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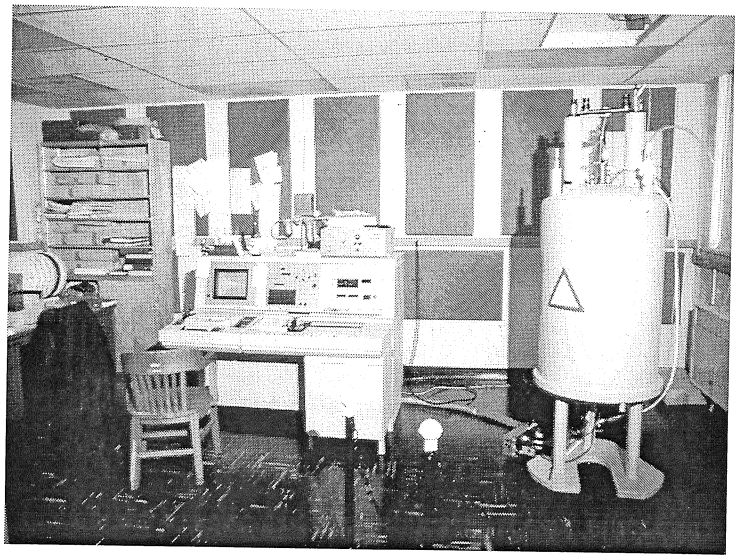
OSU Chemistry Newsletter

Volume 27- Fall 2007

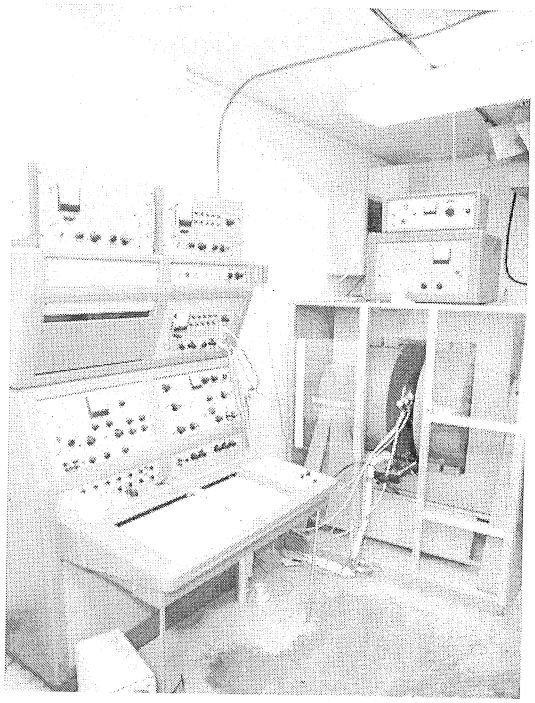
A Day in the Life of the HA-100 NMR

Obtaining a spectrum on the current Fourier transform AVANCE 400 compared to the continuous wave HA-100 NMR is like flying a jet fighter aircraft compared to an open cockpit biplane. For those of you that have not had the chance to play with Nuclear Magnetic Resonance (NMR) spectrometers, I will describe a few terms used in obtaining a NMR spectrum.

All NMR magnets are rated by the **frequency** of the proton spectrum which is directly related to the strength of the magnetic field (read hydrogen here as NMR jocks and organic chemists always run a proton spectrum, never a hydrogen spectrum). For a given magnetic field, the protons will **resonant** at a particular frequency. Thus the AVANCE 400 has the proton spectrum at 400 MHz and the HA-100 has the proton spectrum at 100 MHz. To produce a spectrum, you must either hold the detector at a fixed frequency and sweep the magnetic field or keep the magnetic field constant and sweep the detector frequency to bring the protons into **resonance**. The output of the detector is plotted on the vertical axis of a sheet of paper while the horizontal axis relates to frequency. At this point, you would think that all the protons (remember hydrogen's in the organic molecule) resonant at the exactly the same frequency. Fortunately for the organic chemists, each proton of a particular type will experience a slightly different magnetic field influenced by the location in the molecule and therefore the spectrum will be a series of resonances giving clues as to the structure of the molecule. The term **chemical shift** is used to describe the fact that methyl protons will be located in one part of the spectrum while methylene protons will be located in another part of the spectrum. One could make a list of these frequencies but the values will be dependent on the strength of the main field resulting in a unique list for each NMR magnet in use. The universal solution to this problem is to divide the observed resonances by the base



NMR 400



HA-100

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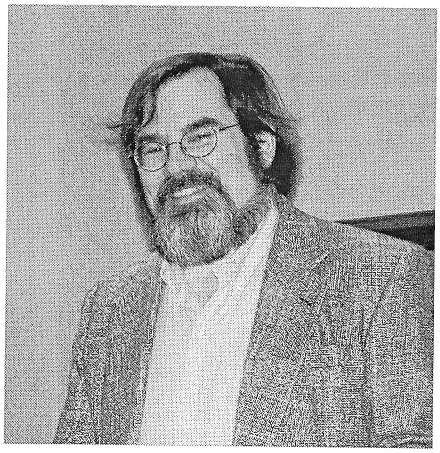

The new academic year begins with a number of major developments. It is a pleasure to announce that by all appearances, we will be getting new space in a joint Chemistry/Linus Pauling Institute building. The Oregon Legislature has committed more than $30 million in funding, and we are well on the way to raising a matching amount from private sources. We are currently in the process of selecting an architectural firm and are confident in our ability to break ground in the near future.

This new building is expected to encompass 120,000 sq. ft. of new space, and will be located west of Nash Hall and the Agricultural and Life Sciences building near 30th Street. Given the very recent developments, we are still in deep consultation about how best to use this. There will be a mix of research and instructional areas, and we want to use this in as strategic a manner as possible to guide the future growth of the Department. It's worth noting that this is the first new space the Department has seen since the construction of Gilbert Addition in 1980. Notably, the University has made several recent investments in renovating labs in Gilbert Hall that we expect will allow us to maintain work here until we can move the remainder of the Department to a hoped-for Phase 2 building.

A second landmark achievement has been to secure funding for a new Nuclear Magnetic Resonance (NMR) instrument, thanks in large part to efforts by Professor Rich Carter. We expect to get a 700 MHz instrument with a cryoprobe that will be the most sensitive instrument in the world for measurement of ^{13}C spectra. You will see inside this newsletter a history of the NMR facility. It's interesting to note that we have often been able to obtain what at the time was cutting edge, state-of-the-art equipment for structure elucidation of organic molecules; we are clearly staying on that path.

Of course, the most important aspect of the Department remains its people. We granted 35 bachelors' degrees, and 15 masters' and Ph. D. degrees in the year ending June 30. Both the undergraduate and graduate programs remain very strong, with the Ph. D. program being the largest on campus. Our best students earned eleven undergraduate scholarships, including four prestigious Hach Scholarships for students in the Chemical Education option. We have sent several of these award winners into the high school teaching ranks over the past couple years, and this pipeline looks to remain healthy.

The faculty remain highly productive, being near the top of University rankings in both instructional service provided and in research dollars raised. We boasted the largest summer program on campus in 2006, and continue to have more than half the student population come through our doors at some point in their academic careers. Professor Doug Keszler was recognized as OSU Distinguished Professor for his groundbreaking work in solid state materials. Professor Walt Loveland was coauthor for a textbook selected as "Best Undergraduate Textbook" by the Association of American Publishers. Professor Staci Simonich received the Savery Award as an Outstanding Young Faculty in the College of Agricultural Sciences.

There are a number of changes in the faces of the Department. Jennifer Travers left us in May. As of this writing we are interviewing candidates for a replacement. Staff members Leah Bandstra and Kristal Young left to pursue different opportunities. While the Chem Stores position is not yet filled, we have welcomed Mary Mucia to the main office. We are continuing efforts to add research faculty.

As always, I hope you find items of interest in this newsletter. I am happy to leave you with my sense that the Department remains a place of vibrant intellectual activity, and that our future is looking every bit as exciting. I hope you are finding success and fulfillment in your activities. And I hope you have a chance to visit over the next few years as we make some major transitions.

Kevin

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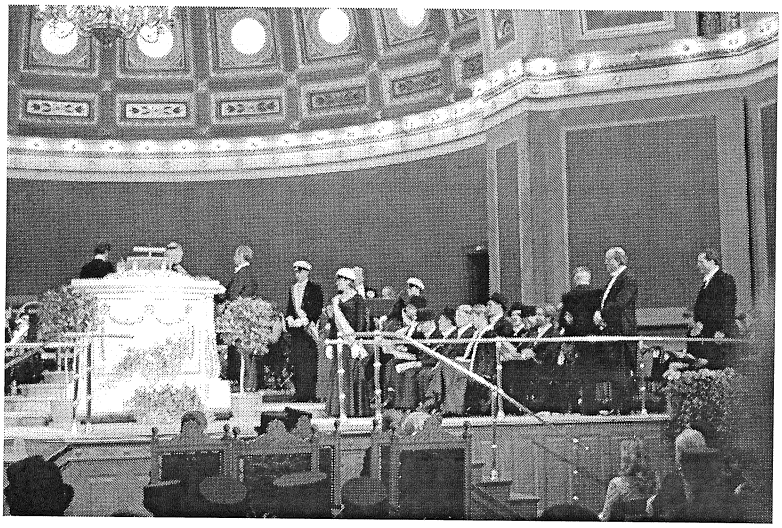
Return Labels

Barofsky receives another PhD

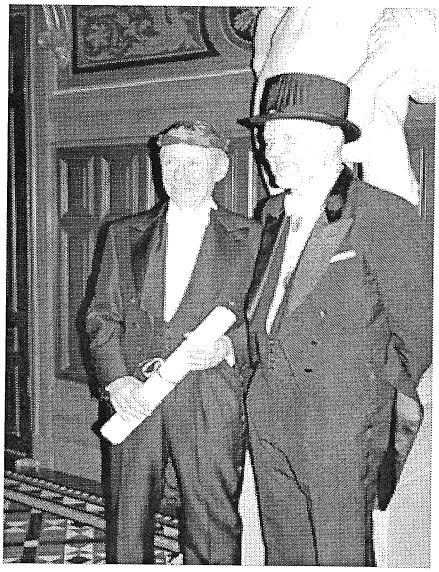
Professor Douglas Barofsky received a Doctor of Philosophy honoris causa from the Faculty of Science and Technology at Uppsala University in Sweden. Doug traveled to Uppsala in January to attend the commencement ceremony on January, 26 2007. The honor was a recognition for all of his past work in ion physics and the physical chemistry of desorption/ionization processes. In addition to his scientific achievements, Doug was recognized for his mentoring of several Ph.D. students at Uppsala University over the past fifteen years. Throughout his mentoring, Doug has traveled to Sweden to participate in the examinations of these students.

The ceremony was very festive and enjoyable as the photos below demonstrate. When asked about traveling to Sweden in January, Doug appeared unfazed. Living in Bend may be good preparation!

Doug appears to be enjoying his retirement. He is staying very active and recently took a rafting trip down the Grand Canyon. It is great to see his enthusiasm for his adventures.



Douglas Barofsky receiving his degree during commencement



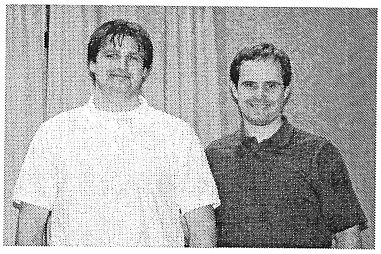
Douglas Barofsky and Bo Sundqvist, (friend, colleague, and former Vice Chancellor of Uppsala University)

Department News

Douglas Keszler presented a lecture, "New Materials for Energy and Process Efficiency" in honor of being named Distinguished Professor on May 22 in the MU Journey room.

Rich Carter and graduate students **Michael Nafzigger**, **Bradley Ashburn** and **Johanna Perkins**' article entitled "Diels-Alder Approach for the Construction of Halogenated, ortho-Nitro Biaryl Templates and Application to the Total Synthesis of anti-HIV Agent Siamenol" will be featured on the cover of the Journal of Organic Chemistry later this year.

Rich Carter has been selected as a 2007 Journal Awardee by the Editorial Board of Synthesis and Synlett. This award is intended to honor promising young professors in organic chemistry. **Rich Carter** is the recipient of the College's Sugihara Young Faculty Research Award. The Dean indicated this will include support for a symposium later this year, so please stay tuned for details.



Michael Naffziger and Rich Carter

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Assets Not Scanned for: 251400 SCH - Chemistry

27-Jan-03

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AI109A	Gilbert Hall Rm 0109a		F8290E6	001100	SI	9/1/1988	PS	

Advanced Degrees 2006/2007

Master of Science

Bin Cao

Bin completed her non-thesis MS and has joined her husband, Guoqiang Wang and her daughter Gina in Michigan where he is working as a Postdoctoral Associate at the University of Michigan (Remcho).

Edgar Lee

Eddie completed his non-thesis MS (Horne).

Ryan Link Cole

Ryan completed his non-thesis MS (Carter).

Yuelong Ma

Synthetic studies on indolic enamide natural products : 1. Total syntheses of coscinamide A, concinamide B and igzamide : 2. Synthetic studies towards the synthesis of halocyamine B (Horne). May is now at the City of Hope pursuing her PhD under the direction of Dr. David Horne.

Arkadiusz Piekarz

Semi-volatile Fluorinated Organic Compounds in Asian and Pacific Northwestern U.S. Air Masses (Simonich).

Doctor of Philosophy

Luke Ackerman

Analysis of Semi-Volatile Organic Contaminants and Their Accumulation in Remote Aquatic Ecosystems of the Western U.S.(Simonich). Luke is working for the US FDA Center for Food Safety and Applied Nutrition in College Park, MD.

James Abbott

Polarization spectroscopy and photodissociation studies of nitroaromatic compounds in the gas phase (Kong).

Carin Huset Ness

Determination of Fluorochemicals in Waste-Dominated Aqueous Systems (Barofsky). Carin is working as a Postdoctoral Associate at the University of South Carolina in the Department of Chemistry and Biochemistry.

Eric Korf

Studies toward the synthesis of halichlorine and pinnaic acid (White).

Tae Hee Lee

Total synthesis of phorboxazole A (White).

Helmars Smits

Studies towards the total synthesis of (-)-kendomycin (White). Helmars is working as a Postdoctoral Associate at the University of Pennsylvania under the direction of Dr. Amos Smith III.

Sorasaree Tonsiengsom

Studies toward the total synthesis of alkaloids : nagelamide A and D, agelastatin D, dragmacidin A-C, salacin and almazoles (Horne). Faye is living in Houston with her husband.

Sascha Usenko

Tracking Semi-Volatile Organic Pollutants in Remote Lake Systems (Simonich).

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AI254	Gilbert Hall Rm 0254	TN1223-21	378052	299999	SI	5/1/1978	PF

2006/2007 Bachelor of Science

Summer 2006

Andy Larkin (BS, forensic science option & German) is in a graduate forensics program at Florida International University.

Preston Skaggs (BS, biochemistry option (MTH Act. Sci minor)) is working at Chemica Technologies in Bend, OR.

Fall 2006

Stefanie Gibson (BS, forensic science option) is working for the Oregon State Police in Springfield.

Paul Heflinger (HBS, ACS certified, advanced chemistry option) is in a graduate biology program at the University of Fairbanks.

Lonnie Hetschel (BS, forensic science option & ANTH) is working for Neilson Research Corp. in Medford.

Winter 2007

Nita Birdsong (BS, environmental chemistry option & BRR (Tox))

Briana Gentry (BS, forensic science option) is working as a lab assistant in Marine Environmental Biology at USC.

Aryanto Herlambang (BS, chemical engineering option) is studying Marine Environmental Biology.

Abigail Joyce (BS, ACS certified, advanced chemistry option (MTH minor)) is working for Bend Research.

Tony Tong (HBS, ACS certified, advanced biochemistry option & BB) is the the graduate Pharmacy program at OSU.

Spring 2007

Joshua Albus (BS, ACS certified, advanced chemistry option) is applying to graduate school.

Britta Anderson (BS, forensic science option) is working at AVI-BioPharma, Inc. in Corvallis.

Marcus Chiodo (BS, pre-medicine option & Biology) is attending OHSU medical school in Portland.

Laura Christ (BS, forensic science option)

Chandra Corley (BS, business option (SPAN minor) is working for Test America in Portland.

Keegan Duff (BS, materials science option) is workig for R&D Biodiesel in Oregon.

Andrew Dunatchik (BS, biochemistry option) is in the graduate Pharmacy program at OSU.

Kendall Dutcher (BS, ACS certified, advanced biochemistry option & BRR (Tox)) is in a Biochemsitry graduate program at UW.

Ian Elliott (BS, chemical engineering option & CHE (MEP-MSE))is in a Chemical Engineering graduate program at UC Davis.

Grant Farr (BS, pre-medicine option (PHL minor)) is attending Medical School at the University of Iowa.

Katherine Fordyce (BS, ACS certified, advanced chemistry option) is working towards her MS in our Department under the direction of Dr. Alexey Shvarev, our newest analytical faculty member.

Elizabeth Haggstrom (BS, forensic science option)

Adam Harney (BS, pre-medicine option)

Nicole Howell (BS, ACS certified, advanced chemistry option) is working at Micron Tech, Inc. in Boise, ID.

Spencer Huff (BS, business option (BA minor))

Matthew Jones (BS, forensic science option) is a Biotech consultant in Albany, OR.

Elizabeth Poore (BS, pre-medicine option & Biology (MTH SCI minor)) is working at AVI BioPharma, Inc.

Tyler Steinke (BS, biochemistry option) is working at AVI BioPharma, Inc. in Corvallis.

Kaleb Stinger (BS, biochemistry option)

Melinda Stoelk (BS, forensic science option) is going to nursing school in Texas.

Jason Warkentin (BS, biochemistry option)

Wesley Williams (BS, environmental chemistry option) is attending a Polymers graduate program at the Univ. of TN.

Shannon Williamson (BS, ACS certified, advanced chemistry option) is in the graduate Material Science program at UO.

Liecong Zhen (BS, ACS certified, advanced chemistry option) is in Portland, OR.

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279177	CENTRIFUGE, REFRIGE, W/ROTOR & ACC		BECKMAN	$7,206.00 **Reconciliation:**				
AU133	Pharmacy Building Rm 0133	GPR	0C016	001100	SI	4/1/1990	PS	
285611	DETECTOR ARRAY, PHOTODIODE		WATERS	$18,000.00 **Reconciliation:**				
AU105	Pharmacy Building Rm 0105	990+	990288D35	299999	SI	1/1/1991	PF	

Department News (continued from page3)

Staci Simonich has received a lot of press regarding her research on the measurement of pesticides and PCBs. An article title, "Asian pollutants found atop Mount Bachelor" was distributed by the Associated Press was included in many papers and peridoicals including Business Week and Forbes detailing the findings of Simonich and other scientists studying pollution patterns.

Walt Loveland will travel to Switzerland in September to "Understanding the synthesis of the heaviest nuclei", presented at the 3rd International Conference on the Physics and Chemistry of the Transactinide Elements, meeting held on the Conference Centre of Davos.

Richard Nafshun facilitated "Chemistry Afternoon." This event consisted of three activities to advance chemistry in the community. Chemistry demonstrations and hands-on laboratories were facilitated for 140 youngsters ages 2-14 and 120 adult chaperons; a social for 30 guests networked members of the OSU community; and ACS Tour Speaker Carolyn Fisher spoke on the Chemistry of Herbs and Spices to 55 guests. Richard hosted "Beaver Buddies" in which Chemistry demonstrations were conducted for 30 students on June 2. "Beaver Buddies is a community outreach event which pairs OSU students with a school-aged child to attend an OSU sporting event, social gathering, or other events of interest. Richard facilitated "Expeditions." For the tenth consecutive year a hands-on chemistry class for talented and gifted 4th /5th grade students was offered. The ten day program runs from 8:45am-noon July 9-20 and has an enrollment of 48 students. Activities include polymers, batteries, crystals, electronic circuits, gases, chemical bonding, acids and bases, the chemistry of art, and chromatography. Richard instructed a General Chemistry course in which one of the recitation sections was a "Learning Community." 24 students participated in the program in which they attended three or more classes together in their field of study (Exercise and Sports Science) and met with the instructors once a week to make connections and foster study skills. This program initiated or increased interaction between students, between students and teachers, and between teachers.

Vince Remcho and his research group had several publications this past year: "Principles and Practice of Capillary Electrochromatography", in Handbook of Capillary Electrophoresis, (**Myra T. Koesdjojo, Carlos F. Gonzalez, Vincent T. Remcho**), "Development of a Semi-automated Procedure for the Synthesis and Screening of a Large Group of Molecularly Imprinted Polymers" in the *Journal of Combinatorial Chemistry* (M. Koesdjojo, Henrik Rasmussen, Adam Fermier, Payal Patel, and V. Remcho), "Organic solvent nanofiltration for microfluidic purification of poly(amidoamine) dendrimers", *Journal of Chromatography* A (**Jack T. Rundel**, Brian K. Paul, V. Remcho), and "Molecularly Imprinted Polymers as Sorbents for Separations and Extractions", in HPLC Method Development for Pharmaceuticals (M. Koesdjojo, **Yolanda H. Tennico**, V. Remcho). The group also gave several presentations: **Corey Koch** and Jack Rundel presented posters at MSB 2007: 21st International Symposium on Microscale Bioseparations January 14-18, 2007 in Vancouver, Canada, "Organic solvent nanofiltration for on-chip extraction of PAMAM dendrimer" (J.Rundel, B.K. Paul, V.Remcho), and "Micro-Flow Injection Device: Integration of Mixing and Optical Detection" (C. Koch, **James D. Ingle** and V. Remcho). Corey also presented his work, "Microfluidics in the Subsurface: towards in-situ analysis of microliter volumes in a miniaturized package" at OSU's Subsurface Biosphere Initiate Graduate Conference, June 17-19, 2007 in Newport, OR. Jack presented, "Fabrication of a Bulk Microfluidic Nanofiltration System for the Processing of Macromolecules" (J. Rundel, B.K. Paul, V. Remcho), at ICOMM 2007: International Conference on Micromanufacturing at Clemson University, in South Carolina. Carlos Gonzalez presented "Development, fabrication, and evaluation of a microfluidic dielectrophoresis device" at the 12th Latin-American symposium on biotechnology, biomedical, biopharmaceutical and industrial applications of capillary electrophoresis and microchip technology, Mexico City, December 2006. **Jintana "Dao" Nammoonnoy** presented "Microfluidic Devices for Selective Photoreversible Extraction of Heavy Metals from Drinking Water" (J. Nammoonnoy, C. Koch, **Jeffrey R. Walker**,and V. Remcho) at the Nano-Micro Breakthrough Conference in Portland in September.

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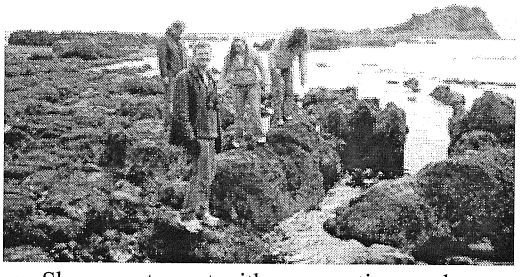
AI302	Gilbert Hall Rm 0302	3700		001100	SI	6/1/1980	PF	
260845	COMPUTER SYSTEM	Z386	ZENITH N/A	$6,301.00 **Reconciliation:**				
AI302	Gilbert Hall Rm 0302			001100	SI	6/1/1988	PS	
AI302	Gilbert Hall Rm 0302	Z386		299999	SI	6/1/1988	PS	

Alexey Shvarev and his graduate students, **Hasini Perera** and **Katy Fordyce** attended Pittcon in March in Chicago. Shvarev was invited to speak at the conference and his talk was titled, "Experimental and Theoretical Investigation of the Response Mechanism of Light-Controlled Ion-Selective Optical Sensors" and covered his collaborative work with **John Westall**. Shvarev's proposal, "Beyond sensing under equilibrium: photoresponsive nanoprobes for rapid localized acid-base titration", Collaborators Oksana Ostroverkhova (OSU) and David McIntyre (OSU). for "ONAMI Nanometrology and Nanoelectronics Initiative" was funded by ONAMI and Office of Naval Research.

Toby Primbs received two awards from the Environmental Chemistry Division of the American Chemical Society: the Environmental Chemistry Graduate Student Award (with a one year subscription to Environmental Science and Technology and membership in the Environmental Division for one year) and the Graduate Student Paper Award (Award winners presented their papers at the American Chemical Society Meeting in Boston, MA in August, 2007. They will also receive a $1,000 cash award at the Environmental Division Dinner).

Myra Koesdjojo extended her research effort through two internships, one at J&J Pharmaceutical Research and Development working on imprinted polymer sorbents for selective extraction of the active pharmaceutical ingredient in pharmaceutical formulations to facilitate impurity profiling, and one at ALZA Corporation (part of J&J), where she worked on development of an automated system for formulation development, salt screening, crystallization and polymorph screening.



Alexey Shvarev at coast with prospective graduate students

Yolanda Tennico enjoyed an internship at ALZA Corporation, CA, from Jan to Dec 2006. Her research focused on analytical method development for oral drug delivery systems.

Three graduate students: **Stephen Meyers (Keszler)**, **Paul Newhouse (Tate)**, and **Annette Richard (Tate)** received fellowships from the NSF Integrative Graduate Education and Research Traineeship (IGERT).

The Department hosted its third Graduate Recruiting Weekend. We hope to continue coordinating this event to recruit promising domestic applicants.

Another promising recruiting event has been the NSF Summer Research Program in Solid State and Materials Chemistry. It is a collaborative venture between **Doug Keszler** and the University of Oregon. The final symposium has been held over a weekend in August at OSU for two years. One of the promising participants from last year, **Alan Telecky**, has joined Keszler's lab for the summer and will be one of our new PhD students this fall.

Dr. Harry Freund, was a professor from 1947 until his retirement in 1980. He died on Monday, August 13, from complications related to Parkinson's Disease.

The Christensen Fund provided travel stipends for several graduate students to attend conferences and present their research. **Carin Huset Ness** attended the North American Meeting of the Society of Environmental Toxicology and Chemistry in Montreal in November to give a platform (oral) presentation and a poster presentation on "Quantitation of Fluorochemicals in Landfill Leachates" and "Mass Flow of Fluorochemicals in a Swiss River Valley". **Robynne Kirkpatrick** attended the International Symposium on Molecular Spectroscopy at Ohio State University in June and presented, "Pieces of the Propellane Puzzle: An Investigation of Rovibrational Coupling.

Robynne Kirkpatrick's research with **J. Nibler** in the area of high resolution molecular spectroscopy was recognized by receipt of a Coblentz Society Student Award, one of 12 nationally. She gave oral presentations on her work at the Western Spectroscopy Association meeting in Asilomar, CA in February.

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AG313	Gilbert Addition Rm 0313		119	299999	SI	3/1/1975	PF	

check dates & degrees

Alumni News

Dean Regier, BS '76, visited last October with his family and received a personal tour of the changes to the chemistry facilities since his graduation from Dr. Pastorek. He was both disconcerted and reassured that the Department has changed so much physically and stated that, "I really missed the distinctive odor that marked the 3rd floor of Gilbert Hall as the organic labs! The renovations are certainly an improvement however. And as the years go by, there are fewer and fewer names on the faculty roster that I recognize". Even Jack Whitney has retired. He and I spent a Christmas vacation doing the annual chemical inventory one long ago Christmas.
I'm sure you have the Integrated Lab Courses ironed out to the point of flawless execution by now but I'll say that being one of the first year guinea pigs during the '74-'76 introduction of that series and having most of the labs crash and burn at some point, was probably better training for real life than you might be willing to admit. Thanks for the training under live fire."

William Bromps, '88 (Yoke), is currently an R&D Chemist with Restek West in Shingle Springs, CA. We are a satellite research facility of Restek Corporation (Bellefonte, PA). My area of research is Porous Layer Open Tubular (PLOT)fused silica capillary columns for the analysis of light hydrocarbons and permanent gases.
It is an exciting time for Restek. In recent years many chromatography companies have been swallowed up by huge comglomerates. The subsequent de-emphasis of chromatography research by these companies has enabled Restek to cherry-pick among the very best researchers in the fields of LC, GC, LC/MS, and GC/MS. I'm excited to help Restek make the most of this opportunity.

Doug Lorenz, BS '85(?) was on campus in April interviewing for a position at Bend Research. Doug was also working on setting up a summer internship program for chemistry undergraduate majors at Bend Research.

John Salinas, '85 (Ingle), continues to teach at Rogue Community College in Grants Pass, OR. Currently he is teaching general chemistry and general science to small classes of wonderful students. He spends summers monitoring and surveying lakes in Southern Oregon. Of special concern presently are the lakes with toxic blue-green algae blooms. "Life is wonderful."

Paul Forster, HBS '98, has accepted a tenure track Assistant Professor position at University of Nevada, Las Vegas. He will start in January. Paul did his thesis research with Gable?, then received a Ph.D. from UCSB, and is currently a postdoc at SUNY Stonybrook.

Tom Whitehead, BS '98, has rejoined AVI BioPharma, Inc. in Corvallis.

Ryan Moser, HBS '99, teaches AP chemistry and physics at Marist HS in Springfield OR and brought his students to campus for some hands on activities last year.

Luke Lavis, BS(ACS) '00, is now a graduate student at UW-Madison and has just been awarded a prestigious ACS Division of Organic Chemistry Graduate Fellowship for 2007.

Tony Masiello, PhD '03, (Nibler) presented a talk at the 62nd International Symposium on Molecular Spectroscopy in Columbus, Ohio on his postdoctoral research at the National Institute for Science and Technology in Gaithersburg, MD in June.

Cristian V. Ion, HBA, '03 is a Legacy Program Associate with Global Green USA (The US Affiliate of Green Cross International, Mikhail Gorbachev, Chairman) cion@globalgreen.org; www.globalgreen.org

Jason Schindler, HBS, 03, is teaching English as a second language in Japan while he makes his way around the world on his bicycle.

Mac Wisdom, BS '03, has joined the Peace Corps.

Veronica Chiu, BS '04, is starting graduate school in Bioanalytical Chemistry at Washington State University. Veronica worked in this area at AVI BioPharma, Inc for the last three years.

Jeff Bilyeu (BS '04) is a Chemistry Instructor at West Linn High School in Oregon

Assets Not Scanned for: 251400 SCH - Chemistry

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Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 01								
158734	SPECTROPHOTOMETER FLUORSCNT 8-A		BAIRD ATOMIC	$6,382.00	**Reconciliation:**			
3B102	Radiation Ctr Wing B Rm 0102	SF-100		001100	SI	11/1/1970	PS	

[BilyeuJ@wlwv.k12.or.us]. Jeff reports that he is doing great and loves teaching. He is sending as many of his students to OSU as possible (60 OSU; 40 UO).

Chris Holms, BS '04, received his MS in Oceanography at OSU.

Darlene Valencia, BS '05, is starting graduate school in Forensic Science at Pace University in NYC. Darlene spent the last year helping us in the chemistry teaching labs as a Laboratory Assistant.

Melissa Schultz, PhD (Barofsky) is an Assistant Professor in the Department of Chemistry at the College of Wooster in Wooster, Ohio.

Kathy VanWormer, HBS, '05, is working on an MS degree in Chemical Engineering and Mechanical Engineering at OSU.

Angela Doneanu, Ph.D. '06 (Remcho), had a daughter, Julia Elena – congratulations!

Mollie Waller, BS '06, is starting graduate school in nanostructures and nanolithography at UC Davis. Mollie worked as an analytical chemist for AVI BioPharma Inc last year before applying to graduate school.

Amanda Wilson, BS '06, graduated from Air Force Intelligence training on 31 July and has move to San Antonio after getting married on Sept 2 in Happy Valley, OR.

Three alums of Chemistry ('06) graduated this spring from the OSU Science Math Education department with MS degrees in Education: **Will Adrian, David Crawford, and Ryan Kanter**. Kanter and Crawford were recipients of the Hach Foundation Scholarships for High School. **Ryan Kanter** has accepted a position teaching chemistry at Crescent Valley High School here in Corvallis. **DJ Crawford** will be teaching middle school somewhere in Oregon.

Department News (continued from page 7)

Hasini Perera had her first publication this year, "Pulsed Galvanostatic Control of Solid-State Polymeric Ion-Selective Electrodes" in *Analytical Chemistry* 2007.

Dr. Alfons Weber from the National Institute for Science and Technology in Gaithersburg, MD spent three weeks at OSU this summer working on a research collaboration with J. Nibler and Dr. Art Maki, former Ph.D student of OSU Professor Jack Decius (dec). In Sept. Dr. Weber will present some of the results of this collaboration at the 20th Colloquium on High Resolution Molecular Spectroscopy at Dijon, France.

Four undergraduates, **Nicole Baker**, **Narumol Jariyasopit**, **Matthew Martin**, and **Robert Zaworski,** worked during the year with **Joe Nibler** on research and on the development of new physical chemistry laboratory experiments involving liquid crystal displays and dynamic light scattering. These experiments will be included in the eighth edition of the Shoemaker, Garland and Nibler laboratory text which is used worldwide.

Michael Chan (Chemistry undergrad) worked with Jack Rundel in the characterization of microfluidic nanofiltration modules (MNMs) during Summer Term 2007. His long hours and scientific curiosity helped push the forward the effort to realize continuous-flow post-synthetic purification of gold nanoparticles in a microreactor system.

Andrew McKenzie, Sheffield U., UK, visited with us an exchange student. While at OSU he learned how to design and fab microfluidic chips for chemical analysis.

The Niobium chapter of Iota Sigma Pi, The National Honor Society for Women in Chemistry, is happy to announce that **Taralyn Tan** (undergraduate in BB) was awarded the 2007 Gladys Anderson Emerson National Scholarship from our chapter. Tari is an outstanding student and has been involved with several undergraduate research projects at OSU. In addition she was instrumental in starting the Sigma Delta Omega Women in Science Sorority which will kick off it's second year this fall.

Assets Not Scanned for: 251400 SCH - Chemistry

27-Jan-03

Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 00								
301621	SAMPLER, FOR HPLC, PROGMBL, AUTO+A	1050-79855A	HEWLETT-PACKARD / 3141A01258	$7,055.00 **Reconciliation:** 001100	SI	7/1/1993	PS	
AU105	Pharmacy Building Rm 0105							
304638	COMPUTER, 100 MHZ W/17" MNTR	PE-30B-MA	DIGITAL EQUIP CORP / AB40300EL9	$7,822.00 **Reconciliation:** 299999	SI	3/1/1994	PF	
3B123	Radiation Ctr Wing B Rm 0123							
309269	SPECTROGRAPH, W/GRAT. 300-1200	CP-200	SPEX INDUSTRIES / 15037-149	$9,009.00 **Reconciliation:** 001100	SI	3/1/1995	PS	
AI241	Gilbert Hall Rm 0241							
310010	COMPUTER, MACINTOSH POWER BOOK	POWER BOOK 1	APPLE / FC34GDGA453	$2,764.00 **Reconciliation:** 890000	OI	8/1/1994	LN	JOHN LOESER
AI109	Gilbert Hall Rm 0109							
312580	Flow Scintillation Analyzer - Radiomatic System	505TR	Packard / 407730	$17,000.00 **Reconciliation:** P0036A	SI	6/26/1995	PF	R1GM32110D
AU135	Pharmacy Building Rm 0135							
312663	Chromatograph, Liquid, Single Instrument 3D Ch	Vectra 486-100	Hewlett Packard / 3423A04955	$10,370.70 **Reconciliation:** P0041A	SI	7/7/1995	PF	R1GM50574A
AI338A	Gilbert Hall Rm 0338a							
315381	Cryostat and Magnet, Super Conducting	n/a	Oxford Industries / n/a	$11,314.00 **Reconciliation:** 299999	SI	1/1/1979	PF	
AK108	Weniger Hall Rm 0108							
318080	Pump, Turbo Molecular	Turbo V701	Varian / 201688	$26,025.87 **Reconciliation:** 001251	SI	8/23/2002	PS	
AI040	Gilbert Hall Rm 0040							
318147	Mass Spectrometer	TSQ700	Finnigan / TS0223	$20,955.00 **Reconciliation:** P0124A	SI	8/21/2002	PF	R1ES09536A
C0085	Ag & Life Sciences Rm 0085							

operating frequency of the spectrometer to produce a unitless entity listed in parts per million or **ppm**. With this in mind, we can directly compare what is seen today at 400 MHz with the spectrum produced by the HA-100 many years ago. Now let's look at several operational terms. The **lock** refers to the signal used to stabilize the magnet field. Today's spectrometers use the deuterium signal from the deuterated solvent that dissolves the sample. For a given magnetic field, the deuterium resonate at about one-sixth the frequency of the proton signal. The HA-100 used instead the signal from protons in tetra-methyl silane (TMS). Thus the stabilization part of the spectrometer is operating on the same frequency as the spectral acquiring part. This is like two trains on the same track headed towards each other. I will tell you more about the train wreck later. The word **shim** or the process of **shimming** refers to getting the magnetic field lines as parallel as possible though the sample region. The sample is normally contained in a 5mm diameter round bottom glass tube suspended in the heart of the magnetic field. The surrounding region has coils of wire carrying small electric currents that reshape the magnetic field. The currents in these coils are under the control of the spectroscopist. This is like focusing a camera, the better the focus the sharper the picture. A sharper spectrum will have more details and will aid chemist in figuring out the molecular structure which is what this is all about.

Let me introduce you to the HA-100, the first NMR spectrometer I ran here at OSU. The magnet was an iron core magnet with 12 inch diameter pole faces. Surrounding the iron core were two 2 foot diameter copper wire coils that were energized by a 30 amp 3 phase power line. The magnet weight exceeded 3000 pounds and produced so much heat that even on cold winter days, I had an air conditioner running continuously. Much of the internal heat was dissipated into city water and sent down the drain. The internal electronics was a collection of vacuum tubes and transistors that only an electronics engineer could understand. I sat in front of two large panels which were covered with dials and switches. As the windowless instrument room was located in the basement of the chemistry building, I could have been in the control room of a submarine or missile launching facility in the heart of Wyoming. You get the picture; it's just me and the equipment. At this point I would like to acknowledge two individuals. The first is Susan Randall, the first NMR operator who taught me how to run the instrument. The second person was Jerry Allison who ran the electronics shop at OSU. Without Jerry's maintenance, I would have had the many fewer years as the HA-100 NMR technician.

This is how a HA-100 day begins. In the morning the first thing you do after opening the door is to see if the instrument is powered up and running. If the power went off for a brief second during the night, the instrument would shut down and I would find a cold magnet the following morning. At this point I would turn on the magnet and console power, get a cup of coffee and make sure that everything turned on during power up. Then I would spend half the day in the library while the magnet warmed up before even thinking about finding the NMR signal and shimming the field.

When the instrument was not in use, you always put the TMS sample back in the magnet and locked the instrument to keep the magnet from drifting. If the power dropped out for less then a tenth of a second, I would find the instrument running but the field is not locked on the TMS sample. The problem is you do not know which way the magnetic field has drifted. It's like you have been dropped off on a country road with no road signs and then try to find your way back to town. You go a bit in one direction then double back and go a bit in the opposite direction. You extend the search in each direction watching the oscilloscope hoping to see the ringing signal from the TMS protons as you sweep past the resonance. What you will see is not just a single signal but a series of signals. For those of you that know about radios, NMR is a radio in the megahertz region using a sideband detector. You see beat frequencies on both sides of the center band frequency. They diminish in intensity the farther you get from the center band. When you see the first small signal in the oscilloscope, you count one. Keep moving the magnetic field, in a few seconds you will see the next signal and it will be stronger, count two. You continue seeing increasing beat signals until the center band is reached. Keep moving the field until the beat signals decrease to nothing and note the number of beat signals seen. Now move the field back to first signal down field from the center signal and lock on to this signal. See why you had to count all the beat signals.

continued on next page

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
	NMR							
PI # /Sub-Org code 71								
303252	PROBE, DUAL, MICROSAMPLING H-C 400M		NALORAC CRYOGEN CO	$19,080.00	**Reconciliation:**			
AI231	Gilbert Hall Rm 0231	40013		001100	SI	11/1/1993	PS	

Final Page

I Certify that all Assets are on Hand, in Use and Scanned with the exceptions as noted above.

Dean/Director/Department Head **Date**

If the instrument was cooperative in the morning, I would find it locked and that the shim condition of the magnetic field would be close to where I had left it last night. But I could also find that the parts of the magnetic field would have shifted in the iron core, requiring me to do a reshim. Shimming is finding the correct combination of currents in the shim coils surrounding the sample region to get the magnetic field lines back into parallel mode. As like a safecracker, I just need to find the correct combination. The only problem here is that the combination changes each day. Unlike the safe combination analogy, some combinations are better than others. Getting a good working shim quickly or spending several hours going nowhere is the difference between a good and bad day. On a good day, I would be shimmed up and ready for the first sample in about 30 minutes.

To run a spectrum, you first removed the TMS sample and replaced it with the tube containing the new sample and 10% TMS dissolved in an appropriate solvent and moved the magnetic field to find the TMS signal. Every new sample requires a minor reshim as each NMR sample interacts with the magnetic field in a slightly different fashion. I would scan through the TMS signal and note the height of the pen deflection on a piece of scratch paper. I then would make a slight adjustment on the main shim current and rescan the TMS signal. If the adjustment was in the correct direction, the TMS signal would get stronger and the line width would decrease. If I when in the wrong direction, the signal would decease with increasing line width. This trial and error method is like climbing a hill. You keep going until you go over the top, then you go backwards to the hilltop. Now that the sample is shimmed the magnetic field is moved to place the TMS signal on to the lock resonance and turn on the lock circuit.

The HA-100 plotted all spectra on a 3 foot long bed plotter with an ink drawing pen. Now I would turn the scratch paper over and move the pen on the left side of the paper. I then set the scan rate to one minute to cover the full spectrum and get a quick view of the NMR spectrum. I now can see the tallest peak in the spectrum. The plotter pen is moved to this peak, the scan rate is now reset to 15 minutes per spectrum and I now need to adjust the signal gain so that this peak stays within the range of the plotter. I only need to scan through this peak a couple of times to get the receiver gain adjusted. The scratch paper is removed and a real sheet of NMR graft paper is placed on the plotter. The pen is moved back to the left side and the real scan of the spectrum is begun. It is about 15 minutes to scan from 10ppm to about .5ppm, plenty of time to almost go to sleep here. You must stop the scan at .5ppm by flipping the scan switch as the scanning frequency is very close to the lock frequency. If you get too close to the lock frequency, the scanning circuitry will blind the lock circuitry and you will lose your lock. This is indeed a train wreck of the first order.

If I have not lost my lock at the end of the scan, then it is time to record the integration of the entire spectrum. The area under each signal is proportional to the number of protons and is very useful to help interpret the spectrum. The pen is returned to the left side of the spectrum and the real chart paper is replaced with the scratch paper. The instrument is now switched into integration mode and the scan rate switched to one minute per scan. With a quick scan the gain of the integrator is adjusted to keep the pen on the paper. Now the real chart paper is returned to the plotter and three repeat scans of the integration are drawn. No train wrecks here as one minute is not enough time to fall asleep.

At this point all the pertinent instrument information is written on the right side of the chart paper and sample is removed from the magnet. Unless I had another sample to run I would replace the TMS sample in the magnet and relock the instrument. At the end of the day I would clean the ink out of the drafting pen and sort through the pile of scratch paper keeping only the usable ones.

The change of summer and winter seasons brought small changes to my routine. The first noticeable change was in the cooling water temperature. During the summer most of the city water comes from the Willamette River and is warmer then the Rock Creek winter water source up in the coast range. The changeover would be over a week period. What I would notice is that my magnet would be running too hot or too cold to get a good shim. The adjustment was to change the flow rate of cooling water through the heat exchanger to where the on-off cycle was back to about half on and half off. It would take a couple of adjustments to get the correct flow rate for the current water temperature. Another change in routine would occur after a heavy

continued on page 12

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 65 Remcho								
318205	Capillary Electrophoresis Instrument		Hewlett Packard	$46,805.00	**Reconciliation:**			
AI244	Gilbert Hall Rm 0244	3DCE	3346G00249	890000	FI	4/1/2001	LN	D00000010, Rev 2

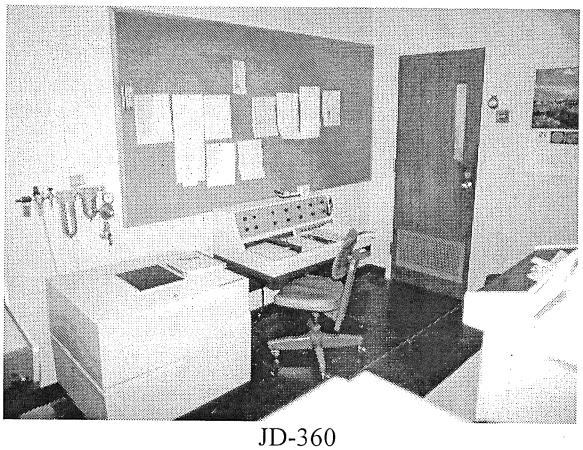
winter rain. As I am in the basement, I am at the same level as the outside window wells. If any of the drain lines to these outside wells plugged up with leaves or debris, I would find 2 inches of standing water in the NMR room. After several floods a new floor drain line was installed.

I think that on a full day I could run nine or ten NMR samples. In about 1980 much of the routine NMR work was done on a smaller 80MHz iron core instrument. This NMR was of a different approach to the problem of detecting the NMR signal. It was a Fourier transform NMR that could obtain a good quality proton spectrum in several minutes on a smaller sample than the HA-100 and had the ability to run a carbon spectrum. The HA-100 finally got too old to produce usable spectra. It was powered down and later the magnet was given to the physics department. The FT-80A carried the department work load for several years until it was superseded by the AM-400 but that is another story. I don't miss the HA-100 at all but it did teach me a lot about NMR as it was completely hands on flying.

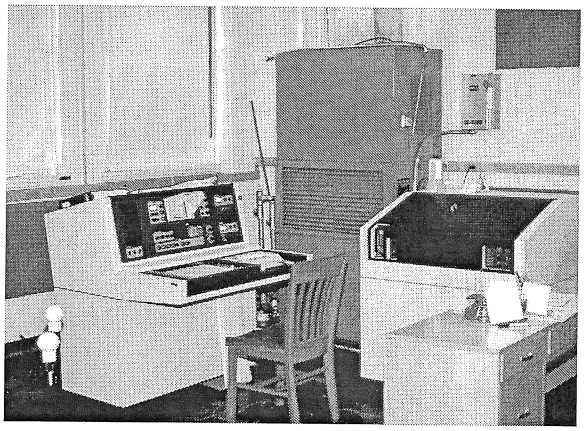
Murdock Charitable Trust and National Science Foundation-Major Research Instrumentation Program have awarded grants to OSU for the purchase of a 700 MHz Nuclear Magnetic Resonance (NMR) Spectrometer with carbon-optimized cryoprobe. Organic chemists depend heavily on NMR for structural assignment of the compounds they synthesize as well as for following the course of a reaction mechanism. Once it is installed, this NMR will be the most sensitive carbon-detecting magnet in the world. **Professor Carter** is the project leader and over 20 different research groups from 10 departments at 5 universities and 3 companies around the state participated in this endeavor.

NMR Timeline (unsure of chronological sequence)

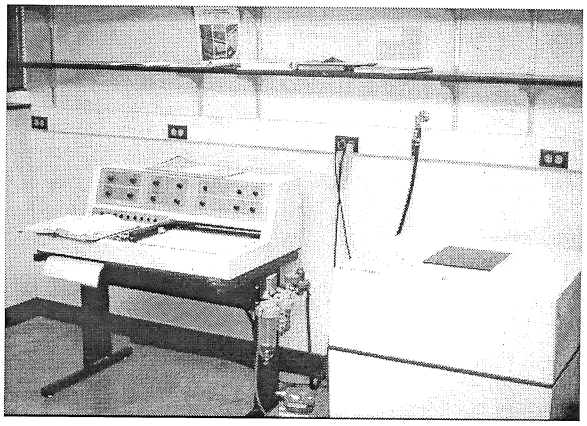
FT80A



JD-360



NR 80



Teaching Lab 360

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 64 Pastorek ?								
197948	CHROMATOGRAPH VARIAN W/ACC	3700	VARIAN	$8,933.00	**Reconciliation:**			
AI302	Gilbert Hall Rm 0302			299999	SI	6/1/1980	PF	
AI302	Gilbert Hall Rm 0302			001100	SI	6/1/1980	PF	

Properties of a NMR Spectrum

I would like to explain several properties of the NMR spectrum. The position of the proton (hydrogen) signal relative to a reference signal is called the **chemical shift** and is used to determine the type of proton in question. Protons on adjacent carbon atoms will influence each other's magnetic environment, resulting in an equal splitting of each other signals and is given the term "**coupling**". The numbers of adjacent protons produce definitive patterns and become recognizable to a trained organic chemist. When the chemical shift between coupled protons is large compared to the proton-proton coupling, the spectrum is said to be "**first order**" and the coupling patterns are not distorted. When the chemical shift separation is close to the coupling or less than the coupling constant, the spectrum is said to be "**second order**". With second order spectra the coupling patterns are distorted and often unrecognizable.

The frequency of the proton signal is directly related to the strength of the magnetic field whereas the proton-proton coupling frequency is dependent only on adjacent protons and is therefore independent of the applied magnetic field. To allow the direct comparision of NMR spectra run on different instruments and at different magnetic fields, the observed proton frequency is divided by the reference frequency to produce a unit less "**ppm**" scale. In Figure 1 the structure of 3-bromoanisole is shown with the four aromatic protons labeled a-d. In Figure 2 the aromatic region of 3-bromoanisole is displayed at 100 MHz, 300 MHz, 400 MHz and 700 MHz. The stacked spectra keep the chemical shifts of each proton in the same place. To demonstrate the increased dispersion at the higher magnetic fields a second plot is shown in Figure 3. Here the spectra are plotted at the same hertz scale with the right most proton signal assigned the value of zero hertz. The width of the proton-proton coupling in this signal stays constant over the range of increasing applied magnetic fields. The chemical shifts of all the proton signals are expanded with the increasing field. What is a complicated second order spectrum at 100 MHz becomes a first order spectrum at 700 MHz.

The signal from the proton at position *c* is coupled to the protons at *b* and *d* by ~8 hertz ortho coupling. The protons at *b* and *d* show the large ~8 hertz coupling and the smaller ~2 hertz meta coupling to each other and the proton at position *a*. The proton at position *a* shows only meta couplings to *b* and *d*.

The NMR spectrum is produced from a very weak radio frequency signal. Detecting this signal from the background noise has always required high quality electronic equipment. This places a minimal sample quantity that can be analyzed by NMR. The NMR signal for a given sample increases with increasing magnetic field. Thus a 700 MHz instrument can get a useable spectrum from a smaller sample than a 100 MHz instrument. The lowering of the detectable sample size with the increased dispersion has been the driving force in the quest of high field NMRs.

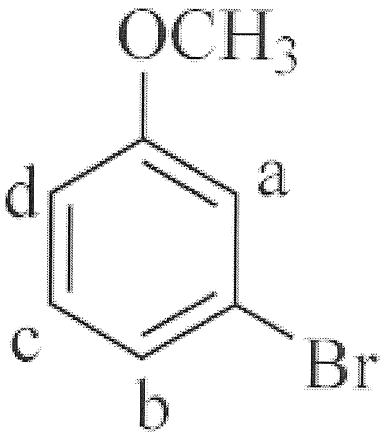


Figure 1 3-Bromoanisole

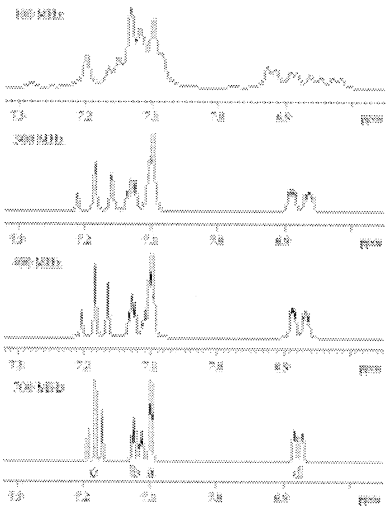


Figure 2 Plotted to the same ppm scale

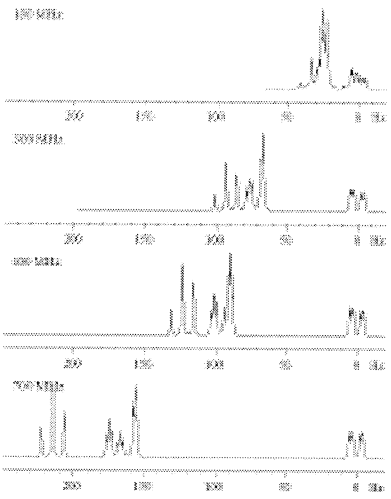


Figure 3: Plotted to the Same Hertz Scale

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

Asset / Location	Description	Model	Manufacturer / Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
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215137	CONTROLLER		BECKMAN	$7,100.00	**Reconciliation:**			
AI307	Gilbert Hall Rm 0307	420	4222093	299999	SI	2/1/1983	PF	
215142	ISOCRATIC SYSTEM		BECKMAN	$5,886.00	**Reconciliation:**			
AI307	Gilbert Hall Rm 0307	341	062-312	299999	SI	2/1/1983	PF	
227607	DETECTOR, VAR-WVGTH UV 394-246		BECKMAN	$6,126.00	**Reconciliation:**			
AI307	Gilbert Hall Rm 0307	164		299999	SI	11/1/1984	PF	PNW 80-85

Honors and Awards

College of Science
Undergraduate Scholarships for 2007/2008

Peter C. Culter Memorial Scholarship
Brian Knight Eric Titus
Jeff Wong

Carroll DeKock Scholarship
Margaret Dalgarno Alex Gilman
Shane Monares

Colleen Spurgeon Scholarship
Garrett Jones

Linda May Oleson Chemistry Scholarship
Layne Clemen

Milton Harris Scholarship
Ben Taucher

Hach Scientific Foundation Education Scholarship
Sarah Bierly Ashley F ulleton
Nicole Rae Tanguileg Dustin Welch

Chemistry Department Awards, Sept. 2006

`05/`06 Employee of the Year Award
Cindy Persson

'05/'06 Milton Harris Teacher of the Year Award
Emile Firpo Christine Pastorek

'05/'06 Harris Graduate Teaching Assistant Award
Corey Koch Susan Genualdi
James Neeway

Chemistry Department Awards, June 2007

William J. Ingram Memorial Fellowship
Khomson Suttisintong

Courtney & Dorothy Benedict Fellowship
Dao Nammoonnoy Hasini Perera

Fall 2006 Laboratory TA Awards
Defne Cakin Jeremey Gunderson
Beth Knight

Winter 2007 Laboratory TA Awards
Christopher Emerson Andrew Smith
Jianyong Wu

Spring 2007 Laboratory TA Awards
Hasini Perera Sheena Strohmayer
Jing Wang

N.L. Tartar Summer Research Fellowships
Bradley Ashburn Chris Emerson
Susan Genualdi Heath Giesbrecht
Damien Kuiper John Melbardis
Johanna Perkins Keith Schwartz
Chad Teters

Milton Harris Summer Research Fellowships
Corey Koch Myra Koesdjojo
Hasini Perera Jack Rundel

David Shoemaker Award
Robynne Kirkpatrick

Bruce Graham Memorial Scholarship
Andrew Smith

Hedberg Fellowship/White Fellowship
Michael Naffziger

Arnold Johnson Fellowship
Jie (Jessica) Zhang

CRC Press Freshman Chemistry Awards
Michelle Adlong Lene Lang

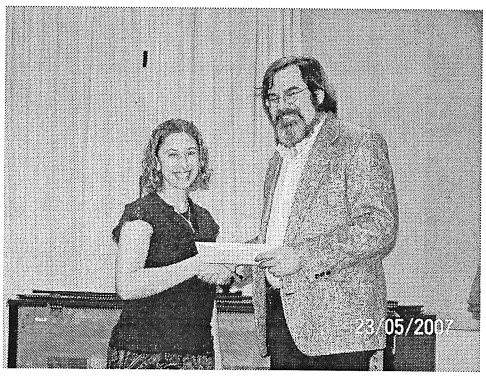
PLU Award
Eric Titus

Analytical Chemistry Award
Sarah Furrer

American Institute of Chemists Award
Ian Elliott

Merck Award
Elizabeth Poore

Hypercube Scholar
Joshua Albus



Kevin Gable and Hach Recipient, Sarah Bierly

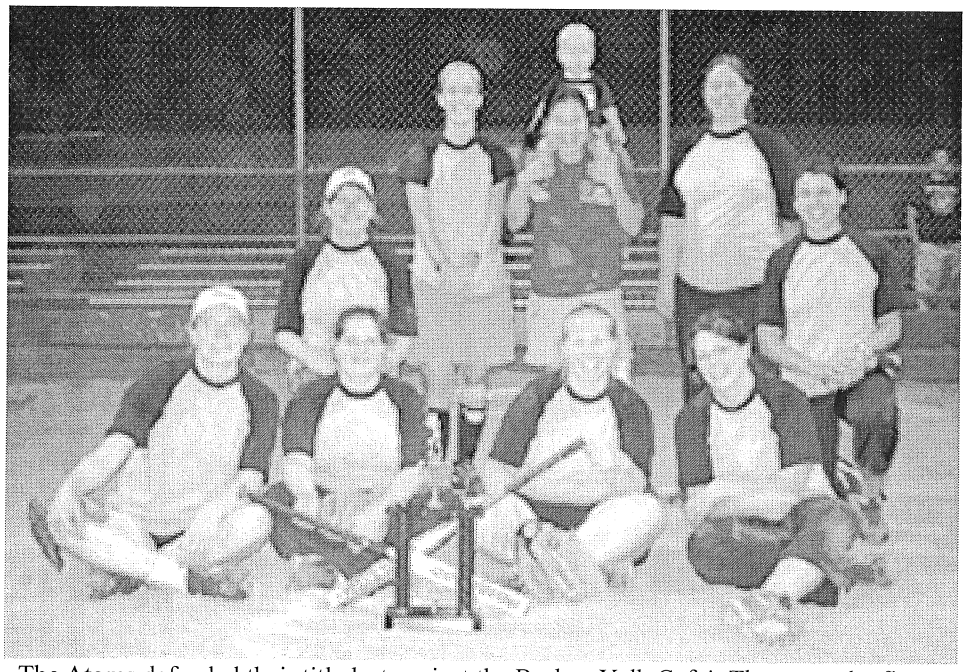
Department Benefactors: Carroll and ??? DeKock, Ken and Lise Hedberg, Clare Shoemaker, and James White

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

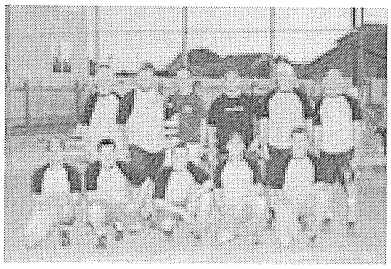
Asset / Location	Description	Manufacturer / Model	Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 34 White								
295004	COMPUTER, IRIS-INDIGO W/ACCESS	SILICON GRAPHICS		$13,251.00 **Reconciliation:**				
AI329	Gilbert Hall Rm 0329	W-4 DRPC	35008654	001100	SI	7/1/1992	PS	

Oregon State UNIVERSITY OSU
Department of Chemistry
Oregon State University
153 Gilbert Hall
Corvallis, OR 97331-4003



Make smaller!

The Atoms defended their title last against the Broken Yolk Cafe! They won the first game 17-15 and dominated in the second game 16-8.



take out?

Assets Not Scanned for: 251400 SCH - Chemistry

11-Aug-04

Asset / Location	Description	Manufacturer / Model	Serial Number	Asset Value / Fund Code	Title-To	Date Acquired	Acqd Code	Grant/Contract
PI # /Sub-Org code 33 Westall								
239103	TROUGH, LANGMUIR	JOYCE-LOEBL		$12,421.00 **Reconciliation:**				
AI243	Gilbert Hall Rm 0243	6AA-4001	342	001100	SI	3/1/1986	PS	
AI243	Gilbert Hall Rm 0243	6AA-4001		359999	SI	3/1/1986	PS	

A Day in the Life of the HA-100 NMR.

Obtaining a spectrum on the current Fourier transform AVANCE 400 compared to the continuous wave HA-100 NMR is like flying a jet fighter aircraft compared to an open cockpit biplane. For those of you that have not had the chance to play with Nuclear Magnetic Resonance (NMR) spectrometers, I will describe a few terms used in obtaining a NMR spectrum.

All NMR magnets are rated by the **frequency** of the proton spectrum which is directly related to the strength of the magnetic field (read hydrogen here as NMR jocks and organic chemists always run a proton spectrum, never a hydrogen spectrum). For a given magnetic field, the protons will **resonant** at a particular frequency. Thus the AVANCE 400 has the proton spectrum at 400 MHz and the HA-100 has the proton spectrum at 100 MHz. To produce a spectrum, you must either hold the detector at a fixed frequency and sweep the magnetic field or keep the magnetic field constant and sweep the detector frequency to bring the protons into **resonance**. The output of the detector is plotted on the vertical axis of a sheet of paper while the horizontal axis relates to frequency. At this point, you would think that all the protons (remember hydrogen's in the organic molecule) resonant at the exactly the same frequency. Fortunately for the organic chemists, each proton of a particular type will experience a slightly different magnetic field influenced by the location in the molecule and therefore the spectrum will be a series of resonances giving clues as to the structure of the molecule. The term **chemical shift** is used to describe the fact that methyl protons will be located in one part of the spectrum while methylene protons will be located in another part of the spectrum. One could make a list of these frequencies but the values will be dependent on the strength of the main field resulting in a unique list for each NMR magnet in use. The universal solution to this problem is to divide the observed resonances by the base operating frequency of the spectrometer to produce a unitless entity listed in parts per million or **ppm**. With this in mind, we can directly compare what is seen today at 400 MHz with the spectrum produced by the HA-100 many years ago. Now let's look at several operational terms.

The **lock** refers to the signal used to stabilize the magnet field. Today's spectrometers use the deuterium signal from the deuterated solvent that dissolves the sample. For a given magnetic field, the deuterium resonate at about one-sixth the frequency of the proton signal. The HA-100 used instead the signal from protons in tetra-methyl silane (TMS). Thus the stabilization part of the spectrometer is operating on the same frequency as the spectral acquiring part. This is like two trains on the same track headed towards each other. I will tell you more about the train wreck later. The word **shim** or the process of **shimming** refers to getting the magnetic field lines as parallel as possible though the sample region. The sample is normally contained in a 5mm diameter round bottom glass tube suspended in the heart of the magnetic field. The surrounding region has coils of wire carrying small electric currents that reshape the magnetic field. The currents in these coils are under the control of the spectroscopist. This is like focusing a camera, the better

the focus the sharper the picture. A sharper spectrum will have more details and will aid chemist in figuring out the molecular structure which is what this is all about.

Let me introduce you to the HA-100, the first NMR spectrometer I ran here at OSU. The magnet was an iron core magnet with 12 inch diameter pole faces. Surrounding the iron core were two 2 foot diameter copper wire coils that were energized by a 30 amp 3 phase power line. The magnet weight exceeded 3000 pounds and produced so much heat that even on cold winter days, I had an air conditioner running continuously. Much of the internal heat was dissipated into city water and sent down the drain. The internal electronics was a collection of vacuum tubes and transistors that only an electronics engineer could understand. I sat in front of two large panels which were covered with dials and switches. As the windowless instrument room was located in the basement of the chemistry building, I could have been in the control room of a submarine or missile launching facility in the heart of Wyoming. You get the picture; it's just me and the equipment. At this point I would like to acknowledge two individuals. The first is Susan Randall, the first NMR operator who taught me how to run the instrument. The second person was Jerry Allison who ran the electronics shop at OSU. Without Jerry's maintenance, I would have had the many fewer years as the HA-100 NMR technician.

This is how a HA-100 day begins. In the morning the first thing you do after opening the door is to see if the instrument is powered up and running. If the power went off for a brief second during the night, the instrument would shut down and I would find a cold magnet the following morning. At this point I would turn on the magnet and console power, get a cup of coffee and make sure that everything turned on during power up. Then I would spend half the day in the library while the magnet warmed up before even thinking about finding the NMR signal and shimming the field.

When the instrument was not in use, you always put the TMS sample back in the magnet and locked the instrument to keep the magnet from drifting. If the power dropped out for less then a tenth of a second, I would find the instrument running but the field is not locked on the TMS sample. The problem is you do not know which way the magnetic field has drifted. It's like you have been dropped off on a country road with no road signs and then try to find your way back to town. You go a bit in one direction then double back and go a bit in the opposite direction. You extend the search in each direction watching the oscilloscope hoping to see the ringing signal from the TMS protons as you sweep past the resonance. What you will see is not just a single signal but a series of signals. For those of you that know about radios, NMR is a radio in the megahertz region using a sideband detector. You see beat frequencies on both sides of the center band frequency. They diminish in intensity the farther you get from the center band. When you see the first small signal in the oscilloscope, you count one. Keep moving the magnetic field, in a few seconds you will see the next signal and it will be stronger, count two. You continue seeing increasing beat signals until the center band is reached. Keep moving the field until the beat signals decrease to nothing and note the number of beat signals seen. Now move the field back to first signal down field from the center signal and lock on to this signal. See why you had to count all the beat signals.

If the instrument was cooperative in the morning, I would find it locked and that the shim condition of the magnetic field would be close to where I had left it last night. But I could also find that the parts of the magnetic field would have shifted in the iron core, requiring

me to do a reshim. Shimming is finding the correct combination of currents in the shim coils surrounding the sample region to get the magnetic field lines back into parallel mode. As like a safecracker, I just need to find the correct combination. The only problem here is that the combination changes each day. Unlike the safe combination analogy, some combinations are better than others. Getting a good working shim quickly or spending several hours going nowhere is the difference between a good and bad day. On a good day, I would be shimmed up and ready for the first sample in about 30 minutes.

To run a spectrum, you first removed the TMS sample and replaced it with the tube containing the new sample and 10% TMS dissolved in an appropriate solvent and moved the magnetic field to find the TMS signal. Every new sample requires a minor reshim as each NMR sample interacts with the magnetic field in a slightly different fashion. I would scan through the TMS signal and note the height of the pen deflection on a piece of scratch paper. I then would make a slight adjustment on the main shim current and rescan the TMS signal. If the adjustment was in the correct direction, the TMS signal would get stronger and the line width would decrease. If I when in the wrong direction, the signal would decease with increasing line width. This trial and error method is like climbing a hill. You keep going until you go over the top, then you go backwards to the hilltop. Now that the sample is shimmed the magnetic field is moved to place the TMS signal on to the lock resonance and turn on the lock circuit.

The HA-100 plotted all spectra on a 3 foot long bed plotter with an ink drawing pen. Now I would turn the scratch paper over and move the pen on the left side of the paper. I then set the scan rate to one minute to cover the full spectrum and get a quick view of the NMR spectrum. I now can see the tallest peak in the spectrum. The plotter pen is moved to this peak, the scan rate is now reset to 15 minutes per spectrum and I now need to adjust the signal gain so that this peak stays within the range of the plotter. I only need to scan through this peak a couple of times to get the receiver gain adjusted. The scratch paper is removed and a real sheet of NMR graft paper is placed on the plotter. The pen is moved back to the left side and the real scan of the spectrum is begun. It is about 15 minutes to scan from 10ppm to about .5ppm, plenty of time to almost go to sleep here. You must stop the scan at .5ppm by flipping the scan switch as the scanning frequency is very close to the lock frequency. If you get too close to the lock frequency, the scanning circuitry will blind the lock circuitry and you will lose your lock. This is indeed a train wreck of the first order.

If I have not lost my lock at the end of the scan, then it is time to record the integration of the entire spectrum. The area under each signal is proportional to the number of protons and is very useful to help interpret the spectrum. The pen is returned to the left side of the spectrum and the real chart paper is replaced with the scratch paper. The instrument is now switched into integration mode and the scan rate switched to one minute per scan. With a quick scan the gain of the integrator is adjusted to keep the pen on the paper. Now the real chart paper is returned to the plotter and three repeat scans of the integration are drawn. No train wrecks here as one minute is not enough time to fall asleep.

At this point all the pertinent instrument information is written on the right side of the chart paper and sample is removed from the magnet. Unless I had another sample to run I

would replace the TMS sample in the magnet and relock the instrument. At the end of the day I would clean the ink out of the drafting pen and sort through the pile of scratch paper keeping only the usable ones.

The change of summer and winter seasons brought small changes to my routine. The first noticeable change was in the cooling water temperature. During the summer most of the city water comes from the Willamette River and is warmer then the Rock Creek winter water source up in the coast range. The changeover would be over a week period. What I would notice is that my magnet would be running too hot or too cold to get a good shim. The adjustment was to change the flow rate of cooling water through the heat exchanger to where the on-off cycle was back to about half on and half off. It would take a couple of adjustments to get the correct flow rate for the current water temperature. Another change in routine would occur after a heavy winter rain. As I am in the basement, I am at the same level as the outside window wells. If any of the drain lines to these outside wells plugged up with leaves or debris, I would find 2 inches of standing water in the NMR room. After several floods a new floor drain line was installed.

I think that on a full day I could run nine or ten NMR samples. In about 1980 much of the routine NMR work was done on a smaller 80MHz iron core instrument. This NMR was of a different approach to the problem of detecting the NMR signal. It was a Fourier transform NMR that could obtain a good quality proton spectrum in several minutes on a smaller sample than the HA-100 and had the ability to run a carbon spectrum. The HA-100 finally got too old to produce usable spectra. It was powered down and later the magnet was given to the physics department. The FT-80A carried the department work load for several years until it was superseded by the AM-400 but that is another story. I don't miss the HA-100 at all but it did teach me a lot about NMR as it was completely hands on flying.