

Journal of Undergraduate Chemistry Research

ISSN: 1541-6003
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KINETICS STUDIES OF 3-AMINOPROPYLDI-METHYLMETHOXYSILANE REACTION WITH SILICA AND A MODEL SILSESQUIOXANE SILANOL: THE ROLE OF SOLVENT

The kinetics of 3-aminopropyldimethylmethoxysilane reaction with a silanol containing silsesquioxane (3,5,7,9,11,13,15-heptacyclopentylpentacyclo[9.5.1.1^{3,9}.1^{5,15}.1^{7,13}]octasiloxan-1-ol) and Cab-O-Sil HS5 nanoparticulate fumed silica was investigated. FTIR spectroscopy was used to monitor the loss of silanol content as a function of time. The reactions of the silsesquioxane with the aminosilane in both hexane and tetrahydrofuran solutions was found to follow overall second order reaction kinetics. The O-H stretching frequency of the silsesquioxane silanol in hexane exhibited a sharp absorbance centered at 3710 cm⁻¹, whereas in THF a broad absorbance was observed at 3310 cm⁻¹, indicating very different hydrogen bonding environments. The reaction rate constant at room temperature was found to be 5.86 x 10⁻⁴ mM⁻¹s⁻¹ in hexane, and 1.06 X 10⁻⁵ mM⁻¹ s⁻¹ in THF. The aminosilane reaction with nanoparticulate fumed silica showed a similar solvent dependence on reaction rate. Results suggest that hydrogen bonded silanols are inherently less reactive than non-hydrogen-bonded silanols.

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FORMATION OF MOLECULAR CLUSTERS BY THERMALLY INDUCED PERCOLATION OF WATER THROUGH DIMETHYLNAPHTHALENE AND DIMETHOXYNAPHTHALENE ADLAYERS ON Al₂O₃

Previously, water was found to form weak van der Waals clusters with substituted benzene and naphthalene molecules on surfaces of Al₂O₃ crystals (1-6). The focus of this study was to determine the composition of clusters between water and two naphthalene-like molecules, dimethyl- and dimethoxynaphthalenes. These compounds were chosen because of the likelihood that water would hydrogen bond to the methoxy substituent, and not as likely with the dimethyl analogue. Results indicated that the composition of water in the clusters with dimethylnaphthalenes ranged from 2:3 to 12:1. These ratios were much higher than with dimethoxynaphthalenes. Molecular modeling calculations demonstrated that the intermolecular attraction of the methoxynaphthalenes was much stronger and excluded water from its structure to a greater extent than molecules of dimethylnaphthalenes.

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QUICKLIME PURITY ANALYSIS BY CALORIMETRY

A calorimeter and test method were designed and calibrated for the determination of CaO in quicklime samples for a local lime processing plant. Existing standard methods were either too cumbersome or results were too vague to meet the needs of this small startup company. The total temperature change as a result of quicklime slaking was normalized to the percent CaO present in the sample. Because CaO can react with moisture in the air, experiments were performed to determine the percent composition of the quicklime samples used in order to correct the observed calibration curve. The normalized ΔT versus % CaO data is now being used on site at the processing plant. In addition to presenting the creation of a reaction specific calorimeter and method, this work can serve as a model for successful collaboration with small businesses in need of single analytical capabilities.

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ISOLATION OF DI (2-ETHYLHEXYL) PHTHALATE (DEHP) FROM CHLOROPHYTES *ULVA LACTUCA* AND *ENTEROMORPHA INTESTINALIS* FROM INDIAN RIVER INLET, DELAWARE, USA

¹³C NMR, ¹H NMR, and GC/MS spectra for a compound isolated from two species of green edible seaweeds, *Ulva lactuca* and *Enteromorpha intestinalis* are analyzed. The seaweed samples, harvested from Indian River Marina, Delaware, USA, are part of a study designed to extract and identify the natural products present in seaweed species of the coastal bays of the Delmarva Peninsula in eastern United States. This paper reports on the preliminary result of the isolation and characterization of a natural product found in the listed species. The spectra are identical to that of di (2-ethylhexyl) phthalate or DEHP, as shown by the analysis. DEHP has been identified as a natural product in other organisms, including species of marine algae. However, it has not yet been reported in edible seaweed species from the Delmarva Peninsula. An analysis of the NMR and GC/MS spectra, leading to the identification of the natural product as DEHP is provided.

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NOSYL AS AN EFFICIENT PROTECTING GROUP FOR THE SYNTHESIS OF BIPYRIDINE AZA-CROWN ETHER MACROCYCLES

Four amine protecting groups (tosyl, Boc, Mtr, and nosyl) were investigated as protecting and cyclization agents in the production of bipyridine aza-crown ether macrocycles. The protected macrocycles were subjected to commonly used deprotection methods to determine the optimal conditions needed to cleave the protecting groups yielding the free amines. One protected spermine macrocycle [2] was used as the model compound for our cyclization and deprotection studies. The macrocycles were characterized using ^1H and ^{13}C NMR spectroscopy, and MALDI-TOF mass spectrometry. The peripheral bipyridine moiety of a deprotected macrocycle [3] was then metallated with K_2PtCl_4 to produce the bipyridine aza-crown coordination complex [5]. This coordination complex was characterized using MALDI-TOF mass spectrometry. The nosyl protecting group efficiently couples to a polyamine framework, promotes cyclization, and may be cleaved using mild deprotection conditions.

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DISSOCIATION ENERGIES OF $\text{N}_8\text{C}_4\text{H}_4$ AND $\text{N}_{12}\text{C}_4\text{H}_4$: HOW DOES NITROGEN ENRICHMENT AFFECT STABILITY?

Complex forms of nitrogen are of scientific interest for their potential as high-energy materials, since dissociation reactions of the type $\text{N}_x \rightarrow (x/2)\text{N}_2$ are strongly exothermic. However, many such N_x molecules are too unstable to serve in a practical application. Previous studies on cage isomers of $\text{N}_6\text{C}_6\text{H}_6$ and $\text{N}_8\text{C}_8\text{H}_8$ have shown the ability of carbon to stabilize nitrogen systems, with a corresponding cost in energy production because of the dilution of the nitrogen content. $\text{N}_6\text{C}_6\text{H}_6$ and $\text{N}_8\text{C}_8\text{H}_8$ are both only 52% nitrogen by mass, and it would be preferable to design stable high-energy materials that are richer in nitrogen because of the additional energy release from the nitrogen. In the current study, cage isomers of $\text{N}_8\text{C}_4\text{H}_4$ (an enriched form of $\text{N}_6\text{C}_6\text{H}_6$) and $\text{N}_{12}\text{C}_4\text{H}_4$ (an enriched form of $\text{N}_8\text{C}_8\text{H}_8$) are studied to determine their stability with respect to dissociation. Theoretical calculations using Hartree-Fock theory and perturbation theory are carried out to calculate dissociation energies of $\text{N}_8\text{C}_4\text{H}_4$ and $\text{N}_{12}\text{C}_4\text{H}_4$. Stability with respect to dissociation, as well as the potential usefulness of each molecule, is discussed.

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THE EFFECT OF SOLVENT TYPE ON QUADRUPOLEAR SPLITTING OF DEUTERATED PROBE MOLECULES IN STRAINED ELASTOMERS

Deuterium Nuclear Magnetic Resonance Spectroscopy has been shown to be a powerful tool to probe molecular order in networks swollen with a solvent. This study focused on characterizing the effect of solvent quality on quadrupolar splitting of deuterated probe molecules. Several deuterated solvents were used as swelling agents and the degree of quadrupolar splitting was measured for each solvent as the degree of elongation was varied. The results suggest that there is a good correlation between the solvent quality and quadrupolar splitting.

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DETERMINATION OF INORGANIC PHOSPHATE SORPTION TO IRON OXIDES AS A FUNCTION OF MINERAL CRYSTALLINITY, SALINITY AND DEPTH IN ESTUARINE SEDIMENTS BASED ON AN HCl EXTRACTION METHOD

Investigating the transport and fate of particulate phosphorus in estuaries is key to mitigating the eutrophication of valued water resources. Changes in particulate phosphorus (P) that occur with changes in estuarine salinity, sediment depth and crystallinity of iron minerals were investigated in this study. Inorganic phosphorus was extracted from sediments collected along the downstream salinity gradient of the Patuxent River, a subestuary of Chesapeake Bay, using three different concentrations of HCl that differentiate fractions of particulate phosphorus. Our findings were also compared to P extracted with citrate-bicarbonate-dithionite. The most dynamic fraction of particulate P occurred in surficial sediments, which were bound to the poorly crystalline iron oxides. This fraction decreased to less than half of total P with increasing salinity, and could account for greater P bioavailability that occurs in estuaries with increasing salinity.

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