

Application of Nuclear Quadrupole Resonance in chemical compounds with special reference to molecular complexes (charge-transfer complex)

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Abstract : Both chemists and physicists use Nuclear Quadrupole Resonance (NQR) technique as frequently as NMR. Considering the vast range, this review is confined to the field of molecular complexes (charge transfer complexes), which is of great interest to chemists. This review encompasses both the theoretical studies and experimental observations of NQR in molecular complexes.

Keywords : Nuclear Quadrupole Resonance spectra (NQR), molecular complexes, ^{35}Cl NQR studies.

Synthesis, spectral characterization and biological screening of a tetraaza-macrocyclic ligand and its complexes with Co^{II} , Ni^{II} and Cu^{II}

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Abstract : Complexes of cobalt(II), nickel(II) and copper(II) with a 12-membered 1,3,7,9-tetraaza-4,6,10,12-tetraphenyl-2,8-diketocyclododecane macrocyclic ligand (L) have been synthesized. These complexes are characterized by the elemental analysis, molar conductance measurements, magnetic susceptibility measurements, mass, IR, electronic and EPR spectral studies.

All the complexes are non electrolytes so they may be formulated as $[\text{M}(\text{L})\text{X}_2]$ [where, $\text{M} = \text{Co}^{\text{II}}$, Ni^{II} and Cu^{II} and $\text{X} = \text{Cl}^-$ and NO_3^-]. On the basis of IR, electronic and EPR spectral studies an octahedral geometry has been assigned for Co^{II} and Ni^{II} complexes and tetragonal geometry for Cu^{II} complexes. The antimicrobial activities of the ligand and its complexes, as growth inhibiting agents have been screened *in vitro* against different species of bacteria and pathogenic fungi.

Keywords : Cobalt(II), nickel(II), copper(II), tetraazamacrocyclic ligand, complex.

Synthesis and characterization of Cu^{II} and Ni^{II} substituted polytungstates and molybdates

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Abstract : Two sodium salts of mixed heteropoly oxometalates of tungstate and molybdate with the molecular formula $\text{Na}_{10}[\text{Cu}^{\text{II}}\text{Ni}^{\text{II}}\text{W}^{\text{VI}}_{11}\text{O}_{40}]\cdot 21\text{H}_2\text{O}$ (1) and $\text{Na}_{10}[\text{Cu}^{\text{II}}\text{Ni}^{\text{II}}\text{Mo}^{\text{VI}}_{11}\text{O}_{40}]\cdot 20\text{H}_2\text{O}$ (2) having Keggin type anion $[\text{X}^{\text{x}}+\text{Z}^{\text{z}}\text{W}_{11}\text{O}_{40}]^{-(14-\text{x}-\text{z})}$ have been synthesized. The compounds were prepared by hydrothermal method from aqueous acidic solution of reagents. The resulting compounds were characterized by elemental and thermal analyses, IR studies and molecular weight determination.

Keywords : Polyanion of tungstate, Ni substituted POM, molybdonickelate, tungstonickelate.

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Synthesis, spectral characterization, catalytic and biological studies of new Ru^{II} carbonyl Schiff base complexes containing N₂O donor ligands

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Abstract : A series of ruthenium(II) Schiff base complexes of the type $[\text{RuCl}(\text{CO})(\text{B})(\text{L})]$ (where B = PPh₃/AsPh₃/py/pip; L = monobasic tridentate Schiff base ligands) were synthesized and characterized. These complexes catalyze oxidation of alcohols with up to 64.17% in the presence of *N*-methyl morpholine-*N*-oxide (NMO) as co-oxidant. Furthermore, *in vitro* toxicity of these complexes was tested against the growth of bacterial species viz. *Escherichia coli*, *Aeromonas hydrophila* and *Salmonella typhi*. An octahedral structure has been proposed for all of the new complexes.

Keywords : Ruthenium(II) Schiff base complexes, characterization, catalytic oxidation, antibacterial activity.

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Kinetics and mechanism of osmium(VIII) catalysed oxidation of guanidine hydrochloride by alkaline permanganate

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Abstract : The reaction between permanganate and guanidine hydrochloride in alkaline medium has a stoichiometry of 2 : 1 with first order dependence each on [permanganate] and [osmium(VIII)] and fractional order in [OH⁻], while zero order dependence on [guanidine hydrochloride]. No effect of added products, ionic strength and dielectric constant has been observed. Investigation at different temperatures allowed the determination of the activation parameters with respect to slow step of the proposed mechanism. The observed kinetic data were compared with uncatalysed reaction. Entirely different observation obtained are discussed with respect to kinetic result and activation parameters.

Keywords : Kinetics, osmium(VIII), mechanism, guanidine hydrochloride, oxidation.

Non-equilibrium growth patterns in a reaction between thiamine and glucose

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Abstract : Reaction between glucose and thiamine has been studied in aqueous solution containing gel matrix. Growth patterns of the reactants and the product have been studied. Glucose crystallizes in the form of fractal with fractal dimension 1.74 and thiamine as spherulitic when observed under polarized light microscope. Glucose-thiamine reaction product crystallized in different morphologies. Following transitions were observed on increasing the concentration.

Spherulite ® Ringed spherulite ® Revert spaced concentric rings

Besides change in the morphology of the reaction product, XRD, IR and DSC studies also revealed the formation of a new product. During the reaction glucose was continuously consumed on addition of thiamine, obeying the relation $[G]^{-1} = m \log V + c$, where m and c are slope and intercept respectively. Heat of fusion DH of the product was calculated using the expression $DH.m = KA$, where m is the mass of the substance, K and A are the calibration constant and area under the curve respectively. The value was found to be 1.36 kcal/mol, which was lower than those of the individual reactants.

Keywords : Growth pattern, fractal, biomaterial, Schiff base.

Synthesis, characterization and thermal degradation kinetics of polyazomethine-ester

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Abstract : A new polyester containing diimine ring, poly(imino isophthaloyl imino (glyoxal bis(2-methyl-4-hydroxyphenyl)imine)) poly[IPIGI] was synthesized by an interfacial polycondensation reaction. The structure of poly [IPIGI] was confirmed by FT-IR and solid state ¹³C NMR techniques. The thermal stability was tested by TG-DTA and solubility was also studied. The kinetics of the thermal degradation of poly[IPIGI] was investigated by thermogravimetric analysis at different heating rates. TG curves showed that the thermal decomposition of poly[IPIGI] occurred in two stages. The apparent activation energies of thermal decomposition for poly[IPIGI], as determined by the Tang method (TM), the Flynn-Wall-Ozawa method (FWD), the Kissenger-Akahira-Sunose method (KOS) and the Coats-Redfern method (CR) are 74.8, 75.2, 76.5 and 83.9 KJ mol⁻¹ for the first stage decomposition and 143.1, 143.7, 147.0, and 156.2 KJ mol⁻¹ for the second stage decomposition, respectively. The mechanisms of each stage decomposition were also investigated by master plots.

Keywords : Thermal degradation, kinetic method, polyester.

A preliminary study on interaction of H₄ histone with DNA in the unirradiated and gamma irradiated states[†]

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Abstract : H₄ histone (100 µg/ml) in 0.9% sodium chloride showed minimum conductance and absorbance values at 230nm, 280 nm and A_{max} at pH 7.02. So exposition of chromophores was minimum and charge neutralization was maximum at this pH. DNA : H₄ complex (50 : 50 µg/ml) in 0.9% sodium chloride was found to be stable in the pH range of 6.68 to 8.86.

Minor variations in binding of H₄ histone with DNA (pH range 6.68 to 7.15) for forming the DNA : H₄ complex (25 : 25 µg/ml) have occurred when 0.9% sodium chloride, 0.15 M and 0.1 M phosphate buffers have been used as solvents. This is evidenced by variation in position of absorption maximum and intensity of absorption maximum. Nature of binding in last two solvents are alike but different for 0.9% sodium chloride. This is due to variations in (i) counter-ion concentration in solution (i.e. PO₄³⁻, HPO₄²⁻, H₂PO₄¹⁻, Na⁺ and Cl⁻), (ii) exposed functional groups and chromophores (both in far and near UV) of H₄ histone (i.e. imidazole group of histidine, positively charged arginine and lysine) and that of DNA (i.e. -C=C-C=O and -C=C-C=N-) and (iii) charge interactions between amino-acids and peptides of H₄ histone with phosphate groups of DNA. The value of the binding constant for DNA-H₄ complex varied from 3.45 LM⁻¹ (for 0.15 M phosphate buffer) to 3.66 LM⁻¹ (0.1 M phosphate buffer) and to 3.77 LM⁻¹ (for 0.9% sodium chloride). The complex formation of DNA with H₄ histone has also been evidenced by increase in melting temperature by 9°C more for DNA : H₄ complex than that of DNA.

H₄ histone (25 µg/ml, pH 6.80, 0.15 M phosphate buffer) can be used as *in vitro* biological dosimeter by measuring A_{660nm} with Folin-Ciocalteu reagent in the dose range 30-80 Gy at 1.12 Gy/s. Similarly depletion of H₄ histone from DNA-H₄ complex (25 : 25 µg/ml) as detected by Folin-Ciocalteu reagent (A_{660nm}) can also be used *in vitro* biological dosimeter in the dose range of 20 to 70 Gy at a dose rate of 1.12 Gy/s. In 0.9% sodium chloride the response for H₄ histone (i.e. A_{280nm}) did not change with dose. But in 0.01 N hydrochloric acid, pH range 3.92- 4.44, the response for H₄ histone (i.e. A_{210nm}, A_{220nm} and A_{680nm}) decreased with dose up to 50 Gy for a dose rate of 0.0103 Gy/s. Here it can be used as a dosimeter for intestinal tract. These biological *in vitro* dosimeters may find use in radiation-induced nucleosomal aberration.

Keywords : DNA, H₄ histone, amino acid, lysine.

Oxidative kinetics of serine and threonine by N-bromonicotinamide

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Abstract : The kinetics of oxidative decarboxylation and deamination of α-amino acids, namely, serine and threonine by N-bromonicotinamide (NBN) in aqueous acetic acid medium (1 : 1) in presence of hydrochloric acid was studied over the temperature range 308-328 K. The oxidation reaction leads to the formation of corresponding aldehydes, CO₂ and NH₃. The reaction does not induce the polymerization of acrylonitrile. The reaction was found to be first order with respect to substrate concentration. Inverse first order was observed with respect to concentration of oxidant

and mineral acid. Added nicotinamide retarded the reaction. Mercuric acetate had no significant effect on the reaction rate. A decrease in the dielectric constant of the medium increases the rate.

HOBr has been postulated as the reactive oxidizing species in the reaction. The activation parameters were evaluated.

Keywords : Serine, threonine, *N*-bromonicotinamide, kinetics, oxidation, mechanism.

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Synthesis and characterization of UO_2^{VI} , Th^{IV} , ZrO^{IV} and VO^{IV} complexes with Schiff base derived from ethylene diamine/orthophenylene diamine and diacetyl monoxime

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Abstract : A series of complexes of the type $[\text{M}(\text{L})(\text{NO}_3)_2] \cdot m\text{H}_2\text{O}$ and $[\text{VO}(\text{L})(\text{SO}_4)] \cdot 2\text{H}_2\text{O}$, where L is a Schiff base 1,10-dihydroxy-2,3,8,9-tetramethyl-1,4,7,10-tetraza-dec-1,3,7,9-tetraene (DHTTDT) or 5:6-benzo-1,10-dihydroxy-2,3,8,9-tetramethyl-1,4,7,10-tetraza-dec-1,3,7,9-tetraene (BDHTTDT) derived from ethylenediamine/*o*-phenylene diamine and diacetyl monoxime have been synthesized ($\text{M} = \text{ZrO}^{\text{IV}}$, UO_2^{VI} and Th^{IV} ; $m = 1, 2$ and 3 respectively). All the complexes are characterized on the basis of elemental analysis, thermal analysis, molar conductivity, magnetic moment, electronic, infrared, ^1H NMR and ESR spectral studies. The results indicate that the VO^{IV} ion is penta co-ordinated yielding paramagnetic complexes where as UO_2^{VI} , ZrO^{IV} ions are hexa co-ordinated and Th^{IV} ion is octa co-ordinated yielding diamagnetic complexes of above composition.

Keywords : Schiff base, electronic, infrared, ESR, ^1H NMR spectra.

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Effect of synthesis time on yield and particle growth of silica nanosphere

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Abstract : The yield and particle growth of silica nanosphere were studied with respect to reaction time. It is found that the yield gives a half S type curve with reaction time. A bimodal particle size was observed through out the reaction time. With time, the minor particles were increased up to certain size. The other particles were stable as such. An addition of octadecyltrimethoxy silane was normalizing the particle size. In both the cases, the particle shape did not change with any time from nanosphere.

Keywords : Silica nanosphere, time, yield, particle size.

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Charge transfer complex of *p*-phenylenediamine and *p*-chloranil

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Abstract : The charge transfer (CT) complex of *p*-phenylenediamine and *p*-chloranil was prepared in acetonitrile by solution growth, diffusion and microwave methods. The CT complex was characterized by its elemental analysis. IR and ¹H NMR spectral studies. The data obtained indicated the formation of 1 : 1 CT complex between the compounds. The d.c. electrical conductivity of the CT complex showed the semiconductor behaviour of complex at room temperature.

Keywords : Charge transfer complex, d.c. electrical conductivity, *p*-phenylenediamine, *p*-chloranil.

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Secondary amines : Synthesis and effect of length of spacer linking two phenyl rings on biological activity

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Abstract : *N*-Benzyl benzylamines (1a-11a) and *N*-benzyl anilines (1b-11b) were synthesized by sodium borohydride reduction of aldimines of benzylamine and aniline respectively. The products were characterized on the basis of elemental analysis and spectral studies and screened for antifungal potential against four fungi and evaluated for nematocidal activity against two nematodes. The former compounds were found to be more effective as compared to the latter thus indicating an enhancement of biological activity due to introduction of an extra methylene group between two phenyl rings of aromatic secondary amines.

Keywords : *N*-Benzyl benzylamines, *N*-benzyl anilines, antifungal potential, nematocidal activity.

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A highly efficient synthesis of 3*H*-1,5-benzodiazepine derivatives using lanthanum(III) nitrate hexahydrate as a catalyst

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Abstract : Sulphonation of 5-(2-ethoxyphenyl)-1-methyl-3-propyl-1,6-dihydro-7*H*-pyrazolo[4,3-*d*]pyrimidin-7-one (1) with chlorosulphonic acid affords 5-[(5-chlorosulphonyl-2-ethoxy)pyrimidin]-1-methyl-3-propyl-1,6-dihydro-7*H*-pyrazolo[4,3-*d*]pyrimidin-7-one (2). Compound (2) condensed with different *b*-diketones/*b*-ketoesters (3a-e) to obtain new *b*-diketones/*b*-ketoesters (4a-e). These *b*-diketones/*b*-ketoesters were condensed with *o*-phenylenediamine (*o*-PDA) in presence of lanthanum(III) nitrate hexahydrate to afford biologically active 3*H*-1,5-benzodiazepines (5a-e) in excellent yield. The compounds (5a-e) have been screened for antimicrobial, antifungal and anthelmintic activities.

Keywords : Lanthanum(III) nitrate hexahydrate, 3*H*-1,5-benzodiazepines, *b*-diketones, *b*-ketoesters, pyrazolo[4,3-*d*]pyrimidin-7-one.

Synthesis, characterization and antimicrobial study of substituted bis-[1,2,4,5]-dithiadiazine and bis-[1,3,4]-thiadiazolidine derivatives

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Abstract : Series of compounds [4-(3-phenylimino-6-aryl/alkylimino-[1,2,4,5]-dithiadiazinane-4-carbonyl)-phenyl]-(3-phenylimino-6-aryl/alkylimino-[1,2,4,5]-thiadiazinan-4-yl)-methanones and [4-(2-phenylimino-5-aryl/alkylimino-[1,3,4]-thiadiazolidine-3-carbonyl)-phenyl]-(2-phenylimino-5-aryl/alkylimino-[1,3,4]-thiadiazolidin-3-yl)-methanones have been synthesized by the interaction of di-(*N*-aryl/alkyl thiocarbamido)terephthalamides with *N*-phenyl-*S*-chloro isothiocarbamoyl chloride and *N*-phenyl isocyanodichloride respectively. These compounds on acetylation afforded bis-acetyl derivatives. These compounds have been assayed for their antimicrobial activity against gram-positive as well as gram-negative microorganisms.

Keywords : Bis-dithiadiazine, bis-thiadiazolidine, synthesis, antimicrobial activity.

New coumarin from *Skimmia anquetelia*

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Abstract : A new coumarin isolated from leaves of *Skimmia anquetelia* has been identified as 6-methoxy-7-(2*c*-hydroxy-3*c*-methyl-butyl) coumarin.

Keywords : *Skimmia anquetelia*, coumarin.

Schmidt reaction of *trans*-3-benzylflavanones : Formation of 3-benzyl-2-phenyl-2,3-dihydro-1,4-benzoxazepin-5(4*H*)ones having both *cis* and *trans* configurations

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Abstract : On treatment with $\text{NaN}_3/\text{c.H}_2\text{SO}_4\text{-HOAc}$, *trans*-3-benzylflavanones yield ring expansion products with alkyl migration and having either *cis* or both *cis* and *trans* configurations.
Keywords : *trans*-3-Benzylflavanones, Schmidt reaction, *cis* and *trans*-3-benzyl-2-phenyl-2,3-dihydro-1,4-benzoxazepin-5(4H)ones.

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Barleriaquinone-I from the heartwood of *Tectona grandis* Linn.

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Abstract : Barleriaquinone-I, a cytotoxic anthraquinone, has been isolated from the heartwood of *Tectona grandis* Linn., along with previously reported lapachol, tecomaquinone-I, deoxylapachol, tectoquinone, stigmaterol and dehydro- α -lapachone. This is the first report on the isolation of barleriaquinone-I from this plant as well as from the family Verbenaceae. A detailed spectral data of the compound together with its HMBC correlations are reported.
Keywords : *Tectona grandis* Linn., Verbenaceae, naphthoquinones, naphthoquinone dimers, anthraquinones, barleriaquinone-I.

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Extraction studies of Pb^{II} from salicylate media using neutral organophosphorous extractant, Cyanex-923 in toluene

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Abstract : The neutral extractant, Cyanex-923 has been used for the extraction of Pb^{II} from sodium salicylate media. This metal ion was found to be quantitatively extracted with Cyanex-923 in toluene in the pH range 6-7 and from the organic phase it is stripped with 1.5 M HCl solution. The effect of pH, sodium salicylate concentration, reagent concentration, equilibration period, diluents, diverse ions and stripping agent on the extraction of Pb^{II} has been studied.
Keywords : Cyanex-923, extraction, sodium salicylate, Pb^{II} .