mL aqueous sodium chloride solution and repeat the process as you did the prior two times. Once again remove the aqueous layer. To remove any traces of water, add ~1 g of magnesium sulfate (MgSO₄). Swirl this mixture and then allow the drying agent to settle at the bottom of the tube. Now you can use a pipette to transfer the organic solution into a clean pre-massed flask.

After all of the organic solution has been transferred, determine the mass of the product.

At this stage, you know what the boiling point of your product is, but you really need to analyze it to determine both what it is and how pure it is. To do this, you’ll need to use the infrared spectrophotometer. To prepare your sample, use a pipette to add a couple of drops of your product to one of the salt plates. Use another salt plate to “sandwich” the product in between the two plates. When you are ready to analyze your sample, your instructor will get the instrument ready to go and in a matter of minutes, you will have a printout of the infrared spectrum of your compound.

Analysis

As mentioned earlier, the IR will help you determine what functional groups are present in your compound. The correlation chart above shows the signals where a variety of functional groups show up. Go to the website under the spectral data link on WebCT and look for an IR reference spectrum for 4-methylcyclohexanol, as well as the compound that you made. You will need to know how to name your product. The key questions to think about are (1) what functional group is in the starting material that isn’t in the product, and (2) what functional group is in the product that isn’t in the starting material? Do the IR spectra of the two compounds look different? Use the correlation chart
on the following page to help explain these differences. Be sure to attach both spectra to your laboratory report.

**Infrared Correlation Chart for Functional Groups**

<table>
<thead>
<tr>
<th>Functional Group</th>
<th>Wave Number Range (cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N–H Primary Amines</td>
<td>3500–3200 cm⁻¹</td>
</tr>
<tr>
<td>N–H Secondary Amines</td>
<td>3500–3200 cm⁻¹</td>
</tr>
<tr>
<td>O–H Alcohols</td>
<td>3400–3200 cm⁻¹ (broad)</td>
</tr>
<tr>
<td>O–H Carboxylic Acids</td>
<td>3300–2500 cm⁻¹ (broad)</td>
</tr>
<tr>
<td>C–H Aromatics (stretch)</td>
<td>3150–3050 cm⁻¹</td>
</tr>
<tr>
<td>C–H Alkenes (stretch)</td>
<td>3100–3000 cm⁻¹</td>
</tr>
<tr>
<td>C–H Alkanes (stretch)</td>
<td>3000–2850 cm⁻¹</td>
</tr>
<tr>
<td>C–H Aldehydes</td>
<td>2850–2700 cm⁻¹</td>
</tr>
<tr>
<td>C=O Ketones</td>
<td>1805–1705 cm⁻¹</td>
</tr>
<tr>
<td>C=O Aldehydes</td>
<td>1740–1720 cm⁻¹</td>
</tr>
<tr>
<td>C=O Esters</td>
<td>1740–1735 cm⁻¹</td>
</tr>
<tr>
<td>C–H Alkenes (bend)</td>
<td>1700–1000 cm⁻¹</td>
</tr>
<tr>
<td>C=C Alkenes</td>
<td>1680–1600 cm⁻¹</td>
</tr>
<tr>
<td>C=C Aromatics</td>
<td>1600–1400 cm⁻¹</td>
</tr>
<tr>
<td>N–H Amides</td>
<td>1650–1550 cm⁻¹</td>
</tr>
<tr>
<td>C–H –CH₃ (bend)</td>
<td>1450, 1375 cm⁻¹</td>
</tr>
<tr>
<td>C–H –CH₂–</td>
<td>1465 cm⁻¹</td>
</tr>
<tr>
<td>C–O Alcohols</td>
<td>1300–1000 cm⁻¹</td>
</tr>
<tr>
<td>C–O Ethers</td>
<td>1300–1000 cm⁻¹</td>
</tr>
<tr>
<td>C–Cl</td>
<td>Chlorides 800–600 cm⁻¹</td>
</tr>
<tr>
<td>C–Br</td>
<td>Bromides &lt; 667 cm⁻¹</td>
</tr>
</tbody>
</table>

**Unsaturation Test**
You’ve already used the unsaturation test with bromine. Perform this test on a small amount of your product. If something happens, can you write the reaction scheme?

**Before the Lab**
- As mentioned, alkenes can be prepared a number of different ways. What type of reaction are you going to do?
- Take special note of where an alcohol (–OH) functional group shows up in an IR spectrum. Given spectral data, can you determine whether or not your compound is an alcohol?
- Distillation is a technique that can be used to separate compounds based on what property?
- Familiarize yourself with writing the mechanism for this reaction.

**After the Lab**
- As mentioned at the beginning of this experiment, this one reaction illustrates the importance of gathering evidence to support any claims you make regarding the identity of your product. Think about how all this evidence can be used when making claims in your laboratory report.
- Calculate the theoretical and the percent yield for your reaction. Using IR data as well as the percent yield, what can you say about the purity of your product?
- In the reaction below, does it matter which way the double bond goes? Will one product be formed over the other?
Lab 5—Electrophilic Aromatic Substitution: The Effect of Directing Groups on Benzene

Introduction

Electrophilic aromatic substitution is a class of reactions in organic chemistry that replaces a hydrogen atom in an aromatic system with another atom called an electrophile. To understand what this means, it is important to know the terminology used. First off, an electrophile (literally ‘electron lover’) is a species that is capable of accepting electrons. You are already aware of this in terms of acid-base chemistry with the definition of a Lewis acid. Most electrophiles carry a positive charge, while many others either contain a partial positive charge or lack an octet of electrons. Some examples of electrophiles are shown below.

\[
\begin{align*}
\text{O} & \quad \text{R} \quad \text{R} \\
\text{X} & \\
\end{align*}
\]

Electronegativity differences create a dipole.

\[
X = \text{halogen}
\]

Aromaticity, on the other hand, is much more difficult to explain in a laboratory setting. We can look at it briefly by considering the compound benzene, shown below. Benzene is a compound that contains three alternating double bonds. These double bonds can “move” by resonance to give benzene with double bonds in different positions. Since this phenomenon happens simultaneously, you may often see the structure of benzene with a circle drawn inside of it to represent this back and forth motion. Now, rather than focusing on aromaticity, you will be experimenting with compounds that contain benzene rings. When different groups are directly attached to the ring, they can alter the resonance structures that the benzene ring can form. The class syllabus dedicates quite a bit of time to the concept of aromaticity, but in the laboratory you should focus on resonance and use some of the techniques you’ve already learned to help you determine how different functional groups attached to the benzene ring can alter the position in which the electrophile gets attached.

The Big Idea

You will be experimenting with two different starting materials, subjecting them to the conditions for electrophilic aromatic substitution. Your starting materials will be acetanilide (from the first experiment) and methyl benzoate. The reactions that you will be performing will be nitration and bromination. More will be said about these reactions later on, but for now it is important to identify the structural differences between the two starting materials. Both have the benzene ring, so what’s the difference between the groups that are attached to the benzene ring?
Notice how acetanilide has a nitrogen atom attached directly to the benzene ring and bears a lone pair of electrons. The carbon attached directly to the ring in methyl benzoate lacks this lone pair, having instead a double bond to the oxygen it is attached to. Without including the ring, the resonance structures for each of the structures can be drawn. In acetanilide, the lone pair of electrons on nitrogen can fold down to form a nitrogen–carbon double bond, which forces a pair of electrons in the carbon–oxygen double bond out onto oxygen.

In methyl benzoate, the electrons move from the single-bonded oxygen onto carbon to produce a carbon–oxygen double bond. This forces a pair of electrons from the original carbon–oxygen double bond out onto oxygen. The charges shown in the two diagrams above come from the concept of formal charges. Notice that all that is happening is the movement of electrons. The arrows being used show the path these electrons take. Now, what happens if the ring is included?

It was mentioned earlier how the electrons in the ring can move around. In that example, no charges resulted. If no charges result, benzene itself is usually not that reactive. Again, we need to look at acetanilide as an example. What would happen if the lone pair of electrons was to fold down in the opposite direction, toward the ring? If no other electrons move, then carbon has five bonds, and you have been taught that cannot happen. So let’s move some electrons.

What has been drawn is another possible resonance structure for acetanilide. In this case, the lone pair of electrons on the nitrogen folded down onto the carbon in the benzene ring. This caused one pair of electrons from the carbon–carbon double bond to fold out onto the neighboring carbon. If you follow the rules for assigning formal charges, carbon has five electrons around it (one from each bond + two from the lone pair). Since carbon is only supposed to have four, the formal charge is $4 - 5$, which gives $-1$. It was mentioned that benzene can move electrons around the entire ring, so shown below is a step-by-step sequence of what happens if the process is carried throughout the ring.

In each step of the sequence, all that is being transferred are electrons. This process distributes electrons around the entire ring. The last step in the sequence transfers electrons back to the nitrogen. Just like the benzene ring example in the introduction, all that has happened is that the double bonds in the benzene ring are in different positions.
The point is that a negative charge was distributed around the ring. Since an electrophile is positively charged, there is now the possibility of a reaction taking place. An electrophile can be generically represented as $E^+$, and if we let “opposites attract” we can come to the following conclusion:

![Resonance structures for acetanilide](image)

Since there were three structures that were drawn with negative charges around the ring, there is the possibility of forming three products. Compounds 1 and 3 are called the *ortho* isomers, and compound 2 is called the *para* isomer. This makes compounds 1 and 3 the same compound, so in reality two possible products are formed, the *ortho* and *para* isomers. Because nitrogen donated electrons into the ring, the entire functional group it is part of is called an activating group. **Activating groups will give either ortho or para isomers as products.**

Now consider methyl benzoate. One resonance structure was already drawn, but now the ring needs to be taken into consideration. The carbon directly attached to the ring has no electrons to donate, so the only thing it can do is accept them. Just as was done for acetanilide, the first resonance structure for methyl benzoate is drawn below.

![Resonance structure for methyl benzoate](image)

A pair of electrons folds out from the ring to form a new carbon–carbon double bond, which causes a pair of electrons in the carbon–oxygen double bond to fold out onto oxygen. Again, if you use the rules for formal charges, you can see that now a positive charge has been generated on carbon, and a negative charge has been generated on oxygen. Once again, the ring is capable of distributing charges, so what follows below is a step-by-step resonance sequence for methyl benzoate.

In this sequence, a positive charge is distributed around the ring by the transfer of a pair of electrons from one atom to the next. The final step in the sequence forms the original carbon-oxygen double bond that you started with and neutralizes the charges in the process. Again, the only difference between the first structure and the last one is where the double bonds in the ring show up.
The point in this second reaction sequence is that a positive charge was distributed around the ring instead of a negative charge as seen with acetanilide. Consequently, there is a difference in the way methyl benzoate reacts. Since the electrophile is positively charged, it will not be attracted to another positive site. The only other options for an incoming electrophile are the two places where no charge exists. This is shown in the following scheme:

\[
\begin{align*}
&\text{O} & & \text{E}^+ & & \text{O} \\
&\text{E} & & \text{O} & & \text{O} \\
&\text{or} & & \text{E} & & \text{O} \\
&\text{O} & & \text{E} & & \text{O}
\end{align*}
\]

The two products formed in this case are both called meta isomers. This makes them identical compounds, so in this sequence only one possible product is formed. Because the functional group attached to the ring accepted electrons from the ring, it is called a deactivating group. **Therefore, deactivating groups will give meta isomers as products.**

The last thing that needs to be addressed is the identity of the electrophiles you will be using in your reactions. For the nitration, you will be mixing nitric acid with sulfuric acid to generate the nitronium ion (NO₂⁺), as shown below.

\[
\begin{align*}
\text{H}^+ & \quad \text{NO}_2^- & \quad \text{H}_2\text{SO}_4 & \rightarrow & \text{O} = \text{N} = \text{O}^- + \text{H}_2\text{O} + \text{HSO}_4^- \\
\text{Nitronium ion}
\end{align*}
\]

For the bromination, you will use a compound that generates Br₂, which in turn can be turned into Br⁺ and Br⁻. In this case the electrophile will be Br⁺.

**Safety**

**General**

- Safety glasses are a requirement for this experiment.
- Lab aprons are optional but are encouraged.
- All reactions are to be performed in the fume hood.

**Nitration**

- Concentrated nitric acid and concentrated sulfuric acid are both very strong acids and can cause burns. Be cautious when handling both of these chemicals.
- Protective gloves are required for this experiment. If any chemicals are spilled, immediately remove your gloves and wash your hands.
- There are two waste containers; one for strong acids and one for methanol. Do not put any methanol into the strong acid waste as this may result in explosive materials.

**Bromination**

- Glacial acetic acid is a weak acid, but still is corrosive. Remove gloves immediately and wash your hands in the event of any spills.
- Pyridinium tribromide is corrosive and is an irritant. Caution should be taken when working with this substance.
Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetanilide</td>
<td>135.17</td>
<td>113–115</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Acetic acid</td>
<td>60.05</td>
<td>16</td>
<td>117</td>
<td>1.05</td>
</tr>
<tr>
<td>Bromine (Atomic)</td>
<td>79.91</td>
<td>−7</td>
<td>59</td>
<td>3.12</td>
</tr>
<tr>
<td>Ethanol</td>
<td>46.07</td>
<td>—</td>
<td>78</td>
<td>0.79</td>
</tr>
<tr>
<td>Pyridinium tribromide</td>
<td>319.84</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Methyl benzoate</td>
<td>136.15</td>
<td>−12</td>
<td>198–199</td>
<td>1.09</td>
</tr>
<tr>
<td>Nitric acid (15.4 Molar)</td>
<td>63.01</td>
<td>—</td>
<td>—</td>
<td>1.40</td>
</tr>
<tr>
<td>Sulfuric acid (18 Molar)</td>
<td>98.08</td>
<td>—</td>
<td>—</td>
<td>1.84</td>
</tr>
<tr>
<td>Methanol</td>
<td>32.04</td>
<td>−98</td>
<td>65</td>
<td>0.79</td>
</tr>
</tbody>
</table>

Procedure

For the most efficient use of time it is advised to run the bromination reaction with acetanilide and the nitration reaction with methyl benzoate. Although it is possible to do both sets of reaction conditions on both starting materials, these reactions tend to take a little bit longer to do. You can work in partners, with each person in the pair using a different starting material. The procedures for both sets of reaction conditions are given below.

Bromination of Acetanilide

In a 25-mL Erlenmeyer flask, dissolve 1.07 g of acetanilide in 4 mL glacial acetic acid. Add a stir bar followed by 1.6 g pyridinium tribromide. Heat the mixture to ~60°C in a warm water bath for 10 minutes.

After the 10 minutes, add 15 mL water and 2 mL saturated sodium bisulfite solution. (This reagent will remove the orange color of the bromine.) If the orange color remains, add an additional 2 mL of the sodium bisulfite solution.

Cool the reaction mixture in an ice bath for 5 minutes and then collect the crystals by vacuum filtration using the Hirsch funnel. Wash the crystals with a small amount of water (~5 mL), and leave on the vacuum to dry (~5 min).

Nitration of Methyl Benzoate

Add 0.5 mL concentrated sulfuric acid to 0.5 mL concentrated nitric acid in a small conical vial. Cap the vial and chill it in an ice water bath. While the nitrating mixture cools, add 0.42 mL methyl benzoate to a 10-mL round-bottom flask. Make sure to record the exact mass of methyl benzoate used, as you will need that value for theoretical yield determination. Place the flask with the methyl benzoate in the ice water bath and allow it to cool to ~0°C. Add a small stir bar to this flask and slowly add 0.9 mL concentrated sulfuric acid. Be sure not to allow the temperature of this mixture to exceed 5°C.
With the two starting materials ready to go, use a pipette to slowly add about half of the nitrating mixture to the flask containing the methyl benzoate. Try to keep the temperature as close to 0°C as possible. After a couple minutes, slowly add the remainder of the nitrating mixture. After complete addition of the nitrating mixture, allow the reaction to stir in the ice bath for 10–15 minutes. After this time, remove the reaction flask from the ice water bath and allow it to warm to room temperature.

Transfer the reaction mixture into a 25-mL Erlenmeyer flask that contains a small handful of crushed ice. Swirl the mixture occasionally and allow it to warm to room temperature. Collect the solid product by using vacuum filtration with the Hirsch funnel. Wash the solid product with two 1-mL portions of cold water.

Up to this point, all of the waste generated in this experiment should be placed in the waste bottle reserved for strong acid waste (sulfuric acid and nitric acid). Do NOT add methanol to the waste bottle reserved for strong acid waste.

Reattach the Hirsch funnel and wash the solid product with two 0.5 mL portions of ice-cold methanol. Allow the solid to dry on the vacuum for about 5 minutes.

**Analysis**

**Bromination**

From the information above, you know it is possible to form two different products. Fortunately, these products have much different melting points. For the sake of argument, a third possibility also exists. These products are shown below. Based on your experimental melting point, can you determine which product you obtained? To help determine the purity of your product, you should take an IR spectrum. The functional groups of the three products are all the same, so the IR won’t tell you which product you have, but it may help you to justify your melting point.

**Nitration**

Based on the information given above, you know you should only obtain one product, the meta isomer. But how can you be so sure? The three possible products that you could obtain all have different melting points. These products are shown below. Based on the melting point you obtained, can you determine which product you made? You should take an IR spectrum. The functional groups are all the same, but the spectrum will help you to analyze the purity of your product.
**Before the Lab**

- This is a lot of information. For now focus on being able to draw resonance structures.
- Where did the charges come from? Become fluent with determining formal charges.
- Know what an electrophile is, and how it reacts.
- Activating groups vs. deactivating groups…what’s the difference?

**After the Lab**

- Draw the mechanism showing how your product(s) was/were formed.
- Given other compounds with different groups attached to a benzene ring, can you predict what the product will be?
- Aromatic compounds…you may not have dealt with these in class, but you will shortly. What makes a compound aromatic?
Lab 6—“Clean” Chemistry: From Margarine to Soap?

Introduction
Soap has been around for quite some time. According to www.cleaning101.com, the earliest known evidence of soap use dates back to the Babylonians from 2800 BC. A formula for soap was written on a Babylonian clay tablet, although its purpose was not recorded. The Egyptians also recorded formulas for making soap around 1550 BC. Around the same time, Moses gave the Israelites detailed laws governing personal cleanliness. Biblical accounts suggest that the Israelites knew that mixing ashes and oil produced a kind of hair gel. Long after, around the 7th century AD, soap making became an established craft in Europe. Vegetable and animal oils were used with ashes of plants, along with fragrances and dyes. Since that time, soap has become one of the most common household “staples” for cleaning things.

Historically, mixing animal fats with lye (NaOH) or potash (KOH) was the most common method for making soap. According to a Roman legend, soap originally received its name from Mount Sapo, which was recorded to be a place where animal sacrifices were held. Rain washed a mixture of melted animal fat and wood ashes down into the clay soil, producing small foamy ponds. Although no one has ever been able to prove this story, and Mount Sapo has never been found, it goes to show the importance soap has had on human civilization since its existence. As mentioned above, vegetable oils came into the picture as time passed. By the middle of the 20th century, detergents started being produced for their resistance to hard water. Much of the “soap” made currently is actually a detergent. For this experiment, you will make soap by one of the three traditional methods and test it by using a series of chemical tests.

The Big Idea
To understand how soap actually works you need to be familiar with the concept of polarity. You have already seen that “like dissolves like,” and you have done some experiments where you tested polarity by using TLC plates. When you made margarine, you started with olive oil and hydrogenated it, as shown below.

If you react the vegetable oil with sodium hydroxide (lye) instead of hydrogen, you get the basic reaction for making soap. For convenience, the long alkyl chains are represented generically by the letter R.
The reaction is called a *saponification* reaction. The hydroxide ion from sodium hydroxide cleaves (breaks) the carbon–oxygen single bonds, resulting in glycerin and three equivalents of the sodium salt of soap. Since the soap has a charged end, that portion of it is polar. This charged end is described as being hydrophilic (water loving). The long chain represented by the letter R is nonpolar and is described as being hydrophobic (water fearing).

Both of these properties can explain how soap works. Grease, grime, and “muck” are essentially nonpolar compounds. The long alkyl chains surround the stain, forming a circular pattern called a micelle. The charged end is oriented to the outside of the circle as shown below.

When water is introduced, the charged ends of the soap are attracted to the water by hydrogen bonds. With the stain trapped in the center of the micelle, the whole complex is washed away.

It was mentioned above that historically the main method for soap making was to use animal fats, which was subsequently replaced by using vegetable oils. It would stand to reason that since vegetable oils came into the picture after animal fats that perhaps soap from vegetable oils has some better quality to it. But is there? This question could be the basis of what you will be trying to do. You will use lard, which is made from animals, in addition to vegetable oil and shortening. Is one method superior over the others, or does it really matter what you start with for making soap? As a class, you will be able to find some answers by making some soap of your own.

**Procedure**

**Lard and Sodium Hydroxide**

Cut a ½-in² piece of lard and place in a pre-weighed 250-mL beaker containing a stir bar. Record the mass of the lard. In a separate beaker, dissolve 2.5 g NaOH in 20 mL 50% ethanol-water solution and add to the beaker containing the lard. Stirring constantly, heat the mixture in a hot water bath for at least 20 minutes. For colored soap, add a few crayon shavings to the soap mixture and mix well to distribute the color. If foaming becomes excessive, add 1–2 mL more of the 50% ethanol-water solution and keep stirring. After heating is complete, remove the beaker from the hot water bath and allow it to cool to room temperature for 5 minutes. Perfume or other fragrance may be added to the soap at this time. Now add 75 mL...
saturated NaCl solution to the soap and stir vigorously to break it up. Collect the soap by vacuum filtration using the porcelain Büchner funnel and filter paper. Wash the soap with two 10-mL portions of ice-cold water and let air-dry. Transfer the soap to a pre-massed weigh boat. Record the mass of the soap.

**Reagent Data**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
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</thead>
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<tr>
<td>Ethanol</td>
<td>46.07</td>
<td>—</td>
<td>78</td>
<td>0.79</td>
</tr>
<tr>
<td>1% Calcium chloride</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>1% Iron (III) chloride</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>1% Magnesium chloride</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Saturated sodium chloride</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Sodium hydroxide</td>
<td>40.00</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Water</td>
<td>18.01</td>
<td>0</td>
<td>100</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Vegetable Oil and Sodium Hydroxide**

Measure 5g of olive oil, cottonseed oil, or other vegetable oil into a pre-weighed 250-mL beaker containing a stir bar. Record the exact mass of the oil. In a separate beaker, dissolve 2.5 g NaOH in 20 mL 50% ethanol-water solution and add to the beaker containing the oil. Stirring constantly, heat the mixture in a hot water bath for at least 20 minutes. For colored soap, add a few crayon shavings to the soap mixture and mix well to distribute the color. If foaming becomes excessive, add 1–2 mL more of the 50% ethanol-water solution and keep stirring. After heating is complete, remove the beaker from the hot water bath and allow it to cool to room temperature for 5 minutes. Perfume or other fragrance may be added to the soap at this time. Now add 75 mL saturated NaCl solution to the soap and stir vigorously to break it up. Collect the soap by vacuum filtration using the porcelain Büchner funnel and filter paper. Wash the soap with two 10-mL portions of ice-cold water and let air-dry. Transfer the soap to a pre-massed weigh boat. Record the mass of the soap.

**Vegetable shortening and sodium hydroxide**

Measure 5g solid vegetable shortening in a 250-mL beaker containing a stir bar. Add 10 mL ethanol and 20 mL 20% NaOH. Heat the reaction mixture while stirring for 20 minutes at 95–100 °C. Maintain the total volume of the reaction mixture by adding small quantities of 50% ethanol. After the 20 minutes of heating you can test your solution to see if the reaction is complete. To do this, add a few drops of the reaction mixture to a large test tube. Add 10 mL cold water and see if any fat droplets form. If they do, add 5 mL 20% NaOH and 5 mL ethanol to the beaker and continue heating for an additional 10 minutes, or until no fat droplets form upon
testing. After the completion of the reaction, add 25 mL water and allow the mixture to cool to room temperature (~5 min.). Add 55 mL saturated NaCl solution to the beaker. Collect the soap by vacuum filtration using the porcelain Büchner funnel and filter paper. Wash the soap with two-20 mL portions ice-cold water. Once dried, transfer the soap to a pre-massed weigh boat and determine the mass.

**Analysis**

There will be two ways to analyze your soap. First, determine the pH of your sample and compare it to a known soap or detergent. Are there any differences? Second, you will determine what effect hard water has on your soap. To do this, make a soap solution using a small piece of your soap and 10 –15 mL water in three separate test tubes. Add 5 drops of 1% calcium chloride solution to the first test tube, 5 drops of 1% magnesium chloride solution to the second test tube, and 5 drops of 1% iron (III) chloride solution to the third test tube. Note whether a precipitate forms in any of the test tubes. Repeat these tests on a known synthetic detergent, and compare the results with what you observed for your soap. Are there any differences?

**Before the Lab**

- What is the reaction classification for making soap; i.e. what is this reaction called?
- Given a long chain fatty acid and a base, could you draw the structure of the products formed?
- Hydrophobic vs. hydrophilic…what’s the difference?
- How can one compound exhibit two opposite polarities?

**After the Lab**

- Not much was said about hard water. What is hard water?
- Is there a difference between a detergent and a soap?
- Which method proved to be the best method for preparing soap?
- Soap was originally made from a substance called lye. Unfortunately, there were no storerooms or chemical warehouses where lye could be purchased. How was this substance made?

**For Further Reading**

Lab 7—Molecular Models: The World of 3-Dimensional Chemistry

Introduction
Have you ever seen those 3-D pictures that promise if you stare at them long enough an image will emerge? On the surface, it may look like a bunch of colors, but embedded within those colors is a 3-D image that you can actually focus in on. Many of these pictures can be found for free online (www.stereoscopy.com or www.vision3d.com). If you cross your eyes a bit, an image with 3-D characteristics seems to magically form in front of your eyes. Some people have no difficulty doing this and can readily see the image with minimal effort. Others, however, are not as fortunate. In organic chemistry, there are molecules that are also 3-dimensional, yet most of the time molecules are just drawn on paper in a 2-D setting. Just as with those 3-D pictures online, some people will have a difficult time seeing the 3-D image of an organic molecule. This is why having a model kit can be important. The structure to the right is for the compound 1-bromo-1-chloroethane. In 2-dimensions, only one structure is drawn. If drawn in 3-dimensions there are two possible structures, both of which can sometimes exhibit different properties. Drawing structures in this manner will lead you into a topic of organic chemistry called stereochemistry. This topic involves the study of the relative spatial arrangements of atoms within molecules. This becomes crucial for scientists who study amino acids, for example, where one form of the amino acid is found almost exclusively over the other one.

One of the most infamous demonstrations of the significance of stereochemistry is the thalidomide disaster. Thalidomide was prepared in 1957 in Germany, and was used to treat morning sickness in pregnant women. Unfortunately, the drug was found to cause serious birth defects in children. After initial testing of the drug, it was deemed to be safe with no known side effects. However, the other form was found to be the culprit. These two structures are shown on the next page. How are these two structures different?

This experiment will help you distinguish between these forms. You will be armed with a model kit so you can build some of these structures and see if any differences exist. Based on the differences you encounter, the structures you build will fall into a different classification. There is quite a bit of terminology that you will need to be introduced to, some of which you may already be familiar with.

The Big Idea
Isomers
Consider the molecular formula C₄H₁₀. Based on this formula alone there are two possible structures you could draw, shown below. As an example of their differences, the boiling point of butane is −0.5°C, and for 2-methylpropane, the boiling point is
These two compounds have the same molecular formula, but different structures. Compounds with the same molecular formula but different structures are called isomers.

![ Constitutional Isomers ]

The three compounds shown above also have the same molecular formula C₄H₁₀O, but notice where the functional groups are. These compounds represent constitutional isomers because the bonding sequence in each case is different. In 1-butanol, one carbon is attached to oxygen in addition to another carbon. In 2-butanol, one carbon is attached to oxygen, but this carbon is also attached to two other carbons. In diethyl ether, oxygen is now bonded to two carbons.

**Positional Isomers**

Positional isomers are a subset of constitutional isomers. Positional isomers contain the same molecular formulas as well as the same functional groups. The functional groups are just attached at different positions. This is illustrated below by using the three isomers of pentanol.

![ Positional Isomers ]

**Functional Group Isomers**

These isomers are yet another subset of constitutional isomers. These isomers have the same molecular formulas, but different functional groups. The three compounds shown below all share the molecular formula C₄H₈O, yet the functional groups represent a ketone, an ether, and an alcohol, respectively.

![ Functional Group Isomers ]

**Stereoisomers**

The two types of constitutional isomers (positional, functional group) result because in each case the bonding sequence was different. When the bonding sequence is the same, you now have a class of isomers called stereoisomers. There are two main classes of stereoisomers.

**Enantiomers**

3-dimensionality becomes critical when beginning a discussion about enantiomers. First, the definition of enantiomers is that they are mirror images that cannot be superimposed. To illustrate this, consider the first compound we looked at, 1-bromo-1-chloroethane, shown below.

![ Enantiomers ]

First of all, notice how one of the carbons is bonded to four different functional groups—hydrogen, bromine, chlorine, and methyl. This carbon is said to be chiral. When drawn in 3-dimensions, you see two possible structures—the original image and its reflected image. You will need to visualize this for now, but you will find that if you try to spin the groups around,
there will be no possible way for you to line up all of the groups. This is what being a non-superimposable mirror image means.

**R, S Nomenclature**

In order to name both enantiomers for a given isomer, the groups bonded to the chiral carbon need to be arranged by a priority. This is done based on the atomic number of each of the atoms directly bonded to the chiral carbon atom. The atom with the highest atomic number is given the number 1. The other three atoms attached are then numbered based on their atomic number. This can be best explained by considering the example of 1-bromo-1-chloroethane.

The atom with the highest atomic number in this example is bromine, so put a number 1 beside it. Next is chlorine, followed by carbon (from the methyl group), and then hydrogen. Using these number designations, you should have something that looks like the following structures.

![Mirror](image)

Now comes the ‘hard’ part. The atom with the lowest priority (hydrogen, labeled as 4) needs to be facing away from you. In order to do this, you’ll need to rotate the compound as a whole, while keeping track of the other groups attached. If hydrogen is facing away from you, the rest of the compound will look like this:

![Mirror](image)

The hydrogen isn’t shown, but just imagine that you are looking at this compound from the bottom of it. If you count the numbers from 1–3, you can draw an arrow showing what direction you are counting in.

![Mirror](image)

With the structure on the left, you will be counting in a clockwise fashion. This isomer is known as the R isomer (R for Right). With the structure on the right you are counting in a counter-clockwise fashion. This isomer is called the S isomer (S for Sinister or Left). To name both of these isomers, you just put the letter designation at the beginning of the name.

**Diastereomers**

Isomers that are not classified as enantiomers fall into the category of diastereomers. You will look at two types—alkenes and rings.

2-butene can exist as either *cis* or *trans* isomers. These two isomers are not mirror images so they are not enantiomers. In *cis*-2-butene, the two methyl groups are on the same side of the double bond. In *trans*-2-
butene, the two methyl groups are on opposite sides of the double bond. When dealing with more complex alkenes it is necessary to use the $E$ and $Z$ naming system. This is needed when the groups on the double bond are not the same.

\[
\begin{align*}
&\text{H}_3\text{C} - \text{CH}_2\text{CH}_3 & &\text{H}_2\text{CH}_2\text{CH}_3\text{C} - \text{Br} \\
&\text{H} & &\text{CH}_3
\end{align*}
\]

(Z)-2-pentene (E)-3-bromo-2-chloro-2-hexene

In this system, the groups are assigned priorities in the same manner as is done for chiral carbon atoms. If the two highest priority groups are on different carbon atoms and on the same side of the double bond, then the configuration is said to be $Z$- ($zusammen$ = together). If the groups are on different carbon atoms and on opposite sides of the double bond, the configuration is said to be $E$- ($entgegen$ = opposite).

When considering how to position two groups at different points along a ring, we discover there are two choices: the groups can lie on (1) the same side or (2) opposite sides of a ring.

\[
\begin{align*}
&\text{cis-1,2-dimethylcyclohexane} & &\text{trans-1,2-dimethylcyclohexane}
\end{align*}
\]

The structures above have the same bonding sequence, but they are not mirror images of each other, nor are they identical. Therefore, they are diastereomers. The compounds are named collectively as 1,2-dimethylcyclohexane but can be distinguished by the $cis$ or $trans$ prefix.

**Meso Compounds**

\[
\begin{align*}
&\text{CH}_3 & &\text{CH}_3 \\
&\text{H} - \text{C} - \text{OH} & &\text{HO} - \text{C} - \text{H} \\
&\text{CH}_3 & &\text{CH}_3
\end{align*}
\]

These structures are mirror images of each other, but they are not enantiomers. These structures are, in fact, superimposable; therefore, they represent the same compound. This compound is an example of a *meso* compound. A meso compound is one whose molecules are superimposable on their mirror images even though they contain chiral centers.

As was mentioned earlier, this is a lot of terminology, and it may be difficult to keep all of this organized. For this reason, a flow chart is included on the following page that will help guide you in determining what the relationships are between the compounds on the following pages.
Do the two compounds have the same molecular formula?
  No  Yes

Are the two molecules identical?
  No  Yes

**ISOMERS**

Is the bonding sequence the same?
  No  Yes

**CONSTITUTIONAL ISOMERS**

Are all the functional groups identical?
  No  Yes

**STEREOISOMERS**

Are all the molecules mirror images?
  No  Yes

**DIASTEREOMERS**

Are the molecules different due to restricted rotation?
  No  Yes

Are the different groups on a C=C?
  No  Yes

(A) Not isomers  (B) Functional group isomers  (C) Positional isomers  (D) All other diastereomers  (E) cis-trans rings  (F) cis-trans Alkenes  (G) Enantiomers  (H) Same compound

Recheck the 2nd question
Isomer Exercises, Part I.

As a class, break up into groups and complete the following 28 problems. Examine each pair of structures and state the relationship between the compounds they represent. Tabulate the answers on the board and make sure everybody agrees on the answers. If someone disagrees with your answer, be prepared to provide reasoning for the answer you have chosen. If you cannot decide from the drawings, use the isomer scheme on the preceding page and/or build models to assist you in deciding the correct answer. Classify each of the following pairs as being either:

A. Not isomers
B. Functional group isomers
C. Positional isomers
D. All other diastereomers (not E or F)
E. Diastereomers (cis–trans rings)
F. Diastereomers (cis–trans alkenes)
G. Enantiomers (nonsuperimposable mirror images)
H. Identical compounds

____ 1. \( \text{CH}_3\text{CHCH}_2\text{CH}_3 \) \( \text{OH} \) \( \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \)

____ 2. \( \text{CH}_3\text{OCH}_2\text{CH}_2\text{CH}_3 \) \( \text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2 \)

____ 3. \( \text{CH}_3\text{C}–\text{OCH}_3 \) \( \text{CH}_3\text{C}–\text{CH}_3 \)

____ 4. \( \text{CH}_3\text{CH}_2\text{C}–\text{CH}_2\text{CH}_3 \) \( \text{CH}_3\text{CH}_2\text{CH}_2\text{C}–\text{CH}_3 \)

____ 5. \( \text{CH}–\text{CH}_2–\text{CH}_3 \) \( \text{CH}–\text{CH}_3 \)

____ 6. \( \text{CH}–\text{CH}_2–\text{CH}_3 \) \( \text{CH}–\text{CH}_3 \)
7. \[
\text{CH}_2=\text{CH}_2
\]

8. \[
\text{CH}_2=\text{CH}-\text{O-CH}_3
\]

9. \[
\text{CH}_2=\text{O-CH}_3
\]

10. \[
\text{O-CH}_2\text{CH}_2\text{O}
\]

11. \[
\text{C}_6\text{H}_10
\]

12. \[
\text{CH}_3\text{Br} \quad \text{CH}_2\text{Br} \quad \text{CH}_3\text{Cl} \quad \text{CH}_2\text{Cl}
\]

13. \[
\text{CH}_3\text{Br} \quad \text{CH}_3\text{Br} \quad \text{CH}_3\text{Cl} \quad \text{CH}_3\text{Cl}
\]

14. \[
\text{HO-CH}_3 \quad \text{HO-CH}_3
\]

15. \[
\text{CH}_3\text{Br} \quad \text{CH}_3\text{Br}
\]
Isomer Problems, Part II.

These questions will each be worth one point extra credit.

29. Use the wedge notation to show the structures of the two enantiomeric forms of 2-bromobutane, CH₃CHBrCH₂CH₃.
30. The structure of carvone is shown below. One enantiomer of carvone is found in caraway seed oil, dill seed oil, and mandarin peel oil. The other enantiomer of carvone is found in spearmint oil. Use wedge notations to show both enantiomers of carvone.

\[
\text{Carvone}
\]

31. The two structures shown below have the same molecular formula. What is the relationship between these compounds?

32. The two structures shown below have the same molecular formula. What is the relationship between these compounds?
Enantiopure Medicinal Agents: The Same Structure but Two Different Drugs? Part III.

After being introduced to the concept of enantiomers and all this 3-D imaging, the question comes about of just how important this really is. This section of the lab will constitute the bulk of your laboratory report. Many compounds in the medical field are composed of different isomers. The interesting part is that one isomer can have remarkably different effects on the body than another. As a result, the same compound can be marketed as two different drugs. Shown below are a few compounds that contain chiral centers. Your job will be to (1) identify the chiral carbon(s) and (2) draw the R and the S isomers.

Ketamine, shown above, is used as a horse tranquilizer. In the 1960s, it was used on humans as an anesthetic. It was quickly found that this drug produced many side effects and as a consequence was labeled as a class 2 chemical. Although it is now illegal, its use is still relatively popular.

Nicotine is a very popular alkaloid that is found naturally in the nightshade family of plants. These include tobacco, tomato, potato, eggplant, and green pepper. Nicotine is named after the tobacco plant *Nicotiana tabacum*, which in turn is named after Jean Nicot, who sent tobacco seeds from Portugal to Paris in 1550.

Norepinephrine is a chemical released from the medulla of the adrenal glands as a hormone into the blood, but is also a neurotransmitter in the nervous system. It is related in part to a similar compound called epinephrine, which is commonly called adrenaline. The naturally occurring isomer is the R isomer.

The last three compounds you will be responsible for are shown below.
Ephedrine has four different enantiomers, but each one of them behaves differently. Amphetamine is a parent to a numerous class of compounds, many of which differ only slightly from the parent. Ibuprofen is also similar to many of the compounds in its family. What structural differences are there in these classes of compounds?

**Before the Lab**

- This is a broad topic, and it is possible that you will not have a chance to discuss it in class prior to coming to the laboratory. If this is the case, the pre-lab quiz will be given at the end of the laboratory.
- Practice the R and S naming system. If necessary look this up in your textbook to find some examples of other problems you can work on.

**After the Lab**

For your lab report, here is how the point system will work out:

- The first 28 problems are practice. These should help you get the hang of seeing these structures in three dimensions.
- Problems 29 – 32 are extra credit, and will be awarded 1 point each for a total of 4 points.
- Part III is worth 25 points:
  - 1 point for correctly identifying the chiral center(s) in each of the six compounds
  - 1 point for drawing the correct R and S configuration using hashes and wedges.
  - 5 points for a reflection concerning one of these six drugs. Do a search on the Internet to see if you can find any information about the two different forms of the drug you chose. Write a paragraph or two summarizing what you found. If you cannot find any information on the drug you chose, then you will need to choose another one.
Lab 8—Substitution vs. Elimination: The Chemical Competition

Introduction

In a substitution reaction, a functional group in a particular compound is replaced by another functional group. This was illustrated earlier during the electrophilic aromatic substitution reactions. In those reactions, one hydrogen atom on the benzene ring was replaced by a nitro or bromo group.

The products in those reactions were determined by going through the mechanism, understanding a bit about activating/deactivating groups, and using resonance to show where the electrons go and how they attack a positive electrophile. Essentially, understanding the mechanism allowed you to predict the product.

Today’s experiment is going to focus on a reaction that can happen by three different possible pathways. The problem you will be trying to solve will be determining which of the three pathways the reaction proceeded by. Earlier in the course you treated an alcohol with an acid to dehydrate it and form an alkene.

It was mentioned that this dehydration reaction could also be classified as an elimination reaction since water was being eliminated. You were able to use the IR Spectrometer to prove that your product was an alkene. But why didn’t substitution happen? What was the driving force for the alkene to form instead of the substituted product?

The Big Idea

As mentioned above, there are three possible pathways to consider. The first is elimination and is one pathway that we’ve already studied. The other two fall under the umbrella of substitution. To understand how elimination and substitution reactions differ, consider the following reaction:
With the formation of two possible products, this reaction wouldn’t appear to be very useful if you only wanted one product. In the dehydration experiment, you used a small amount of acid so that it acted as a catalyst.

In the mechanism, a water molecule attacks a proton on a neighboring carbon to regenerate the acid catalyst and keep the reaction going:

\[
\begin{align*}
\text{CH}_3 & \quad \text{H} \\
\text{Carbocation} & \quad \text{Intermediate} \\
\text{O} & \quad \text{H} \\
\text{H} & \quad \text{H} \\
\text{CH}_3 & \quad \text{H} \quad \text{H} \\
\end{align*}
\]

In order for substitution to take place, the chloride ion (Cl⁻) would need to “win the race” and beat the water molecule to the starting material. If it does, then substitution can occur. What are some factors that could affect this? Is there any way to control this?

In the reaction outlined above you can see that a 3° carbocation is formed. You know that 3° carbocations are relatively stable. If a 3° carbocation can form, then generally it is favored to do so. This means that you would see a carbocation in the mechanism.

So what happens if the starting material isn’t able to form a 3° carbocation? Consider the following reaction:

\[
\begin{align*}
\text{OH} & \quad \text{HCl} \\
\rightarrow & \quad \text{or} \\
\text{Cl} & \quad \text{H}_2\text{O}
\end{align*}
\]

Once again the possibility of forming two products exists—one for elimination and the other for substitution. The big difference is that the starting material cannot form a carbocation. If it did it would be 1° and in terms of stability, that’s not a good thing. In terms of a mechanism, we can think of this in two ways, just as we did in the first example above.

The starting material cannot form a carbocation, so something else must be happening. It is reasonable to assume that the first step in the sequence is the protonation of the alcohol. If the incoming chloride nucleophile attacks carbon and causes the protonated alcohol to leave at the exact same time, then there is no carbocation forming.

**So What Does it All Mean?**

You will be exploring the differences between elimination and substitution reactions. There are two types of substitution reactions, and although they were not named or described, you may be able to determine what property in a molecule would dictate one type or the other. There are two parts to this experiment. As a class, you will need to pool your data and see if you can reproduce each other’s work so you can make some definitive claims and be able to support them logically with evidence you obtain. The experiment will end with a discussion on these types of reactions, so you will be able to judge your experimental techniques.
Safety

- HCl is a very strong acid that can cause severe burns on skin and put holes in your clothing. Gloves are required.
- When working with reagents, be sure to close the bottles when you are done with them.
- All waste needs to be drained into a waste jug. Nothing goes down the drain.
- Use gloves when working with AgNO₃

Procedure

Part 1: tert-butyl Alcohol with HCl

Determine the mass of two clean, dry centrifuge tubes and caps. Pour 4 mL of tert-butyl alcohol into each of two centrifuge tubes. Cap the tubes securely and determine the exact mass of starting material you are using.

Add 10 mL of cold concentrated hydrochloric acid. Gently mix the components—but avoid shaking the tubes vigorously.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>tert-butyl alcohol</td>
<td>74.12</td>
<td>25–26</td>
<td>83</td>
<td>0.775</td>
</tr>
<tr>
<td>Hydrochloric acid (12 M)</td>
<td>—</td>
<td>−26</td>
<td>48</td>
<td>1.18</td>
</tr>
<tr>
<td>Calcium chloride</td>
<td>Drying agent</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2-methyl-2-propene</td>
<td>56.10</td>
<td>−140</td>
<td>−6.9</td>
<td>1.997</td>
</tr>
<tr>
<td>tert-butyl chloride</td>
<td>92.57</td>
<td>−25</td>
<td>51–52</td>
<td>0.851</td>
</tr>
</tbody>
</table>

Carefully vent each tube and allow them to stand for 20 minutes. At this stage, you may see two layers in each of the centrifuge tubes. Use the reagent table above to help you determine which one you want to throw away. Combine the layers you keep in a 50-mL Erlenmeyer flask. Add ~1 g of anhydrous calcium chloride and allow 2–3 minutes for the cloudy liquid to dry and become clear. Transfer the dried material to a distilling flask using a disposable pipette, and distill the product as you did during the dehydration experiment (apparatus shown above). When no more product comes over the sidearm, take apart the distillation apparatus and record the mass of your product. Be sure to record the boiling point as well.

Part 2: Qualitative Analysis of Substitution Reactions

There are six different alkyl halides that you will need to experiment with. There are also two different reagents you will use to characterize the alkyl halides. One of the reagents you will be using is silver nitrate (AgNO₃). Since AgNO₃ is soluble in water, it exists as ions in solution (Ag⁺ and NO₃⁻). Some silver salts, however, are insoluble so they form precipitates in water. If there are some free Cl⁻ or Br⁻ ions in solution, for example, they will react with Ag⁺ to form either AgCl or AgBr.

The other reagent you will use is sodium iodide (NaI) in acetone. In acetone, NaI exists as the ions Na⁺ and I⁻. I⁻ is an exceptional nucleophile and can attack a partially positive compound to kick off another leaving group (Cl⁻ or Br⁻). The Na⁺ ions in solution can then react with the negatively charged leaving group to produce either NaCl or NaBr, both of which are insoluble in acetone.

**NaI in Acetone**

Label a series of six clean and dry test tubes 1–6 and in each tube place 2 mL of a 15% NaI in acetone solution. Now add 4 drops of one of the alkyl halides from the table to each of the test tubes. After adding the alkyl halide, gently shake the test tube and record your observations. If a precipitate forms, be sure to record the time required for the formation of one. After about 5 minutes, place any test tubes that do not contain a precipitate in a 50°C water bath. Make sure that the temperature of the bath does not exceed 50°C, otherwise all of the acetone will boil off. After about 1 minute of heating, cool the
tube to room temperature and note whether anything happened.

*AgNO₃ in Ethanol*

The same procedure can be followed as previously described substituting AgNO₃ for NaI. For heating the tubes that do not contain a precipitate, heat the water bath to ~70 °C. Tabulate your results in the table below.

**Analysis**

**Part 1**

- IR Spectroscopy—What do you see? (Use the chart on page 156.)
- Boiling point—how does it compare to the value in the reagent table?
- Product Identification—use the boiling point in the reagent table to identify your product. The IR will help assess purity.
- Theoretical and percent yield.

**Part 2**

- You are looking to develop a trend. What is different about each of the six starting materials?
- If a precipitate formed, what does that tell you?
- Arrange the six starting materials in terms of time. What do you notice?

<table>
<thead>
<tr>
<th>Compound</th>
<th>NaI in Acetone</th>
<th>AgNO₃ in Ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Time @ 25°C</td>
<td>Time @ 50°C</td>
</tr>
<tr>
<td>1-chlorobutane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-chlorobutane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tert-butylchloride</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-bromobutane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-bromobutane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tert-butylbromide</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Before the Lab**

- Look up S₅1 and S₅2 reactions in your book to find out a little about them.
- Given the structure of the starting material, predict whether it would undergo an S₅1 or and S₅2 reaction.
- Given a starting material and the amount of acid it is reacted with, predict whether you would more likely obtain the elimination product or the substitution product.
- Carbocation stability—familiarize yourself with the trend.
After the Lab

• Write out the mechanism for the product you obtained.

• Did your reaction undergo $S_N1$, $S_N2$, or elimination? Is there anything in terms of the structure of the starting material that would help you predict this? (See Part 2 data)

• What can you use as evidence to support the structure of your product? (Find a reference spectrum.)

• Based on class data, what are some of the governing factors that dictate what type of reaction (substitution vs. elimination) these two starting materials undergo?
Lab 9—Artificial Scents: The Chemistry of Fragrances

Introduction

Have you ever wondered how a shampoo company makes their shampoo smell like some fruit? Or maybe why different perfumes can have different scents? The answer lies within organic chemistry, by way of a reaction called esterification. Simply put, this is a reaction type that produces esters. A generic structure of an ester is shown below.

\[
R - O - C - O - R
\]

A general ester

\[
\text{Methyl benzoate}
\]

Esters are carbonyl compounds (meaning they have a C=O). The R groups in the general form can be any carbon-bearing group. You have already used methyl benzoate while experimenting with aromatic substitution. In this compound, the benzene ring and the methyl group are the R groups that the general structure represents. Most esters have distinctive scents, leading to their widespread use as artificial flavorings and fragrances. Depending on what the R groups are, the compound will have a different scent and can be added to such commodities as shampoo, soap, lotion, and food (non-toxic esters, of course). For today’s experiment, you will be making a variety of esters and analyzing them on the basis of their scent.

The Big Idea

A wide variety of scented products are on the market. Due to the high cost or unavailability of natural flavor extracts (scents), companies employ scientists to synthesize compounds that are identical to those that are found in nature. This process is much more affordable, and leads to much cheaper products. You will be asked to prepare an ester, in a similar fashion as a scientist working for a shampoo company. The process used in industry requires an alcohol and a carboxylic acid as starting materials. In the presence of a catalytic amount of acid, these two compounds will react as shown in the mechanism on the following page. Once again you will be setting up a reflux apparatus to speed up the process. IR spectroscopy will be used to analyze the purity of your scented compound.

Mechanism for Esterification

In the first step of the reaction, the carbonyl oxygen attacks a proton from the acid, which makes the carbonyl group more reactive. The alcohol then attacks the carbonyl group, causing the electrons in the double bond to fold out onto oxygen. One of the OH groups pulls the proton off the incoming alcohol to form a good leaving group (water). The oxygen from the free OH group then folds electrons back onto carbon to regain the double bond, which causes water to leave. This water molecule then attacks the proton on oxygen to regenerate the acid and keep the reaction going.

There are a wide variety of alcohols and carboxylic acids for you to choose from. As a class, see how many different combinations you can come up with.
Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alcohols</td>
<td>See bottle</td>
<td>See bottle</td>
<td>See bottle</td>
<td>See bottle</td>
</tr>
<tr>
<td>Carboxylic acids</td>
<td>See bottle</td>
<td>See bottle</td>
<td>See bottle</td>
<td>See bottle</td>
</tr>
<tr>
<td>Sulfuric acid (18 M)</td>
<td>—</td>
<td>10</td>
<td>337</td>
<td>1.84</td>
</tr>
<tr>
<td>Aq. sodium bicarbonate</td>
<td>For extraction</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Magnesium sulfate</td>
<td>Drying agent</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

Safety
- Carboxylic acids severely stink! Keep these compounds in the fume hood whenever possible.
- Concentrated sulfuric acid is corrosive and causes burns. Use caution when working with it.
- Avoid inhaling large amounts of your product. Although many of these esters are non-toxic, there may be some impurities in your final product that are toxic.

Procedure
Record the mass of a 10-mL round-bottom flask. Place approximately 12.5 mMol of an alcohol into this flask and record the mass again to determine an accurate mass of your alcohol. Add 26 mMol of a carboxylic acid to the flask. Then add 5 - 7 drops of concentrated sulfuric acid. Add a small magnetic stirrer to the flask and assemble the reflux apparatus. Reflux the reaction for 40 minutes.

After the 40-minute reflux period, turn off the heat and allow the reaction mixture to cool. Using a disposable pipette, transfer the cooled reaction mixture to a centrifuge tube. Add 3 mL of water to the centrifuge tube. After mixing the contents of the tube, withdraw the aqueous layer and discard it. Slowly, and in small portions, add a total of 3 mL of 5% aqueous sodium bicarbonate to the reaction mixture. Agitate the mixture until the fizzing stops. Using a disposable pipette, remove the aqueous layer and discard it. Repeat this extraction twice using a fresh 3-mL portion of 5% aqueous sodium bicarbonate.
each time. Try to remove as much water from the centrifuge tube as you possibly can. Add a small amount of anhydrous magnesium sulfate to dry the ester (about 10% by volume). Allow the mixture to dry for a few minutes. Transfer the dry ester to a clean, dry, pre-massed 10-mL round-bottom flask while leaving the drying agent in the centrifuge tube. Determine the theoretical and percent yield of your ester.

Analysis

Scents

• IR Spectroscopy—a carboxylic acid contains an OH group. If any is unreacted you will see a peak in roughly the same area as you would find an alcohol.

• With the structures of the starting materials, go through the mechanism to determine the structure of your ester.

• Scent—What does your ester smell like? As a class, tabulate the results on the chalkboard.

• Theoretical and percent yield

Before the Lab

• Given the structure of an ester, determine the structures of the alcohol and the carboxylic acid that were required to make it.

• Given the structures of a carboxylic acid and an alcohol, draw the structure of the ester they would produce.

After the Lab

• Write out the mechanism for the product you obtained (No R groups).

• How accurate are your results? Can you find a table anywhere to compare against? (Use the Internet)

• Flavorings, scents, and perfumes all take advantage of this reaction. Name the ester that you made, and see if you can find any information about it on the Internet. Is it made on a larger scale? What is it used for?

• Look at the ingredient list on a shampoo bottle. Can you determine if there are any esters in the shampoo?
Lab 10—Sodium Borohydride: From a One-Time Accident to H₂ Fuel Cells

Introduction

NaBH₄ was first synthesized in the early 1940s. Its reactive properties were discovered by accident. At the time, H.C. Brown and Hermann Schlesinger were searching for a solvent to purify sodium borohydride. When they tried acetone, they found that the acetone was converted into isopropyl alcohol (2-propanol). After further researching, they became pioneers in the use of boron compounds in organic synthesis. H.C. Brown would later go on to be awarded a Nobel Prize in chemistry for related work. This example illustrates the importance of asking questions and doing experimental work to make claims. As mentioned, the reactivity of NaBH₄ was discovered by accident. Many of the most promising advances in science are discovered in this fashion. As it turns out, this one-time accidental chemical is now being studied further as an energy source for direct borohydride fuel cells.

NaBH₄ is a reagent that will reduce carbonyl compounds. You have already reduced double bonds in olive oil, so this could be thought of as an extension. While the class of reactions is somewhat similar, the products are quite a bit different.

The Big Idea

A carbonyl compound is one that contains a C=O bond. You have been introduced to this functional group in the form of an ester and a carboxylic acid, but you have also seen it in the structures of olive oil, acetonilide, methyl benzoate, and benzoic acid. The numbers of compounds that contain a carbonyl group are numerous, so much so that this group is found in several different classes of compounds.

\[
\begin{align*}
\text{Ketone} & : O=CH_3 \\
\text{Aldehyde} & : CH=O \\
\text{Carboxylic acid} & : COOH \\
\text{Ester} & : COOCH_3 \\
\text{Acyl chloride} & : COCl \\
\text{Amide} & : CONH_2 \\
\text{Anhydride} & : COOCH_3
\end{align*}
\]

Oxygen is more electronegative than carbon, so it pulls electron density away from carbon to increase the double bond’s polarity. Therefore, the carbonyl carbon becomes more electrophilic, making it more reactive to nucleophiles. All of the compounds above share this trend. Despite this similarity, all of these compounds can react differently in the presence of various reagents. You will be reacting NaBH₄ with compounds that contain carbonyl groups to test what effect this reagent has on the different groups. This reagent can be thought of as a hydride (H⁻) donor. The hydride ion can be thought of as a nucleophile. This sets the stage for the reaction that you will be performing.

Safety

- NaBH₄ is hygroscopic, meaning it reacts quickly with water. If the humidity is high, you need to work quickly with it. Try not to leave it out in the open for long periods of time.
Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>para</em>-tolualdehyde</td>
<td>120.15</td>
<td>–6</td>
<td>205</td>
<td>1.02</td>
</tr>
<tr>
<td>Benzophenone</td>
<td>182.22</td>
<td>48.5</td>
<td>305.4</td>
<td>1.11</td>
</tr>
<tr>
<td>Vanillin acetate</td>
<td>194.19</td>
<td>77–79</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Sodium borohydride</td>
<td>37.83</td>
<td>400</td>
<td>500</td>
<td>1.07</td>
</tr>
<tr>
<td>95% ethanol Solvent</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Methanol Solvent</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Diethyl ether Solvent</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Aq. sodium chloride</td>
<td>For extraction</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Magnesium sulfate Drying agent</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

- Concentrated HCl is corrosive and can cause burns. Handle with care.
- Diethyl ether is highly volatile. Handle this reagent in the hood whenever possible.

Procedure

*para*-tolualdehyde and Benzophenone

Mix 2.5 g starting material and approximately 30 mL methanol in a 250-mL beaker containing a magnetic stir bar. Slowly add (in small portions) about 0.5 g NaBH₄. Keep the temperature of the reaction below 60°C by using an ice bath if necessary. Allow the reaction to stir for 25 minutes. Pour the reaction mixture into a 400-mL beaker that contains a small handful of crushed ice and 10 mL concentrated hydrochloric acid. After the frothing has ceased, collect the solid product by vacuum filtration. Record the mass of the product.

Vanillin Acetate

Mix 2.5 g vanillin acetate and approximately 20 mL 100% ethanol in a 250-mL beaker containing a magnetic stir bar. Cool the mixture in an ice bath and then slowly add (in small portions) 1.5 g NaBH₄. Allow the reaction to stir for 15
minutes, being careful to maintain the reaction temperature below 60°C. After the reaction, add 50 mL H₂O and allow this solution to continue stirring for 3–5 minutes.

Use a separatory funnel to extract the aqueous solution twice with diethyl ether (2 × 30 mL). Combine the two organic extracts in the separatory funnel and add 10 mL water. Remove the aqueous layer. To the organic layer, add 30 mL aqueous sodium chloride solution. Remove the aqueous layer and drain the organic layer into a 100 mL beaker that contains ~1 g magnesium sulfate. Pour the solution into a 100-mL round-bottom flask and use a rotary evaporator to remove the solvent. Determine the mass of your product.

**Analysis**

- IR spectroscopy—What peaks are present? What peaks are missing?
- Melting point analysis for solids
- TLC
- Structure determination—What is the structure of your product? How do you know?
- Of the functional groups tested, which ones reacted with NaBH₄?
- Theoretical yield and percent yield (NaBH₄ is not the limiting reagent)

**Before the Lab**

- Look up NaBH₄ in your textbook to get an understanding of its reactivity.
- What is the mechanism? How does it react?
- What products are produced?
- What functional groups will react with NaBH₄? Are there some that will not?

**After the Lab**

- NaBH₄ does have its limits when reacting with carbonyl compounds. What other reagent can be used to reduce carbonyl compounds that won’t react with NaBH₄?
- For solids, compare the melting point you obtained with the accepted melting point of your product.
- As was mentioned, NaBH₄ is currently being researched for use in hydrogen fuel cells. How is this possible?
Lab 11—Chemical Inventory: Preparing for the EPA Inspection

Introduction
At typical research universities, it can be quite common for faculty members to be in charge of their research labs for considerable amounts of time. New chemicals are purchased on almost a daily basis to serve the needs of the current projects. Since many graduate students work in the research laboratory, a large number of chemicals can come in, each finding a place to occupy. As the projects change or finish up, new projects begin, resulting in the acquisition of new chemicals. But what happens to the old chemicals? Maybe the project only called for a few milligrams of a particular compound, but the chemical company only sold gram quantities. If the excess chemical isn’t needed anymore, perhaps it finds its way into the chemical supply room where it may sit dormant for a period of years. As the time passes by, the label may wear off and no one would notice it until a chemical inventory was completed. So what do you do if you find a bottle that doesn’t have a label on it? You can’t just throw it in the trash or dump it down the drain; state and federal laws prohibit that. The people in charge of disposing it need to know what it is for their safety. It becomes your job to find out what this mystery compound is. You need to do this in relatively short order, especially if the EPA is coming for an inspection. Recently the EPA fined a university $1.7 million because chemicals were improperly labeled and stored. This experiment will introduce you to some qualitative techniques you can use that will help you figure out what the unknown chemical is.

The Big Idea
This experiment will allow you to focus in on different functional groups and will aid you in determining the differences in reactivity they exhibit. Consider the following functional groups:

- **Ketone**
- **Aldehyde**
- **Carboxylic acid**

Throughout the course of this term, you have worked with compounds that represent all of these functional groups. The most common way you have analyzed these compounds was by using infrared spectroscopy. IR could certainly tell the difference between alcohols and carbonyl compounds, but how could IR tell the difference between 1°, 2°, or 3° alcohols? Similarly, the carbonyl peak usually shows up in the range of 1600–1800 cm⁻¹, and is different depending on whether you have a ketone, aldehyde, or a carboxylic acid. Depending on the structures, though, sometimes the IR frequency of these three classes of compounds can overlap. This being said, it could prove rather difficult to identify your compound by IR alone.

Fortunately, the above compounds can react differently with a wide variety of reagents. Some will produce colorful products, precipitates, or changes in pH.
These are all observations that you can see or measure that will make the task a little easier. In addition, you will now be able to use $^1$H NMR to put together a structure of your sample. Using $^1$H NMR in conjunction with the observational tests, you should be able to prove with great certainty what that unlabeled compound is.

**Safety**

- The unknown compounds you will be working with all fall into one of the functional group categories mentioned earlier. Standard safety procedures should be followed when working with these compounds.
- You will be working with a variety of chemical tests. Specific safety precautions will be pointed out for each of these in the procedure section.

**Procedure**

You and your partner will each need to choose a vial containing a sample of an unlabeled compound. There are many of these unlabeled compounds available, and as a class you will be responsible for identifying them all. For this reason, it may be beneficial to create a table. It will be important for you to remember how to know when a reaction takes place. In general chemistry, you performed an experiment where you looked for signs that a reaction took place (color change, production of a gas, a precipitate, heat, etc.). Some of these signs may be evident.

The first thing you should do is to take an IR of your sample. This should help narrow down your choices. However, since the IR is sensitive to impurities, you should also do the following test with 2,4-dinitrophenylhydrazine (2,4–DNP).

Add 1–2 drops of a known alcohol to a small test tube. To this, add 1 mL of 2,4–DNP solution and gently agitate the mixture for 30 seconds and note whether anything happens. Repeat this test using acetone and report your results. Now follow the same procedure using 1–2 drops of your unknown solution. Based on what you observed for the two known solutions, can you draw any conclusions regarding your unknown sample?

1. **Oxidation**

You have already reacted ketones and aldehydes with sodium borohydride to produce alcohols. You learned that this process was a reduction of the carbonyl group. In the reverse reaction, alcohols can be oxidized to form carbonyl compounds. Depending on what type of alcohol you have, you could get different results. When experimenting with NaBH$_4$, it was mentioned that 3º alcohols could not be formed by a reduction reaction. Similarly, 3º alcohols cannot be oxidized to form carbonyl compounds. For this test, you will use a reagent known as the Jones reagent. This reagent is a good reagent to use to oxidize an alcohol to a carbonyl group. Since this reagent contains chromium, be advised to wear gloves and exercise caution. For any test tube you use for chromium compounds, be sure to dump the waste in a waste jug specifically designated for chromium.

Place 5 drops of each known alcohol (1º, 2º, and 3º) into separate small test tubes. To each of the tubes, add 1 mL of acetone followed by 1–2 drops of the Jones reagent. Note the color of the Jones reagent before the addition, as well as the color after. A reaction should take place immediately, and with the known samples you will be able to tell which of the three alcohols react with the Jones reagent.
Once a trend is established, repeat this test on your unknown sample following the same procedure.

2. S$_{N}$1 Reaction

Earlier in the semester, you converted tert-butyl alcohol into tert-butyl chloride, an example of an S$_{N}$1 reaction. You knew a reaction took place when you saw two layers in the centrifuge tubes. What structural features favor an S$_{N}$1 reaction? This test will introduce you to a reagent known as the Lucas reagent. If you know the trends in an S$_{N}$1 reaction, then you will be able to utilize this test to narrow down your results from part 1 above.

Add 0.5 mL of each of three different known alcohols (1°, 2°, and 3°) to three different test tubes. Add 3 mL of the Lucas reagent into each of the test tubes, one at a time. Stopper the test tube, shake it, and allow it to stand for a few minutes. Carefully inspect your solution, looking for the formation of two layers. Also note the time required for the layers to form. If no layer forms, place the test tube in a hot water bath for a few minutes. If after 5 minutes of heating no layers form, then the test can be considered negative. Repeat this test using your unknown sample following the same procedure. Based on your results, can you establish a trend based on the structure of the starting alcohol? If your unknown sample forms two layers, can you fit it into the trend that you developed?

With the information gained from the two tests above for alcohols, you should know what the general structure of your alcohol is (1°, 2°, or 3°). To determine the absolute structure you will now need to use the $^1$H NMR. You will be given a printout of a spectrum for your compound.

If You Have a Carbonyl Compound

If the IR spectrum you obtained from your sample indicates the presence of a carbonyl peak, you could have either an aldehyde or a ketone. There are two tests you can do to distinguish between the two. They are very sensitive to impurities, however, so it is recommended to do both of them to be absolutely certain of the identity of your unknown.

1. The Schiff Reagent

Add 2 mL of the Schiff reagent to each of two small test tubes. Add 1–2 drops of a known aldehyde to one of the tubes and 1–2 drops of a known ketone to the other. Shake the test tube and allow the mixture to stand for a few minutes. Note any changes that happen. Look specifically for differences in color. Repeat this test on your unknown sample, following the same procedure.

2. Oxidation

Aldehydes can further be oxidized to form another class of compounds that you have worked with. Ketones, on the other hand, cannot. To understand the difference, you’ll need to know your functional groups. In addition, you will need to know what oxidation means. If you imagine oxidation meaning “the addition of oxygen,” then what functional group could form from the oxidation of an aldehyde?

The Jones reagent will serve as the reagent to run the oxidation reaction. Since the Jones reagent contains chromium, be sure to follow all safety procedures (gloves, goggles, etc.). Any test tube that contains this reagent needs to be dumped into the waste jug specifically reserved for chromium.

Add 0.5 mL of a known aldehyde into one small test tube and 0.5 mL of a known
ketone in the other. Add 2 mL acetone, followed by a few drops of the Jones reagent. Record your results, specifically looking for any color changes to happen. Repeat this test on your unknown using the same procedure.

By using the two tests mentioned above, you should have a pretty good idea of what the general structure of your unknown chemical is. To determine the absolute structure, you will now need to use the $^1$H NMR. You will be given a printout of a spectrum for your unknown.

**Analysis**

- Using the chemical tests in addition to the $^1$H NMR, determine the structure and the name of your unknown sample.
- Compare the $^1$H NMR spectra of different functional groups (aldehyde vs. alcohol, etc.). Are there any differences in target peaks?
- Once you know the structure of your unknown, what products were produced when you subjected it to the conditions for oxidation?
- Many of these tests are sensitive to impurities. Given this information, can you conclude if your unknown sample is pure or not? Did you get any positive tests where you think you should not have?

**Before the Lab**

- What is the reactivity order in an $S_{N1}$ reaction when working with alcohols?
- $^1$H NMR—Given a structure, can you predict (1) how many signals you expect to see, (2) where the signals show up, and (3) the splitting pattern observed for each signal?
- $^1$H NMR—Given a spectrum, can you assign a structure to the compound?

**After the Lab**

- One of the reagents you worked with was the Jones reagent. What product forms if a $^1$° alcohol is treated with this reagent? $^2$° alcohol? Aldehyde?
- Another reagent used for the oxidation of alcohols is pyridinium chlorochromate (PCC). It is similar to the Jones reagent, but there is one big difference you should be aware of. Look up these two reagents in your textbook to find out what this difference is.
Lab 12—The Aldol Condensation: The Carbon Construction Company

Introduction

The aldol reaction is a very important reaction in organic synthesis because it constructs new carbon-carbon bonds. In the simplest sense, the reaction involves the addition of an aldehyde or a ketone to another aldehyde or ketone. Since the carbon atoms of a carbonyl group are partially positive, they make excellent electrophiles. When ketones are treated with a base, they can easily lose a proton, since the resulting anion can be stabilized by resonance. This is illustrated below using a simple ketone.

A base (hydroxide ion) will attack a proton that is α to the carbonyl group. When hydroxide attacks a proton it becomes water (\( \text{OH} + \text{H}^+ \rightarrow \text{H}_2\text{O} \)). Left behind is the negatively charged carbon anion. If no other electrophile is around, this negatively charged anion could attack water and reproduce the starting material. This is why an equilibrium arrow is shown; this reaction can go back and forth.

It was mentioned above that this reaction is very important to scientists who specialize in organic synthesis. A simple definition of organic synthesis is the construction of organic molecules via chemical processes. More precise however would be an aspect of organic synthesis called total synthesis in which complex organic molecules are constructed from commercially available and often times cheap starting materials.

This type of research is critical in the pharmaceutical industry, for example, where very complex structures are produced on a very large scale. If the price of the starting materials is very expensive, then that price trickles down to the consumer once the product is made.

The Big Idea

There are two aldehydes and two ketones listed in the procedure section. Your job will be to select one of each of them and react them together. You will be responsible for determining what the structure of your product is. The melting point is given so you have something to compare against, but without the structure, you cannot do a theoretical yield calculation and you cannot do any type of analysis by instrumentation. This leads us into a discussion about mechanisms. Since you have been providing mechanisms for each reaction you have done this semester, you should consider this as something you would have to do anyway. Understanding the mechanism will help you in determining what the structure of your product is. The melting point analysis and spectral data you collect will provide evidence to help support your claim of what the structure is.

\[
\begin{align*}
\text{Resonance-stabilized Anion}
\end{align*}
\]
The scheme above showed what happened to a ketone after it was reacted with a base. It was mentioned that the anion is stabilized by resonance and two structures were drawn. Again, the two structures are drawn below, but now a little more detail needs to go into the discussion.

The two possible resonance structures formed are called tautomers. The structure that contains the ketone is called the keto tautomer \((\text{keto} = \text{ketone})\). The structure that contains the alkene is called the enol tautomer \((\text{en} = \text{alkene}; \text{ol} = \text{alcohol})\). With a negative charge in the molecule, a reaction is waiting to take place; all that is required is an electrophile. Since you will be using aldehydes in addition to ketones, you know that the only thing you have left is the aldehyde. So what happens if an aldehyde is dropped into a reaction mixture containing a negative charge? From experimenting with sodium borohydride, you know that aldehydes are electrophiles. The scheme below illustrates what happens when a generic aldehyde is added to the mix.

With the keto form of the starting ketone, the negative charge is capable of attacking the carbonyl carbon of the aldehyde that was added into the mix (step 2). Since water is present from the first acid-base reaction, it loses a proton to the \(O\) so that the base can be regenerated and this reaction can continue on. This results in the formation of a new compound that contains both a ketone and an alcohol. This compound is called a \(\beta\)-hydroxy ketone. This reaction could be stopped at this point, but under the reaction conditions a second reaction may take place. Recall what happened when you dehydrated an alcohol by mixing it with sulfuric acid. The resulting compound was an alkene. This same reaction can happen under basic conditions (step 3).

The excess base present in the reaction mixture can attack a proton \(\alpha\) to the carbonyl group. The resulting negative charge folds down expelling the hydroxide group, and forming water in the process. Even though the hydroxide group is a poor leaving group, the basic conditions of the reaction allow this to happen. From the margarine lab you were introduced to the concept of saturated vs. unsaturated carbon atoms. This convention can be extended to help name the compound that was formed. The double bond that was formed in the sequence above touches both the \(\alpha\) carbon and the \(\beta\) carbon (relative to the carbonyl). You know that you have a ketone. Putting the terms together gives you an \(\alpha,\beta\)-unsaturated ketone.
The ketones you will use contain acidic hydrogens on both sides of the carbonyl group. This adds more variety to the reaction you will do. Let’s take another look at that $\alpha,\beta$-unsaturated ketone you made from the last step. What happens if $R_1$ represents a group containing hydrogens, such as a methyl group? We can essentially start from the beginning, using the extra base in the reaction to deprotonate the ketone (step 4).

This reaction should look very similar to the first reaction that was drawn in the beginning. Once again, a base is attacking the protons $\alpha$ to a carbonyl group, resulting in a compound that has a negative charge that can be stabilized by resonance. Again the two different forms (keto and enol) could be drawn to show this. Under the conditions employed in this experiment, you will be using twice as much aldehyde as ketone. In forming the $\alpha,\beta$-unsaturated ketone, one equivalent of the ketone and one equivalent of the aldehyde were consumed. If however, another negative charge is floating around with some aldehyde, this reaction can keep going.

**Reagent Data**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>58.08</td>
<td>−94.9</td>
<td>56.3</td>
<td>0.79</td>
</tr>
<tr>
<td>Cyclopentanone</td>
<td>84.12</td>
<td>−51</td>
<td>131</td>
<td>0.95</td>
</tr>
<tr>
<td>Benzaldehyde</td>
<td>106.13</td>
<td>−26</td>
<td>178.1</td>
<td>1.04</td>
</tr>
<tr>
<td><em>trans</em>-cinnamaldehyde</td>
<td>132.16</td>
<td>7.4</td>
<td>251</td>
<td>1.04</td>
</tr>
<tr>
<td>95% ethanol</td>
<td>Solvent</td>
<td>—</td>
<td>—</td>
<td>0.78</td>
</tr>
<tr>
<td>Diethyl ether</td>
<td>Solvent</td>
<td>−116.3</td>
<td>34.6</td>
<td>0.71</td>
</tr>
<tr>
<td>2M sodium hydroxide</td>
<td>2 mol / liter</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Magnesium sulfate</td>
<td>Drying agent</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>
Just as was the case in the beginning, a compound is formed that contains both a ketone and an alcohol. In forming the alcohol this time, –OH is again produced. In a similar fashion as before, under the conditions we can eliminate the OH group to form the alkene (step 6).

It should be noted that even though several different reactions were drawn out in this description, many of them are all identical. Depending on the conditions that you use in your procedure any one of these reactions can happen. For this reason, it is critical that you use precision in measuring out the amounts of materials you are using.

**Safety**

- 2M Sodium hydroxide is a very strong base; handle this solution with care
- Ethanol, ether, and acetone are all flammable liquids. No flames will be allowed during the laboratory period.

**Procedure**

In order for the reaction to proceed in a smooth manner, it is important to make sure you are using exact amounts. It is critical to use at least a 1:2 mole ratio between the ketone and the aldehyde you choose to work with. There are two aldehydes and two ketones to choose from, but the general procedure given will apply to all four possible products.

Add 0.023 mol of the aldehyde and 20 mL 95% ethanol to a 125-mL Erlenmeyer flask. Swirl the solution to mix and then add 10 mL 2M sodium hydroxide. Swirl this solution for about 1 minute and then add 0.01 mol of a ketone. Swirl this solution for a few minutes to mix it, and then allow it to sit undisturbed. Be sure to look for any signs indicating a reaction happening.

Once you see a reaction happening, cool the Erlenmeyer flask in an ice water bath for about 15 minutes. Depending on what type of product you have there will be two ways for you to isolate it. If you have a solid, then you can simply filter your solution using either the Hirsch funnel or the Büchner funnel. You can wash the product as well as the Erlenmeyer flask with several portions of ice-cold 95% ethanol (3 portions, ~2 mL each portion). If you have a liquid, you can use extraction to collect your product. To do this, transfer your liquid from the Erlenmeyer flask to the separatory funnel. Add 5mL diethyl ether along with 10mL water. Extract the aqueous solution twice using another 5 mL addition of diethyl ether. Keep the organic layer in a 50-mL beaker and add about 0.5 g of anhydrous magnesium sulfate (MgSO₄). Transfer this solution to a pre-massed round-bottomed flask and use the rotary evaporator to remove the solvent.

**Analysis**

- ¹H NMR – this can tell you what structure you have
- IR – this can tell you what functional groups you have
- Melting point analysis – the melting points of the expected products are given, but may not resemble the melting points for the compounds that you make
- Draw out the mechanism for the reactants you use. Not only will this help you practice your mechanism writing skills, it may also lead you to the structure of the product.
• Fill in the table below with the structures for all four compounds that the class prepared.

• Theoretical yield and percent yield

**Before the Lab**

• Certainly, this may seem like quite a bit of information. Look up the aldol reaction in your textbook and read a little about it.

• The process of tautomerization can happen under both basic and acidic conditions. Given the structure of a ketone, could you draw both the keto and enol tautomers?

• $^1$H NMR is the main method of analysis. Given the structure of a compound, can you predict how many signals would show up in the $^1$H NMR spectrum?

• Given the structure of a simple aldehyde and a ketone, can you predict what the structure of their reaction product would be?

**After the Lab**

• Total synthesis and organic synthesis – just what exactly do these terms mean?

• Can you draw the mechanism that shows the formation of your product?

• Based on the IR data, was your compound dehydrated or is there an alcohol still present?

• How pure is your product? Use the percent yield and the spectral data.

<table>
<thead>
<tr>
<th></th>
<th>acetone</th>
<th>cyclopentanone</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzaldehyde</td>
<td>Product #1</td>
<td>m.p. 113 °C</td>
</tr>
<tr>
<td></td>
<td>m.p. 189 °C</td>
<td></td>
</tr>
<tr>
<td>trans-cinnamaldehyde</td>
<td>Product #3</td>
<td>m.p. 144 °C</td>
</tr>
<tr>
<td></td>
<td>m.p. 225 °C</td>
<td></td>
</tr>
</tbody>
</table>
Lab 13—Radical Chemistry: Bromine vs. the Hydrocarbons

**Introduction**

Radicals are atomic or molecular species that contain an unpaired electron. In the simplest sense, radicals are formed when a bond between two atoms breaks, with one electron going to each of the two atoms. Radicals are not all that stable compared to the original atom, which leads them to be highly reactive in order to pair up the lone electron. To do this, they can act as scavengers, essentially stealing an electron from other chemical species. This will stabilize the radical that is doing the stealing, but it will create another radical that can go and do the same thing. What happens is a continuous cycle, which is outlined below using a chlorine molecule and methane as an example.

With light or heat, the first step (initiation) gets the cycle going by generating two chlorine radicals.

These two radicals could just recombine to form Cl₂ or as shown in the second reaction, one of the chlorine radicals can grab a hydrogen atom from methane. Since the chlorine radical only needs one electron to pair up, the other electron that was in the carbon – hydrogen bond goes to methane, which now becomes a methyl radical. In the third reaction the methyl radical can attack a different Cl₂ molecule, which produces another chlorine radical, essentially starting the whole process over again. These processes are all illustrated by using arrows to show the movement of electrons. These arrows contain a single-headed hook at the end to show that only one electron is doing the scavenging.

This cycle can be stopped whenever two radicals join together. As shown above, the termination step can involve either two methyl radicals joining to form ethane, a methyl radical joining with a chlorine radical, or two chlorine radicals combining to form Cl₂.
The Big Idea

Free radicals are of paramount importance when talking about human health. When radicals steal an electron from your cells, oxidative stress can result. A particularly destructive aspect of oxidative stress is the production of reactive oxygen species such as radicals and peroxides. Since these radicals are highly reactive, they will basically take an electron from any source they can find. In terms of physiology there is evidence to suggest that radicals can increase the appearance of aging. This theory was first proposed in the 1950's by Denham Harman, a biogerontologist at the University of Nebraska Medical Center. Although this theory has the most consistent experimental support, models do exist that demonstrate increased oxidative stress without any effect on lifespan. According to data from Business Communications Company, in 2004 Americans spent nearly $45 billion on anti-aging products. In addition to aging, free radicals are also thought to be contributors to the development of a wide range of diseases including Alzheimer's disease, Parkinson's disease, diabetes, and arthritis. This shines the spotlight on antioxidants, molecules that slow or stop the oxidation (e.g. oxidative stress) of other chemicals. What this means is that antioxidants increase the likelihood of a radical cycle being terminated. This has led to a report compiled by the National Institute of Health (NIH) that shows in 2006, Americans spent nearly $23 billion on dietary supplements, many of which contain antioxidants. In the same report however, the NIH claims that there is no convincing evidence to support the claim that taking supplements is a good idea for the general population.

Although this experiment is not going to answer any of the questions concerning antioxidants or their role in human health, it should provide you with an introduction to radicals. For now, focus on the six substances that you will use to experiment with. Are there factors that determine the rate of reaction? If there are, could these results be extended to human health, slowing down the spread of disease or even the aging process?

Safety

- Take caution when using glass pipettes, as the tips can be very sharp.
- A solution of bromine in methylene chloride is very dense. It is also volatile. Extra caution needs to be taken when transferring this solution with pipettes. Wear protective gloves and remove them immediately if any spills should happen.
• All compounds need to stay in the fume hood. Hydrocarbons are toxic.
• All waste generated is considered halogenated waste, with the exception of acetone. Be sure to use a separate waste container for acetone.

Reagent Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Mass (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclohexane</td>
<td>84.16</td>
<td>6.6</td>
<td>80.7</td>
<td>0.78</td>
</tr>
<tr>
<td>Methylcyclohexane</td>
<td>98.19</td>
<td>-126</td>
<td>101</td>
<td>0.77</td>
</tr>
<tr>
<td>Toluene</td>
<td>92.14</td>
<td>-93</td>
<td>110.6</td>
<td>0.87</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>106.17</td>
<td>-95</td>
<td>136</td>
<td>0.87</td>
</tr>
<tr>
<td>Isopropylbenzene</td>
<td>120.19</td>
<td>-96</td>
<td>152</td>
<td>0.86</td>
</tr>
<tr>
<td>tert-Butylbenzene</td>
<td>134.22</td>
<td>-58</td>
<td>169</td>
<td>0.87</td>
</tr>
<tr>
<td>Br₂ in CH₂Cl₂ solution</td>
<td>1 mol / liter</td>
<td>--</td>
<td>--</td>
<td>~3.11</td>
</tr>
<tr>
<td>Sodium bisulfite</td>
<td>10% Aq. Solution</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

Procedure

Part 1: Room Temperature

Label six of your small test tubes so they correspond to the six hydrocarbons listed in the reagent table. Add three drops of each of the hydrocarbons to its corresponding test tube, making sure that the drops fall directly in the bottom of the test tubes. To each of the tubes, add 1 mL methylene chloride. In a separate conical vial, add ~1.0 – 1.5 mL of the bromine solution. Cap the vial and place in your fume hood.

Add six drops of the bromine solution to one of the test tubes containing a hydrocarbon. You will want to do this quickly, but again, try to make sure these drops fall directly into the hydrocarbon solution. After adding the final drop of bromine, have your lab partner note the time the last of the bromine was added. Agitate the mixture until the reaction appears to be complete. The maximum time to spend on any of these solutions should only be 20 minutes. If there is no apparent reaction after 20 minutes, then the hydrocarbon can be considered non-reactive. If this is the case, then add a small amount (~0.5 mL) of sodium bisulfite solution to the tube.

As soon as this reaction is complete, pour the solution into the waste jug.

• The UV lamps produce harmful radiation. Do not look directly at the bulb and avoid shining the light directly on exposed skin.
Part 2: Elevated Temperature

If the reaction does not occur after this time, then the hydrocarbon can be considered non-reactive.

Part 3: Room Temperature With Radiation

Repeat the same procedure you followed in parts 1 and 2. Place all 6 samples in an empty beaker, add the bromine solution, and immediately shine them with a UV lamp. Once again, begin a timer after the addition of the bromine, to monitor the reaction.

Analysis

- As a class, pool your data on the chalkboard. Which solutions reacted faster? Were there any hydrocarbons that did not react?
- What happened when the reaction conditions were altered (2 & 3)?
- Propose structures for the products that were formed.

Before the Lab

- Look up the phrase "free radical halogenation" in your textbook to familiarize yourself with this reaction ahead of time.
- Predict the order in which you think these hydrocarbons will react in, arranging them from fastest to slowest.
After the Lab

• How do you explain the differences in reactivity? Why do some of the hydrocarbons react faster than others?

• Look back at your prediction you made before coming to the lab. Were you correct?

• Two topics in medicine were mentioned earlier, and in both cases, more data is needed to identify the role of free radicals. If the two reports were summative, then Americans spent $68 billion on combating the two of them. Do some digging on the Internet to see if you can shed some light on these two issues.

References


For Further Reading

http://www.livescience.com/health/060912_bad_vitamins.html
APPENDIX B: PREPARATORY NOTES FOR TEACHING ASSISTANTS

TA Notes – Checking In

Pre Lab

- Introduce yourself; write your name, email, etc on the board. If you want, take attendance – this can help you get to meet them.
- Go over the syllabus with them. No need to spell it all out to them, but do emphasize the grading. Make sure to tell them about the late lab report penalty and what they can do should they miss a lab.
- Make sure to tell them they need to bring a lock; perhaps tell them how soon they need it by (by next time or 2 weeks; whichever is appropriate). Safety glasses, too!
- Tell them what you are trying to do in your role as a TA. How do you want to run your lab each week? Are you going to make sure they come with a procedure written in advance? Make sure to tell them your expectations.
- Mention WebCT – it may not be fully functional just yet, but in a few days it will be.
- Give a brief overview of the format (beg. questions, claims, etc.). If they have their lab manuals you can just point them to the first few pages. No need to completely spell it out just yet, it’s better to do this when there is some data.
- Tell them about the point system (20 per report, 5 per quiz, 50 for the practical). You may want to break the 20 points for the lab report down: Beginning Questions – 2; Safety – 1, Procedure – 1, Data – 4, Claims – 2, Evidence – 5, Reading & Reflection – 5.
- Hand out survey (I’ll supply this). After they finish take them up to the lab.

In the Lab

- Assign them to a locker (one from the sheet that Allen gave out)
- If they don’t have a lab manual, you will need to give them a check-in sheet (I’ll bring copies)
- When they finish, you will want to show them around the lab (point out all of the safety equipment.)
TA Notes – Prep of Acetanilide

Pre Lab

• Rationale: Have your students make something on day one and analyze it
• Be vague in your discussion – you want to try to get beginning questions out of them (preparation on your end is a must). Typically something to do with solvent selection, polarity, or purity.
• The *before the lab* bullets should not be used for beginning questions

Concepts

• Solvent selection
• Precision (technique, obviously)
• Melting point determination
• Theoretical yield calculation

Reaction / Procedure

• No reaction time (this is a pretty fast reaction)
• Make sure they are recording exact masses (not just the 1mL, or 1.12g in the procedure)
• They simply make two separate solutions and then combine the two to make the product
• Product is a white solid, able to be filtered

Things to Do

• Modify the mass of the starting materials (theoretical/percent yield)
• Compare melting points before and after recrystallization
• Compare experimental melting point with a known sample

Solvent Selection

• Students are told how much acetanilide will dissolve in cold and hot water as well as in cold and hot ethanol. Have them figure out which solvent is suitable to use based on this information. If they don’t know, or can’t figure it out, have them experiment. A procedure is given to them to do this, but if they are thinking, they won’t need it.
• Bring in the concept of polarity – they should know something about the “like dissolves like” concept. Can they say anything about the polarity of acetanilide?
• They will need to recrystallize their entire sample, but they are not told how much solvent to use. Try to pull this out of them by referring them to the ratios given in the procedure.

**Melting Point Determination**

• Minimum of two melting points are needed (before/after recrystallizing)
• The melting point of acetanilide is given. Have them compare their sample with the given value, or have them take a melting point of a known sample.

**Wrap-Up**

• Do not let anyone leave until you have a discussion with the class
• Go through the format with them (beginning question, claim, evidence)

**TA Notes – Caffeine Extraction**

**Pre Lab**

• The procedure is lengthy. Perhaps it would be a good idea to draw a diagram or a flow chart showing how this is all supposed to happen.
• There are two waste jugs – make sure to emphasize this throughout the lab

**Concepts**

• Extraction
• Density
• Polarity
• TLC

**Things to Do**

• The class can work as a group. Results can be put on the board so students can see how much caffeine is in each of the beverages.
• Make sure everybody isn’t picking the same beverage; try to get a variety.
• The actual melting point of caffeine is given in the reagent table. It may take awhile for students to get the Mel Temps up to 237 °C.

**Extraction**

• They will be doing a total of four extractions – time will become an issue.
• Make sure to emphasize not to shake up the solution in the separatory funnel. An emulsion occurs if the substances are shaken up.
• Don’t tell them which layer to keep. Given the density of the solutions, as well as the volumes, this should be obvious.

**TLC**
• Have students draw two lines on the TLC plate – one on the bottom (where they spot the solution) and one on the top (so they know how far the solvent travels).
• Only small spots are needed – try to emphasize to the students that they don’t need to put huge spots on the plate.
• Each pair of students can use one TLC plate – they should see two spots showing up in the same place.
• It will take at least 5 minutes for the solvent to travel up the TLC plate.

**Rotovapping**
• Make sure to put dry ice into the trap; otherwise CH₂Cl₂ goes into the pump and out into the lab.
• Students only have a 100-mL flask, but should be obtaining ~120mL of solution. Have them remove the solvent in two trials – ~60mL at a time.

**Wrap-Up**
• Use the data the students accumulate to have them tell you which substance had the most caffeine in it.

**TA Notes – Hydrogenation**

**Pre Lab**
• There are two waste jugs – make sure to emphasize this throughout the lab
• You may need to say something about refluxing (acid reflux disease?)
• Try to outline the goal…

**Concepts**
• Reactions of alkenes (hydrogenation, bromination)
• Saturation vs. unsaturation; effect on melting points
• *trans* fats
Things to Do

- Ideally, there would be more than just olive oil to choose from. Have the class split up the workload to see if any differences can be found with different oils.
- When centrifuging, make sure students keep it balanced.
- Unsaturation test – this is the main method for analysis

The Reaction

- This reaction requires 50 minutes of refluxing; you’ll need to keep them “entertained” during this time.
- When they finish, they should be left with some margarine that is stuck in the flask; they don’t need to use it all for the unsaturation test

Unsaturation Test

- They will need two different test tubes, one for the starting material and one for the product
- Have the students put 10 drops of 1.0M bromine in each test tube
- They are looking at how many drops of starting material and product are required to turn the bromine colorless
- They should find out that it takes more product to turn the bromine colorless than it does starting material. This is because the starting material has more double bonds.
- Depending on the error (cyclohexene) and the starting oil used, a ratio of 1:2 or 1:3 should be found, which can determine the degree of unsaturation.

Rotovapping

- Make sure to put dry ice into the trap; otherwise petroleum ether goes into the pump and out into the lab.
- Shouldn’t have to use water in the condenser jackets; that’s what the dry ice trap is for.

Wrap-Up

- Try to get them into a discussion about trans-fats, and all of the media buzz that is going on about them.
• The manual mentions that they need to save their margarine for a later experiment. This works well in the summer, since the next experiment involves soap making, but during the regular semesters they don’t do the soap experiment for a while.

TA Notes – Dehydration

Pre Lab
• You’ll need to explain distillation (separation of compounds by boiling point).
• Emphasize what they need to gather for evidence (bp, IR).
• Try to walk them through the mechanism (negative attacks positive).
• The product isn’t given to them.

Concepts
• Formation of alkenes
• Boiling point analysis
• IR spectroscopy
• Mechanism writing

Things to Do
• Have each pair work together – one reaction per pair.
• For reference spectra, we can use the computer in the lab. Go to WebCT and click on the spectral data link. Have the students put the name of their product and starting material in to search for reference spectra.
• Unsaturation test – they will have time to do this. Have them write the reaction that happens when bromine is reacted with their alkene.

The Reaction
• The procedure calls for 6g of starting material, but the starting material is a liquid…They need to do a calculation with the density.
• Fairly short as far as time is concerned – typical yield should be around 4 – 5g.
• The boiling point will fluctuate. They will ask you about this…explain it in terms of Le Chatlier’s principle.
Work-Up
• This is probably where the most time will be spent. Don’t just tell them what layer to keep. They need to be able to figure this out for themselves

IR
• You’ll need to run this for them. If you don’t know how to operate the instrument, it would be a good idea to come in before your lab meets.
• Most of them will still see a broad peak in the 3300cm⁻¹ range. They will assume it to be starting material, but depending on the % yield it could also be water.

Wrap-Up
• If they don’t get a printout of a reference spectrum, be sure to emphasize to them that there are links on WebCT.

TA Notes – Aromatic Substitution

Pre Lab
• Resonance structures – arrow pushing
• For each reaction, three possible products are shown, but they aren’t told which one they are “supposed” to get. Try to keep this a mystery to them.

Concepts
• Resonance structures of aromatic compounds
• Melting point analysis
• Mechanism writing
• IR spectroscopy

Things to Do
• Have two groups team up – one does nitration, the other does bromination.
• To take an IR of their sample, students will need to dissolve a small amount of the solid product in CH₂Cl₂. Have them do this in a small test tube.

The Reactions
• For brominating acetanilide, the procedure calls for 1.07g of starting material. Some students may still have this in their locker from the first experiment. If they do, they can use it, otherwise there will be some in the reagent hood for them.
• For the nitration, they need 0.5mL of the acids, and 0.42mL of methyl benzoate. They cannot measure this with their equipment. Have them do a density calculation to get the mass.

Waste Management
• Two separate waste jugs – one for strong acid waste and the other for methanol. Do not let students put methanol into the strong acid waste jug.

Analysis
• Melting point analysis – the melting points of all possible products are given. This should be pretty straightforward.
• Acetanilide – they may have a melting point somewhere in between 99 – 167. Try to get them to explain why (activating groups = ortho/para substitution)
• Methyl benzoate – they should be able to eliminate one of the products entirely (what does it mean if the melting point is negative?). Again, they might not have a melting point exactly what it is supposed to be which may indicate a mixture of products.

IR
• This can only tell them if water is present. Doing this is optional, but if they want to, then run their sample for them.

TA Notes – “Clean Chemistry” (Soap)

Pre Lab
• Try to relate this to what students have already done (hydrogenation of margarine).
• Draw a generic structure of a soap; have students tell you about hydrophobic and hydrophilic interactions.
• Try to frame the discussion around the three methods used; see if anyone wants to predict which is better.

Concepts
• Saponification
• Drawing reactions

Things to Do
• There are 3 different methods to use; have the students split it up.
• Supply crayon shavings or any fragrances if they are in the laboratory.

Analysis
• Hard water test – there are three solutions for students to test with. What does it mean if their soap forms a precipitate?
• For the hard water test, have the students compare the soap they made with commercial soaps or detergents.
• pH test – try to have them tell you why the pH is basic.

Things to Look out For
• If experimenting with solid NaOH make sure students use it quickly.
• Watch out for foaming – emphasize moderate heating.

Report
• There are 3 methods under study; probably the most common beginning question will have something to do with one of these three being better than the other.
• There really isn’t much for them to claim, and consequently not much for evidence – perhaps emphasizing the reading and reflection part would be a good idea.
• Make sure they say something about hard water, and the pH – these are really the only two methods of analysis they have – we need to make sure they understand why they are doing what they are doing.

TA Notes – Models

Pre Lab
• Students may not get to this topic until later on during the week.
• Have the quiz at the end of lab.

Concepts
• Isomers
• Enantiomers
• Drawing in 3-D

Things to Do
• Stay in the pre-lab room if possible; have students get into groups or teams
• Part I consists of 28 problems, the first 15 or so are pretty simple—try to get the students to divide this up (each group responsible for 4 or so).
• Insist that the class must reach a consensus on the first 28 before moving on.
• Part II consists of four problems—each are worth one point extra credit.
• Part III is the big deal—try to get the students to stay in class to work on them.

Model Kits
• Allen has these; you’ll need to get them from his office.
• Some of them are in shabby shape, but students can share.

Analysis
• For some of the larger structures in part III, students will want to build a model of the whole thing; tell them just to construct the area around the chiral center.
• The majority of their lecture deals with enantiomers – try to focus in on this concept.

Report
• It won’t follow the typical SWH format – the manual tells students to do some research, so let’s see just how good their research skills are.
• Do you want to collect the first 28?

Grading
• There are six drugs in part III; they need to locate the chiral centers and draw the R and S enantiomers—1 point for each (ephedrine has two chiral centers) = 6.
• For drawing R and S—each structure is worth 1 point: 2 points for the first five plus 4 points for Ephedrine = 14 points (they must have hashes and wedges).
• They need to pick one of the six drugs and do some research on it, identifying what characteristics each enantiomer has; 5 points (make sure they cite their source).
• This report will be worth 25 points with a possible 4 points extra credit.

TA Notes – The Chemical Competition

Pre Lab
• Two possible products can form for the reaction, but based on the conditions, only one will form; keep this a mystery from the students.
• Work on the general mechanism – highlight the difference between substitution and elimination.
This lab leads to a discussion about SN1 and SN2 reactions. Depending on the schedule, students may not have talked about this in lecture.

Make sure the students give equal weight to both parts; pool class data on the board

Concepts
- Substitution reactions, and the conditions required for them
- Product identification (this should be obvious, given the data in the reagent table, but based upon prior labs, this won’t be so easy for students to do)
- Back to density (once again, the layers are not identified)
- Mechanism

Things to Do
- \(t\)-butylchloride – reaction time of 20min; nothing special, but they will need to figure out which layer to keep and use distillation to get the product.
- Reactions – can use test tubes; just looking for precipitates to form and to see how long it takes.

Analysis
- IR – disappearance of OH, no C=C, formation of C—Cl.
- Boiling point – HCl boils at a similar temperature as \(t\)-butylchloride, can use pH paper?
- Have them use the reagent table – based on the bp given, they should intuitively know what to expect.
- For the reactions, they should see some trends. Try to focus on the two different leaving groups as well as 1°, 2°, and 3° structures.

Look out For
- \(t\)-butylalcohol melts at 25 °C. It’s easier to use in the liquid form, but students need to work quickly with it, otherwise it will solidify in their graduated cylinders.

Report
- They need to write out the reaction they did as well as the mechanism for it.
- Make sure in the data they are using exact values, not just those given in the procedure.
- Keep them going with theoretical and percent yield.
• Make sure they attach IR data to the report.
• Try to get them to tell you the difference between $S_N1$ and $S_N2$ reactions (elimination too).

**TA Notes – Artificial Scents**

**Pre Lab**
- Start with the structure of an ester on the board. Show students how you can break it into the acid and the alcohol it is composed of.
- Work the other way…start with an alcohol and an acid and see if they can predict the structure of the product that would form.

**Concepts**
- Esterification
- Drawing reaction schemes
- Mechanisms
- Theoretical / percent yield

**Things to Do**
- Have the students construct a grid on the board. If we have three alcohols and three acids, then they can make a total of nine esters. (Have the class split up the work load)
- No structures of the products are given…have students predict the products that can form once they have the grid on the board.
- The procedure uses mMol instead of mL. Most likely, students will not know how to do the conversion – have them look at the bottle to get the density and the formula weight.

**Analysis**
- IR – looking for the disappearance of the $–OH$ group; make sure students understand this when you run their sample…don’t just tell them.
- Have students go to the spectral database to find a reference spectrum (link on WebCT).
• Students will need to draw a mechanism for the formation of their ester. Work with them on this, since they may have just started talking about this in lecture.

**Things to Look out For**

• All of these products should be a liquid
• Some of the carboxylic acids will no doubt really stink…be wary of any spills, because these things tend to hang around for a while.
• Emphasize to the students that the carboxylic acids need to stay in the fume hoods.

**Report**

• BQ – most likely will be something about the identity of the product.
• For evidence, make sure they reference the mechanism and the IR somewhere in their report.
• R&R – they can use the Internet to find tons of tables that show structures of various esters and the scents they produce. Make sure students generate a class table that everyone copies down.
• Have them look at the ingredient list on a shampoo bottle or some food item to see if they can identify any esters present.

**TA Notes – Reductions with NaBH₄**

**Pre Lab**

• Usually, there is an exam at the beginning of the lab week. If the trend holds, pass out the quiz either after the pre-lab or even after the lab.
• Simple procedures. Have the students focus on predicting the product, especially with vanillin acetate. Don’t tell them NaBH₄ leaves esters alone.
• Mechanism

**Concepts**

• Reduction
• Reactions of carbonyl compounds
• Mechanisms
• Theoretical / percent yield
• IR
Things to Do

• Three reactions; have the students split up the workload and create some sort of class data table.
• No structures of the products are given. Students will need to figure out what the structures of the products are so they can calculate theoretical and percent yield.

Analysis

• IR – looking for the appearance of the –OH group and disappearance of C=O; make sure students understand this when you run their sample…don’t just tell them.
• Have students go to the spectral database to find a reference spectrum (link on WebCT).
• Students will need to draw a mechanism for the formation of their alcohol. Work with them on this, since they may have just started talking about this in lecture.
• We can do TLC; the reaction is fairly short – we’ll need the proper developing solvent.

Things to Look out For

• \( p \)-tolualdehyde is a liquid – students need a calculation to get mL.
• The product of the vanillin acetate reaction is a liquid – make sure dry ice is available for the rotovaps.

Report

• Beginning Question – Effect of NaBH\(_4\) on different carbonyl compounds?
• For evidence, make sure they reference the mechanism and the IR somewhere in their report.
• Reading and Reflection – see if you can get them thinking of hydrogen fuel cells – NaBH\(_4\) is currently being researched in this area.
• They are asked to compare NaBH\(_4\) with LiAlH\(_4\).

TA Notes – Chemical Inventory

Pre Lab

• This is basically an “Identify the Unknown” laboratory; students can work in pairs, but each student gets their own unknown sample.
• Try to discuss these reactions in terms of the pathway that they will take (perhaps drawing a flowchart could be helpful).
• The only reagent they are probably familiar with is the Jones reagent.
• Depending on the lecture schedule, starting $^1$H NMR with this experiment may be challenging. If you need to explain $^1$H NMR, don’t get into theory – this will only confuse your students. Instead, focus on three topics that will be useful to them: the number of signals, where they would show up, and the splitting pattern ($N + 1$ Rule).

Concepts
• Logic and reasoning
• Structure determination
• Writing reaction schemes
• IR
• $^1$H NMR

Things to Do
• Usually, there are 20 or so unknown samples. Have each pair of students obtain two or three unknown samples depending on your class size.
• Each test uses comparisons with known compounds – based on these results, students should know what to be looking for when they test their unknowns.

Analysis
• IR – won’t tell them anything about structure, but they can use this in conjunction with the DNP test to tell whether their compound contains an alcohol or carbonyl.
• Rather than running $^1$H NMR samples we usually supply them with printouts.
• Have them draw the product if their compound reacted with the Jones reagent.

Things to Look out For
• Depending on the schedule, $^1$H NMR could be a brand new topic. If this is the case, students will most likely be shaky; come prepared to explain (they usually have an exam during the week this experiment is performed).
• They will be using chromium – there will be a separate waste jug.
• These tests are very sensitive to impurities – be ready for some “false positives”.
Report

- Beginning question will most likely be “What is the unknown?”
- They will need to be rather descriptive. Make sure that they are backing up their claim by describing what happened with all of the tests.
- They have to explain their $^1$H NMR spectra; not just refer to it.
- Reflection – compare the Jones reagent to PCC, especially in the context of $^1$° alcohols.

TA Notes – Aldol Reaction

Pre Lab

- Tautomerization – go through both acid–catalyzed and base–catalyzed.
- Guide them through a mechanism; try to explain the importance of the molar ratios being used and how that can determine what happens in the reaction.
- $^1$H NMR; depends on the lecture schedule

Concepts

- $^1$H NMR
- Logic and Reasoning
- Structure determination
- Writing reaction schemes
- Theoretical & percent yield

Things to Do

- There are four possible combinations – I think students should be responsible for writing the structure of all of them, but maybe the mechanism for just their own.
- Rather than obtaining $^1$H NMR spectra we can supply them with printouts.
- We can run the IR if we really want to; if we do try to get them to tell you what they are looking for (why they are taking one).

Analysis

- IR – should show a peak for a ketone, as well as some for double bonds. Based on the purity of the compound, you could see a peak from the alcohol.
• They need to draw a reaction scheme with the expected product; this means that they will need to calculate the molecular mass of the product in order to do the theoretical yield calculation; you will need to emphasize this to them.
• They need to draw a mechanism; if they don’t they should lose 2 points in the data.
• Melting points of the products are given – they will need to compare their experimental melting point with the known.

**Things to Look out For**

• Make sure they use ice-cold ethanol to wash their product; they need to do this several times to remove the excess base – if it isn’t cold, it may dissolve their product.
• The reaction happens almost instantly, but the students don’t know this. Tell them to look for signs of a reaction happening.
• All products are solid, but they are given a procedure for extraction – they may have to do this if something goes wrong.

**TA Notes – Radical Chemistry**

**Pre Lab**

• Try to get them to make a connection between the structure of the starting material and the reactivity.
• Three different variables – see if they can ask a question concerning them.
• Focus your discussion on radicals and antioxidants (i.e. how antioxidants work).
• Emphasize safety – especially with bromine, glass pipettes, and the UV lamp.

**Concepts**

• Radical halogenation
• Reactivity

**Things to Do**

• The class will have to work as a group. Have them pair up to focus on one of the three parts. One person will have to add the bromine; the other will have to focus on the time.
• Make sure everybody isn’t picking the same method; try to get a variety.
• Students should just be looking for the color of Br\textsubscript{2} to disappear, but this isn’t given to them in the manual.

**Analysis**

• The six hydrocarbons each will take different amounts of time. Some react faster than others. See if you can get them to tell you what causes that.
• Have the students develop a trend. Have them tell you whether it agrees with the trend that they can find in their textbook.

**Special Notes**

• Br\textsubscript{2} in CH\textsubscript{2}Cl\textsubscript{2} is very dense and has a tendency to push itself out of the pipette. Have the students practice dripping it…It will be extremely difficult for them to add six drops.
• If students need to use acetone, they need to put that in a separate waste jug. Bromine and acetone form tear gas.
• One of the hydrocarbons will not react. The fastest one will take 45 – 55 seconds (under normal conditions). With heat or light this rate will be cut roughly in half. They will need to work quickly.

**Wrap-Up**

• Students should come in with a prediction about the order of the reactivity. Have them analyze the data obtained to see if their prediction was accurate.
• If their trend is not accurate, have them hypothesize why.
• For reading and reflection, have them mention something about radicals and aging, or about the cost of antioxidants.

**TA Notes – Lab Practical Exam**

**Pre Lab**

• Give students the exam in the prelab room.
• Allow students to go through the exam with their group or partner. Don’t let them write anything, but give them 5 – 10 minutes to look it over.
• Ask them if they have any questions – don’t tell them how to do anything; only address issues concerning safety.
Concepts

- Notebook keeping
- Data interpretation
- Independent work

Things to Do

- Just make sure no one is cheating.
- Remember…don’t tell them how to do anything. It is their job to put the pieces together. If they ask you something, ask them what their lab notebook says.
- Make sure that students next to each other are doing a different unknown.

Reagents

- They are usually given printouts of both $^1$H NMR and IR data in addition to the chemical formula and physical properties for each unknown.
- Different aldehydes and ketones (two or three of each); all qualitative test reagents from the unknown experiment, TLC plates & developing solution, etc…it will be critical to make sure everything they need is out and available for them to use.

Things to Look out For

- Try to practice oversight…if they are doing something they shouldn’t be doing, don’t just let them do it, but try to guide them in the right direction if they are off track.
- Remind students that they need to come back the following week to check out and complete evaluations.
- They will ask for known aldehydes and ketones to test; remind them they’ve already done it before during the unknown experiment.

Grading

- Much easier and more consistent to get together as a group. It usually only takes a little more than an hour to grade.

Note

If you have a student who has dropped the lab, email them and tell them they need to check out of their drawer. Unless you want to make arrangements there should be a deadline for checking out. If a student fails to check out, then the TA will need to do it.
APPENDIX C: SAMPLE LABORATORY REPORTS

<table>
<thead>
<tr>
<th>GRADING GUIDELINES FOR</th>
<th>Name ____________________________</th>
</tr>
</thead>
<tbody>
<tr>
<td>PREPARATIVE EXPERIMENTS</td>
<td>Section Number ____________________</td>
</tr>
<tr>
<td>Date __________________</td>
<td>GRADING DISTRIBUTION</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>I. Format</th>
<th>1 (complete)</th>
<th>0 (poor)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(title, relevant headings, permanent ink, overall neatness, clarity)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>II. Purpose &amp; Balanced Main Reaction</td>
<td>1 (complete)</td>
<td>0 (poor)</td>
</tr>
<tr>
<td>III. Reaction Mechanism (showing flow of electrons)</td>
<td>1 (complete)</td>
<td>0 (poor)</td>
</tr>
<tr>
<td>IV. Reagents and Products</td>
<td>1 (complete)</td>
<td>0 (poor)</td>
</tr>
<tr>
<td>Table of relevant reagent and product data</td>
<td>(name, MW, density, mp, bp, solubility, etc.)</td>
<td></td>
</tr>
<tr>
<td>V. Procedure</td>
<td>4 (complete)</td>
<td>3 (major omissions)</td>
</tr>
<tr>
<td>a. Full references, brief experimental details, any changes in the procedure(s)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. An abbreviated listing of the amounts of each reagent used</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. Good English spelling and grammar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VI. Observations and Data</td>
<td>4 (complete)</td>
<td>3 (major omissions)</td>
</tr>
<tr>
<td>a. Relevant observations recorded during the experiment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. Measurement of physical properties of the main product</td>
<td>(mp, bp, infrared spectra, NMR spectra, etc.)</td>
<td></td>
</tr>
<tr>
<td>c. Presentation of data in an orderly manner using tables</td>
<td></td>
<td></td>
</tr>
<tr>
<td>d. Calculation of theoretical and percent yields</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VII. Conclusions and Product Sample</td>
<td>8 (complete)</td>
<td>6 (major omissions)</td>
</tr>
<tr>
<td>a. Logical interpretation of observations and data</td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. Do the observations and data support your conclusions?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. Error discussion</td>
<td></td>
<td></td>
</tr>
<tr>
<td>d. Label including Name, Date, Name of Material, Amount</td>
<td></td>
<td></td>
</tr>
<tr>
<td>e. Quality and Appearance of Product</td>
<td></td>
<td></td>
</tr>
<tr>
<td>f. Good English spelling and grammar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VIII. TA's initials (0 points, but -1 if not there)</td>
<td>0 (TA's initials present)</td>
<td>-1 (TA's initials missing)</td>
</tr>
<tr>
<td>Total Score (20 points total)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TA Evaluation**

a. The student was adequately prepared to do this experiment
b. The student demonstrates a reasonable understanding of this experiment
c. The student complies with the safety guidelines outlined
d. The student has good lab practices
e. The student works with a minimum amount of confusion

5 (Exceptional) 4 (Good) 3 (Fair) 2 (Major Problems) 1 (Very Poor)

Figure 1: Traditional laboratory report grading rubric
Preparation of 4-methylcyclohexene (traditional)

Shown below are screen images to show a comparison between the traditional laboratory manual and the corresponding laboratory report that received a perfect score. The traditional laboratory report was graded using the rubric outlined in Figure 1.

In this experiment, 4-methylcyclohexanol, phosphoric acid, and sulfuric acid are mixed and heated to produce 4-methylcyclohexene according to the equation

\[
\text{OH} \quad \text{H}_2\text{PO}_4 / \text{H}_2\text{SO}_4 \quad \text{heat} \quad \text{CH}_3 \\
\text{CH}_3 \quad \rightarrow \\
\text{OH} \quad \text{H}^+ \quad \text{OH}_2 \quad \text{H}_2\text{O} \quad \text{slow} \quad \text{CH}_3 \\
\text{CH}_3 \quad \rightarrow \\
\text{CH}_3 \quad \rightarrow \quad \text{CH}_3
\]

**Reagent and Product Data**

<table>
<thead>
<tr>
<th>compound</th>
<th>mol. weight</th>
<th>b.p. (°C)</th>
<th>density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-methylcyclohexanol</td>
<td>114.19</td>
<td>173</td>
<td>0.91</td>
</tr>
<tr>
<td>4-methylcyclohexene</td>
<td>96.17</td>
<td>101</td>
<td>0.81</td>
</tr>
<tr>
<td>phosphoric acid/sulfuric acid</td>
<td>(catalyst)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>aq. 10% sodium carbonate</td>
<td></td>
<td></td>
<td>&gt; 1</td>
</tr>
<tr>
<td>aq. saturated sodium chloride</td>
<td></td>
<td></td>
<td>&gt; 1</td>
</tr>
</tbody>
</table>

**Theoretical Yield**

\[
\text{Theoretical Yield} = \frac{\text{# moles of limiting reagent}}{\text{molecular weight of product}} \times \frac{\text{product coefficient}}{\text{limiting reagent coefficient}}
\]

**Percent Yield**

\[
\text{Percent Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100\%
\]
**I. Purpose**: Prepare 4-methylcyclohexene from 4-methylcyclohexanol using catalysts (sulfuric acid, phosphoric acid) and distillation. Verifying identity of product by infrared spectroscopy.

*We were supposed to perform an unsaturation test but there was no cyclohexane available so this test was not done.*

**II. Chemical Equation**: 

\[ \text{4-Methylcyclohexanol} + \text{H}_2\text{PO}_4/\text{H}_3\text{PO}_4 \xrightarrow{\text{heat}} \text{4-Methylcyclohexene} + \text{H}_2\text{O} \]

(B.P. 173°C) (B.P. 101°C)

**III. Reaction Mechanism**:

\[ \text{OH} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3 \text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{O}} \text{CH}_3\text{CH}_2\text{CH} = \text{CH}_2 \xrightarrow{\text{O}_2} \text{CH}_3\text{CH} = \text{CH}_2 \]

rate-determining step = $E_1$ rxn.

**IV. Reagents and Products**

<table>
<thead>
<tr>
<th>Compound</th>
<th>mol. wt</th>
<th>B.P. °C</th>
<th>Density</th>
<th>Amt. Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Methylcyclohexanol</td>
<td>114.19g</td>
<td>173</td>
<td>0.91</td>
<td>6.6 mL or 5.71g</td>
</tr>
<tr>
<td>4-Methylcyclohexene</td>
<td>96.17g</td>
<td>101</td>
<td>0.81</td>
<td>1.0 mL</td>
</tr>
<tr>
<td>Phosphoric acid (85%)</td>
<td></td>
<td></td>
<td></td>
<td>1.8 mL</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td></td>
<td></td>
<td></td>
<td>1.0 mL</td>
</tr>
<tr>
<td>10% Sodium carbonate</td>
<td></td>
<td></td>
<td></td>
<td>2.0 mL</td>
</tr>
<tr>
<td>Anhydrous sodium sulfate</td>
<td></td>
<td></td>
<td></td>
<td>0.0 mL</td>
</tr>
</tbody>
</table>

*anhydrous sodium sulfate: drying agent*
Laboratory manual conclusion

The infrared spectrum of 4-methylcyclohexene (shown below) features bands in the region of 2800 to 3000 cm\(^{-1}\). These are due to the C-H stretches that occur on the carbon-carbon single bonds. The presence of a sharp peak near 3050 cm\(^{-1}\) is indicates the presence of a C-H stretch on a carbon-carbon double bond. The interpretation of the “fingerprint region” of these spectra, which occurs below 1500 cm\(^{-1}\), is beyond the scope of this course.

The infrared spectrum of cyclohexane (shown below) features bands in the region of 2800 to 3000 cm\(^{-1}\). These bands are attributed to the C-H stretching vibrations. The -CH\(_2\)- (methylene) groups have a characteristic absorption near 1450 cm\(^{-1}\). The absence of an intense, sharp peak near 3050 cm\(^{-1}\) is indicates that a carbon-carbon double bond is not present in cyclohexane.

The infrared spectrum of 4-methylcyclohexanol (shown below) features a wide band from 3600 to 3000 cm\(^{-1}\). This band is indicative of an O-H stretching vibration due to intermolecular hydrogen bonding and is prominent in the infrared spectra of alcohols.

Student written conclusion

![Student written conclusion image]
Alkenes: Gathering a wealth of evidence to support claims (SWH)

Beginning Questions:

1. What are different methods and techniques that can be used to determine what a product is?

2. What evidence can be used to support claims of a product consists of?
**Observations**

1. When we added the 4-methylcyclohexan and 85% phosphoric acid and concentrated sulfuric acid, I could feel a temperature change take place. The round bottom flask felt warm.

2. The reaction material in the round bottom flask changed from a yellowish color to a dark brown-black color.

**Data**

**Product**: 4-methylcyclohexanol

- **Formula mass**: 96.14 g/mol
- **Boiling Point**: 104°C
- **Density**: 0.815 g/mL

**Mechanism**

The mechanism involves the reaction of 4-methylcyclohexan with concentrated sulfuric acid and phosphoric acid to form 4-methylcyclohexanol.

**Data (continued)**

- **Theoretical yield**: 0.0526 mol x \( \frac{112}{1} \) = 0.0526 mol x \( \frac{91.177}{1} \) g/mol = 5.058 g
- **Actual yield**: 3.52 g product x \( \frac{1 \text{ mol}}{91.177 \text{ g}} \) = 0.0385 mol x
- **Product mass**: 40.697 g - 23.117 g = 3.52 g of product
- **Percent yield**: \( \frac{0.0367 \text{ mol product}}{0.0526 \text{ mol}} \times 100\% = 69.47\% \)

**Recorded boiling point of product**: 98°C good!

**Unsaturation test**: 2 drops of purple
<table>
<thead>
<tr>
<th>Claims:</th>
</tr>
</thead>
<tbody>
<tr>
<td>0. There are different methods that can be used to determine what a product is. Some of these methods to determine a chemical reaction product are IR spectroscopy, unsaturation test, percentage yield, and mechanisms.</td>
</tr>
<tr>
<td>2. Evidence from an infrared spectrophotometer can support claims of what a product consists of. Also, performing an unsaturation test will provide evidence if a product contains double bonds or not.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Evidence:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. IR Spectroscopy is a method that can be used to determine what a product is by graphing out different bands in a substance. Using the IR Spectrometer for my substance, I discovered my product had C-H stretch, C-C stretch and a C=O stretch. The unsaturation test confirmed that double bonds were indeed present in the product. By drawing out a mechanism we can predict the product. Once we have predicted the product we can compare it to published IR spectroscopy.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Evidence (continued):</th>
</tr>
</thead>
<tbody>
<tr>
<td>- When comparing graphs if the same peaks are present in the same stretches than most likely it will be the same product. Using percentage yield and IR spectroscopy can help determine the purity of the product. For example, the product I obtained with a 69.27% yield had some impurities. Looking at the IRs my product looks very similar to 4-methylcyclohexene but not exactly indicating there were impurities present.</td>
</tr>
<tr>
<td>2. After performing an unsaturation test I knew that my product had double bonds because it only took 2 drops of the product to turn the bromine colorless. I also used the IR spectroscopy to determine what my product was. I was able to compare my products IR with what I predicted it was - 4-methylcyclohexene IR. By comparing these IRs I was able to notice my product was indeed 4-methylcyclohexene because it had the same peaks (see attached IR Spectras).</td>
</tr>
</tbody>
</table>
Reading / Reflection:

1) Setting up the distillation apparatus was quite the challenge. This was the first lab that we used the different glassware: connectors. At first I was a bit skeptical of the set up but with the tightened connectors our product boiled off and flowed right into the collecting centrifuge tube.

2) When we added 1 mL of distilled water to our distilled product there were 2 distinct layers which were both colorless. Even though both substances were the same color you could see the different layers that formed because of the different densities.

Reading / Reflection (continued)

3) I felt like the most exciting part of this lab was putting all of the evidence together to figure out what my product was. In the process of putting everything together I at first came to the conclusion that I had produced 1-methyl-cyclohexene. I came to this conclusion before I worked out the mechanism for the reaction. After working out the mechanism I realized my error and looked back at all the other evidence. The IR graph’s unsaturation test being supported with evidence that the product of this chemical reaction was 1-methyl-cyclohexene. I felt like a detective.

4) Be serious, it is hard to determine the real structure depending on only IR and nuclear test.

In this reaction, Product B will be formed more likely than Product A because of the double bond location. The double bond on product B has more substituents than A therefore it is a more stable product.
Preparation of 2-chloro-2-methylpropane (traditional)

In this experiment, tert-butyl chloride (2-chloro-2-methylpropane) will be prepared from tert-butyl alcohol. This substitution reaction proceeds rapidly at room temperature with concentrated hydrochloric acid.

The mechanism of the reaction involves protonation of the alcohol. What happens next is the slow, rate-determining step of this reaction. A water molecule is lost and a carbocation is formed. The carbocation then reacts rapidly with a nucleophile like Cl⁻ to form the substitution product.
The rate-determining step is formation of the cation and depends only on the concentration of the alcohol. Hence, this is a unimolecular substitution or $S_n1$ reaction. The unimolecular substitution of an alcohol (R-OH) readily occurs when R is a tertiary alkyl group because a tertiary alkyl group forms a stabilized carbocation. An $S_n1$ mechanism may also be involved when R is a secondary group or when R forms a resonance-stabilized carbocation, such as an allyl or benzyl carbocation. Hence the general relationship for the substitution reactions of alcohols which proceed by an $S_n1$ mechanism is

\[
\text{tertiary} > \text{secondary} > \text{primary} \quad (3^\circ > 2^\circ > 1^\circ)
\]

---

**Purpose & Balanced Moins Run**

The purpose of this experiment is to make 2-chloro-2-methoxypropane from tert-butyl alcohol, an example of an $S_n1$ Run.

---

**Run Mechanism**

\[
\begin{align*}
\text{CH}_3 \text{C} & \text{O} - \text{H} + \text{H}^+ \to \text{CH}_3 \text{C} & \text{O} - \text{H}^+ \to \text{CH}_3 \text{C} & \text{O}^- \text{CH}_3 \\
\text{CH}_3 \text{C} & \text{O}^- \text{CH}_3 + \text{H}^+ \to \text{CH}_3 \text{C} & \text{Cl}^- \to \text{CH}_3 \text{C} & \text{Cl}^- \text{CH}_3 \\
\end{align*}
\]

---

**Reagents & Products**

<table>
<thead>
<tr>
<th>compound</th>
<th>mol. weight</th>
<th>m.p. (°C)</th>
<th>bp. (°C)</th>
<th>density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>tert-butyl alcohol</td>
<td>74.12</td>
<td>25-26</td>
<td>83</td>
<td>0.775</td>
</tr>
<tr>
<td>conc. hydrochloric acid</td>
<td>drying agent</td>
<td></td>
<td></td>
<td>1.18</td>
</tr>
<tr>
<td>calcium chloride</td>
<td></td>
<td></td>
<td></td>
<td>0.85</td>
</tr>
<tr>
<td>tert-butyl chloride</td>
<td>92.57</td>
<td>-25</td>
<td>51-52</td>
<td>0.851</td>
</tr>
</tbody>
</table>
Laboratory manual conclusions

The product is insoluble in concentrated hydrochloric acid and forms a distinct upper layer. After the layers are separated the product is dried using anhydrous calcium chloride and then purified by distillation. The product will be analyzed using the technique of infrared spectroscopy.

The infrared spectrum of 2-methyl-2-propanol (shown above) features bands in the region of 2800 to 3000 cm\(^{-1}\). These are due to the C-H stretches that occur on the carbon-carbon single bonds. The wide absorption band from 3600 to 3000 cm\(^{-1}\) is indicative of an O-H stretching vibration due to intermolecular hydrogen bonding.

The absence of a wide O-H absorption band from 3600 to 3000 cm\(^{-1}\) indicates the absence of an O-H functionality in 2-chloro-2-methylpropane.

Student written conclusion

The exp. was successful because the % yield was relatively high at 83.46%, the b.p. of 45°C was close to the actual value of 51-52°C for tert-butyl chloride and the infrared spectrscopy shows the lack of the OH spike at around 3500 wave numbers. The sample of tert-butyl chloride obtained was a clear liquid and it had a mass of 6.619g.
Substitution vs. Elimination: The Chemical Competition (SWH)

![Image of a table and graphs]

**Table: Reactions in Acetone and Butanol**

<table>
<thead>
<tr>
<th>Compound</th>
<th>NaI in Acetone</th>
<th>AgNO₃ in Ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Time 25°C</td>
<td>Time 35°C</td>
</tr>
<tr>
<td>1-Chlorobutane</td>
<td>None, cloudy</td>
<td>None, cloudy</td>
</tr>
<tr>
<td>2-Chlorobutane</td>
<td>None, cloudy</td>
<td>None, cloudy</td>
</tr>
<tr>
<td>1-Chlorobutanol</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>2-Chlorobutanol</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>1-Bromobutane</td>
<td>1.5 min</td>
<td>None</td>
</tr>
<tr>
<td>2-Bromobutane</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>1,2-Dibromoethane</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>1-Haloalkanes</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>Bromobenzene</td>
<td>None</td>
<td>None</td>
</tr>
</tbody>
</table>

2.717 g tert-butyl alcohol + 1.413 g tert-butyl chloride → 3.39 g theoretical yield

92.57 g tert-butyl chloride = 3.39 g theoretical yield

1.413 g × 43.45% → 43% yield

![Graphs showing data distributions]
Claims

Part one of the lab demonstrates an SN1 reaction. SN1 reactions work when the molecule is in a 1° or 2° molecule, not in a 3° molecule. SN2 reactions are opposite and occur under 1° molecules. Therefore, we can say that SN1 reactions work when a tertiary alkyl halide is present and an SN2 reaction occurs when a primary alkyl halide is present. SN1 reactions are also a 2° step. The halide leaves the molecule making it into a carbocation. Then in the second step the nucleophile attacks the positive charge and replaces the halide. SN2 reactions are just one step. The nucleophile attacks the back side of the carbon that is attached to the halogen. Steric hindrance plays a major role in the nucleophile group being attacked. The bigger the nucleophile the more the halide leaves. The SN1 reaction is much more efficient and it has a higher yield than the product and the back side of the carbon. The peak at 810 showed the addition of the C-Cl bond, therefore our molecule went from tert-butyl chloride to tert-butyl chloride.

Evidence

We are able to see the differences in the two reactions by looking at Part I and Part II of our lab. As shown in the mechanism, SN1 reaction occurred with tert-butyl alcohol coming into butyl chloride. In the first step a pair of electrons left the carbon and the HO came in. Then in the 2nd step the C-H leaves creating a carbocation in the 3rd step. Finally, the nucleophile attacks creating tert-butyl chloride. In Part II, SN2 reactions occurred with NaCl and KNO3. This means that we would not have an SN1 reaction since the substrate reacts with the nucleophile to create a carbocation which makes that an SN1 reaction.
Electrophilic aromatic substitution: The nitration of methyl benzoate (traditional)

Because of the mechanism for this reaction, we can see why the principal product in the nitration of methyl benzoate is methyl 3-nitrobenzoate. To a much lesser extent, other isomers of methyl 3-nitrobenzoate are also formed.
In Step 2, a sigma bond is formed between the aromatic ring and the electrophile. The reaction of the electrophile $E^+$ with the arene is the slow step since it results in the loss of aromaticity even though the resulting cation is still resonance stabilized.

### Reagent and Product Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Weight (g/mole)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>methyl benzoate</td>
<td>136.15</td>
<td>17</td>
<td>108-100</td>
<td>1.00</td>
</tr>
<tr>
<td>nitric acid (15.4 Molar)</td>
<td>63.01</td>
<td>-</td>
<td>-</td>
<td>1.40</td>
</tr>
<tr>
<td>sulfuric acid</td>
<td>98.08</td>
<td>-</td>
<td>-</td>
<td>1.84</td>
</tr>
<tr>
<td>methanol</td>
<td>32.04</td>
<td>-98</td>
<td>65</td>
<td>0.791</td>
</tr>
<tr>
<td>methyl 3-nitrobenzoate</td>
<td>181.15</td>
<td>78-79</td>
<td>279</td>
<td>-</td>
</tr>
</tbody>
</table>
**Purpose:** In this lab we are going to observe an electrophilic aromatic substitution through the nitration of Methyl Benzoate.

**Table of Reagents:**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula Weight (g/mole)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methyl Benzoate</td>
<td>136.15</td>
<td>-11</td>
<td>198-199</td>
<td>1.09</td>
</tr>
<tr>
<td>Nitric Acid</td>
<td>63.01</td>
<td>-</td>
<td>-</td>
<td>1.40</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>98.08</td>
<td>-</td>
<td>-</td>
<td>1.84</td>
</tr>
<tr>
<td>Methanol</td>
<td>32.04</td>
<td>-98</td>
<td>65</td>
<td>0.79</td>
</tr>
<tr>
<td>Methyl Nitrobenzene</td>
<td>181.15</td>
<td>78-79</td>
<td>274</td>
<td>-</td>
</tr>
</tbody>
</table>

\[ d = \frac{\text{m}}{\text{dV}} \]

- 0.5 mL H₂SO₄ \( \rightarrow \) \( m = (0.5 \text{mL})(1.84 \text{ g/mL}) = 0.92 \text{ g} \) H₂SO₄
- 0.5 mL HNO₃ \( \rightarrow \) \( m = (0.5 \text{mL})(1.40 \text{ g/mL}) = 0.70 \text{ g} \) HNO₃
- 0.9 mL H₂SO₄ \( \rightarrow \) \( m = (0.9 \text{mL})(1.84 \text{ g/mL}) = 1.65 \text{ g} \) H₂SO₄
- 0.47 mL Methyl Benzoate \( \rightarrow \) \( m = (0.42 \text{mL})(1.07 \text{ g/mL}) = 0.458 \text{ g} \) Methyl Benzoate

**Actual Mass:**

- 0.5 mL H₂SO₄: 0.927 g
- 0.5 mL HNO₃: 0.700 g
- 0.9 mL H₂SO₄: 1.655 g
- 0.47 mL Methyl Benzoate: 0.457 g

**After Adding Electrophile:** turned light yellow

**Mass of Extracted Product:** 0.775 g.

**Melting Point Range:** 60-66 °C.
TLC Plate Results:

<table>
<thead>
<tr>
<th>Spot</th>
<th>Distance (cm)</th>
<th>Rf Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>6.4</td>
<td>0.625</td>
</tr>
<tr>
<td>B</td>
<td>6.4</td>
<td>0.625</td>
</tr>
<tr>
<td>C</td>
<td>4.0</td>
<td>0.313</td>
</tr>
<tr>
<td>D</td>
<td>4.0</td>
<td>0.313</td>
</tr>
</tbody>
</table>

Production of Methyl-3-nitrotoke:

\[
\text{OCH}_3
\]

\[
\text{OCH}_3
\]

Results:

<table>
<thead>
<tr>
<th>Calculated Yield</th>
<th>0.458 g Methyl Borate</th>
<th>1 mol methylborate</th>
<th>1 mol Methyl-3-nitrotoke</th>
<th>1 mol Boric Acid</th>
<th>1 mol Methyl-3-nitrotoke</th>
</tr>
</thead>
<tbody>
<tr>
<td>Theoretical Yield</td>
<td>0.609 g Methyl-3-nitrotoke</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

% Yield Calculation:

\[
\% \text{ Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100\%
\]

\[
\text{Actual} = 0.275 \text{ g Methyl-3-nitrotoke}
\]

\[
\text{Theoretical} = 0.609 \text{ g Methyl-3-nitrotoke}
\]

\[
\text{Yield} = \frac{0.275 \text{ g Methyl-3-nitrotoke}}{0.609 \text{ g Methyl-3-nitrotoke}} \times 100\% = 45.27\% \text{ Yield}
\]
Analysis of Results:

Based on my results, I got a relatively pure product of methyl-3-nitrobenzoate. This can be seen when looking at the TLC plate. When comparing the Rf values for the starting material and the product, it is clear that a new compound was formed. The spots for the product (methyl-3-benzoate) are much closer to the starting line than that of the starting material (methyl-3-benzoate). This means that the product is much more polar and it didn't travel as far because of a higher attraction to the TLC plate. In determining the melting point of the product, I found it to be in between the range of 60-66°C. The actual melting point of methyl-3-benzoate is 78-79°C. My low melting point could have been a result of not letting the solid dry completely, causing it to melt faster. However, overall the M.P. is close to the actual M.P., and this reinforces my conclusion that I obtained a relatively pure product of methyl-3-benzoate. I ended up attaining a yield of 0.775 g, methyl-3-nitrobenzoate and this turned out to be a 68.45% yield.

Conclusion:

Overall, my results for the lab were good. I obtained a relatively pure sample of methyl-3-nitrobenzoate by nitro-phenyl methyl benzoate. My only regret was that I did not obtain in a very high % yield. This low % yield could have been caused by a number of errors in the laboratory techniques. I know I lost some of the product in the filtering process due to not being able to remove all of the solid from the filtration flask. I also lost some when trying to weigh out the yield of the solids. When transferring it to the weighing dish some fell onto the lab table. Another possibility is that I did not allow a long enough time for the run to take place, I also might have kept the run at the exact right temperature. Besides this low % yield, I do see slight errors in my lab technique. I did a good job of producing a pure sample of methyl-3-nitrobenzoate by using an electrophilic aromatic substitution reaction.
Electrophilic aromatic substitution: The effect of directing groups on benzene (SWH)

Beginning Questions:
1. What is the difference between activating and deactivating groups?
2. What is an electrophile and how does it react?
3. What effect do directing groups (functional groups) have?

The purpose of this lab is to understand resonance structures and the formal charges associated with them.

Data/Observations:
- Exconimation
- 1.073g of pyridinium tribromide, mass of starting material = 1.073g
- 1.271g = Mass of product
- Melting point of product = 160.7°C
- Melting point range = 161.0° - 163.3°C
- Percent yield = 70%

Molar Mass of product = 135.17 - 1.00 = 79.9 = 214.07g
Mol of starting material = 1.073g / 135.7 = 0.0079
14.07 x 0.0079 = 1.071g = Theoretical yield
1.271g = Exp. yield

% yield = Act/theor. x 100
C7H7Br x 100 = 76%
1.70

Claims:
As a result of our experiment, I was able to answer the beginning questions. The differences between activating groups and deactivating groups are that activating groups produce either ortho or para isomers, and deactivating groups produce meta isomers as products. The answer to my second question (What is an electrophile and how does it react?) is that an electrophile is a species that accepts electrons.

Also, we were able to see the effects a directing group (functional group) has. The functional group affects resonance structures which includes formal charges and where electrons are distributed on an aromatic ring.
Evidence: +5 Very insightful and well written!!

In the two different experimental procedures (Elevation and Nitration), we were able to gain information to determine what type of structure we produced. Heather and I performed a Bromination experiment. From our melting point data of our product and the known melting points of the three different structures given in the lab manual, we deduced that our product was the para product. Our melting point range was between 194°C–195°C. The given melting point in the lab manual reports para structures have a melting point of 197°C. Below is a picture of the para isomer we generated:

![Para Isomer](image)

The accuracy of our experiment is good with a percent yield calculated on the previous page of 75%.

This product is a result from a deactivating group because the nitrogen in the functional group has electrons available to donate to the ring. The functional group used in the Elevation was acetamide, methyl benzene, the functional group used in the Nitration experiment, on the other hand, was from a deactivating group producing a meta isomer because there was no electron pair attached to the ring to share.

Mechanisms:

**Bromination**:

\[ \text{[ortho]} \rightarrow \text{[para]} \rightarrow \text{[ortho]} \]

\[ \text{[ortho]} \rightarrow \text{[para]} \rightarrow \text{[ortho]} \]
This is the mechanism for the bromination experiment we performed. Here the bromine is the electrophile that replaces hydrogen giving the para product shown above. The mechanism for the nitration experiment performed by another group is on the next page.

**Evidence Cont.**

Nitration:

\[
\begin{align*}
& \text{Ar}^+ \quad \text{NO}_2^- \\
& \text{Ar}\text{CH}_2 \quad \text{Ar}\text{CH}_2 \\
& \text{H}_2\text{SO}_4 + \text{HNO}_3 = \text{NO}_2
\end{align*}
\]

In this mechanism of a nitration, the NO\textsubscript{2} is the electrophile that replaces hydrogen. This is a meta product coming from a deactivating group.

**Reading & Reflection: +5**

I researched aromatic compounds to learn more about what characterizes an aromatic compound. For example, an aromatic compound contains at least one ring with six carbon atoms. Each carbon bond is joined adjacent by a double and single bond. Also, the bonds in an aromatic ring are not as reactive as regular double bonds. Aromatic rings usually undergo electrophilic substitution instead of addition. Originally, aromatic compounds
were so-called this because they were once only associated with good-smelling chemicals from vegetables; aroma = an odor arising from spices, plants, cooking, etc., esp. an agreeable odor; fragrance; http://www.dictionary.com

References:

APPENDIX D: ASSESSMENT AND EVALUATION

Introductory Survey
Chem 231L

Section: ____________

Note: Questions 1 – 5 are based on a 5-point scale (5 – strongly agree, 4 – agree, 3 – neutral, 2 – disagree, 1 – strongly disagree). Please provide comments to your responses. Thank you!!

1. I would rather learn concepts in the laboratory before going to the lecture.
2. The laboratory should help me understand concepts that are covered in the lecture.
3. I expect my TAs to be effective teachers. Can you mention at least two traits you believe an effective teacher would have?
4. My past experience in the general chemistry laboratory should help me with this laboratory course.
5. The format of the laboratory report (claims, evidence, etc.) is something I have seen before. (If so, what comments do you have concerning it?)

**Questions 6 – 11 are open ended (No 5-point scale)**

6. On average, how much time do you expect to spend writing your laboratory report each week?
7. On average, how much time outside of class do you expect to spend each week studying for the lecture portion of the course?
8. Can you think of an experiment that you would like to do, or is there some topic that you think could be discussed in the laboratory that may be relevant to you?
9. What are your expectations for this laboratory course? Have you talked with someone who took it before?
10. Concerning your previous laboratory course(s), were they “practical”? Were the topics discussed and the experiments completed things you could relate to?

11. What is your primary reason for taking this course? (i.e. would you take this course if it wasn’t required?)

Mid-Term Evaluation

Chem 231L

Section: ___________

Note: Questions 1 – 4 are based on a 5-point scale (5 – strongly agree, 4 – agree, 3 – neutral, 2 – disagree, 1 – strongly disagree). Please provide comments to your responses. Thank you!!

1. The laboratory is helping me understand topics discussed in the lecture.

2. My TAs lead an effective and understandable pre-lab discussion. (Mention both TAs)

3. My TAs grade my laboratory reports fairly.

4. My TAs leave constructive comments on my reports if I don’t receive full credit.

** (Open Ended) **

5. Can you comment on the format of the laboratory report? (beginning questions, etc.) Does it help you organize and put together your report?

6. On average, how much time have you spent writing your lab report each week?

7. On average, how much time outside of class have you spent each week studying for the lecture portion of the course?

8. Of the experiments completed so far, is there one or two in particular that stand out as either fun to do or relevant to some topic you may already be familiar with?

9. How has this laboratory compared with the expectations you had for it at the beginning of the semester?
10. So far, how does this laboratory compare to any previous laboratory classes you have taken?

End of Semester Evaluation

Chem 231L

Section: __________

The following questions are in regard to the laboratory in general. Please provide comments to your responses.

1. Did the laboratory help you understand topics discussed in lecture?

2. Did the format of the lab report (beginning questions, etc.) help you organize and put together your report?

3. On average, how much time did you spend writing each lab report?

4. On average, how much time did you spend studying for the lecture? (per day/week)

5. How has this lab compared with the expectations you had for it at the beginning of the semester? (easier, more difficult, etc.)

6. Of the experiments completed, were there any in particular that stood out as either fun to do or relevant to some topic you may already have been familiar with?

7. Do you have any suggestions as to how the lab portion of this course could be improved?

End of Semester Evaluation for Lecture

1. Did you find the activities helpful in understanding the material?

2. Did group work seem more beneficial for understanding concepts, or would more lecturing been beneficial?

3. In terms of the Activity assignments, can you rate the level of:
   - Difficulty (5 – way too difficult, 3 – about right, 1 – way too easy)
• Lengthiness (5 – way too long, 3 – about right, 1 – way too short)

4. Was the 10 minutes spent prior to exams in groups beneficial?

5. If there were one thing you could change about this class, what would it be?
APPENDIX E: LABORATORY PRACTICAL EXAMINATION

Exam
Lab Practical Exam Name: ________________________
Chem 231L Section:_______________________

Unknown starting material (number or letter of unknown): _____________________

1. (10 pts) The first thing you will need to do is to identify what your starting material is.

   There are a variety of reagents and instruments you can use. Using all of this
   information, draw the structure of your starting material. Be sure to provide evidence to
   back up your structure.
   - Unknowns used: Acetophenone, 4-methylacetophenone, isobutyrophenone, 4-
     chlorobenzaldehyde, 2-chlorobenzaldehyde, cuminaldehyde
   - 5 points for correct structure; 3 points for partial credit (incorrect substitution pattern,
     correctly identify aldehyde or ketone). 5 points for evidence (¹H NMR and IR
     interpretation, Jones oxidation test, Lucas reagent or Schiff test)

2. (5 pts) Write the reaction scheme showing the product you expect to produce.

   Starting material NaBH₄
   \[ \text{CH}_3\text{OH} \rightarrow \text{Product} \]

   2 points 1 point 2 points

   - No double jeopardy. If the product is correct for their starting material, then 5 points
     can be given (they lose points in question 1 for an incorrect starting material)

3. (2 pts) Give a general outline of what you are going to do to complete the reaction. Be
   specific with amounts of reagents you are using. How are you going to identify your
   compounds?
   - 2 points for a brief procedure
4. (4 pts) Set up your reaction. After it is complete, set up a jar for TLC. What are you trying to find out by performing this test? Draw the plate below and explain what it tells you.

- 2 points for drawing the plate, indicating where the spots are. If they did not get a product, then they cannot do this. But they should explain what the test would have told them (polarity and/or purity). 2 points for explanation.

5. (8 pts) Based on your data, what is the structure of your product? Be sure to include all relevant evidence to support your claim.

- 4 points for the correct structure. This should match what they have drawn for a product in question 2.
- 4 points for evidence. Primarily from the IR they obtain (disappearance of carbonyl group, appearance of alcohol group) but they can also use the Jones oxidation test or the Lucas reagent.

6. (10 pts) Once you are certain of the identity of your product, calculate the theoretical yield and the % yield.

- 7 points for the calculation (2 points per step, 1 point for correct answer)
- 3 points for the percent (if no product obtained, they need to write 0%)

7. (4 pts) Using your % yield, spectral data, TLC data, melting point, etc., can you say anything about the purity of your product?

- Purity based on % yield (1), TLC (1), spectral data (1), anything else reasonable, i.e. melting point (1)

8. (5 pts) Draw out the mechanism showing how your product formed.

- Two step mechanism, 2 points per step; arrows pointing the correct direction (1)
9. (2 pts) Based on your data, do you believe you have a case to get the job? How would you convince Blake to use your compounds for his research and not ones someone else made?

- As long as what they say fits with the data they collected, give them 2 points. Just a yes or no answer receives zero credit.

**Materials List**

1. **Unknowns**
   - Any simple aldehydes or ketones. Students generally use 2.5 g of the unknown to experiment with

2. **Solvents and reagents**
   - Sodium borohydride
   - 100% ethanol
   - methanol
   - diethyl ether (extraction)
   - aqueous sodium chloride (work-up)
   - magnesium sulfate (drying agent)
   - 50:50 (volume percent) ethyl acetate / hexanes solution (for TLC)
   - methylene chloride (for use in IR spectometer if reagents or products are solid)
   - 2,4-dinitrophenylhydrazine (qualitative test to distinguish alcohols from carbonyls)
   - Chromium trioxide in sulfuric acid (qualitative test for alcohols and aldehyds; caution: chromium is toxic. This works best if the solution is diluted with acetone)
   - Lucas reagent (zinc chloride and HCl) (qualitative test for 1º, 2º, and 3º alcohols)
   - Schiff reagent (qualitative test for aldehydes)
   - 3 known alcohols (1º, 2º, 3º), 1 known aldehyde, acetone (for comparisons)

3. **Other Materials (in addition to standard chemical equipment)**
   - dry ice and 2-propanol (for rotary evaporator condensor)
   - wet ice
   - thermometer, TLC plates, TLC spotters, UV lamp
Starting Material A:

C₈H₈O
F.W. = 120.15 g/mol
m.p. = 19-20 °C
b.p. = 202 °C
d = 1.03 g/mL

Infrared Spectrum:

¹H NMR (CDCl₃): 2.58 ppm (3H), 7.32-7.68 ppm (3H), 7.94 ppm (2H)
Starting Material B:
C₉H₁₀O
F.W. = 134.18 g/mol
m.p. = 22-24 °C
b.p. = 226 °C
d = 1.00 g/mL

Infrared Spectrum:

\(^1\)H NMR (DMSO-\(d_6\)):

2.38 ppm (3H), 2.54 ppm (3H), 7.23 ppm (2H), 7.84 ppm (2H)
Starting Material C:
C_{10}H_{12}O
F.W. = 148.2 g/mole
b.p. = 217 °C
d = 0.99 g/mL

Infrared Spectrum:

\[\text{\textsuperscript{1}H NMR (CDCl\textsubscript{3})}:\]

1.22 ppm (6H), 3.54 ppm (1H), 7.31-7.67 ppm (3H), 7.95 ppm (2H)
Starting Material D:
C\textsubscript{7}H\textsubscript{5}OCl
F.W. = 140.57 g/mol
m.p. = 45-50 °C
b.p. = 213-214 °C

Infrared Spectrum:

\textsuperscript{1}H NMR (CDCl\textsubscript{3}):

7.52 ppm (2H), 7.82 ppm (2H), 9.99 ppm (1H)
Starting Material E:
C₇H₅OCl
F.W. = 140.57 g/mol
m.p. = 12.4-12.8 °C
b.p. = 211-212 °C
d = 1.25 g/mL

Infrared Spectrum:

¹H NMR (CDCl₃):

7.34 ppm (1H), 7.40 ppm (1H), 7.47 ppm (1H), 7.86 ppm (1H), 10.42 ppm (1H)
Starting Material F:
C_{10}H_{12}O
F.W. = 148.20 g/mol
b.p. = 235-236 °C
d = 0.98 g/mL

Infrared Spectrum:

\[^1H\text{ NMR (CDCl}_3\text{)}:

1.28 \text{ ppm (6H), 2.97 ppm (1H), 7.38 ppm (2H), 7.80 ppm (2H), 9.96 ppm (1H)}