

Synthesis, spectral, thermal and antimicrobial studies of oxomolybdenum(V) and dioxomolybdenum(VI) complexes of Schiff base derived from 4-aminoantipyrine and 2,4-dihydroxyacetophenone

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Abstract : Some novel metal complexes of oxomolybdenum(V) and dioxomolybdenum(VI) with a Schiff base derived from 4-aminoantipyrine and 2,4-dihydroxyacetophenone have been synthesized and characterized by elemental analyses, molar conductance, magnetic susceptibility, IR, UV-Visible, ¹H NMR and ESR spectral studies. The X-ray diffraction studies of the complex [MoO(AAPDHA)Cl₃] (where AAPDHA = aminoantipyrine dihydroxyacetophenone) indicated orthorhombic crystal lattice with unit cell dimensions $a = 6.3314 \text{ \AA}$, $b = 8.5059 \text{ \AA}$ and $c = 15.405 \text{ \AA}$. The thermal behaviour of one of the complexes has also been examined. The ligand and the complexes were screened for their possible antibacterial and antifungal activities. The Schiff base ligand behaves as a neutral bidentate chelating agent in all the complexes, coordinating through carbonyl oxygen and azomethine nitrogen.

The complexes have distorted octahedral geometry.

Keywords : Oxomolybdenum(V), dioxomolybdenum(VI), Schiff base, 2,4-dihydroxyacetophenone.

Spectrophotometric determination of tungsten(VI) using 3-hydroxy-6-methyl-2-(4-tolyl)-4-oxo-4H-1-benzopyran

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Abstract : A new, simple, sensitive and selective spectrophotometric method is developed for the trace determination of tungsten. A light yellow (1 : 2) complex of tungsten(VI) is formed with 3-hydroxy-6-methyl-2-(4-tolyl)-4-oxo-4H-1-benzopyran (HMTB) in perchloric acid medium. The coloured species is extracted into chloroform and absorbance is measured at 396-402 nm. The method obeys Beer's law in the range 0-9.5 mg W^{VI} ml⁻¹, with a molar absorptivity and Sandell's sensitivity of $1.43 \times 10^5 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and 0.0013 mg W^{VI} cm⁻², respectively at 398 nm. The method is free from the interference of a large number of elements and has been applied for the determination of tungsten in a variety of samples with satisfactory accuracy.

Keywords : W^{VI}, spectrophotometric, extraction, determination.

Kinetics and mechanism of oxidation of benzyl ethers by N-chloronicotinamide in aqueous acetic acid medium

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Abstract : Kinetics of oxidation of benzyl ethers by *N*-chloronicotinamide (NCN) in aqueous acetic acid medium have been investigated by potentiometric method. The observed rate of oxidation is first order in both [NCN] and [benzyl ethers]. An increase in the dielectric constant of the medium increases the rate. Addition of nicotinamide (NA), the reduction product of NCN, have a negligible effect on the rate of oxidation. A small increase in the rate is observed with increase in [HClO₄] and [NaCl]. Benzaldehyde is identified as the major product of oxidation.

Keywords : Oxidation, benzyl ethers, *N*-chloronicotinamide.

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Viscometric investigations and molecular interactions of triphenodioxazine dyes in mixed organic solvents

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Abstract : Colloid chemical behaviour of 6,13-dichloro-3,10-dimethyl triphenodioxazine (TPDO-I) and 6,13-dibromo-3,10-dimethyl triphenodioxazine (TPDO-II) in non-aqueous solvent mixture benzene-methanol of varying composition has been investigated by viscometric measurements at 303 ± 0.1 K. The viscosity of the system increases with the increase in triphenodioxazine concentration. The Trend Change Point (TCP) values have been determined by intersection of two straight lines which are found to be dependent on the composition of solvent mixtures. The study confirms that the nature of triphenodioxazine agglomerate formed below and above 50% benzene concentration is quite different. The viscometric data have been analysed in terms of Einstein, Vand, Moulik and Jones -Dole equations. These well known equations have been successfully applied to explain the results of viscosity measurements and the viscometric parameters show that the behaviour of triphenodioxazine dyes changes in the proximity of 50% benzene concentration.

Keywords : Triphenodioxazine dye, trend change point, dye aggregation, viscosity, solute-solute interaction, solute-solvent interaction.

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Dye-sensitized photochemical transformation of some pharmaceutical drugs by singlet molecular oxygen

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Abstract : Dye-sensitized photo-oxygenation of thiopentone and phenytoin by singlet molecular oxygen has been carried out under various reaction conditions, including variation of solvents and sensitizers. The product has been isolated and characterized by elemental analysis, physical, chemical and spectral data. A suitable mechanism has been proposed for the formation of photo product. To confirm the participation of singlet oxygen in the reaction, singlet oxygen scavengers have been used in the photo-oxygenation reaction.

Keywords : Photo-oxidation, singlet molecular oxygen, thiopentone, phenytoin, dye sensitizer.

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Study of azo ester mesogenic homologous series : 2-(4*n*-alkoxy benzoyloxy)-naphthyl-1-azo-4*m*-methyl benzenes

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Abstract : Mesogenic homologous series : 2-(4*n*-alkoxy benzoyloxy)-naphthyl-1-azo-4*m*-methyl benzenes is synthesized to understand the effects of structural variation on mesogenic characteristics. All the members of the homologous series exhibit enantiotropic nematic mesophase with threaded type of textures. Smectic mesophase does not occur in any homologue of the series even in the monotropic condition. Well-known odd-even effect is observed in the nematic isotropic transition curve. Thermal stabilities and liquid crystalline properties are compared with other structurally identical homologous series. Melting and transition temperatures are determined by hot stage polarizing microscope. Analytical data support the structures of molecules.

Keywords : Azomesogens, smectic, nematic, mesomorphism.

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Oxidative decarboxylation and deamination of essential amino acids by *N*-Bromonicotinamide – A kinetic study

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Abstract : The kinetics of oxidation of valine, leucine, isoleucine and phenylalanine by *N*-Bromonicotinamide (NBN) in acetic acid-water in presence of hydrochloric acid has been studied. The reaction shows inverse order dependence on oxidant and [H⁺]. Increase in [amino acid] has a slight positive effect on the rate, indicating fractional order dependence. Addition of salts like K₂SO₄, Na₂SO₄, KCl to the reaction medium has no effect on the rate. Increase in temperature increases the rate of the reaction. The activation parameters have been computed. A mechanism consistent with the results has been proposed.

Keywords : Kinetics, oxidation, valine, leucine, isoleucine, phenylalanine, *N*-bromonicotinamide, mechanism.

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Oxidation of benzhydrols by *N*-bromophthalimide : A kinetic and mechanistic study

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Abstract : Kinetic investigations of oxidation of benzhydrol and *p*-substituted benzhydrols by *N*-bromophthalimide (NBP) in aqueous acetic acid medium in presence of mercuric(II) acetate as a scavenger have been studied. In absence of mineral acids, the oxidation kinetics of benzhydrols by NBP shows a first order dependence on NBP and fractional order on benzhydrols. The variation of ionic strength, $\text{Hg}(\text{OAc})_2$, H^+ and phthalimide (reaction product) have insignificant effect on reaction rate. Activation parameters for the reaction have been evaluated from Arrhenius plot by studying the reaction at different temperatures. A mechanism involving transfer of hydride ion in rate determining step is suggested.

Keywords : Kinetics, oxidation, benzhydrol, *N*-bromophthalimide.

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Acid-catalysed rearrangements of allyl 4-hydroxybenzoate and 3-methylbut-2-enyl-4-hydroxybenzoate

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Abstract : On treatment with 75% polyphosphoric acid or TFA- H_2SO_4 (50%) allyl 4hydroxybenzoate and 3-methylbut-2-enyl-4-hydroxybenzoate afforded 4-hydroxy-3-(2 α -hydroxypropyl)benzoic acid and 4-hydroxy-3-(3 α -hydroxy-3 α -methylbutyl)benzoic acid, respectively. Mechanisms of these reactions are discussed.

Keywords : Polyphosphoric acid, trifluoroacetic acid, acid-catalysed rearrangement, Reimer-Tiemann reaction.

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CuSO₄·5H₂O : A novel and efficient catalyst for the synthesis of bis-indolylmethanes

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Abstract : bis-Indolylmethanes are synthesized in a one pot coupling of carbonyl compounds and indoles using catalytic amount of CuSO₄·5H₂O at ambient temperature. It is first ever use of CuSO₄·5H₂O in this reaction yielding products in excellent yields without the formation of any side products.

Keywords : bis-Indolylmethanes, CuSO₄·5H₂O, indole, carbonyl compounds.

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Synthesis, characterization and antimicrobial activity of substituted 3-(phthalazine-1-ylamino)alkanoic acid containing imidoxy moiety

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Abstract : Hydrozinolysis of phthalyl derivatives of amino acid 1a-c afforded [(4-oxo-3,4-dihydrophthalazine-1-yl)amino]substituted acetic acid 2a-c, which on reaction with POCl₃/PCl₅ gave [(4-chlorophthalazine-1-yl)amino]substituted acetic acid 3a-c. This when condensed with *N*-hydroxyphthalimide/ *N*-hydroxysuccinimide 4a-b yielded phthalimido or succinimido-2-((4-phthalazine-1-yl)amino)substituted acetic acid 5a-f. Condensation of compounds 2a-c with *w*-bromoalkoxyphthalimide 6a-b furnished the corresponding 2-[(3-{2-[(1,3-dioxo-1,3-dihydro-2*H*-isindol-2-yl)oxy]ethyl}-4-oxo-3,4-dihydrophthalazine-1-yl)amino]substituted acetic acid 7a-f. All the compounds were screened for antimicrobial activities.

Keywords : Phthalazine, *w*-bromoalkoxyphthalimide, condensation, antimicrobial activity, *N*-hydroxyphthalimide.

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Synthesis and biological evaluation of 3-(phthalimidoethyl)-4-(5-substituted isoxazoline and pyrazoline) substituted benzanilides

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Abstract : 3-Phthalimidoethyl benzoic acids 2a-g were prepared by treating *N*-hydroxyethylphthalimide 1 with substituted benzoic acids. The corresponding acid chlorides 3a-g were condensed with 4-aminoacetophenone in the presence of anhydrous potassium carbonate to give 3-phthalimidoethyl-4-acetyl substituted benzanilides 4a-g. The substituted benzanilide derivatives 4a-g were condensed with diverse aromatic aldehydes to afford compounds 5a₁-d₇. Compounds 5a₁-d₇ on treatment with hydroxylamine hydrochloride and hydrazine dihydrochloride separately in the presence of sodium acetate afforded titled compounds 6a₁-d₇ and 7a₁-g. The structures of the newly synthesized compounds have been established by elemental analysis and spectral data and some of the selected compounds have been screened for their antibacterial, antifungal, anthelmintic and hypoglycemic activities.

Keywords : Phthalimide, isoxazole, pyrazole, antimicrobial, anthelmintic activity.

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Electrochemical determination of chitosan based on its supramolecular interaction with thorin

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Abstract : In this paper the diazo dye thorin was selected as a new electrochemical probe for the determination of chitosan (CTS). The electrochemical behavior of thorin with CTS was investigated on a dropping mercury working electrode in 0.2 M Britton-Robinson (B-R) buffer solution (pH 3.7). In B-R buffer thorin could be easily reduced on mercury electrode and showed a sensitive linear sweep voltammetric reductive peak at -0.57 V (vs SCE). In the presence of CTS, the reductive peak current of thorin decreased without the shift of the reductive peak potential and no new peaks appeared, which was due to the formation of a supramolecular complex of thorin with CTS. The binding constant and the binding ratio of thorin with CTS were calculated by the electrochemical data and found to be 1.07×10^9 and 2 : 1, respectively. Under the selected conditions, the decrease in second order derivative linear sweep voltammetric reductive peak current of thorin was in proportion to the CTS concentration in the range 2.5 to 30.0 mg L⁻¹ and the detection limit of CTS was calculated as 1.35 mg L⁻¹ (3s). The relative standard deviation (RSD) for 10 replicate determination of 20.0 mg L⁻¹ CTS was 0.79%. Different kinds of samples containing CTS were detected satisfactorily with this method.

Keywords : Chitosan, thorin, supramolecular, interaction, linear sweep voltammetry.

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Effect of physical parameters on alcohol dehydrogenase and aldehyde dehydrogenase during the production of acetic acid from ethanol by an ethanol resistant strain *Saccharomyces cerevisiae* AB₁₀₀

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Abstract : An ethanol resistant strain of *Saccharomyces cerevisiae* AB₁₀₀ was examined to ascertain its acetic acid production. The influence of physical parameters on acetic acid production and

activities of alcohol dehydrogenase (alcohol: NAD⁺ oxidoreductase; E.C. 1.1.1.1) and aldehyde dehydrogenase (aldehyde : NAD⁺ (P⁺) oxidoreductase; E.C. 1.2.1.5) were also observed. The optimal conditions finally found were : initial pH = 4.5, incubation temperature = 30 °C, incubation time = 48 h, volume of medium = 85 ml, age of inoculum = 48 h and cell density = 10.25 × 10⁵. These optimal conditions were also optimum for the activities of the two main regulatory enzymes i.e. alcohol dehydrogenase and aldehyde dehydrogenase.

Keywords : Acetic acid, physical parameters, alcohol dehydrogenase, aldehyde dehydrogenase.

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Synthesis and characterization of manganese(II) complexes derived from 2-substituted benzaldehyde semicarbazones and thiosemicarbazones

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Abstract : The metal complexes of manganese(II) with 2-chlorobenzaldehyde/2-ethoxybenzaldehyde semicarbazone (cbsc, ebsc) and thiosemicarbazone (cbtsc, ebtsc) have been synthesized and characterized by means of various physico-chemical techniques viz. elemental analysis, magnetic moment, infrared and electronic spectral studies. IR spectral studies showed bidentate chelating behaviour of the ligands coordinating through azomethine nitrogen and carbonyl oxygen/thio keto sulphur atoms. Magnetic moment of these complexes indicates high spin configuration. The probable structure for all the complexes is proposed here.

Keywords : Semicarbazone, manganese(II), thiosemicarbazone, synthesis, complex, benzaldehyde.

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Polymetallic complexes. Part-LXXXXIII : O[⊖]C[⊖]O[⊖]N[⊖]O and O[⊖]C[⊖]O[⊖]N donor azodye dimeric complexes of Co^{II}, Ni^{II}, Cu^{II}, Zn^{II}, Cd^{II} and Hg^{II}

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Abstract : Twelve dinuclear complexes of Co^{II}, Ni^{II}, Cu^{II}, Zn^{II}, Cd^{II} and Hg^{II} with one tetradentate O[⊖]C[⊖]N[⊖]O donor azodye ligand (LH₃), 3-(2-hydroxynaphthyl-4-sulphonic acid azo)-2-hydroxy-5-chlorobenzoic acid (HNSAHCB) and one tridentate O[⊖]C[⊖]N donor azodye ligand (L₃H₂), 3-(naphthyl azo)-2-hydroxy-5-chlorobenzoic acid (NAHCB) have been prepared. The complexes have been characterized by analytical, conductance, magnetic susceptibility, IR, electronic spectra, ESR, NMR and X-ray diffraction (powder pattern) data. The cobalt(II) and nickel(II) complexes are found to be octahedral. Copper(II) complexes are distorted octahedral and a tetrahedral stereochemistry has been assigned for Zn^{II}, Cd^{II} and Hg^{II} complexes.

Keywords : Polydentate azodye ligands, polymetallic complexes.

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Semi-empirical (PM3) calculations on b-diketones and their Zn^{II} complexes

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Abstract : Semi-empirical calculations on β -diketones (acetylacetone, benzoylacetone and dibenzoylmethane) and their Zn^{II} complexes $Zn(acac)_2$, $Zn(acac)(bzac)$, $Zn(bzac)_2$, $Zn(acac)(dbzm)$ and $Zn(bzac)(dbzm)$ have been carried out using PM3 method with the help of MOPAC 6.0 program. The values for the heats of formation, total energies, ionization potentials, atom electron densities, atomic charges, bond lengths and bond angles have been determined and optimized geometries have been worked out. The heats of formation are -117.0306 to -6.3592 kcal, total energies are -4469.3733 to -2602.5726 eV and ionization potentials are 8.9802 to 9.1501 eV. Atom electron densities on the oxygen atoms surrounding zinc atom are 6.2991 to 6.3187 and atomic charges are -0.3187 to -0.2991 . Zn-O bond lengths are in the range 1.9796 to 2.0348 Å. O-Zn-O bond angles are close to 109.5° (99.1724° to 122.7135°) supporting distorted tetrahedral geometry for the complexes.

Keywords : Semi-empirical calculations, PM3 method, zinc.

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A study of oxidation of primary and secondary alcohols by diethylammonium chlorochromate

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Abstract : Kinetics of oxidation of primary and secondary alcohols by diethylammonium chlorochromate (DEACC) has been studied in aqueous-acetic acid medium. The oxidation of various primary as well as secondary alcohols by DEACC resulted in the formation of corresponding aldehydes and ketones respectively in good yields. The reaction is first order in DEACC and Michaelis-Menten type in alcohols. The reaction is first order with respect to hydrogen ion concentration. A suitable mechanism of the reaction has also been proposed.

Keywords : Diethylammonium chlorochromate, mechanism, alcohol, oxidation.

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Synthesis and biological potential of nitrones of 4-chlorobenzaldehyde and ethylvanillin

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Abstract : Nitrones of 4-chlorobenzaldehyde (1a-6a) and ethylvanillin (1b-6b) were synthesized by condensing 4-chlorobenzaldehyde (a) and ethylvanillin (b) with phenylhydroxylamines (1-6) in equimolar ratio. The synthesized nitrones were characterized on the basis of elemental analysis and spectral studies and screened for antifungal potential against six phytopathogenic fungi and nematocidal activity against two nematodes.

Keywords : Nitrones, 4-chlorobenzaldehyde, ethylvanillin, antifungal potential, nematocidal activity.

Flavonoid glycosides from *Myrica esculenta* leaves

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Abstract : Two flavonoid glycosides identified as flavone 4*o*-hydroxy-3*o*,5*o*,5*o*-trimethoxy-7-*O*- β -D-glucopyranosyl(1 \rightarrow 4)- α -L-rhamnopyranoside (1) and flavone 3*o*,4*o*-dihydroxy-6-methoxy-7-*O*- α -L-rhamnopyranoside (2) with three known compounds β -sitosterol, β -sitosterol- β -D-glucopyranoside and quercetin have been isolated from the leaves of *Myrica esculenta*. Their structures were elucidated on the basis of spectroscopic evidences and chemical studies.

Keywords : *Myrica esculenta*, flavonoid glycosides.

Synthesis of 2-aryl-3-*N*¹⁰-[(acetylamino)-1,3-thiazolidin-4-ones] and 5-arylidine-2-aryl-3-*N*¹⁰-[(acetylamino)-1,3-thiazolidin-4-ones] and their antimicrobial and diuretic activities

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Abstract : Thirty-nine new phenothiazino-4-oxo-thiazolidines and their 5-arylidines were synthesized and evaluated for their antimicrobial and diuretic activity. The structure of all the products was corroborated by IR, ¹H NMR, mass spectra and elemental analysis. Some of the newly synthesized compounds show promising antimicrobial and diuretic activity.

Keywords : Synthesis, antimicrobial, diuretic, thiazolidino-arylidines.

Study of Friedel-Crafts acylation and Baeyer-Villiger oxidation reactions on benzocycloheptanones and 1-tetralones in acid media

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Abstract : Synthesis and characterization of some new substituted naphthopyrones (2a-d) and 4,5-dihydro-3*H*-benzo[*b*]oxocin/oxepin-2-ones (3a-d), starting from 1-tetralone and benzocycloheptanones have been reported. Biological testing of these compounds for their antimicrobial activity has been carried out.

Keywords : Friedel-Crafts acylation, Baeyer-Villiger oxidation, naphthopyrones, antimicrobiol activity, synthesis.

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Performance of phenylthiourea as corrosion inhibitor for aluminum in trichloroacetic acid

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Abstract : The corrosion of aluminum in trichloroacetic acid (TCA) containing phenylthiourea has been studied. In plain TCA, the corrosion rate increases with the acid concentration. At constant acid concentration, the inhibition efficiency (I.E.) of phenylthiourea increases with the inhibitor concentration. At constant inhibitor concentration, the I.E. decreases with the increase of acid concentration. As temperature increases, percentage of inhibition decreases. Plot of $\log (q/1 - q)$ versus $\log C$ results in a straight line, suggesting that the inhibitors cover both the anodic and cathodic regions through general adsorption following Langmuir isotherm. The curves show very little anodic but significant cathodic polarization.

Keywords : Corrosion, aluminum, trichloroacetic acid, phenylthiourea.