

## Novel Proton Transfer Compounds Containing 2,6-Pyridinedicarboxylic Acid and Melamine and Their Pb<sup>II</sup> Complex: Synthesis, Characterization, Crystal Structure and Solution Studies

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Two novel proton transfer compounds **LH<sub>2</sub>**, (tataH<sub>2</sub>)(pydc), and **L'H<sub>2</sub>**, (tataH)<sub>2</sub>(pydc), (pydcH<sub>2</sub> = 2,6-pyridinedicarboxylic acid and tata = melamine, 2,4,6-triamino-1,3,5-triazine), were synthesized and characterized by IR, <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy and solid state CPMAS <sup>13</sup>C NMR. The (tataH<sub>2</sub>)<sub>2</sub>[Pb(pydc)<sub>2</sub>]<sub>2</sub>·2tata·4H<sub>2</sub>O complex (**I**) was prepared using **L'H<sub>2</sub>** and lead(II) nitrate, and characterized by IR and NMR spectroscopy and single crystal X-ray analysis. The Pb<sup>II</sup> complex is a binuclear anionic complex [Pb(pydc)<sub>2</sub>]<sub>2</sub><sup>4-</sup>, in which two metal fragments are linked *via* a central ten-membered PbN(13)C(18)C(22)O(24)Pb(A)N(13A)C(18A)C(22A)O(24A) ring. Extensive hydrogen bonding between carboxylate groups, (tataH<sub>2</sub>)<sup>2+</sup> and water molecules throughout the Pb<sup>II</sup> complex together with ion pairing play important roles in stabilizing the corresponding lattice. The protonation constants of the reactants, the equilibrium constants for the reaction of pydc with tata and the stoichiometry and stability of the Pb<sup>II</sup> complex with **L'H<sub>2</sub>** in aqueous solution were investigated by potentiometric pH titrations. The stoichiometry of one of the most abundant complexed species in solution was found to be the same as that of the crystalline lead complex.

**Key words:** lead, dipicolinic acid, melamine, X-ray crystal structure, hydrogen bonds, ion pairs, solution studies

## Synthesis, Characterization and Antiferromagnetic Interaction in Oxalato-Bridged Chromium(III)–Chromium(III) Binuclear Complexes

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Two new  $\mu$ -oxalato-bridged binuclear chromium(III) complexes,  $[\text{Cr}_2(\text{ox})(\text{salen})(\text{bpy})_2](\text{NO}_3)_2$  (**1**) and  $[\text{Cr}_2(\text{ox})(\text{salen})(\text{phen})_2](\text{NO}_3)_2$  (**2**) [where salen = N,N'-ethylenebis(salicylideneimine), ox = oxalato, bpy = 2,2'-bipyridine and phen = 1,10-phenanthroline], have been synthesized by the strategy of 'complex as ligand', and characterized by elemental analyses, molar conductance and magnetic moments of room-temperature measurements, and spectroscopic studies. It is proposed that the two complexes have extended ox-bridged structures consisting of two chromium(III) ions, each in an octahedral environment. The two complexes were further characterized by variable temperature magnetic susceptibility (4.2–300 K) measurements and the observed data were successfully simulated by the equation based on the spin Hamiltonian operator,  $\hat{H} = -2J\hat{S}_1 \cdot \hat{S}_2$ , giving the exchange integrals  $J = -2.78 \text{ cm}^{-1}$  for (**1**) and  $J = -3.85 \text{ cm}^{-1}$  for (**2**). This result indicates the presence of weak antiferromagnetic interaction between the metal ions within each molecule.

**Key words:**  $\mu$ -oxalato-bridge, chromium(III), binuclear complexes, antiferromagnetic interaction

## Synthesis, Characterization of Di-*t*-butyltin Piperazine Carbodithioato Complexes $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NR}$ and Crystal Structures of $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NEt}$ and $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NBz}$

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Ten di-*t*-butyltin piperazine carbodithioato complexes  $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NR}$  (R = Et **1**, Bz **2**, H3, Me **4**, Ph **5**, 4-CIPh **6**, 2-CIPh **7**, 3-CIPh **8**, <sup>n</sup>Pr **9**, <sup>t</sup>Bu **10**) have been synthesized and characterized by elemental analysis, UV, IR and NMR spectroscopies. The structures of  $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NEt}$  (**1**) and  $t\text{Bu}_2\text{Sn}(\text{Cl})\text{S}_2\text{CN}(\text{CH}_2\text{CH}_2)_2\text{NBz}$  (**2**) have been determined by X-ray diffraction. The crystals of complexes **1** and **2** belong to monoclinic and triclinic system with space group  $P2(1)/c$  and  $P\bar{1}$ , respectively, with cell dimensions: **1**,  $a = 13.066(2)$  Å,  $b = 12.560(2)$  Å,  $c = 13.066(2)$  Å,  $\beta = 97.043(10)^\circ$ ,  $Z = 4$ ,  $V = 2128.0(6)$  Å<sup>3</sup>,  $F(000) = 936$ ,  $R_1 = 0.0375$ ,  $wR_2 = 0.1011$  and **2**,  $a = 8.2658(16)$  Å,  $b = 10.927(2)$  Å,  $c = 14.182(3)$  Å,  $\alpha = 97.29(3)^\circ$ ,  $\beta = 91.05(2)^\circ$ ,  $\gamma = 98.54(3)^\circ$ ,  $Z = 2$ ,  $V = 1255.6(4)$  Å<sup>3</sup>,  $F(000) = 532$ ,  $R_1 = 0.0543$ ,  $wR_2 = 0.1617$ . In the crystals of the two complexes, the tin atoms are all rendered five-coordination in a trigonal bipyramidal configuration by coordinating with both S atoms of dithiocarbamate groups.

**Key words:** diorganotin, heterocyclic dithiocarbamate, synthesis, crystal structure

## **Studies on Transport of Pb(II) Ions Using Different Crown Ethers**

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Membrane based separation processes represent a sophisticated way to limit industrial wastes in the natural environment. Crown ethers have been recognized as very effective class of compounds to achieve selective separation of heavy metal ions from aqueous solutions. Lead (Pb) is a well known toxic metal and is heavily used in industry. Therefore, finding ways to selectively remove Pb(II) from aqueous solutions is important. In the present paper, different crown ethers of varying cavity size, substituent groups, donor atoms, and ring number are compared for their ability to transport  $\text{Pb}^{2+}$  through a supported liquid membrane. All experiments were carried out in a laboratory scale membrane reactor with crown ether solution immobilized on a polypropylene porous sheet interposed between feed and strip solutions. It was observed that when O atoms of the same sized macrocycle are replaced with N or S atoms, percentage recovery of Pb(II) increases significantly. By substituting a benzene ring on the same sized macrocycle, the percentage of Pb(II) transport increased from 78 to 86%. The cavity size of the crown ether seems to affect the rate of transport. After different trials, it was found that transport is maximal where the ionic radius of Pb(II) matches maximum with the cavity diameter of the macrocycle. These interactions of crown ethers with Pb(II) are explained on the basis of metal-ligand coordination chemistry.

**Key words:** membrane transport, lead, crown ethers, separation

## Synthesis of New Derivatives of 1,2,4-Triazol-3-one

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Reactions of nucleophilic substitution on 2,5-diphenyl-, 1,5-diphenyl-, and 5-phenyl-1,2,4-triazol-3-one were studied. Both *O*- and *N*-derivatives were obtained. Molecular structure proposed for this group of compounds was confirmed by X-ray structure analysis of selected crystals (**4a**, **2b**, **4b**, **5c**).

**Key words:** 1,2,4-triazol-3-one derivatives, reaction of substitution, X-ray structure analysis

## **An Improved Method for the Synthesis of Macrocyclic Benzodiamides and Dibenzotetraamides**

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Seventeen new macrocyclic diamides and tetraamides have been synthesized *via* amidation reactions of three various methyl phenoxyacetates, readily available from simple dihydroxybenzenes, with  $\alpha,\omega$ -diamines in methanol or ethylene glycol as solvents.

**Key words:** amidation reaction, macrocycles, diamides, tetraamides

## ***N*-Substituted 5-Aryl-1,3-oxazolidin-2-ones from Arylethylene Oxides**

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Substituted 5-aryl-1,3-oxazolidin-2-ones can be efficiently prepared from arylethylene oxides and *tert*-butyl alkylcarbamates in the presence of catalytic amount of potassium *tert*-butoxide as a base. Their further hydrolysis under basic conditions provides aryl substituted aminoethanols in high yields.

**Key words:** epoxide, carbamate, aminoalcohol

## **From *o*-Substituted Bromoselenobenzenes to Selenaheterocycles**

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The reactions of two *o*-substituted bromoselenobenzenes: 2-(bromoseleno)benzene sulfonyl chloride and 1,2-di(bromoseleno)benzene with bisnucleophiles having amino, thiol and active methylene group have been studied. In most cases five membered rings, containing selenium as well as nitrogen and sulfur atoms, such as 1,3,2-benzothiaselenazole, 1,2,3-dithiaselenazole, benzo-1,3(2H)-thiaselenole, 1,3,2-benzodiselenazole and benzo-1,3(2H)-diselenole, were formed. The elaborated procedures have synthetic value.

**Key words:** selenium compounds, selenenylation, selenaheterocycles, azaheterocycles, thiaheterocycles

## Interactions of the Oxidation Products of Papaverine with Guanine Quadruplexes

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The interactions of G-quadruplex DNA with two oxidation products of papaverine, 6a,12a-diazadibenzo-[a,g]fluorenylium derivative (**1**) and 2,3,9,10-tetramethoxy-12-oxo-12H-indolo[2,1-a]isoquinolinium cation (**2**) were investigated. Effect of the organic modifier (EtOH) and NaCl on the spectral properties and aggregation of free ligands and on the DNA-binding affinity were assessed. Ligand **1** exhibited tendency for aggregation and showed higher binding selectivity for G-quadruplex DNA over double-stranded DNA. Both ligands were capable of interacting with DNA according to three binding modes: (i) electrostatic interactions, (ii) classical intercalation to dsDNA, and (iii) the specific binding to quadruplex DNA. The cytotoxic activity of ligand **2** was generally higher than that for ligand **1**, approaching a micromolar concentration level that may suggest its telomerase inhibition activity.

**Key words:** DNA binding, G-quadruplex DNA, papaverine, telomerase inhibitor

## Kinetics and Mechanism of *trans*-[Cr(cyca)(OH)F]<sup>+</sup> Complex Oxidation by [Fe(CN)<sub>6</sub>]<sup>3-</sup> Ion in Strongly Alkaline Aqueous Solutions

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Oxidation of the *trans*-[Cr(cyca)(OH)F]<sup>+</sup> complex, where cyca = *meso*-5,5,7,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane, by [Fe(CN)<sub>6</sub>]<sup>3-</sup> ion in strongly alkaline media has been studied. A long-lived intermediate chromium(V) species was detected basing on the EPR signal at  $g_{\text{iso}} = 1.986$ . The hyperfine structure of the main EPR signal is consistent with the d<sup>1</sup>-electron interactions with four equivalent or nearly equivalent nitrogen nuclei and [Cr<sup>V</sup>=O(cyca<sub>ox</sub>)]<sup>3+</sup> formula, where cyca<sub>ox</sub> = oxidized cyca, can be postulated for the intermediate Cr<sup>V</sup> complex. Kinetics of the rate limiting stage – electron transfer from the chromium(III) complex – has been studied in details applying an excess of the reductant and [OH<sup>-</sup>] at ionic strength equal 2.0 M. The rate law:

$$-\frac{d[\text{Fe}^{\text{III}}]}{dt} \approx \frac{d[\text{Cr}^{\text{IV}}]}{dt} = \frac{\frac{1}{2}k_{\text{cb}}K[\text{OH}^-]}{1 + \frac{1}{2}(K-1)[\text{OH}^-]}[\text{Cr}^{\text{III}}][\text{Fe}^{\text{III}}] \text{ is consistent with an outer-sphere}$$

electron transfer from the conjugate base (cb) of the reductant formed in a preequilibrium (*K*) step. Reactivity of the deprotonated chromium(III) complex is characterized by  $k_{\text{cb}}^{298} = 9.7$ ,  $\Delta H^\ddagger = (44.1 \pm 0.5) \text{ kJ}\cdot\text{mol}^{-1}$ ,  $\Delta S^\ddagger = (-78.0 \pm 2.0) \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$  and the preequilibrium step – by  $K^{298} = 0.237$ ,  $\Delta H^0 = (11.5 \pm 1.8) \text{ kJ}\cdot\text{mol}^{-1}$ ,  $\Delta S^0 = (26.6 \pm 5.8) \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$ .

**Key words:** chromium(IV), chromium(V), electron transfer from a tetraazamacrocyclic chromium(III), EPR, hexacyanoferrate(III), kinetics

## Investigation of the $Y_2X_3-Cu_2X-SnX_2$ ( $X = S, Se$ ) Systems

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The interactions between the components in the  $Y_2X_3-Cu_2X-SnX_2$  ( $X = S, Se$ ) systems at 870 K were determined using X-ray powder diffraction. The existence of the compound  $YCuS_2$  and the  $Y_{(2+x)/3}Cu_{2-x}S_2$  ( $0 \leq x \leq 0.52$ ) solid solution was confirmed in the  $Y_2S_3-Cu_2S$  section. The existence of the solid solution  $Y_{(2+x)/3}Cu_{2-x}Se_2$  ( $0 \leq x \leq 1$ ) was confirmed in the  $Y_2Se_3-Cu_2Se$  section. No compounds were found in the  $Y_2S_3-SnS_2$  and  $Y_2Se_3-SnSe_2$  sections. The formation of the compounds  $Cu_4SnS_4$ ,  $Cu_2SnS_3$ ,  $Cu_4Sn_7S_{16}$  in the  $Cu_2S-SnS_2$  system and the  $Cu_2SnSe_3$  compound in the  $Cu_2Se-SnSe_2$  system was confirmed. The formation of the  $Y_3Cu_{1-4x}Sn_{1+x}S_7$  ( $0 \leq x \leq 0.09$ ) solid solution and the  $Y_2Cu_{0.20}Sn_{0.95}S_5$  compound was determined in the  $Y_2S_3-Cu_2S-SnS_2$  system. The existence of the compound  $Y_3CuSnSe_7$  in the  $Y_2Se_3-Cu_2Se-SnSe_2$  system was confirmed.

**Key words:** X-ray analysis, isothermal section, crystal structure, phase diagram

## **Polymer–Surfactant Interaction and Physico-Chemical Properties of Polymethacrylic Acid–Triton X-100 and Polymethacrylic Acid–Poly(ethylene)oxide Aqueous Systems**

by **A.V. Tugay, V.P. Zakordonskiy and M.M. Soltys**

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The interaction of both Triton X-100 and poly(ethylene)oxide (PEO-2000) with polymethacrylic acid (PMAA) in aqueous solutions has been investigated by different methods: tensiometry, conductivity, viscometry, pH- and optical density measurement. The obtained results testify, that interaction of PMAA with the nonionic surfactants of different nature is accompanied by non-additive changes of physico-chemical properties and phase separation of binary systems. Thus, the character of influence of nonionic component depends on its nature. These changes take place in the clearly defined concentration ranges ( $1 \cdot 10^{-4}$ – $1 \cdot 10^{-3}$  M for Triton X-100 and  $1 \cdot 10^{-2}$ – $1 \cdot 10^{-1}$  M for more high-molecular PEO-2000) and they are caused by intramolecular PMAA–surfactant complex formation. The experimental results clearly reveal, that important role in complex formation is played by hydrogen bonds. For investigated systems both the critical aggregation concentration (CAC) and the saturation concentration (C2) were calculated, and the idealized schema of the mechanism of polymer–surfactant interaction was proposed.

**Key words:** polymethacrylic acid, Triton X-100, PEO, polymer–surfactant interaction, H–H binding complex, hydrophobic interaction

## **Magnetic Susceptibility Measurements for Polycrystalline $[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_4)_2$**

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The molar susceptibility of  $[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_4)_2$  (powder) was investigated between 2–300 K using superconducting quantum interference. The magnetic moment of the sample was also measured at various magnetic strengths, until the sample became saturated. At low temperatures the sample shows an antiferromagnetic alignment. The effective magnetic moment connected with  $\text{Ni}^{2+}$  corresponds well with the value predicted by theory. No anomalous behavior of the molar susceptibility of  $[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_4)_2$  was found at *ca* 225 K (as was previously stated by others authors).

**Key words:**  $[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_4)_2$ , molar susceptibility vs temperature, antiferromagnetic alignment, saturated magnetic moment

## **Displacement of CO from Ni(CO)<sub>4</sub> by Diphosphine Disulfide Derivatives *via* a Photochemical Reaction**

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A series of compounds with formulas [Ni(CO)<sub>2</sub>R<sub>4</sub>P<sub>2</sub>S<sub>2</sub>] and [Ni<sub>2</sub>(Co)<sub>6</sub>-μ-R<sub>4</sub>P<sub>2</sub>S<sub>2</sub>] (R = Me, Et, Pr<sup>n</sup>, Bu<sup>n</sup> and Ph) have been prepared photochemically by the reaction of Ni(CO)<sub>4</sub> with R<sub>2</sub>PSPSR<sub>2</sub> using a 400 W medium pressure mercury lamp through a quartz-walled immersion well reactor. They were characterized by elemental analyses, IR and <sup>31</sup>P NMR spectroscopy. The results reveal, that the diphosphine disulfide ligands show disubstitution and, thus, act as a bidentate species or monosubstitution and so act as a bridging ligand.

**Key words:** reaction mechanism, metal carbonyl

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**Synthesis and Physicochemical Study of *3d*  
Metal Coordination Polymers with Dinegative  
Tetradentate [NSNS] Schiff Base**

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