

Anion radical of naphthyl-azo-imidazole in ruthenium and osmium carbonyls

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Abstract : The reaction of $[M(H)(Cl)(CO)(PPh_3)_3]$ ($M = Ru, Os$) with alkyl-2-(naphthyl-a/b-azo)imidazole (a-NaiR 1; b-NaiR, 2) in boiling dry heptane has afforded $[M(Cl)(CO)(PPh_3)_2(a/b-NaiR-)]$ (3/4 and 5/6) (a/b-NaiR-, azo anion radical) as the major product. The radicals are oxidized upon treatment with NH_4PF_6 in dichloromethane-acetonitrile medium to one electron oxidized non-radical salts ($3^+/4^+$ and $5^+/6^+$). The complexes display redox couple in the range -0.3 to -0.6 V vs SCE. The magnetic properties are studied by bulk (m BM) and EPR measurements. The anion radicals are fluorescent and free ligands do not fluoresce (1, 2). DFT calculation shows that anion radical non-coordinated ligand is very much unstable while metal-carbonyls stabilize them.
Keywords : Naphthylazoimidazole, azo anion radicals, electrochemistry, EPR and DFT calculation.

Spectroscopic studies and characterization of Ni^{II} and Cu^{II} complexes with a nitrogen donor new azamacrocyclic ligand with pendent arms

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Abstract : The newly synthesised azamacrocyclic ligand L [2,6,12,16,21,22-hexaaza-3,4,5,13,14,15-hexamethyltricyclo-[15,3,1,1⁷⁻¹¹]-docosa-1(2,10,2,5,7,9,11(22),12,15,17,19-decene] was prepared by the reaction of 3-methyl-2,4-pentadione and 2,6-diaminopyridine. The complexes of Cu^{II} and Ni^{II} were synthesised with the new macrocyclic ligand. All the complexes were characterized by the molar conductance measurements, magnetic susceptibility measurements, mass, IR, electronic and EPR spectral studies. The molar conductance measurement of the complexes in DMF solution is corresponding to non-electrolytic nature. Thus these complexes may be formulated as $[M(L)X_2]$ (where, $M = Ni^{II}$ and Cu^{II} and $X = Cl^-$ and NO_3^-). On the basis of spectral studies, an octahedral geometry for Ni^{II} complexes and tetragonal for Cu^{II} complexes is assigned. The biological activity of the ligand and complexes were screened *in vitro* against different pathogenic fungi and several bacteria to study their comparative capacity to inhibit the growth.
Keywords : Spectroscopic, EPR, 2,6-diaminopyridine, 3-methyl-2,4-pentadione, Ni^{II} and Cu^{II} complexes.

Electro-organic synthesis of 1-(3-chloro-2-hydroxy propyl)-2-methyl-5-aminoimidazole in acidic medium

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Abstract : Electro-organic synthesis of 1-(3-chloro-2-hydroxy propyl)-2-methyl-5-aminoimidazole has been carried out by the constant current electrolysis at copper and amalgamated forms of copper and lead electrodes under acidic conditions. The electro reduction of 1-(3-chloro-2-hydroxy propyl)-2-methyl-5-nitroimidazole (Ornidazole) has been also studied by cyclic voltammetry at glassy carbon electrode under acidic conditions which indicate the reducible behaviour of it. Isolated product was characterized by TLC, usual laboratory qualitative tests and IR, NMR spectral analysis. Effect of different parameters like current density, temperature, depolarizer concentration and nature of cathode material on yield percentage and current efficiency have been investigated. Number of electrons have also been calculated to confirm the isolated product, which is 1-(3-chloro-2-hydroxy propyl)-2-methyl-5-nitroimidazole.

Keywords : Constant current electrolysis, Ornidazole, cyclic voltammetry.

Solvent hydrogen bonding and structural effects on nucleophilic aromatic substitution reactions. Part-2 : Reaction of benzenesulphonyl chloride with anilines in propan-2-ol/2-methylpropan-2-ol mixtures[†]

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Abstract : Substitution reactions of fourteen *para*- and *meta*-substituted anilines with benzenesulphonyl chloride in different mole fractions of propan-2-ol in 2-methylpropan-2-ol have been investigated conductometrically. The second order rate constants correlates satisfactorily with pK_a values of the anilines and also with the Hammett's substituent constant. The *para*-substituted anilines shows a satisfactory correlation with Charton's LDR equation. The results of these correlations indicate the formation of an electron deficient transition state. The rate data correlate satisfactorily with macroscopic solvent parameters such as relative permittivity, ϵ_r and polarity, E_T^N .

Multiple correlation analysis of the rate data via Kamlet-Taft's solvatochromic parameters reveals that the solvent dipolarity/polarizability plays a dominant role in governing the reactivity.

Keywords : Aniline, benzenesulphonyl chloride, substitution reaction, solvent effect.

Solvent hydrogen bonding and structural influences on the Cr^{VI} oxidation of anilines in aqueous acetic acid medium

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Abstract : The oxidation of *meta*- and *para*-substituted anilines by Cr^{VI} oxidant, imidazolium fluorochromate (IFC), in aqueous acetic acid mixtures of varying compositions in the presence of *p*-toluenesulfonic acid (PTS) is first order in IFC and PTS. Michaelis-Menten type kinetics is observed with all of the anilines. The IFC oxidation of 15 *meta*- and *para*-substituted anilines at 299–322 K complies with the isokinetic relationship but not to any of the linear free energy relationships. The isokinetic temperature lies within the experimental range. The rate data failed to correlate with macroscopic solvent parameters such as relative permittivity, ϵ_r , and ionizing power, Y , correlation of rate data with Kamlet-Taft solvatochromic parameters (hydrogen bond donor acidity, α , hydrogen bond acceptor basicity, β , and dipolarity/polarizability, ρ^*) is linear which suggests that the specific solute-solvent interactions play a dominating role in governing the reactivity.

Keywords : Aniline, kinetics, oxidation, solvent effect.

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Electronic structure and nonlinearity of Schiff bases of furfural and α -amino acids

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Abstract : A series of Schiff bases of furfural (fur) with α -amino acids viz. glycine (gly), alanine (ala), valine (val), phenylalanine (phy), threonine (thr), glutamine (gln), glutamic acid (glu), aspartic acid (asp) and histidine (his) are optimised using RHF/6-31G*. The polarizability and hyperpolarizability are calculated by the finite-field method using AM1 Hamiltonian. It was found that the conformation selectivity of the compounds is due to the side chain in the amino acid. A *trans* conformation along the imino group is stable. The molecules look like a helix with the cavity at the center. The effects of substitution are mainly of steric in nature. The torsion twists of the back bones are characteristic of the substitution. Except fur-phy and fur-his, all the Schiff bases have maximum dipolemoment along the x -axis. The fur-phy and fur-his are found to have dipolemoment maximum along y -axis. The linear, first and second order hyperpolarizabilities follow the dipolemoment order.

Schiff bases with acid side chain have very high polarizability and comparable with metal clusters.
Keywords : Nonlinear optical properties, Schiff base, amino acid.

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Oxidation of 2-hydroxynaphthaldehyde by alkaline *N*-bromosuccinimide – A kinetic and mechanistic study

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Abstract : The kinetics of the oxidation of 2-hydroxynaphthaldehyde (2-HNA) by *N*-bromosuccinimide (NBS) in aqueous alkaline medium at a constant ionic strength of 0.60 mol dm⁻³ was studied titrimetrically. The reaction is of first order in [NBS] and of fractional order in both [2-HNA] and

[alkali]. Addition of products has no significant effect on the reaction rate. However, increasing ionic strength and decreasing dielectric constant of the medium increases the rate. The oxidation process in alkaline medium has been shown to proceed via the formation of a complex between the active species of the oxidant and the substrate followed by the decomposition of the complex in a slow rate determining step to yield the product. Some reaction constants involved in the mechanism were determined. The calculated and observed rate constants agree excellently. The activation parameters were computed with respect to the slow step of the mechanism.

Keywords : 2-Hydroxynaphthaldehyde, *N*-bromosuccinimide, kinetics, oxidation.

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CuSO₄·5H₂O catalyzed efficient one pot synthesis of α-amino nitriles

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Abstract : α-Amino nitriles are synthesized in a one pot three component coupling of aldehydes, amines and trimethylsilyl cyanide using catalytic amount of CuSO₄·5H₂O at ambient temperature.

Keywords : α-Amino nitriles, CuSO₄·5H₂O, aldehydes, amines, trimethylsilyl cyanide.

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Multi-component reactions of formyl-4-aryloxymethylcoumarins under microwave irradiation

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Abstract : Ethylacetoacetate and ammonia/urea were reacted with formyl-4-aryloxymethylcoumarins 2a-e by microwave irradiation to give 1,4-dihydropyridyl-4-aryloxymethylcoumarins 3a-e/3,4-dihydropyrimidin-2-one-4-aryloxymethylcoumarins (Biginelli compounds) 4a-e respectively. These compounds have been screened for their potential as microbial growth inhibitors against six bacterial and six fungal strains. The growth inhibition was observed against the Gram positive species and all the fungal strains even at a concentration of 6.25 mg ml⁻¹ in some cases.

Keywords : Coumarin, 1,4-dihydropyridine, 3,4-dihydropyrimidin-2-one, Biginelli reaction, Hantzsch condensation, 4-bromomethylcoumarin.

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Sodium borohydride reduction of *E*-3-cinnamylidene flavanone in alcohols : Formation of structurally interesting alkoxydienes

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Abstract : Sodium borohydride reduction of *E*-3-cinnamylidene flavanone, separately in methanol and ethanol, gave alcohol dependent reduction products. A plausible mechanism for formation of the products has been suggested. Thermal [4+2]-cycloaddition of one of such products with *N*-phenylmaleimide was studied and in that reaction only one cycloaddition product could be isolated.
Keywords : *E*-3-Cinnamylidene flavanone, sodium borohydride, cycloaddition, *N*-phenylmaleimide.

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Extraction and spectrophotometric determination of molybdenum(V) as its thiocyanate complex in industrial, environmental and soil samples

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Abstract : A simple and rapid extractive spectrophotometric method for micro determination of molybdenum using thiocyanate as a complexing agent has been worked out for industrial, environmental and soil samples. The Mo^V-thiocyanate complex is extracted into 2,2'-dipyridyl in chloroform. The vermilion colour of the extract is measured at λ_{\max} 505 nm against a similarly prepared reagent blank. The 1 : 4 (Mo : SCN) complex is found to be stable for more than one hour in the extract and obeys Beer's law over the concentration range of 0–11 mg Mo ml⁻¹. The molar absorptivity and Sandell's sensitivity of the proposed method are found to be 1.24×10^4 dm³ mol⁻¹ cm⁻¹ and 0.0075 mg Mo cm⁻², respectively. A large number of cations, anions and complexing agents of major analytical importance such as Re^{VII}, Cr^{III,VI}, Cu^{II}, Zr^{IV}, Mn^{II}, Ni^{II}, Fe^{II,III}, W^{VI}, U^{VI}, V^V and platinum metals, sulphate, chloride, fluoride, bicarbonate, phosphate, tartrate and EDTA do not interfere. Only oxalate, acetate, hydrogen peroxide, tin, cobalt, bismuth, titanium, osmium and ruthenium interfered in the procedure. The method has been applied satisfactorily for the determination of molybdenum in varieties of samples.
Keywords : Molybdenum(V), extraction, thiocyanate complex, sample.

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Synthesis, spectroscopic and antibacterial studies on bis(cyclopentadienyl)-hafnium(IV) derivatives with dithiocarbamates derived from α -amino acids

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Abstract : The reactions of bis(cyclopentadienyl)hafnium(IV) dichloride with novel dithiocarbamate ligands [Na₂(S₂C-NH-(R)-COO)], derived from α -amino acids (glycine, leucine, b-alanine and dl-alanine), have been studied in THF/DMF mixture and binuclear complexes of type [(C₅H₅)₂HfCl]₂(S₂C-NH-(R)-COO) have been isolated. Tentative structures of the complexes have been proposed on the basis of analyses, electrical conductance, magnetic moment and spectral (UV-Vis, FT-IR, ¹H NMR, ¹³C NMR and FAB-mass) data. NMR spectra indicate that there is rapid rotation of the cyclopentadienyl ring around the metal-ring axis at 25 °C. Studies were conducted to assess the growth inhibiting potential of the ligands and complexes against various bacterial strains. The complexes are found to be potent bactericides than the ligands.
Keywords : Dithiocarbamate, α -amino acid, bis(cyclopentadienyl)hafnium(IV), synthesis.

Synthesis and antimicrobial studies of 4-oxo-thiazolidine derivatives

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Abstract : Ethyl (4-chlorophenoxy)acetate (1) upon condensation with hydrazine hydrate gave 2-(4-chlorophenoxy)-acetohydrazide (2) which by condensation with aromatic aldehydes gave 2-(4-chlorophenoxy)-*N*-(substitutedbenzylidene) acetohydrazide (3a-k). Derivatives (3a-k) fused with thioglycolic acid and thiolactic acid gave 4-thiazolidinone derivatives (4a-k) and 5-methyl-4-thiazolidinone derivatives (5a-k) respectively. 4-Thiazolidinone derivatives (4a-k) treated with formaldehyde and *p*-fluoroaniline gave 2-(4-chlorophenoxy)-*N*-{2-(substitutedphenyl)-5-[(4-fluorophenyl)aminomethyl]-4-oxo-1,3-thiazolidine-3-yl}acetamide (6a-k). The structures of the synthesized compounds were assigned on the basis of IR, ¹H NMR, LC-MS and elemental analysis data.

Keywords : Cycloaddition, Schiff base's derivatives, Mannich reaction, thiazolidinones, microwave method, antimicrobial activity.

Studies on *Phyllanthus emblica* seed oil

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Abstract : GLC has been used to determine the fatty acid profile. The oils due to its saturated/unsaturated fatty acids combine and nutritional properties, have been tried for its possible utility in confectionary (chocolate making) as an alternative to traditional cocoa fat. ¹³C NMR has been used to evaluate the olefinic content of the seed oil. High temperature heating of the oil produces an aldehyde. Physico-chemical properties show variation after heat treatment.

Keywords : Seed oil, *Phyllanthus emblica*.

Studies on non-traditional seed oil of *Boswellia serrata*

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Abstract : The saponifiable part of the seed oil apart from usual fatty acids, contained two unusual fatty acids – which have been identified as methyl hexadecanoic (1) and octa decadienoic (2) acids, on the basis of mass spectral study of their piconyl ester derivatives. The unsaponifiable part contained stigmastadiene.

Keywords : *Boswellia serrata*, seed oil.

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Synthesis of some newer thiazolidinones

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Abstract : Some new 2-[2*c*-substituted -4*c*-thiazolidinon-3*c*-yl]-5,6-dimethyl-8,9-dihydro-10*H*-benzo[7]annuleno[9,8-*d*]thiazoles (5a-f) have been synthesized by 1,3-cyclocondensation of thioglycolic acid with 1-substituted-*N*-(5,6-dimethyl-8,9-dihydro-10*H*-benzo[7]annuleno[9,8-*d*]thiazol-2-yl)methanimines (4a-f). The structures of all these compounds have been delineated by elemental analysis and spectral studies (IR, ¹H NMR and Mass).

Keywords : Benzosuberones, thiazolidinone.

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Microwave assisted synthesis of 2,3,4-trisubstituted 1,2-dihydropyrimido-[1,2-*a*]benzimidazole

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Abstract : A new methodology for the synthesis of 2,3,4-trisubstituted 1,2-dihydropyrimido[1,2-*a*]benzimidazole has been described. All the synthesized compounds were characterized on the basis of IR, ¹H NMR and elemental analyses. Synthesized compounds exhibited moderate to good anti-fungal and anti-bacterial activities.

Keywords : 2-Aminobenzimidazole, microwave-assisted synthesis, pyrimidobenzimidazoles.

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Microgram determination of ticlopidine hydrochloride in single pharmaceutical preparation by HPLC method

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Abstract : A new HPLC method for the determination of ticlopidine in single and in their pharmaceutical preparations has been described. The method is based on reverse phase liquid chromatography using C-18 column and a suitable mobile phase. The detection is done at 220 nm. The flow rate is adjusted at 1.0 mL/min and the linearity is established.

Keywords : Microgram, HPLC, ticlopidine.

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Spectrophotometric estimation of celecoxib in bulk and its pharmaceutical formulation

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Abstract : A new simple, precise, sensitive, highly specific and economical ultraviolet spectrophotometric method for the determination of celecoxib in bulk and its pharmaceutical formulation (dispersible tablets and capsules) has been developed. The absorbance maxima of celecoxib in a mixture of methanol and 0.01 N sodium hydroxide (1 : 1 v/v) were determined at 253.1 nm. Beer's law is obeyed over concentration range of 8-22 mg/ml with correlation coefficient $r > 0.999$. The results have been validated statistically and by recovery studies.

Keywords : Spectrophotometry, celecoxib, sodium hydroxide, methanol, correlation coefficient.

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Spectrophotometric determination of cobalt in biological samples using 2-acetylpyridine semicarbazone

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Abstract : Analytical application of 2-acetylpyridine thiosemicarbazone (APT) and 2-acetylpyridine semicarbazone is described for the direct non-extractive spectrophotometric determination of cobalt. The reagents react with cobalt in acidic medium (pH 6.0, sodium acetate-acetic acid buffer) to form orange yellow colored 1 : 2 (M : L) complexes. The colour reactions are instantaneous and absorbance values remain constant for over 24 h. The molar absorptivity and Sandell's sensitivity of APT and APS methods are found to be 1.25×10^4 and 1.45×10^4 L mol⁻¹ cm⁻¹ and 0.0047 and 0.004 mg cm⁻² of Co^{II} respectively. The systems obey Beer's law in the range of 0.236-2.357 mg/ml of Co^{II}. Since APS method is more sensitive it was applied for the determination of cobalt in biological samples.

Keywords : Spectrophotometry, cobalt, 2-acetylpyridine semicarbazone, biological samples.