

THE NUCLEUS

March 2009

Vol. LXXXVII, No. 7

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

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Please carefully review the following information:

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- 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.

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Office: Marilou Cashman, 23 Cottage St., Natick, MA 01360. 1-800-872-2054 (Voice or FAX) or 508-653-6329.

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Editor: Michael P. Filosa, Ph.D., ZINK Imaging, Inc., 16 Crosby Drive, Building 4G, Bedford, MA 01730 Email: Michael.filosa(at)zink.com; Tel: 508-843-9070

Associate Editors: Myron S. Simon, 20 Somerset Rd., W. Newton, MA 02465, Tel: 617-332-5273
Sheila E Rodman, Konarka Technologies, Inc., 116 John St. Suite 12, Lowell, MA 01852 email: srodman(at)konarka.com tel 978-569-1414
Stefan G. Koenig, Mindy Levine

Assistant Editors: David Cunningham (Chair), Mary Mahaney, Vivian K. Walworth

Board of Publications: Karen Piper, 19 Mill Rd., Harvard, MA 01451, Tel: 978-456-8622

Business Manager: Vincent J. Gale, P.O. Box 1150, Marshfield, MA 02050, Tel: 781-837-0424

Advertising Manager: Morton Hoffman, Feature Editor; Dennis Sardella, Book Reviews

Calendar Coordinator: Sheila Rodman, email: srodman(at)konarka.com

Photographers: Morton Z. Hoffman and James Phillips

Proofreaders: Donald O. Rickter, Vivian K. Walworth, Mindy Levine

Webmaster: David Cunningham, webmaster(at)nesacs.org

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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 univer-

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sities 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

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Monthly Meeting

The 895th Meeting of the Northeastern Section of the American Chemical Society

Thursday – March 12, 2009

Courtyard Boston-Cambridge,

777 Memorial Drive, Cambridge, MA 02139,
(617) 492-7777

4:30 pm Board Meeting

5:30 pm Social Hour

6:30 pm Dinner

7:45 pm Evening Lecture, NESACS Chair, E. Joseph Billo, Presiding

Speaker: John C. Warner, President and Chief Technology Officer,
Warner Babcock Institute for Green Chemistry,
President, The Beyond Benign Foundation,
Wilmington, Massachusetts

Title: *Green Chemistry: Sustainability and Innovation at the Molecular Level*

Dinner reservations should be made no later than noon, Thursday, March 5, 2009. Please call or fax Marilou Cashman at 800-872-2054 or e-mail at Mcash0953(at)aol.com. Please specify vegetarian. Reservations not cancelled at least 24 hours in advance must be paid. Members, \$30; Non-members, \$35; Retirees, \$20; Students, \$10.

THE PUBLIC IS INVITED

Directions:

Public Transportation: Take MBTA red line to Central Square Station. Walk 0.5 mile down River Street to Memorial Drive. The hotel is a short distance to the left. A shuttle will be available to take people back to the T stop at the end of the meeting.

Anyone who needs special services or transportation, please call Marilou Cashman a few days in advance so that suitable arrangements can be made. ◇

Biography

John Warner received his BS in Chemistry from UMass Boston and his M.S. and Ph.D. in Organic Chemistry from Princeton University. He worked at the Polaroid Corporation in exploratory research and media research for 10 years. In 1997 he accepted a position at the University of Massachusetts (Chemistry, Boston Campus and Plastics Engineering, Lowell Campus), where he helped start the world's first Green Chemistry Ph.D. program. John is now President and Chief Technology Officer of the Warner Babcock Institute for Green Chemistry (a research and development laboratory designing industrial solutions) and the Beyond Benign Foundation (a non-profit group dedicated to promoting Green Chemistry education and outreach). He has published nearly 150 patents, papers and books and is co-author of *Green Chemistry: Theory and Practice*. His recent patents in the fields of semiconductor design, biodegradable plastics, personal care products and polymeric photoresists are examples of how green chemistry principles can be immediately incorporated into commercially relevant applications. He received *The 2004 Presidential Award for Excellence in Science Mentoring* from President Bush, the American Institute of Chemistry's Northeast Division's *Distinguished Chemist of the Year* for 2002, and the Council of Science Society President's *2008 Leadership award*. Warner is editor of *Green Chemistry Letters and Reviews* and associate editor of the journal *Organic Preparations and Procedures International*. Warner serves on the Board of Directors of the ACS Green Chemistry Institute in Washington DC and is chair of California's science advisory board for Green Chemistry and Chemicals Policy Initiative. ◇

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. ◇

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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
400 The Fenway
Emmanuel College, 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.

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Royal Sonesta Hotel, 40 Edwin Land Boulevard
Cambridge, MA 02142 (617) 806-4200

Speakers Will Include:

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David Dolphin, University of British Columbia
Paul Wender, Stanford University
Pat Confalone, 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

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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

Women in Chemistry

Continued from page 7

the efforts of the Women in Chemistry group, Ms. Schefiliti says, that number has decreased dramatically.

Women's colleges can also create a supportive environment for women. Students at these colleges can receive more encouragement to pursue their career aspirations, including careers in fields where women are the minority. Professor Doerrer, who previously was a professor at Barnard College, says, "I think they (women's colleges) play an invaluable role in creating a steady state of women who go out into the world with the attitude that they can do anything." Professor Herzfeld, who attended Barnard College, agrees that at Barnard, "we were taught to believe we could be anything we want."

Concluding thoughts

When asked whether she has seen resentment from male colleagues regarding efforts to promote and hire women faculty, Professor Allen says, "There will always be people who find that they want to go back to what is familiar and comfortable...but everyone understands that this is what we need to ensure a diverse, creative, multi-tasking department of chemistry...that meets the needs of students."

Others agreed that any resentment only makes women's work more important. "That's why I think it is so important for people to get more women faculty, so that the next generation of graduate students will take women faculty seriously," Ms. Ruff said.

Professor Beuning agreed, "When you make things better for women, you make things better for everyone."

Author's note: I understand that everyone who works in chemistry is exceptionally busy. I am extremely grateful to the graduate students and professors who took the time to talk with me about the issues discussed in this article. Thank you for all of your advice and encouragement. ◇

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. ◇

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National Chemistry Week 2008 Report

The Northeastern Section Celebrates the Chemistry of Sports

By Christine Jaworek-Lopes, Emmanuel College, Boston, MA

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 Shakhshiri, 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

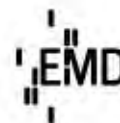
School, Greater Lowell Technical HS, Malden HS, Old Rochester Regional HS, Parkway Academy of Technology and Health, St. Joseph High School Open Bible Academy. These students participated in a number of hands-on activities and demonstrations related to the yearly theme. In addition, the students attended 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

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Ian Schwartz and Lori Conway (Head of Odyssey Day School) Photo by: Brian Carpenter

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 Shakhshiri, 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. ♦

Notable New England Chemists

We continue to present here biographies of New England chemists written by Lyman G. Newell of BU and Tenney L. Davis of M.I.T. for the guidebook for the 76th ACS Meeting held in September 1928 in Swampscott, Massachusetts. Transcribed for the Nucleus by M. S. Simon.

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 CF_3 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**.

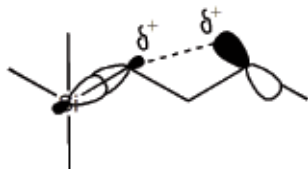
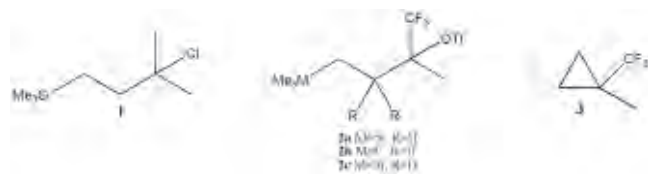


Figure 1: Percaudal Interactions



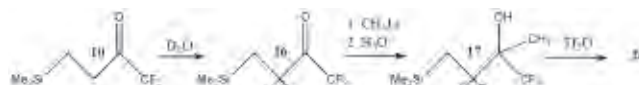
Background

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

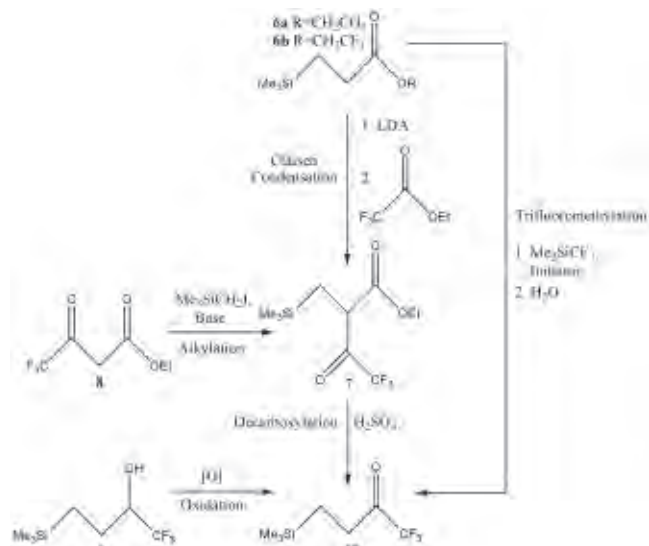
overlaps with the developing carbocation p-orbital, as shown in Figure 1. This effect leads to increased rates of solvolysis relative to carbon analogs and the formation of cyclopropanes through trimethylsilyl group elimination. Moreover, an inverse β -deuterium kinetic isotope effect is observed. The 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 destabilizing³ CF_3 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 D_2O , then

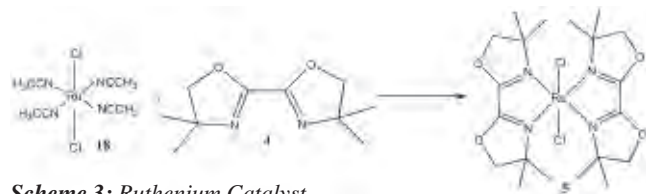


Scheme 1: Deuteration

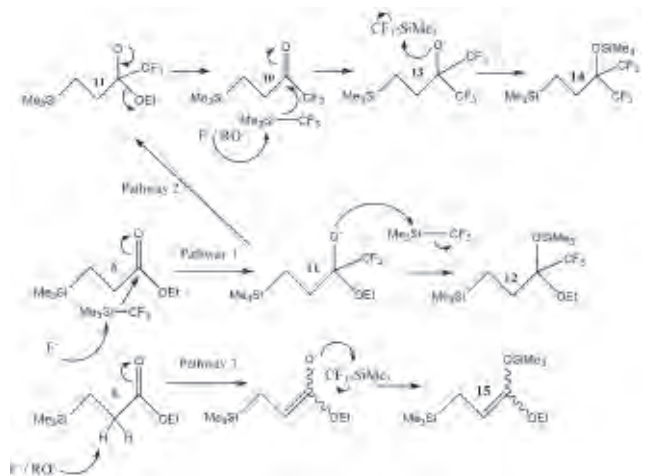


Scheme 2: Schematic Representation of Reactions

reacted with methyl lithium to yield alcohol **17**, which yields **2c** on reaction with triflic anhydride, as shown in Scheme 1.



Scheme 3: Ruthenium Catalyst



Scheme 4: Trifluoromethylation

Synthesis

Several routes to the synthesis of **10** can be envisioned as seen in Scheme 2, including oxidation^{5, 6, 7, 8} Claisen condensation, alkylation followed by decarboxylation^{6, 8} or nucleophilic trifluoromethylation.^{9, 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**.

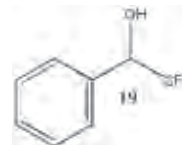


Table 1: Summary of Experiments

Solvent	Initiator	Products				SM	Temp °C
		15	12	14	10		
THF	TBAF	1%	4%	30%	42%	23%	-80 to RT
THF	CsF	NA	4%	30%	43%	23%	-80 to RT
CH ₂ Cl ₂	TBAF	NA	NA	24%	56%	20%	-80 to RT
CH ₂ Cl ₂	TBAF	NA	NA	23%	54%	23%	RT
CH ₂ Cl ₂	CsF	1%	8%	28%	40%	25%	-80 to RT
EtO ₂	TBAF	3%	14%	24%	37%	22%	-60 to RT
EtO ₂	CsF	4%	11%	22%	34%	29%	-50 to RT
EtO ₂	(CH ₃) ₂ COK	NA	12%	24%	38%	26%	-60 to RT
Neat	TBAF	11%	16%	14%	30%	29%	-60 to RT
Neat	TBAF	NA	NA	NA	NA	100%	RT
Neat	CsF	12%	15%	14%	31%	28%	-60 to RT
DME	TBAF	NA	NA	NA	NA	100%	-60 to RT
Pentane	CsF	NA	14%	24%	48%	19%	-60 to RT
Pentane	(CH ₃) ₂ COK	1%	3%	26%	48%	22%	-60 to RT
Benzene	(CH ₃) ₂ COK	NA	13%	23%	45%	19%	-60 to RT

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. This 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, 1762

Jeremias B. Richter, who was born on this date, discovered the law of equivalent proportions, was the first to establish stoichiometry, and founded the basis of quantitative chemical analysis.

March 12, 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 14, 1984

Twenty-five years ago on this date, the first atom of element of hassium (Hs, 108) was observed at GSI Laboratory, Darmstadt.

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. Halban 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 on this date, 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 Debye 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

Notable NE Chemists

Continued from page 11

under Stockhardt, Bunsen, and Boussingault. In 1857 he opened a laboratory in Boston for analysis and general consulting and maintained it successfully until February, 1865, when he assumed the duties of Professor of General and Industrial Chemistry at the Massachusetts Institute of Technology. Here Charles W. Eliot joined him in October. The two professors developed methods of laboratory instruction and worked together in writing the manuals of Inorganic 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.

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

continued on page 16

burner, filter pump, a galvanic battery, and with Henry E. Roscoe, the actinometer.

Additional historical events can be found at Dr. May's website, <http://faculty.cua.edu/may/Chemistrycalendar.htm>. ◇

Summer Scholar

Continued from page 13

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.^{9,10} 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 **6a** generated 56% of **10**; while this represented a fair yield, the fact that the ketone was being formed in the reaction mixture instead of **12** led to problems with double addition.

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 trifluoroethyl 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-butanone 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 revis-

ited 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.

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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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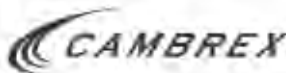
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Notable NE Chemists

Continued from page 16

in the Massachusetts Institute of Technology. He accepted. His colleague was Storer. These men began the chemistry department of the Institute in a small, poorly equipped room upstairs, in an old building on Summer Street, nearly opposite the present location of C.F. Hovey and Co. Here they wrote the famous "Eliot and Storer books." Eliot remained at the Institute four years, resigning in 1869 to become President of Harvard University. His active work as a chemist then ceased, but he was a friend and supporter of his favorite science up to the time of his death. ◇

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Calendar

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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)

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

Mar 11

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)
"Bioinspired Macromolecules: Protein Catalysts
to Materials"
Boston College, Merkert 130 4:00 pm
Abbott Lecture in Organic Chemistry:
Michael T. Crimmins (University of North
Carolina at Chapel Hill)
Title: TBA
Kevin Cusack (Abbott Bioresearch 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 P 106 4:30 pm

**Notices for The Nucleus
Calendar of Seminars
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