

Synthesis and Magnetic Properties of Binuclear Lanthanide(III) Complexes Bridged by μ -2-Chloroterephthalato Groups

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Ten new binuclear lanthanide(III) complexes described by the overall formula $[\text{Ln}_2(\text{CTPHA})(\text{Mephen})_4(\text{ClO}_4)_2](\text{ClO}_4)_2$ ($\text{Ln} = \text{La, Ce, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er}$), where Mephen stands for 5-methyl-1,10-phenanthroline, and CTPHA represents the 2-chloroterephthalate dianions, have been synthesized and characterized by elemental analyses, molar conductance measurements, IR, ESR and electronic spectra. It is proposed that these complexes present CTPHA-bridged structures consisting of two lanthanide(III) ions. The $[\text{Gd}_2(\text{CTPHA})(\text{Mephen})_4(\text{ClO}_4)_2](\text{ClO}_4)_2$ complex has been further characterized by variable-temperature magnetic susceptibilities (4–300 K), demonstrating a very weak antiferromagnetic spin-exchange interaction between Gd(III)–Gd(III) ions within the complex. Based on the spin Hamiltonian operator, $\hat{H} = -2J\hat{S}_1\hat{S}_2$, the exchange parameter (J) was evaluated as -0.16 cm^{-1} .

μ -phth- μ -X Bridged Dicobalt(II) Complexes: Synthesis, Properties and Catalase-Like Activity

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μ -phth (phthalic acid dianion), having two isolated metal-binding groups, together with X ligand (X = CH_3CO_2^- , CF_3CO_2^- and Ph-CO_2^-) formed dicobalt(II) complexes $[\text{Co}_2(\text{II})(\mu\text{-phth})(\mu\text{-CH}_3\text{CO}_2^-)(\text{bipy})_2]\text{ClO}_4$ (**1**), $[\text{Co}_2(\text{II})(\mu\text{-phth})(\mu\text{-CF}_3\text{CO}_2^-)(\text{bipy})_2]\text{ClO}_4$ (**2**) and $[\text{Co}_2(\text{II})(\mu\text{-phth})(\mu\text{-Ph-CO}_2^-)(\text{bipy})_2]\text{ClO}_4$ (**3**). Their structure was characterized spectroscopically and magnetically, presenting a μ -phth- μ -X-bridged dinuclear structure. The pair of cobalt ions bridged by CH_3COO undergoes antiferromagnetic coupling. Catalytic properties for H_2O_2 dismutation are reported for complex **1–3**.

Thermochemical and Spectral Properties of Hydrates of Co(II), Ni(II) and Cu(II) Maleate. Crystal Structure of Co(II) Maleate Trihydrate

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The crystal and molecular structure of Co(II) maleate trihydrate, $\text{Co}(\text{C}_4\text{H}_2\text{O}_4) \cdot 3\text{H}_2\text{O}$, was determined by direct method and Fourier techniques. The structure was refined by full-matrix least-squares methods to a weighted R factor of 0.0263, based on independent reflections. The compound is a three-dimensional coordination polymer with distorted octahedron geometry at each cobalt(II) centre. Each cobalt atom is six-coordinated through two oxygen atoms from water molecules and four oxygen atoms from four different maleate groups. The thermal decompositions of $\text{Co}(\text{mal}) \cdot 3\text{H}_2\text{O}$, $\text{Ni}(\text{mal}) \cdot 3\text{H}_2\text{O}$ and $\text{Cu}(\text{mal}) \cdot \text{H}_2\text{O}$ (mal = maleate) have been investigated by means of TG, DTG, DSC and IR spectra. The release of volatile molecules after heating is either a three-step process for $\text{Co}(\text{mal}) \cdot 3\text{H}_2\text{O}$ and $\text{Ni}(\text{mal}) \cdot 3\text{H}_2\text{O}$ or a two-step process for $\text{Cu}(\text{mal}) \cdot \text{H}_2\text{O}$.

Syntheses, Structures and Physico-Chemical Properties of 2-Mercaptopyridine *N*-Oxide (Hmpo) Chelated Co- and Ni-Complexes with Phosphine Mediation

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2-Mercaptopyridine *N*-oxide (Hmpo) chelated complexes [Co(mpo)₂{P(OMe)₃}₂]PF₆ (**1**), [Co(mpo)₃]⁺Bu₄NPF₆⁻ (**2**), [Ni(mpo)(dppe)]Cl·0.5H₂O·MeOH (**3**) and [Ni(mpo)₂]⁺Bu₄NPF₆⁻ (**4**) were synthesized and their crystal structures characterized. The octahedral Co in **1** and **2** and the square planar Ni in **3** and **4** are chelated by mpo⁻, while phosphite or diphosphine coordinated in *cis*-orientation in **1** and **3**, respectively. The co-crystallized salt ⁺Bu₄NPF₆⁻ in **2** and **4** extrude any solvent molecules that might form hydrogen bonds within the crystal lattices.

Synthetic, Structural, Thermal and Electrical Properties of Some Transition Metal Polymeric Chelates of Bis(mercaptoacetamido)diaminobutane

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Polymeric chelates of bis(mercaptoacetamido)-1,4-diaminobutane (BMADB) with manganese(II), cobalt(II), nickel(II), copper(II), zinc(II) and cadmium(II) ions have been synthesized and characterized by elemental analysis, magnetic moment, infrared, electronic spectra and thermal analysis. In the present investigation the ligand BMADB exhibits bis-bidentate character and coordinates symmetrically with metal through nitrogen of amido and sulfur of mercapto groups. The thermogravimetric studies indicate a two stage decomposition and the presence of water molecules. Thermal data have been analyzed by Freeman-Carroll and Sharp-Wentworth methods and various kinetic and thermodynamic parameters have also been evaluated. Comparable values of parameters indicate common decomposition reaction mode in all chelates. Their d.c. electrical conductivity in pellet form over a wide range of temperatures have also been studied.

Synthesis, Structure and Tuberculostatic Activity of N'-(Amino-pyridyl-methylene)-hydrazinecarbodithioic Acid Methyl Esters

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The methods of preparation of the title esters from imidates or amidrazones are described, and structures of the compounds are elucidated on the basis of ¹H, ¹³C-NMR, 2D-NMR spectra and X-ray diffraction method. Tuberculotic activity was also studied.

New Tetraoxacyclophanes with α,α' -Di(para-hydroxyphenyl)-1,4-diisopropylbenzene Structural Unit

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α,α' -Di(para-hydroxyphenyl)-1,4-diisopropylbenzene (**1**) was used as a precursor for synthesis of six macrocyclic compounds (**5–10**). Low-temperature crystal structures were determined for α,α' -di(4-ethoxyphenyl)-1,4-diisopropylbenzene (**3**), C₃₀H₃₈O₂, monoclinic $P2_1/c$, $a = 12.831(4)$, $b = 8.708(2)$, $c = 11.221(3)$ Å, $Z = 2$, and 32,32,39,39-tetramethyl-13-oxo-1,6,20,25-tetraoxa[6.1.6.1.1]paracyclophane (**7**), C₄₉H₅₃O₅, triclinic $P\bar{1}$, $a = 10.506(2)$, $b = 14.770(2)$, $c = 14.917(2)$ Å, $\alpha = 111.91(2)$, $\beta = 108.75(2)$, $\gamma = 95.30(2)^\circ$, $Z = 2$. Compound **7** forms 2:1 clathrate-type compound with *para*-xylene. IR, Raman and UV spectra were recorded for all compounds.

Novel Heterocyclic Aminophosphonic Acids Derived from Furan and Thiophene

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Heterocyclic derivatives of aminomethylphosphonic acid were obtained in a one-pot procedure, by treatment of the corresponding heterocyclic aldimines with a mixture of trimethyl phosphite and bromotrimethylsilane (BrTMS). A reagent for phosphorylation of the imines in this case was the tris(trimethylsilyl)phosphite, formed *in situ* in a reaction mixture. The silylated esters formed were hydrolyzed to the final aminophosphonic acids.

Why γ - and δ - Are Less Active than β -Lactams? An *ab Initio* Study

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Ab initio calculations (HF/6-31G** and MP2/6-31G**) were performed to investigate the intramolecular hydrogen bonding in the model β -, γ -, and δ -lactam molecules. It was found that the intramolecular (C=O)O–H...O=C hydrogen bond stabilizes much more the γ - and δ -lactam fused ring systems than the β -lactam penicillin and cephalosporin-like systems. This observation suggests that γ - and δ -lactams block themselves by the intramolecular hydrogen bond and therefore are less active toward receptor active site than β -lactams. It is also likely that this factor can discriminate the β -lactamase inhibitors.

Synthesis of *N*-Benzoylphenylisoserinates of Lactarorufin B (a Sesquiterpene of *Lactarius* Origin) and Its Derivatives

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Lactarorufin B, which is a *Lactarius* sesquiterpene possessing primary, secondary and tertiary hydroxyl groups on its lactarane skeleton, was selectively transformed into (*2'R,3'S*)-*N*-benzoylphenylisoserinates, using protective group methodology described below.

Investigation of (β -Cyclodextrin)-Lappaconitine Inclusion Complex

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The structure of the inclusion complex of Lappaconitine (Lap) and β -cyclodextrin (β -CD) was studied by the UV, infrared, and NMR spectroscopy, as well as X-ray powder diffractometry. The stability constant of the complex in water is 275 M^{-1} , determined from the straight line portion of the phase-solubility diagram.

Synthesis of N-Alkyl Thiocarbamates, Derivatives of Monosaccharides

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Thiocarbamates, derivatives of monosaccharides are conveniently prepared by treatment of sugars with allyl or methyl isothiocyanates in the presence of DBU.

**Syntheses and Some Reactions
of 3-Amino-6-chloro-7-methyl-1,1-dioxo-1,4,2-
benzodithiazine**

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Syntheses and some reactions of new 3-amino-6-chloro-7-methyl-1,1-dioxo-1,4,2-benzodithiazine **2** with formaldehyde and alkyl halides are described. The chemical properties of obtained new 3-hydroxymethylamino-6-chloro-7-methyl-1,1-dioxo-1,4,2-benzodithiazine **3** were established, as well the conversion of **2** to the new 2-mercapto-benzenesulphonylcyanamide potassium salts **5–10** was discussed.

**Isoquinoline Syntheses via Δ^2 -Oxazolines. Part VIII.
Cyclization of 2-Acetamido-1,2-diphenylethan-1-ol
Derivatives into Isoquinoline Systems**

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The results of the conversion of 2-acetamido-1,2-diphenylethan-1-ol derivatives (**1**) into 1-methyl-4-phenylisoquinoline derivatives (**2**) have been described. The mechanism proposed for these reaction assumes the existence of protonated Δ^2 -oxazolines (**3**), carbonium ions (**4**), and unsaturated amides (**5** and **6**) as intermediates.

Transformations of 2-Alkoxy-6-chloro- and 6-Alkoxy-2-chloro-7-methylpurines Under the Action of Methyl Iodide

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2-Alkoxy-6-chloro-7-methylpurines **3** were prepared by 6-chloro-dealkoxylation of 2,6-dialkoxy-7-methylpurines **4**. Reactions of alkoxy-chloro-7-methylpurines **2** and **3** with methyl iodide led as N-9 methylation and as N-3 methylation to give *N*-methyl purinium salts **9** and **11** from purine **2** or **6** and **7** from purine **3**. The formation of purinium salts was accompanied by 6-iodo-dechlorination leading from **3** to 6-iodopurinium salt **5** and by alkyl iodide elimination leading to hypoxanthine **10**.

Carbon-13 Isotope Fractionation in the Oxidation of Acetic Acid of Natural Isotopic Composition with Homogeneous Solution of Chromic Anhydride in Orthophosphoric Acid

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^{13}C -isotope fractionation in the oxidation of acetic acid, (AA), of natural isotopic composition with homogeneous solution of chromic anhydride, CrO_3 , in 85% orthophosphoric acid has been investigated in the temperature interval 30–90°C and the measured ^{13}C fractionation factors, located in the range 1.021–1.016, have been compared with the earlier kinetic isotope effect (KIE) data for the oxidation of acetic acids labelled with carbon-14 in the carboxyl and in the methyl groups. A comparison of the ^{13}C KIE found to be of 1.026 for oxidation of $^{13}\text{CH}_3\text{COOH}$ at 51°C with the ^{13}C KIE of 1.029 ± 0.002 at 45°C and of 1.0266 at 21°C observed in the oxidation of formic acid of natural isotopic composition in the gas phase and in liquid phase oxidation respectively has been done also. These comparisons of the rate isotope effects, k_{12}/k_{13} , for acetic acid and for formic acid oxidation led the authors to conclusion that the cleavage of carbon to hydrogen bond in acetic acid is the rate determining step and the oxidation of AA proceeds with complete loss of the $^{13}\text{C}-^1\text{H}$ bond in the activated state. Two oxidation schemes, both involving the rupture of the $^{13}\text{C}-^1\text{H}$ bond in the TS have been proposed to rationalise the methyl- and carboxyl-carbon isotope effects in oxidation of AA with chromic acid-orthophosphoric acid solution. It has been shown also that the resistance of AA towards oxidation with Cr(VI) is caused by the higher by 5–6 kcal/mol activation energy relative to the oxidation of the methylene groups of the aliphatic carboxylic acids similarly as it has been observed in the oxidation of methyl group of acetate with alkaline permanganate.

**A Potentiometric Study of the Acid Dissociation and
Cationic Homoconjugation Equilibria in
Substituted Pyridine N-Oxide Systems in
Binary Polar Solvents, Acetonitrile – Acetone and
Acetonitrile – Dimethyl Sulfoxide**

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Acid dissociation constants of protonated substituted pyridine N-oxides and cationic homoconjugation constants of these acids with conjugate N-oxides have been determined by potentiometric titration in mixed polar non-aqueous solvents (acetonitrile – acetone and acetonitrile – dimethyl sulfoxide) with increasing (from 0 to 1) mole fraction of acetonitrile. In acetonitrile – dimethyl sulfoxide mixtures, the acid dissociation constants (expressed as pK_a) and cationic homoconjugation constants (expressed as $\log K_{BHB^+}$) vary non-linearly with increasing mole fraction of acetonitrile. In systems with the least basic N-oxides, local minima of pK_a have been observed. In acetonitrile-acetone mixtures, both the pK_a and the cationic homoconjugation constant values changed irregularly in the acid-base systems studied and the character of those variations could not be unambiguously determined.

X-ray Structural Investigation of 4-Phenyl-, 4-(*o*-Tolyl)- and 4-(2',4',6'-Trimethylphenyl)[2.2]paracyclophanes

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Crystal structures of 4-phenyl[2.2]paracyclophane (**1**), C₂₂H₂₀, monoclinic, *P*2₁/*c*, *a* = 14.7168(3), *b* = 7.8504(1), *c* = 15.1773(3) Å, β = 118.100(1), *Z* = 4; 4-(*o*-tolyl)[2.2]paracyclophane (**3**), C₂₃H₂₂, monoclinic, *Cc*, *a* = 11.354(1), *b* = 22.960(2), *c* = 7.7156(7) Å, β = 128.824(1), *Z* = 4; and 4-(2',4',6'-trimethylphenyl)[2.2]paracyclophane (**6**), C₂₅H₂₆, orthorhombic, *Pbca*, *a* = 12.892(4), *b* = 8.091(2), *c* = 35.895(9) Å, β = 90, *Z* = 8 have been determined. The interplanar angles between the aryl substituent and the cyclophane ring, to which they are bonded, are 38.0, 50.2 and 56.9° in **1**, **3** and **6**, respectively. For **3**, the methyl group of the substituent points away from the cyclophanyl cavity.

Crystal and Molecular Structures of Two Antiarrhythmic α -[(Diarylmethoxy)methyl]-1-piperidineethanols

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The crystal structures of two antiarrhythmic piperidineethanols, α -{[(2-methylphenyl)phenylmethoxy)methyl]-2,6-dimethyl-1-piperidineethanol (**1**) and α -[(bis(2,6-dimethylphenyl)methoxy)methyl]-2,6-dimethyl-1-piperidineethanol (**2**) have been determined by X-ray structure analysis of single crystals. The piperidine rings are close to ideal chair conformations, the methyl substituents are in equatorial positions. Overall shapes of the molecules differ significantly: in compound **1** the oxygen atoms are in *anti* position, while in **2** their mutual disposition is *gauche*. Dihedral angles between the phenyl rings and C–O–C plane are close to 90° for the mono- or di-substituted phenyl rings, while for the unsubstituted phenyl ring in **1** this value is smaller, equals 27.8(3)°. The bond angles in phenyl rings are influenced by the presence of methyl substituents. In both crystal structures the molecules make centrosymmetric dimers connected by strong O–H...N hydrogen bonds (piperidine nitrogen atoms act as acceptors).

Synthesis and Structure of Zinc Complex of 1-Phenyl-3-methyl-4-benzoyl-pyrazolone-5

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**Molecular Structure
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**Energy Transfer Evidence for Cross-Linking of DNA by
1,4-Bis((N-methylquinolinium-4-yl)vinyl) Benzene**

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Crystal Structure of Dichloro(1-phenylpyrazole-*N*²)copper(II)

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**Synthesis, Spectroscopy of New Push-Pull Ferrocene
Complexes Containing Different Conjugation Bridge
Between Ferrocenyl Donor and Pyridinium Acceptor**

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