Monthly Meeting
Dr. John Warner Speaks on “Green Chemistry: Sustainability and Innovation at the Molecular Level”

Women in Chemistry
By Mindy Levine

Summer Scholar Report
By Devon A. Heath and Leon J. Tilley

National Chemistry Week Report
By Christine Jaworek-Lopes
2009 Eastern Analytical Symposium
November 16 - 19, 2009
Garden State Exhibit Center, Somerset, New Jersey

CALL FOR PAPERS
Deadline – April 15, 2009

The Eastern Analytical Symposium and Exposition is the second largest conference and exposition for laboratory science in the U.S. dedicated to the needs of analytical chemists and those in the allied sciences. We offer high quality cutting-edge technical sessions and state-of-the-art short courses, workshops and seminars. We invite you to be a part of the program by contributing a paper for oral or poster consideration. Please note that all abstracts must be submitted electronically via the EAS web site at www.EAS.org. The abstract submission deadline is April 15.

To submit a contributed paper for the 2009 EAS Technical Program, please submit abstracts through our web site at www.EAS.org, between March 1 and April 15, and follow the instructions for abstract submission.

Invited speakers must not submit abstracts to EAS until requested.

Please carefully review the following information:

- All contributed abstracts must be submitted through our web site at www.EAS.org between March 1 and April 15, 2009. No faxed, e-mailed, or mailed abstracts will be accepted.
- Please note that no one author may submit and present more than two posters.
- All abstracts will be acknowledged via e-mail.
- The title of the presentation and the list of authors that you submit are final, and may not be changed.
- The abstract that you submit will be considered to be your final abstract that will be printed in the abstract book for the 2009 Eastern Analytical Symposium.
- Presenting authors of contributed submissions will be notified in June 2009 of the status of the abstract and its session assignment.

TOPIC AREAS

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Capillary Electrophoresis
Chromatography
Chromometrics
Chiral Analysis
Comprensive Methods
Conservation Science
Data Analysis/LIMS
Dissolution
Environmental Analysis/Pollutants
Food Analysis
Forensic Analysis
Gas Chromatography
HPLC
Immunochemistry
Industrial Hygiene
Ion Chromatography
IR Spectroscopy
Laboratory Automation
Laboratory Management
LC-MS
Mass Spectrometry
Microchemistry
Microscopy
Near Infrared (NIR)
NMR Spectroscopy
Organic Chemistry
Pharmaceutical Analysis
Pharmaceutical Impurities
Process Analytical Science
Quality by Design
Quality/Regulatory/Compliance
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Deadlines: May 2009 Issue: March 13, 2009
Summer 2009 Issue: June 16, 2009

The Nucleus is published monthly, except June and August, by the Northeastern Section of the American Chemical Society, Inc. Forms close for advertising on the 1st of the month of the preceding issue. Text must be received by the editor six weeks before the date of issue.

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Women in Chemistry

By Mindy Levine, Ph.D.,
Massachusetts Institute of Technology, Cambridge, MA

“There is no good time for any woman who is a professional chemist to have a child,” says Professor Richard N. Zare, chair of the Stanford University chemistry department, in a recent Chemical and Engineering News article.

Jennifer Schefiliti, a sixth year graduate student in the MIT chemistry department and former chair of the Graduate Women in Chemistry group, agrees. “I want to be actively involved in my children’s life,” she explains, “and I don’t see academic university life as being compatible with that.” Thus, Ms. Schefiliti has decided not to pursue a career in academia, instead planning to become a management consultant. In the field of management consulting, she will have the option of working part-time for a few years. She can take a break and get “back on track” afterwards. In short, she sees her career in management consulting as affording her a work-life balance that academic chemistry may not.

Nonetheless, Ms. Schefiliti’s decision to stay out of academia comes with regrets. Ms. Schefiliti spends a significant amount of time tutoring undergraduate women in their science studies, and she is amazed “to see how excited they get about science.” “I would love to be able to give back, to encourage more women to go into science,” she notes. “I would love a non-tenure track position where they just let me stay and do research.”

Work-life balance

Professor Christine Thomas of Brandeis University agrees that the greatest challenge for women in academia is trying to balance career and family responsibilities. “While this is a problem for women in all careers, it is particularly pronounced in academia because of the overlap of the tenure timeline with the time when society (and biology) dictate that women should be starting a family,” she says. While many universities now offer professors the option of taking time off of the tenure clock for having children, not everyone may feel comfortable availing herself of that option.

In any case, even with time off from teaching and committee work, the job of a chemistry professor is immensely time-consuming. As Professor Judith Herzfeld of Brandeis University explains, “You have to work more than a forty hour week to stay competitive...because grant funding is so tight.”

There are advantages to a career in academia. In academia, one has the option of working from home or bringing children to work. Professor Karen continued on page 6
ABSTRACT

The field of Green Chemistry is over 15 years old. There are many textbooks, journals and conferences dedicated to the subject. Most universities across the world have faculty that are integrating the principles of green chemistry into their curricula. Industry has formed external collaborative roundtables and hold routine internal workshops on the subject. Federal and state governments are creating programs and legislation to promote green chemistry as a solutions based approach to sustainability. While these policy efforts recognize the potential for Green Chemistry to protect human health and the environment, they also seek to leverage economic and workforce development as well as educational objectives. The fact that green chemistry has captured the attention of a diverse group of organizations that do not typically interact with the traditional chemistry infrastructure has provided many unique opportunities and challenges. This presentation will discuss the history and science of Green Chemistry as well as its relationship with the general society.
Contest Announcements

Chemists Celebrate Earth Day Haiku Contest

The Northeastern Section will participate in the Illustrated Haiku Contest as part of the Chemists Celebrate Earth Day celebration.

For detailed contest rules, please visit:
http://portal.acs.org/portal/acs/corg/content?_nfpb=true& pageLabel=PP_ARTICLEMAIN&node_id=1573&use_sec=false& uuid=1374c1eb-1268-4570-b1ff-012f383d497d

Please submit your entries by Friday, April 3, 2009 to:
Christine Jaworek-Lopes
Emmanuel College
400 The Fenway
Boston, MA 02115

Winning entries will be published in a future edition of the Nucleus. Authors of winning submissions will receive a CCED 2009 t-shirt and a gift certificate to one of the following: www.amazon.com, www.teachersource.com, or the ACS online store.

NCW 2009: Design a t-shirt contest

Would you like to design the National Chemistry Week 2009 t-shirt worn by all NESACS National Chemistry Week volunteers?

The winning design will be on the front of the t-shirt. The Northeastern Section of the American Chemical Society Logo and NCW 2009 will be on the back of the t-shirt. This contest is open to all students K-12 in the Northeastern Section.

Contest rules:
1. Your design must capture the NCW 2009 theme which is the chemistry of the elements.
2. You may use up to 4 colors in your design and your design must be on an unlined 8.5” x 11” sheet of paper.
3. The deadline for submission is May 1, 2009. The winner will be announced by June 1, 2009.
4. Please mail your original design to:
   Christine Jaworek-Lopes
   Emmanuel College
   400 The Fenway
   Boston, MA 02115
5. All entries must have the following information included with the entry: student’s name, grade, home address, telephone number, school name, school address, teacher’s name, email, and school telephone number.
6. Have fun!!!

Women in Chemistry

Continued from page 4

Allen of Boston University said sometimes she meets collaborators at her home when she is responsible for watching her children. Her collaborators are, in general, understanding. “Flexibility is the key,” she says.

However, a career in industry may still be seen as more family-friendly. Professor Herzfeld explains that having children is not trivial in any profession, but in industry, there is maternity leave. In academia, it is difficult to take a real maternity leave without falling behind in research. “Hour-to-hour there is flexibility in academia,” she says, since professors can take time off or work from home if necessary. However, due to the necessity of maintaining research momentum in the laboratory, there is not the same flexibility from year to year in academia as is available in other careers.

Other issues

Professor Penny Beuning of Northeastern University, and President of the local chapter of Sigma Delta Epsilon—Graduate Women in Science, explains that there are other issues that face women in academia, in addition to figuring out how to navigate a work-life balance successfully. In particular, female chemistry professors tend to serve on more committees than their male colleagues. This trend is due to the fact that the committees are designed to have a certain percentage of female faculty, to ensure that women’s voices are heard. Given that there are fewer women than men on the faculty, the women ultimately serve on more committees. Consequently, their research productivity may decline. This phenomenon, says Professor Beuning, is “the unintended consequence of good intentions.” Professor Linda Doerrer of Boston University concurs that “most women carry an extra burden of community service.”

Moreover, there may be subtle, even subconscious, sexism that women chemistry faculty experience. “Things that are taken seriously when a male

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Women in Chemistry
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colleague says it are ignored when a woman does,” observes Professor Herzfeld, “but I think it is getting better.” Professor Liz Hedstrom of Brandeis University believes that people who are ten years older had a much tougher time, but that the environment has gotten better. Professor Beuning adds that, “sometimes women are not automatically assumed to be competent.”

Proactive measures
One of the proactive measures taken to encourage more women to pursue careers in science is to reach out to them at a young age. Allison Wensley, a fourth year graduate student at Boston University and head of the Women in Chemistry (BUWIC) group explains that the group has begun to do outreach in high schools and hopes to soon extend their program to middle schools as well. “I think it is a valuable thing to reach young women at an earlier stage,” she declares. “Middle school is really the best time. We can get girls excited about science…and they realize that they can do it.”

Graduate women’s groups like BU’s Women in Chemistry may also help attract and retain women in chemistry. Karen Ruff, a graduate student at Harvard University and former chair of Harvard Women in Chemistry (HWIC) says that when HWIC started ten years ago, the graduate student body was only 5-10% female. Now, the number of female graduate students has increased to 30%. The goals of HWIC, Ms. Ruff explains, “are to keep more women in science, provide mentors and support, and make the environment better for everyone.”

Similarly, the Women in Chemistry group at MIT seeks “to promote healthy social interaction with women in the department,” Ms. Schefiliti explains. A few years ago at MIT, the number of women leaving graduate school before the end of their second year was substantially higher than the number of men leaving. As a result of

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Announcement
The NESACS Committee on Continuing Education will present Two Short Courses for Advanced Microsoft Excel® Users:

**Advanced Excel® for Scientists and Engineers**
A course that focuses on the ways to apply Excel® to scientific problems Thursday, April 23 & Friday, April 24, 2009, 8:30 AM – 4:00 PM

**Excel Visual Basic Macros for Scientists**
An introduction to programming using Excel’s Visual Basic Thursday, May 21 & Friday, May 22, 2009, 8:30 AM – 4:00 PM
Place: The Courtyard Marriott, 342 Speen Street, Natick, MA

**Instructor: Dr. E. Joseph Billo, Boston College**
Dr. Billo is the author of Excel® for Chemists, 2nd Ed., and Excel® for Scientists and Engineers: Numerical Methods, both published by J. Wiley and Sons.
He has taught these courses to over 2,000 scientists at locations including Amoco, Bayer, Chevron, Hercules, Kodak, Genzyme, National Cancer Institute, NIST, Procter & Gamble, Shell, Texaco, Unilever, and numerous others.
Details and registration forms will be in next month’s issue of The Nucleus.
For further information contact Dr. Billo at joseph.billo@verizon.net

The 13th Annual Andrew H. Weinberg Memorial Lecture

Lee J. Helman, M.D.
Scientific Director for Clinical Research
Center for Cancer Research
National Cancer Institute

“Targeting IGF signaling in sarcomas: past, present and future”

Tuesday, April 7th, 2009
4:00 pm – 5:00 pm

Dana 1620 Conference Room
Dana-Farber Cancer Institute

Additional information about the Weinberg Memorial Lecture will be published in the April Nucleus.
Robert Burns Woodward Memorial Symposium

Marking the 30th anniversary of his death

Sponsored by the Northeastern Section of the American Chemical Society
Co-sponsored by the Royal Society of Chemistry-US, Indus Entrepreneurs Group, Chemical Heritage Foundation and IUPAC

Thursday, April 9th, 2009
Symposium - 9:00 AM – 5 PM
Royal Sonesta Hotel, 40 Edwin Land Boulevard
Cambridge, MA 02142 (617) 806-4200

Speakers Will Include:
William Roush, Scripps Florida
David Dolphin, University of British Columbia
Paul Wender, Stanford University
Pat Conflaloe, Bristol-Myers Squibb
Peter Jacobi, Wesleyan University
Thomas Hoye, University of Minnesota
Robert Williams, Colorado State University

Symposium Registration fee: $50.00
Graduate Students/Post Docs: $25.00

Attendance is Limited – Advanced Registration is Required
Please send your registration form along with your check made payable to NESACS to
Marilou Cashman, Administrative Secretary, 23 Cottage Street, Natick, MA 01760
For information, contact any of the Organizing Committee
Chorghade@comcast.net  William.greenlee@spcorp.com
jv.heck@gmail.com

Followed by the Third Annual Advances in Chemical Sciences Symposium

Sponsored by the Northeastern Section of the ACS
Cosponsored by IUPAC and the Royal Society of Chemistry-US

Friday, April 10th, 2009
The James Flack Norris and Theodore William Richards Undergraduate Summer Research Scholarships

The Northeastern Section of the American Chemical Society (NESACS) established the James Flack Norris and Theodore William Richards Undergraduate Summer Scholarships to honor the memories of Professors Norris and Richards by promoting research interactions between undergraduate students and faculty.

Research awards of $3500 will be given for the summer of 2009. The student stipend is $3000 for a minimum commitment of ten weeks of full-time research work. The remaining $500 of the award can be spent on supplies, travel, and other items relevant to the student project.

Institutions whose student/faculty team receives a Norris/Richards Undergraduate Summer Research Scholarship are expected to contribute toward the support of the faculty members and to waive any student fees for summer research. Academic credit may be granted to the students at the discretion of the institutions.

Award winners are required to submit a report (~5-7 double-spaced pages including figures, tables, and bibliography) of their summer projects to the NESACS Education Committee by November 6, 2009 for publication in The Nucleus. They are also required to participate in the Northeast Student Chemistry Research Conference (NSCRC) in April 2010.

Eligibility: Applications will be accepted from student/faculty teams at colleges and universities within the Northeastern Section. The undergraduate student must be a chemistry, biochemistry, chemical engineering, or molecular biology major in good standing, and have completed at least two full years of college-level chemistry by Summer, 2009.

Application: Application forms are available on the NESACS web site at http://www.nesacs.org. Completed applications are to be submitted no later than April 3, 2009 to the Chair of the Selection Committee:

Professor Edwin Jahngen
University of Massachusetts Lowell
Chemistry Department, Room 520, Olney Hall
1 University Avenue
Lowell, MA 01854-5047

Notification: Applicants will be notified of the results by e-mail on April 24, 2009, with written confirmation to follow.

The NESACS website
Updated frequently • Late-breaking news • Position postings
Back issues of the Nucleus archived • Career-related Links • Awards and Scholarships
WWW.NESACS.ORG
In preparation for National Chemistry Week 2008, a volunteer preparation day was held at Emmanuel College on Saturday, October 4, 2008. More than 30 individuals attended this event, which allowed volunteers to practice the hands-on activities and demonstrations in advance of the October celebration. Staff members from the Museum of Science-Boston and the Boston Children’s Museum were on hand to choose which activities worked best for their respective venues.

On Saturday, October 18, 2008, the Northeastern Section of the American Chemical Society sponsored a National Chemistry Week 2008 Kick-Off Event at Museum of Science-Boston (MoS). More than 45 volunteers (from ACS, the Brauner Committee, Emmanuel College, Malden High School, Stonehill College, Suffolk University, Tufts University) ensured that the more than 450 visitors to the daylong event enjoyed a number of hands-on activities. Among the highlights of the day were the two Phyllis A. Brauner Memorial lectures, presented by Dr. Bassam Shakhashiri, Professor of Chemistry at the University of Wisconsin-Madison. These captivating lectures were enjoyed by children and adults alike. Approximately 400 individuals attended these lectures.

Five hundred and ninety-three students attended the High School Science Series event at the MoS-Boston on October 24, 2008. The students were from: Abington High School (HS), Advocates for Home Education in MA, AOK (Home schoolers in Cambridge), Arlington HS, Codman Academy Charter Public School, East Boston High School, Boston Day and Evening Academy, John D O’Bryant School, Greater Lowell Technical HS, Malden HS, Old Rochester Regional HS, Parkway Academy of Technology and Health, St. Joseph High School Open Bible Academy. These students participated in a number of hands-on activities and demonstrations related to the yearly theme. In addition, the students attended a lecture-demonstration given by David Sittenfeld, MoS-Boston, and Patrick Drane, Baseball Research Center at UMass-Lowell regarding materials used in sports. Admission for all students was covered by a generous donation from Creagen Biosciences, Inc. and the MoS-Boston. The students also had the opportunity to participate in the High School Series Optional Chemistry Problems. The questions were based on two of the activities in which the students participated. July Merizier and Janeisa Lashley of John D. O’Bryant School of
Mathematics and Science correctly solved the questions and each received a $25.00 gift certificate to www.amazon.com and a National Chemistry Week t-shirt.

In addition, an NCW event was held at the Boston Children’s Museum on Saturday, October 25, 2008. Approximately 500 individuals participated in NCW hands-on activities and demonstrations. A particular favorite at the Boston Children’s Museum was making bouncing balls. More than 30 volunteers from Creagen Biosciences, Inc., Emmanuel College, Gordon College, Merck Research Laboratories-Boston, Pingree School, Suffolk University, and Tufts University were available to assist visitors throughout the day.

The activities and demonstrations that were performed throughout the week included: determining the starch content in foods, learning about the sugar content of a variety of foods, UV beads and sunscreen, make your own bouncing balls, make your own Gatorade, learning about electrolytes, water resistant materials, and the chemistry of hot and cold packs.

Children in grades K-12 were able to participate in the national poster competition. The winning poster from the NESACS was submitted by Jordan Sheehan from Bishop Guertin High School. Children in grades K-12 had the option of participating in two puzzle contests. The puzzles were designed by Dr. Christopher Morse. Drew Fuchs from Tantasqua Regional High School was the winner of the Element Sudoku. Alex Frezza from Attleboro High School was the winner of the Sports Word Square.

More than 100 pairs of used sneakers were collected at Emmanuel College, Hugs Plus II, and Odyssey Day School, as part of the NCW Sneaker Recycling Event. For more information about sneaker recycling, please see: http://www.nikereuseashoe.com/

Special thanks to all of our volunteers, Boston Children’s Museum, Lynn Baum, Creagen Biosciences Inc., Alissa Daniels, Patrick Drane, Meghan Moriarty, Museum of Science-Boston, Nina Nolan, Northeastern Section of the American Chemical Society, David Sittenfeld, Dr. Bassam Shakhashiri, Strem Chemicals, and the Phyllis A. Brauner Memorial Lecture Committee.

The theme for NCW 2009 is “Chemistry – It’s Elemental,” to be celebrated from October 18-24, 2009.

Francis Humphreys Storer 1832-1914

Francis Humphreys Storer, agricultural chemist and first professor of chemistry at the Massachusetts Institute of Technology, received the Harvard B.S. degree on examination in 1855 after one year at the Lawrence Scientific School, two years as assistant to Professor Josiah Parsons Cooke, Jr., and a year at sea as chemist to the United States North Pacific Exploring Expedition. Thereafter he studied in Europe

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SUMMER SCHOLAR REPORT

Electron-deficient γ-silyl Systems: Methods for Synthesis of 1,1,1-trifluoromethyl-4-trimethylsilyl-2-butanone

Devon A. Heath and Leon J. Tilley, Department of Chemistry, Stonehill College, Easton, MA 02357

Abstract

The presence of a γ-silyl substituent is known to increase the stabilization of carbocation intermediates during solvolysis reactions of secondary compounds by way of a bridged “percaudal” ion; this effect is reduced or absent in tertiary alkyl systems. As an effort to increase the synthetic utility of this reaction and to study further mechanistic ramifications, we have been investigating this effect on a tertiary system destabilized by an electron-withdrawing trifluoromethyl substituent. Rate and product studies previously conducted in our laboratory have demonstrated a large γ-silyl bridging effect. We wished to further characterize the extent of bridging by measuring the β-deuterium kinetic isotope effect, which requires the synthesis of the corresponding isotopically labeled triflate 2c. We investigated several routes to the ketone 10, a key intermediate in this synthesis, including oxidation of alcohol 9 and nucleophilic trifluoromethylation of ester 6, with the main focus being on the latter route.

On reaction with (trifluoromethyl)trimethylsilane and tetrabutylammonium fluoride, the initial CF₃ group is transferred to the ester, producing a deprotonated hemiacetal, which proceeds to the transfer of additional trifluoromethyl substituents. While the correct ketone was achieved, several side reactions, including formation of the silyl enol ether 15, and double addition product 14, have thus far precluded trifluoromethylation as a viable method for synthesis of 10.

Background

The electropositive nature of silicon has an influence on solvolysis reactions involving carbocations. The nature of this effect depends upon whether or not the silicon is in the α, β, or γ position. ¹,² Secondary γ-silyl systems have been found to exhibit stabilization by way of “percaudal participation,” in which the back lobe of the silicon carbon bond overlaps with the developing carbocation p-orbital, as shown in Figure 1. This effect leads to increased rates of solvolysis relative to carbon analogs and the formation of cyclopropanes through trimethylsilyl group elimination. Moreover, an inverse β-deuterium kinetic isotope effect is observed. The conformation required for bridging removes hyperconjugation, leading to an inductive effect and slight rate acceleration compared to the hydrogen analogue. ¹,²

A study of the tertiary system 1, however, showed no or little evidence of the gamma-silyl effect. ¹,² Consequently, our group decided to investigate the solvolysis of 2a, in which the strongly destabilizing CF₃ group would be expected to enhance the bridging effect of the silyl group. Indeed, rate constant studies have shown a 200-fold increase compared to the carbon analog 2b, and product studies have shown cyclopropane 3 to be almost the exclusive product. ⁴ In addition to academic interest, this reaction may be of use synthetically for cyclopropane formation.

In order to confirm percaudal participation in this tertiary system and complete the mechanistic picture, we wished to synthesize and study the β-deuterium kinetic isotope effect for the solvolysis of system 2c. A key intermediate in the synthesis of this system is the trifluoromethyl ketone 10, which could be deuterated by exchange with D₂O, then

![Figure 1: Percaudal Interactions](image-url)

![Scheme 1: Deuteration](image-url)

![Scheme 2: Schematic Representation of Reactions](image-url)
reacted with methyllithium to yield alcohol 17, which yields 2c on reaction with triflic anhydride, as shown in Scheme 1.

Scheme 3: Ruthenium Catalyst

Synthesis

Several routes to the synthesis of 10 can be envisioned as seen in Scheme 2, including oxidation5, 6, 7, 8; Claisen condensation, alkylation followed by decarboxylation6, 8 or nucleophilic trifluoromethylation9, 10, 11. Prior to this summer, our research group had investigated a number of these methods. Alkylation of 8 with (iodomethyl) trimethylsilane proved unsuccessful due to the poor nucleophilicity of the enolate ion. Claisen condensation of 6 with ethyl trifluoroacetate appeared to form some of the acetoacetate 7, but decarboxylation required harshly acidic conditions, leading to decomposition. Oxidation of 9 with alkaline permanganate was not reproducible. While oxidation using Dess-Martin periodinane5 was successful on a small scale, the shock sensitive nature and high cost of this reagent led us to search for a more practical, economical synthesis. Some previous work had also been done in our laboratory on the trifluoromethylation reaction. This method appeared promising but did not completely convert the reactants to product. Consequently, the focus of our group this summer was twofold: to investigate other possible oxidizing agents for the oxidation of 9 to 10 and to further investigate conditions which would improve the trifluoromethylation of 6 to 10.

Oxidative Methods

One alternative oxidizing agent for 9 that was investigated was the ruthenium catalyst 5, shown in Scheme 3, which has been reported to oxidize trifluoromethyl alcohols using sodium periodate as a co-oxidant.7, 12 We were able to successfully prepare the ligand13 4, but attempts to prepare 18 by different procedures failed to produce a product which could be definitively characterized.14, 15

As an alternative, we decided to reinvestigate the use of permanganate in both aqueous and non-aqueous media, including 18-crown-6 in benzene.16 Oxidation of the aryl trifluoromethyl alcohol 19 was found to be complete within six hours by GC/MS, but was unsuccessful with compound 9.

Scheme 4: Trifluoromethylation

Table 1: Summary of Experiments

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Initiator</th>
<th>Products 6a</th>
<th>SM</th>
<th>Temp °C</th>
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<td>TBAF</td>
<td>15</td>
<td>42</td>
<td>23</td>
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<tr>
<td>THF</td>
<td>CsF</td>
<td>NA</td>
<td>23</td>
<td>80 to RT</td>
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<td>CH3(CF3)</td>
<td>TBAF</td>
<td>NA</td>
<td>23</td>
<td>80 to RT</td>
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</tr>
</tbody>
</table>

Trifluoromethylation

By far, the most thoroughly investigated reaction this summer was that of the nucleophilic trifluoromethylation of ethyl 3-trimethylsilylpropionate 6a. (Trifluoromethyl) trimethylsilane is well known for adding a CF3 equivalent to aldehydes and ketones.9 Ideally, the product would be envisioned by Pathway 1 of Scheme 4. The initiator, either fluoride or tert-butoxide, attacks the Me3SiCF3, which transfers a trifluoromethyl group to the ester, generating a deprotonated hemiketal 11, which reacts with additional molecules of Me3SiCF3 to produce 12 and continue the chain reaction. Cleavage of the resulting silyl ether 12 with acid or aqueous fluoride would lead to the desired ketone via the hemiketal.

Two problems with this reaction are the unreactivity of esters toward nucleophilic trifluoromethylation, as well as the potential for double addition if the reaction does occur.9, 10, 11 In this case, 11 eliminates ethoxide during the course of the reaction, prematurely forming 10, which can add a second equivalent of CF3. The mechanism of such addition continued on page 15.
March Historical Events in Chemistry

by Leopold May, The Catholic University of America, Washington, DC

March 1, 1896
Antoine Henri Becquerel discovered the radioactivity of uranite in pitchblende on this day.

March 3, 1709
Three hundred years ago, Andreas S. Marggraf was born. He isolated zinc from calamine; distinguished between potash and soda by flame test; found alumina in clay; and discovered beet sugar in beetroot.

March 3, 1918
Fifty years ago, Arthur Kornberg shared the Nobel Prize in Physiology or Medicine with Severo Ochoa for their discovery of the mechanisms in the biological synthesis of ribonucleic acid and deoxyribonucleic acid. He was born on this date.

March 6, 1869
Aleksei E. Favorskii, a researcher in the anionic rearrangements of acetylenes and α-haloketones, was born on this date.

March 10, 1824
One hundred and fifty years ago, Gustav R. Kirchhoff invented the spectroscope with Robert Bunsen, with which they discovered cesium (Cs, 55) in 1860 and rubidium (Rb, 37) in 1861. He was born on this date and discovered that substances, which emit radiation, absorb the same type of radiation when cool (Kirchhoff’s Law).

March 12, 1834
One hundred and fifty years ago on this date, Hermann W. Vogel was born. He invented the orthochromatic photographic plate in 1873; designed a photometer; and was a researcher in spectroscopic photography.

March 16, 1834
One hundred and fifty years ago on this date, Hermann W. Vogel was born. He invented the orthochromatic photographic plate in 1873; designed a photometer; and was a researcher in spectroscopic photography.

March 19, 1900
Seventy-five years ago, Frédéric J. Joliot (Joliot-Curie), H. Haiban and L. W. Kowarski proved experimentally that neutron emission occurs in nuclear fission. In 1935, Joliot shared the Nobel Prize in Chemistry with his wife Irène Joliot-Curie for production of artificial radioisotopes. He was born on this date.

March 19, 1984
Twenty-five years ago on this date, the ten millionth CA Abstract was published in volume 100, issue number 12 of Chemical Abstracts.

March 20, 1834
One hundred and seventy-five years ago, Charles W. Eliot, a teacher of chemistry and president of Harvard University, was born.

March 24, 1884
One hundred and twenty-five years ago, Peter Joseph William Deby was born. He was a researcher in dipole moments and the powder method of x-ray diffraction. He was awarded the Nobel Prize in Chemistry in 1936 for his contributions to our knowledge of molecular structure through his investigations on dipole moments and on the diffraction of X-rays and electrons in gases.

March 31, 1811
One hundred and fifty years ago, Robert Bunsen invented the spectroscope with Gustav R. Kirchhoff, with which they discovered cesium (Cs, 55) in 1860, and rubidium (Rb, 37) in 1861. He was born on this date and invented the Bunsen burner, filter pump, a galvanic battery, and with Henry E. Roscoe, the actinometer.

Notable NE Chemists

Charles William Eliot
1834-1926

Charles William Eliot was born in Boston, Mass. His whole life was spent in New England, chiefly in Cambridge. Eliot entered Harvard University in 1849. Chemistry, at that time was taught in the Medical School, but not in the College. Eliot was fortunate, however, in being admitted to the Chemistry and of Qualitative Analysis, which were the first textbooks of their kind, the joint production of two co-authors. These books went through many editions before the end of the nineteenth century. In 1870 Storer became Professor of Agricultural Chemistry at the newly founded Bussey Institution of Harvard University. In 1871 he assumed the additional duties of Dean and continued to occupy both positions until 1907. Storer’s “First Outlines of a Dictionary of Solubilities of Chemical Substances,” published in 1864, is the first dictionary of solubilities in any language. His “Agriculture in some of its Relations with Chemistry,” first published in 1887, was the fruit of his leisure and of his lectures at the Bussey Institution.

Additional historical events can be found at Dr. May’s website, http://faculty.cua.edu/may/Chemistrycalendar.htm.
is similar to what occurs when a Grignard reagent is added to an ester and is seen in Pathway 2 of Scheme 4.

These problems had reportedly been circumvented by the use of polar aprotic solvents under very dry conditions, or the use of no solvent at all with a cesium fluoride initiator. As a result, we investigated this reaction using a variety of solvents and initiators, as shown in Table 1. GC/MS was used to determine relative amounts of product from each reaction.

We did indeed encounter problems with both double addition and low reactivity for the starting material. In cases where double addition was not observed, the reaction took place to only a very small extent. Attempts to force completion using excess Me₃SiCF₃ also led to more double addition product. In the best case, a 1:1 ratio of the Me₃SiCF₃ to the ester addition product. In the best case, a 1:1 ratio of the Me₃SiCF₃ also led to more double addition only a very small extent. Attempts to observed, the reaction took place to cases where double addition was not reactivity for the starting material. In

An additional problem we encountered was the formation of silyl enol ethers, wherein the initiator functioned as a base rather than a nucleophile leading to formation of 15, as seen in Pathway 3 of Scheme 4. Upon hydrolysis of 15, starting material is regenerated.

As a last attempt to increase reactivity, we used the trifluororoethyl ester 6b instead of the ethyl ester. This compound was much more reactive, but double addition was also significantly increased.

**Conclusion**

The synthesis of the ketone 1,1,1-trifluoromethyl-4-trimethylsilyl-2-butanol was envisioned and attempted through several methods. Trifluoromethylation appeared to be promising, but competition between the low reactivity of the starting material and double addition in more reactive conditions made synthesis of the desired ketone difficult. In the future, the Dess-Martin reagent will be revisited for the oxidation of 1,1,1-trifluoromethyl-4-trimethylsilyl-2-butanol. Additionally, acetonitrile will be applied in the trifluoromethylation reaction as an alternative solvent. A silylating agent, such as hexamethyldisiloxane, might also be utilized to trap the anion before it eliminates the ethoxide group and forms the ketone. The successful production of the ruthenium catalyst will also be pursued. Once obtained, the resulting ketone 10 can be isotopically labeled and used to synthesize 2c for solvolysis studies.

**Acknowledgements**

Devon Heath would like to thank Professor Leon Tilley for his mentorship, intellectual contribution, and extreme patience while guiding her work. Also, she would like to acknowledge the other members of her research team, the S.U.R.E. (Stonehill Undergraduate Research Experience) program, Stonehill College, and the Northeastern Section of the American Chemical Society, specifically the Norris Richards Award, for funding and supporting this endeavor.

**References**


**Summer Scholar**

Continued from page 13

**Nesacs Candidates for 2009**

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Patrick Gordon
Liming Shao

**Secretary (vote for 1)**

Michael Singer

**Trustee (vote for 1)**

Joe Lima
Peter Meltzer

**Councilor/Alternate (vote for 10)**

Catherine Costello
Pam Mabrouk
Michaelene Chen
Dorothy Phillips
Amy Tapper
Jerry Jasinski
Ed Brush
Ira Krull
Ruth Tanner
Norton Peet
Raj Rajur
Mick Hurrey
Gary Weisman
Marietta Schwartz

**Dean-at-Large (vote for 2)**

James Phillips
Alfred Viola

**Nominating Committee (vote for 2)**

Michael Filosa
Chris Moreton
Ralph Scannell

**Esselen Award Committee (for 2)**

Anna Strome
Howard Mayne
Shainaz Landge

**Richards Medal Committee (for 2)**

Pam Mabrouk
Ira Krull
Jeff Steinfield

**Candidates**

Notable NE Chemists

Continued from page 14

small private chemistry laboratory of Professor Josiah Parsons Cooke, Jr., who had just begun his life work at the College. Eliot studied chemistry and mineralogy for nearly four years as an undergraduate. After graduation he continued his studies at Harvard and took his A.M. in 1856. He also assisted Professor Cooke at the Cambridge laboratory and later at the Medical School in Boston. From 1858 to 1861 Eliot gave instruction in mathematics and chemistry, and from 1861 to 1863 he was Assistant Professor of Chemistry and had charge of the laboratory of the Lawrence Scientific School. During this later period he conducted several investigations in analytical chemistry, some alone and some jointly with Francis H. Storer. From 1863 to 1865 he studied chemistry and investigated educational methods in Europe. While in Vienna, he was offered a position as Professor of Chemistry and Metallurgy...
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Mar 2
The Pfizer Symposium
Timothy Jamison (Massachusetts Institute of Technology)
TBA
Harvard Univ., Pfizer Lecture Hall 4:00 pm

Mar 3
Prof. Daniel Rabinovich (Univ. North Carolina, Charlotte)
“Hooked on Sulfur Ligands: Novel Complexes and Unexpected Applications”
Tufts Univ., Pearson P106 4:30 pm
Prof. Art Greenberg (UNH)
“Fifty Years That Transformed Organic Chemistry from a ‘Primeval Forest’ to a Modern Science V”
UNH Iddles, Room L103 11:10 am

Mar 4
Dan Yang,(University of Hong Kong)
TBA
Harvard Univ. Pfizer Lecture Hall 4:00 pm
Dr. Pinghua Liu (Boston Univ.)
“Isoprenoid biosynthesis: mechanistic studies and metabolic profiling.”
UMass Dartmouth, Building Group II, Room 115 4:00 pm

Mar 5
Dan Yang (University of Hong Kong)
TBA
MIT, Room 6-120 4:00 pm

Mar 6
Prof. Dan Yang (University of Hong Kong)
TBD
Boston College, Merkert 130 4:00 pm

Mar 9
James Chen (Stanford University)
“Chemical Probes of Embryonic Signaling and Patterning”
Pfizer Lecture Hall
4:00 pm to 5:00 pm
Dianne Newman (MIT)
Brandeis University, Gerstenzang 122 3:45 pm

Mar 10
Professor Jeffrey Katz (Colby College)
UNH Iddles, Room L103
11:10 am
Prof. Jin K. Montclare (Polytechnic Institute of Technology)

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Nathan Lewis (California Institute of Technology)
TBA
Harvard Univ., Pfizer Lecture Hall 12:30 pm
Wonwoo Nam (Ewha Woman’s University, Korea)
TBA
MIT, Room 6-120 4:00 pm
Dr. Matthias Brewer (Univ. Vermont)
“Hydrazones and Tethered Aldehyde Ynoates: Structurally Simple Precursors of Polycyclic Nitrogen-Containing Heterocycles”
UMass Dartmouth, Building Group II, Room 115 4:00 pm

Mar 12
Prof. Jin Montclare (Polytechnic Institute of NYU)
“Biosnippet Macromolecules: Protein Catalyststo Materials”
Boston College, Merkert 130 4:00 pm
Abbott Lecture in Organic Chemistry:
Michael T. Cinninos (University of North Carolina at Chapel Hill)
Title: TBA
Kevin Cusack (Abbott Biorsearch Center)
Title: TBA
MIT, Room, 6-120 4:00 pm

Mar 16
Kai Johnsson, (Swiss Federal Institute, Lucerne)
TBA
Harvard Univ. Pfizer Lecture Hall 4:00 pm
James Morken (Boston College)
Brandeis University, Gerstenzang 122 3:45 pm

Mar 18
John Hartwig (University of Illinois)
TBA
MIT, Room, 6-120 4:00 pm
Klaus Mullen (Max-Planck Institute for Polymer Research)
“Molecular Electronics”
MIT, Room, 6-120 4:00 PM

Mar 24
Prof. Christine Thomas (Brandeis Univ.)
UNH, Iddles, Room L103 11:10 am

Mar. 25
Dr. Xudong Yao (University of Connecticut, Storrs)
“Chemical control of peptide fragmentation for mass spectrometry”
UMass Dartmouth, Building Group II, Room 115 4:00 pm

Mar 31
Prof. Josef Michl (University of Colorado)
“From Molecular Rotors to Molecular Bubbles”
Tufts Univ., Pearson P106 4:30 pm

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Notable NE Chemists
Continued from page 16

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“Bio-Inspired Macromolecules through Protein Engineering”
Tufts Univ., Pearson P106 4:30 pm