

## Molecular machines based on transition metal complexes : Rotaxanes and catenanes<sup>†</sup>

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**Abstract :** Human beings since ambiguity have been persistently designing mechanical devices which can perform certain functions useful to them. Recently along with this endeavour, emphasis has also been directed towards miniaturizing these devices leading to the possibility of constructing machines on nanoscale level, but for the past four decades it has been realized that miniaturization has reached its practical and fundamentals limit. Owing to the progress made to better understanding of the operation and mechanism of natural molecular machines of the biological world, it has however become possible to artificially design simple prototype of such device using molecular machines which provide atom-by-atom (bottom-up) approach towards the construction of microdevices.

These imitate biological systems which play essential roles in a wide variety of biological events particularly those related to the activities of cells and realize specific functions through their responses of mechanical motions. Such studies have led to an enormous interest not only in basic research but also in the growth of nanosciences, in particular, towards designing molecular machines using "bottom-up" rather than 'top-down' approach. These are based on the synthesis of various molecular rotaxanes, catenanes, rotors, and other systems. In synthesizing the latter, various systems have been proposed based on redox reactions (electro-chemical; chemical) involving in particular the formations of dissociation of transition metal complexes. It will not be out of place to refer that a very large volume of work has also been carried out using organic and inorganic molecules.

In this paper attempts have been made to describe a few examples of Cu<sup>I</sup>, Cu<sup>II</sup>, Ru<sup>II</sup>, Zn<sup>II</sup> and other metal complexes using electrochemical, chemical and light-driven molecular motions. However the review will not be a complete one in any sense because of the very large and fast work going on in this area.

**Keywords :** Endowment lecture, catenanes, molecular machines, rotaxanes.

## Synthesis and characterization of organotin(IV) complexes of 2-phenylphenol

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**Abstract :** The *n*-dibutyl and triphenyltin(IV) complexes of composition  $n\text{-Bu}_2\text{SnCl}_{2-x}(\text{OAr})_x$  and  $\text{Ph}_3\text{Sn}(\text{OAr})$

(where

$\text{OAr} = \text{OC}_6\text{H}_4\text{-Ph-2}$ ;  $x = 1$  and  $2$ ) have been synthesized in quantitative yields by the reaction of their respective chlorides,  $n\text{-Bu}_2\text{SnCl}_2$  and  $\text{Ph}_3\text{SnCl}$  with trimethylsilyl derivative of 2-phenylphenol in carbon tetrachloride. On the other hand, the good yields of the complexes of composition  $\text{Me}_2\text{SnCl}_{2-x}(\text{OAr})_x$  and  $n\text{-Bu}_3\text{Sn}(\text{OAr})$  have been obtained from the reaction of  $\text{Me}_2\text{SnCl}_2$  and  $n\text{-Bu}_3\text{SnCl}$  with 2-phenylphenol in the presence of  $\text{Et}_2\text{NH}$  in tetrahydrofuran. The characterization of the complexes has been accomplished by elemental analyses, molar conductance measurements, molecular weight determinations and IR, <sup>1</sup>H, <sup>13</sup>C, <sup>119</sup>Sn NMR and mass spectral methods. The thermal behaviour of the complexes has been studied by TG-DTA techniques. The reactions of parent complexes with nitrogenous bases viz. imidazole and benzimidazole have yielded 1 : 1 and 1 : 2 molecular adducts authenticated by physicochemical and IR spectral data.

**Keywords :** Organotin(IV) complexes, 2-phenylphenol, nitrogenous bases, imidazole, benzimidazole.

## Densities, viscosities, viscosity deviations and excess thermodynamic properties of ternary liquid mixtures of ethylenediamine/propylenediamine + 1,4-dioxane + (benzene or + toluene or + carbon tetrachloride) and their constituent binaries at 303.15 K

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**Abstract :** Densities and viscosities of ternary liquid mixtures of ethylenediamine and propylenediamine with 1,4-dioxane, as the second common component, and benzene/toluene/carbon tetrachloride as the third component and their constituent binaries have been measured at 303.15 K. The viscosity deviations ( $D_h$ ) and thermodynamic excess properties ( $V^E$  and  $DG^{#E}$ ) have been determined and fitted to Cibulka's equation. The experimental values of  $D_h$ ,  $V^E$  and  $DG^{#E}$  have been found to be in close agreement with the theoretical values predicted by Cibulka's equation. The nature and strength of molecular interactions between the mixing components of the ternary liquid mixtures are also discussed.

**Keywords :** Viscosities, viscosity deviations, thermodynamic excess properties, ternary mixtures.

## Aqueous hydrogen peroxide activated by ammonium heptamolybdate catalyst – A mild clean effective reagent system for the deprotection of oximes, semicarbazones and phenylhydrazones at room temperature

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**Abstract :** Oximes, phenylhydrazones and semicarbazones of a wide variety of aldehydes and ketones are cleanly cleaved to the corresponding carbonyl compounds in good to acceptable yields using environmentally safe and convenient oxidizer, 30% hydrogen peroxide in catalytic combination with 20 mol% ammonium heptamolybdate in aqueous acetic acid or tetrahydrofuran at room temperature. Absence of overoxidized by-products in case of oxidation-prone aromatic aldehydes, a,b-unsaturated aldehydes and compatibility with various common functional groups such as hydroxy, methoxy, epoxy, amino, methylenedioxy, conjugated and unconjugated C=C bonds are some of the key advantageous features of the method.

**Keywords :** Deprotection, oximes, semicarbazones, phenylhydrazones, 30% hydrogen peroxide, ammonium heptamolybdate.

## Reduction of some flavanones and E-3-benzylidene flavanones under modified Clemmensen reduction condition

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**Abstract** : A mild and efficient method for reduction of flavanones and *E*-3-benzylidene flavanones has been developed by using modified Clemmensen reduction condition [Zn(Hg), CH<sub>3</sub>COOH, EtOH, reflux, 1.5 h].

Flavanones gave flavans and *E*-3-benzylidene flavanones gave 3-benzylflavans and 3-benzylflav-3-enes.

**Keyword** : Flavanone, flavan, *E*-3-benzylidene flavanone, flav-3-ene, modified Clemmensen reduction, de-oxygenation.

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## **Role of physico-chemical parameters in Quantitative Structure-Activity Relationship based modeling of CYP26A1 inhibitory activity**

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**Abstract** : Quantitative Structure-Activity Relationship (QSAR) studies were performed on CYP26A1 inhibitory activity using physico-chemical parameters like Balaban centric index (BAC), mean Weiner index (WA), Balaban distance connectivity index (J), information theoretic index (Id), zero order (<sup>0</sup>c), first order (<sup>1</sup>c) and second order (<sup>2</sup>c) of molecular connectivity, polarizability (Pz) and partition coefficient (log P) along with appropriate dummy parameter. The QSAR models were tested for their statistical significance and reliability by using leave one out cross validation method. Some significant models have been reported.

**Keywords** : QSAR, CYP26A1, physico-chemical parameters.

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## **Use of a new 3-hydroxy-2-[1*ϕ*-phenyl-3*ϕ*-(*p*-chlorophenyl)-4*ϕ*-pyrazolyl]-4-oxo-4*H*-1-benzopyran (HPCPB) as an analytical reagent for trace determination of vanadium(V)**

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**Abstract** : Vanadium(V) in trace amounts forms an intense green 1 : 3 (M : L) complex with 3-hydroxy-2-[1*ϕ*-phenyl-3*ϕ*-(*p*-chlorophenyl)-4*ϕ*-pyrazolyl]-4-oxo-4*H*-1-benzopyran (HPCPB) in acetic acid medium (0.1 M) which is extractable into carbon tetrachloride and is stable for more than 3 h. Beer's law is obeyed in the vanadium concentration range of 0-1.4 mg mL<sup>-1</sup>. Molar absorptivity, detection limit and Sandell's sensitivity when applying spectrophotometric determination at the wavelength of 410 nm, are 2.2 × 10<sup>4</sup> L mol<sup>-1</sup> cm<sup>-1</sup>, 5.48 × 10<sup>-5</sup>

g L<sup>-1</sup> and 0.0023 mg V cm<sup>-2</sup>, respectively. The correlation coefficient being  $r = 1.0001$ . Ascorbic acid, dithionite, oxalate, EDTA and hydrogen peroxide interfere seriously in the determination. The method handles satisfactorily the analysis of various samples of varying complexity.

Keywords : Vanadium(V), HPCPB, determination, analytical reagent.

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## Oxovanadyl(IV) complexes of 4-benzylamidothiosemicarbazide and 1-a-furyl-4-benzylamidothiosemicarbazone

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Abstract : Oxovanadyl(IV) complexes of 4-benzylamidothiosemicarbazide (BTSC) and 1-a-furyl-4-benzylamidothiosemicarbazone (FBTS) have been prepared. The chemical analyses of the complexes correspond to the general formula VO(L)<sub>2</sub>Cl<sub>2</sub>, where L = BTSC and FBTS.

Keywords : Oxovanadyl(IV), thiosemicarbazone, complex.

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## Synthesis and characteristics of Ni<sup>II</sup> and Co<sup>II</sup> complexes of Schiff base ligands containing triphenylphosphine

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Abstract : The reactions of nickel(II) and cobalt(II) complexes, [MCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] (M = Ni or Co) with bidentate Schiff base ligands derived from acetylacetone or benzoylacetone with aniline, *o*-, *m*-nitroaniline have been carried out. The complexes were characterized by elemental analysis, spectral (IR, electronic) and cyclic voltammetric studies and are formulated as [MCl(PPh<sub>3</sub>)(L)] (M = Ni or Co; L = bidentate Schiff base ligand). A square planar structure has been tentatively proposed for all the new complexes.

Keywords : Metal complexes of Schiff base ligands, spectral studies, square planar, cyclic voltammetry.

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## Stability constant of ternary complexes of copper(II) involving Schiff bases as primary ligands and amino acids as secondary ligands

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**Abstract :** The formation constant of mixed ligand complexes of copper(II) with Schiff bases 1-(2-hydroxyl benzamido)-2-phenyl azomethine ( $L_1$ ) and 1-(2-hydroxyl benzamido)-2-(2-methyl phenyl) azomethine ( $L_2$ ) as primary ligands and glycine ( $R_1$ ), DL-naline ( $R_2$ ) as secondary ligands have been determined potentiometrically in 40% (v/v) THF-water mixture at 30 °C and ionic strength of 0.1 M NaClO<sub>4</sub>. The protonation constants of free ligands and stability constant for ternary systems involving Schiff bases and amino acids were also determined under identical conditions. The pH titration data were analysed using the computer SCOGS programme. The relative stability of ternary complexes as compared to that of corresponding binary complexes has been quantitatively explained in terms of  $D \log K$ ,  $K_R$ ,  $K_L$  and  $K_T$  values.

**Keywords :** Schiff base, copper(II), amino acids, stability constant, ternary complex.

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## Synthesis of *N*-galactosylated isodithiobiurets

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**Abstract :** Several 1-aryl-5-tetra-*O*-acetyl- $\beta$ -D-galactosyl-2-*S*-benzyl-2,4-isodithiobiurets have been prepared by the interaction of aryl-*S*-benzyl isothiocarbamides and tetra-*O*-acetyl- $\beta$ -D-galactosyl isothiocyanate. The structures of the newly synthesized compounds have been established on the basis of usual chemical transformations and IR, NMR and Mass spectral studies.

**Keywords :** *N*-Galactosides, aryl-*S*-benzyl isothiocarbamides, galactosyl isothiocyanate, isodithiobiurets.

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## Synthesis and study of new mesogenic homologous series : 4-(4-*n*-Alkoxy benzoyloxy)-3-chlorophenylazo-4-*n*-acetylbenzenes

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**Abstract :** A new homologous series of liquid crystals viz. 4-(4-*n*-alkoxy benzoyloxy)-3-chlorophenylazo-4-*n*-acetylbenzenes are studied with a view to understand mesomorphic characteristics of the molecule. The nematic mesophase appeared as threaded type of texture. The first five and sixteenth homologues are nonmesomorphic. Mesomorphic property commences from the sixth member of the series as nematic type without exhibition of any smectic property. Transition temperatures are observed through hot stage polarizing microscope. Analytical data supports the structure.

**Keywords :** Liquid crystal, mesogens, mesomorphic, nematic, smectic, polymesomorphism.

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## Solvent free synthesis of new-1-acetyl-3-(4-fluoronaphthyl)-5-substituted aryl pyrazolines as spermicides

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**Abstract :** In this paper we wish to report the solvent free synthesis of 1-acetyl-3-(4-fluoronaphthyl)-5-substituted aryl pyrazolines by using simple and efficient Green Chemistry techniques (Grindstone method and microwave irradiation). These methods have been developed for the rapid solvent free synthesis of title compounds from corresponding chalcones and hydrazine hydrate or phenylhydrazine in acetic acid. Considerable increase in the reaction rates have been observed with better yields compared with sample prepared from conventional method. Structure of synthesized new compounds have been confirmed on the basis of spectral (IR, <sup>1</sup>H NMR) and analytical data. Representative compounds have been screened for their spermicidal activity in HF cattles and Murrah buffalo.

**Keywords :** Aldol condensation, chalcone, pyrazoline, Grindstone chemistry, microwave irradiation.

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## Synthesis, characterization and biological screening of some new aryloxyacetic acid analogs

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**Abstract :** A novel series of 2-(4-bromo-2-formyl-phenoxy)acetyl amino acid and peptide analogs was synthesized by the coupling of 2-(4-bromo-2-formyl-phenoxy)acetic acid with different amino acid methyl ester hydrochlorides, dipeptide and tripeptide methyl esters using dicyclohexylcarbodiimide as the coupling agent and N-methylmorpholine as the base. Structures of all the newly synthesized compounds were elucidated on basis of IR, NMR and MS spectral data as well as elemental analysis. On pharmacological screening, some peptide derivatives were found to exhibit potent bioactivity against gram-negative bacterium *Pseudomonas aeruginosa*, pathogenic fungus *Candida albicans* and earthworms *Megascolex konkanensis*, *Pontoscotex corethruses* and *Eudrilus* sp. Dermatophytes were found to be moderately sensitive towards the newly synthesized analogs.

Hydrolyzed peptide derivatives displayed better antimicrobial activity in comparison to corresponding esters.

**Keywords :** 5-Bromosalicylaldehyde, aryloxyacetic acid, peptide, coupling, biological activity.

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## Photochemistry of 3-alkoxy chromones : Photocyclisation of 3-benzyloxy-6-chloro-2-(1- $\zeta$ -methyl-2- $\zeta$ -phenylvinyl)-4-oxo-4H-1-benzopyran

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**Abstract :** Photocyclisation of 3-benzyloxy-6-chloro-2-(1- $\zeta$ -methyl-2- $\zeta$ -phenylvinyl)-4-oxo-4H-1-benzopyran with pyrex filtered UV light to yield linear tricyclic pyranopyrone and angular fused tetracyclic compound is described. The product formation occurred via cyclisation of 1,4-biradical produced through H-abstraction by C=O group and conrotatory 6pe-electrocyclisation.

**Keywords :** Styrylchromones, pyranobenzopyrones, photocyclisation.

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# A distinction between 1,5- and 1,8-dimethoxy anthraquinones using IR of the demethylated product

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**Abstract** : When spectral data for a naturally occurring compound suggests two alternative structures involving 1,5- and 1,8-dimethoxy anthraquinones; IR of the demethylated compound helps in choosing the correct structure.

**Keywords** : Dimethoxy anthraquinones, IR, demethylation.

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# Voltammetric study and electrode kinetics of [Mn-sulfonamide-cephazolin] system at dropping mercury electrode

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**Abstract** : Stability constants ( $\log b$ ) of ternary complex of  $Mn^{II}$  with sulfadiazine, sulfoxazole, sulfamethaxazole, sulfamethazine, sulfathiazole, sulfacetamide and sulfanilamide as primary ligands and cephalosporin as secondary ligands were reported at  $pH = 7.30 \pm 0.01$  at  $m = 1.0 M NaClO_4$  at  $25^\circ C$  by polarographic technique. The kinetic parameters like transfer coefficient ( $\alpha$ ), degree of irreversibility ( $\lambda$ ), diffusion coefficient ( $D$ ) and rate constant ( $k$ ), were determined. The rate constants ( $k$ ) that varied from  $3.61 cm s^{-1}$  to  $9.93 cm s^{-1}$  confirmed the quasi-reversible nature of electrode processes. The study was carried out at  $25^\circ C$  and  $35^\circ C$ . Thermodynamic parameters such as entropy change ( $\Delta S$ ), enthalpy change ( $\Delta H$ ) and free energy change ( $\Delta G$ ) were determined. The values of stability constants ( $\log b$ ) that varied from 1.75 to 9.13 showed these drugs or their complexes might be effective against Mn toxicity.

**Keywords** : Electrode kinetics, [ $Mn^{II}$ -sulfonamides-cephazolin] system.

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# New spectrophotometric methods for the simultaneous determination of amlodipine and ramipril

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**Abstract** : Three new analytical methods were developed based on the simultaneous estimation of drugs in a binary mixture without prior separation of amlodipine and ramipril. Quantitative estimation of these drugs in marketed brands of the tablets was carried out using first order derivative, simultaneous equations and multi wavelength methods. Amlodipine and ramipril have absorbance maxima at 360 nm and 206.5 nm respectively in methanol. Both the drugs obey Beer's law in the concentration range employed for these methods. The results of analysis have been validated statistically and also by recovery studies.

**Keywords** : Ramipril, amlodipine.