

Chemistry Seminar Abstracts for the Year 2011

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Treatability of Environmental Contaminants and Removal of Disinfection By-Products in Drinking Water Using LC-MS/MS

Yinfa Ma, Dept. of Chem., MS&T

Abstract: Environmental contaminants in natural and drinking waters, such as pesticides, herbicides and pharmaceuticals and personal care products (PPCPs), may exert possible health impacts on humans. In addition, many disinfection byproducts (DBPs), such as N-nitrosamines, halonitromethanes (HNMs), could also exist in drinking water after water treatment processes at water treatment facilities. Therefore, efficient treatment and removal of these emerging contaminants and DBPs in natural and treated waters become an important issue for environmental researchers so to provide safe drinking waters for human and animals.

In our recent study, chemical and physical methods for removal of selected PPCPs and DBPs in natural and drinking waters have been investigated by using liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) and other analytical techniques. The details on method development, quality control/assurance, removal efficiency, and potential solutions for a clean drinking water will be discussed.

Organofluorine Chemistry in the Design of Cell Cycle Inhibitors

Dr. V. Prakash Reddy, Dept. of Chem., MS&T

Abstract: In view of the unique applications of organofluorine compounds in bioorganic and medicinal chemistry, a series of novel purine-based fluoroaryl triazoles have been synthesized using the Cu(I) catalyzed 1,3-dipolar cycloadditions, and assayed for their potency in cell cycle inhibition using Ab cultures. Some of these triazole derivatives are found to have comparable effect as that of Roscovitine, and Flavopiridol, the widely used cyclin-dependent kinase (CDK) inhibitors currently under clinical trials, in cell cycle suppression. The beneficial effects of these triazole based purine derivatives on the Amyloid-beta (Ab) induced neurotoxicity is significant, and thus they would have potential therapeutic applications. General synthetic methodologies for organofluorine compounds will also be discussed.

Optimization of the Response Times in Photorefractive Polymeric Composites through the Inclusion of Semiconductor Nanocrystals

Jeffrey G. Winiarz, Dept. of Chem., MS&T

Abstract: The phenomenon of photo refractivity, which involves the creation of a reversible hologram generated by the crossing of two coherent beams in an appropriate medium, can be realized in polymeric composite materials which simultaneously exhibit photoconductive and electro-optic properties. The *nanocomposite* approach offers opportunities to produce high performance and relatively low cost optoelectronic media, suitable for many applications. Especially promising are nanocomposites of polymers and inorganic semiconductor nanocrystals commonly known as quantum dots. Initially, experiments focused on the optical absorption properties of quantum dots and their dependence on

composition and size. The broad tunability of the spectral response and increased photo-charge generation efficiency were particularly interesting with respect to photorefractive composites. More recently, however, time-of-flight experiments have revealed that the mere inclusion of semiconductor nanocrystals significantly enhances the mobility of charge carriers in polymeric composites. This enhanced mobility translates into an improved response time; an issue which has plagued this class of materials since their inception and has precluded their use in many practical applications. This seminar will present experimental evidence confirming that through judicious selection of an appropriate semiconductor material, the loading content of the photosensitizing nanocrystals can be increased while avoiding detrimental effects such as substantial optical absorption losses. Through increased loading, the enhanced charge-carrier mobility associated with the nanocrystals can be more fully exploited leading to improved response times. In addition, data will be presented which demonstrates the ability to achieve unprecedented response-times through enhanced charge-carrier mobility via the inclusion of semiconductor nanocrystals in more traditionally organically photosensitized photorefractive composites.

A General Scheme for Representing Potential Energy Surfaces for Multichannel Reactions

Dr. Richard Dawes, Dept. of Chem., MS&T

Abstract: The potential energy surface (PES) is central to how chemists think about the structure and dynamics of molecular systems, in terms of minima or asymptotes connected by paths across landscapes and over energetic barriers. The concept of an adiabatic energy hypersurface is a result of the Born-Oppenheimer approximation and is usually sufficiently accurate even for high-resolution spectroscopic applications. When necessary, most treatments of non-adiabatic dynamics begin with a set of (coupled) adiabatic electronic states, making the potential energy surface ubiquitous to theoretical chemistry.

I will describe some methods used to compute accurate molecular electronic energies and fit them into analytic functional forms (PESs). A strategy for multichannel reactions and representative results from studies of spectroscopy and dynamics will be shown.

Further Materials Research at MS&T

Dr. Wayne Huebner, Dept. of Material Sciences and Engineering, MS&T

Abstract: Advances in the development of man can be traced to his mastery of the world of materials around him. This presentation will initially focus on the ages of man from the Stone Age through today's Nano/Bio Age, with particular emphasis on the evolution of our understanding of structure-processing-property relationships. Then I will present many examples of leading-edge materials research at S&T.

In vivo Inhibition of L-Buthionine-(S,R)-Sulfoximine(BSO)-Induced Cataracts by a Novel Antioxidant, N-acetylcysteine Amide

Joshua W. Carey, Dept. of Chem., MS&T

Abstract: Cataracts are the most common cause of treatable blindness worldwide. It has been shown that cataracts likely develop due to the effects of oxidative stress. Oxidative stress is caused by an imbalance between the production of reactive oxygen and a biological system's ability to readily detoxify the reactive intermediates or easily repair the resulting damage. A normal lens has several antioxidant enzymes and a high concentration of ascorbate and GSH that protect it from the damages of oxidative stress. The ability of the lens to resist oxidative damage decreases dramatically with the age related decrease in the production of GSH by the cell. GSH is an important thiol for protecting against GSH and maintaining the transparency of the lens. The effects of GSH on normal lens function can be studied by depleting the intercellular levels of GSH using L-Buthionine-(S,R)-sulfoximine (BSO). BSO is an inhibitor of γ -glutamyl-cysteine synthetase, which is known to deplete the levels of GSH both in vivo and in vitro. The role of GSH can be studied by inducing BSO generated cataracts in an animal model. When GSH levels are depleted the formation of a cataract occurs; however it has been suggested that by increasing the levels of antioxidants in the cell it is possible to prevent the formation of cataracts. Recent research using N-acetylcysteine amide, (NACA) a novel antioxidant, have led to the theory that it may be possible to reverse the effects of oxidative damage in low grade cataracts.

Inorganic Chemistry for the Mineral Collector

Dennis L. Whitney, Environmental Engineer, American Airlines, Retired

Abstract: The collection and classification of minerals may be either a hobby or a profession that involves the study of geology, mineralogy, and inorganic chemistry. The history of mineral collecting and the Old Dana, Strunz, and New Dana classification systems used to classify a systematic mineral collection will be discussed.

The assay of a mineral typically does not provide information about its systematic mineralogy, but rather provides only its bulk chemical composition in terms of its corresponding elemental oxides. The reasons for this unusual approach to reporting the composition of a mineral will be discussed. Exotic and unusual mineral specimens will be displayed and the processes involved in assembling a mineralogical study collection will be discussed.

Biomarkers for Noninvasive Early Cancer Detection

Sanjeeva Gamagedara, Dept. of Chem., MS&T

Abstract: Cancer is the second leading cause of death, accounting 13% of all deaths worldwide. Deaths from cancer are projected to continue rising, with an estimated 12 million deaths in 2030. Cancer develops rapidly and early diagnosis and treatment greatly improve the patient's chances of survival. Biomarkers are good potential candidates for this early cancer diagnosis.

Part I: Recently pteridines become a focal point of cancer screening research because certain pteridines levels have been shown to reflect the presence of cancers. This study analyzed eight pteridines; 6,7-dimethylpterin, 6-biopterin, D-(+)-neopterin, 6-hydroxymethylpterin, pterin, isoxanthopterin, xanthopterin and pterin-6-carboxylic acid using a house-built high-performance capillary electrophoresis with laser-induced fluorescence detection (HPCE-LIF). The levels of pteridines were reported as a ratio of pteridine to creatinine. Statistical hypothesis testing was conducted and P-values were calculated to analyze the data.

Part II: Sarcosine and related metabolomic profiles recently drew a lot of attention because a debate regarding their possible role as potential clinical markers for prostate cancer. In this study, levels of sarcosine and related metabolites in 126 patients with genitourinary malignancies (63 prostate cancers & 63 bladder cancers) were analyzed using a validated LC/MS/MS method. Statistical hypothesis testing, Multivariate analysis of variance, correlation study, and principal component analysis were conducted to analyze the data. For prostate cancer these biomarker levels were compared in T1, T2 stages and Gleason scores <7 , ≥ 7 . The detailed experimental conditions and results will be presented at the seminar.

The Art and Science of Microscopy

Clarissa Wisner, Electron Microscope Specialist, MS&T

Abstract:

In 1661, Henry Powers, wrote a poem "In Commendation of ye Microscope,"

"Of all the Inventions none there is Surpassed
the Noble Florentine's Dioptrick Glasses
For what a better, fitter guift could bee
in this World's Aged Luciosity.
To help our Blindnesse so as to devise
a paire of new & Artificial eyes,
by whose augmenting power wee
now see more than all the world Has euer donn Before."

This poem is still so true today, with all the innovations to the world of microscopy. But not all work in microscopy is scientific in nature. There is a growing industry that uses the microscope to enhance walls, textiles, pottery and other ordinary everyday things. The merging of these two dissimilar words is not only exciting, but interesting as one follows the history of the science of microscopy to the pop culture art world.

We will take a journey from the beginning of the microscope in the 16th century to the present that will not only give historical facts but beautiful images to entice everyone to take up microscopy as a hobby!

An Overview of the Library Resources for the Students of Chemistry

Christopher Jocius, Head of the Reference Department C.L. Wilson Library, MS&T

Abstract: An overview of the resources available for chemistry graduate students at the Missouri University of Science and Technology library will be presented. This will include how to access and use the ACS based Scifinder to search the chemical scientific literature.

Organic/Inorganic Aerogels through Ring Opening Metathesis Polymerization

Dhairiyashil Mohite, MS&T

Abstract: Aerogels are open-pore, low-density nanostructured solids with high surface areas, low thermal conductivities, low dielectric constants and high acoustic attenuation. The most common type, silica aerogels, are made by base-catalyzed gelation of tetramethylorthosilicate (TMOS) and consist of a self-assembled, 3D skeletal framework of silica nanoparticles. Despite their attractive bulk properties, these materials are extremely fragile, which limits their applications. Strength of aerogels can be increased by providing interparticle polymeric tethers which covalently connect the skeletal nanoparticles. These new, mechanically strong porous materials are referred to as polymer crosslinked aerogels. Organic aerogels on the other hand are derived solely from organic polymers, mainly based on resorcinol-formaldehyde (RF), melamine-formaldehyde (MF), and phenol-furfural (PF) resins. Other polymer-based aerogels reported include polyurethane/polyurea, polystyrene, polydicyclopentadiene, and lately polyimides.

Part A: Strong Silica Aerogels Crosslinked with Polynorbornene via Ring Opening Metathesis Polymerization (ROMP) Crosslinking of silica aerogels is demonstrated by ring opening metathesis polymerization (ROMP) by providing the surface of silica particles with the norbornene functionality using a new nadimide derivative of 3-aminopropyltriethoxysilane (APTES). Norbornene monomer is introduced in the mesopores and a ROMP process is started using 2nd generation Grubbs' catalyst at ambient temperature. The growing polymer engages norbornene moieties bound on the surface of

silica forming a conformal coating of polynorbornene on the mesoporous surfaces throughout the entire skeletal framework. The amount of polymer incorporated in the mesoporous structure is controlled by the concentration of the monomer in the mesopores. Despite the increase in bulk density (up to 0.6-0.7 g cm⁻³), decrease in porosity (down to ~50% v/v), and decrease in surface area (down to ~150 m² g⁻¹), the materials remain mesoporous. The mechanical properties in terms of strength, modulus and the energy absorption capability relative to the native (non-crosslinked) counterparts are increased dramatically.

Part B: Organic (Polyimide and Polydicyclopentadiene) Aerogels by ROMP Polyimide aerogels are synthesized by crosslinking through ROMP of a bisnadimide bifunctional monomer, **bis-NAD**, using a second generation Grubbs' catalyst. Aerogels with different bulk densities (ranging from 0.13 to 0.66 g cm⁻³) were obtained by varying the monomer concentration in the sol (from 2.5% to 20% w/w). With

increasing density, the interconnected nanoparticle structure changed from macro- to mesoporous and the percent ratio of the micropore area relative to the BET surface area decreased from 28% to 9%. **bis-NAD** aerogels demonstrate excellent mechanical strength and high specific energy absorptions.

Synthesis of mechanically strong polydicyclopentadiene (pDCPD)-based organic aerogels is carried out through ROMP by reinforcing through post-gelation grafting with polymethylmethacrylate (PMMA) using free radical chemistry initiated with AIBN. Solid ^{13}C CPMAS NMRs confirm the grafting of PMMA on pDCPD structure. Homogeneous aerogels with bulk density $\sim 0.43 \text{ g cm}^{-3}$ were obtained consisting of interconnected nanoparticles with narrow pore size distribution. Such **pDCPD-X-MMA** aerogels demonstrate excellent mechanical strength.

Organofluorine Compounds: From Superacids to Medicinal Chemistry

V. Prakash Reddy, Dept. of Chem., MS&T

Abstract: Due to the unique properties of C-F bond, organofluorine compounds have unusual physicochemical properties. They are components of superacids, widely used polymeric materials, anesthetics, agrochemicals, as well as numerous pharmaceuticals. Understanding the effects of C-F bond on the stabilization of reactive intermediates is essential in designing new synthetic methods, and for the designing of the biologically and medicinally active compounds. In general, fluoroalkyl groups such as trifluoromethyl and *gem*-difluoromethylene groups are expected to destabilize the carbocationic intermediates. However, experimental evidence is of limited scope for the fluorinated carbocation intermediates due to the difficulty of their formation. Insights gained from the effect of substituents on the relative stabilization or destabilization of the carbocations and other reactive intermediates, in conjunction with factors such as hydrophobic and lipophilic nature of fluorinated compounds are of fundamental interest in the design of the biologically and medicinally relevant novel organofluorine compounds. In this context, our studies on some of the fluorinated carbocations in superacid media and overview of our recent synthetic strategies for organofluorine compounds will be discussed.

The Characterization of Ultrafine Particulate Matter from Combustion Processes

Philip D. Whitefield, Dept. of Chem., MS&T

Abstract: This presentation will discuss the need for ultrafine particulate matter (PM) characterization for both environmental, economic and defense related purposes. It will provide a basic introduction to the fast, real time experimental methods employed to determine PM, size distribution, number and mass concentration, composition and hydration properties using extractive sampling methods. The application of these methods will be described using examples from the work of the Aerospace Emissions Program at MS&T. In particular gas turbine engine emission characteristics using conventional and alternative aviation fuels will be described as will the changes observed in the PM properties as the engine exhaust expands and disperses in the engine exhaust plume.

Correlation for Yield of Competitive Reactions in Reactors with Turbulent Mixing

Gary K. Patterson, Dept. of Chemical and Biological Engineering, MS&T

Abstract: A correlation for yield of a product of competitive reactions with turbulent mixing resulted in a plot of [yield/perfect yield] versus a mixing Damkoehler number, the ratio of mixing time to reaction time, written as follows: $Da_M = \tau_M/\tau_R$, where $\tau_M \approx 2(L^2/\varepsilon)^{1/3}$ and $\tau_R = 1/(k_1k_2C_{Af}C_{Bres})^{1/2}$. Such competitive reactions are represented by $A + B \rightarrow R$; $R + B \rightarrow S$ and $A + B \rightarrow R$; $C + B \rightarrow S$. The scale of mixing L is approximated in a stirred vessel by $D_f = (4Q_f/\pi U_{bt})^{1/2}$ and the rate of turbulence energy dissipation rate in a stirred vessel ε is given by $N_i N_p D^2 N^3 / [(T/D)^3 (\pi/4)]$. [Symbols are defined as follows: k_1 and k_2 are rate constants for reactions 1 and 2, C_{Af} and C_{Bres} are molar concentrations of the feed A and the resident B, D_f is the diameter of the feed jet as stretched or compressed by the flow, Q_f is the volumetric feed rate, U_{bt} is the blade tip speed of an impeller, N_i is the ratio $\varepsilon_{feed\ point}/\varepsilon_{average}$, N_p is the impeller power number, N is the impeller rotation rate, T is the vessel diameter, and D is the impeller diameter.]

A recent more detailed dimensional analysis has shown the need to include the effect of the ratio of reaction rate constants, k_2/k_1 , in the correlation, even though the ratio of yield to perfect yield removes most of the divergence. The new correlation effectively separates experimental data for different k_2/k_1 -ratios. The seminar will include the details of the dimensional analysis, consequences for other types of mixed reactors, for instance static mixers, and sources of data used.

Learning in the Undergraduate Chemistry Laboratory: Perspective from a South African Research University

Karen Wallace, University of Western Cape, South Africa

Abstract: First-year chemistry students at most South African universities typically had limited or no exposure to laboratory work while at school. This applies especially to students from lower socio-economic beginnings who attended township or rural schools. The University of the Western Cape (UWC), a historically black university (HBU), draws at least half of its students from this demographic.

My presentation will focus on two aspects of laboratory learning that is addressed by my research in the above context, namely:

- The role of the postgraduate students as facilitators in the first-year laboratory experience; and
- The use of online video clips to support student preparation for laboratories.

Addressing the first of these, I will report on a qualitative study performed in my department to characterise postgraduate participation in the undergraduate laboratories. Teaching assistants (or demonstrators as they are called at South African universities) have become indispensable to the delivery of teaching particularly to first-year students, due mainly to the growing need in tertiary institutions to balance increasing student numbers and needs with pressure on academic staff time and institutional resources. At most universities the role of teaching assistants falls to postgraduate students who are being

trained in disciplinary research. In addition to funding their own studies, their participation in teaching activities is increasingly being recognised as preparation for possible careers in tertiary teaching.

A theoretical framework that views learning as participation in a *community of practice* was used to characterise demonstrators' engagement with their task of facilitating student learning. Learning as participation is more than engagement in the activities and practices of a social or professional group of people; it encompasses both *active participation* and the *construction of an identity* in relation to the group and its practices. I will attempt to present a qualitative interpretation of what learning in a demonstrator/postgraduate community might mean: from emerging conceptions about student learning and the learning of chemistry, to a deeper understanding of the meanings of professional behaviour and academic enterprise. I will also report on the outcomes of an intercessionary process consisting of various measures aimed at improving demonstrating practice in the first-year laboratories at my institution.

The second aspect of laboratory learning that will be addressed is student preparation for laboratory work. Students attending the first-year chemistry laboratory course at UWC are required to prepare a flow diagram, showing the procedure they intend to follow during the execution of the practical, prior to each laboratory session. The rationale behind this requirement is to increase the students' level of preparation for laboratories, with at least some of the transformative tasks occupying the working memory during the execution of the practical taken care of beforehand. In order to assist students in the transformation of procedural texts into flow diagrams, short video clips were uploaded to UWCs e-learning platform. The results of a pilot experiment in which the flow diagrams of students who had watched the clips were compared with those of students who had not watched the clips will be presented.

Chemistry of the Solar System

Dennis L. Whitney, Environmental Engineer, American Airlines, Retired

Abstract: The nature of the creation of the environment we inhabit has intrigued philosophers since the dawn of our conciseness.

The application of the principles of nuclear, inorganic, and geological chemistry to the study of geological specimens and meteorites has led to new insight into the nature of the process that have lead to our current environment. The fact that the earth supports life seems to have an important effect on the nature of this environment.

Probing Actinide Electronic Structure Using High-resolution Photoelectron Spectroscopy

Dr. Michael C. Heaven, Dept. of Chem., Emory University

Abstract: High-level theoretical models of the electronic structures and properties of actinide compounds are being developed by several research groups. This is a challenging problem due to the need for explicit treatment of relativistic effects, and the circumstance that many of these molecules exist in states where the *f* and/or *d* orbitals are partially filled. Current theoretical models are being evaluated through

comparisons with experimental results. Gas phase data are most suitable for this purpose, but there have been very few gas phase studies of actinide compounds. We are addressing this issue by carrying out spectroscopic studies of simple uranium and thorium compounds. Multiple resonance spectroscopy and jet cooling techniques are being used to unravel the complex electronic spectra of these compounds.

Our recent studies have focused on the electronic structures of the oxides, sulfides fluorides and nitrides of Th and Hf. Comparisons between isoelectronic species indicate that relativistic effects play a relatively minor role in the ionic bonding of ThO, ThO⁺, ThF and ThF⁺. ThO and the ions HfF⁺ and ThF⁺ have been identified as excellent candidates for measurements that probe the electric dipole moment of the electron (a manifestation of CP symmetry violation). The characteristics that render these molecules favorable for studies of fundamental constants will be reviewed, and the first gas phase spectra for the Th fluoride and nitride species will be presented.

Photorefractive Polyvinyl Carbazol Composites Using PbS Nanocrystals as a Photosensitizer

Jong-Sik Moon, Dept. of Chem., MS&T

Abstract: Inspired by the promise of enhanced spectral response, photorefractive polymeric composites photosensitized with semiconductor nanocrystals have emerged as an important class of materials. In addition to providing efficient photosensitization, however, extensive study of these hybrid composites has indicated that the inclusion of nanocrystals also provides an enhancement in the charge-carrier mobility, and subsequent reduction in the photorefractive response time. Unfortunately, the included nanocrystals have also been shown in most cases to increase the deep trap concentration, resulting in a significant decrease in the photorefractive performance, specifically diminished diffraction efficiencies as well as reduced two beam coupling gain coefficients. Nonetheless, evidence suggests that this problem can be largely avoided through the inclusion of semiconductor nanocrystals possessing a relatively narrow band-gap. Here, we fully exploit this quality by doping PbS nanocrystals into a newly formulated photorefractive composite based on molecular triphenyldiamine photosensitized with C₆₀. Through this approach, unprecedented response times of 400 ms are observed, opening the door for video and other high-speed applications. It is further demonstrated that this improvement in response time occurs with little sacrifice in photorefractive efficiency, with internal diffraction efficiencies of 72% and two-beam-coupling gain coefficients of 500 cm⁻¹ being measured. A thorough analysis of the experimental data is presented, supporting the hypothesized mechanism of enhanced charge mobility without the accompaniment of superfluous traps. It is anticipated that this approach can play a significant role in the eventual commercialization of this class of materials.

The Chemistry and Remarkable Applications of Cyanoximes and their Metal Complexes

Nikolay Gerasimchuk, Dept. of Chem., Missouri State University

Abstract: During the last two decades my research interests were dedicated to the new class of weak organic acids – cyanoximes. These low molecular weight organic molecules represent series of new ligands for coordination chemistry.^[1] Both unbound to metal free ligands, their Na⁺ and K⁺ salts and other metal complexes show a large spectrum of biological activity from growth regulation in plants to *in vitro* cytotoxicity. Currently 37 cyanoximes are known, and were more than two hundreds cyanoxime complexes synthesized and studied using a variety of different spectroscopic methods and x-ray analysis. Stereochemistry of cyanoxime ligands,^[2] structures and properties of the most interesting coordinations compounds are reviewed in this presentation.^[3,4] A broad spectrum of applications of both cyanoximes and their metal complexes is outlined in this lecture as well.^[5-11]