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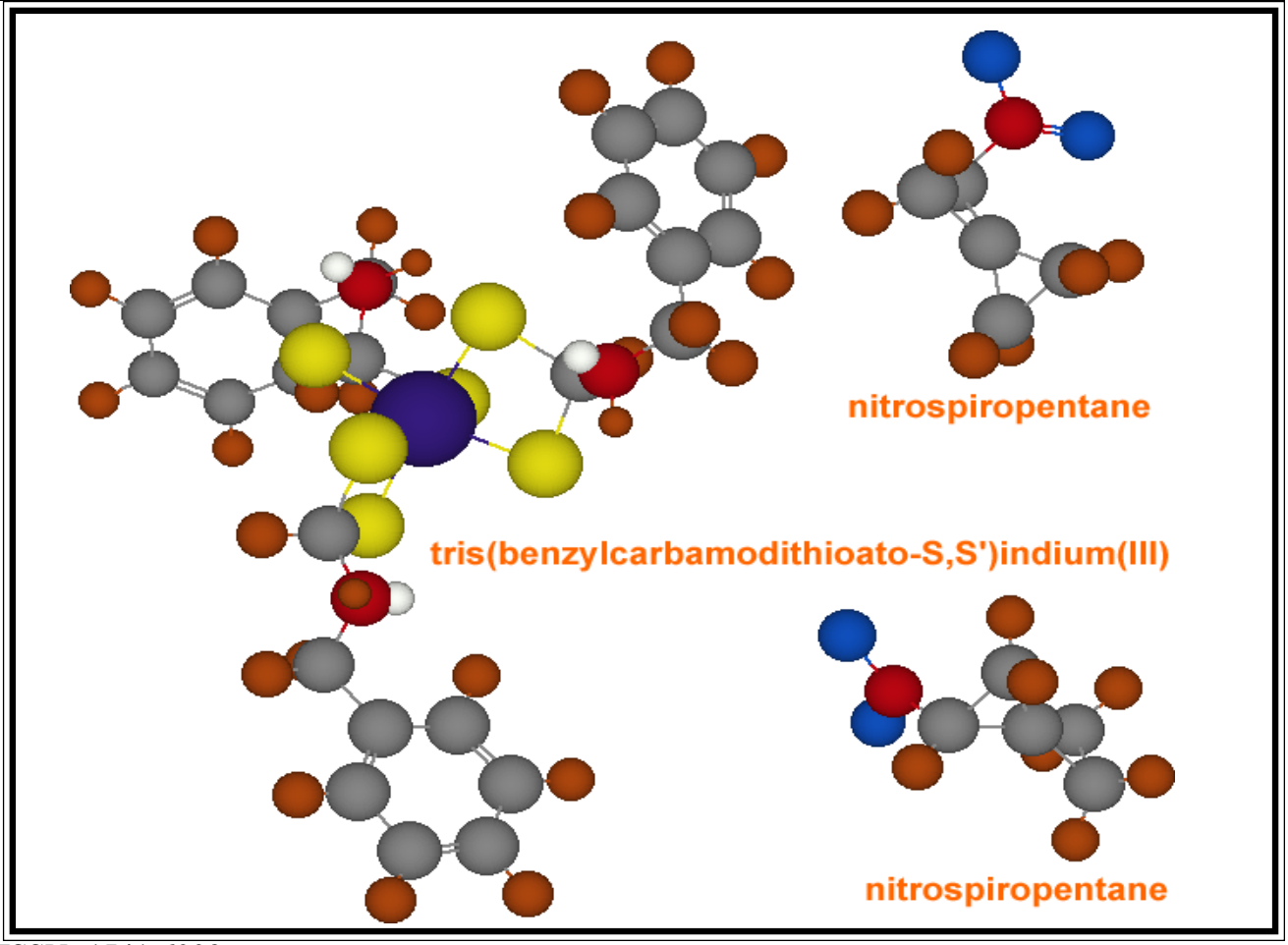

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SAMPLE MATRIX INTERFERENCE PHENOMENON IN THE HEADSPACE-GAS CHROMA TOGRAPHY OF ALDEHYDES

The presence of primary amines in the solutions of volatile aldehydes caused the complete suppression of the GC peaks upon analysis by headspace - gas chromatography when compared to the analysis of solutions containing no amines. After observing that the headspace-gas chromatography detector response of propanal was greatly reduced in the presence of 3-amino-1-propanol, two additional aldehydes and one additional amine were utilized to determine whether this was a general phenomenon. Thus, a 3 by 2 study was designed with the three aldehydes - propanal, butanal, and heptanal - and the two amines 3-amino-1-propanol and 1-aminohexane. Although the headspace-gas chromatography analyses of each of the three aldehydes (in the absence of amines) yielded reproducible GC traces with quantifiable peaks, the aldehyde peaks were completely suppressed when amines were added to the samples. All runs employed toluene as an internal standard and the high-boiling N,N-dimethylacetamide as a solvent. It was concluded that this amine interference phenomenon was general in regard to the headspace-gas chromatography analysis of volatile aldehydes and the *in situ* formation of Schiff bases is proposed, which would result in the reduction of the aldehyde concentrations from the headspace of the sample vials.

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THERMAL DECOMPOSITION PATHWAYS FOR INDIUM(III) AND GALLIUM(III) DITHIOCARBAMATES DERIVED FROM PRIMARY AMINES II. THERMOGRAVIMETRIC ANALYSIS AND INFRARED SPECTRA

Thermogravimetric analyses of seven indium(III) and gallium(III) dithiocarbamate complexes derived from primary amines are reported. These complexes undergo thermal decomposition at significantly lower temperatures than the corresponding complexes derived from secondary amines. The mass loss on decomposition generally corresponds to production of the trivalent sulfide, M_2S_3. The infrared spectra of these complexes are reported along with the preparation and characterization of the previously unreported tris(t-butyldithiocarbamato)indium(III) complex.

Emilie M. Schierloh*[a], Scott M. Brothers*[b], DoBin Choi*[c], Aloysius F. Hepp[d], Michael J. Baird[e], Michael D. Hoops[f] and Norman V. Duffy†
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COMPETITIVE INHIBITION BY DIVALENT METAL CATIONS IN THE HORSERADISH PEROXIDASE/ *O*-PHENYLENEDIAMINE SYSTEM

Studies of the interaction between metal ions and enzyme systems provides a window into the world of metabolism in living systems and the toxicological effects of heavy metals on plants and organisms. Elucidating the structure and function of enzymes is also important in identifying potential drugs and optimizing clinical tests. In this study, we observed the inhibition of horseradish peroxidase (coupled with *o*-phenylenediamine) by metal cations and measured the kinetic parameters, including the specific inhibition constants. All of the cations that were studied (Ni^{2+}, Pb^{2+}, Mn^{2+}, Co^{2+}, and Cu^{2+}) exhibited competitive inhibition. We have compared our values for the inhibition constants with studies in the literature and found that our specific inhibition constants are of the same order of magnitude, in some cases, as the K_I values and IC_{50} values reported in the literature for these metals. Some differences were observed, presumably due to the different forms of peroxidase, different substrates, and varying reaction conditions (pH and temperature).

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NITROSPIROPENTANES AS POTENTIAL HIGH ENERGY MATERIALS

Density functional calculations were performed on a series of nitro-substituted spiropentane molecules to assess their possible value as new high energy materials. Calculations on 64 isomers indicate that, as with other highly nitro-substituted systems, the specific enthalpy of combustion decreased with increasing nitro content. Calculations on 10 of the isomers (with six or more NO_2 groups) having sufficient oxygen for complete oxidation also showed a decrease in specific enthalpies of decomposition with increasing nitro content.

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EFFECT OF DESORPTION OF ALKANES ON THE FLUORESCENCE OF METHYLNAPHTHALENE ON AL_2O_3

If vacuum deposited on dielectric surfaces, many aromatic molecules form excimers when electronically excited. Bilayers of 1- and 2-methylnaphthalenes and homologous series of alkanes were formed on the surface of a single crystal of alumina. When optically pumped, the methylnaphthalenes were fluorescent while the alkanes were non-emissive. Then the surface was heated in a temperature programmed desorption (TPD) experiment and enhancement of the excimer and trap emission from the methylnaphthalene occurred between 4-41 K prior to the desorption of the alkane. The postulated mechanism for this enhanced emission is that the desorbing alkane molecule transfers its kinetic energy to affect the surface morphology of the fluorescing methylnaphthalene adlayer.

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DETERMINATION OF THE EFFECT OF WATER-MISCIBLE ORGANIC SOLVENTS ON THE STABILITY OF DNA DUPLEXES VIA UV THERMAL DENATURATION AND CIRCULAR DICHROISM

Water-miscible organic solvents are commonly used to dissolve insoluble organic molecules to obtain homogenous solutions in the studies of small molecule-DNA interactions. Although it is well known that organic solvents such as DMSO destabilize duplex DNA, reports on the effect of various water-miscible solvents on DNA stability are limited. In the present work, we survey the effect of five common water-miscible organic solvents (methanol, ethanol, DMSO, DMF, and acetonitrile) on the stability of intact DNA and DNA containing a lesion. UV thermal denaturation studies show that the rank order for the destabilizing effect on DNA by these solvents is acetonitrile > DMF > ethanol > DMSO > methanol. Methanol causes minimal destabilization of DNA whereas acetonitrile and DMF dramatically destabilize the investigated DNA duplexes. Ethanol and DMSO have an intermediate effect on the stability of DNA. Circular dichroism results reveal that the destabilization does not result from the conformation change of the DNA duplexes.

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ODMR LINEWIDTH OF 2-INDANONE

The optically detected magnetic resonance (ODMR) spectral linewidth is a good indicator of the uniformity in molecular environment in single crystals of aromatic molecules. The nature of the broadening can be determined by saturating the zero-field transition with continuous wave, cw, energy, while the spectrum is taken and then varying the power of the cw field. For 2-indanone, the entire linewidth decreases asymmetrically with increasing cw power. This observation gives support to the crystallographically inequivalent sets of molecules that are present in the unit cell. Deconvolution of the lineshape indicate that the ODMR line is inhomogeneously broadened and the broadening is attributed to the unresolved zero-field transitions caused by the inequivalent set of molecules.

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AN INEXPENSIVE ROOM-TEMPERATURE CHIRPED-PULSE FOURIER TRANSFORM MICROWAVE (RT-CP-FTMW) SPECTROMETER

An inexpensive room-temperature chirped-pulse Fourier transform microwave (RT-CP-FTMW) spectrometer has been designed and constructed. The majority of the research in the field of microwave spectroscopy today uses pulsed valves to cool molecular samples to extremely cold (~ 1K) temperatures before the microwave measurement is made. We have developed a new RT-CP-FTMW design for measuring room-temperature microwave spectra. The design is similar to that of a recently-developed spectrometer, but is significantly less expensive. Thus, it is well-suited for use at primarily undergraduate institutions (PUIs). The design of the spectrometer is presented, as well as data from representative chemical samples.

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EFFECTS OF VARIOUS LIGANDS ON SPECTROSCOPIC PROPERTIES OF THE URANYL ION

The linear uranyl UO_2^{2+} ($D_{\infty h}$ symmetry) ion has been characterized by electronic absorption spectroscopy in different media. In the media of glacial acetic acid (HOAc)-sodium acetate (NaOAc) mixtures, the magnitude of splitting (cm^{-1}) of the vibronic peaks in the UO_2^{2+} spectra decreased linearly as molar concentration of NaOAc in the media increased from 0 M to 0.30 M. This shows the weakening of the U=O bonds due to coordination of the acetate anion to the central uranium atom of UO_2^{2+}. As a result, a dissociative excited state of UO_2^{2+} was likely formed in the HOAc-NaOAc media, evidenced by a borderline (convergence limit) between the observed lower-energy side vibronic peaks and a higher-energy side continuous absorption band in the spectra. In the media of acetone $[(CH_3)_2CO]$- water (H_2O) mixtures, as the volume ratio of $(CH_3)_2CO:H_2O$ reached 2:1, the UO_2^{2+} spectra exhibited an analogous feature attributable to dissociative excited state. This is reasonably due to a strong coordination of the acetone carbonyl oxygen to uranyl $[(CH_3)_2C=O:ÆUO_2^{2+}]$ likely forming a $[UO_2(acetone)_5]^{2+}$ complex and weakening the U=O bonds.

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IMPROVED SYNTHESIS OF 5-ALKYLIDENE-MALONATE DERIVATIVES OF MELDRUM'S ACID

Several known 5-alkylidenemalonate derivatives of Meldrum's acid were synthesized using a room temperature stirring method in a minimal amount of DMF via a Knoevenagel condensation method using cyclic ketones and aromatic aldehydes as reagents. The corresponding products were characterized via ^1H and ^{13}C NMR studies. The yield of the reaction performed was ideal and compared well with reported literature yields that were obtained from more tedious methods. This method may be a potential route to biologically active analogs of g-aminobutyric acid.

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