

## Chemistry of molecular precursors for compound semiconductor nanoparticles<sup>†</sup>

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**Abstract:** This lecture intends to present recent results on design and development of molecular precursors for the synthesis of some binary and ternary compound semiconductor nanoparticles. A wide variety of complexes of Group II, III, IV and V elements with dithiocarboxylate, xanthate, dithiocarbamate, selenocarboxylate and *N,N*-dimethylaminoalkylchalcogenolates have been prepared and characterized by spectroscopic and single crystal X-ray diffraction techniques. Several of these complexes on thermolysis yield bulk as well as nanoparticles of metal chalcogenides (ME : M = Zn, Cd, Hg, Pb; E = S, Se, Te) and M<sub>2</sub>S<sub>3</sub> (M = In, Sb, Bi)). The latter have been characterized by XRD, EDAX, SEM, TEM, SAED and photoluminescence.

**Keywords :** Endowment lecture, molecular precursors, nano particles, metal chalcogenides, photoluminescence.

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## Target directed enediynes : Chemical and biological significance<sup>†</sup>

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**Abstract:** The novel chemical framework and potent antitumor activity of the enediyne natural products such as calicheamicin, dynemicin, esperamicin, and neocarzinostatin has fostered interest in the development of simple enediynes with low thermal cyclization temperature. It is well established that thermally labial enediynes exhibit anticancer activity, while there are few scattered examples in the literature about the biological importance of thermally stable enediynes. The present article deals with the synthesis, thermal reactivity of metalloenediynes, and antibacterial activity of thermally stable cyclic enediynes.

**Keywords :** Endowment lecture, enediynes, thermal reactivity, cyclization, antibacterial activity, copper complexes.

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## Template synthesis of novel 16-membered tetraazamacrocyclic transition metal complexes: DNA cleavage and antimicrobial studies

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**Abstract:** A new series of 16-membered tetraazamacrocyclic complexes of type  $[ML]X_2$  [where  $M=Ni^{II}$ ,  $Co^{II}$ ,  $Cu^{II}$ ,  $Zn^{II}$ ,  $Hg^{II}$ ,  $VO^{IV}$ ,  $Cd^{II}$  and  $Mn^{II}$ ;  $X_2= 2Cl^-$ ,  $SO_4^{2-}$ ] by incorporating an  $N_4$  donor site has been synthesized via the template condensation of *m*-phenylenediamine, formaldehyde, *p*-anisidine and metal salts in 1 : 2 : 1 : 0.5 molar ratio. The structural features of the complexes have been confirmed by microanalytical data, IR, UV-Vis,  $^1H$  NMR, ESR and CV techniques. The UV-Vis, magnetic susceptibility and ESR spectral data of the complexes suggest a square-planar geometry around the central metal ion except  $VO^{IV}$  complex which has square-pyramidal geometry. The electrochemical behaviour, the anodic and cathodic potential and the number of electrons transferred were calculated using cyclic voltammogram. The X-band EPR spectrum of copper complex in DMSO at 300 K and 77 K were recorded and its salient features are reported. The antimicrobial data show good results. The nuclease activity of the above metal complexes shows that only copper complex cleaves CT-DNA through redox chemistry in the presence of oxidant.

**Keywords :** Tetraazamacrocyclic complexes, CT-DNA, nuclease activity.

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## Design, synthesis and spectral characterization of chelates of $Pd^{II}$ , $Pt^{II}$ and $Rh^{III}$ with 6-guanidino-2,4-dimethyl-3,5-diazine and 6-phenyl guanidino-2,4-dimethyl-3,5-diazine-potential ligands with biological interest

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**Abstract:** Complexes of 6-guanidino-2,4-dimethyl-3,5-diazine and 6-phenyl guanidino-2,4-dimethyl-3,5-diazine with  $Pd^{II}$ ,  $Pt^{II}$  and  $Rh^{III}$  have been reported. Complexes have been characterised on the basis of analytical, magnetic and spectral characterization and powder diffraction studies. Crystal field parameters have also been calculated. The  $Pd^{II}$  and  $Pt^{II}$  complexes are square planar as expected and  $Rh^{III}$  complexes are pseudo-octahedral. IR data indicate that the imino nitrogen of the guanidine residue and one of the pyrimidyl nitrogen atoms acts as bonding sites in the formation of these complexes.

**Keywords :** Palladium, platinum, rhodium, guanidino pyrimidines.

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## Cyclic voltammetric studies of oxovanadium(IV) complexes derived from 2,2'-bipyridine and 1,10-phenanthroline in nonaqueous media

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**Abstract:** The electrochemical behaviour of oxovanadium(IV) complexes,  $[\text{VO}(\text{bipy})_2]^{2+}$  and  $[\text{VO}(\text{phen})_2]^{2+}$  (where, bipy = 2,2'-bipyridine and phen = 1,10-phenanthroline) have been examined in DMSO and DMF with 0.1 M TBAP using cyclic voltammetry at a Pt working electrode. It is found that  $[\text{VO}(\text{bipy})_2]^{2+}$  and  $[\text{VO}(\text{phen})_2]^{2+}$  complexes are irreversibly oxidized ( $\text{VO}^{2+/3+}$ ) with anodic peak potential,  $E_{\text{pa}1} \sim 1.10$  V vs SCE at 200 mV s<sup>-1</sup>. The initial reduction of these complexes in DMSO and DMF media revealed two irreversible cathodic peaks with  $E_{\text{pc}1} = -0.96$  to  $-1.13$  V and  $E_{\text{pc}2} = -1.17$  to  $-1.30$  V vs SCE. It should be mentioned that dependence of  $I_{\text{pa}1}$  or  $I_{\text{pc}1}$  on  $n^{1/2}$  is linear without any intercept. This suggests that the redox processes  $a_1$  and  $c_1$  are diffusion controlled. It is also observed that the oxidation of bipy complex is easier while reduction is difficult in comparison to phen complex in a given medium.

**Keywords:** Cyclic voltammetry, electrochemistry, imines,  $\text{VO}^{2+}$  complexes.

## Metal ion interaction with tridentate ONO donor azo ligands : Part I. Equilibrium study on the complex formation of $\text{Co}^{\text{II}}$ , $\text{Ni}^{\text{II}}$ , $\text{Cu}^{\text{II}}$ and $\text{Zn}^{\text{II}}$ with azo ligands

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**Abstract:** The ionization constants of three ONO donor azo ligands ( $\text{H}_2\text{L}^{1-3}$ ) containing different functional groups, viz. 2-hydroxy-2'-carboxy-5-methylazobenzene ( $\text{H}_2\text{L}^1$ ), 2,2'-dihydroxyazobenzene ( $\text{H}_2\text{L}^2$ ) and 2-hydroxy-2'-hydroxymethyl-5-methylazobenzene ( $\text{H}_2\text{L}^3$ ) and their formation constants with  $\text{M}^{2+}$  ions ( $\text{M} = \text{Co}, \text{Ni}, \text{Cu}$  and  $\text{Zn}$ ) have been determined in dioxane-water (50% v/v) medium at 25 °C at a fixed ionic strength ( $I = 0.1$  mol dm<sup>-3</sup> NaNO<sub>3</sub>) by pH-potentiometric method. The nature of the species present in solution have been elucidated on the basis of their electronic spectra. Job's method of continuous variation study indicated the formation of 1 : 1 complex with  $\text{Cu}^{2+}$  ion and 1 : 2 complex with  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions with all these three ligands. Formation constants of these metal ions with all these three ligands have been found to follow the order :  $\text{Co}^{2+} < \text{Ni}^{2+} < \text{Cu}^{2+} > \text{Zn}^{2+}$ . With respect to any particular metal ion, the formation constant values follow the order :  $(\text{L}^1)^{2-} < (\text{L}^2)^{2-} < (\text{L}^3)^{2-}$  which is explained on the basis of overall basicity of these ligands.

**Keywords:** Formation constants, cobalt(II), nickel(II), copper(II), zinc(II), azo ligands.

## Mixed ligand peroxovanadium complexes containing L-carnosine

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**Abstract:** Mixed ligand peroxy complexes containing L-carnosine, either in its neutral or deprotonated form have been synthesized and characterized. The complexes have been formulated as  $K[VO(O_2)L].H_2O$  (1),  $K[VO(O_2)_2(H_2L)].H_2O$  (2),  $[V_2O_2(O_2)_3(H_2L)_3].H_2O$  (3), where  $H_2L = L$ -carnosine. The ligand in its dideprotonated form in the complex (1), acts as a tridentate ligand, coordinating to vanadium through three donor atoms namely the oxygen atom of the carboxylate group, two nitrogen one each from the primary amine and the other from the amide group of the ligand. The ligand in acidic pH, behaves as a zwitterions, acts as bidentate through both oxygen of the carboxylate group in the complex (2) and as monodentate through oxygen of the carboxylate group in the complex (3). The peroxide ligand in all the complexes are chelated to vanadium and in the triperoxy complex, additionally it also bridge two vanadium atoms.

**Keywords :** Peroxovanadium, L-carnosine, synthesis.

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## Oxovanadium(IV) complexes of Schiff base derived from 3-aminopropanol and derivatives of salicylaldehyde

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**Abstract:** Oxovanadium(IV) complexes were synthesised by reacting vanadyl acetylacetonate with Schiff bases obtained by condensation of 3-aminopropanol with salicylaldehyde and its derivatives, 3-methoxysalicylaldehyde, 5-nitrosalicylaldehyde, 5-chlorosalicylaldehyde and 3,5-dichlorosalicylaldehyde. The complexes were characterised by IR and UV-Visible spectra. The effects of the substituents on salicylaldehyde and the changes in electronic spectra in different solvents with respect to donor properties of the solvents were studied. The reaction of the complexes with hydrogen peroxide was also observed.

**Keywords :** Schiff base, oxovanadium(IV), salicylaldehyde.

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## Physico-chemical studies on nanosized material from glucose-cupric sulphate reaction

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**Abstract:** Glucose reacts with cupric sulphate to form a new product as evident by growth morphologies, UV-Vis spectra, XRD and DSC studies. Reaction between glucose and cupric sulphate is also evident by conductivity measurements which showed a decrease in electrical conductivity on addition of glucose. There was also a decrease in glucose concentration on addition of cupric sulphate. Glucose crystallizes as fractal in agar-agar matrix, which on addition of cupric sulphate crystallizes rhythmically. Surface morphology of the product was studied by Atomic Force Microscopy (AFM), which revealed the formation of nanosized material. The average thickness of the particle aggregates was in the range of 50–115 nm.

**Keywords :** Glucose, cupric sulphate, nanosized material, growth morphology, biomaterial.

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## Apparent molar volumes of mono- and di-saccharides in water and in aqueous oxalic acid solutions at 293.15, 303.15, 313.15 and 323.15 K

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**Abstract:** Apparent molar volumes ( $V_r$ ) have been determined for mono- and di-saccharides, [D(+)-glucose, D(-)-fructose and sucrose] in water and in aqueous oxalic acid solutions at different concentrations in the range 0.2–1.0 mol kg<sup>-1</sup> by measuring the densities at (293.15, 303.15, 313.15 and 323.15) K. The limiting apparent molar volumes ( $V_r^0$ ) and experimental slope ( $S_v$ ) have been obtained in each case and their significance has been discussed briefly. The partial molar volumes ( $V_r^0$ ) have been used to calculate the partial molar volumes of transfer ( $ΔV_r^0$ ) of mono- and di-saccharides from water to aqueous oxalic acid solutions at different temperatures. The values of limiting apparent molar expansibilities ( $f_E^0$ ) and that of ( $∂^2V_r^0/∂T^2$ )<sub>p</sub> have been determined from temperature-dependence of  $V_r^0$ . It is concluded that all the saccharides behave as structure makers in water as well as in aqueous solutions of oxalic acid.

**Keywords :** Apparent molar volumes, saccharides, aqueous oxalic acid medium.

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## Synthesis of hollow microsphere containing of cavity

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**Abstract:** A novel procedure is reported for hollow silica microsphere containing cavity. Reaction with polystyrene, water, cetyltrimethylammonium chloride and tetraethylorthosilicate at room temperature give silica hollow microsphere containing cavity. Initially polystyrene synthesis was achieved by dispersion polymerization. Calcinations will eliminate the organics and leave microsphere as hollow. Scanning electron micrograph was used to identify the morphology.

**Keywords:** Polystyrene, silica microsphere, cavity, SEM.

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## Viscometric and thermodynamic studies of ion-ion and ion-solvent interactions in the solutions of uni-univalent and bi-univalent electrolytes in purely aqueous and aqueous thiourea media at 293.15, 303.15 and 313.15 K

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**Abstract:** Viscosities ( $\eta$ ) and apparent molar volumes ( $V_p$ ) of solutions of uni-univalent and bi-univalent electrolytes (NaCl, KCl,  $\text{NH}_4\text{Cl}$ ,  $\text{NaNO}_3$ ,  $\text{KNO}_3$ ,  $\text{NH}_4\text{NO}_3$  and  $\text{BaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{Ba}(\text{NO}_3)_2$  and  $\text{Mg}(\text{NO}_3)_2$ ) in purely aqueous and aqueous thiourea solutions have been determined at (293.15, 303.15 and 313.15) K. These data have been used to calculate the constants of Jones-Dole and Masson's equations. Activation thermodynamic quantities ( $\text{Dm}_1^{0\#}$ ,  $\text{Dm}_2^{0\#}$ ,  $\text{DS}_2^{0\#}$ ,  $\text{DH}_2^{0\#}$ ) of viscous flow have also been obtained. From the values of these parameters conclusions in regard to ion-ion and ion-solvent interactions have been obtained. It has also been found that all the electrolytes behave as structure makers in purely aqueous and aqueous thiourea solutions.

**Keywords:** Viscosity, electrolyte, aqueous thiourea.

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## Monomeric and dimeric stilbenoids from the orchid *Dendrobium amplum*

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**Abstract:** Amplumthrin, a new dimeric 9,10-dihydrophenanthrene derivative, was isolated from the orchid *Dendrobium amplum* which also afforded the known 9,10-dihydrophenanthrene dimer flavanthrin and the monomeric stilbenoids gigantol, batatasin III, its 3*o*-methyl ether and 3,3*o*-dimethyl ether, 2,7-dihydroxy-3,4,6-trimethoxyphenanthrene and its 9,10-dihydro derivative, 2,3,7-trihydroxy-4,6-dimethoxyphenanthrene and its 9,10-dihydro derivative and coelonin. The structure of amplumthrin was established as 2,2*o*,7,7*o*-tetrahydroxy-3,3',4,4*o*,6,6*o*-hexamethoxy-9,9',10,10*o*-tetrahydro-1,1*o*-biphenanthryl from various spectral and chemical evidence. The structure of amplumthrin was finally confirmed by regioselective biomimetic synthesis of the compound from its monomeric congener 2,7-dihydroxy-3,4,6-trimethoxy-9,10-dihydrophenanthrene by oxidative phenol-coupling reaction with CuCl(OH).TMEDA in very good yield (80%). Similar biomimetic synthesis of flavanthrin was also achieved in 82% yield from its monomeric congener coelonin using the same oxidant. The co-occurrence of amplumthrin and flavanthrin with their respective monomers in the same orchid *Dendrobium amplum* provides a strong circumstantial evidence in support of the proposed biogenesis of the naturally occurring biphenanthryl derivatives assumed to have arisen from their corresponding monomers through enzymatic oxidative phenol-coupling reaction.

**Keywords:** *Dendrobium amplum*, orchidaceae, amplumthrin, flavanthrin, dimeric 9,10-dihydrophenanthrene derivatives, biomimetic synthesis, oxidative phenol-coupling reaction with CuCl(OH).TMEDA.

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## Complexation equilibria of ciprofloxacin with proton and metal ions in aqueous-organic mixtures

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**Abstract:** The proton-ligand stability constants ( $\log K_1^H$ ,  $\log K_2^H$ ) of ciprofloxacin (CIP) and its metal-ligand stability constants ( $\log K_1$ ) in aqueous, acetonitrile-water, alcohol-water, DMSO-water and 1,4-dioxan-water (25 : 75 and 50 : 50% v/v) mixtures have been determined potentiometrically at  $25 \pm 0.1$  °C and 0.1 mol dm<sup>-3</sup> (NaCl) ionic strength. Ciprofloxacin formed 1 : 1 (M : L) complexes with VO<sup>II</sup>, Cu<sup>II</sup>, Zn<sup>II</sup>, Ni<sup>II</sup>, Mn<sup>II</sup> and Co<sup>II</sup>. In general, the values of  $\log K_1$  follow Irving-Williams stability order in each solvent system.

**Keywords:** Ciprofloxacin, metal ions, stability constant, ionization constant.

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## Zn<sup>II</sup> complexes of cyclic tetradentate thioethers

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**Abstract:** A new series of 14- and 15-membered tetrathia macrocyclic complexes, [MLX<sub>2</sub>] [M = Zn<sup>II</sup>; X = Cl or NO<sub>3</sub>; L = L<sup>1</sup> = 1,4,8,11-tetrathiacyclotetradecane; L<sup>2</sup> = 13,14-benzo-1,4,8,11-tetrathiacyclotetradecane; L<sup>3</sup> = 3,6,10,13-tetrathiacyclotetradecane-1-ol; L<sup>4</sup> = 4,5-benzo-3,6,10,13-tetrathiacyclotetradecane-1,8-diol; L<sup>5</sup> = 4,5,11,12-dibenzo-3,6,10,13-tetrathiacyclotetradecane-1,8-diol] have been prepared. The complexes have been characterized on the basis of elemental analyses, conductivity, IR and X-ray photoelectron spectra. Octahedral structures have been proposed for all the prepared metal complexes.

**Keywords:** Metal complexes, X-ray photoelectron spectra.

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## Trinuclear complexes of alkali metals with anhydrous nickel salicylate and copper salicylate

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**Abstract:** Ni(HSal)<sub>2</sub> and Cu(HSal)<sub>2</sub> complexes have been used as ligands in the synthesis of trinuclear alkali metal complexes having general formula [Ma(HL)<sub>2</sub>.2MbL], where Ma = Cu<sup>II</sup> or Ni<sup>II</sup>; HL = salicylic acid, Mb = Li<sup>+</sup>, Na<sup>+</sup> and K<sup>+</sup>; L = a-nitroso-b-naphthol (1N2N), 8-hydroxyquinoline (8HQ), NaSCN and KBr. These trinuclear complexes are considerably less soluble in ethanol than the complex ligand. Magnetic spectral and infrared spectral data suggest that they are oxygen bridged trinuclear complexes.

**Keywords:** Nickel salicylate, copper salicylate, alkali metal complexes.

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## Kinetic of base hydrolysis of some(aminomonocarboxylato)bis(ethylene-diamine)/(trimethylenediamine) Co<sup>III</sup> complexes in aqueous medium

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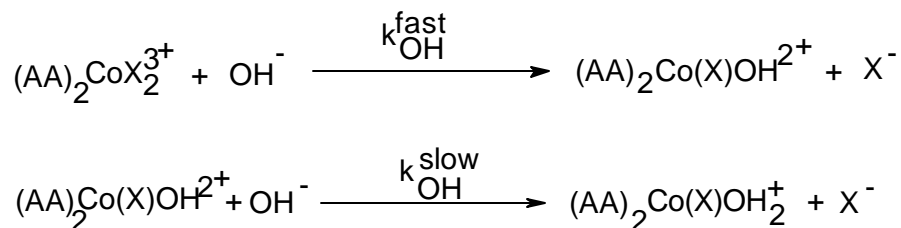
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**Abstract:** *cis*-[(en)<sub>2</sub>Co(O<sub>2</sub>CCH<sub>2</sub>NH<sub>3</sub>)<sub>2</sub>]<sup>3+</sup>, *cis*-[(en)<sub>2</sub>Co(B-O<sub>2</sub>C(CH<sub>2</sub>)<sub>2</sub>NH<sub>3</sub>)<sub>2</sub>]<sup>3+</sup> and *trans*-[(tmd)<sub>2</sub>Co(O<sub>2</sub>CCH<sub>2</sub>NH<sub>2</sub>)<sub>2</sub>]<sup>3+</sup> were synthesized. The base hydrolysis of these complexes proceed through a two step mechanism.

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A D<sub>cb</sub> mechanism has been proposed for the reaction.

**Keywords :** Kinetics, hydrolysis, amino acid complex, cobalt(III).

## Aromatic dianionic oxy-Cope rearrangement of 1,2-dinaphthyl-1,2-diols of acyclic diketones

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**Abstract :** We report the first instance where 1,2-dinaphthyl-1,2-diols of benzil and biacetyl undergo aromatic dianionic oxy-Cope rearrangement where the p bonds of the two naphthyl rings form the 1,5-hexadiene system necessary for oxy-Cope rearrangement.

**Keywords :** Benzil, biacetyl, 1,2-dinaphthyl-1,2-diol, aromatic dianionic oxy-Cope rearrangement.

## X-Ray diffraction studies of Schiff base 4-[2'-hydroxysalicylidene 5-(2'-thiazolyazo)]nitrobenzene

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**Abstract:** X-Ray diffraction studies have been carried out for 4-[2'-hydroxysalicylidene 5-(2'-thiazolyazo)]nitrobenzene. This ligand is synthesized and purified by condensing 5-(2'-thiazolyazo)salicylaldehyde and *p*-nitroaniline. The structure of compound is found to be tetragonal belonging to non-primitive system. The strain broadening effects are also examined and discussed.

**Keywords:** Azo Schiff base, X-ray diffraction, *p*-nitroaniline.

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## Synthesis and fungitoxicity of aldimines and 4-thiazolidinones derived from 4-bromoaniline

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**Abstract:** Condensation of 4-bromoaniline with benzaldehyde and substituted benzaldehydes (1-8) in equimolar ratio resulted in the formation of benzal-4-bromoaniline and its C-phenyl derivatives (1a-8a). Addition of thioglycolic acid to 1a-8a yielded the respective 4-thiazolidinone (1b-8b). The synthesized compounds were characterized on the basis of elemental analysis and spectral studies and were screened against five phytopathogenic fungi.

**Keywords:** Al dimines, fungitoxicity, thiazolidinone, bromoaniline.

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## Reaction of diethyl mesoxalate with active methylene compounds : an interesting outcome

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**Abstract:** Diethyl mesoxalate reacts separately with each of Meldrum's acid, indan-1,3-dione and diethyl malonate – all active methylene compounds – on neutral alumina either at room temperature or under microwave irradiation to furnish expeditiously the corresponding tertiary carbinols in very good yields. However, when the reaction of diethyl mesoxalate with a number of other active methylene compounds was tried similarly or by heating at 60–70 °C, the reactions either led to single products, which were too unstable to be isolated, or simply failed. Our findings, shedding new light on the reactivity of diethyl mesoxalate, are presented below.

**Keywords :** Diethyl mesoxalate, active methylene compounds, neutral alumina, *tert*-carbinols.

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## Microdetermination of palladium(II) using 5-chloro-8-hydroxy-7- iodoquinoline as a complexing agent

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**Abstract:** A simple, rapid, highly selective and sensitive method for the spectrophotometric determination of palladium(II) in trace amounts is worked out employing 5-chloro-8-hydroxy-7-iodoquinoline as a complexing agent for the metal ion and extracting the coloured complex into chloroform from 1 M H<sub>2</sub>SO<sub>4</sub> solution, whose absorbance is measured at 450 nm. Beer's law is obeyed in the range 0–2.6 mg Pd<sup>II</sup> mL<sup>-1</sup>. Molar absorptivity and Sandell's sensitivity of the complex are  $0.9 \times 10^4$  dm<sup>3</sup> mol<sup>-1</sup> cm<sup>-1</sup> and 0.0119 mg Pd cm<sup>-2</sup>, respectively. The method is free from the interference of a large number of elements including molybdenum, tungsten, tantalum, silver and other platinum metals. The proposed method handles satisfactorily the analysis of several samples of varying complexity.

**Keywords :** Palladium, 5-chloro-8-hydroxy-7-iodoquinoline, extractive spectrophotometry.

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