



THE NUCLEUS

February 2019

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Monthly Meeting

A Joint Meeting of NESACS and NOBCCHE at the Broad Institute

LaShanda Korley to Speak

2018 NESACS Process Chemistry Symposium

By Mindy Levine

Protein Kinase Inhibitors: Inventing New Patent Strategies

By Katherine Ann Rubino

Summer Scholar Report

By Yingzi Huang and Jia Niu, Boston College

2018 NESACS Process Chemistry Symposium Features Cutting-Edge Science

By Mindy Levine, 2018 NESACS Chair



From left to right: Kami Hull, Frances Arnold, Robert Knowles, Alan Cherney, David Leahy, Ken Drew, Matthew Beaver, Scott Plummer, Stefanie Roeper, Theodore Martinot, Suzie Opalka, Eric Meggers, Cristian Harrison, Erin O'Brien, and Katherine Lee

The NESACS Process Chemistry Symposium was held on October 19, 2018 at Novartis in Cambridge, MA and brought together almost 200 people from over 50 different companies and academic institutions for an exciting daylong symposium.

The symposium featured presentations by eight speakers who represented academic institutions from across the United States as well as Germany, and biotech and pharmaceutical companies from the local scientific community: Robert Knowles, Princeton University; Cristian Harrison, Vertex; Theodore Martinot, Merck; Kami Hull, University of Texas; Scott Plummer, Novartis; Alan Cherney, Amgen; Eric Meggers, Philipps-Universität Marburg; and 2018 Nobel Prize in Chemistry Laureate Frances Arnold, California Institute of Technology.

The topics included cutting-edge research on proton-coupled electron transfer, process development to overcome challenges with potentially energetic materials, synthesis of substituted oxadiazoles and benzocyclobutyl amines, transition metal-catalyzed amination and amidation reactions, aqueous surfactant technology, development of branch-selective cross-coupling of alkylzinc reagents, asymmetric catalysis with “chiral at metal” complexes, and directed evolution to optimize and invent new enzymes.

The 2018 NESACS Process Chemistry Symposium was generously supported by the following sponsors: Amgen, Bio-

gen, Merck, Novartis, Takeda, Vertex, Navin Fluorine, Johnson Matthey, Cambrex, SK BIOTEK, Flamma, PharmaBlock, Mettler Toledo, J-Star Research, PCI, and Strem Chemicals, Inc.

The symposium was designed to allow ample time for discussion after each talk as well as opportunities for attendees to network throughout the day over breakfast and lunch, and during coffee breaks. Following the last talk, there was a reception which included opportunities for sponsors to display literature and to share information about products and services with the attendees.

We would like to acknowledge the members of the organizing committee: Matthew Beaver (Amgen), David Leahy (Takeda), Katherine Lee (Pfizer and NESACS), Matthew Maddess (Merck), Scott Plummer (Novartis), Erin O'Brien (Biogen), Suzie Opalka (Biogen), and Stefanie Roeper (Vertex).

Thank you also to NESACS colleagues Anna Singer (Administrative Coordinator), Ken Drew (PayPal registration), Ashis Saha (Treasurer), Jim Piper (Past Treasurer), and Roy Hagen (Webmaster). Thank you to Novartis for hosting the symposium, with special thanks to Karin Briner, Mark Hellberg, Jocelyn Williams, and Pete Delgado from Novartis, for joining with us to make the 2018 NESACS Process Chemistry Symposium a success. ◇

The Northeastern Section of the American-Chemical Society, Inc.

Office: Anna Singer, 12 Corcoran Road,
Burlington, MA 01803
(Voice or FAX) (781) 272-1966.
e-mail: secretary@nesacs.org
NESACS Homepage:
http://www.NESACS.org

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Andrew Scholte
Sanofi-Genzyme
153 2nd Ave
Waltham, MA 02451-1122
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Immediate Past Chair

Mindy Levine
35 Cottage St
Sharon, MA 02067-2130
(516)697-9688
mindy.levine@gmail.com

Secretary

Michael Singer
MilliporeSigma
400 Summit Drive, Burlington, MA 01803
(781)-229-7037,
Michael.singer@milliporesigma.com

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Ashis Saha
67 Bow St
Arlington, MA 02474-2744
(978)212-5462
sahaashish1909@gmail.com

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Editor: Michael P. Filosa, Ph.D., 18 Tamarack Road, Medfield, MA 02052 Email: mpf1952@gmail.com; Tel: 508-843-9070

Associate Editors: Myron S. Simon, 60 Seminary Ave. apt 272, Auburndale, MA 02466
Morton Z. Hoffman, 23 Williams Rd., Norton, MA 02766

Board of Publications: Ajay Purohit (Chair), Mary Mahaney, Ken Drew, Katherine Lee, Katherine Rubino

Business Manager: Vacant: contact Michael Filosa at mpf1952@gmail.com

Advertising Manager: Vacant: contact Michael Filosa at mpf1952@gmail.com

Calendar Coordinator: Samurdhi Wijesundera, Email: samu.amameth@gmail.com

Photographers: Brian D'Amico, Morton Z. Hoffman

Proofreaders: Donald O. Rickter, Morton Z. Hoffman

Webmaster: Roy Hagen, Email: webmaster@nesacs.org

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Chad A. Mirkin to Receive 2019 Theodore Richards Award for Conspicuous Achievement in Chemistry

Northeastern Section, American Chemical Society



Northwestern University's Chad A. Mirkin has been selected to receive the 2018 Theodore Williams Richards Award and Medal, in recognition of "conspicuous achievements in chemistry," from the Northeastern Section of the American Chemical Society (ACS).

Mirkin, the George B. Rathmann Professor of Chemistry and the director of the International Institute for Nanotechnology (IIN) at Northwestern, will receive the award during the ACS Northeastern Section monthly meeting on Thursday, March 14, 2019, in Cambridge, Mass. Mirkin will be the 45th recipient of the biennial award established in 1928. Recipients of the prestigious honor include 11 Nobel laureates and other scientific luminaries.

Mirkin is a world-renowned chemist and expert in nanotechnology, most known for his discovery and development of spherical nucleic acids, and the many medical diagnostic, therapeutic and materials applications that have derived from them: Dip-Pen Nanolithography (recognized by National Geographic as one of the "top 100 scientific discoveries that changed the

world") and numerous other contributions to supramolecular chemistry. Mirkin is the author of more than 730 manuscripts and more than 1,100 patent applications worldwide (more than 310 issued), and he is the founder of seven companies.

Among an elite group of scientists, engineers and medical doctors to be elected to all three branches of the U.S. National Academies — the National Academy of Sciences, the National Academy of Engineering and the National Academy of Medicine — Mirkin also is a professor of medicine at the Feinberg School of Medicine and a professor of chemical and biological engineering, biomedical engineering and materials science and engineering in the McCormick School of Engineering at Northwestern. Mirkin served on President Barack Obama's Council of Advisors on Science and Technology, from 2009 through 2016.

His scientific achievements have been recognized with more than more than 130 national and international awards, including the NAS Raymond and Beverly Sackler Prize in Convergence Research, the Dan David Prize (Israel), the Wilhelm Exner Medal (Austria), the RUSNANOPRIZE (Russia), the Friendship Medal (China), the Dickson Prize in Science, the American Institute of Chemists Gold Medal and the \$500,000 Lemelson-MIT Prize.

Mirkin has served on the editorial advisory boards of more than 20 scholarly journals and is a current associate editor of the *Journal of the American Chemical Society* and the founding editor of the journal *Small*, one of the premier international nanotechnology journals. Mirkin has co-edited multiple best-selling books. ◇

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Monthly Meeting

The 985th Meeting of the Northeastern Section of the American Chemical Society

A Joint NOBCCHE-NESACS Event

Thursday, February 28, 2019

Broad Institute

415 Main Street, Cambridge, MA 02142

4:30 pm **NESACS Board Meeting**

5:30 pm Social Hour

6:30 pm Dinner

7:30 pm Andrew Scholte., NESACS Chair, Presiding

Keynote Presentation (Auditorium, 415 Main St):

Dr. LaShanda Korley, Distinguished Associate Professor, Department of Materials Science and Engineering, University of Delaware
Title: *Utilizing concepts of mechanics, transport, and assembly in Nature – towards responsive materials via strategic control of architecture and alignment*

**YOU MUST REGISTER IN ADVANCE TO ATTEND THE MEETING;
THERE IS NO REGISTRATION FEE TO ATTEND THE MEETING;**

DINNER RESERVATIONS ARE REQUIRED.

THE PUBLIC IS INVITED

- For those who would like to join us for dinner, register by noon, Thursday, February 21, at <https://lashanda-korley.eventbrite.com>. Cost: Members, \$30; Non-members, \$35; Retirees, \$20; Students, \$10. Dinner reservations not cancelled at least 24 hours in advance will not be refunded. For additional information, contact the Administrative Coordinator, Anna Singer, via e-mail at secretary@nesacs.org.
- If you wish to join us for this meeting and not eat dinner, please register by noon, Thursday, February 21, at <https://lashanda-korley.eventbrite.com> Select "Seminar only".
- Directions to Broad Institute: 1. From Route 90 take exit 18 toward Cambridge onto Cambridge Street for 0.6 miles 2. Turn right onto Memorial Drive and continue for 2.1 miles. 3. Make a U-turn at Wadsworth Street and continue for 0.2 miles. 4. Turn right onto Ames Street and continue for 0.2 miles. Turn left onto Main Street and continue for 300 feet; the Broad Institute will be on the right. Parking is available for \$10 after 4pm at Kendall Center Green Garage (0.2m).
- From Kendall Center Green Garage: 5 min walk (0.2 m)
- Additional Nearby parking:
<http://greaterbostonparking.com/kendall.html>
<http://www.pilgrimparking.com/boston-parking-garages/kendall-square-south-garage.htm> ◇

The NESACS website

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Biography:

LaShanda T.J. Korley joined the Departments of Materials Science and Engineering, and Chemical and Biomolecular Engineering at the University of Delaware (UDel) in January 2018 as a Distinguished Associate Professor. Prior to Prof. Korley's appointment at UDel, she held the Climo Associate Professorship of Macromolecular Science and Engineering at Case Western Reserve University, where she started her independent career in 2007. Taking inspiration from nature, her research program involves understanding the design rules employed by nature and applying these strategies to the development of mechanically-enhanced and tunable materials. Prof. Korley is the Principal Investigator of the recently awarded NSF PIRE: Bio-inspired Materials and Systems.

She received a B.S. in both Chemistry & Engineering from Clark Atlanta University as well as a B.S. in Chemical Engineering from the Georgia Institute of Technology in 1999. Dr. Korley completed her doctoral studies at MIT in Chemical Engineering and the Program in Polymer Science and Technology in

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Abstract:

Taking cues from biological materials, we are interested in understanding the design rules employed by nature and applying these strategies to the development of mechanically-enhanced and tunable materials.

Motivated by the pine cone, we have explored the fabrication of responsive composite systems utilizing high modulus, electrospun and low molecular weight gelators as fillers. Here, we discuss new insights into hygromorphic (e.g. hydration/humidity) response in composites utilizing concepts of interfacial assembly, transport, bias, and orientation. We have fabricated a strategically interfaced hygromorphic composite utilizing an active electrospun filler and a passive, low molecular weight gelator layer in an elastomeric matrix. The impact of material parameters on water front progres-

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Protein Kinase Inhibitors: Inventing New Patent Strategies

By Katherine Ann Rubino, NESACS Board of Publications, Patent Attorney, Caldwell Intellectual Property Law

Protein kinase inhibitors are the second most targeted group of drug targets, after G-protein coupled receptors, as the human body encodes over 500 protein kinases. Protein kinase inhibitors work by blocking the action of one or more protein kinases. Protein kinases are enzymes that add a PO_4 phosphate group to an amino acid, generally serine, threonine, or tyrosine. Protein kinase inhibitors were first developed to treat cancer, which causes hyperactive protein kinases. By inhibiting overactive protein kinases, kinase inhibitors can regulate cell signaling pathways and stop uncontrolled cell growth and oncogenesis.

There are several different types of protein kinase inhibitors, classified based on the structure of the enzyme bound antagonist complex. The majority of inhibitors target the APT-site of the kinase in its active state. Type II inhibitors induce a distinguishable complex, as they occupy an additional hydrophobic pocket. Recently, naturally occurring kinase inhibitors have been discovered as a new class of kinase inhibitors, which can bind directly to tyrosine kinases to alter phosphorylation. Many of these naturally occurring kinase inhibitors have antioxidant properties and are sold on pharmacy shelves including resveratrol, quercetin, curcumin, and chrysin.

Protein kinase inhibitors were first discovered for the treatment of cancer in the late 1970's and early 1980's, when the first oncogene was found to be a protein kinase. Following this discovery, the first protein kinase inhibitors, known as naphthalene sulphonamides were synthesized. This led to further research and development until the first protein kinase inhibitor was approved by the Food and Drug Administration (FDA) in 2001, known as imatinib or Gleevec. This medication is used to treat a number of cancers, for example, chronic myeloid leukemia, gastrointestinal stromal tumors, acute lymphoblastic leukemia, as well as several others. Protein kinase inhibitors are ideal treatment options for patients diagnosed with cancer as they provide targeted therapy and limited toxicity, as compared to chemotherapy which can have a very narrow therapeutic index.

Currently, 37 kinase inhibitors have received FDA approval for different oncology-based indications, and over 150 kinase inhibitors are currently in clinical trials. Roughly 25% of all drug development is currently directed towards kinase inhibitors. Kinase inhibitors are being studied for many uses beyond oncology, including diseases such as liver ischemia, Crohn's disease, osteoarthritis, and ischemia reperfusion. At the present moment, 2 kinase inhibitors have been approved for non-oncology-based treatments. These include Xeljanz approved for arthritis and Ofef approved for pulmonary fibrosis.

As kinase inhibitors have exhibited potentials far beyond oncology uses, the question remains, does a drug found to

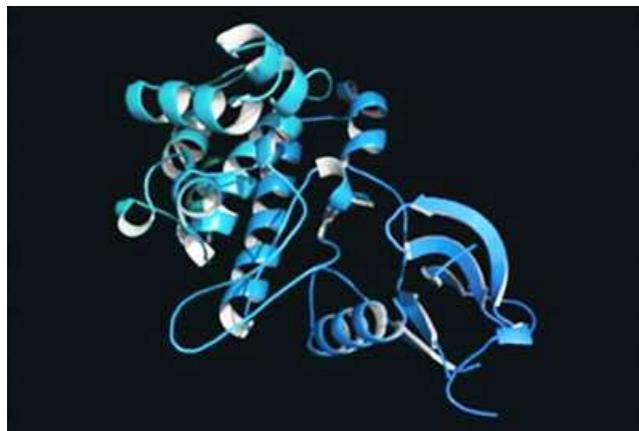


Illustration of Protein kinase inhibitor

treat a new indication need a new patent? This is a complex problem, that can be traced back to aspirin.

Aspirin, also known as acetylsalicylic acid, was synthesized in 1899, and a patent was granted to Frederick Bayer & Company on March 6, 1899 for acetylsalicylic acid. Under the Patent Act of 1836, a patent filed in 1899 would have been valid for 14 years with an extension of up to an additional 7 years. However, patents for aspirin continued to be filed today. This is done through a process known as drug repositioning; whereby, approved drugs and compounds are found to treat a different disease state. For example, Viagra was originally discovered to treat pulmonary hypertension, and only later on was it discovered to also be able to treat erectile dysfunction. Furthermore, the well-known antibiotic erythromycin, used to treat bacterial infections of the skin and respiratory tract, was repositioned to treat a condition of delayed gastric emptying known as gastroparesis.

Through the process of drug repositioning, new methods of treatments are discovered and utilized. When a drug is first discovered for its initial use, a patent is generally obtained to cover the chemical compound, as well as patents that may cover methods of treating a disease state with the compound. After patents for the original compound have expired, new filings can be obtained by others pertaining to new methods of treatment or use that were not covered originally. For example, a search of recent aspirin filings shows patents obtained in the past five years showing unique methods for preparing aspirin, combining aspirin together in dosage forms with other medications, as well as stable aspirin preparations. These filings come almost 120 years after the original patent for acetylsalicylic acid was first obtained.

A review of patent filings at the United States Patent and Trademark Office (USPTO) indicates that the number of ki-

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February 2019 Medicinal Chemistry Symposium

A Medicinal Chemistry Symposium organized by the Medicinal Chemistry Section of the Northeastern Section of the American Chemical Society

Recent Advances in Cancer Therapies

Wednesday, February 6, 2019

Bristol-Myers Squibb

100 Binney Street, Cambridge, MA 02142

3:00 pm Refreshments

3:15 pm **Welcome**-Raj Rajur, Medicinal Chemistry Program Chair, CreaGen Inc., Woburn, MA

3:20 pm Introductory Remarks - Gregory Vite, Ph.D. Head, Oncology Discovery Chemistry and New Modalities
Bristol-Myers Squibb

3:30 pm Jared Cumming, Ph.D., Executive Director, Discovery Chemistry Merck, Boston, MA
Title: "Novel *STING* agonists: Activating the Innate Immune System to Fight Cancer"

4:15 pm Vasanthi Viswanathan, Ph.D., Broad Institute of MIT and Harvard, Cambridge, MA
Title: "Chemical Biology-Based Approach to Understanding and Overcoming Cancer Therapy Resistance"

5:00 pm Scott Biller, Ph.D., Chief Scientific Officer, Agios, Cambridge, MA
Title: "IDH Inhibitors: from Bench to Bedside and Back"

6:00 pm Social Hour

6:30 pm Dinner

7:30 pm **Keynote Presentation**

Prof. Nathanael Gray, Ph.D., Harvard Medical School and Dana-Farber Cancer Institute, Boston, MA

Title: "Targeted Protein Degradation as a New Drug Development Strategy"

Symposium Organizing Committee: Kap-Sun Yeung, Matthew Hill, Swanee Jacutin-Porte, Andrew Scholte, Raj (SB) Rajur

THE PUBLIC IS INVITED - RESERVATIONS ARE REQUIRED

- Dinner reservations should be made no later than 11:30 pm, Wednesday, January 30, 2019. Reservations are to be made using Eventbrite: <https://nesacs-bms-oncology.eventbrite.com> Members, \$30, Non-members, \$35; Retirees, \$20; Students, \$10.
- If you wish to join us for this meeting and not dinner, please register by 11:30 pm, Wednesday, January 30, 2019 at <https://nesacs-bms-oncology.eventbrite.com> Select "Seminar Only."
- New members or those seeking additional information, please contact the NESACS Administrative Coordinator, Anna Singer, at secretary@nesacs.org.
- For questions about the symposium, please contact Kap-Sun Yeung at kapsun.yeung@bms.com or Andrew Scholte at Andrew.Scholte@sanofi.com

Directions to Bristol-Myers Squibb Cambridge Research Site:

- The Bristol-Myers Squibb Cambridge research site is located at 100 Binney Street, Cambridge, MA 02142, between Second Street and Third Street.
- It is within walking distance of the MBTA Kendall station on the Red Line (about 0.3 miles) or the Lechmere station on the Green Line (about 0.5 miles).
- **Parking Garage:** There are several parking garages within walking distance from BMS. The closest one is behind the BMS building on Linskey Way, between East Kendall Street and Second Street. ◇

Summer Scholar Report

Synthesis of Heterotelechelic Polymers via RAFT Polymerization for Tagging Red Blood Cells as Drug Carriers

Yingzi Huang and Jia Niu, Boston College, Chestnut Hill, MA 02467

Abstract

Heterotelechelic polymers with functionalized chain ends were synthesized using the reversible addition-fragmentation chain transfer (RAFT) chemistry. A trithiocarbonyl chain transfer agent was designed to introduce reactive polymer chain ends for future bioconjugation to red blood cells. NMR and gel permeation chromatography (GPC) were used to study the molecular weight, dispersity, and chain end group fidelity.

Introduction

Tagging red blood cells (RBC) using nanoparticles can offer great advantages for potential applications in drug delivery. The abundance, biocompatibility, and longevity in circulation made the RBC an excellent candidate as a drug shuttle¹. Also, the drug-carrying agents attached to RBC can be cleared through the pathways that normally eliminate old and damaged red blood cells, which provides a novel approach for targeting these pathways.² Therefore, anchoring the drug-loading nanoparticle to the surface of RBCs via a synthetic polymer linker is a promising strategy to improve the solubility of the loaded drugs, extend the time of their circulation, reduce the unintended side effects of the drug, and enhance their efficacy.

It has been widely studied and developed that controlled radical polymerization (CRP) gives predictable molecular weights, low polydispersity (\mathcal{D}) and precise control of the reaction rate.³ For reversible addition-fragmentation chain transfer (RAFT) reaction, the reaction kinetics is controlled by the chain transfer agent (CTA). The result of RAFT polymerization is a well-defined polymer with low \mathcal{D} .⁴

In this work, we propose to synthesize heterotelechelic polymers with thiol and alkyne functionalities on the chain ends. The thiol can form conjugate with a drug-loading agent via thiol-maleimide reaction and the alkyne can undergo click chemistry with an azide-functionalized affinity reagent.

The Experimental Section

Materials. All organic solvents used were from Sigma and used as received. All commercially available chemicals were from Sigma and TCI. Azobisisobutyronitrile (AIBN) was recrystallized in methanol. 2-hydroxyl acrylate (HEA) was purified according to literature. Poly(ethylene glycol) acrylate (PEGA, $M_w=480$) and *N,N*-dimethylacrylamide (DMA) were purified by alumina column.

Characterization. ¹H and ¹³C NMR spectra were recorded in CDCl₃, on either a Varian Gemini-600 (600 MHz) or Varian Inova-500 (500 MHz) and calibrated residual solvent peaks. Size-exclusion chromatography (SEC) measurements were performed using Tosoh's high-performance SEC system HLC-8320GPC with TSKgel Alpha-M columns at 50 °C and a flow rate of 1 mL/min. HPLC grade dimethylformamide (DMF) with 0.01 M LiBr (anhydrous, purchased from Sigma-

Aldrich) as used as the eluent. Polystyrene standards (Ready-Cal Kit, Sigma-Aldrich#81434) were used to determine the molecular weight and molecular weight distribution of polymers. The polymers were dissolved in the above DMF solution and filtered through a 0.20 μm PTFE filter before being injected into the SEC system.

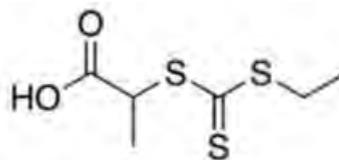


Figure 1. Structure of PAETC

Synthesis of 2-(propionic acid)ylethyl trithiocarbonate (PAETC). Sodium hydroxide (6.25 M, 3.20 mL) and ethanethiol (1.28 g, 20 mmol, 1.53 mL) were combined in H₂O (20 mL) and acetone (50 mL). methanedithione (1.83 g, 24.00 mmol, 1.45 mL) was added dropwise. The reaction mixture was then left to react at room temperature for 40 min, affording a dark orange solution. 2-bromopropanoic acid (3.37 g, 22.00 mmol, 1.98 mL) was added dropwise on ice bath over 10 min. The resulting yellow reaction mixture was stirred overnight. 5.0 mL of 10M HCl was added, and the reaction mixture is extracted into ethyl acetate. The organic layer was washed with 100 mL brine three times and dried with a layer of sodium sulfate. The residue was concentrated in vacuo and recrystallized in hexane at 50 °C to yield 2-ethylsulfanylcarbothiolsulfanyl-propanoic acid (2.89 g, 13.74 mmol, 68.70% yield). ¹H-NMR (500 MHz, CDCl₃) δ 4.86 (1H, q, CH₃CH(S)COOH), 3.38 (2H, q, CH₃CH₂S), 1.63 (3H, d, CH₃CH(S)COOH), 1.36 (3H, t, CH₃CH₂S).

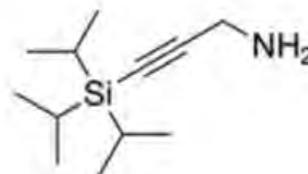


Figure 2. Structure of TIPS-PA

Synthesis of 3-triisopropylsilylprop-2-yn-1-amine (TIPS-PA). prop-2-yn-1-amine (851.73 mg, 15 mmol, 990.38 μL) in anhydrous THF (40 mL) was cooled to -78 °C. butyllithium (2.5 M, 6.00 mL) were added dropwise. The solution was stirred for 30 minutes and warmed to 0 °C. chloro(triisopropyl)silane (3.58 g, 18.00 mmol, 3.97 mL) was added dropwise. The reaction was left to react overnight and concentrated in vacuo. 50 mL of H₂O were added and the aqueous layer was extracted with ethyl acetate 3 times. The combined organic phase was dried over sodium sulfate. The residue was

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Summer Scholar

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purified by chromatography on silica gel (1:20 to 1:1 gradient EtOAc: Hexane) to give 3-triisopropylsilylprop-2-yn-1-amine (2.18 g, 10.31 mmol, 69% yield) as a yellow oil. ^1H NMR (600Hz, CDCl_3) δ 1.05- 1.09 (21 H, m, 3x Si-CH(CH_3) $_2$), 1.43 (2H, br s, NH_2), 3.45 (2H, s, CH_2). ^{13}C NMR (600Hz, CDCl_3) δ 13.20 ($\text{CH}_3\text{CH}_2\text{S}$), 16.68 (CH_3CHS), 31.95 ($\text{CH}_3\text{CH}_2\text{S}$), 47.71 (CH_3CHS), 177.66 (CHCOOH), 221.77 (SC(S)S)

Synthesis of PAETC-alkyne-TIPS protected (PAETC-yn).

3-triisopropylsilylprop-2-yn-1-amine (1.16 g, 5.5 mmol), 1-hydroxypyrrolidine-2,5-dione (575.45 mg, 5.00 mmol), N,N' -dicyclohexylmethanediiimine (1.13 g, 5.50 mmol) were dissolved in DCM (30 mL). The solution was cooled to 0 °C and stirred for 15 minutes. Then the reaction was allowed to stir at room temperature for one hour, during which it turned cloudy. The reaction was then filtered, cooled to 0 °C, then 3-triisopropylsilylprop-2-yn-1-amine (1.16 g, 5.5 mmol) was added. The reaction was then stirred overnight and washed with brine three times. The organic layer was dried over anhydrous sodium sulfate. The residue was purified by silica gel column chromatography (1: 9 to 1:1 gradient EtOAc:Hexane) to yield 2-ethylsulfanyl-carbothioylsulfanyl-N-(3-triisopropylsilylprop-2-ynyl)propanamide (2.00 g, 4.95 mmol, 99% yield). ^1H NMR (500 MHz, CDCl_3): δ 1.05- 1.09 (21 H, m, 3x Si-CH(CH_3) $_2$), 1.36 (3H, t, $\text{CH}_3\text{CH}_2\text{S}$), 1.63 (3H, d, $\text{CH}_3\text{CH(S)COOH}$), 3.37 (2H, dd, CH_2NH), 4.05 (2H, q, $\text{CH}_3\text{CH}_2\text{S}$), 4.76 (1H, q, $\text{CH}_3\text{CH(S)COOH}$). ^{13}C NMR (600 MHz, CDCl_3) δ 219.35 (SC(S)S), 172.49 (NHCOCH), 102.45 (Si-CC), 84.84 ($\text{CC-CH}_2\text{NH}$), 47.37 (COCH_2S), 31.79 ($\text{C-CH}_2\text{NH}$), 30.14 ($\text{S-CH}_2\text{CH}_3$), 18.52 ($\text{Si-CH(CH}_3)_2$), 16.00(SCH(O)CH_3), 11.26 ($\text{S-CH}_2\text{CH}_3$), 11.08 ($\text{Si-CH(CH}_3)_2$). DART+(m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{18}\text{H}_{34}\text{SiNOS}_3$ 404.7492 found 404.1572.

Synthesis of Poly(PEGA) and Poly(HEA): RAFT polymerization was conducted using standard Schlenk techniques. Monomer, CTA and AIBN were loaded in a Schlenk tube with 1mL DMF as solvent. The sealed tube was subjected to three freeze-pump-thaw cycles. The polymerization was initiated at 70 C. The reaction was stopped by exposure to air at certain time points. The polymers were purified against H_2O (MWCO 3500 Da). Polymer conversion was calculated from the signal from vinyl protons in the polymer versus the monomer in ^1H NMR (CDCl_3). Polymer molecular weight and the polydispersity (\bar{D}) was measured by SEC.

Synthesis of Poly(DMA): Reactions were carried in a 3 mL clear glass vial with PP Hole Cap & PTFE/Silicone Septa with

all reaction components. The vial was covered in aluminum foil and degassed by N_2 for 30 minutes, then irradiated by a blue LED strip (4.8 Watts, $\lambda_{\text{max}} = 435 \text{ nm}$) at room temperature. The polymer was analyzed by ^1H NMR (CDCl_3) and GPC to measure the conversions, number average molecular weights (M_n) and \bar{D} .

Result and Discussions:

CTA Design and Synthesis

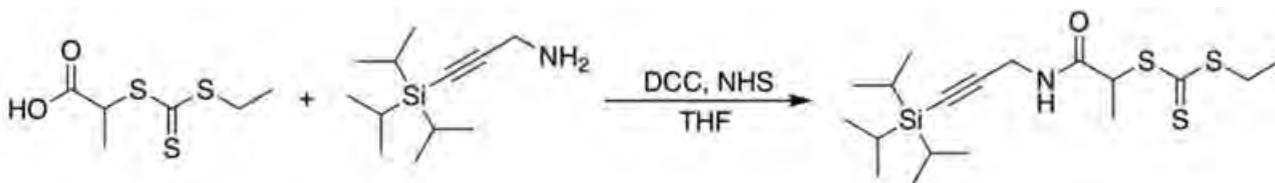
The CTA was designed to contain a trisopropylsilyl (TIPS) protected-alkyne group and a trithiocarbonate group. The TIPS protecting group can be conveniently deprotected by tetrabutylammonium. The alkyne will couple with the antibody that with an azide modification via copper(I)-catalyzed alkyne-azide cycloaddition (CuAAC). Trithiocarbonate chain transfer agent was chosen for its high reactivity with acrylates and acrylamide monomers.⁵ The thiocarbonate chain end could be removed to yield a free thiol group, which then can be coupled to a maleimide-containing poly(lactic-co-glycolic acid) (PLGA) drug loading molecule. The synthesis of 2-(ethyl trithiocarbonate) propionic acid was conducted following literature procedures. The resulting trithiocarbonate was subsequently coupled using N,N' -dicyclohexylcarbodiimide and N -hydroxysuccinimide to form the final CTA in high yield (Scheme 1).

Synthesis of Poly(PEGA) and Poly(HEA) via RAFT Poly-

merization: The CTA was used to polymerize monomers poly(ethylene glycol) acrylates (PEGA), and 2-hydroxyl acrylate (HEA) using azobisisobutyronitrile (AIBN) as initiator to form poly(PEGA) and poly(HEA) (Table 1, entry 1-4). With $[\text{PEGA}]:[\text{CTA}]:[\text{AIBN}] = 200:1:0.1$, from ^1H -NMR the monomer conversion is calculated to be 77% after 2 hours. \bar{D} was found to be 1.43 for the poly(PEGA) from SEC. The broad \bar{D} occurred in the polymerization of PEGA is a sign that the reaction started to lose control at higher molecular weight (Table 1, entry 1). A lower degree of polymerization at the ratio of $[\text{PEGA}]:[\text{CTA}]:[\text{AIBN}] = 50:1:0.1$ was subsequently studied.

However, SEC still showed $\bar{D} = 1.29$ at 63% monomer conversion by NMR (Table 1, entry 2). For poly(PEGA), the M_n calculated by SEC using the polystyrene standards was lower than the predicted value by NMR. Such a disparity was attributed to the structural difference between the branched hydrophilic chain on the poly (PEGA) and polystyrene standards. I reason that the high molecular weight of PEGA monomer limits the monomer concentration and reactivity in polymerization, so I decided to try a smaller monomer HEA. The polymerization at the ratio of $[\text{HEA}]:[\text{CTA}]:[\text{AIBN}] = 200:1:0.1$ and $50:1:0.1$ were studied. However, both polymers

continued on page 12



Scheme 1. Synthesis of TIPS protected chain-transfer agent.

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Invite for the CCEW event at the MoS, Boston: Saturday April 13, 2019

Dear All:

The NESACS and MoS will be celebrating Chemists Celebrate Earth Week at the Blue Wing of MoS on Saturday April 13, 2019 from 11.00 am - 3.00 pm. The event starts at 9.00 am with the training session.

The theme for Chemists Celebrate Earth Week event this year is Paper "Take Note: The Chemistry of Paper". We encourage teams to bring their own activities related to the current theme. If you don't have one, please mention it on the sign-up sheet. We can provide you with activities. Looking forward to having science at the fun-filled STEM outreach event. After this event, the science educators can enjoy the Cambridge Science Festival happening in the same area.

Here is the schedule:

Arrival/Training: 9:00 – 10:30am

1st shift activities: 10:30am – 1:00pm (includes set-up time)

2nd shift activities: 12:30pm – 3:30pm (includes clean-up time)

Please complete the online form by Feb 15, 2019 (Friday). This would greatly assist the organizers with ordering t-shirts for our science educators (volunteers).

SIGN UP GOOGLE FORM: <https://goo.gl/forms/9gDDJIrynonR1JfO2>



Please enter the Name of your Institution, Title of your activity, Name & e-mail of the contact person from your Institute, names of all the science educators from your Institute, number of t-shirts (+ t-shirt sizes) for your team members at the above link.

We request the team leader from your institution to e-mail the organizers (Jay: jranga@salemstate.edu, David: dsittenfeld@mos.org & Emily: ehostetler@mos.org) the following information by March 1, 2019 (Friday).

- (a) Title of your activity
- (b) Abstract
- (c) List of chemicals to be used for the planned activity

We hope to have as many activities as possible at the Earth Day Event. All the science educators will receive an Earth Day t-shirt. As always, thanks for your commitment and enthusiasm.

Dr. Ranga

On behalf of Salem State University, NESACS, and MoS ◇

Summer Scholar

Continued from page 9

still exhibited $\bar{D} > 1.2$ (Table 1, entry 3-4). The short half-life of the AIBN initiator is known to limit the molecular weight of the polymer and cause broad dispersity.⁶ To overcome this challenge, I decided to employ the photoelectron transfer-RAFT (PET-RAFT) method to generate the heterotelechelic polymers. As an emerging technology, PET-RAFT allows the continuous single electron transfer from the excited photocatalysts over the course of the irradiation, leading to a constant concentration of the propagating chain ends and excellent control over polymerization for high molecular weight polymers.^{7,8} Dimethyl-acrylamide (DMA) was chosen as the monomer due to compatibility for photo-RAFT and water-solubility, which makes a good candidate for making a hydrophilic linker. The photocatalyst tris(2,2'-bipyridine) ruthenium(II) was used to initiate the reaction after irradiated by blue LED light ((4.8W, $\lambda_{\text{max}} = 435$ nm). Three poly(DMA) polymers were synthesized with [M]:[CTA]:[I] ratio between 50 and 400, and the polymers achieved reasonable conversion and low polydispersity even at high degree of polymerization ($\bar{D} < 1.3$) (Table 1, entry 5). NMR spectrum and SEC traces are shown in Figure 3. The NMR spectrum of dialyzed polymer shows characteristic signals of functional chain ends (Figure 3A). Triisopropyl groups, propargyl amine, trithiocarbonate signals were confirmed by the characteristic signals at δ 1.09 ppm, δ 3.3 ppm, and δ 4.0 ppm respectively.

Conclusion and Future directions:

I have developed a simple method to synthesize polymers with an alkyne group for conjugating proteins site-specifically later in the project. The triisopropylsilyl protected

poly(PEGA), poly(HEA) and poly(DMA) were successfully synthesized via RAFT chemistry. Since poly(DMA) demonstrated low \bar{D} at high molecular weight, poly(DMA) will be deprotected the thiol and alkyne chain ends to conjugate to the drug loading nanoparticle and RBC-binding antibody, respectively. The alkyne would be first deprotected and coupled to the antibody in order to increase the solubility of the polymer complex. Protein SDS gels could be used to detect the presence of the antibody. The polymer-antibody conjugate will then be conjugated to drug-loading PLGA nanoparticles. Transmission electron microscopy will be employed to study the structure of the conjugated nanoparticles. Further investigations for studying for drug delivery agents such as drug loading efficiency, and the stability of the conjugates will be performed in collaboration with Samir Mitragotri's lab at Harvard University School of Engineering.

Bibliographic Citations:

1. Pan, D. C. et al. Nanoparticle Properties Modulate Their Attachment and Effect on Carrier Red Blood Cells. *Scientific Reports* **8** (2018).
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4. Convertine, A. J.; Lokitz, B. S.; Lowe, A. B.; Scales, C. W.; Myrick, L. J.; McCormick, C. L. *Macromol. Rapid Commun* **26**, 791–795 (2005).
5. Mayadunne, R. T. A. et al. Living Polymers by the Use of

continued on page 13

Table 1.
Polymer linkers generated using different hydrophilic monomers.

Entry	M	Initiator	[M]:[CTA]:[I]	Conv (%) 1H NMR	M_n (NMR)	Conv (%) SEC	M_n (SEC)	\bar{D}
1	PEGA	AIBN	200/1/0.1	77	74,100	68	34,500	1.43
2	PEGA	AIBN	50/1/0.1	63	15,600	50	12,800	1.29
3	HEA	AIBN	200/1/0.1	74	17,700	64	12,200	1.38
4	HEA	AIBN	50/1/0.1	69	4,400	68	2,600	1.27
5	DMA	Ru(bpy) ₃ Cl ₂	400/1/1x10 ⁻⁴	68	27,600	55	22,300	1.23
6	DMA	Ru(bpy) ₃ Cl ₂	200/1/1x10 ⁻⁴	76	15,600	56	11,500	1.14
7	DMA	Ru(bpy) ₃ Cl ₂	50/1/1x10 ⁻⁴	63	3,550	57	3,300	1.08

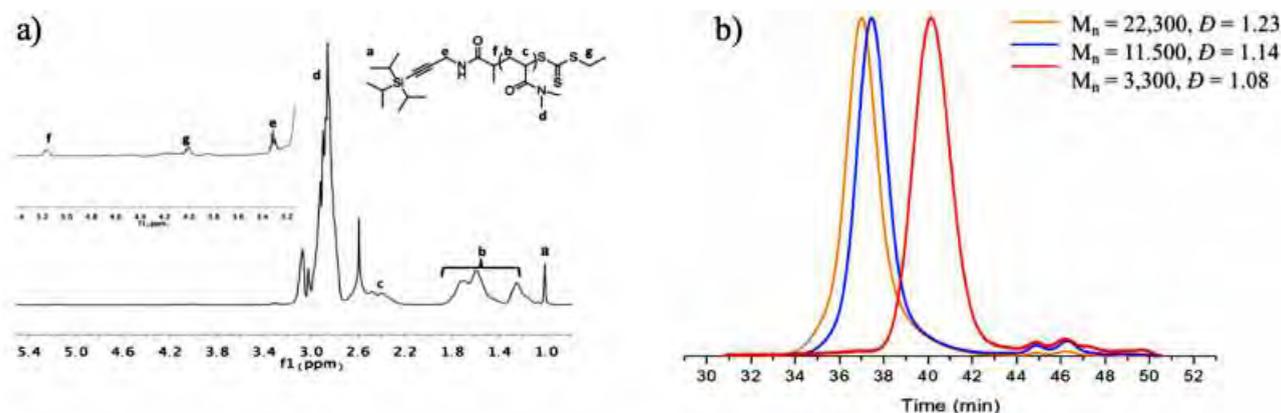


Figure 3. a) ¹H NMR Spectrum (CDCl₃) of PAECT-poly(DMA). b) GPC result of PAECT-poly(DMA).

Announcement

Norris-Richards Undergraduate Summer Research Scholarships March 29, 2019 Deadline

The Northeastern Section of the American Chemical Society 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 2019. The student stipend is \$3000 for a minimum commitment of ten weeks of full-time research work. The remaining \$500 of the award is for 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 Editor of *The Nucleus* by November 1, 2019 for publication in *The Nucleus*. They are also required to participate in

the Northeast Student Chemistry Research Conference (NSCRC) in April 2020.

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, 2019.

Criteria for Selection:

- **Scientific merit** - important factors include the originality of the project, the depth of the investigation, the significance of the scientific questions you pose, and the methods you propose to use.
- **Feasibility** - evidence must be provided to demonstrate that the project can be completed by you in the time available and with the facilities at

your disposal.

- **Preparation** - your academic record, your ability to handle the project, and the background study you have made on your research problem will be taken into consideration.
- **Commitment** - the depth of your commitment, and that of your department, faculty, and institution to independent research as a vital component of science education will be assessed.

Application for 2019:

Application available at: http://www.nesacs.org/awards_norris-richards.html

Completed applications are to be received by the Chair of the Selection Committee no later than March 29, 2019. Please note that applications via email (PDF format) are strongly preferred. Applicants will be notified of the results by email by April 12, 2019, with written confirmation to follow.

Selection Committee Chair:

Professor Jonathan Rochford
Department of Chemistry
University of Massachusetts Boston
100 Morrissey Boulevard Boston, MA
02125-3393

Email: jonathan.rochford@umb.edu ◇

Summer Scholar

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- Trithiocarbonates as Reversible Addition-Fragmentation Chain Transfer (RAFT) Agents: ABA Triblock Copolymers by Radical Polymerization in Two Steps. *Macromolecules* **33**, 243-245 (2000).
6. Fischer, A.; Brebilla A.; Lochon, P.; *European Polymer Journal* **37**(1), 33-37 (2001).
 7. Chen, M.; Zhong, M.; Johnson, J.A.; *Chem Rev* **116**(17), 10167-10211(2016).
 8. Pan, X. et al. Photomediated controlled radical polymerization. *Progress in Polymer Science* **62**,73-125 (2016). ◇



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Abstract

Continued from page 5

sion and actuation were probed theoretically and experimentally in their design. Via this approach, preferential coiling was observed, although two challenges were encountered due to the isotropic nature of the PVA mat: (1) slow response times, and (2) non-uniformity in hydration-induced response. To overcome these challenges, we explored the impact of the alignment of the PVA electrospun fibers as a handle to control rate of hydration and program shape change. These engineered hygromorphic composites exhibited predictable curvature, and much faster response times (2 - 3 min). It is anticipated that these water-responsive systems may have unique applications in therapeutic delivery and chemical/biological protection.

Inspired by spider silk, we have designed a series of polymer-peptide polyurethane/ureas to explore the hierarchical arrangement critical to energy ab-

sorption and mechanical enhancement. We have developed chain-extended and non-chain extended peptide-polyurea hybrids with tunable secondary structure, modulating extensibility, toughness, and stiffness. The sheet-dominant hybrid materials were typically tougher and more elastic due to intermolecular H-bonding, while the helical-prevalent systems generally exhibited higher modulus. We have also explored the impact of a molecular design strategy that overlays a covalent and physically crosslinked architecture in these hybrids, demonstrating that physical constraints in the network hybrids influences hydrogen bonding and morphology. More recently, tailored physical associations within the soft and hard phases were engineered as a function of peptide content, leading to a rheological response dictated by block ordering and highlighting their potential as structural and injectable hydrogels. These structural features have enabled new thrusts in injectable gels and responsive actuators. ◇

A Cartoon by Sidney Harris



The incongruity of the reviewers' comments with the criteria that one thinks should govern the evaluation of a paper or proposal is what makes the cartoon funny. But, as is always true in Sidney Harris's cartoons, there is another message under the surface: Perhaps scientists should embrace the "broader audience" of their papers, or the "outreach" criteria of the granting agencies more openheartedly. Nothing in the nature of the world or the understanding of scientists would be damaged if humor and suspense were allowed to enter scientific papers. Gatekeepers relax!

-- Roald Hoffmann, Cornell University

Biography

Continued from page 5

2005. LaShanda Korley was the recipient of the Provost's Academic Diversity Postdoctoral Fellowship at Cornell in 2005, where she completed a two-year postdoctoral appointment in the Department of Chemical and Biomolecular Engineering. ◇

Protein Kinase Inhibitors

Continued from page 6

nase inhibitor filings is on the rise. Since 2001, there have been 23,198 patent applications relating to kinase inhibitors filed at the USPTO. Over the past five years the number of patent applications filed relating to kinase inhibitors has continued to rise, with the exception of 2015. Furthermore, the number of patent applications filed relating to kinase inhibitors over the past 5 years accounts for 40% of all filings of patent applications relating to kinase inhibitors since 2001. As of early December 2018, there have been 1,915 patent applications relating to kinase inhibitors filed.

Year	# of Patent Applications
2014	1,847
2015	1,796
2016	1,960
2017	2,024
2018	1,915

Number of patent applications filed relating to Kinase Inhibitors

As scientists continue to discover and explore drug discovery in this area, we can expect to see an increase in patent filings over the next few years. This may include new drugs that have been discovered, as well as old drugs that have been shown to do a new trick. Either way, as 2018 has drawn to a close, we can expect 2019 to be a year filled with exciting medical breakthroughs and innovation. ◇

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<http://www.brandeis.edu/departments/chemistry/events/index.html>

<http://chemistry.harvard.edu/calendar/upcoming>

<http://www.northeastern.edu/cos/chemistry/events-2/>

<http://chemistry.mit.edu/events/all>

<http://chem.tufts.edu/seminars.html>

<http://engineering.tufts.edu/chbe/newsEvents/seminarSeries/index.asp>

<http://www.chem.umb.edu>

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<http://www.uml.edu/Sciences/chemistry/Seminars-and-Colloquia.aspx>

<http://www.unh.edu/chemistry/events>

<https://www.wpi.edu/academics/departments/chemistry-biochemistry>

February 1

Prof. Andy McNally (Colorado State)
Boston College, Merkert 130, 4:00 pm

Prof. Min Chen (UMass-Amherst)
UMass-Lowell, Olney 316, 3:30 pm

February 4

Prof. Alex Grenning (Univ. Florida)
Boston University, Metcalf 113, 4:00 pm

Prof. James L. Skinner (Univ. Chicago)
Harvard, Pfizer Lecture Hall, 5:00 pm

Dr. Angad P. Mehta (Scripps Research
Institute)

*Exploring Evolution Through Synthetic
Approaches*

MIT, Building 6-120, 4:00 pm

February 5

Prof. Michelle Farkas (UMass-Amherst)
Tufts, Pearson, Rm P-106, 4:30 pm

February 6

Justin Provazza (Boston University)
Boston University, Metcalf SCI 512, 2:00 pm

Prof. Douglas Stephan (Univ. Toronto)
MIT, Building 4-370, 4:15 pm

Graduate Research Innovation Exchange
(GRIE)

WPI, Rubin Campus Center
8:00 am – 5:00 pm

February 11

Prof. Muralee Murugesu (Univ. Ottawa)
Boston University, Metcalf 113, 4:00 pm

Natalia Shustova (Univ. South Carolina)
Brandeis, Gerstenzang 121, 3:40 pm

Prof. Ke Xu (UCal-Berkeley)
Harvard, Pfizer Lecture Hall, 4:15 pm

February 12

Dr. Giulia Galli (Univ. Chicago)
*At the edge: energy conversion at interfaces
from first principles*
MIT, Building 6-120, 4:00 pm

Prof. Jeffrey Moore (Univ. Illinois)
Tufts, Pearson, Rm P-106, 4:30 pm

February 13

Dr. Robert Best (NIH)
MIT, Building 4-237, 4:15 pm

Prof. Xavier Roy (Columbia)
MIT, Building 4-370, 4:15 pm

February 14

Prof. Charles Sykes (Tufts)
Boston College, Merkert 130, 4:00 pm

February 15

Prof. Young Jo Kim (UNH)
UMass-Lowell, Olney 316, 3:30 pm

February 19

Prof. Paula Diaconescu (UCLA)
Boston College, Merkert 130, 4:00 pm

Prof. Thomas Kodadek (Scripps Research
Institute)
Tufts, Pearson, Rm P-106, 4:30 pm

February 20

Prof. Dan Kahne (Harvard)
Boston College, Merkert 130, 4:00 pm

February 21

Thesis/Dissertation Writing Boot Camp
WPI, Founders Hall, 12:00 pm

February 25

Prof. Jennifer Roizen (Duke)
Boston University, Metcalf 113, 4:00 pm

Prof. Michael Nippe (Texas A&M)
Harvard, Pfizer Lecture Hall, 4:15 pm

February 26

Dr. Joshua Vura-Weis (Univ. Illinois)
*Ultrafast extreme ultraviolet spectroscopy
reveals short-lived states in transition metal
complexes and organohalide perovskite
semiconductors*
MIT, Building 6-120, 4:00 pm

Prof. Ander Peterson (Brown)
Tufts, Pearson, Rm P-106, 4:30 pm

February 27

Prof. Brett Fors (Cornell)
Boston College, Merkert 130, 4:00 pm

Prof. Laura Gagliardi (Minnesota)
MIT, Building 4-237, 4:15 pm

Prof. Stosh Kozimor (Los Alamos National
Laboratory)
MIT, Building 4-370, 4:15 pm

February 28

**Sigma-Aldrich Lecture in Organic
Chemistry**

Prof. Brett P. Fors (Cornell)
MIT, Building 6-120, 4:00 pm

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