

Polymetallic complexes. Part-LXXXXIV : Hexadentate O O N–N O O donor azodye tetrameric complexes of Co^{II}, Ni^{II}, Cu^{II}, Zn^{II}, Cd^{II} and Hg^{II}

Bipin B. Mahapatra* and Ashish K. Sarangi

P. G. Department of Chemistry, G. M. College, Sambalpur-768 004, Orissa, India

E-mail : ashishsbp_2008@yahoo.com

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Abstract : Twelve tetrameric complexes of Co^{II}, Ni^{II}, Cu^{II}, Zn^{II}, Cd^{II} and Hg^{II} with two hexadentate (bis-tridentate) O O N–N O O donor azodye ligands 4,4'-bis-(2'-hydroxy-3'-carboxy-5'-chlorophenylazo)-diphenyl (LH₄) and 4,4'-bis-(2'-hydroxy-3'-carboxy-5'-chlorophenylazo)-diphenylmethane (L(H₄)) have been synthesized. The complexes have been characterised by analytical, conductance, magnetic susceptibility, IR, electronic spectra, ESR, NMR and X-ray diffraction (powder pattern) data. Antifungal and antibacterial activity of the ligands and the complexes were also established. 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 to zinc(II), cadmium(II) and mercury(II) complexes.

Keywords : Polymetallic complexes, azodye complexes.

Synthesis and characterization of oxothiomolybdenum(VI) complexes of some ONS chelating ligands by *in situ* oxo removal and sulphido insertion in the corresponding dioxomolybdenum(VI) complexes

Nikhil Ranjan Pramanik, Saktiprosad Ghosh, Tapas Kumar Raychaudhuri and Sudhanshu Sekhar Mandal*

Department of Chemistry, University College of Science, 92, Acharya Prafulla Chandra Road, Kolkata-700 009, India

E-mail : ssmandal2000@yahoo.com Fax : 91-33-23519755

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Abstract : This work reports the synthesis, characterization and electrochemical studies of several [Mo^{VI}OS]²⁺ complexes of some tridentate ONS donor ligands obtained by the condensation of salicylaldehyde/2-hydroxyacetophenone with S-benzyl and S-methyl dithiocarbazates. In this work PPh₃ and PPh₃S mixture is used as the synthetic reagent. The general strategy of synthesizing the complexes of the [MoOSL] type with the [MoOS]²⁺ core from corresponding complexes with the [MoO₂]²⁺ core by *in situ* oxoremoval-sulphide addition process. Presence of the oxothio core in all the [MoOSL] complexes is established by their reaction with KCN resulting in the formation of the CNS⁻ species. All the complexes are characterized by various spectroscopic (NMR, IR, UV-Vis) techniques and also by cyclic voltammetry. All the complexes exhibit an irreversible overall 2 electron reductive response probably by proton assisted loss of the sulphido group leading to the formation of the corresponding [MoOL] complex.

Keywords : Oxothiomolybdenum(VI) complexes, dithiocarbazate, Schiff bases, Ph₃S.

Synthesis, characteristic, catalytic and antimicrobial activities of imidazolo substituted benzylidene imines with ruthenium(II) complexes

K. P. Balasubramian^{a*}, V. V. Raju^a and V. Chinnusamy^b

^aDepartment of Chemistry, Gobi Arts & Science College, Gobichettipalayam-638 453, Tamilnadu, India

^bP. G. Department of Chemistry, Sri Ramakrishna Mission Vidyalaya College of Arts & Science, Coimbatore-641 020, Tamilnadu, India

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Abstract : The synthesis and characterization of several hexa-coordinated Ru^{II} complexes of the type [RuCl(CO)(EPh₃)₂(L)] (E = P or As; L = monobasic bidentate anion) are reported. IR, electronic, ¹H NMR, ³¹P NMR and catalytic activity of the complexes are discussed. An octahedral geometry has been tentatively proposed for all these complexes. The new complexes exhibit catalytic activity for the addition of benzyl alcohol and cyclohexanol in the presence of *N*-methylmorpholine-*N*-oxide as co-oxidant. The new complexes were also exhibited antimicrobial investigations.

Keywords : Ruthenium(II) complex, benzylidene imine, synthesis, imidazolo

Oxidation of 4-chlorobenzyl alcohol by polymer supported chromic acid –Kinetic and mechanistic study

S. A. Kakade, A. S. Varale, V. Y. Sonawane and N. P. Hilage*

Department of Chemistry, Shivaji University, Kolhapur-415 004, Maharashtra, India

E-mail : shital-kakade09@yahoo.com

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Abstract : The kinetics of oxidation of 4-chlorobenzyl alcohol with polymer supported chromic acid in 1 : 4 dioxane has been studied. The reaction is found to be zero order each in [alcohol] and [oxidant]. The effect of substituent on the rate of oxidation and the % of cross linking in polymeric resin (oxidant) are studied. Thermodynamic parameters are evaluated. The reaction product has been isolated and characterized by its derivative. A probable mechanism of oxidation is proposed.

Keywords : Kinetic, oxidation, polymer supported chromic acid, 4-chlorobenzyl alcohol, Ambersep 900 (O H).

Cetyl pyridinium chloride catalysed reaction of acridine yellow with acidic chloramine-T

Brijesh Pare*, Manisha Ayachit and S. B. Jonnalagadda^a

Post Graduate Department of Chemistry, Madhav Science College (Vikram University),
Ujjain-456 010, Madhya Pradesh, India

E-mail : brijesh_pare@hotmail.com

^aUniversity of KwaZulu-Natal, Durban, South Africa

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Abstract : The kinetics and mechanism of the uncatalyzed and cetyl pyridinium chloride (CPC) catalyzed oxidation of acridine yellow (3,6-diamino-2,7-dimethyl acridine hydrochloride) dye by chloramine-T in acidic media has been studied spectrophotometrically. With excess concentrations of other reactants, the reaction rate follows pseudo-first order kinetics with respect to acridine yellow. The uncatalyzed reaction has fractional order dependence on chloramine-T as well as on H⁺ concentration. The catalyzed reaction follows fractional order kinetics in [CPC]. The cetyl pyridinium chloride, a cationic surfactant, catalysed the reaction even before its critical micelle concentration. This pre-micellar kinetics has been rationalized in the light of Piszkievicz positive co-operativity model. The positive co-operativity index $n = 1.95$ has also been computed. The spectral shifts are the evidences of dye-surfactant and dye-surfactant-oxidant interactions. Variations of ionic strength has no influence on the reaction rate suggesting that neutral charged species are involved in the rate-determining step. Increase in the addition of *p*-toluenesulfonamide resulted in decrease in rate constant values. The basic stoichiometric equation is as follows : $AY^+ + ArSO_2NHCl + H_2O = P^+ + ArSO_2NH_2 + CH_3CH_2OH + CH_3CN + 2HCl$, where $P^+ = 7$ -aminoquinoline-2,3-dicarboxylic acid. On the basis of the product analysis pertinent mechanisms are proposed.

Keywords : Chloramine-T, acridine yellow, cetyl pyridinium chloride.

Ultrasonic velocity and molecular interaction study of biphenol derivatives at 30 °C

D. D. Madhvi, F. D. Karia and P. H. Parsania*

Department of Chemistry, Saurashtra University, Rajkot-360 005, Gujarat, India

E-mail : phparsania@aol.com, phparsania22@gmail.com

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Abstract : The density (ρ), viscosity (η) and sound velocity (U) (2 MHz) of pure solvents : 1,4-dioxane, methyl ethyl ketone (MEK) and *N,N*-dimethyl formamide (DMF) and solutions of 4,4'-biphenyl diacetate (BPDA) and 4,4'-dihydroxy-3,3'-diethanone biphenyl (DHDEBP) were determined at 30 °C. Various acoustical parameters namely specific acoustical impedance (Z), isentropic compressibility (k_s), Van der Waals constant (b), intermolecular free path length (L_f), internal pressure (p), free volume (V_f) and solvation number (S_n) were derived from ρ and U data and correlated with concentration (C). A fairly good to excellent correlation is observed between a specified parameter and C . Linear increase of U , Z , b and V_f and linear decrease of k_s , L_f and p confirmed presence of strong molecular interactions in the solutions. Both BPDA and DHDEBP possess structure forming tendency as confirmed by positive values of S_n . Powerful solute-solute interaction is observed above 0.75 mol/L.

Keywords : Ultrasonic velocity, acoustical parameters, solvation number, molecular interactions.

Use of mustard cake as adsorbent for adsorption of patent-blue dye from aqueous solution and its desorption studies

Rajeev Jain^{a*}, S. Malathi^b and Deepak Pathak^c

^aSchool of Studies in Chemistry, Jiwaji University, Gwalior-474 002, Madhya Pradesh, India

E-mail : rajeevjain54@yahoo.co.in *Fax* : 91-751-2346209

^bGovt. Kamla Raja Girls Autonomous P.G. College, Lashkar, Gwalior, Madhya Pradesh, India

^cS. L. P. Govt. P.G. College, Morar, Gwalior, Madhya Pradesh, India

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Abstract : The adsorption of patent-blue on de-oiled mustard was investigated to assess the possible use of this adsorbent for the processing of dye industry waste water. The efficiency of de-oiled mustard in the dye colour sorption was compared with activated carbon i.e. charcoal. The influence of various factors such as adsorbent dose, contact time, pH, adsorbate concentration, particle size, temperature on the adsorption capacity has been studied by batch experiments. The adsorption studies revealed that the ongoing adsorption validates both Langmuir and Freundlich adsorption isotherms at temperatures 40, 50 and 60 °C. Thermodynamic parameters such as $ΔG^{\circ}$, $ΔH^{\circ}$ and $ΔS^{\circ}$ for the adsorption process were calculated. Desorption profile revealed that a significant portion of the dye (92%) was recovered by using 1.0M NaOH as eluting agent.

Keywords : Adsorption, de-oiled mustard (DOM), patent-blue, desorption.

Kinetics and mechanism of uncatalyzed and silver(I) catalyzed oxidation of lysine by cerium(IV) in acid perchlorate medium

M. B. Yadav^a, Vijay Devra^b and Ashu Rani^{c*}

^aP. G. Department of Chemistry, Govt. College Kota, Kota-324 001, Rajasthan, India

^bP. G. Department of Chemistry, Govt. J. D. B. Girls College, Kota-324 001, Rajasthan, India

^cDepartment of Pure and Applied Chemistry, Kota University, Kota-324 005, Rajasthan, India

E-mail : ashu.uok@gmail.com

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Abstract : The kinetics of the uncatalyzed and silver(I) catalyzed oxidation of lysine with cerium(IV) has been studied in perchloric acid medium. The reaction is second order, that is first order with respect to each reactant. The mode of electron transfer has been indicated through an adduct between Ag^I and lysine via oxygen atom of the carboxyl group rather than the amino group. Uncatalyzed reaction simultaneously occurs with the silver(I) catalyzed reaction and the following rate law conforms to all experimental data observed in the reaction,

$$k = \frac{k_1[H^+]}{K_h + [H^+]} + \frac{k_2[Ag^I][H^+]}{K_h + [H^+]}$$

where, k is an observed second order rate constant. A reaction mechanism accounting for all these experimental observations has been suggested.

Keywords : Lysine, cerium(IV), silver(I), perchloric acid.

Engineering and physico-chemical properties of soils of deltaic region of West Bengal. Part-II : Physico-chemical and agricultural properties of soils and attempts for correlation with engineering properties

Late Sabari Biswas^a, Surajit Ghoshal^{a,b}, S. S. Dedalal^b and S. C. Lahiri^{a,c*}

^aDepartment of Chemistry, Kalyani University, Kalyani-741 235, West Bengal, India

^bSoil Mechanis Section, River Research Institute, West Bengal, P.O. HRRI, Nadia, West Bengal, India

^cEmeritues Fellow, AICTE, Kalyani Govt. Engineering College, Kalyani-741 235, West Bengal, India

E-mail : sujitclahiri@yahoo.com

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Abstract : Index and engineering properties (like moisture content, liquid limit, plastic limit, plasticity etc.) of soils of deltaic region of West Bengal were studied and reported in Part-I of this paper. In Part-II of this paper, the physico-chemical properties of soils like moisture content pH, EC, exchangeable cations (K^+ , Na^+ , Ca^{2+} , Mg^{2+}) and cations under stress conditions were determined. The results were utilized to determine SAR (sodium absorption ratio), K_c (Gapon constant), PBC^K (potential buffering capacity of K) and K-status of soils. The parameters are important for the proper management of soils in agronomy.

Exchangeable Na^+ ions appears to be considerable and may affect K_c but it is not usually taken into consideration. Attempts to correlate the engineering property of soils (like particle size distribution) with concentrations of exchangeable ions are not successful.

Keywords : Correlation, cations under stress conditions, engineering properties, exchangeable cations, K_c (Gapon constant), K-status, PBC^K , physico-chemical properties, SAR (sodium absorption ratio).

Synthesis and antimicrobial activity of pyridopyrazole and pyrazolo[3,4-d]dihydrothiazole

M. B. Deshmukh*, S. A. Deshmukh, S. S. Jagtap, A. W. Suryavanshi, S. D. Jadhav and P. V. Anbhule

Department of Chemistry, Shivaji University, Kolhapur-416 004, Maharashtra, India

E-mail : m_deshmukhl@rediffmail.com

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Abstract : 6-Methyl-1,3-dihydro-4H,7H-pyrazolo[2,3-c]pyridine-4-one (4) were synthesized by microwave as well as conventional method. The reaction time reduced 10 times than that of conventional method. The pyrazolo[3,4-d]dihydrothiazole (8) were synthesized by reacting phenylisothiocyanate with 1-carbomethoxy-5-amino-pyrazol-4-ene-3-one and were screened for their antimicrobial activities.

Keywords : Microwave assisted synthesis, pyrazolo[2,3-c]pyridine-4-one, pyrazolo[3,4-d]dihydrothiazole, anti-microbial activity, amino pyrazole.

Synthesis and antimicrobial activities of novel formazans incorporating pyrimidine ring

N. M. Goudgaon* and CH Upendar Reddy

Department of Studies and Research in Chemistry, Gulbarga University, Gulbarga-585 106, Karnataka, India

E-mail : naganna_g@yahoo.com

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Abstract : Reaction of 5-substituted-4(3H)-one-2-mercaptopyrimidines 1a and 1b with hydrazine hydrate furnishes 5-substituted-4(3H)-one-2-hydrazinopyrimidines 2a and 2b. Condensation of compounds 2a and 2b with salicylaldehyde and anisaldehyde yields 2-hydroxybenzaldehyde-(5-substituted-4(3H)-one)-2- π -pyrimidazolylhydrazones) 3a and 3b and 4-methoxybenzaldehyde-(5-substituted-4(3H)-one)-2- π -pyrimidazolylhydrazones) 3c and 3d respectively. Compounds 3a-d were reacted with diazonium salts of an appropriate primary amine in presence of base furnished the target formazans 4a-l. All the synthesized compounds were evaluated for antimicrobial activities.

Keywords : Pyrimidine analogs, formazans, antimicrobial agents.

Synthesis and structural characterization of Schiff base complexes of Pd^{II}, Rh^{III} and Ru^{III}

B. H. Mehta* and J. A. Shaikh

Department of Chemistry, University of Mumbai, Vidyanagari, Santacruz (E), Mumbai-400 098, India

E-mail : bipin_281050@yahoo.com

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Abstract : A bidentate ligand, 2-hydroxy-1-naphthalidene-4-nitroaniline (HNNA) was synthesized from 2-hydroxy-1-naphthaldehyde and 4-nitroaniline. Its metal complexes of general formula M(HNNA)₂ where M = Pd^{II} and [M(HNNA)₂.X.H₂O].2H₂O where M = Rh^{III} and Ru^{III} and X = Cl have been prepared. All the complexes were characterized using various physico-chemical methods such as elemental analysis, thermal analysis and spectral (IR, electronic and NMR) analysis. The Rh^{III} and Ru^{III} complexes have octahedral structures while Pd^{II} complex has square planar geometry.

Keywords : Schiff base, 2-hydroxy-1-naphthaldehyde, 4-nitroaniline, Pd^{II}, Rh^{III} and Ru^{III} complexes.

Halocobaloximes containing axially coordinated imidazole or histidine : Microwave assisted synthesis, characterization and antibacterial activity

A. Dayalan^{a*}, P. Meera^a, K. Balaraju^b, P. Agastian^b and S. Ignasimuthu^c

^aDepartment of Chemistry, ^bDepartment of Plant Biology and Biotechnology, ^cEntomology Research Institute, Loyola College (Autonomous), Chennai-600 034, India

E-mail : dayalan77@gmail.com Fax : 91-44-28175566

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Abstract : The complexes of the type *trans*-[Co(dmgH)₂(L)X] where dmgH⁻ = dimethylglyoximate, L = imidazole (Imi) or histidine (His) and X = Cl⁻, Br⁻ or I⁻ were synthesized and characterized by elemental analysis, TG/DTA, FABMS, UV-Visible, IR and ¹H NMR spectra. The FABMS analysis confirmed the expected molecular weight for the complexes. The thermal analysis of the complexes reveals that the bromo complexes dissociate at higher temperatures compared to the chloro complexes, the iodo cobaloximes being unstable even at low temperature decomposing without any sharp change in mass. The free ligands viz. dmgH₂, Imi and His showed no antibacterial activity against the bacteria tested but, cobaloximes were found to be active against most of the microbes. Iodocobaloximes were found to be more active than the corresponding chloro- and bromo-cobaloximes with the antibacterial activity order for the axial halides as I⁻ > Cl⁻ > Br⁻ and that of the axial nitrogen heterocycles as histidine > imidazole.

Keywords : Cobalt(III) complexes, cobaloximes, imidazole, histidine.

Synthesis, characterization and reactivity of dichloro bis(4-methoxy-pheno)tin(IV)

S. C. Chaudhry*, Kumari Bandna, S. S. Bhatt and Neeraj Sharma

Department of Chemistry, Himachal Pradesh University, Summer Hill, Shimla-171 005, Himachal Pradesh, India

E-mail : scchaudhry@sancharnet.in

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Abstract : The dichloro bis(4-methoxypheno)tin(IV) complex of composition SnCl₂(OC₆H₄OMe-4)₂ has been synthesized in quantitative yield by the reaction of tin tetrachloride with bimolar amount of 4-methoxyphenol in benzene under reflux and characterized by elemental analyses, molar conductance measurement, molecular weight determination, IR, ¹H NMR and FAB-mass spectral techniques. The thermal behaviour of the complex has been studied by TG-DTA techniques. The reactions of parent complex with nitrogenous bases viz. diethylamine, triethylamine, imidazole, benzimidazole (L), 2,2'-bipyridyl and 1,10-phenanthroline (B) yielded addition compounds of composition SnCl₂(OC₆H₄OMe-4)₂.L and SnCl₂(OC₆H₄OMe-4)₂.B characterized by physicochemical and IR spectral data.

Keywords : Tin(IV) complex, 4-methoxyphenol, spectroscopic studies, nitrogenous bases, addition compounds, FAB-mass.

Volumetric and viscometric studies on 1,4-dioxane and methanol binary mixtures at different temperatures

A. G. Peshwe^{a*} and B. R. Arbad^b

^aP. G. Department of Chemistry, Netaji Subhashchandra Bose College, Nanded-431 602, Maharashtra, India

E-mail : ajitpeshwe@gmail.com

^bDepartment of Chemistry, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad-431 004, Maharashtra, India

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Abstract : Densities and viscosities of binary liquid mixtures of 1,4-dioxane with polar solvent viz. methanol have been measured at 303.15, 308.15, 313.15 and 318.15 K. From the density and viscosity data, the values of various properties viz. excess molar volume (V^E), excess viscosity (η^E) and excess Gibb's free energy of activation of flow (DG^E) have been determined. Further the viscosities of binary mixtures have been correlated to various viscosity models.

On the basis of the values of interaction parameters of these viscosity models and also on the basis of the values of various excess properties, the nature of molecular interactions between the components of mixtures have been explained.

Keywords : Viscometric studies, mixed solvent, binary mixture (1,4-dioxane and methanol), viscosity models (equations).

Synthesis of new *N*-lactosylated isodithiobiurets and dithiazolidines (hydrobromide)

Poonam T. Agrawal and Shirish P. Deshmukh*

P. G. Department of Chemistry, Shri Shivaji College, Akola-444 001, Maharashtra, India

E-mail : poonam.agrawal2008@rediffmail.com

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Abstract : A series of new 1-hepta-*O*-benzoyl- β -D-lactosyl-5-aryl-2-*S*-benzyl-2,4-isodithiobiurets and 3-hepta-*O*-benzoyl- β -D-lactosylimino-5-arylimino-1,2,4-dithiazolidines (hydrobromide) have been synthesized by the interaction of hepta-*O*-benzoyl- β -D-lactosyl isothiocyanate with 1-aryl-*S*-benzyl isothiocarbamides followed by oxidative cyclisation of 1-hepta-*O*-benzoyl- β -D-lactosyl-5-aryl-2-*S*-benzyl-2,4-isodithiobiuret. The identities of these new *N*-lactosides have been established on the basis of usual chemical transformations and IR, NMR and Mass spectral studies.

Keywords : Lactosyl isothiocyanate, 1-aryl-*S*-benzyl isothiocarbamides, isodithiobiurets, dithiazolidines.

Kinetics of oxidation of nicotinic and isonicotinic acids by permanganate in acidic medium

Ashok Sharma^a, Anil K. Sharma and K. S. Gupta*

Department of Chemistry, University of Rajasthan, Jaipur-302 004, Rajasthan, India

E-mail : guptaks14@rediffmail.com

^aGovt. College, Dausa, Rajasthan, India

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Abstract : The most important feature of this study of the oxidation of nicotinic acid (Nic) and isonicotinic acid (Inic) by permanganate in acidic medium is the complete solution of the rate laws (A) for Nic and (B) for Inic by varying $[H^+]$ over an extended concentration range.

$$-d[MnO_4^-]/dt = \{k_0 K_1 [H^+] + k_1 K_1 K_2 [H^+]^2 + k_3 K_1 K_2 K_3 [H^+]^3\} [MnO_4^-]_t [Nic]_0 / \{1 + K_1 [H^+] + K_1 K_2 [H^+]^2\} \quad (A)$$

$$-d[MnO_4^-]/dt = \{k_1 K_2 [H^+] + k_3 K_2 K_3 [H^+]^2\} [MnO_4^-]_t [Inic] / \{1 + K_2 [H^+]\} \quad (B)$$

The relationships of the rate parameters and their values are as follows : K_1 ($NC_5H_4COO^- + H^+ \rightleftharpoons HN^+C_5H_4COO^- [B]$), 1.78×10^4 (30°) (Nic); K_2 ($HN^+C_5H_4COO^- [B] + H^+ \rightleftharpoons HN^+C_5H_4COOH [C]$), 46.2 (30°) (Nic), 33.6 (40°) (Inic); k_0 ($[B] + MnO_4^- \rightarrow$ products), $1.56 \times 10^{-3} L mol^{-1} s^{-1}$ (30°) (Nic); k_1 ($[C] + MnO_4^- \rightarrow$ products), 1.64×10^{-2} (30°) (Nic), $1.68 \times 10^{-3} L mol^{-1} s^{-1}$ (40°) (Inic) and $k_3 K_3$ ($[C] + HMnO_4 \rightarrow$ products), 2.02×10^{-2} (30°) (Nic), $3.2 \times 10^{-3} L^2 mol^{-2} s^{-1}$ (40°) (Inic). The reactivity of Nic was greater than that of Inic.

Keywords : Kinetics, oxidation, hydrogen ion dependence, permanganate, nicotinic acid, isonicotinic acid, N-protonation.

Gangetial, a new pterocarpan from the roots of *Desmodium gangeticum*

Medapati Vijaya Varaprasad^a, Kedike Balakrishna^b, Ethirajan Sukumar^c and Amarendra Patra^{d*}

^aSree Vidyaniketan College of Pharmacy, A. Rangampet, Via Tirupathi-517 102, Andhra Pradesh, India

^bCentral Research Institute for Siddha (CCRAS), Arumbakkam, Chennai-600 106, India

^cDepartment of Applied Sciences, Higher College of Technology, P.B. 74, P.C. 133, Muscat, Sultanate of Oman

^dDepartment of Chemistry, University College of Science, 92, Acharya Prafulla Chandra Road, Kolkata-700 009, India

E-mail : amarendra@sify.com

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Abstract : A new pterocarpan gangetial (1) has been isolated from the chloroform extract of the roots of the plant *Desmodium gangeticum* together with gangetinin. The structure of the new compound was elucidated as (5bR,10bR)-5b,10b-dihydro-8-hydroxy-5-methoxy-2,2-dimethyl-2H,11H-benzo[4,5]furo[2,3-d]pyrano[3,2-g]chromene-7-carbaldehyde based on the chemical and spectroscopic data.

Keywords : *Desmodium gangeticum*, Leguminosae, pterocarpan, gangetial, (5bR,10bR)-5b,10b-dihydro-8-hydroxy-5-methoxy-2,2-dimethyl-2H,11H-benzo[4,5]furo[2,3-d]pyrano[3,2-g]chromene-7-carbaldehyde.