

Synthesis, spectral and antibacterial studies of bis(cyclopentadienyl)titanium(IV)/zirconium(IV) and mono(cyclopentadienyl)titanium(IV) complexes of antipyrine Schiff bases

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Abstract : The reactions of mono(cyclopentadienyl)titanium(IV) trichloride and bis(cyclopentadienyl)titanium(IV)/zirconium(IV) dichloride with Schiff bases, derived by condensing 4-aminoantipyrine with benzaldehyde (L_1), furfuraldehyde (L_2), pyridine-4-carboxaldehyde (L_3) or salicylaldehyde (L_4), have been studied in anhydrous tetrahydrofuran or dichloromethane and products of types $[Cp_2M(L)Cl]Cl$, $[CpTiCl_3(L)]$ ($L = L_1, L_2$ or L_3); $[Cp_2M(L_4)]Cl$, $[CpTiCl_2(L_4)]$, $[CpTi(L_4)_2]Cl$ [$M = Ti^{IV}$ or Zr^{IV}] have been isolated. Tentative structures are proposed for these complexes based upon elemental analyses, electrical conductance and spectral (electronic, IR, 1H NMR and ^{13}C NMR) data. Attempts have been made to establish a correlation between antibacterial activity and the structures of the products.

Keywords : Titanium(IV), zirconium(IV), antipyrine, antibacterial study.

Binuclear complexes of Cu^{II} and Ni^{II} Schiff base of salicylaldehyde and 1,2-propylenediamine with alkali metal salts of oxygen and nitrogen containing organic acids

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Abstract : A number of new heterobinuclear alkali metal complex of general formula $M_1PS.M_2L$ have been synthesized and characterized, where $M_1 = Cu^{II}$ or Ni^{II} , $PS = N,N'$ -1,2-propylene-bis(salicylaldimine), $M_2 = Li, Na$ or K and $L =$ deprotonated *o*-nitrophenol, 2,4-dinitrophenol, 2,4,6-trinitrophenol and 1-nitroso-2-naphthol. These complexes have been characterized by elemental analysis, electronic and IR spectral analysis, magnetic moment and molar conductance measurements. 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 *E. coli*, *S. aureus*, *C. albicans* and so these may be treated as good antibacterial agents and fungicides. It was therefore, thought interesting to synthesize the title compounds and examine their antimicrobial activity.

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

Synthesis and mesomorphic properties of azoester mesogens : 4-(4*n*-alkoxy benzoyloxy)-2-methyl phenylazo-4*o*-methoxy benzenes

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Abstract : A new homologous series of azomesogens, 4(4*n*-alkoxy benzoyloxy)-2-methyl phenylazo-4*o*-methoxy benzenes is synthesized. Mesomorphism commences from the very first homologue of the series and continues upto tetradecyl derivative. Last hexadecyl derivative is nonmesomorphic. All mesomorphic homologues are enantiotropic nematic. Thus, present homologous series is purely nematogenic. The usual odd-even effect is observed in nematic-isotropic transition curve. Transition curve behaves in a normal manner. The average thermal stability and mesomorphic characteristics are compared with structurally similar homologous series. Series is of middle ordered melting type without display of smectic character even in the last member of the series.

Keywords : Mesomorphism, nematic, liquid crystal, azomesogens, mesophase.

Study of binary systems and determination of latent transition temperatures (LTTs) of nonmesogenic components

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Abstract : The latent ability to exhibit mesophase transition temperatures of a substance is determined for non-mesomorphic components of the binary systems, viz. Schiff's bases (B₁, B₂, B₃) by mixing them with a monotropic nematic component (A) as a common component. Components of the binary systems were prepared by established methods of preparation. The transition temperature of pure components and binary mixtures were observed through hot stage polarizing microscope. The phase diagrams are drawn for transition temperatures versus mole% of component (A). Analytical data support the structure of the molecules.

The latent transition temperature of each non liquid crystal component is determined by the extrapolation of nematic-isotropic (or vice versa) transition curve to zero mole% of (A). The values of latent transition temperatures (LTTs) for different Schiff's bases are determined and well agree with previously work. Thus, reliability of extrapolation method is raised and credibility to the conclusions already drawn earlier is supported by present investigation.

Keywords : Nematic, smectic, mesogen, mesomorphism.

Study of binary systems including mesophase and determination of latent transition temperature (LTT)

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Abstract : Six binary systems consisting of common component A₁ and A₂ (mesogen) are mixed with uncommon component (B) (nonmesogen), i.e. *para*-substituted Schiff's bases. The results shows that the mixed melt have the latent ability to exhibit mesomorphism. This latent transitions temperatures of nonliquid crystal substances are determined by the extrapolation method. The transition temperatures of the binary systems as well as the constituent components were observed through hot stage polarising microscope. LTTs determined are mutually comparable and well compared with others.

Keywords : Mixed mesomorphism, nematic, liquid crystal, mixed melt, mesogen.

Preparation of data base for the determination and estimation of high explosives and a comparative study of different methods of estimation of RDX

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Abstract : Analysis of post explosion residues containing high explosive and quick disposal of cases require a suitable analytical data base in the laboratory for the rapid detection and estimation of explosives in the residues. GC-MS is found to be appropriate for the separation, detection and estimation of explosives and a suitable data base for the detection and estimation of high explosives is presented. RDX has been found to be one of the main constituents of the explosives. Depending on the availability of RDX in the post explosion residues, suitable analytical method is needed for its detection and estimation. A comparative study of the different methods of detection and quantitation of RDX using UV-Vis, FTIR, GC-MS, HPLC and HPTLC instruments has been made for proper selection of the analytical method.

The range of detection and estimation of RDX is 100 mg – 1 ng or less depending on the method used.

Two chromogenic agents tryptophan and chromotropic acid used for the detection of CH₂O and RDX (which decompose to 3CH₂O in presence of strong H₂SO₄) were examined for the quantitation of CH₂O and traces of RDX.

Formaldehyde in presence of H₂SO₄ and traces of FeCl₃ solution reacts with tryptophan to form a red colored complex with absorption maximum at 451 nm. Similarly, CH₂O (RDX) reacts with chromotropic acid in 16N H₂SO₄ to form a blue-violet colored complex with an absorption maximum at 577 nm. However, CH₂O can be completely converted into the complex with large excess of tryptophan or chromotropic acid and the extinction coefficients of the complex are utilized for the quantitation of CH₂O or RDX. The stability constant of the complex at 298 K between CH₂O and chromotropic acid has been determined.

Keywords : CH₂O, chromotropic acid, FTIR, GC-MS, HPLC, HPTLC, RDX, tryptophan.

Studies of solar effect of safranine, methylene blue and Azur-B with reductants and their photogalvanic effect

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Abstract : Photochemical studies of dyes has contributed to the understanding of the mechanism of electron transfer reactions in photoelectro-chemical devices¹⁻⁶. A number of photoinduced redox reactions have been studied. These reactions prove to be helpful in electrochemical cell for the conversion of solar energy to electrical energy by chemical means⁷⁻⁹. It has been found that phenazine dyes in conjunction with different reducing agents is efficient photosensitizers for probing charge transfer in photochemical cells¹⁻⁸. Studies carried out by Isabel *et al.*⁹ on the photo redox reactions of thionine with iron(II), cobalt(II) and manganese(II) by flash photolysis techniques provided better understanding of the mechanism of thionine based photogalvanic cell. A detailed literature survey¹⁻²³ reveals that different photosensitizers mainly cationic and neutral dyes and reductants have been used in photogalvanic cells but the use of combination of anionic, cations and neutral dyes as photosensitizers in the photogalvanic cell for solar energy conversion and storage is rare, and hence the present work is undertaken. The system consisting of anionic dye safranine in aqueous solution generates photovoltage when studied in photogalvanic cell. The open circuit photovoltage, the short circuit current and solar energy efficiency of these systems have been determined. The effect of different parameters on electrical output of the cell were observed and a mechanism has also been proposed for the generation of photocurrent in photogalvanic cell.

Keywords : Soalr effect, Azur-B, safranine, methylene blue, photogalvanic effect.

Kinetics and mechanism of oxidation of some aldoses by hexacyanoferrate(III) ions in aqueous alkaline buffered medium

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Abstract : Kinetics of oxidation of some aldoses like glucose, galactose, xylose and ribose by hexacyanoferrate(III) ions in aqueous alkaline buffered medium has been investigated. The kinetic results indicate the zero-order kinetics in hexacyanoferrate(III) and first-order in aldoses and OH⁻. The ionic strength of the medium has no influence on oxidation rate. Various activation parameters were also calculated at four different temperatures : 30, 35, 40 and 45 °C. The corresponding acids were identified as the main oxidation products of the reaction. A suitable mechanism consistent with experimental findings has been proposed.

Keywords : Glucose, galactose, xylose, ribose, hexacyanoferrate(III) ions, buffer.

Synthesis of amino acid derivatives of 2-mercaptobenzothiazole

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Abstract : The reaction of ethyl benzothiazole-2-yl-thioacetate and methyl benzothiazole-2-yl-thiopropionate with various amino acids in presence of alkali at 0–5 °C to form peptide has been described.

Keywords : Peptides, 2-mercaptobenzothiazole, amino acids.

Synthesis and characterization of polyhalogen copper phthalocyanine

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Abstract : Polyhalogen copper phthalocyanine has been synthesized by bromination with liquid bromine and chlorine gas at 156–158 °C under acidic melt media, made with eutectic mixture of aluminium chloride and sodium chloride, followed by chlorination at 156–158 °C. The resultant halogenated mass isolated by pouring it into the mixture of water and hydrochloric acid. The crude pigment thus obtained evaluated analytically in terms of degree of halogenation and number of atomic substitution.

Keywords : Polyhalogen, eutectic, Pigment Green 36.

Modern Friedel-Crafts chemistry. Part-29. Cyclialkylation of some triphenylated propane, butane and pentane substrates to diphenylated indans under Friedel-Crafts conditions

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Abstract : Reactions under Friedel-Crafts cyclialkylation conditions produced : isomeric 1,2-diphenylindans from 1,2,3-triphenyl-(1- or 2-)propanol and 1-chloro-2,3,3-triphenylpropane, *trans*-2-benzyl-1-phenylindan from 2-benzyl-1,3-diphenyl-2-propanol, isomeric 1-methyl-1,2-diphenylindans from 2,3,4-triphenyl-2-butanol, 1,1-dimethyl-3,4-diphenylindan from 2-methyl-3,4,4-triphenyl-2-butanol, and isomeric 1-ethyl-1,3-diphenylindans from 1,1,3-triphenyl-3-pentanol. Mechanistic interpretations and product identifications are offered.

Keywords : Cyclialkylation, Friedel-Crafts conditions, triphenylated alkane substrates, diphenylated indans.

Synthesis and biological significance of 2-aminobenzothiazole derivatives

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Abstract : Several new 2-N-(substituted benzylidene)imino-1,3-benzothiazole 1; 2-[2-(substituted phenyl)-4-oxo-1,3-thiazolidine]-1,3-benzothiazole 2 and 2-[5-(arylidene)-2-substituted phenyl-4-oxo-1,3-thiazolidine]-1,3-benzothiazole 3 have been synthesized and evaluated for their antifungal activity against *Aspergillus niger*, *Aspergillus flavus*, *Fusarium oxysporum* and *Trichoderma viride* and antibacterial activity against *Bacillus subtilis*, *Escherichia coli*, *Klebsiella pneumoniae* and *Staphylococcus aureus*.

Keywords : 2-Aminobenzothiazole, antimicrobial activity.

Tabebuin and tecomaquinone-III-dimeric quinones from *Tabebuia rosea*

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Abstract : Two dimeric C₃₀ quinones-tabebuin and tecomaquinone-III have been isolated from the heartwood of *Tabebuia rosea* in addition to the previously reported 1,4-naphthoquinone derivatives. Both the dimeric quinones are reported for the first time from this plant.

Keywords : Tabebuin, tecomaquinone-III, *Tabebuia rosea*, Bignoniaceae.

New triterpene glycosides of *Clematis montana* and their cytotoxic activities

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Abstract : Two new triterpene glycosides designated as montanosides 1 and 2 were isolated from methanolic extract of *Clematis montana*. Their structures have been elucidated as 3-O-b-D-glucopyranosyl-oleanolic acid-28-O-b-D-glucopyranosyl-(1 \otimes 4)-[α -L-rhamnopyranosyl-(1 \otimes 2)]-b-D-xylopyranoside and 3-O-b-D-glucopyranosyl-oleanolic acid-28-O-b-D-glucopyranosyl-(1 \otimes 4)-[β -D-glucopyranosyl-(1 \otimes 3)- α -L-rhamnopyranosyl-(1 \otimes 2)]-b-D-xylopyranoside, respectively by NMR and MS data. Cytotoxic activity of the compounds were evaluated against various squamous carcinoma cells (HSC-2) and human gingival fibroblast cells (HGF). Both of the compounds showed potent cytotoxic activity against the test cells.

Keywords : *Clematis montana*, Ranunculaceae, cytotoxic activity, triterpene glycosides.

Spectroscopic studies of some VO^{II} complexes

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Abstract : The reaction of 2-formylpyridine semicarbazone (HFPS), 2-formylpyridine thiosemicarbazone (HFPTS), 2-acetylpyridine semicarbazone (HAPS) and 2-acetylpyridine thiosemicarbazone (HAPTS) with VOSO₄ in ethanolic solution affords the complexes of formula VOL₂, where L = FPS, APS, FPTS and APTS. The infrared spectra of complexes in comparison to that of the free ligand show the mode of coordination of the ligands. The appearance of three bands in their electronic spectra is indicative of their square pyramidal geometry which is further substantiated by their electron spin resonance spectra. The presence of eight lines in the hyperfine ESR spectra shows that they are mononuclear complexes.

Keywords : VO^{II} complexes, semicarbazone, ESR spectra.

Synthesis and pharmacological evaluation of imidazole derivatives of some non-steroidal anti-inflammatory drugs

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Abstract : A series of imidazole derivatives of NSAIDs (4a-l) have been prepared by condensation of NSAIDs hydrazides (1,2) with substituted oxazolones and evaluated for anti-inflammatory and gastrointestinal toxicity, which showed good anti-inflammatory activity and reduced gastrointestinal toxicity as compared to parent drug.

Keywords : Non-steroidal anti-inflammatory drugs, carragenin, oxazolone, imidazole.

Synthesis and pharmacological evaluation of (N-phenothiazinomethyl)-4-[N-(3-chloro-2-oxo-4-substituted-azetidin)]-5-mercapto-1,2,4-triazoles

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Abstract : (N-Phenothiazinomethyl)-4-[N-(3-chloro-2-oxo-4-substituted-azetidin)]-5-mercapto-1,2,4-triazoles (2a-s) have been synthesised by cycloaddition of (N-phenothiazinomethyl)-4-(arylideneamino)-5-mercapto-1,2,4-triazoles (1a-s) with chloro ketene generated *in situ* from chloroacetyl chloride and triethylamine. All the products have been evaluated *in vitro* for their antibacterial and antifungal activities against some selected microbes and *in vivo* for their antiinflammatory activity by rat paw oedema method.

Keywords : Phenothiazinomerapto-1,2,4-triazoles, antimicrobial, antiinflammatory activities.

A convenient synthesis of crystalline 4-ethyl-2,3-dioxopiperazine-1-carboxamido-*p*-hydroxyphenylacetyl chloride using triphosgene

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Abstract : The crystalline 4-ethyl-2,3-dioxopiperazine-1-carboxamido-*p*-hydroxyphenylacetyl chloride was prepared in 97.6% yield when triphosgene was reacted with D(-)-2-[(4-ethyl-2,3-dioxo-1-piperazineyl)-carbonylamino]-2-(4-hydroxyphenyl)acetic acid (HO-EPCP) in methylene dichloride at 0-5 °C in the presence of triethylamine (TEA). This new method without using chlorinating agent such as phosgene, diphosgene, phosphorus oxychloride or oxalyl chloride enjoys a number of advantages in that the reaction is carried out under mild conditions and is clean.

Keywords : 4-Ethyl-2,3-dioxopiperazine-1-carboxamido-*p*-hydroxyphenylacetyl chloride, D(-)-2-[(4-ethyl-2,3-dioxo-1-piperazineyl)-carbonylamino]-2-(4-hydroxyphenyl)acetic acid, triphosgene.

Microwave assisted synthesis of 2,4-disubstituted pyrimido-[1,2-*a*]-benzimidazoles

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Abstract : A new series of 2,4-disubstituted pyrimidobenzimidazoles were synthesized by conventional as well as microwave methods. The synthesized compounds exhibited moderate to good antifungal and antibacterial activities.

Keywords : 2-Aminobenzimidazoles, pyrimidobenzimidazoles, microwave assisted synthesis, antimicrobial activity.

Synthesis and antimicrobial activity of 2-(6-substituted-1,3-benzothiazol-2-yl)-N-(4-halophenyl)hydrazinecarbothioamide

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Abstract : A number of new 2-(6-substituted-1,3-benzothiazol-2-yl)-N-(4-halophenyl)hydrazinecarbothioamide (5a-o) have been synthesized and screened for antibacterial activity.

Keywords : Benzothiazole, synthesis, antimicrobial activity.

Cyclopeptide alkaloids of *Zizyphus xylopyra*

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Abstract : 14-Membered cyclopeptide alkaloids, franganine, frangufoline, amphibine-D and mauritine-A has been isolated from the whole plant of *Zizyphus xylopyra* and their structures established by spectral evidences. This is the first report of these alkaloids from *Z. xylopyra*.

Keywords : *Zizyphus xylopyra*, alkaloid, cyclopeptide.
