

**Studies on Mononuclear Chelates Derived from
Substituted Schiff Bases Ligands (Part 8):
Synthesis and Characterization of a New
5-Chlorosalicyliden-*p*-aminoacetophenoneoxime
and Its Complexes with Co(II), Ni(II), Cu(II) and Zn(II)**

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Co(L)₂, Ni(L)₂, Cu(L)₂ and Zn(L)₂ were synthesized from *p*-aminoacetophenoneoxime and 5-chlorosalicylaldehyde and Co(AcO)₂·4H₂O or Ni(AcO)₂·4H₂O or Cu(AcO)₂·H₂O or Zn(AcO)₂·2H₂O in 1:2 molar ratio, employing a template approach. Based on elemental analyses, molar conductivity and magnetic susceptibility data, IR, ¹H- and ¹³C-NMR and UV spectra, as well as thermal analyses (TG), a tetrahedral geometry for the complexes was determined. The data show that the ligand coordinates to the metal ion *via* -C=N- and -C-O- groups. All complexes are soluble in common organic solvents such as DMF and DMSO.

Key words: Schiff bases, tetrahedral complexes

Multinuclear NMR and Thermal Studies of New Silver(I) Silylated Carboxylates with Triphenylphosphine

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$\text{Me}_3\text{SiCH}_2\text{COOAg}$, $[\text{Ag}(\text{Me}_3\text{SiCH}_2\text{COO})(\text{PPh}_3)]$ and $[\text{Ag}(\text{Me}_3\text{SiCH}_2\text{CH}_2\text{COO})(\text{PPh}_3)]$ were prepared and characterized in solution by ^1H , ^{13}C , ^{29}Si , ^{31}P and ^{29}Si , ^{31}P CPMAS NMR. Spectral analysis suggests bidentate bridging carboxylates and monodentately bonded PPh_3 in solution and in the solid state. Two crystallographically inequivalent molecules of PPh_3 appeared in the solid state, what is evident from ^{31}P CPMAS NMR. Thermal studies of both complexes demonstrate two stage decomposition process (exothermic followed by endothermic) with formation of metallic silver in the range 483–548 K. TGA/IR and MS studies of $\text{Me}_3\text{SiCH}_2\text{COOAg}$ and $[\text{Ag}(\text{Me}_3\text{SiCH}_2\text{COO})(\text{PPh}_3)]$ revealed volatile products of decomposition (CO_2 , $\text{Me}_3\text{Si-}$) and recombination reactions such as $(\text{Me}_3\text{SiCH}_2\text{CO})_2\text{O}$, $(\text{Me}_3\text{Si})_2\text{O}$. The mechanism of complexes thermal decomposition is proposed.

Key words: Ag(I) complexes, triphenylphosphine, silylated carboxylates, thermal analysis, NMR

Nickel(II) Dithiocarbamates with AsI_3 and SbI_3 as Mixed Ligands

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Ni(II) dithiocarbamates with AsI_3 or SbI_3 as ligands of the composition $[\text{Ni}(\text{hmidtc})_2(\text{AsI}_3)]$ (1), $[\text{Ni}(\text{bzbutdtc})_2(\text{AsI}_3)]$ (2), $[\text{Ni}(\text{bzbutdtc})_2(\text{SbI}_3)] \cdot 2\text{CHCl}_3$ (3), $[\text{Ni}(\text{bz}_2\text{dtc})_2(\text{AsI}_3)]$ (4), $[\text{Ni}(\text{bz}_2\text{dtc})_2(\text{SbI}_3)] \cdot \text{CS}_2$ (5), $[\text{Ni}(\text{hmidtc})_2(\text{AsI}_3)_2] \cdot \text{CHCl}_3$ (6), $[\text{Ni}(\text{hmidtc})_2(\text{SbI}_3)_2]$ (7) and $[\text{Ni}(\text{cetdtc})_2(\text{AsI}_3)_2]$ (8), (hmidtc = hexamethyleneiminedithiocarbamate, bzbutdtc = benzylbutyldithiocarbamate, bz₂dtc = dibenzylidithiocarbamate, cetdtc = cyclohexylethyldithiocarbamate, dtc = S_2CN^-) have been synthesized. The complexes have been characterized by elemental analyses, thermal analysis, IR and UV/VIS spectroscopies, magnetochemical and conductivity measurements as square-pyramidal (1–5) and octahedral (6–8) complexes. X-ray analysis of $[\text{Ni}(\text{bz}_2\text{dtc})_2(\text{AsI}_3)]$ and $[\text{Ni}(\text{bz}_2\text{dtc})_2(\text{SbI}_3)] \cdot \text{CS}_2$ confirmed square-pyramidal arrangement around nickel in both complexes with the NiS_4As and NiS_4Sb chromophore, respectively.

Key words: Nickel(II), dithiocarbamate, AsI_3 , SbI_3 , synthesis, X-ray structures

Ability of 2,4D and 2,4DP Herbicides to Combine with Metal Ions of Biological Interest (Part 2): Zinc(II) Complexes

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Zinc(II) complexes with the commercial auxin herbicides 2,4-dichlorophenoxyacetic acid (2,4D; $C_8H_6O_3Cl_2$) and 2-(2,4-dichlorophenoxy)-propionic acid (2,4DP; $C_9H_8O_3Cl_2$) were prepared and characterized. The formulae of the solid complexes $Zn_2(C_8H_5O_3Cl_2)_3 \cdot 5H_2O$ (**Zn-2,4D**) and $Zn(C_9H_7O_3Cl_2)_2 \cdot 1.5H_2O$ (**Zn-2,4DP**) were established on the basis of the results of elemental analysis and Zn(II) determination. Water solubility of synthesized complexes at room temperature was examined. X-ray powder analysis was carried out. The discussion of IR spectra and conductivity data is presented. Thermal decomposition of these complexes in air was studied by TG, DTG, DTA and TG/MS methods with simultaneous analysis of the solid and gaseous products. The formation of complexes Zn-herbicide in the water solution by potentiometric pH titration was also investigated. The dissociation constants (pK) of 2,4D and 2,4DP were determined at 25°C and I = 0.1 (KNO₃).

Key words: 2,4-dichlorophenoxyacetic acid, 2-(2,4-dichlorophenoxy)-propionic acid, zinc(II) complexes, IR spectra, conductivity, solubility, TG, DTA and TG/MS data, potentiometric titration, dissociation constant

**Synthesis, Spectroscopic and Structural Characterization
of Cobalt(II) Complex with
Bis(3,5-dimethyl-1-pyrazolyl)methane and
Its Application in Catalytic Alkanes Oxidation with
tert-Butyl Hydroperoxide**

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A novel cobalt complex, [Co(bdpm)(OBz)](ClO₄) (bdpm = bis(3,5-dimethyl-1-pyrazolyl)methane, OBz = benzoate), was synthesized and characterized by elemental analysis, UV–Vis, IR spectroscopy and X-ray diffraction. The complex crystallizes in the trigonal system, space group P3₁21 with $a = 19.2583(9)$, $b = 19.2583(9)$, $c = 25.5269(17)$ Å and $\gamma = 120^\circ$. The complex can catalyze the selective oxidation of alkanes to corresponding alcohols and ketones, and a presumptive mechanism was proposed.

Key words: catalytic oxidation, crystal structure, biomimetic hydroxylation, cobalt complex

Synthesis and Characterization of Complexes of Isatin Schiff Base

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Complexes of Mn(II), Fe(III), Co(II), Ni(II), Cu(II) and Zn(II) with Schiff bases derived from isatin and ethylenediamine (L¹) or diethylenetriamine (L²) have been synthesized and characterized by microanalysis, IR, UV-Vis, ¹H NMR, EPR and conductance measurements. Electrical conductance of the Fe(III) chelates indicated them to be 1:1 electrolyte whilst those of divalent metal ions are non-electrolytes in DMSO. On the basis of magnetic moment and spectral data a pseudooctahedral geometry has been suggested for all of the complexes. The EPR spectrum of [Cu(L¹)Cl₂] complex indicates a considerable exchange interaction in the solid state.

Key words: isatin, spectroscopic measurements, Schiff base

**Synthesis and Spectral Studies on 2:1 Adducts Involving
Cadmium Dithiocarbamates and 4,4'-Bipyridine.
Single Crystal X-ray Structural Studies
on Bis(piperidinecarbodithioato-S,S')cadmium(II)
Benzene Solvate**

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Mixed ligand complexes of the general type $[\text{Cd}_2(\text{dtc})_4(4,4'\text{-bipy})]$ (dtc = diethyl-dithiocarbamate or piperidinedithiocarbamate anions; 4,4'-bipy = 4,4'-bipyridine) have been synthesized and characterized. IR spectra of the complexes show that the thioureide $\nu(\text{C-N})$ bands are observed at lower frequency than the values obtained for the parent dithiocarbamates. Charge transfer transitions are observed in the region 310–340 nm. Thermogravimetric studies show that the 4,4'-bipyridine adducts are more stable than the 2,2'-analogues. Also, the crystal structure of the benzene solvate of $[\text{Cd}(\text{pipdtc})_2]_2$ (pipdtc = piperidinedithiocarbamate anion) is reported. The coordination geometry can be described as a distorted trigonal bipyramid around each cadmium.

Key words: cadmium, dithiocarbamate, 4,4'-bipyridine adduct, crystal structure

Magnetic, Electrochemical and Antimicrobial Properties of Some Cu(II) Complexes with TPMC

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Three complexes: $[\text{Cu}_2(\text{C}_6\text{H}_5\text{COO})\text{tpmc}](\text{ClO}_4)_3 \cdot \text{CH}_3\text{OH}$, (**A**), (tpmc = *N,N',N'',N'''*-tetrakis(2-pyridylmethyl)-1,4,8,11-tetraazacyclotetradecane), $[\text{Cu}_2(\text{H}_2\text{pht})\text{tpmc}](\text{ClO}_4)_3 \cdot 3\text{H}_2\text{O}$ (H_2pht = phthalic acid) (**B**) and $[\text{Cu}_4(\text{ipht})(\text{tpmc})_2](\text{ClO}_4)_6 \cdot \text{NaClO}_4 \cdot 2\text{CH}_3\text{CN}$ (H_2ipht = isophthalic acid) (**C**) previously described were investigated by magnetic measurements, cyclic voltammetry (CV) and were tested towards some strains of microorganisms. It is found that all three complexes were electrochemically stable in the investigated ranges. The least stable was μ -ipht-complex (**C**) due to the reaction of central ion, which is seen from weak peaks in its cyclic voltammogram. Complexes were characterized by variable temperature magnetic measurements (4.2–300 K) and the observed data were successfully simulated by the equation based on the spin Hamiltonian operator, $\hat{H} = -2\hat{S}_1\hat{S}_2$, giving the ferromagnetic exchange parameters $J = 0.92 \text{ cm}^{-1}$ for **A**, $J = 0.55 \text{ cm}^{-1}$ for **B** and $J = 0.65 \text{ cm}^{-1}$ for **C**. For the complex **C** model of molecular magnet with two magnetically isolated dimeric subunits was used. These results indicate the presence of weak ferromagnetic spin exchange interaction between the Cu^{II} magnetic centers within each molecule.

Key words: Cu^{II} complexes, tpmc, redox properties, carboxylato bridged ligands

Keto-Enol Tautomerism in Pyruvic Acid – Theoretical (HF, MP2 and DFT in the *Vacuo*) Studies

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Extended quantum-chemical calculations {HF, MP2, DFT(B3LYP) in *vacuo*} were performed for neutral pyruvic acid and its enol forms. Among various tautomers-rotamers considered, three keto (*Tce*, *Tte* and *Cte*) and six enol structures (E1–E6) are found to be thermodynamically stable. The stability order for the keto and enol isomers: *Tce* > *Tte* > *Cte* > E1 > E2 > E3, E4, E5 > E6 is the same at each level of computations. The keto *Tce* structure has the lowest Gibbs free energy (G). The G value of the most stable enol E1 structure is larger than those of the three keto structures by a few kcal mol⁻¹.

Key words: pyruvic acid, keto-enol tautomerism, stability order, *ab initio* calculations

The Kinetic Studies of Complexation Reaction of Polyoxaalkylphosphates as P-Podands with Copper Ions in Ethanol

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The new P-podands (P3.1, P3.2, OP3.1 and OPH2.1) have been used as complexation agents in reactions with copper cations and the complexes obtained have been studied by NMR and kinetic methods. Two types of reactions, one between P-ligands and Cu(I) and the other between OP-podands and Cu(II), have been observed for the ligands studied.

Key words: P-podand, copper ions, kinetics, complexes

Synthesis and Antiproliferative Activity *in vitro* of New 2-Aminobenzimidazole Derivatives. Part 2 [1]

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A series of 2-methylpyrimido[1,2-a]benzimidazole derivatives has been synthesized in the reactions of 2-aminobenzimidazole (**1**) with selected halogeno β -diketones: 1,1,1-trifluoro- **2**, 1-chloro-1,1-difluoro- **3**, 3-chloro-2,4-pentadione- **4** and with 4-fluorobenzoylacetone **5**. 2-Aminobenzimidazole (**1**) in the reactions with α -chloro- and α -bromocinnamaldehyde gave Schiff bases **10** and **11** which have been subjected to reduction using NaBH₄ and 3-benzylideno-1,2-dihydro- (**12**) and 3-benzylideno-1,2,9,10-tetrahydroimidazo[1,2-a]benzimidazole (**13**) were obtained. The structures **2–13** were identified by the results of elemental analysis and their IR, ¹H NMR and MS spectra. Compounds **2–13** were examined for their antiproliferative activity *in vitro* against the cells of 3 human cancer cell lines, using SRB (sulphorhodamine B) or MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide) technique. Four out of all tested compounds revealed cytotoxic activity *in vitro*.

Key words: 2-aminobenzimidazole, halogeno-1,3-diketone, pyrimido[1,2-a]benzimidazole, imidazo[1,2-a]benzimidazole, antiproliferative activity *in vitro*

Kinetics and Mechanism of Hydrolysis Process of Podand Ligands

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The influence of ion complexation on the hydrolysis process of Si-podands was studied. On the basis of kinetic measurements a mechanism of the hydrolysis as well as a mechanism of the ion complexation effect were proposed.

Key words: supramolecular chemistry, kinetics, silicon podands, hydrolysis, ion complexes

Application of Dialkyltin Hydrides Containing the Chiral 2-(4-Isopropyl-2-oxazolinyl)-5-phenyl Ligand in Radical Cyclizations at the Steady State

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New *N*-coordinated dimethyl and di-*n*-butyl tin hydrides containing the oxazole moiety appeared to be useful in radical cyclizations of acyclic dihalides at the steady state. Both the hydrides were prepared in three steps starting from (–)-(S)-(2-bromophenyl)-4-isopropyl-4,5-dihydrooxazole. The advantages of the hydrides are at least twofold. First, a resolution of the radical intermediates allows generating higher stereomeric excess than in the case of commercially available hydrides. Second, tin by-products are easily removed.

Key words: diastereoselection, steady state, Sn-N coordination, tin hydrides

Analogues of Arginine Vasopressin (AVP) Modified in the N-terminal Part of the Molecule with Stereoisomers of 4-Aminopyroglutamic Acid

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The present work is a part of our studies aimed at clarifying the influence of sterical constraints in N-terminal part of AVP analogues on the pharmacological activity of the resulting peptides. In this study we described the synthesis of four new analogues of AVP substituted at positions 2 and 3 or 3 and 4 with two diastereoisomers of 4-aminopyroglutamic acid and four peptides in which we combined the above modification with the placement of 3-mercaptopropionic acid at position 1. All new peptides were not active in bioassays for pressor, antidiuretic and uterotonic activity.

Key words: analogues, arginine vasopressin (AVP), conformational constraints, V_{1a}-agonists, V₂-agonists

Synthesis of 2,4-Diamino-1,3,5-triazine Analogues of Methotrexate with Potential Antitumor Activity

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A series of analogues of methotrexate **23–31** in which aminopteridine ring was replaced by 2,4-diamino-1,3,5-triazine moiety were obtained. The syntheses of the analogues were accomplished by alkylation of *p*-aminobenzoic acids with appropriate 6-bromo-methyl-triazines **1–5** followed by conjugation with alkyl glutamate, aspartate or methioninate to give esters **14–22**. Saponification of the esters afforded target compounds **23–31** which were evaluated for their *in vitro* antitumor activity against MCF-7 (breast), DAN-G (pancreas) and LCLC-104 (lung) cancer cell lines.

Key words: classical antifolate analogues, 2,4-diamino-1,3,5-triazines, synthesis

**On n - π Electron Distribution in Simple Keto-Enol
Tautomeric Systems: $\text{H}_3\text{C}-\text{C}(\text{R})=\text{O} \rightleftharpoons \text{H}_2\text{C}=\text{C}(\text{R})-\text{OH}$
and $\text{RH}_2\text{C}-\text{CH}=\text{O} \rightleftharpoons \text{RHC}=\text{CH}-\text{OH}$**

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Variations of the n - π electron distribution during the tautomeric interconversion and substituent effects were analysed for simple keto-enol tautomeric systems of general formulae $\text{H}_3\text{C}-\text{C}(\text{R})=\text{O} \rightleftharpoons \text{H}_2\text{C}=\text{C}(\text{R})-\text{OH}$ and $\text{RH}_2\text{C}-\text{CH}=\text{O} \rightleftharpoons \text{RHC}=\text{CH}-\text{OH}$ using the geometry-based HOMA index.

Key words: tautomerism, n - π electron distribution, HOMA indices, substituent effects

Preparation and Some Properties of Cu–Te–S Thin Films on the Polyamide (PA) Surface

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The process and results of the formation of thin Cu–Te–S films on the polyamide (PA) surface are described. For clarifying the influence of various factors (the concentration of the initial solution of sodium telluropentathionate dihydrate, $\text{Na}_2\text{TeS}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$, pH, an exposure time and temperature) the kinetics of the sorption-diffusion of tellurium and sulfur into PA from freshly prepared solutions of sodium telluropentathionate was studied. Some structural properties and electrical resistance's values of obtained films have been investigated also. Cu–Te–S polymer composites indicate p-type properties of electrical conductivity and have electrical resistance in the range of $1.5 \cdot 10^3$ – $4.0 \cdot 10^3$ $\text{k}\Omega/\square$ to 3 – 5 $\text{k}\Omega/\square$ after to 2–3 h of an exposure and 1.0 – 1.5 $\text{k}\Omega/\square$ after 24–72 h of exposure in 0.01 – 0.1 mol dm^{-3} solutions of $\text{Na}_2\text{TeS}_4\text{O}_6$.

Key words: telluropentathionate, polyamide, chalcogenation, sorption-diffusion, layers of copper sulfide-copper telluride

A Study of the Properties of Tri-*n*-octylphosphine Oxide (TOPO) Monolayers at the Air/Water Interface

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Tri-*n*-octylphosphine oxide (TOPO) was found to be capable of Langmuir monolayer formation at the air/water interface. Surface pressure-area (π -A) isotherms obtained at different experimental conditions (barrier speed, number of deposited molecules, temperature and presence of metal ions in the aqueous subphase), compression modulus (C_s^{-1}) values and BAM images have been analyzed. The stability of TOPO monolayers has also been thoroughly examined. The obtained results indicate a liquid state of TOPO monolayers independently of subphase temperature or ionic strength. Such experimental conditions as the barrier speed or the number of deposited molecules were found to have no influence on the shape and position of the isotherms. Moreover, the performed experiments proved a high stability of monolayers from TOPO at the air/water interface.

Key words: Langmuir monolayers, air/water interface, tri-*n*-octylphosphine oxide, Brewster angle microscopy

Limited Penetration Depths of Hydrides in Cylindrical Pd-Ni Alloy Samples

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The penetration depths of hydrides formed in 6.8 kbar of gaseous hydrogen in cylindrical samples was investigated at 25°C in Pd-Ni alloys of 80, 120, 150, 200 and 250 μm diameters. The exposition time was about 4 months, thus, sufficient for the stationarity in respect to equilibration of the sample composition. In all alloys the penetration depth was limited, exhibiting a linear increase with radii of the wires applied with the slope of about 0.6 μm per 1 μm of the wire radius. These results are interpreted in terms of the coherent character of the hydride formation, supporting the thermodynamic reason for the limited penetration depth of metallic hydrides.

Key words: nickel hydride, high pressure, penetration depth, thermodynamics