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SEQUENTIAL SIMPLEX OPTIMIZATION OF A GC SEPARATION OF HEXANE, CHLOROFORM, AND 2-METHYL-2-PENTENE

The optimization of a GC separation of hexane, chloroform, and 2-methyl-2-pentene using the sequential simplex method is presented. Sequential simplex optimization is a local hill-climbing algorithm that moves a figure with (k + 1) vertexes through a k-dimensional factor space toward a region that will produce an improved response. The goal of this study is to achieve chromatographic separation of each component in as few manipulations of the variables as possible. Baseline separation of all three analytes was achieved within six iterations of the simplex.

S. Lakhani*, J.A. Palasota†, and W.N. Tinnerman II
†Department of Chemistry & Physics, University of St. Thomas, 3800 Montrose Boulevard, Houston, Texas 77006 (USA), japalaso@stthom.edu

COMPARISON OF ACRYLAMIDE CONTENT IN CORN AND POTATO SNACK CHIPS VIA HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Acrylamide has been shown to be present in fried, starchy foods. Since acrylamide is a known carcinogen and neurotoxin, a recently developed LC/MS method to detect and quantify its presence in foods is an important achievement. We have quantified the acrylamide content of selected corn and potato snack chips using a previously reported reversed-phase HPLC/UV detection method. Samples of aqueous extracts (n=6) obtained from homogenized chips were processed through a series of high-speed centrifugations followed by filtration with a 0.2 µm HPLC syringe filter. Reversed phase HPLC was performed on dilutions of these samples using an isocratic method and UV detection. It was found that corn chips contained the lowest acrylamide concentration (0.0441±.01 µg/mL) and interestingly the baked potato chips had a higher concentration (1.1916±.35 µg/mL) than fried potato chips (0.3891±.027 µg/mL). The exact mechanism and causative factors behind this difference in acrylamide content deserve further attention.

Jonathan Wagner* and Nick Flynn†
†Nick Flynn, Angelo State University, San Angelo, TX 76909, Nick.Flynn@angelo.edu

ENANTIOPURE CYCLOPALLADATED and COORDINATION COMPLEXES of (*S*)-4-ETHYL-2-PHENYL-2-OXAZOLINE

The reaction of L(-)-2-amino-1-butanol and benzonitrile in the presence of $ZnCl_2$ afforded (*S*)-4-ethyl-2-phenyl-2-oxazoline [1] in 85% yield. Direct *ortho*-palladation of 1 using $Pd(OAc)_2$ and NaOAc in AcOH provided (*S,S*)-di-µ-acetatobis{2-[2-(4-ethyl)oxazolinyl]phenyl-*C,N*}dipalladium(II) [2], which was converted *in situ* into the corresponding µ-chloro analog 3 using LiCl in MeOH with an overall yield of 57%. The coordination complex dichlorobis[(*S*)-4-ethyl-2-phenyl-2-oxazolinyl]palladium(II) [4] was obtained by the reaction of 1 with $PdCl_2$ in MeOH in 68% yield. Dimer 3 was converted into its mononuclear PPh_3 adduct 5 in 94% yield. The structures of complexes 3-5 were proven by NMR and IR spectroscopy.

Christopher J. Dehen*, Kristopher J. Keuseman, Irina P. Smoliakova†
†Chemistry Department, University of North Dakota, P.O. Box 9024, Grand Forks, ND 58202, ismoliakova@chem.und.edu

THERMAL DECOMPOSITION PATHWAYS FOR INDIUM(III) AND GALLIUM(III) DITHIOCARBAMATES DERIVED FROM SECONDARY AMINES

Three dithiocarbamate complexes (the dibenzyl, morpholine and pyrrolidine derivatives) of indium(III) and gallium(III) have been prepared and characterized by elemental analysis, infrared spectra and melting point. Each complex was decomposed thermally and its decomposition products separated and identified with the combination of gas chromatography/mass spectrometry to determine their suitability as precursors for the fabrication of photovoltaic cells.

Michael D. Hoops,*[a] Aloysius F. Hepp,[b] and Norman V. Duffy†
Department of Chemistry, Wheeling Jesuit University, Wheeling WV, 26003, nduffy@wju.edu
[a] Department of Chemistry, Northwestern University, Evanston IL, 60208
[b] Photovoltaic and Space Environments Branch, NASA Glenn Research Center at Lewis Field, Cleveland OH, 44135

OXIDATION of SELECT PAHS BY $[(bpy)_2(py)Ru^{IV}(O)]^{2+}$ IN SOLUTION AND SILICA SOL-GEL MEDIA

Silica sol-gel derived hybrid materials containing a ruthenium-oxo metal complex in a porous network were prepared and tested for efficacy in the stoichiometric oxidation reaction of anthracene and phenanthrene. GC-MS data reveal the primary oxidation products to be anthraquinone and phenanthrene quinone. A ruthenium-aqua complex was anchored to the silica matrix by condensation of $Si(OR)_4$, where R = Et, Pr or Bu, under either acidic and basic conditions. The reactions between $[(bpy)_2(py)Ru^{IV}(O)]^{2+}$ {(bpy = 2,2'-bipyridine, py = pyridine } and the organic substrates were investigated in both acetonitrile solution and with sol-gel media as the support for the catalyst. Inhibition of the metal-based comproportionation process was observed with the catalysts entrapped in the gels as compared to the solution studies.

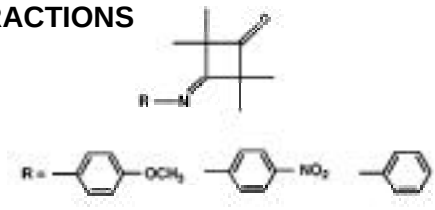
Rachel Kusowski*, Danniel Renne*, and Estelle Lebeau†
268 Dow Science Complex, Central Michigan University, Mt. Pleasant, MI 48859, lebea1el@cmich.edu

CHEMICAL AGENT DECONTAMINATION CHEMICAL TERRORISM ATTACK DECONTAMINATION– HOUSEHOLD OXIDIZERS: WHAT WORKS

Several household oxidizer cleaners were examined as potential decontamination agents for use against chemical weapons. The household oxidizers were bleach (5.25%), OxiClean, a sodium percarbonate formulation, and standard 3% hydrogen peroxide solution. Diazinon was selected as the model organophosphate for the nerve agents. The results show that bleach is the best oxidizer under various conditions, though Oxiclean may have some value in cases where bleach is not suitable. Dilute bleach (0.525%) recommended by military manuals for localized decontamination of skin does work, but it requires longer than 10 minutes for effectiveness, a time factor that may not be practical for the more poisonous military nerve agents.

Fred Walters & Eric R. Taylor†
Department of Chemistry, University of Louisiana at Lafayette, Lafayette, LA 70504-4370, ertaylor@louisiana.edu

FREE ENERGY OF ACTIVATION FOR THE INVERSION OF GROUPS ATTACHED TO NITROGEN IN 3-IMINO-CYCLOBUTANONES: LONG RANGE LONE PAIR ORBITAL INTERACTIONS



[1] [2] [3]

Variable temperature NMR was used to determine the coalescence temperature for the methyl signals of the compounds shown. The free energy of activation was calculated and the values were directly correlated to the long range orbital interactions in the transition state between the heteroatoms in the 1,3-positions.

Hing-Lun A. Chong*, Monica Phelps* and James J. Worman†
Department of Chemistry, Rochester Institute of Technology, 85 Lomb Memorial Drive, Rochester, NY 14623, JJWSCH@RIT.edu

THE CONTROL OF THE WAGNER-MEERWEIN REARRANGEMENT IN A RITTER REACTION

It is known that the treatment of a solution of a-isopropylbenzyl alcohol [1] in acetic acid with benzonitrile in the presence of concentrated sulfuric acid at 70 ºC (Ritter reaction) gives a 1:1.4 mixture of *N*-(a-isopropyl-benzyl)benzamide [2] and *N*-(a,a-dimethylphenethyl) benzamide [3] (under kinetic control) or essentially pure [3] (under thermodynamic control). We describe herein that the treatment of [1] in dichloromethane solution, in the presence of benzonitrile, with triflic anhydride at –30ºC gives a 16.5:1 mixture of [2] and [3]. The control of the Wagner- Meerwein rearrangement described in this report could be useful, in other cases, for the preparation of a specific regioisomer increasing the versatility of the Ritter reaction.

Valeria Justribó*, María I. Colombo and Edmundo A Rúveda†
Instituto de Química Orgánica y de Síntesis (CONICET-UNR), Facultad de Ciencias Bioquímicas y Farmacéuticas, Suipacha 531, 2000 Rosario, Argentina, eruveda@fbioyf.unr.edu.ar