

## Synthesis, Structure and Magnetic Property of a New Neodymium-Adipate Coordination Polymer with Large Guest Molecules

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A new lanthanide-adipate coordination polymer,  $\{[\text{Nd}_2(\text{ad})_3(\text{H}_2\text{O})_4](\text{H}_2\text{ad})\cdot 4\text{H}_2\text{O}\}_n$  (**1**) ( $\text{H}_2\text{ad}$  = adipic acid), has been hydrothermally synthesized and structurally characterized by X-ray single crystal structure analysis, elemental analysis, infrared spectrum, thermogravimetric analysis and temperature-dependent magnetic susceptibility. The three-dimensional structure of compound **1** is based on edge-sharing  $\text{NdO}_8(\text{H}_2\text{O})_2$  polyhedron chains, which were interconnected by twisted or extended carbon chains of the adipate ligands in [010] and [001] directions, respectively. The network contains large channels with adipic acid and lattice water acting as guest molecules. Above 50 K, the magnetic susceptibility data of **1** can be fitted to the Curie-Weiss law with  $\theta = -41.0$  K and  $c = 3.52$  emu  $\text{Kmol}^{-1}$ .

**Key words:** neodymium compound, adipic acid, guest molecules, hydrothermal synthesis

## Characterization and Antifungal Study of Genuine Oxovanadium(IV) Mixed-Ligand Complexes with Schiff Bases

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New oxovanadium(IV) complexes [VO(L)(A)] (KHL = potassium salt of o-hydroxyacetophenoneglycine, A<sup>1</sup> = 2,2'-bipyridylamine, A<sup>2</sup> = bis(benzylidene)ethylenediamine, A<sup>3</sup> = thiophene-o-carboxaldeneaniline, A<sup>4</sup> = thiophene-o-carboxaldene-p-toluidine, A<sup>5</sup> = bis(benzylidene)-1,8-diaminonaphthalene, A<sup>6</sup> = bis(acetophenone)ethylenediamine) have been isolated and characterized by elemental analyses, infrared spectra, electronic spectra, magnetic measurement and thermogravimetric analyses. An octahedral geometry has been assigned to all the VO(IV) complexes. The antifungal activity of Schiff bases, oxovanadium(IV) complexes, vanadyl sulphate, control (DMSO) and fungicides (bavistin and emcarb) have been evaluated against *A. niger*, *F. oxysporum* and *A. flavus*, which show clear enhancement in the antifungal activity upon complexation with Schiff bases but moderate activity as compared to the standard fungicides bavistin and emcarb.

**Key words:** oxovanadium(IV) complexes, Schiff bases, antifungal activity, fungicides, octahedral geometry

**Determination of Stability Constants of  
Copper(II)/Cobalt(II)–Methionine and  
Copper(II)/Cobalt(II)–Methionine–Cysteine  
(Binary and Mixed) Complexes by Paper  
Electrophoretic Technique**

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The stability constants of copper(II) and cobalt(II) with methionine and cysteine were determined by paper ionophoretic technique. Beside binary, mixed ligand complexes have also been studied, in which methionine and cysteine act as primary and secondary ligand, respectively. The stability constant of Cu(II)–methionine–cysteine and Co(II)–methionine–cysteine mixed ligand complexes were found to be  $2.80 \pm 0.07$  and  $2.44 \pm 0.11$  (logarithm of stability constant values), respectively at ionic strength 0.1 M and temperature of 35°C.

**Key words:** paper electrophoretic technique, copper(II) complexes, cobalt(II) complexes, methionine, cysteine, stability constants

## **Equilibrium and Thermodynamic Studies of Ternary Systems Involving Metal Ions, *N*-(2-Acetamido)iminodiacetic Acid and Pyrimidine Bases**

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Complex formation in the ternary systems:  $M^{n+} + N$ -(2-acetamido)iminodiacetic acid ( $H_2ADA$ ) + some pyrimidine bases of nucleic acid (HL) [thymine, thymidine and uracil], where  $M^{n+} = Cu^{II}, Zn^{II}, Ni^{II}, Co^{II}, Cd^{II}, Pb^{II}, UO_2^{II}$ , and  $Ce^{III}$  have been investigated pH-metrically. The thermodynamic functions ( $\Delta H$ ,  $\Delta G^\circ$ ,  $\Delta S^\circ$ ) associated with the complex formation of  $[Ni^{II}-N$ -(2-acetamido)iminodiacetic acid – thymine or uracil] were also calculated and discussed.

**Key words:** *N*-(2-acetamido)iminodiacetic acid, pyrimidine bases, metal ions, complex formation

## Complexes of Adenine with Metal Ions: Stability and Excited States

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Molecular geometry optimizations and excited state energy calculations were performed for the N9H and N7H tautomers of adenine (Ade) and their complexes with selected transition metal ions ( $\text{Cu}^+$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$  and  $\text{Ag}^+$ ). The ground state geometry optimizations were carried out at the HF and UHF (for open-shell metal ions) levels, while transition energies were calculated using the configuration interaction involving single excited configurations (CIS method). Single point ground state energy calculations were also performed at the full MP2 (UMP2 for open-shell systems) level. The 6-31+G(d,p) basis set was used for all but  $\text{Ag}^+$  containing complexes for which the LANL2DZ basis set was used. Experimental UV-spectra of adenine complexes with  $\text{Cu}^{2+}$  and  $\text{Ag}^+$  metal cations in the water solution were also obtained. These spectra show red-shift of approximately  $1000\text{ cm}^{-1}$  in comparison with that of the adenine. The analysis of the first electronic singlet  $\pi\text{-}\pi^*$  transition energies suggests that metal ion binding to the N1 or N7 atomic sites of adenine generally leads to the red-shift, while the binding at the N9 site leads to blue shift of the absorption maximum of complexes. The most probable binding site for the  $\text{Cu}^+$  and  $\text{Zn}^{2+}$  ions is found to be the N7 atomic site of the N9H tautomer of adenine. Divalent copper  $\text{Cu}^{2+}$  ion would bind at the N1 site. Co-existence of the two forms of adenine complexes with silver ion ( $[\text{N9H-Ade-AgN1}]^+$ , and  $[\text{N9H-Ade-AgN7}]^+$ ) is also revealed.

**Key words:** adenine, metal–adenine complex, *ab initio*, transition energy

## Brauhenefloroside C: A New Acylated Flavone Glycoside from the Fruits of *Stocksia brauhica*

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Phytochemical investigations of the fruits of *Stocksia brauhica* (Sapindaceae) resulted in the isolation of a new acylated flavone glycoside. The structure of the new compound brauhenefloroside C (**1**) was established as 7-[(6-*O*-(2*E*-3-(4-hydroxyphenyl)-1-oxo-2-propenyl)- $\beta$ -D-glucopyranosyl)]-(1 $\rightarrow$ 2)-[(6-deoxy- $\alpha$ -L-mannopyranosyl)]-(1 $\rightarrow$ 3)-[(6-deoxy- $\alpha$ -L-mannopyranosyl)-oxy]-5-hydroxy-2-(4'-hydroxyphenyl)-4H-1-benzopyran. The structure elucidation of the new compound was based primarily on 1D and 2D NMR analysis, including COSY, 2D *J*-resolved, HMBC and HMQC correlations.

**Key words:** *Stocksia brauhica*, Sapindaceae, acylated flavone glycoside, 1D and 2D NMR analysis

**Unexpected Formation of  
*N*-(4-Cyano-2-phenyloxazol-5-yl)benzamide  
from Benzoyl Cyanide. Evidence for Sulfonate-Assisted  
Rearrangement of Benzoyl Cyanide  
into Benzoyl Isocyanide**

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Experimental evidence for rearrangement of benzoyl cyanide into corresponding isocyanide is provided by the reaction of benzoyl cyanide with triethylammonium 2-hydroxylaminoimidazolidine-*O*-sulfonate (**2**) which affords *N*-(4-cyano-2-phenyloxazol-5-yl)benzamide (**4**) as a result of the trimerization of benzoyl isocyanide. Compound **4** was further converted into *N*-acetyl-*N*-(4-cyano-2-phenyloxazol-5-yl)acetamide (**6**) upon heating in acetic acid anhydride. Structure of **6** was confirmed by X-ray crystallographic analysis.

**Key words:** benzoyl cyanide, benzoyl isocyanide, 4-cyano-2-phenyl-oxazoles, sulfonate-assisted rearrangement of benzoyl cyanide

## **Synthesis of (1*S*,2*S*)- and (1*R*,2*S*)-3-Amino-1,2-dihydroxypropylphosphonic Acids**

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(1*S*,2*S*)- and (1*R*,2*S*)-3-amino-1,2-dihydroxypropylphosphonic acids were synthesized from (*S*)-3-azido-2-benzyloxypropanal in two steps, which involved addition of dibenzyl phosphite, clean separation of dibenzyl (1*S*,2*S*)- and (1*R*,2*S*)-3-azido-2-benzyloxy-1-hydroxypropylphosphonates followed by azide reduction with simultaneous hydrogenolysis of benzyl protecting groups.

**Key words:** 3-aminophosphonic acids, 1-hydroxyphosphonates, configuration, conformation

## **Diastereoselective Addition of Grignard Reagents to Chiral $\alpha$ -Ketoimides Derived from Oppolzer's Sultam**

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Additions of various Grignard reagents to *N*-pyruvoyl- (**3**) and *N*-phenylglyoxyloyl-(2*R*)-bornane-10,2-sultam (**4**) under thermal and Lewis-acid catalytic conditions are studied. High diastereoselectivity was observed in these reactions, and in the case of vinylmagnesium bromide additions to  $\alpha$ -ketoimide **4** a change of direction of asymmetric induction was found.

**Key words:** Grignard reaction, asymmetric synthesis, diastereoselection, chiral auxiliary

## **A Simple Synthesis and Determination of Alkali Metal Binding Selectivities of Chiral Macrocyclic Tetraamides Derived from L-alanine**

by **A. Szczepańska<sup>1</sup>**, **P. Tarnowski<sup>2</sup>** and **J. Jurczak<sup>1,2</sup>**

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Chiral  $\alpha,\omega$ -diamines react under high-pressure conditions (10 kbar) with  $\alpha,\omega$ -dicarboxylates to give chiral cyclic tetraamides having  $C_2$ -symmetry. The alkali metal ( $Li^+$ ,  $Na^+$ ,  $K^+$ ,  $Rb^+$ , and  $Cs^+$ ) binding selectivities of tetraamides are evaluated by way of electrospray ionization mass spectrometry. The relative binding selectivities are estimated from the mass spectral intensities of the metal complexes.

**Key words:** macrocycles, chiral ligands, cations, complexation selectivity, mass spectrometry

## **Asymmetric Synthesis of Multi-substituted Indolizidines by Intramolecular Addition of Allylsilane to Chiral 2,3-Dihydro-4-pyridones**

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A short and synthetically attractive asymmetric synthesis of substituted indolizidines is described. The discovered reaction sequence consists of the asymmetric aza-Diels-Alder reaction of aldimines with Danishefsky's diene and the intramolecular Hosomi-Sakurai allylation. The absolute configuration of cycloadducts was established by CD-spectroscopy.

**Key words:** asymmetric synthesis, chiral dihydropyridones, conjugate addition, allylsilanes

## Decomposition of Ionic Liquids by Photocatalysis

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A method for the decomposition of ionic liquid (ILs) by photocatalysis has been demonstrated. The participation of ILs in light absorption by TiO<sub>2</sub> was shown. The IL molar mass influence on photocatalytic decomposition was proven. The samples with a lower molar mass had higher stability under UV light in the presence of TiO<sub>2</sub>. The degree of decomposition depends upon the cation and anion. The easiest to degrade proved to be ILs with a long alkyl substituent. Ammonium ILs (choline derivatives) with hydroxyl group proved to be more stable than with acetyl group. The most readily decomposable salts are phosphonium ionic liquids.

**Key words:** imidazolium salts, pyridinium salts, phosphonium salts, quaternary ammonium salts, ionic liquids, photocatalysis

## **Dimerization of Copper Octacarboxyphthalocyanine in Aqueous Solutions**

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Spectral absorption UV-Vis investigations on association of copper(II) octacarboxyphthalocyanine in aqueous solutions are reported. In  $5.0 \cdot 10^{-7}$ – $5.0 \cdot 10^{-5}$  mol/dm<sup>3</sup> concentration range monomers and dimers are mainly present. The calculated dimerization constant  $10^8$  dm<sup>3</sup>/mol at 25°C and the hypsochromic spectral shift for the dimer  $1164 \text{ cm}^{-1}$  (688 nm → 637 nm) indicate a stack-like association of molecules and suggest  $\pi$ – $\pi$  interactions between Pc macrocycles in dimer and higher aggregates, in contrast to hydrogen bonding between carboxylic groups of adjacent Pc molecules.

**Key words:** copper octacarboxyphthalocyanine, association, dimerization, aqueous solutions, UV-VIS spectra

**Spectroscopic Studies of the Interaction Between  
Chloranil and 4-Aminopyridine**

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**Plasma Treatment of Polycarbonates Surface  
in Oxidizing Conditions**

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**Catalytic Hydrogen Transfer Over Magnesia. XIX.  
Liquid Phase Reduction of  $\alpha,\beta$ -Unsaturated Ketone**

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