The Synthesis and Spectral Characterization of 
$N,N'$-Bis(2-[(2-methyl-2-phenyl-1,3-dioxolan-4-yl)methyl]amino)ethyl)$N'$,$N''$-dihydroxyethanediimidamide
and Its Complexes

by E. Canpolat$^1$, M. Kaya$^1$ and Ö.F. Öztürk$^2$

$^1$Department of Chemistry, Faculty of Arts and Sciences, University of Firat, 23119, Elazığ, Turkey
$^2$Department of Chemistry, Faculty of Arts and Sciences, University of Onsekiz Mart, 17100, Çanakkale, Turkey

(Received January 14th, 2004; revised manuscript June 2nd, 2004)

$N,N'$-Bis(2-[(2-methyl-2-phenyl-1,3-dioxolan-4-yl)methyl]amino)ethyl)$N'$,$N''$-dihydroxyethanediimidamide (LH$_2$) has been synthesized and its mononuclear complexes with Co$^{2+}$, Ni$^{2+}$, Cu$^{2+}$, Zn$^{2+}$ and Cd$^{2+}$ ions have been obtained. The mononuclear complexes indicated that the metal ions are ligand coordinated through the two N atoms, as do most of the vic-dioximes. The authenticity of the ligands and their complexes have been established by IR, $^1$H- and $^{13}$C-NMR spectra, UV-Visible spectroscopy, elemental analyses and magnetic susceptibility measurements.
Vitexoside, new flavonoid glycoside and hexatetracontanoic acid derivative have been isolated from the roots of *Vitex negundo* and assigned structures sakuranetin 4'-(6'-O-α-L-rhamnopyranosyl)-β-D-glucopyranoside 1 and 3-(3-methoxy-4-hydroxyphenyl) propyl hexatetracontanoate 2 respectively. In addition agnoside 3, 5-hydroxy-1,3-benzenedicarboxylic acid 4, R-dalbergiphenol 5 and R-4-methoxy dalbergione 6 were also isolated for the first time from this species.
Alkoxyalumoxanes (ROAlO)$_n$ – Synthesis and Properties

by A.R. Kunicki, S. Pasynkiewicz, J. Jankowski and B. Mańk

Faculty of Chemistry, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland

(Received May 18th, 2004)

Two reaction methods were employed to prepare alumoxanes of general formula [(RO)AlO]$_n$. The first one from Et$_3$Al, H$_2$O and ROH [R = CH$_3$, (CH$_3$)$_2$CH]; the second one from Et$_3$Al, ROH [R = (CH$_3$)$_2$CH, CH$_3$(CH$_2$)$_2$] and H$_2$O. The properties, chemical composition, aggregation and proposed structure of the final products are discussed based on gel permeation chromatography, molecular weight determination and spectroscopic data.
An efficient synthesis of 2-alkyl-5-nitrobenzofurans from 5-nitrosalicylaldehyde and 2-bromoesters via 2-(2-formyl-4-nitrophenoxy)alkanoic acids is described.
Synthesis of Fluorinated Dialkyl 1-Aryl-4-alkoxy-5-oxo-2,5-dihydro-1H-pyrrole-2,3-dicarboxylates

by I. Yavari, F. Nasiri and H. Djahaniani

Department of Chemistry, Tarbiat Modarres University, PO Box 14115-175, Tehran, Iran
Fax: +98(21)8006544; E-mail: isayavar@yahoo.com

(Received May 31st, 2004)

Alkyl 2-(2-fluoro-anilino)-2-oxo-acetates or ethyl 2-oxo-2-(trifluoromethylanilino)-acetate undergo a multistep reaction with dialkyl acetylenedicarboxylates in the presence of triphenylphosphine to produce dialkyl 1-(2-fluorophenyl)-4-alkoxy-5-oxo-2,5-dihydro-1H-pyrrole-2,3-dicarboxylates or dialkyl 4-ethoxy-5-oxo-1-[2-(trifluoromethyl)-phenyl]-2,5-dihydro-1H-pyrrole-2,3-dicarboxylates in good yields. Dynamic NMR study of dimethyl 4-ethoxy-5-oxo-1-[2-(trifluoromethyl)-phenyl]-2,5-dihydro-1H-pyrrole-2,3-dicarboxylate shows a fairly high energy barrier ($\Delta G^* = 60.9 \text{ kJ mol}^{-1}$) for rotation around the N-aryl single bond, which leads to an observable atropisomerism.
Derivatization of Aminoacids with Fluorine Containing Markers – a Way for Their Determination by $^{19}$F NMR Spectroscopy

by P. Szczeciński and D. Bartusik

Warsaw University of Technology, Faculty of Chemistry,
Noakowskiego 3, 00-664 Warszawa, Poland

(Received June 8th, 2004; revised manuscript June 22nd, 2004)

A new, water soluble marker, 2, suitable for analysis of aminoacid mixtures, as well as its derivatives 3 and 4 with several aminoacids have been prepared and characterized by NMR spectroscopy. Remarks on benzimidazole ring formation have been given.
Luminescence of Bismuth, Cerium and Terbium in Alkaline Earth Borates

by Y. Xie\textsuperscript{1}, S. Zhang\textsuperscript{2} and Q. Zeng\textsuperscript{3}

\textsuperscript{1}Shekou Laboratory, Intertek Testing Services Shenzhen Ltd., 518067, P. R. China
\textsuperscript{2}Department of Chemistry, Shaoguan College, 512005, P. R. China
\textsuperscript{3}Key Laboratory of Rare Earth Chemistry and Physics, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, 130022, P. R. China

(Received November 24th, 2003; revised manuscript March 29th, 2004)

The luminescence of Bi\textsuperscript{3+}, Ce\textsuperscript{3+} and Tb\textsuperscript{3+}-activated BaB\textsubscript{8}O\textsubscript{13} and Sr-borates are studied. The emission peak of Ce\textsuperscript{3+} in alkaline earth borates shifts to higher energy side with decreasing ratio of SrO/B\textsubscript{2}O\textsubscript{3}. The energy transfer from Ce\textsuperscript{3+} to Tb\textsuperscript{3+} in BaB\textsubscript{8}O\textsubscript{13} is studied.

PHYSICAL CHEMISTRY
The State Dependency of the Effective Pair Potential Parameters

by E. Keshavarzi\textsuperscript{1}, F.S. Hashemi\textsuperscript{2} and Z. Ghazvini\textsuperscript{3}

\textsuperscript{1}Dept. of Chemistry, Isfahan University of Technology, Isfahan, 84154, Iran
\textsuperscript{2}Dept. of Chemistry, Faculty of Sciences, Azad University, Tehran, Iran
\textsuperscript{3}Dept. of Chemistry, Faculty of Basic Sciences, Mazandaran University, Babolsar, Iran

(Received November 12th, 2003; revised manuscript April 26th, 2004)

The state dependency of the effective pair potential parameters $\varepsilon_{\text{eff}}(T, \rho)$, $\kappa_{\text{eff}}(T)$ has been studied for gas, liquid and supercritical regions for different types of fluids. We have assumed that the configuration of potential energy between $N$ molecules can be divided into independent pair clusters with an effective pair potential, in which the parameters of the potential are state dependent. We have obtained the values of $\varepsilon_{\text{eff}}(T, \rho)$ and $\kappa_{\text{eff}}(T)$ using $p-V-T$ data. The results show, that $\varepsilon_{\text{eff}}(T, \rho)$ increases with density for those thermodynamic states, at which pair interactions are dominant, while it decreases with density for those systems, where triplet and higher clusters are dominant. Both $\varepsilon_{\text{eff}}(T, \rho)$ and $\kappa_{\text{eff}}(T)$ decrease with temperature, which coincides with the literature data. A remarkable result of the present work is the determination of density, at which triplet clusters come into account. We have also shown new corresponding states for the effective well depth parameter, which is held for all examined fluids, including Ar, Xe, CH\textsubscript{4}, N\textsubscript{2}, CO, H\textsubscript{2}O, CO\textsubscript{2}, CH\textsubscript{3}OH, C\textsubscript{2}H\textsubscript{6} and C\textsubscript{6}H\textsubscript{6} for different isotherms. A linear dependence of $\varepsilon_{\text{eff}}(T, \rho)$ versus density and temperature on the zeno-line is predicted.

Quantum Chemical Modelling of the Oxidation of Myoglobin

by E. Ilkowska¹, M. Witko², R. Tokarz-Sobieraj² and G. Stochel¹

¹Faculty of Chemistry, Jagiellonian University, ul. Ingardena 3, 30-060 Cracow, Poland
fax: +48 12 636 53 92; e-mail: stochel@chemia.uj.edu.pl

²Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences,
ul. Niezapominajek 8, 30-239 Cracow, Poland
fax: +48 12 425 19 23; e-mail: ncwitko@cyf-kr.edu.pl

(Received February 9th, 2004; revised manuscript April 29th, 2004)

The electronic structure (charge distribution, bond indices) and the geometry (bond distances and angles) of the deoxyheme and the oxyheme with coordinated proximal histidine in their reduced and oxidized form were determined by the INDO method. The effect of the distal histidine (in the case of the oxyheme) and a water molecule (in the case of the metheme) on the geometry, charge distribution and stability of the systems was investigated. The method was adopted to model the oxidation of myoglobin in biological systems. The results revealed that both deoxy- and oxymyoglobin could spontaneously undergo one-electron oxidation. The mechanistic considerations based on the charge distribution and energetic effects led to the conclusion, that in oxymyoglobin’s case the electron transfer are followed by dissociation of a dioxygen molecule and addition of a water molecule, where both processes proceed in parallel.
Photoresponse of Undoped and W-Doped TiO$_2$

by M. Radecka, P. Sobaś, A. Trenczek and M. Rękas

AGH University of Science and Technology, Faculty of Materials Science and Ceramics,
al. Mickiewicza 30, 30-059 Kraków, Poland

(Received March 2nd, 2004; revised manuscript May 13th, 2004)

Undoped and W-doped polycrystalline TiO$_2$ were investigated. The kinetics of recombination processes has been studied. The flat band potential of TiO$_2$ was determined (−0.11 V in respect to saturated calomel electrode, at pH = 7.8). Basing on the photocurrent experiments, the effect of W concentration on solar energy conversion efficiency was discussed. It was found, that addition of W leads to an increase of solar-to-hydrogen energy efficiency. The maximum energy conversion efficiency has been observed for voltage bias ca 0.65 V. The highest energy conversion efficiency has been observed for TiO$_2$ doped with 0.1 at % W.
Crystal Structure, Infrared Spectra and Density Functional Theory Study on 2,3-Diketo-benzopiperazine Dimer

by P.S. Zhao\textsuperscript{1}, F.F. Jian\textsuperscript{2}, H.L. Xiao\textsuperscript{2} and P.P. Sun\textsuperscript{2}

\textsuperscript{1}Department of Chemistry, Huaiyin Teachers’ College, Huai’an, Jiangsu, 223001, P.R. China
\textsuperscript{2}New Materials & Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao Shandong 266042, China
E-mail: ffj2003@163169.net

(Received April 15th, 2004; revised manuscript May 18th, 2004)

2,3-Diketo-benzopiperazine, which exists as dimeric form in its crystal structure has been synthesized. The calculated results on the dimer at B3LYP/6-31G\textsuperscript{*} level show that the average strength of the double hydrogen bonds is of medium-grade. Natural bond orbital analyses have been performed. The predicted harmonic vibration frequencies support the experimental values. The thermodynamic properties of the dimer at different temperatures have been calculated and the change of Gibbs free energy for the aggregation from the monomer to the dimer \( \Delta G_T = -30.86 \text{ kJ/mol} \) at 298.15 K, which implies the spontaneous process of the dimer formation.
Crystal Structure and *Ab Initio* Studies on \((p\text{-Methoxyphenyl})\text{thiosemicarbazide}\)

by P.S. Zhao\(^1\), F.F. Jian\(^2\) and Y.X. Hou\(^2\)

\(^1\)Department of Chemistry, Huaiyin Teachers’ College, Huai’an, Jiangsu, 223001, P. R. China
\(^2\)New Materials & Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao Shandong 266042, P. R. China
E-mail: ffj2003@163169.net

(Received May 28th, 2004)

\((p\text{-Methoxyphenyl})\text{thiosemicarbazide} \left[\text{CH}_3\text{OC}_6\text{H}_4\text{NHNHCSNH}_2\right] \) has been prepared and characterized by elemental analysis, IR, electronic absorption spectra and X-ray single crystal diffraction. In the crystal lattice, there exist some intermolecular hydrogen bonds, \(\pi-\pi\) stacking interactions and \(C-H-\pi\) supramolecule interactions, which stabilize the crystal structure. *Ab initio* calculations at HF/6-31G* level of the structure, charges distribution, electronic spectra, natural population analysis and thermodynamic properties at different temperatures have been performed. The calculated results show that the sulfur atom and nitrogen atoms have bigger negative charges, which result in they are the potential sites to react with the metallic ions. The electronic transitions associated with the electronic absorption spectra are mainly derived from the contribution of bands \(\pi \rightarrow \pi^*\).
Temperature Programmed Desorption of Pyridine and 2,6-Dimethylpyridine from Differently Pretreated Pd/Al₂O₃ Catalysts

by M. Skotak¹ and Z. Karpiński¹,²

¹Institute of Physical Chemistry of the Polish Academy of Sciences, ul. Kasprzaka 44/52, PL-01224 Warszawa, Poland
²Faculty of Mathematics and Natural Sciences, Cardinal Stefan Wyszyński University, ul. Dewajtis 5, PL-01815 Warszawa, Poland

(Received June 28th, 2004)

The temperature programmed desorption (TPD) of pyridine and 2,6-dimethylpyridine from alumina and two alumina-supported palladium catalysts of different metal loadings (0.3 and 2.77 wt.% Pd) confirmed the presence of strong acid sites in the samples subjected to high temperature reduction at 600°C. Roughly similar amounts of both organic bases were desorbed from the catalysts which underwent similar pretreatments. However, 2,6-dimethylpyridine appears less strongly bonded than pyridine to Lewis acid sites in alumina, apparently because of some steric hindrance produced by the presence of methyl substituents in 2- and 6-position to the nitrogen lone pair. Thus, pyridine is better suited for probing evolution of Lewis acidity in alumina, brought about by high temperature reduction at 600°C. With increasing temperature during thermodesorption, both organic bases adsorbed on palladium-containing samples undergo transformation, leading to desorption of several products, among which hydrogen and nitrogen predominate. Introduction of increasing amounts of palladium to alumina makes the acidity probing difficult, because a considerable part of adsorbed organic base is decomposed on metal sites. Decomposition of pyridine and 2,6-dimethylpyridine may serve as a convenient probe of availability of palladium surface. After high temperature reduction of Pd/Al₂O₃ a considerable part of Pd surface is blocked by support species.
Water-Gas Shift Reaction over the Carbon-Based Magnetite Catalysts Doped with Ruthenium or Palladium

by K. Stołecki¹, W. Raróg-Pilecka², A. Jedynak-Koczuk², E. Miśkiewicz¹ and Z. Kowalczyk²

¹Fertilizers Research Institute, Al. Tysiąclecia P.P., 24-100 Puławy, Poland
²Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, 00-664 Warsaw, Poland

(Received June 7th, 2004)
The Space Group of
Tribenzyl[4-phenyl-1,3,4-thiodiazolyl-2-
mercapto-5(II)-thione]tin(IV)

by S.W. Ng

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
seikweng@um.edu.my

(Received July 13th, 2004)