

## Regioselective synthesis of bioactive heterocycles by radical cyclization†

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**Abstract :** In recent years free radical cyclization has emerged as one of the versatile and simplest methodology for the construction of carbon-carbon bond in organic synthesis. In multi-step synthesis its use is significant as the key step of the synthesis involve the formation of carbon-carbon or carbon-hetero bond employing radical cyclization strategy. Regioselective synthesis of heterocycles viz. uracil, pyrone, coumarin, quinolone indole, benzofuran-annulated oxygen, nitrogen and sulfur heterocyclic compounds by tri-*n*-butyl tin hydride and thiol-mediated radical cyclization will be presented.

Keywords : Endowment lecture, regioselective synthesis, heterocycles, bioactive, radical cyclization.

## Synthesis, spectral characterization and electrochemical behavior of some cobalt(III) complexes of the type *cis-b*-[Co(trien)(RC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>)Cl]Cl<sub>2</sub>

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**Abstract :** Seven new cobalt(III) complexes of the type *cis-b*-[Co(trien)(RC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>)Cl]Cl<sub>2</sub>, where R = H, *p*-Me, *p*-OMe, *p*-Et, *p*-OEt, *p*-F and *m*-Me have been prepared and characterized by elemental analysis, UV-Vis, FT-IR and <sup>1</sup>H NMR spectral techniques. The results of the spectral studies reveal that these complexes possess a *cis-b* arrangement of ligands around the cobalt ion. Molar conductance measurements support the 1 : 2 natures of these complexes. TGA and DTA studies reveal that these complexes are thermally stable up to 175 °C. Cyclic voltammetric studies of these complexes and a fairly linear correlation (*r* = 0.93) of reduction potential data with the Hammett's substituent constants indicates that the electroreduction depends on the nature of the sixth ligand.

Keywords : Cobalt(III) complexes, substituted anilines, electrochemical and spectral studies.

## Binuclear complexes of Cu<sup>II</sup> and Ni<sup>II</sup> Schiff base of 2-hydroxy-1-naphthaldehyde and *o*-phenylenediamine with alkali metal salts of oxygen and nitrogen containing organic acids

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**Abstract :** A number of stable heterobinuclear alkali metal complexes of general formula  $M_aPhN.M_bL$ , where  $M_a = Cu^{II}$  or  $Ni^{II}$ ;  $M_b = Li, Na$  or  $K$ ;  $PhN = N,N'$ -1,2-phenylene-bis(2-hydroxy-1-naphthaldimine);  $L =$  deprotonated *o*-nitrophenol (ONP), 2,4-dinitrophenol (DNP), 2,4,6-trinitrophenol (TNP) or 1-nitroso-2-naphthol (1N2N) have been synthesized using  $Cu^{II}$  or  $Ni^{II}$  metal chelates of Schiff base and alkali metal salts of ONP, DNP, TNP and 1N2N. The IR spectra suggest bonding between the  $Ni^{II}$  or  $Cu^{II}$  metal chelate and alkali metal which appear by dative bond via oxygen atoms of C–O (phenolic). Low value of molar conductance would suggest them to be non-electrolyte. These complexes are biologically active against bacteria viz. *E. coli* and *S. aureus* and fungus viz. *C. albicans* and so these may be treated as good antibacterial agents and fungicides. It was therefore, though interesting to synthesize the title compounds and examine their antimicrobial activity.

Keywords : Heterobinuclear alkali metal complex, antimicrobial studies, MIC.

## Synthesis and structural evaluation of transition metal complexes of thiosemicarbazone and semicarbazone derived from pyrrole-2-carboxaldehyde

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**Abstract :** This paper presents the synthesis and characterization of cobalt(II), nickel(II) and copper(II) complexes of thiosemicarbazone ( $L^1$ ) and semicarbazone ( $L^2$ ) derived from pyrrole-2-carboxaldehyde. All the complexes reported here had been characterized by elemental analysis, molar conductance, magnetic moment, IR, electronic and EPR spectral studies. The molar conductance measurements of the complexes in DMSO correspond to non electrolytic nature. All the complexes are of high-spin type. On the basis of spectral studies various six coordinated geometry may be assigned for all the complexes except  $Co(L)_2(SO_4)$  and  $Cu(L)_2(SO_4)$  [where  $L = L^1$  and  $L^2$ ] which are of five coordinated square pyramidal geometry.

Keywords : Thiosemicarbazone, transition metal complex, pyrrole, semicarbazone.

## Synthetic, spectral, thermal and antimicrobial activity studies of some transition metal complexes derived from 2-hydroxy-methylbenzaldehyde *N*-(4-phenyl-1,3,4-thiazol-2-yl)semicarbazone

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**Abstract** : The Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup>, Zn<sup>II</sup>, Cd<sup>II</sup> and Hg<sup>II</sup> complexes of Schiff base 2-hydroxy-5-methylbenzaldehyde *N*-(4-phenyl-1,3,4-thiazol-2-yl)semicarbazone have been synthesized and characterized by elemental analyses, conductivity measurements, magnetic susceptibility, UV-Visible, IR, NMR, ESR, TGA and Mass spectral data. The Schiff base behaves as tridentate ONO donor ligand and forms the complexes of the type ML stoichiometry for Zn<sup>II</sup>, Cd<sup>II</sup> and Hg<sup>II</sup> complexes and ML<sub>2</sub> (metal-ligand) stoichiometry for Co<sup>II</sup>, Ni<sup>II</sup> and Cu<sup>II</sup>, complexes and are non-electrolytic in nature. It is found that Zn<sup>II</sup>, Cd<sup>II</sup> and Hg<sup>II</sup> complexes exhibited tetrahedral geometry whereas Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup>, complexes exhibited octahedral geometry. The ligand and its complexes were tested for their antibacterial activity against *Escherichia coli* and *Staphylococcus aureus* and antifungal activity against *Aspergillus niger* and *Candida albicans*.

**Keywords** : Antimicrobial activity, Schiff base, semicarbazone, ESR.

## Synthesis, reactivity and physico-chemical properties of novel chloro complex of nickel(II) containing benzoxazole ligand

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**Abstract** : New chloro complex of nickel(II) of the type [(m-Cl)<sub>2</sub>Ni<sub>2</sub>{h<sup>2</sup>-(pbox)}<sub>2</sub>] (1) has been synthesized by the interaction of nickel(II) chloride with {2-(*o*-hydroxyphenyl)-benzoxazole (pboxH) in equimolar ratio (in refluxing EtOH). Further, it was treated with sodium salts of Schiff bases (sb) [sb = smabH and saph], isopropoxide and aluminiumtetraisopropoxide and aryloxo- and alkoxo-salts in the presence of THF/pyridine to produce new hydrocarbon soluble mixed ligand complexes of the types : [(smab)Ni{h<sup>2</sup>-(pbox)}] (2), [(saph)Ni{h<sup>2</sup>-(pbox)}] (3), [(m-OPr<sup>i</sup>)<sub>2</sub>Ni<sub>2</sub>{h<sup>2</sup>-(pbox)}<sub>2</sub>] (4), [(m-OAr)<sub>2</sub>Ni<sub>2</sub>{h<sup>2</sup>-(pbox)}<sub>2</sub>] (5) and [(m-OPr<sup>i</sup>)<sub>2</sub>Al(OPr<sup>i</sup>)<sub>2</sub>]{Ni{h<sup>2</sup>-(pbox)}} (6). These new products have been characterized by spectroscopic [IR, electronic (UV-visible), FAB-mass] and magnetic studies. A tentative T<sub>d</sub> geometry around nickel(II) are proposed.

**Keywords** : Nickel(II), benzoxazole, synthesis, spectroscopic studies.

## **Removal of copper(II) from water using SrWO<sub>4</sub> — A photocatalytic process**

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**Abstract** : Photocatalytic removal of copper(II) over semiconductor SrWO<sub>4</sub> was carried out. The progress of reaction was observed spectrophotometrically. The effect of variation of different parameters like pH, concentration of copper(II), amount of photocatalyst, light intensity etc. on the rate of reaction was observed. A tentative mechanism for this reaction has been proposed.

Keywords : Photocatalyst, reduction, copper(II), semiconductor.

## **Charge-transfer complexes of ofloxacin, chlorpheniramine, azacyclonol and indapamide drugs with 2,3-dichloro-5,6-dicyanobenzoquinone**

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**Abstract** : Charge-transfer (CT) complexes of ofloxacin, chlorpheniramine, azacyclonol and indapamide drugs with 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) were investigated spectrophotometrically in different organic solvents at different temperatures. The spectral characteristics and formation constants of the formed 1 : 1 CT-complexes were examined and discussed in terms of the solvent properties. The oscillator and transition dipole strengths of the complexes have been determined from the CT absorption spectra. The  $DH^0$ ,  $DS^0$  and  $DG^0$  values are all negative, indicating stability and exothermic character of the complexes. The ionization potential value of the donors calculated using spectral data are in good agreement with those computed using molecular orbital package MOPAC (PM3) method. Infrared analysis of the CT-complexes have also been carried out.

Keywords : Charge-transfer, solvent effect, MOPAC method, drugs, electronic spectra, molecular complexes.

## Syntheses of 1,5-benzothiazepines : Part-XXXIV. Syntheses and antimicrobial studies of 8-substituted-2,5-dihydro-2-(4-methoxyphenyl/3,4-dimethoxyphenyl)-4-(2-thienyl)-1,5-benzothiazepines

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**Abstract :** Reactions of the 5-substituted-2-aminobenzenethiols 4a-f with the a,b-unsaturated heterocyclic ketones 3a,b were carried out in dry ethanol saturated with dry HCl gas. The products 5a-l were characterized by microestimations for C, H, N, S and IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>19</sup>F NMR and mass spectral studies. The synthesized compounds were screened for antimicrobial activity against the bacteria, *Staphylococcus aureus* and *Pseudomonas aeruginosa* and the fungus, *Candida albicans*. All the compounds showed moderate activity against the bacteria *Staphylococcus aureus* and *Pseudomonas aeruginosa* with reference to standard drugs, gatifloxin and natilmicin but showed good activity against the fungus, *Candida albicans* with reference drug fluconazole. The maximum antifungal activity has been shown by the compound 5k having maximum number of methoxyl groups and 5g and 5a having fluoro in addition to methoxyl groups.

Keywords : Cardiovascular, 1,5-benzothiazepines, diltiazem, methoxyphenyl.

## Mild and efficient synthesis of β-amino alcohols by antimony trichloride catalysed opening of epoxides

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**Abstract :** Nucleophilic opening of epoxides with aniline derivatives in the presence of catalytic amount of antimony trichloride in acetonitrile at room temperature afford the corresponding β-amino alcohols in excellent to very good yield.

Keywords : Epoxide, antimony trichloride, nucleophilic opening, aromatic amine, β-amino alcohols

## Comparative voltammetric behaviour of isatin and some of its Schiff bases at a solid electrode

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**Abstract :** The electrochemical behaviour of isatin (1) and its Schiff base, 3-arylimino-2H-indol-2-ones (2a-g), has been investigated and compared using cyclic (CV) and differential pulse voltammetry (DPV) techniques at glassy carbon electrode (GCE) in different solvent systems. CV of isatin (1) solution in DMF in 0.1 M LiCl showed two irreversible reduction step due to successive transfer of two electrons leading to formation of dioxindole whereas Schiff bases in this system gave a single two-electron transfer irreversible reduction peak at all scan rates studied. This is due to high basicity of nitrogen atom of imine bond, proton abstraction and second electron transfer are both rapid. The product was secondary amine in case of Schiff bases. However, in acidic BR buffer, isatin (1) is preprotonated and showed a single irreversible one-electron reduction wave due to formation of its dimer, isatide. Controlled potential electrolysis was carried out to find the number of electrons transferred and products were isolated and identified by spectroscopy. Kinetic parameters i.e. charge transfer coefficient ( $\alpha_{na}$ ), forward rate constant ( $K_{r,h}$ ), diffusion constant ( $D_0^{1/2}$ ) have also been calculated.

**Keywords :** Isatin, Schiff base, electro reduction, oxindole, secondary amine.

## Adsorbing micellar interface for enhanced removal of lead from aqueous solution

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**Abstract :** An enhanced removal of lead with polymerized saw dust (PSD) in the presence of micellar solutions of sodium dodecylsulfate (SDS), cetyl trimethylammonium bromide (CTAB) and Criton X-100 has been investigated. The PSD with micellar solution was found to be an effective sorbent for removal of lead from wastewaters in laboratory-scale experiments. Batch studies of PSD with and without micellar systems indicate that Pb removal is greater with micellar systems. The percentage removal of Pb on PSD with micelles increased on decreasing lead concentration and increasing sorbent dose, micellar concentration and electrolyte addition. The single and binary micellar systems also showed marked variations in percentage removal. In single systems of SDS, CTAB and Criton X-100 percentage removal enhancements of lead were 68.5, 65.0 and 3.2% respectively. The binary mixtures of Criton X-100 with SDS and with CTAB enhanced removal upto 74.0% and 69.3% respectively. The combination of SDS and CTAB had no significance effect. The adsorption equilibrium followed Freundlich and Langmuir isotherms. The enhancement in removal was possibly due to interaction between Pb and micellar media and then adsorption on PSD. So, PSD is a good sorbent for both Pb and micelles.

**Keywords :** Polymerized saw dust (PSD), sodium dodecylsulfate (SDS), cetyl trimethylammonium bromide (CTAB).

## Long term leaching studies on Coal Combustion Residues from Balco Captive Power Plant, Korba

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**Abstract** : The study was conducted to assess the leaching of coal combustion residues (CCRs) from Balco Captive Power Plant, Korba, India through five different standard techniques. The leaching procedures included acid digestion, short term (24-hour shake test, TCLP) and long term leaching (Open column percolation experiments, ASTM column experiments) studies for a period of more than three years. The physico-chemical characteristics (pH, conductivity and TDS) were observed within the permissible limits. The leachates were analysed for twenty-three elements like Na, K, Mg, Ca, Fe, Ni, Cu, Zn, Pb, Mn, Cr, Co, Cd, Se, Al, Ag, As, B, Ba, V, Sb, Mo, W and compared with their compositional analysis. Among these elements, only Na, K, Mg and Ca were found to be leaching throughout the study period and were within permissible limits of the prescribed standards (IS : 2490). However, Fe, Pb, Ni, Cu and Mn were observed at significant concentration levels on a few occasions. Overall, this study establishes the CCRs of Balco Captive Power Plant as environmentally benign material and encourages its large-scale utilisation.

**Keywords** : Coal Combustion Residues, trace elements, leaching.

## Synthesis of CL-20 by clean nitrating agent dinitrogen pentoxide

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**Abstract** : CL-20 is a high energy material. It is usually prepared via nitration with concentrated nitric acid and sulphuric acid, but this technique pollutes the environment. In this article, CL-20 was synthesized with 2,6,8,12-tetraacetylhexaazatetracyclo[5,5,0,0<sup>3,11</sup>,0<sup>5,9</sup>]dodecane (TAIW) and clean nitrating agent dinitrogen pentoxide. By the use of new nitrating agent, the reaction eliminated the use of concentrated sulphuric acid and was environmental friendly with high atom economy. The structure of the compound was characterized by elemental analysis, IR, <sup>1</sup>H NMR and MS. Meanwhile, the effects of reaction temperature, time and other factors on yield were analyzed. Through the separation of intermediate, the nitration course and mechanism were also discussed.

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**Keywords** : CL-20, dinitrogen pentoxide, synthesis, explosive .

## Synthesis and characterisation of palladium(II) and platinum(II) complexes with triphenylphosphine sulphide and triphenylstibine

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**Abstract :** The complexes of palladium(II) and platinum(II) with triphenylphosphine sulphide ( $\text{Ph}_3\text{PS}$ ) and triphenylstibine ( $\text{Ph}_3\text{Sb}$ ) of composition  $[\text{ML}_2\text{X}_2]$  ( $\text{M} = \text{Pd}^{\text{II}}, \text{Pt}^{\text{II}}$ ;  $\text{L} = \text{Ph}_3\text{PS}$  or  $\text{Ph}_3\text{Sb}$  and  $\text{X} = \text{Cl}, \text{Br}, \text{I}$  or  $\text{SCN}$ ) have been prepared and characterized. The triphenylstibine sulphide complexes like former ligand could not be isolated as the ligand get desulphurized in aqueous medium and the complexes of desulphurized ligand triphenylstibine have only been isolated in pure form. The complexes have been characterized by UV, IR,  $^1\text{H}$  NMR spectroscopic methods and resembles with square planar geometry.

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**Keywords :** Triphenylphosphine sulphide, triphenylstibine, palladium(II), platinum(II).

## Computation studies on Schiff base ligands and equilibrium studies on their metal complexes

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**Abstract :** Schiff bases of furfural with L-threonine (thr) and L-glutamine (gln) were studied by quantum mechanical method using GAMESS program. Geometry of the Schiff bases was optimized by HF and MP2 methods. The Schiff bases are non-planar. Based on the Mulliken's charge density calculations, furan oxygen, imino nitrogen and carboxylato oxygen atoms were found to be the potential binding sites. Equilibrium studies of  $\text{Co}^{\text{II}}, \text{Ni}^{\text{II}}, \text{Cu}^{\text{II}}$  and  $\text{Zn}^{\text{II}}$ -furfural (A)- L-threonine (thr), L-tryptophane (trp) and L-glutamine (gln) (B) systems were done by batch wise titration method at 25 °C and ionic strength of 0.1 m ( $\text{KNO}_3$ ). All the systems have MAB,  $\text{MAB}_2$  or  $\text{MAB}_2$  types of Schiff base complexes. The results indicate that the Schiff base (AB) binds to the metal ions in terdentate manner through furan oxygen, imino nitrogen and carboxylato oxygen atoms.

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**Keywords :** Computation, equilibrium, Schiff base, furfural, amino acid, potentiometry.

## **Insect antifeedant potent chalcones**

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**Abstract** : Some halogen substituted 2-propen-1-ones were synthesized by solvent free eco-friendly method. They are characterized by physical constants, analytical and spectral data. Their antifeedant activity against castor *semilooper* and *Achoea janata* L. has been studied. The chlorinated chalcones were found to be most effective.

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**Keywords** : Chalcones, eco-friendly synthesis, grind stone chemistry, insect antifeedant activity.

## **One step process for starch fermentation employing the starch digesting yeast *Saccharomyces diastaticus***

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**Abstract** : The production of industrial and fuel ethanol from starchy biomass commonly involves three steps. The present investigation was to assess the possibility of developing a direct one step process for starch fermentation employing the starch digesting yeast *Saccharomyces diastaticus*, a haploid strain. Direct fermentation of cassava waste by a pure culture of *S. diastaticus* produced the maximum of 17 g/L ethanol at 36 h fermentation.

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**Keywords** : One step process, fermentation, *Saccharomyces diastaticus*, cassava waste.

## Sensitive determination of chromium by the catalytic hydrogen wave in the presence of thiocompounds at DME

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**Abstract** : A sensitive polarographic method for the determination of chromium is proposed, based on the catalytic hydrogen wave of Cr<sup>VI</sup>-xanthate complexes in the presence of NH<sub>4</sub>Cl at mercury electrode. The Cr<sup>VI</sup>-xanthate complexes produce a catalytic hydrogen wave at -1.52 V vs SCE with potassium propyl xanthate (Kpxan) and at -1.47 V vs SCE with potassium cyclohexyl xanthate (Kchxan) in NH<sub>4</sub>Cl-NH<sub>4</sub>OH medium (pH 9.6 for Kpxan and 9.2 for Kchxan). The peak height is proportional to metal ion concentration. The proposed method is free of interference from other metal ions except Mo<sup>VI</sup> and is evaluated to micro level Cr<sup>VI</sup> down to 0.1 ppm by the determination of chromium content in drinking water samples, industrial effluents, tannery waste water and agricultural materials.

**Keywords**: Polarographic catalytic hydrogen wave, chromium(VI), xanthate, environmental samples.

## Kinetics and mechanism of the oxidation of DL-methionine by quinolinium bromochromate

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**Abstract** : The oxidation of methionine (Met) by quinolinium bromochromate (QBC) in dimethylsulphoxide (DMSO) leads to the formation of corresponding sulphoxide. The reaction is of first order with respect to each QBC and Met. The reaction is catalysed by hydrogen ions. The hydrogen-ion dependence has the form :  $k_{\text{obs}} = a + b [\text{H}^+]$ . The oxidation of methionine was studied in nineteen different organic solvents. The solvent effect was analyzed by Kamlet's and Swain's multiparametric equations. Solvent effect indicated the importance of the cation-solvating power of the solvent. A suitable mechanism has also been postulated.

**Keywords** : Mechanism, quinolinium bromochromate, DL-methionine, chromium(VI).

## Studies of natrolite and its cation-exchanged and adsorbed derivatives with $\text{Co}^{\text{II}}$ and liquor $\text{NH}_3$ and $\text{H}_2\text{S}^\dagger$

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**Abstract** : These studies are based on the investigation carried out on the cation exchange and adsorption behaviour of a natrolite which is natural zeolite collected from Sinner, Nasik (Maharashtra) as a geological specimen.  $\text{Co}^{\text{II}}$ -natrolite was prepared by treating a saturated aqueous  $\text{Co}^{\text{II}}$ -nitrate solution with natrolite by continuous shaking at 60 °C for maximum interaction. A portion of this exchanged derivative was then heated over a maker burner for several days in a platinum crucible. Both, the original and the preheated  $\text{Co}^{\text{II}}$ -natrolite samples were also interacted with gaseous  $\text{H}_2\text{S}$  and liqueor  $\text{NH}_3$  to study their sorption capacity and preheating affects. All the derivatives and the original natrolite before and after heating were analyzed by XRD and FTIR.

**Keywords** : Natrolite, XRD, cation exchange, adsorption behaviour.