

Lanthanide complexes of tetradentate macrocyclic ligand : Synthesis and spectroscopic investigation

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Abstract : In the present paper, the lanthanide complexes derived from (1,5,8,12-tetraaza-2,4,9,11-tetramethyl cyclotetraaza-1,4,8,11-tetraene) were synthesized. The general composition of the complexes is $[Ln(L)X_2 \cdot H_2O]X$, where Ln = La, Ce, Nd, Sm and Eu and X = NO_3^- and Cl^- . The ligand was characterized on the basis of elemental analyses, IR, Mass and 1H NMR spectral studies. All the complexes were characterized by elemental analyses, molar conductance measurements, magnetic susceptibility measurements, IR, Mass, electronic spectral techniques and thermal studies. The ligand acts as a tetradentate chelate and coordinates through four nitrogen atoms of azomethine groups. The lanthanum complexes are diamagnetic while the other Ln^{III} complexes are paramagnetic due to the presence of $4f^n$ unpaired electrons. The spectral parameters i.e. nephelauxetic effect (ϕ), covalency factor ($b^{1/2}$), Sinha parameter (d%) and covalency angular overlap parameter (h) have been calculated from absorption spectra of Nd^{III} and Sm^{III} complexes. These parameters suggest the metal-ligand covalent bonding. In the present study, the complexes show the coordination number seven.

Keywords : Tetradentate macrocycle, metal complexes, lanthanide(III).

A mechanistic and non-mechanistic approach on the solid state thermal decomposition of transition metal complexes of 3-(3-furan-2-yl-acryloyl)-4-hydroxy-6-methyl-pyran-2-one

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Abstract : The thermal decomposition of Mn^{II} , Fe^{III} , Cu^{II} , Ni^{II} and Cu^{II} chelates of 3-(3-furan-2-yl-acryloyl)-4-hydroxy-6-methyl-pyran-2-one was studied by TG-DTA and X-ray powder diffraction methods. The thermal stability of the complexes was studied using thermogravimetry and the decomposition schemes of the complexes were given. The mechanism of the decomposition has been established from TG data. The kinetic parameters i.e. n (order of reaction), E (energy of activation), Z (pre-exponential factor), DS (entropy change) and DG (free energy change) were calculated from the TG curve using mechanistic (Sestak-Berggren and Satava) and non-mechanistic (Coats-Redfern, MacCallum-Tanner and Horowitz-Metzger) integral equations.

Keywords : Dehydroacetic acid, chalcones, thermal study, TG-DTA, kinetic parameters, powder X-ray diffraction.

Synthesis and spectroscopic studies of cobalt(II), nickel(II) and copper(II) complexes with N-donor (N₄) macrocyclic ligand (DSL_F)

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Abstract : Complexes of Co^{II}, Ni^{II} and Cu^{II} containing a tetradentate macrocyclic nitrogen ligand have been prepared via template reaction of 2,3-pentanedione, *m*-phenylenediamine and transition metal ions. The complexes have been characterized on the basis of elemental analysis, molar conductance measurements, magnetic susceptibility measurements, infrared, EPR and UV-Visible electronic absorption spectral studies.

Keywords : Cobalt(II), nickel(II), copper(II), macrocyclic ligand, spectral studies.

Synthesis and spectroscopic characterization of bromodioxotriphenylphosphine oxide (*N,N*-dialkyldithiocarbamate/*O*-alkyldithiocarbonate)molybdenum(VI) complexes and the crystal structure of 2,5-dimethylpyridium bromide

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Abstract : A new series of bromodioxomolybdenum(VI) complexes, MoO₂Br(S₂CNR₂)(OPPh₃) (NR₂ = NEt₂, N(*i*-Pr)₂, NCH₂(CH₂)₃CH₂) and MoO₂Br(S₂COR)(OPPh₃) (R = Et, *n*-Pr, *i*-Pr) were prepared by the reaction of dibromodioxobis(triphenylphosphineoxide)molybdenum(VI) with sodium/potassium salts of the corresponding dithiocarbamates or dithiocarbonates in 1 : 1 molar ratio in dichloromethane. These newly synthesized complexes were characterized on the basis of elemental analysis and spectral (IR and ¹H NMR) studies. The crystal structure of 2,5-dimethylpyridium bromide is also described. [CMeCHCMeCHNH]Br crystallizes as monoclinic in the space group P2₁/c with cell parameters *a* = 8.6591(9), *b* = 13.252(1), *c* = 7.258(1) Å, β = 101.94(1)°, *V* = 814.9(2) Å³, *Z* = 4, *R* = 0.0393, *R_w* = 0.0885.

Keywords : Molybdenum, dithiocarbamate, dithiocarbonate, triphenylphosphineoxide, 2,5-dimethylpyridium bromide.

A planar 2,2'-dipyridylammonium salt : Synthesis and structure

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Abstract : Reaction of 2,2'-dipyridylamine (DPA) with hexafluorophosphoric acid (HPF₆) in methanol generates white rod-shaped crystals of HDPA.PF₆.H₂O. The salt is characterised by C, H and N analyses, FT-IR, ¹H NMR, mass spectroscopy and molar conductance measurement. The X-ray crystal structure of the salt has been determined. The structure shows that the salt is monomeric. The amine nitrogen is strongly hydrogen bonded with oxygen atom of water. Two pyridyl rings are almost planar which is a rarity in the coordination chemistry of DPA. Semi-empirical AM1 calculation was also performed to delve into the novel situation.

Keywords : 2,2'-Dipyridylamine, hexafluorophosphate salt, planar structure, hydrogen bonding, AM1 calculation.

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Reactivity ratios determination and NMR characterization of 2-hydroxy ethyl methacrylate and methyl methacrylate copolymers

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Abstract : NMR has proven to be one of the most informative technique for the investigation of copolymer microstructure as chemical shifts are sensitive to compositional and configurational sequences of copolymers. The HEMA/MMA copolymers were prepared by free radical bulk polymerization at 70 °C. The copolymer composition was determined from ¹H NMR spectrum which is then used to determine the reactivity ratios. The reactivity ratios obtained from KT, EVM and least square methods are $r_{\text{HEMA}} = 0.98$, $r_{\text{MMA}} = 0.82$; $r_{\text{HEMA}} = 0.88$, $r_{\text{MMA}} = 0.81$ and $r_{\text{HEMA}} = 0.92$, $r_{\text{MMA}} = 0.81$ respectively. ¹H and ¹³C{¹H} NMR spectra were analyzed with the help of DEPT, 2D HSQC and 2D TOCSY spectrum. The long range interactions of carbonyl carbon with methoxy protons and oxy methylene carbon with hydroxyl proton were determined with the help of heteronuclear multiple bond coherence (HMBC) spectrum. With the help of 2D spectra it was found that carbonyl, α -methyl and quaternary carbon of HEMA and MMA unit were sensitive to various configurational sequences as in poly(2-hydroxy ethyl methacrylate) and poly(methyl methacrylate). The carbonyl and oxymethylene carbons showed long range interactions with methoxy and hydroxyl protons respectively.

Keywords : Reactivity ratios, NMR, 2-hydroxy ethyl methacrylate, methyl methacrylate, microstructure, polymerization.

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Studies on the photochemical interaction of Fuchsin Basic dye with Triton X-100

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Abstract : A new solarchemical cell consisting of Fuchsin Basic, a cationic triphenyl methane dye, and triethanol amine as reductant in solution of surfactant separated from a saturated aqueous solution of iodine by a pyrex sintered glass membrane, has been developed. The surfactants used was Triton X100 (non-ionic). The results show that the photopotential and photocurrent of the cell is increased in Triton X100 and decreased with the cell without surfactant. The photopotential and photocurrent of the cell were observed as 690 mV, 175 mA respectively and power of the cell was 63.4 mA/min. The possible mechanisms of the cell in the various surfactants are discussed.
Keywords : Fuchsin Basic, Triton X-100, triethanol amine, electrical performance.

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Study of hydrogen bonding interactions of methaqualone with alcohols, naphthols (a and b) and nitrophenols

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Abstract : Methaqualone is a non-addictive drug widely abused by the miscreants. However, the principle of drug action is not known with certainty. H-bonding appears to be an important property for drug action. Methaqualone is found to form H-bonds with alcohols, naphthols, nitrophenol as apparent from the determination of the association constants of methaqualone with the ligands. Attempt has been made to suggest a probable mechanism of drug action based on water structure, H-bonding and hydrophobic interactions.
Keywords : Methaqualone, hydrogen bonding, naphthols, alcohols, drug mechanism.

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Effect of ethylenediaminetetra acetic acid, 2,2'-bipyridyl and manganese(II) on the reduction of chromium(VI) by D-glucose in presence of HClO₄

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Abstract : Kinetic data for the chromium(VI)-D-glucose redox system in presence of complexing agents are reported for the first time. The reaction is first-order each in [Cr^{VI}] and [D-glucose]. The kinetics reveal complex order dependence with [HClO₄]. The zero-order kinetics with respect to [HClO₄] at low concentrations shifts to higher order at higher concentration. Ethylenediaminetetra acetic acid (EDTA) and 2,2'-bipyridyl (bpy) catalyze the reaction whereas Mn^{II} has no effect. In the EDTA- and bpy-catalyzed paths, Cr^{VI}-EDTA and Cr^{VI}-bpy complexes have respectively been suggested as the active oxidant species. On the basis of various experimental data and the product characterization, the most plausible mechanisms have been proposed for the reactions.
Keywords : Manganese(II), 2,2'-bipyridyl, chromium(VI), D-glucose, EDTA, HClO₄.

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QSAR of substituted N-benzyl piperidines in the GBR series

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Abstract : Quantitative structure activity relationship (QSAR) studies on substituted piperidine in the GBR series have been made. Significant correlations were been obtained by multiparametric regression by using structural parameters with indicator parameters. The dopamine transporter (DAT) inhibitory activity of these compounds was found to increase with equalized electronegativity, hydrophobicity and bulk. The generated models were statistically validated using leave one out technique and cross validation methods.

Keywords : *N*-Benzyl piperidines in the GBR series, QSAR.

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Electrochemical reactions at sacrificial electrodes. Part-XXIII[†] : Direct electrochemical synthesis of bismuth(III) alkoxides

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Abstract : Bismuth(III) alkoxides have been synthesized by electrochemical reactions of methanol, ethanol, *n*-propanol, *n*-butanol, *n*-pentanol, *n*-hexanol, *n*-heptanol, *n*-octanol, *n*-nonanol, *n*-decanol at sacrificial bismuth anode and inert platinum cathode using tetrabutylammonium chloride as supporting electrolyte and acetonitrile as solvent. The solid products separated in the anode compartment have been isolated and characterized by elemental analysis and infrared spectral studies. Current efficiencies of these reactions are quite high.

Keywords : Bismuth(III) alkoxide, electrochemical reaction, sacrificial electrodes.

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Ultrasonic study of vanadium myristate in liquor ammonia

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Abstract : The ultrasonic velocity of vanadium myristate confirms that there is a significant interaction between the solute-solvent molecules in dilute solutions and the carboxylate molecules do not aggregate appreciably in dilute solutions. The value of Critical Micelle Concentration (CMC) for vanadium myristate is in agreement with those obtained from other physical parameters. The effects of concentration and chain length of soap on ultrasonic velocity and the various acoustic parameters (adiabatic compressibility, specific acoustic impedance, intermolecular free length, apparent molar compressibility, apparent volume, molar sound velocity, molar sound compressibility and solvation number) have been investigated.

Keywords : Vanadium myristate, ultrasonic velocity, liquor ammonia, acoustic parameters.

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Kinetics and mechanism of oxidation of aliphatic aldehydes by morpholinium chlorochromate

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Abstract : Oxidation of six aliphatic aldehydes by morpholinium chlorochromate (MCC) in dimethyl sulfoxide (DMSO) leads to the formation of corresponding carboxylic acids. The reaction is first order each in MCC and the aldehyde. The reaction is catalysed by hydrogen ions. The hydrogen-ion dependence has the form : $k_{\text{obs}} = a + b[\text{H}^+]$. The oxidation of deuteriated acetaldehyde, MeCDO, exhibited a substantial primary kinetic isotope effect $k_{\text{H}}/k_{\text{D}} = 5.95$ at 298 K). The oxidation of acetaldehyde has been studied in 19 different organic solvents. The solvent effect has been analysed using Taft's and Swain's multiparametric equations. The rate constants correlate well with Taft's σ^* values and reaction constants are negative. A mechanism involving transfer of hydride ion has been suggested.

Keywords : Kinetics, morpholinium chlorochromate, aldehydes, oxidation, mechanism.

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Spectral, thermal, XRD and biological studies on 4-(*p*-fluorophenyl)-2-amino-thiazole[†]

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Abstract : 4-(*p*-Fluorophenyl)-2-aminothiazole (FPAT) was synthesized and characterized by spectral (UV-Visible, IR, NMR and Mass), thermal and X-ray diffraction analyses. From the TG-DTA curve, various kinetic parameters were calculated using Coats-Redfern (C.R.), MacCallum-Tanner (M.T.) and Horowitz-Metzger (H.M.) methods. The compound possesses a triclinic crystal system. It exhibits antibacterial activity against *S. aureus* and *K. pneumoniae*, and antifungal activity against *A. niger* and *C. albicans*.

Keywords : Aminothiazoles, thermal analysis, X-ray diffraction studies, biological activity.