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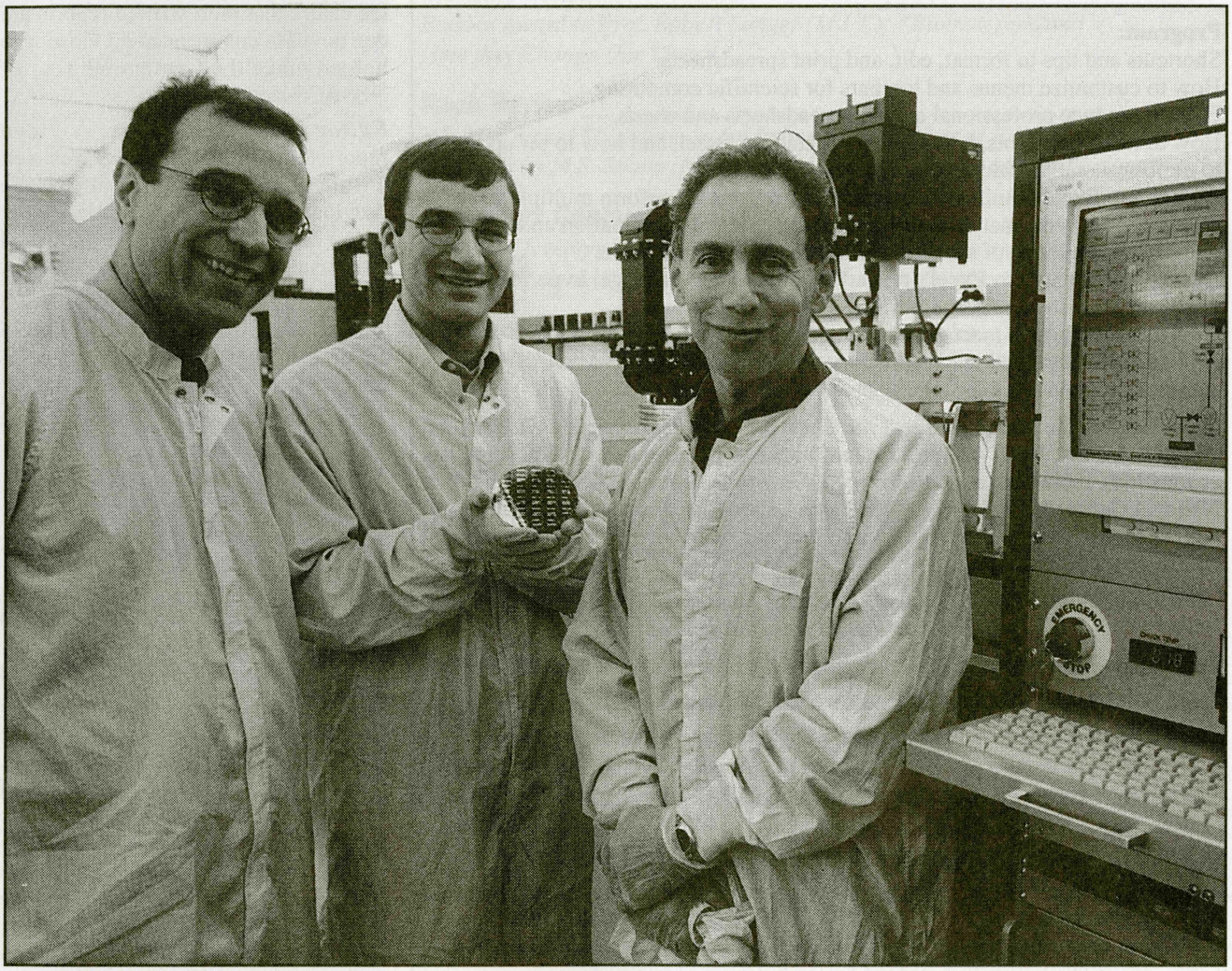
NESACS

THE NUCLEUS

April 1999 *Vol. LXXVII, No. 8*



Monthly Meeting

Esselen Award to Robert Langer

Book Review

Insisting on the Impossible: The Life of Edwin Land

Meeting Report

Robert Langer's February Meeting Talk on his research

Summer Scholar Report

Khai Bui and John C. Warner on Green Chemistry

Microsoft Excel® for Scientists and Engineers

A Two-Day Hands-On Short Course

Instructor: Prof. E. Joseph Billo, Boston College

Thursday, May 13 - Friday, May 14, 1999

8:00 AM - 4:00 PM

Computer Career Institute, 4 California Avenue, Framingham MA

Program:

Shortcuts and tips to format, edit, and print spreadsheets.
How to customize menus and toolbars for scientific computing.
How to produce professional quality spreadsheets and charts.
How to link documents, how to import data into Excel, and how to set up Excel to perform iterative calculations automatically.
How to use Excel's built-in mathematical functions to perform multiple linear regression analysis, matrix mathematics, numerical differentiation and integration, solutions of systems of linear equations, and more.
How to use the Solver, Excel's powerful optimization package, to perform nonlinear least squares curve fitting.
How to create simple macros to automate routine spreadsheet tasks.
How to write advanced macros to carry out complex, repetitive calculations automatically.

Prof. Billo is the author of *Excel for Chemists: A Comprehensive Guide*, John Wiley and Sons, 1997, 480 pp. He has taught this course to over 500 scientists at locations including Amoco, Biogen, Chevron, Eastman Kodak, Genzyme, National Cancer Institute, Naval Research Laboratory, Procter & Gamble, Shell and Texaco. This is the same course that has been offered at ACS National Meetings for $925.

This is not a course for beginners. You must have some familiarity with Excel in order to benefit from this course.

Pre-registration required. Registration is limited to 24 attendees.

Each participant will have his/her own PC workstation.

Registration Fee (add $50 to receive a copy of *Excel for Chemists*):

	On or before Apr. 30	After Apr. 30
A. C. S Members	$375.00	$425.00
Non - A. C. S Members	$475.00	$525.00

For further information: (617) 552-3619 or joseph.billo@bc.edu

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Registration form for **MICROSOFT EXCEL FOR SCIENTISTS**

☐ Check to order a copy of *Excel for Chemists* (add $50 to registration fee)

Name _____ ☐ ACS Member

Mailing Address _____ Tel.: _____

_____ Total enclosed:

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Mail with remittance to: Dr. E. J. Billo, 13 Shattuck Street, Natick MA 01760
(Please make check payable to E. J. Billo)

Board of Directors

NOTE: Board Meetings are held on the Monthly Meeting day at 4:30 p.m. Section members are invited to attend.

Annual Meeting, January 14, 1999

Chair: M. Hearn reminded all committee chairs that their written reports are due now. He commended all those who helped make the Centennial Year of NESACS a complete success.

Editor of the NUCLEUS: A. Heyn reported that the NUCLEUS was well within budget and, compared to the previous year, had increased pages by 20% (268 pages total) and increased advertising by 33.7%.

J. Neumeyer MOVED to commend The NUCLEUS staff for their 1998 accomplishments; VOTED by acclamation.

The meeting was recessed.

Notes of the regular Meeting of January 14, 1999

Officers' Reports:

Chair: D. Rickter, 1999 Chair, presented M. Hearn the NESACS Past-Chair pin and thanked M. Hearn for his efforts as Chair of NESACS in 1998. Chairs for current committees are to be announced shortly.

Chair-Elect: D. Lewis reviewed the speaker line-up for 1999: February: Dr. Langer, who subsequently was selected to receive the Esselen Award; March: Dr. Charles Kolb; April: Esselen Award Meeting; May: Education Night, Dr. Eric Marz (U. Mass-Amherst); September: A joint meeting with the Electrochemical Society is under discussion; October: Madeleine Jacobs (Editor of C&ENews); November: Norris Award for Teaching; December: Joint meeting with Medicinal Chemistry Group.

It was MOVED and VOTED to hold the November Award meeting at the scheduled date of November 11 in spite of the holiday.

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Contents

Cover: *(L. to r.) Prof. Michael Cima, Grad. Student John Santini, Prof. Langer in the MIT Microsystems Technology Laboratory where the chip was fabricated. Mr. Santini holds a silicon wafer containing 21 dime-sized chips for releasing chemicals on demand. (M.I.T. Photo by Donna Coveney)*

Deadlines: *Summer issue: June 11, 1999*

THE NUCLEUS

The Nucleus is distributed to the members of the Northeastern Section of the American Chemical Society, to the secretaries of the Local Sections, and to editors of all local A.C.S. Section publications. Forms close for advertising on the 1st of the month of the preceding issue. Text must be received by the editor six weeks before the date of issue.

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Meeting Report

Report of Prof. Robert S. Langer's talk at the February 11, 1999 meeting of the Northeastern Section

Advances in Drug Delivery and Tissue Engineering

Reported by Dr. Myron S. Simon

This year's recipient of the Gustavus John Esselen Award for Chemistry in the Public Interest opened his bag of good things just a little in February, promising to tell us the full story at the Esselen Award meeting in April. He reported on two major themes from his recent work: methods for delivering drugs either through the skin or by aerosols, and devising ways to grow new organs using polymeric armatures.

Ultrasound Enhanced Delivery of Drugs

The use of transdermal delivery has blossomed in the past twenty years from virtually nothing to a two billion dollar business. The technique had been restricted to low molecular weight, highly lipophilic drugs. Langer wondered whether transfer of molecules through materials such as human skin might be aided by ultrasound. The use of ultrasound had already found use in diagnosis, some therapeutic applications and liposuction. He and his former post doctoral fellow, Joseph Kost, found, using radiolabelled mannitol and insulin and unlabelled physostigmine on rats and guinea pigs, that ultrasound caused enhanced transport of the drugs through skin. Following Langer's early work, in the 80's other laboratories subsequently studied this approach, with mixed results. Success was dependent on the nature of the drug. For example, estradiol worked well.

In 1992 Langer, graduate student Samir Mitragotri, and colleague Daniel Blankenstein began studies to understand the mechanism for the enhanced movement of molecules through skin. They examined four possibilities: 1. Temperature effects. 2. Convection through hair follicles. 3. Mechanical oscillation of lipid bilayers. 4. Cavitation effects on bilayers. Measurement showed that there was no temperature change due to sonophoresis. The role of convection was eliminated by studying drugs with charges and finding no enhancement. Sonophoresis was found to be inversely proportional to ultrasound frequency, suggesting that mechanical effects were not the answer. If cavitation were the answer, Langer and his colleagues reasoned, then there should be a threshold of activity. Indeed, they found that above 2.5 MHz the enhancement disappeared. At 1 MHz a thirteenfold increased flux was observed, while at 3 MHz there was no effect. A second experiment, evacuating gases from the system, led to loss of the effect, another support for the cavitation explanation. Furthermore, skin compression destroyed the effect. The conclusion: The mechanism of sonophoresis consists in cavitation disordering of the skin's lipid bilayers. The model chosen:

Enhancement Rate = f $[D^{l}/D^{b}]$, where D^{l} is the diffusivity through the disordered lipid and D^{b} that through the normal lipid bilayers, and f the fraction of bilayers disordered.

Realizing that cavitation was inversely proportional to the frequency of ultrasound, they reduced the frequency to 20 kHz and were rewarded with an amazing thousandfold enhancement! Many compounds responded to the lower frequency experiments: insulin, γ-interferon, erythropoeitin. The levels of insulin and interferon delivered by sonophoresis were in the therapeutic range when tested on cadaver skin. Testing using a diabetic rat showed that insulin delivery was indeed effective. Histologic experiments proved that the treatment did not damage the skin, and water permeability experiments demonstrated that the skin resumed normal penetration within two hours. Tests on 40 patients using saline showed no ill effects.

Having esablished that sonophoresis can be used for drug delivery, Langer and his colleagues asked whether diagnostic samples could be *removed* by this technique. Indeed, by positioning a reservoir between a rat's skin and the transducer, interstitial fluid could be drawn out and theophylline, glucose, cholesterol, urea, and calcium could be measured (An evacuated reservoir increased the effect.) The low frequency was essential. Studies using the Clark Grid Test showed that this was an acceptable measurement route. Six patients had equivalent glucose measurements by the ultrasound and direct blood sampling methods. Future work will study insulin delivery in diabetic patients. Langer had used an ordinary laboratory sonicator, and he and his colleagues are presently studying the possibility of a very small sonicator that could be worn on a wrist.

Porous Aerosols for Inhalation Therapy

Conventional aerosol treatment, e.g. for asthmatics, is very inefficient. The aerosol particles are small, 3-4 μ, having a density of 1. They tend to

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Monthly Meeting

The 806th Meeting of the Northeastern Section of the American Chemical Society

The Gustavus J. Esselen Award for Chemistry in the Public Interest

Thursday, April 8, 1999

Harvard University

5:30pm Social Hour, Harvard Faculty Club, 20 Quincy St., Cambridge, Mass.
A table of Career Services Literature and Aids will be available

6:30pm Dinner

8:15pm Evening meeting, Harvard Undergraduate Science Center, 1 Oxford St.
Dr. Donald O. Rickter, Chair, presiding
The Gustavus J. Esselen Award Dr. Myron S. Simon
Introduction of Dr. Robert Langer, Dr. Gordana Vunjak- Novakovic, Dr. Prasad Shastri, Massachusetts Institute of Technology
Presentation of the Award to Dr. Langer, Gustavus J. Esselen III
Award Address: *Biomaterials and How They Change Our Lives*

Dinner reservations should be made no later than April 1, noon. Please call or fax Marilou Cashman at 800-872-2054. Reservations not canceled at least 24 hours in advance must be paid. Members, $30.00; Non-members, $35.00; Retirees, $20.00; Students, $8.00. THE PUBLIC IS INVITED.

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

Free Parking: Felton St. Garage (3rd level or higher); enter from Cambridge St.

***Next Meeting: Education Night**: May 13, 1999 at Holiday Inn, 1200 Beacon St., Brookline Mass.: 5:30 Social Hour and Dinner, 8:00 Evening Meeting, Dr. Eric Marz, UMass-Amherst, on molecular visualization.*

Abstract

Biomaterials and How They Change Our Lives

Nearly every one has used some type of biomaterial, whether it be in the form of a dental filling, suture, or something more complex. However, there is an ongoing revolution in the biomedical sciences, as well as in the design of novel biomaterials, that together may enable new therapies that people once would not have thought possible. These include new plastic-based systems that can deliver an extensive array of new therapeutics for prolonged time periods and perhaps right to their intended targets in the human body. Such approaches are already leading to new therapies for certain types of cancer. There is also the exciting possibility of creating human replacement parts, such as new tissues like cartilage or skin by synthesizing the right biomaterials and combining these with the appropriate mammalian cells. This lecture will explore my personal experiences in this field, what has been accomplished at this time, and where I hope the future will lead us. ◇

Biography

Robert Langer is the Kenneth J. Germeshausen Professor of Chemical and Biomedical Engineering at MIT. He received a Bachelor's Degree from Cornell University in 1970 and a Sc.D. from MIT in 1974, both in chemical engineering. Dr. Langer has received honorary doctorates from the ETH (Switzerland) in 1996 and the Technion (Israel) in 1997. Dr. Langer has written 590 articles, 400 abstracts, 350 patents, and has edited 12 books.

Dr. Langer has received over 70 major awards, including the Gairdner Foundation International Award, the Lemelson-MIT prize, the American Chemical Society (ACS) Polymer Chemistry and Applied Polymer Science Awards, Creative Polymer Chemistry Award (ACS, Polymer Division), the Pearlman Memorial Lectureship Award (ACS, Biochemical Technology Division), and the A.I.Ch.E's Walker, Professional Progress, Bioengineering, and Stine Materials Science and Engineering Awards. In 1989, Dr. Langer was elected to the Institute of Medicine, and in 1992 he was elected to both the National Academy of Engineering and to the National Academy of Sciences. He is the only active member of all three United States National Academies. ◇

NESACS Election

For elected positions in 2000

The Nominees' statements will be in the May 1999 *NUCLEUS* along with the ballots. **Be sure to vote!**

ACS Members: If you haven't submitted your 1999 membership dues, the March 29 C&ENews is the last issue you will receive. Pay your dues now (and don't forget to include your optional Local Section Dues; these dues help us maintain a healthy *NUCLEUS*). ◇

Meeting Report

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aggregate, making aerosolization poor. Only 5% of the medication is utilized, and repeated use is required. The bulk of the drug is chewed up by macrophages in the lungs, phagocytosis.

David Edwards, a former postdoctoral fellow in Langer's group, calculated that lower density, larger, porous particles should be a major improvement. The aerodynamics should be better, so the drugs could get to a deep part of the lungs, there should be less aggregation because of less total surface area, and the macrophages would have a more difficult time eating the larger particles. Langer showed a slide of porous particles, and likened them to whiffle balls, pointing out that the same weight of drug would occupy a very much larger space. Spray drying produced these whiffle balls. The porous balls improved drug availabilty from 5% to 60-95%! *In vivo* results were tenfold better. For example, insulin bioavailability was 86%, and the blood sugar remained lower for four days! Clinical work using Estralast (estradiol) and Albuterol have already shown that a single inhalation is effective for three days.

Tissue Engineering

Langer showed a slide of a child dying of liver failure because of a lack of donors. To save the child someone else had to die. The need for liver donors annually in the United States is about 30,000. The supply is about 3,000. The challenge is to find ways to synthesize organs.

Langer explained that if mammalian cells could be made to line up close together, they can reform tissue structures. For example, mammary epithelial cells when lined up can produce milk. The route Langer and his collaborator, Jay Vacanti, take is to put cells close together on synthetic polymers so that they can self-organize. Their approach is to use biodegradable polymers in appropriate configurations to bring the cells into close proximity so that they can engineer a new organ.

The concept is that three-dimensional scaffolds, like a sculptor's armature, can be plastered with cells, and in a bioreactor the cells will go to work and build the appropriate organ, while the armature will biodegrade. The scaffolds must have a huge surface area for their volume to get the necessary cell packing density. Copolymers of lactic and glycolic acids have been found to have too little affinity for some cells. Langer decided to synthesize polymers with the ability to attach a small peptide, ligands containing biological information. A graduate student, Denise Barrera, synthesized a copolymer of lactic acid and lysine, in order to have dangling amino groups on the polymeric backbone. Use of carbodiimide in DMSO and dichloromethane attached appropriate peptides via the amino groups, giving a modified polymer which allowed endothelial cells to stick to and flatten on the polymer surface.

Tony Atala, a urologist, seeded modified polymer tubes with urothelial cells and generated a replacement for an incomplete urethra. Valves have been bioengineered to replace damaged valves that allow urine backflow, by local injection of cells and a polymer matrix. The reverse problem, incontinence, which affects 1-3 million people, 85% women, can be similarly treated by augmenting the weakened tissue. To make replacement heart valves two cell types, endothelial and fibroblast, were employed. Collaborator John Meyer at Children's Hospital has found that after six months the synthetic valves are functional in lambs.

Blood vessels have been another goal. Three millimeter diameter tubes of the modified polymer were cultured with the two types of cells by Laura Niklason, one of Langer's former staff members. She found that using pulsatile radial stress in the bioreactor, mimicking the heartbeat, was more successful than static treatment. The synthesized vessels were similar to normal blood vessels: 50% collagen, high rupture strength, ability to be sutured. When transplanted into pigs the vessels retained the ability to allow

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Book Review

Victor L. McElheny, Insisting on the Impossible: The Life of Edwin Land,

Perseus Books, 1998, 510 pages, 25 black and white illustrations.
ISBN 0-7382-0009-3, $30.

Reprinted with permission from the Journal of Imaging Science and Technology Reports, Feb. 1999.

Reviewed by Myron S. Simon

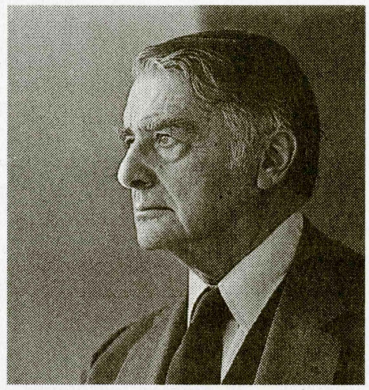

photo: Polaroid Corp.

"Recounting his life is a meditation on the nature of innovation." With this introduction Victor McElheny proceeds to write his biography of Edwin Herbert Land, founder of the Polaroid Corporation, inventive genius, entrepreneur, philanthropist, scientific adviser to presidents. That his subject preferred to limit his recordable life to the papers and actions for which he himself was responsible does not seem to have quenched McElheny's thirty year quest. This book is not the ultimate history of the Polaroid Corporation, but it is probably as good a biography of Land as we are likely to see. If the picture is solely that which Land wished to leave for posterity, perhaps that is far better than a picture distorted by minor, unimportant and even belittling trivia.

McElheny has outlined the scientific work in sufficient detail so that members of NESACS will be able to read, understand and enjoy the science he describes.

The story is assembled in eight major parts, each consisting of two to five chapters.

The first part covers Land's childhood, early influences, the Land-Wheelright Laboratory, the invention of the first sheet polarizer, and the formation of the Polaroid Corporation. His mother's interest in physics and his exposure at summer camp to Barney Girden seem to have been the principal stimulants that led him to the choice of optics and, in particular, polarized light, as his initial field of work. His early attempts to make a sheet polarizer took him from Harvard after the fall term of his freshman year to the reading room of the New York Public Library and experimentation. His first polarizer, made by aligning very small crystals of herapathite in nitrocellulose by a magnetic field, was described in 1929 in a joint patent application with Joseph S. Friedman, an organic chemist, later a historian of photography. The patent was issued in 1933, the first of Land's 535 issued U.S. patents. Back at Harvard in 1929 he continued his experiments. His college career at Harvard as a member of the Class of 1930 was entirely unorthodox, and in 1932 he gave up Harvard to form a joint effort with George Wheelwright to manufacture and sell the polarizer he had invented, now improved by aligning the crystals in the polymer matrix by extrusion. Wheelwright paid him a salary of $2000 a year. (Land had to wait until 1957 to get a Harvard degree, a Doctorate of Science, *causa honoris.*)

Next came the frustrating and ultimately failed attempts to sell the use of polarizers to the automobile industry to solve the night-time glare problem in automobile driving. Land's sheet polarizer made polarizing the headlights and using a polarized filter in front of the driver to eliminate glare practical, and the promise of this approach led Wall Street to fund the Polaroid Corporation. The golden opportunity at the end of World War II to switch over to polarized headlights, when the prewar cars on the road were ready for replacement, came and went. Detroit was not buying.

The story of three dimensional movies started in the thirties when Land and Wheelwright showed their system to Kodak. The Land-Wheelwright Laboratory had already developed a fruitful friendship with Kodak, and the first polarizer sales had been photographic filters sold to Kodak, but the 3-D movies, while exciting to see, were a product which Kodak did not know what to do with. (3-D movies had a brief outing in the fifties and sold very many Polaroid viewers.)

The sale of Polaroid sunglasses, desk lamps and filters for scientific and photographic purposes kept the company breathing during the thirties, but in 1940 the company underwent a big change. The war in Europe led many to recognize that the United States had a role to play. Land devoted the entire company to the work the government required. McElheny quotes Land (p. 127), "We didn't exist for any profit, nor singly for the welfare of our employees, or to provide the consumer market with sunglasses; that had been our start. We now existed for one purpose: to win this war." Land's and Polaroid's efforts in the Second World War led to many valuable products for the war effort: variable density and dark-adaptation goggles, the optical ring sight, 3-D Vectograph for aerial survey and training, plastic optics, heat-homing bombs, Land's support of the Woodward-Doering synthesis of quinine.

The story of instant photography is the story of a company reborn. Land had conceived of the camera during the war, as a new beginning for the post-war Polaroid. By stripping down the war-swollen population of the company and conserving capital wherever he could, Land managed to get the

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Meeting Report

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the flow of blood.

Other outgrowth from this work has been the preparation of synthetic skin which has already won FDA approval. Other areas of research include cartilage and nerve replacements. ◇

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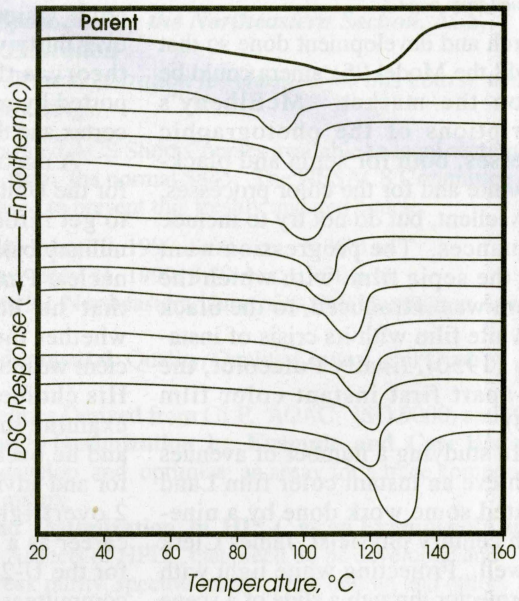

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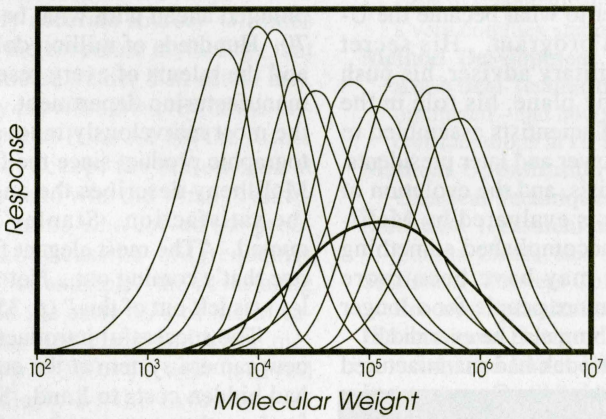

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Book Review

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research and development done so that in 1948 the Model 95 camera could be put on the market. McElheny's descriptions of the photographic processes, both for sepia and black-and-white and for the color processes, are excellent, but do not try to include the nuances. The progression went from the sepia film, with which the camera was introduced, to the black and white film with its crisis of instability (1950), finally Polacolor, the peel-apart first instant color film (1963).

In studying a number of avenues to achieve an instant color film Land repeated some work done by a nineteenth century physicist, James Clerk Maxwell. Projecting white light with one projector through a slide of a scene taken with a green filter, and projecting red light with a second projector through a slide of the same scene taken through a red filter gave "a remarkably extensive palette of colors" rather than the pink monochrome traditional color theory would have predicted. The experiment began Land's most important fundamental research program, to understand and develop a theory of color vision. The pursuit continued over thirty years, and led to his retinex theory, a theory that has been supported by recent studies on the visual cortex and the brain.

A major problem of the Cold War for the United States was our inability to get information about the Soviet military build-up. Eisenhower feared a nuclear Pearl Harbor, and recognized that he had no way of knowing whether the United States had sufficient weapons to deter any such attack. His choice of civilian scientists to examine the problem drew Land in, and he became a leading spokesman for and adviser to what became the U-2 overflights program. His secret career as a military adviser, his push for the U-2 spy plane, his role in the committees of scientists assembled to advise Eisenhower and later presidents, the Sputnik crisis, and the evolution of spy satellites is evaluated by McElheny: "Land accomplished something secretly that may have been more important for more people for a longer time than anything else he ever did."

Eastman Kodak had manufactured the negative for the Polacolor film from the start. There was no alternate supplier. When Polaroid's growth seemed to a new Kodak management team to threaten Kodak's preeminent position in photography, a challenge was made: Kodak would cease to provide negative unless allowed to sell an instant peel-apart film under its own label. Land had two arrows in his quiver to meet this threat. One was the work being done at Polaroid, Project Cardinal, learning to manufacture his own negative. The second was his dream of a new medium which would be truly one-step photography. Agreeing in 1969 to let Kodak sell its own peel-apart product in 1975, Land plunged ahead with what became SX-70. Hundreds of million dollars later, and the talents of every research and manufacturing department, produced the most marvelously innovative photographic product since the first Leica. McElheny describes the anguish and the satisfaction. Stanley Bloom is quoted, "The most elegant form is the one that's coming out....Not a hell of a lot was left out of this." (p. 356)

The successful introduction of the new camera system at the end of 1972 had hidden costs to Land. Sales were far less than expected, and by 1974 shares of the company had fallen drastically. He lost the unconditional support of his board of directors and the presidency to William McCune in 1975, and when his next great effort, instant movies, Polavision, failed to sell, in 1980 Land was forced to give up his position as CEO and Director of Research and he resigned from Polaroid. The Rowland Institute for Science which he had established in 1979 was his parachute.

At a forum, "The Future of Industrial Research", in 1944, Land had enunciated his vision of how research should be carried out in an industrial laboratory: Fifty scientists "inspired by curiosity", aware of recent advances in almost any field, "determined to make something useful" could "invent and develop an important new field in about two years." Their patented inventions would support their laboratory. Perhaps this was the paradigm

Book Review

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for the Rowland Institute. Land took his color vision research and a number of Polaroid employees with him to the Institute and went back to fundamental scientific work.

Land made a final gift to the Polaroid Corporation, his defense of Polaroid patents against Kodak in the famous Polaroid *vs.* Kodak lawsuit. The outcome was that Kodak was forced out of the instant photography business and required to pay $925,000,000.

The book, although heavily researched, cannot be accepted as a historically accurate source. Ofttimes it reflects the corporate mythology that Land promoted, rather than actual fact. McElheny provides a survey of the scientific work at Polaroid but the reader should not accept the statements at face value as to who did what. Names of the inventors who solved the problems under discussion are frequently omitted. For example, the devastating problem in the early 50's of the instability of the first black-and-white pictures (pp. 209) was solved commercially by the invention by Howard Haas of a stabilizing polymeric fluid. Lloyd Taylor made major contributions to Polaroid photography, both black and white and color. His polymeric interlayers in the negative and temperature-independent timing layers in the positive were key to the success of the SX-70 film, and his contributions to coating problems made the positive manufacturable (p.354).

Those men are barely mentioned (p.236-7). The commercially successful "chemical curtain" which protected SX-70 pictures while they were developing in the light, McElheny's "perhaps the second greatest invention made by a Land associate..." (p.368) was invented by Myron Simon. Simon's step-by-step solutions of the problems inherent in the use of phthalein dyes irritated Land, who had to wait longer than he liked for a successful opacification system. (The latest opacification system uses the most

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ACS SHORT COURSE

Methods Development, Validation Procedures, and Conformity Assessment in the Analytical Laboratory

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Save more than $500 over the registration fee charged for this course when it is offered at ACS National Meetings.

National ACS is making top-rated ACS Short Courses available to local sections at tuition fees greatly reduced from the normal $845. The NESACS Committee on Continuing Education is pleased to present this freshly updated course.

DATES and TIME: Monday, May 10, 1999, 8:00 a.m. - 5:30 p.m.
and Tuesday, May 11, 1999, 8:30 a.m. - 5:30 p.m.

PLACE: Snell Library, Room 90, Northeastern University, 360 Huntington Ave., Boston, MA

PROGRAM AGENDA: **Fundamental**: Quality, Quality Control, and Quality Assurance

Method Validation: Guidelines Derived from GLP, AOAC, ISO 9000, and ICH

Method Development and Optimization by Example and Case Histories: How to systematically develop and optimize an assay for a trace component in a very complex sample matrix

Method Development and Optimization in HPLC as an Example of Current Analytical Technique: Aspects of HPLC that need to be developed and optimized - and how; Peak purity, spectral match, peak tailing, and other considerations in HPLC

Methods Optimization Considerations in Spectroscopic (UV-VIS, AA) and Classical Techniques

Statistical Treatment of Analytical Data - Mean, Mode, Standard Deviation, Control Charts, etc.

Statistical Process Control (SPC) Applications for Process Improvement and Process Optimization

International Harmonization Efforts and Their Relevance to Current Analytical Issues and Trends

Instructor: Dr. Shib Mookherjea, an international speaker on issues related to quality assurance and validation, has extensive experience in R&D, quality assurance, and quality management in major pharmaceutical and chemical industries. He has held positions in analytical research, QA, and R&D support at Johnson & Johnson, Colgate-Palmolive, and BASF Corporation over the past 25 years. He also has several years of experience in academic institutions and government agencies. He has participated in numerous task forces and subcommittees on laboratory quality assurance and ISO 9000 and has authored numerous technical papers, articles and reports.

Pre-registration Required - Registration Fees:

ACS Members if received before April 23 .....$325.00; after April 23 .....$375.00
non-ACS Members if received before April 23 .....$425.00; after April 23.....$475.00

There will be a limited number of scholarships for unemployed ACS Members on a space-available basis.

Parking Fee $6.00/day **University cafeterias will be available for lunches.**

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Prof. Alfred Viola, Chair
NESACS Committee on Cont. Ed.
Department of Chemistry
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Boston, MA 02115

Book Review

continued from page 11

recent of Simon's and David Waller's phthaleins.) The structure behind the instant movies (p.410) and instant slides owed much to the work of William Ryan and Vivian Walworth twenty years earlier. That these were omitted from the Black and White and Collaborators or Instant Movies chapters is probably more a shield for the corporate mythology than a failing of McElheny. Still, a skillful reading of Polaroid patents, admittedly a major undertaking, would have led to a better understanding of the staffs on which Land leaned.

A major weakness of the book is that at no time does the reader experience an understanding of the spirit, the joy, the excitement of working with Land.

Land was outstanding in so many areas: his scientific genius was not limited to optics, his chosen field of physics. He was a charismatic leader of people. He had the ability to choose and train people to do the work just the way he wanted it done, the ability to select people to fill in the voids in his own scientific background, and the understanding of the need for and attempt to carry out a revolution in corporate structure. Withal that, he was very much aware of the fact that he would be judged by others, and he was determined to control that judgment; witness his reluctance to allow a biography to be researched, witness his insistence that his papers be destroyed after his death.

McElheny took on an impossible task, writing the biography of a perceptive man who was desperately interested in protecting his privacy while at the same time providing a public image that could stand the rigors of the future. Did McElheny succeed in portraying Edwin H. Land? Maybe not; but what he did was provide us with Land's version of Land, that public image, in a very well documented form. We owe him much for that.

Myron S. Simon, Ph.D.,
Research Fellow, Emeritus,
Polaroid Corporation ◇

Historical Notes

We continue the biographical sketches of chemical scientists whose deaths have been reported to us.

by Edward R. Atkinson, Amherst, MA

Anthony Mulone, Sr., 80, died of leukemia on May 15, 1998. He was a Boston native, and a chemistry major at Northeastern University. For service as a U.S. Army medic in the Philippines during World War II he received the Bronze Star, the Purple Heart, and the Medal of Honor. After a 40 year career as plant manager for the Polaroid Corporation he became active in volunteer work with the United Way, the Service Corporation of Retired Executives (SCORE), the Polaroid retiree association, where he served as president, and an association for parents of deaf children. He was active in yacht clubs in Winthrop and Boston and in the Coast Guard Auxiliary. ◇

Member News

Jeremy R. Knowles of Harvard University, Chemistry Department and Dean of the Faculty of Arts & Sciences, received the ACS Nakanishi Prize for his solution of biochemical problems – how enzymes accelerate reactions with such astonishing efficiency and specificity.

Robert S. Langer, our February speaker, is to receive the ACS Award in Polymer Chemistry. The report of his talk in this *NUCLEUS* describes his work.

Hearty congratulations to these distinguished award recipients. ◇

Summer Scholar Report

Green Chemistry: Deoxygenation of Organic Oxides via Reactions with Benzaldoximes.

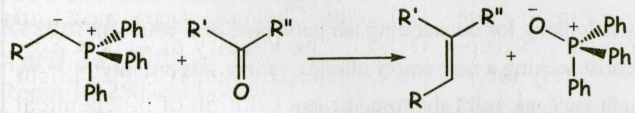
A Progress Report

By Khai Bui* and John C. Warner, Department of Chemistry, University of Massachusetts Boston

Scientists and engineers from both the chemical industry and the academic world have made a great deal of progress in reducing pollution by designing new and efficient methods of waste control and treatment. The environment, and society, have certainly benefited from the efforts of these individuals. In addition to these waste treatment techniques, there has been a growing effort in a new focus of pollution prevention called Green Chemistry.

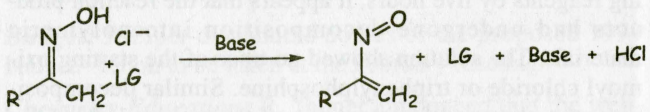
Green Chemistry is a philosophy that expands the efforts of pollution prevention to involve synthetic chemists. The philosophy of preventing pollution at the molecular level through designing more efficient and environmentally benign chemical processes, aims to lessen the burden of those who must deal with hazardous waste by generating less in the first place. Inspired by the concept of Green Chemistry,[1] I decided to work on a research project under the direction of Professor John C. Warner at the University of Massachusetts Boston with the hope that we will contribute to the mission of bringing about a cleaner and better living environment. More specifically, we are interested in inventing methods for reducing organic wastes by designing environmentally benign reactions.

There are many industrial processes that generate organic oxides as stoichiometric byproducts. For example the Wittig reaction[2] is a standard method for the preparation of alkenes from aldehydes or ketones. This reaction often generates the olefin product in very high yield and purity.

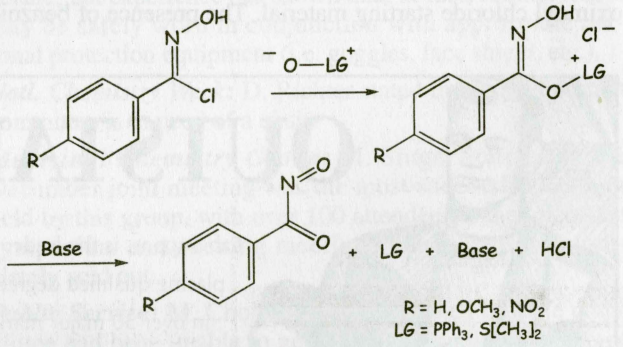


From the perspective of atom economy,[3] however, this reaction is not very efficient. For every molecule of product produced, one molecule of triphenylphosphine oxide is also generated. This by-product material must ultimately be dealt with somehow. The processing of organic waste, in addition to obvious environmental problems, has economic consequences involving regulatory compliance and other handling and treatment costs. In the case of the Wittig reaction, triphenylphosphine is the reagent required to prepare the initial phosphonium ylide. We are interested in designing an effective deoxygenation scheme so that triphenylphosphine may be regenerated from the triphenylphosphine oxide.

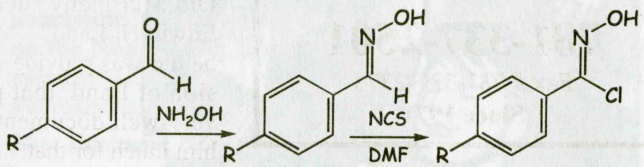
One of the mechanisms that we are interested in using is based on the known elimination of β-substituted oximes under alkaline conditions.[4]



We would like to extend this reaction where the methylene group adjacent to the oxime is replaced by an oxygen atom (ultimately from the organic oxide in our case). In order to explore the feasibility of this process we wanted to investigate the reaction of various substituted benzaldoximoyl chlorides with the organic oxides. It is hoped that displacement of the chlorine by the organic oxide will lead to the oxonium-oxime intermediate. Base promoted 1,4-elimination should give rise to the deoxygenated species, triphenylphophine or dimethyl sulfide and the reactive acyl nitroso intermediates.[5]



In order to explore the experimental parameters of this reaction, we chose to study the impact of substitution of the benzaldoxime, by using the electron-rich methoxy, electron-deficient nitro and unsubstituted benzaldehyde as starting materials. The oximoyl chlorides were synthesized by chlorination of the respective oximes.[6] The oximes were obtained by simple condensation of the substituted benzaldehyde with hydroxylamine .[7]



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*1998 Norris/Richards Summer Scholar

Summer Scholar

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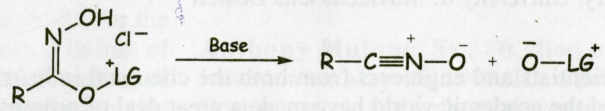
The substituted oximoyl chlorides were refluxed with a molar equivalent of triphenylphosphine oxide in dimethyformamide under nitrogen. The reactions were monitored by TLC. Although TLC showed the disappearances of starting reagents by five hours, it appears that the reaction products had undergone decomposition into polymeric materials. The solution showed no trace of the starting oximoyl chloride or triphenylphosphine. Similar decomposition was observed for reactions of the oximoyl chlorides with molar equivalents of dimethyl sulfoxide in dimethyformamide.

Assuming that the temperature of refluxing dimethylformamide (bp 153° C) was too high for this reaction, we chose to try refluxing acetonitrile (bp 82° C) as the reaction medium. Equimolar equivalents of benzaldoximoyl chloride and dimethyl sulfoxide were heated at reflux in acetonitrile under nitrogen. After approximately 6 hours of reflux, a white solid began to precipitate from the hot solution. This solid continues to form over an additional 18 hours of reflux. The solid (sublimes at 340° C) was identified as ammonium chloride. Repeat reactions confirmed production of this material in approximately 50% yield based on oximoyl chloride starting material. The presence of benzoic acid in the reaction mixture can be detected by TLC.

We continue to explore this reaction. Preliminary experiments replacing acetonitrile with toluene appear to lead to similar products. This seems to rule out acetonitrile as a nitrogen source for the ammonium chloride. Further studies characterizing the reaction products with both solvents are currently underway. Aware of the potential production of nitrile oxides under these conditions,[8] experiments to detect its formation are being carried out.



Other plans include "blocking" 1,4-elimination by using O-methyloximoyl chlorides.[9] We hope that by elucidating this mechanism we will be able to continue our original plans to exploit this reaction as a method to deoxygenate organic oxides.

References:

[1] Anastas, P. T.; Warner, J. C., *Green Chemistry: Theory and Practice*, Oxford University Press, Oxford, 1998.

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Summer Scholar

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[4] (a) Hassner, A.; Alexanian, V., *J Org. Chem.* **1979**, *44*, 3861.
(b) Baczynskyj, L. et al., *J Org. Chem.* **1972**, *37*, 4104.
(c) Boggs, R. A. et al., *J. Chem. Soc., Perkin Trans 2* **1992**, 1271.
[5] (a) Kirby, G. W.; Mackinnon, J. W. M., *J Chem. Soc. Chem. Comm.* **1977**, 23.
(b) Corrie, J. E. T. et al., *Chem. Soc. Perkin Trans. 1* **1985**, 883.
(c) Corrie, J. E. T. et al, . *J. Chem. Soc. Perkin Trans. 1* **1982**,1571.
[6] Armand, J., *Bull. Chim. Soc. France.* **1966**, *5*, 882.
[7] Liu, K.C. et al., *J Org. Chem.* **1980**, *45*, 3916.
[8] Grundmann, C. and Grunanger, P., *The Nitrile Oxides*, Springer-Verlag, New York, 1971.
[9] Johnson. J. E. et al., *J Org. Chem.* **1996**, *61*, 45. ◇

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For more information, schedules, etc.: http://chem.tufts.edu/nsycc/nscrc.html or: Tel: 617-627-3123 or -3046

Board of Directors

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Treasurer: The Treasurer's report was APPROVED as presented. The Section is in sound financial shape.

Standing Committees:

Bd. Of Publications-NUCLEUS: A. Heyn reported that the February issue will be 24 pages.

Budget: J. Piper distributed copies of the proposed 1999 budget. Action to be taken at the February meeting.

Chemistry Education: R. Tanner announced that the well-attended organizing meeting of the Younger Chemists Committee (YCC) was taking place this afternoon.The April meeting of the Northeast Student Chemistry Research Conference is to be held April 24 (at M.I.T. as later announced).

Other Committees:

Safety: J. Kaufman announced that the next free safety seminars for students will be held at Simmons College on January 16, and Boston College on February 13. The Committee welcomes offers for housing future student safety seminars. M. Solstad stated that the ACS Division of Chemical Health and Safety has changed its stand on the use of contact lenses in the laboratory. Formerly this was considered unsafe, but experience has shown that, in fact, contact lenses may be safely worn in conjunction with appropriate personal protection equipment (i.e. goggles, face shield, etc.).

Natl. Chemistry Week: D. Rickter stated that this important committee is in need of a chair.

Medicinal Chemistry Group: M. Singer stated that the December joint meeting was the most successful meeting held by this group, with over 100 attendees at the afternoon symposium and evening meeting. The dinner was completely sold out.

Public Service: M. Chorghade. He regretted that the committee had been unable to get sufficient media coverage of the August National ACS Meeting in Boston. Efforts for better media coverage will continue. The committee plans to organize afternoon meetings at various companies in the area. He mentioned that many companies, while willing to pay national ACS dues of their professional employees, are reluctant to do so for local dues since these are voluntary dues.

Old Business: The Nominating Committee presented Danielle Simonelli for the Board's consideration for filling the vacancy in the office of Director-at-Large. The Board unanimously APPROVED this nomination. Two positions on the Nominating Committee, to be filled with and by members of the Board of Directors are to be elected in at the February meeting.

New Business: None.

Minutes prepared by secretary pro-tem Dr. Michael Singer. ◇

Puzzle Column

WATER TREATMENT

By Paris Svoronos

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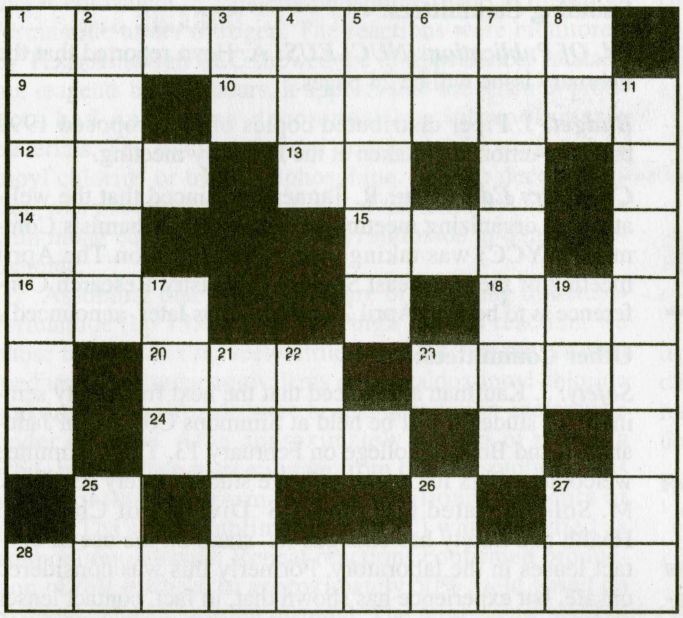

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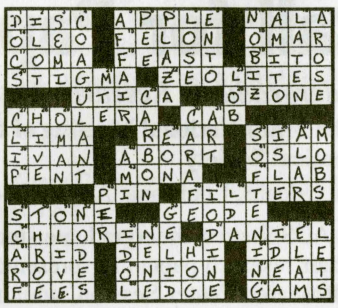
9. Noble gas
10. The product of the reaction of a 2° amine and a carbonyl compound
12. ____ addition: The hydrogenation of an alkene is such a reaction
13. Noble gas (reversed)
14. State where Oak Ridge Labs are located
15. Human organ that deals with sonography
16. Electric snake
18. Suffix of alkenes
20. Letters following C
23. Tautomerizes to a ketone or aldehyde
24. Opposite of 12 across
27. Hybridization seen in alkynes
28. Groups that shift the UV and visible bands of chromophores

DOWN

1. 19th century French scientist
2. Benzyne is such a species
3. The heteroatom in selenophene
4. Same as 18 across
5. ____nickel: reduces carbonyls to alkanes
6. Two-thirds of rim
7. Nitrogen analog of carbene
8. Element atomic number 49
11. Puckered conformation
17. Reagent used in alpha-substitutions of carbonyls
19. Negative answer
21. Prefix in bicyclo compounds that indicates the position of a substituent
22. Fourier Transform
25. Element atomic number 44
26. Same as 9 across
27. Element atomic number 62

Solution For March



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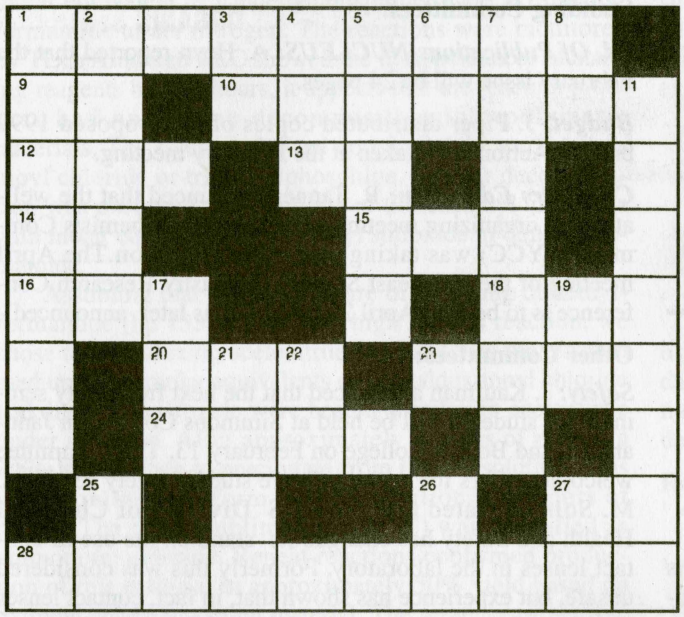

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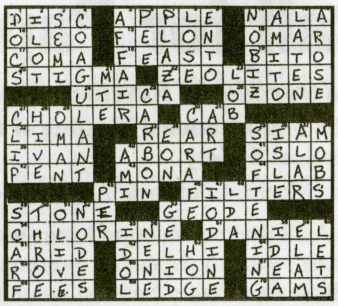

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Mar. 22

Prof. Glen Martyna (Univ. Indiana)
"Computer Simulation of Conformational Equilibrium in Peptides and Proteins"
Boston University
Science Ctr. Auditorium, at 4 PM

Bristol-Myers Squibb Symposium
Prof. Masakatsu Shibasaki (Univ. Tokyo)
"Recent Progress in Multifunctional Asymmetric Catalysis"
Dr. Scott Biller (Bristol-Myers Squibb)
"The Interplay of Human Genetics, Serendipity and Design, Leading to a Potential New Approach to Lipid Lowering Therapy"
Harvard Univ.
Pfizer Lecture Hall, at 3:00 PM

Mar. 22 and 23

Humic Substances Seminar III
Northeastern University
Raytheon Amphitheater, 8:30 AM-6:00 PM
Information at 617-373-7988, registration fee

Mar. 23

Prof. Bibhuti N. Singh (SUNY Syracuse)
"Lipophosphoglycans in Trichonomads"
Boston Glycobiology Discussion Group
MIT Faculty Club, at 6:30 PM
For dinner reservations, call (781) 642-0025

Prof. Louis A. Carpino (UMass, Amherst)
"A New Family of Base- and Nucleophile-sensitive Amino-protecting Groups"
UMass Boston, Harbor Campus
Science Bldg, 1st Fl, Rm. 089, at 4:30 PM

Mar. 29

Prof. James McCusker (Univ. Cal., Berkeley)
"Electronic Structure Effects on the Photo-induced Dynamics of Transition Metal Complexes"
Boston University
Science Ctr. Auditorium, at 4 PM

Mar. 30

Prof. Janice Hicks (Georgetown Univ.)
"Ice Surface Chemistry Relevant to Stratospheric Ozone Depletion Studied by Surface Second Harmonic Generation"
Tufts University
Pearson Hall, Room 106, at 4:30 PM

Mar 31

Prof. Scott Sieburth (SUNY, Stony Brook)
"Organosilanes: From Reagents to Pharmaceuticals"
UMass, Dartmouth
Science & Eng. Bldg., Rm. 305, at 4 PM

Apr. 1

Prof. Chris Miller (Brandeis Univ.)
Title TBA
Mass. Inst. of Technology
Room 6-120, at 4 PM

Dr. S.-R. Holmes-Farley (GelTex Pharmaceuticals)
"Novel Polymeric Pharmaceuticals: From Startup to Market"
UMass, Lowell
Olney Hall, Rm. 428 at 3:30 PM

Apr. 5

Prof. Ken Dill (Univ. Cal., San Francisco)
"Sightseeing on the Energy Landscapes of Protein Folding"
Boston University
Science Ctr. Auditorium, at 4 PM

Prof. Craig Forsyth (Univ. Minnesota)
"Synthesis of Cytologically Interesting Natural and Non-natural Products"
Harvard Univ.
Pfizer Lecture Hall, at 4:15 PM

Apr. 6

Prof. Giacinto Scoles (Princeton Univ.)
"Using Helium Atom Reflectivity to Study Adsorption on Metallic Surfaces"
Tufts University
Pearson Hall, Room 106, at 4:30 PM

Prof. Leverett Zompa (UMass, Boston)
"Metal Complexes of 'Earmuff' Ligands"
UMass Boston, Harbor Campus
Science Bldg, 1st Fl, Rm. 089, at 4:30 PM

Apr. 7

Prof. Craig Hill
"Self Repairing and Buffering Catalysts: Selective Biomimetic O_2-Based Delignification"
Mass. Inst. of Technology
Room 6-120, at 4 PM

Prof. Timo Ovaska (Conn. College)
"Intramolecular Cyclization-Claisen Rearrangement Sequence: A Novel Approach to the Tetracyclic Ring System of Phorbol"
UMass, Dartmouth
Science & Eng. Bldg., Rm. 305, at 4 PM

Apr. 13

Prof. Michael Sailor (Univ. Cal., San Diego)
"Nanocrystalline Porous Si: Luminescent Materials and Chemical Sensors"
Tufts University
Pearson Hall, Room 106, at 4:30 PM

Apr. 16

Prof. Melissa Moore (Brandeis Univ.)
Title TBA
Boston College
Merkert Chem. Ctr., Rm. 127, at 4 PM

Apr. 19

Prof. Michael Fayer (Stanford Univ.)
"Protein Dynamics of Myoglobin: Ultrafast Infrared Vibrational Echo Experiments"
Boston College
Merkert Chem. Ctr., Rm. 127, at 4 PM

Prof. Shu Kobayashi (Univ. Tokyo)
"New Dimensions of Organic Synthesis Toward the 21st Century"
Harvard Univ.
Pfizer Lecture Hall, at 4:15 PM

Apr. 20

Prof. Gregory Verdine (Harvard Univ.)
"The Secret Life of the Genome: Enzymatic Processing of DNA"
Tufts University
Pearson Hall, Room 106, at 4:30 PM

Prof. James Pavlik (Worcester Polytechnic Inst.)
"Photochemistry of Thiazoles and Isothiazoles"
UMass Boston, Harbor Campus
Science Bldg, 1st Fl, Rm. 089, at 4:30 PM

Notices for the Nucleus Calendar should be sent to:

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