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THE NUCLEUS

January 2015 *Vol. XCIII, No. 5*

Monthly Meeting

Dr. Christopher J. Doona, US Army-Natick Soldier RDEC, to speak at Amgen

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Photos from NESACS Events

By Morton Z. Hoffman

Summer Scholar Report

Toward New Organic Solid State Reactions

Tyler S. Harrison and D. J. Sandman, Center for Advanced Materials, Department of Chemistry,
University of Massachusetts Lowell, Lowell, Massachusetts 01854-5046

Introduction

The objective of this research is the discovery and development of new lattice-controlled organic solid-state reactions. Reasons for interest in such reactions include that the rigid structure of the solid state allows for considerable regiospecificity and stereoselectivity, as well as the possibility to synthesize systems that are otherwise difficult to access. Moreover, such reactions are "environmentally friendly" as they do not utilize a solvent. These reactions can be initiated either thermally or by ultraviolet light.

The [2+2] pi dimerization of cinnamic acids[1,2] **1** (X= -OH) and amides, usually accomplished with ultraviolet (uv) light or sunlight, is the best known of molecular solid-state reactions. Since the detailed role of the chromophore in the cinnamic reaction is not clear, a modification of the chromophore by substitution with nitrogen is of interest, and the initial subjects for investigation are shown in Figure 1. The molecular structure of cinnamic acid is regarded as consisting of an aromatic ring, a multiple bond, and a hydrogen-bonding group. In the current research, the aromatic groups could include benzene, naphthalene, pyridine, quinoline, thiophene, furan, and pyrrole. The multiple bonds could include carbon-carbon double and triple bonds and carbon-nitrogen double bonds. The hydrogen-bonding groups would be carboxyl, amide, and urea.

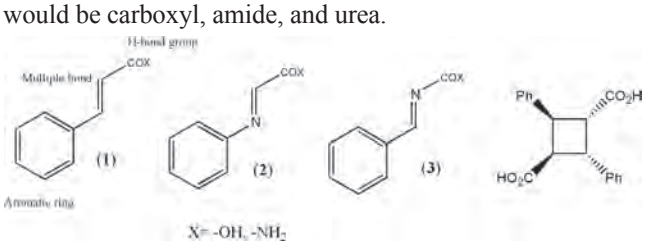

Figure 1: *Cinnamic acid, amide, and related structures; structure of the photodimer of the α-form of* ***1***

Imesatin

A synthesis of **2** (X=-OH) was attempted by reaction of aniline with glyoxylic acid monohydrate[3]. From its IR spectrum, the major product was a determined to be a carboxylic acid; the ^{1}H NMR showed only aromatic resonances and was initially uninformative. Titration of the product with a standard base gave a neutralization equivalent of 238, and structure **4** (calculated MW 240) was assigned to this compound. Heating **4** led to the known compound imesatin **5**, which was synthesized by the reaction of aniline with isatin[4] and compared to **4**. Thermogravimetric analysis (TGA) of **4** led to a weight loss of 7%, as is expected for the postulated chemistry, which is summarized in Figure 2.

While **2** has not yet been obtained, imesatin has the same chromophore as **2**, albeit in a cyclic array. Accordingly, it was irradiated with a 254 nm uv lamp, but no thermal or uv initiated solid state reactivity was found. A search of the Cambridge Structural Database[5] revealed that the double bonds in the imesatin crystal structure are not oriented for solid state reactivity.

Reaction of Benzaldehyde and Urea

The synthesis of **3** (X= -NH_2), benzylidene urea, was reported in the literature. Juaristi[6] refluxed benzaldehyde, urea, and p-toluenesulfonic acid monohydrate in toluene with a Dean-Stark trap to synthesize benzylidene urea, providing reasonable evidence to the structure in the form of IR spectral data, ^{1}H NMR, and ^{13}C NMR.

A synthesis of **3** was attempted by simply heating benzaldehyde and urea in a test tube at 100°C. In identifying the product's melting behavior, it was found that the melt was not reversible, suggesting thermal activity. Differential scanning calorimetry (DSC) confirmed the thermal activity—the endotherm associated with the second heating revealed less heat than that of the initial heating, indicating thermal activity. However, a repetition of this synthesis using freshly distilled benzaldehyde resulted in no reaction occurring. It is believed that the initial reaction was initiated by benzoic acid, a frequent impurity in benzaldehyde, so a few milligrams of that were added. The resulting solid was suspended in hot water, followed by warm acetone to isolate the thermal product. TGA and DSC show that this thermal product is itself thermally reactive, displaying a weight loss above 178°C and a difference in the heat absorbed between subsequent heatings. Following this, a repetition of Juaristi's synthesis was performed. The resulting solid, practically insoluble in numerous solvents, also exhibited thermal reactivity, and TGA and DSC confirmed a decomposition, which is at variance with Juaristi's report. The TGA shows a 5% decrease in weight from 125°C to 169°C, followed by a 75% decrease in weight at 252°C. IR (cm^{-1}): 3434, 3307, 1642, 1595, 1529, 1447, 1366, 1311, 1230, 1200, 1140, 1091, 1053, 1010, 1001, 914, 814, 748, 734, 695, 591, 542,

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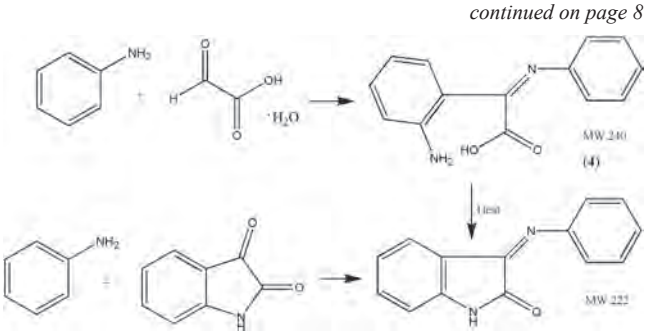

Figure 2: *Formation of imesatin by two routes*

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Office: Anna Singer, 12 Corcoran Road, Burlington, MA 01803
(Voice or FAX) 781-272-1966.
e-mail: secretary(at)nesacs.org
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April 2015 Issue: February 15, 2015

THE NUCLEUS

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Editor:	Michael P. Filosa, Ph.D., 18 Tamarack Road, Medfield, MA 02052 Email: filosam(at)verizon.net; Tel: 508-843-9070
Associate Editors:	Myron S. Simon, 60 Seminary Ave. apt 272, Auburndale, MA 02466, Mindy Levine, 516-697-9688, mindy.levine(at)gmail.com
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Calendar Coordinator:	Xavier Herault, Email: xherault(at)netzero.net
Photographers:	Morton Hoffman and James Phillips
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2015 Chair's Statement

Katherine Lee
katherine.lee@pfizer.com

It is an honor to serve as Chair of the Northeastern Section of the American Chemical Society (NESACS).

- Did you know that NESACS is the largest local section, with over 6,000 members?
- Did you know that if you are an ACS member residing within the geographical boundaries of NESACS, that you are automatically a NESACS member?
- Did you know that one of my goals, as NESACS Chair, is to increase engagement and participation of our members in NESACS activities?

We are here to serve you. I will work to continue to deliver diverse scientific programming through our monthly meetings and beyond; to host career workshops and foster networking opportunities; to support science outreach and inspire young and old alike with the wonders of chemistry; and to recognize excellence in chemistry, be it in teaching, research, or student achievement. This is just a sampling of what NESACS has to offer.

We support going green. Did you know that you can opt for electronic delivery of The Nucleus? Go to: http://www.nesacs.org/home_electronic_delivery.html

We need you. NESACS is a vibrant organization, one that relies on the participation of its members to accomplish its mission, for the advancement of chemistry and chemical engineering, the promotion of research, the improvement of the qualifications of members through high standards of educational and professional ethics, the increase and diffusion of chemical knowledge, and the promotion of scientific interests and inquiry. How can you help? Get involved. Attend a NESACS event. Visit http://www.nesacs.org to learn more about upcoming NESACS activities. Step up and volunteer, as I did – you will see that opportunities abound. Tell me what you would like to see from NESACS. NESACS is your local section. ◇

IUPAC-SOLVAY Award for Young Chemists

Applications for the 2015 IUPAC-SOLVAY International Award for Young Chemists are now being solicited with a deadline of February 1, 2015.

The Award is intended to encourage young research scientists at the beginning of their careers by recognizing outstanding Ph.D. theses in the chemical sciences. Up to five cash prizes of $1,000 each will be awarded together with paid travel expenses to the next IUPAC Congress. In keeping with the status of IUPAC as a global organization, efforts will be made to assure fair geographic distribution of the prizes.

Each awardee will be invited to present a poster about his/her research and to participate in a plenary award session, and is expected to submit a review article for possible publication in Pure and Applied Chemistry.

The 2015 awards will be presented at the 45th IUPAC World Chemistry Congress, August 9-14, 2015, in Busan, Korea; see <www.iupac2015.org>.

More information about the Award can be found at <http://www.iupac.org/news/news-detail/article/2015-iupac-solvay-international-award-for-young-chemists-announced.html>. ◇

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ACS Scholars Program

The ACS Scholars Program, which provides renewable scholarships to underrepresented minority (African American, Hispanic, and American Indian) students in the chemical sciences, is now accepting online applications for the 2015-2016 academic year.

Awards of up to $5,000 (amount depending on individual financial need and available funding) are given to qualified high school seniors or college freshman, sophomores, or juniors who wish to pursue a degree in the chemical sciences or chemical technology.

For complete details and access to the online application system, go to <www.acs.org/scholars>. The deadline for receipt of completed applications and supporting documents is March 1, 2015. ◇

Monthly Meeting

The 947th Meeting of the Northeastern Section of the American Chemical Society

Thursday, January 8, 2015

Amgen, Inc.

360 Binney Street, Cambridge, MA 02142

4:00 pm Annual Board Meeting (Amgen Conference Center)

4:30 pm Monthly Board Meeting (Amgen Conference Center)

5:30 pm Social Hour (Amgen Conference Center)

6:15 pm Dinner

7:15 pm Dr. Katherine Lee, NESACS Chair Presiding
Dr. Christopher J. Doona, US Army-Natick Soldier RDEC
Seminar: "Army Chemistry Helps in the Fight Against Ebola"

RESERVATIONS ARE REQUIRED (for both Dinner AND/or Symposium) BY NOON, WEDNESDAY, DECEMBER 31

To register use PayPal: http://acssymposium.com/paypal.html. Select the pay with credit or debit card option and follow the additional instructions on the page. Cost: Members, $30; Non-members, $35; Retirees, $20; Students, $10. Dinner reservations not cancelled at least 24 hours in advance must be paid.

If you wish to join us for this meeting and not eat dinner, please register by noon, Thursday, January 1. Use PayPal: select "seminar only". The fee is $1.

New members or those seeking additional information, contact the NESACS Administrative Coordinator, Anna Singer, at secretary@nesacs.org (preferred) or at (781) 272-1966, 9 AM - 6 PM. PLEASE DO NOT CALL AFTER HOURS.

THE PUBLIC IS INVITED

Directions to Amgen Massachusetts:
By Public Transportation: Kendall Square is located on the MBTA's Red Line. Amgen-Massachusetts is a five-minute walk from the Kendall/MIT stop.
From the Mass Pike: From US 90E, take Exit 18. Follow the Cambridge signs over the Charles River and take the right at the Mobil station onto Memorial Drive. Continue for 2 miles on Memorial Drive, pass under the Longfellow Bridge and continue onto Edwin Land Blvd. Stay left and follow the signs for Kendall Square.
Parking: One Kendall Square Garage is located on Binney Street directly opposite the entrance to the Kendall Square Courtyard. Follow the signs for Cinema Parking. ◇

Biography

Dr. Christopher Doona serves as Senior Research Chemist for the US Army Natick Soldier Research, Development, and Engineering Center carrying out basic and applied research with colleagues in academia and industry.

Since 1995, Dr. Doona has investigated chemical heating for military rations, novel technologies for food preservation, textile decontamination, and graywater recycling using High Pressure Processing, Ohmic heating, Natick's Intrinsic Chemical Markers, Magnetic Resonance Imaging (MRI), mathematical modeling for Food Safety (featuring Natick's Quasi-chemical model), and "hurdle" technology to develop a 3-year Challenge Test to ensure the safety of military sandwiches.

Dr. Doona earned his undergraduate degree in Biology from Ripon College and Ph.D. in Physical Chemistry from Brandeis University. He has been a Visiting Scientist at Loránd University in Budapest, an NSF Physical Chemist at University of Würzburg (Germany), a Postdoctoral Fellow at Auburn University, and Professor at Middlebury College, teaching 9 courses and mentoring undergraduate Environmental Chemistry research.

Dr. Doona has over 80 peer-reviewed publications, 3 books, and 15 patents, including novel chemical disinfectant technologies being used by international public health organizations to sterilize medical equipment during the current Ebola crisis in West Africa. He is a Professional member of ACS and IFT (Past-Chair of the Nonthermal Processing Division, 2013 Service award winner). ◇

Abstract

The spread of Ebola to the U.S. in recent weeks has intensified the public's concern of this deadly virus, especially among airport screening personnel and healthcare workers simply doing their jobs. The comingle of concern and compassion are nowhere more evident than in West Africa, the epicenter of the Ebola crisis (see link 1 below).

Chemistry and Microbiology researchers at the U.S. Army – Natick Soldier RD&E Center invented an ensemble of power-free, portable chemical decontamination technologies for use in austere environments. Currently, this technology is being used by Doctors without Borders, World Health Organization (WHO), Public Health Canada, and National Institute of Health (NIH-with support from the US government) to sterilize medical equipment and electronics in remote clinical sites where Ebola is prevalent (see link 2 below).

These technologies derive from

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Historical Note

David O. Ham
(1939-2014)

By James L. Kinsey

David Ham, 75, physical chemist, environmental research scientist, and science educator, a resident of Williamsburg, Massachusetts, died suddenly October 15, 2014 in Houston, TX. He was attending a workshop to set up an International Baccalaureate Program at Pioneer Valley Chinese Immersion Charter School (PVCICS), where he taught physics, chemistry and mathematics.

In 1961 Ham graduated with a B.S. degree in chemistry from the University of California, Berkeley, and in 1968 was awarded a Ph.D. from the Department of Chemistry at the Massachusetts Institute of Technology.

From 1970 to 1980 he worked at the University of Rochester, initially as an assistant professor in the Chemistry Department and then as a Senior Research Associate, Laboratory for Laser Energetics and Department of Mechanical and Aerospace Sciences in the College of Engineering. After 10 years in Rochester, Ham spent one year at the National Laboratory in Los Alamos, New Mexico.

In 1980 he resettled in the Boston area, working as a research scientist and as Vice President of Energy and Environmental Sciences for Physical Sciences, Inc. (PSI) in Andover, MA. Subsequently, he founded his own company, Envirochem, Inc., conducting contract research for environmental projects.

Ham wrote a monthly column about global warming for *The Nucleus*. Throughout his career as a research scientist, he maintained his commitment to bringing science to underprivileged youth and his enthusiasm for instilling his love of science in all students.

He is survived by his wife, MaryAnna, of 52 years, his daughters Audrey and Lesley, and a grandson, Wei David. ◇

Summer Research Abroad

The ACS International Research Experience for Undergraduates (IREU), an NSF-sponsored program, will begin accepting applications soon for research in Germany, Italy, Scotland, and Singapore in Summer 2015.

Up to 17 undergraduate students will be chosen to receive a $4,000 stipend and airfare/housing compensation, a pre-departure orientation session in Washington, DC, language training and medical insurance, and travel to the Spring 2016 ACS National Meeting in San Diego, CA, to present the results of the research.

The criteria for student participation in the program are:

- Be a citizen or permanent resident of the United States.
- Be enrolled full-time in a chemistry, materials science, biochemistry, or chemical engineering program at a U.S. university or college.
- Have one semester or summer of prior experience working in a research laboratory outside of normal coursework.
- Be a rising junior or senior by the end of the program with at least one semester of enrollment remaining after the summer.
- Please note that membership in ACS is not a criterion and all students, regardless of membership status, are welcome to apply.

For more information, go to <www.acs.org/ireu>. ◇

For late breaking news, job postings and the latest meeting and event information please visit us at

WWW.NESACS.ORG

Photos from November 2014 Meeting

photos by M.Z. Hoffman

Former colleagues at what was Southeastern Massachusetts University, now the University of Massachusetts Dartmouth: (l-r) Toby Dills, Jim Golen, Thomas Greenbowe (Iowa State University), Don Boerth.

Morton Hoffman (at right) receiving the Arno Heyn Memorial Book Prize from Mike Filosa, Editor of The Nucleus. The book presented with a bookplate honoring Arno Heyn was "Fritz Haber: Chemist, Nobel Laureate, German, Jew" by Dietrich Stoltzenberg.

Thomas Greenbowe (third from the right) with members of his family: (l-r) son Joel, daughter Meagan, wife Lynette, son Benjamin, and daughter-in-law Sue. Daughter Kaitlyn and son-in-law Eric could not attend.

Doris Lewis, Chair, Norris Award Committee (at right) and Myron Simon, 2007 recipient of the Heyn Memorial Book Prize.

Thomas Greenbowe (Iowa State University), at left, with Michael Abraham (University of Oklahoma).

(l-r) Doris Lewis (Suffolk University), Chair, Norris Award Committee; Ruth Tanner (University of Massachusetts Lowell), 2012 NESACS Chair; Kathy Lee (Pfizer), NESACS Chair-Elect.

Ken Mattes, NESACS Archivist and presenter of the history of the Norris Award, at left, with Kathy Lee, NESACS Chair-Elect.

Morton Hoffman (Boston University) with his wife, Sandy.

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Summer Scholar

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428. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 5.41 (s), 7.62 (t), 7.73 (t), 7.92 (d). Analysis by electrospray ionization mass spectrometry (ESI-MS) in 0.05% acetic acid indicates the presence of a $[M]^+$ *m/z* of 191.339, which does not suggest that **3** was synthesized. This is illustrated in Figure 3.

Figure 3: *The unsuccessful synthesis of **3***

Due to the discrepancy between the literature and our experiments, a more comprehensive literature survey into both benzylidene urea and the reaction of benzaldehyde and urea was performed. A paper by Ji[7] was found, which claims that a reaction between p-chlorobenzaldehyde and urea in the presence of p-toluenesulfonic acid in toluene results in the formation of a bisurea, with two equivalents of urea adding to the carbonyl carbon. Also, several papers by Schiff[8] were found, in which he calls the product of the reaction between benzaldehyde and urea benzylidenebiuret. In addition, Das-Gupta[9] reports that benzaldehyde and urea condense to form benzylidenebiuret, which decomposes above 270°C and is practically insoluble in a variety of solvents. A search of *March's Advanced Organic Chemistry* revealed that amides frequently react with aldehydes in the presence of bases or acids to form alkylidene or arylidene bisamides via an acylated amino alcohol[10]. Considering this information, it is possible that benzylidene bisurea was formed to some extent when Juaristi's synthesis was performed—the molecular ion at 191.339 may be the result of a reaction between benzylidene bisurea and acetic acid, resulting in the cyclization of the bisurea and the loss of ammonia, m/z calculated for $C_9H_9O_2N_3$ 191.19. This is summarized in Figure 4. Pandey[11] reports a synthesis of benzylidene bisurea, which was repeated for comparison; however, the material obtained was not identical to the product obtained from Juaristi's synthesis in either IR or NMR. At the present time, it cannot be said with absolute certainty that the product obtained from Juaristi's synthesis is one component, which may be the cause of some of these discrepancies.

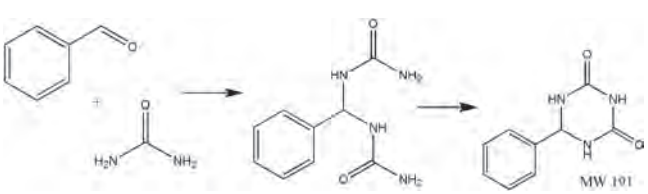

Figure 4: *Reaction of benzaldehyde and urea to produce the cyclic bisurea.*

Reaction of Thiophene-2-carboxaldehyde and Urea

Another avenue considered was the reaction of thiophene-2-carboxaldehyde and urea. Upon refluxing in toluene with p-toluenesulfonic acid as a catalyst, a tan solid was isolated. Thermogravimetric analysis showed that the product is inconsistent with thiophenylidene urea **5**. On the basis of NMR and MS, the compound is assigned the structure **6**. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 5.78 (s, 1H)), 7.01 (t, 1H), 7.08 (s, 1H), 7.53 (t, 1H), 8.28 (s, 2H), 9.46 (s, 1H). ESI-MS: *m/z* calculated for $C_7H_7O_2N_3S$ 197.22, found $[M]^+$ 197.270.

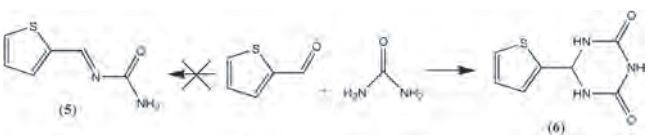

Figure 5: *Reaction of thiophene-2-carboxaldehyde and urea*

Semicarbazones

Another series of compounds with the desired carbon-nitrogen double bond are the semicarbazones, synthesized from the reaction of an aldehyde with semicarbazide hydrochloride in a basic solution[12]. Two semicarbazones were synthesized—benzaldehyde semicarbazone and p-chlorobenzaldehyde semicarbazone, identified by melting temperature and IR spectroscopy. Upon irradiation of the two semicarbazones with a 254 nm uv lamp, it was discovered that there was no apparent change in either melting behavior or in the infrared spectra, indicating the lack of a uv initiated reaction. In addition, the melt was reversible, suggesting that no thermal reaction is taking place either. A search of the Cambridge Structural Database[5] revealed that the double bond of benzaldehyde semicarbazone is not oriented for solid state reactivity.

Further Work

Other avenues of obtaining pure samples of **3** are under consideration. Sahu and Sahu[13] report the synthesis of **3** by using various acidic clays, such as hydrotalcite, an aluminum magnesium hydroxide carbonate hydrate[14], and kaolin, a hydrated aluminum silicate[15]. They offer a reasonable structure proof, including proton and carbon NMR and a molecular ion in the mass spectrum. Repetition of their work would be informative, but the clays have not yet been obtained. Additionally, Lu[16] reports the synthesis of the ethyl ester of **3** using benzaldehyde, urethane, and sodium benzenesulfinate, which may potentially be treated with ammonium hydroxide to yield **3**. A derivative, substituting urea for urethane, may also be attempted to synthesize **3**.

Summary

We have found imesatin and the semicarbazones are unreactive to ultraviolet light for structural reasons. The synthesis of **3** as described by Juaristi is not reproducible. Our efforts to obtain a pure sample of **3** continue.

Acknowledgements

The authors would like to thank Dr. Sammaiah Thota and Dr. Joshna Chittigori for their assistance in obtaining NMR spectra and thermal analyses, Dr. Jin Xu and his students for their assistance in obtaining mass spectra, Mr. James Hall

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SE-MA NESACS Meeting Photos

photos by M.Z. Hoffman

Anna Singer (NESACS Administrative Secretary), at left, and Sandy Hoffman.

From Stonehill College: (l-r) Everton Pacheco, Cheryl Schnitzer, and Taylor Williams.

Listening to Nocera's lecture.

Dan Nocera (Harvard University) giving his talk, "The Artificial Leaf: Personalized Energy for 1 (x 6 Billion)."

Summer Scholar

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for his assistance in providing standard base for the titrations, and Aaron Oakley for his assistance in the reaction of thiophene-2-carboxaldehyde and urea. The authors would also like to thank Dr. Bruce Foxman of Brandeis University for his assistance in searching the Cambridge Structural Database and his support of our structural work. The authors would also like to thank the Northeastern Section of the American Chemical Society for support of this work.

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Abstract

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the controlled chemical production of chlorine dioxide (ClO_2) by unique "effector" reaction chemistry, as demonstrated recently in the Journal of Visualized Experiments (see link 3 below). The Portable Chemical Sterilizer (PCS) is a next-generation field autoclave that sterilizes surgical instruments without power and water in far-forward deployments.

The "Disinfectant-sprayer For Environmentally-friendly Sanitation (D-FENS) is a collapsible handheld sprayer that cleans and disinfects hard surfaces in kitchens, galleys, showers, and latrines. The "Disinfectant for Environmentally-Friendly Decontamination. All-purpose (D-FEND ALL) is used for textiles disinfecting water.

The inventions were licensed to ClorDiSys Solutions (see link 4 below) and commercialized under the product name CHEM-CD. The ontogeny of these technologies from basic research to commercial innovation, as used in West Africa, will be discussed.

Links:

1. http://www.army.mil/article/136641/Natick_plays_key_role_in_helping_to_fight_spread_of_Ebola/
2. http://www.necn.com/news/new-england/Mass-Researchers-Create-Disinfectant-to-Fight-Ebola-280284792.html
3. http://www.jove.com/video/4354/the-portable-chemical-sterilizer-pcs-d-fens-d-fend-all-novel-chlorine
4. www.clordisys.com ◇

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Calendar

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Note also the Chemistry Department web pages for travel directions and updates.
These include:
http://www.bc.edu/schools/cas/chemistry/seminars.html
http://www.bu.edu/chemistry/seminars/
http://www.brandeis.edu/departments/chemistry/events/index.html
http://www.chem.harvard.edu/courses/seminars.php
http://chemcalendar.mit.edu/index.php
http://chem.tufts.edu/seminars.html
http://engineering.tufts.edu/chbe/newsEvents/seminarSeries/index.asp
http://www.chem.umb.edu
http://www.umassd.edu/cas/chemistry/
http://www.uml.edu/Sciences/chemistry/Seminars-and-Colloquia.aspx
http://www.unh.edu/chemistry/events

January 12
Prof. Daniel Siedel (Rutgers University)
Boston University, Metcalf, Rm 113
4:00 pm

January 13
Prof. Doug Stephan (University of Toronto)
Boston College, Merkert 130
4:00 pm

January 15
Prof. Varinder Aggarwal (University of Bristol)
Prof. Cristina Nevado (University of Zurich)
MIT, Room 34-1001
4:00 pm

January 20
Prof. Brian McNaughton (Colorado State)
Boston College, Merkert 130
4:00 pm
Prof. Heather Maynard (University of California, Los Angeles)
Harvard University, Pfizer Lecture Hall
4:15 pm

January 27
Prof. Tom Muir (Princeton University)
Boston College, Merkert 130
4:00 pm
Prof. Brian Stolz (California Institute of Technology)
University of New Hampshire, N104
11:10 am

Notices for The Nucleus Calendar of Seminars should be sent to:
Xavier Herault, email: xherault(at)netzero.net ◇