

## Studies of some electron transfer reactions of bis(dihydrogentellurato)-argentate(III): A kinetic and mechanistic approach

Kalyan K. Sen Gupta<sup>a</sup> and Biswajit Pal<sup>b\*</sup>

<sup>a</sup>Department of Chemistry, Jadavpur University, Kolkata-700 032, India

<sup>b</sup>Department of Chemistry, St. Paul's C. M. College, 33/1, Raja Rammohan Roy Sarani, Kolkata-700 009, India

E-mail : palbiswajit@yahoo.com

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**Abstract :** The review by Lancashire<sup>1</sup> on silver is devoted to a small section on kinetics and mechanistic aspects. Since then a great deal of work has been done on kinetics and mechanism of oxidation of different reducing substrates by silver(III) complexes. The present review deals with studies on some electron transfer reactions involving bis(dihydrogentellurato)argentate(III) and 37 reducing organic compounds in alkaline medium which focuses on investigations during the period 1992-2006. The reactions have been carried out under different experimental conditions. Kinetic results and activation parameters of different redox processes have been incorporated and analyzed. The reduction of silver(III) may occur by either one electron or one step two electron transfer process. Although the oxidations appear to proceed through outer-sphere electron transfer process in the rate determining step, the reduced species of silver(II) ultimately decomposes to give black silver compounds. Attempts have been made to compare the results obtained earlier with the oxidations of similar substrates by bis(dihydrogentellurato)cuprate(III).

**Keywords :** Electron transfer, kinetics and mechanism, bis(dihydrogentellurato)argentate(III), reducing substrates.

## Synthesis and characterization of transition metal complexes of a 16-membered (N<sub>4</sub>) macrocycle with tetrapeptide features

A. K. Panda\*, A. Panda, S. Sutar<sup>a</sup>, P. Mishra, S. Pradhan, S. Ghosh and S. Pany

School of Chemistry, Sambalpur University, Jyoti Vihar-768 019, Orissa, India

<sup>a</sup>Government Autonomous College, Rourkela-769 004, Orissa, India

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**Abstract :** A series of macrocyclic complexes of the titled ligand, [16]1,5,6,8,9,13,14,16-octaza-2,4,10,12-tetraoxo-cyclo decahexane (OTCH) with cobalt(II), nickel(II) and copper(II) have been prepared by the action of formaldehyde on the respective bis(1,5-diamino-1,5-diaza-2,4-dioxopentane) metal complexes. The coordination template effect governs the steric course of the reaction. The structure of the complexes has been elucidated on the basis of IR, electronic and ESR spectral data, magnetic properties and electrochemical studies. All the complexes except copper(II) give two well defined reduction steps corresponding to the formation of a species where the metal can be formally assigned to an oxidation state of +1 and zero. Copper(II) complexes show one reduction step corresponding to zero oxidation state.

**Keywords :** Tetrapeptide, macrocycle, complex, synthesis.

## Synthesis, spectral, thermal and antibacterial studies of copper(II) complexes of a Schiff base derived from 2,3-dimethyl-1-phenyl-4-aminopyrazol-5-one

M. L. Hari Kumaran Nair\* and L. Shamlala

Department of Chemistry, University College, Thiruvananthapuram-695 034, Kerala, India

E-mail : drmlhnair@gmail.com

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**Abstract :** The Schiff base 2,3-dimethyl-1-phenyl-4-(2,5-dihydroxyacetophenone)-5-pyrazolone (DHAAP) was prepared by reacting 4-aminoantipyrine with 2,5-dihydroxy acetophenone and a series of metal complexes with this ligand were synthesized by reaction with copper(II) having the compositions  $[\text{Cu}(\text{DHAAP})_2(\text{Cl})_2]$ ,  $[\text{Cu}(\text{DHAAP})_2(\text{NO}_3)_2]$ ,  $[\text{Cu}(\text{DHAAP})_2(\text{ClO}_4)_2]$ ,  $[\text{Cu}(\text{DHAAP})_2(\text{CH}_3\text{COO})_2]$  and  $[\text{Cu}(\text{DHAAP})_2(\text{NCS})(\text{Cl})]$  (where DHAAP = dihydroxy-acetophenoneazoantipyrine). The complexes were characterized on the basis of elemental analyses, molar conductance, magnetic susceptibility data, UV-Visible, IR and ESR spectral studies. The thermal behaviour of the complex,  $[\text{Cu}(\text{DHAAP})_2(\text{NO}_3)_2]$  was investigated by thermogravimetric technique. X-Ray diffraction studies of the complex,  $[\text{Cu}(\text{DHAAP})_2(\text{Cl})_2]$  indicated orthorhombic crystal lattice with  $a = 13.6118 \text{ \AA}$ ,  $b = 5.2759 \text{ \AA}$  and  $c = 3.2758 \text{ \AA}$ . The ligand exhibits a neutral bidentate behaviour in all the complexes, coordinating to the metal ion through carbonyl oxygen and azomethine nitrogen. The X-band ESR spectrum of the complex,  $[\text{Cu}(\text{DHAAP})_2(\text{CH}_3\text{COO})_2]$  shows an axial ligand field symmetry. The ligand and the complex  $[\text{Cu}(\text{DHAAP})_2(\text{NCS})(\text{Cl})]$  were screened for their antibacterial activities against gram positive and gram negative bacteria.

**Keywords :** Copper(II), Schiff base, thermogravimetry.

## Medium effect on deprotonation equilibrium of HA-type acid-indicators in aqueous $\text{NaNO}_3$ solutions – a model aquo-ionic system

Kalyan Pandey, Amitava Bhattacharya, Swapan Bhattacharya\* and M. B. Saha\*

Department of Chemistry, Jadavpur University, Kolkata-700 032, India

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**Abstract :** The saturated solubility and the indicator constant of four newly synthesized HA-type azo-dye acid indicators, have been determined in  $\text{NaNO}_3$  aquo-ionic model solvent system. The relative dissociation equilibrium of HA is found to be a function of transfer Gibb's energy of all the involved species in equilibrium for each indicator, although it is mainly dictated by that of conjugate anion  $\text{A}^-$ . The greater stability of the oxo anion  $\text{A}^-$  of 2-carboxy-2,4,4-diaminoazo benzene with respect to other indicators due to increasing interaction with the solvated  $\text{Na}^+$  ions with increased  $\text{NaNO}_3$  concentration, differentiates it from others.

**Keywords :** Acid indicators, equilibrium,  $\text{NaNO}_3$ , aquo-ionic system.

## Correlation analysis of reactivity in the oxidation of substituted benzaldehydes by morpholinium chlorochromate

Anurag Choudhary<sup>a</sup>, N. Malani<sup>a</sup>, S. Agarwal<sup>b</sup>, M. Sharma<sup>c</sup> and Vinita Sharma<sup>a\*</sup>

<sup>a</sup>Department of Chemistry, J. N. V. University, Jodhpur-342 005, Rajasthan, India

E-mail : drpkvs27@yahoo.com

<sup>b</sup>Govt. J.D.B. Girls College, Kota-342 001, Rajasthan, India

<sup>c</sup>Modi Institute of Technology, Kota-342 001, Rajasthan, India

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**Abstract :** Oxidation of thirty six monosubstituted benzaldehydes by morpholinium chlorochromate (MCC) in dimethylsulphoxide (DMSO), leads to the formation of corresponding benzoic acids. The reaction is of first order with respect to MCC. A Michaelis-Menten type kinetics is observed with respect to benzaldehydes. The reaction is promoted by hydrogen ions; the hydrogen-ion dependence has the form  $k_{obs} = a + b [H^+]$ . The oxidation of [P]benzaldehyde (PhCDO) exhibited a substantial primary kinetic isotope effect. The reaction was studied in nineteen organic solvents and the effect of solvent was analysed using Taft's and Swain's multi-parametric equations. The rates of the oxidation of *para*- and *meta*-substituted benzaldehydes showed excellent correlation in terms of Charton's triparametric LDR equation, whereas the oxidation of *ortho*-substituted benzaldehydes were correlated well with tetraparametric LDRS equation. The oxidation of *para*-substituted benzaldehydes is more susceptible to the delocalized effect than is the oxidation of *ortho*- and *meta*-substituted compounds, which display a greater dependence on the field effect. The positive value of  $\rho$  suggests the presence of an electron-deficient reaction centre in the rate-determining step. The reaction is subjected to steric acceleration by the *ortho*-substituents. A suitable mechanism has been proposed.

**Keywords :** Morpholinium chlorochromate, benzaldehyde, oxidation, correlation analysis.

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## **Oxidation of ethylene glycol by quinolinium dichromate in aqueous perchloric acid medium – A kinetic and mechanistic study**

**H. V. Rajeshwari, S. T. Nandibewoor and S. A. Chimatadar\***

P.G. Department of Studies in Chemistry, Karnatak University, Dharwad-580 003, Karnataka, India

*E-mail :* schimatadar@gmail.com

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**Abstract :** The oxidation of ethylene glycol by quinolinium dichromate has been studied spectrophotometrically in aqueous perchloric acid medium. The stoichiometry is 2 : 3 i.e., two moles of quinolinium dichromate requires three moles of ethylene glycol. The reaction is first order with respect to quinolinium dichromate and ethylene glycol concentrations. Increase in perchloric acid concentration increases the reaction rate. The order with respect to acid concentration is nearly two. Added products do not have any significant effect on the rate of reaction. Increasing ionic strength and decreasing dielectric constant of the medium increases the rate. A possible mechanism is proposed and reaction constants involved have been determined. The activation parameters were determined with respect to slow step of mechanism.

**Keywords :** Perchloric acid, ethylene glycol, quinolinium dichromate, oxidation.

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## **Density, viscosity and ultrasonic velocity studies of cardo group containing symmetric double Schiff bases solutions at 303, 308 and 313 K**

**B. J. Gangani 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, viscosity and ultrasonic velocity (2 MHz) of pure solvents e.g. chloroform, DMF, 1,4-dioxane and symmetric double Schiff bases solutions have been investigated to understand

the effect of solvents on molecular interactions at 303, 308 and 313 K. Various acoustical parameters such as  $U$  (ultrasonic velocity),  $Z$  (specific acoustical impedance),  $k_s$  (isentropic compressibility),  $R$  (Rao's molar sound function),  $b$  (Van der Waals constant),  $p$  (internal pressure),  $V_f$  (free volume),  $L_f$  (intermolecular free path length),  $(a/f^2)_{C1}$  (classical absorption coefficient) and  $t$  (viscous relaxation time) have been determined and correlated with concentration ( $C$ ). Good to excellent correlation between a given parameter and concentration is observed at all temperatures ( $T$ ) and solvent systems studied. Linear or non-linear increase or decrease of acoustical parameters with concentration and temperature indicated the existence of strong molecular interactions. The linear or non-linear increase of  $S_n$  with  $C$  and decrease with  $T$  further supported the existence of molecular interactions.

**Keywords :** Ultrasonic velocity, acoustical parameters, density, viscosity, solute-solvent and solvent-solvent interactions, Schiff bases.

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## Tandem *N*-allylation/[3,3]-sigmatropic shift route to the synthesis of allyl ethers of dihydropyrimidines

A. Rasheeth, G. Yogeswari and C. A. M. A. Huq\*

Post Graduate and Research Department of Chemistry, The New College, Chennai-600 014, India

(Affiliated to University of Madras, Tamil Nadu, India)

*E-mail* : drmdabdulhuq@yahoo.com *Fax* : 91-44-28352883

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**Abstract :** We report herein the first instance of tandem *N*-allylation/[3,3]-sigmatropic rearrangement of dihydropyrimidones leading to the facile synthesis of functionalized allyl ethers and thioallyl ethers of dihydropyrimidine wherein C=O and C=S form a part of [1,5]-hexadiene system necessary for [3,3]-sigmatropic shift.

**Keywords :** Tandem reaction, [3,3]-sigmatropic shifts, Biginelli reaction, *O*-allylation, *S*-allylation.

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## Synthesis and antimicrobial activity of thiazolidinones derived from 4-methylcoumarinyl-7-oxyacetic hydrazide

P. Manojkumar\*, T. K. Ravi and S. Gopalakrishnan

Department of Pharmaceutical Chemistry, College of Pharmacy,  
Sri Ramakrishna Institute of Paramedical Sciences, Coimbatore-641 044, Tamilnadu, India

*E-mail* : kmano1975@rediffmail.com

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**Abstract :** A series of new thiazolidin-4-ones (3a-j), 5-carboxymethyl-4-thiazolidinones (4a-l) have been synthesised via the condensation of 4-methylcoumarinyl-7-oxyacetic acid [(substituted phenyl)methylene]hydrazides (2a-l), with thioglycolic acid and thiomalic acid respectively. The compounds (2a-l) were synthesised from 4-methylcoumarinyl-7-oxyacetic hydrazide using various aromatic aldehydes. The structure of all synthesised compounds has been determined by spectral methods. The compounds were screened for their antibacterial activity against *S. aureus* and *B. subtilis* bacteria.

**Keywords :** Thiazolidin-4-ones, antibacterial activity, 4-methylcoumarinyl-7-oxyacetic hydrazide, thiomalic acid.

## A facile microwave-induced synthesis of heterocyclic compounds and a dialdehyde from a naturally occurring limonoid

Nayan Kamal Bhattacharyya<sup>a\*</sup> and J. C. S. Katakya<sup>b</sup>

<sup>a</sup>Department of Chemistry, Sikkim Manipal Institute of Technology, Majitar, Rangpo-737 132, East-Sikkim, India

*E-mail* : nkamalbhatt@yahoo.com    *Fax* : + 91-3592-246112

<sup>b</sup>Synthetic Organic Chemistry Division, Regional Research Laboratory, Jorhat-785 006, Assam, India

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**Abstract** : Paniculatin (1) (6a-acetoxiazadirone), a naturally occurring meliacin, was subjected to chemical transformations under microwave irradiation to give heterocyclic compounds and also a dialdehyde derivative. These include 2-methyloxazolo[4,5-d]-1,2,20,21,22,23-hexahydropaniculatin (4), 2-methyloxazolo[4,5-d]-6a,7a-dihydroxy-1,2,20,21,22,23-hexahydropaniculatin (5) and the dialdehyde (9) derivative of paniculatin. These reactions give excellent yields in a very short time when carried out under microwave irradiation under solvent-free condition.

**Keywords** : Microwave, Meliaceae, limonoids, paniculatin, heterocycles.

## Use of cipher and cipher-key in carbohydrate nomenclature

Amalendu Banerjee<sup>a</sup>, Tanya Das<sup>b</sup> and Rina Ghosh\*

Department of Chemistry, Jadavpur University, Kolkata-700 032, India

*E-mail* : ghoshrina@yahoo.com

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**Abstract** : The steric structures of the carbohydrates and related compounds have been concised and generalised through a proposed cipher and cipher-key to make the study easier.

**Keywords** : Carbohydrates, nomenclature and configuration, cipher and cipher-key.

## Synthesis and characterization of $\text{UO}_2^{\text{VI}}$ , $\text{ZrO}^{\text{IV}}$ and $\text{Th}^{\text{IV}}$ complexes with 3-(pyridyl/thienyl-2- $\zeta$ -methylene/furfurylidene)imino-2-thiohydantoin

D. C. Dash\*, (Mrs.) P. Naik, S. K. Naik, R. K. Mohapatra and (Ms.) S. Ghosh

P.G. Department of Chemistry, Sambalpur University, Jyoti Vihar, Burla, Sambalpur-768 019, Orissa, India

*E-mail* : dhruba\_dash@yahoo.co.in

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**Abstract** : A few complexes of the type  $[\text{ML}_2]_n \cdot n\text{H}_2\text{O}$  and  $[\text{ThL}_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$  where HL = 3-pyridyl-2- $\zeta$ -methyleneimino-2-thiohydantoin (Hpmit), 3-thienyl-2- $\zeta$ -methyleneimino-2-thiohydantoin (Htmit) and 3-furfurylideneimino-2-thiohydantoin (Hfit), M =  $\text{UO}_2^{\text{VI}}$  and  $\text{ZrO}^{\text{IV}}$ ; n = 1 or 2 have been synthesized and characterized on the basis of elemental analysis, thermal analysis, conductance, <sup>1</sup>H NMR, IR and electronic data. The results indicate that the ligands behave as monobasic tridentate coordinating

through azomethine nitrogen deprotonated enolic oxygen and ring hetero atom (N or S or O) to  $\text{UO}_2^{\text{VI}}$ ,  $\text{ZrO}^{\text{IV}}$  and  $\text{Th}^{\text{IV}}$  yielding these complexes.

Keywords : 3-Substituted (pyridyl/thienyl-2 $\epsilon$ -methylene/furfurylidene)imino-2-thiohydantoin,  $\text{UO}_2^{\text{VI}}$ ,  $\text{ZrO}^{\text{IV}}$ ,  $\text{Th}^{\text{IV}}$ .

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## Kinetics of hydrolysis of *N*-phenylbenzohydroxamic acid in acetone-water medium

Nitin Choure, P. Awadhiya and S. A. Bhoite\*

School of Studies in Chemistry, Pt. Ravishankar Shukla University, Raipur-492 010, Chhattisgarh, India

E-mail : nitin.choure1@gmail.com; sa.bhoite10@gmail.com

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Abstract : The kinetics of hydrolysis of *N*-phenylbenzohydroxamic acid have been studied in 1.8 to 8.1 mol dm<sup>-3</sup> H<sub>2</sub>SO<sub>4</sub> at 55 °C. The rate of reaction increases with increasing concentration of H<sub>2</sub>SO<sub>4</sub> nearly linearly up to 7.2 mol dm<sup>-3</sup> and then increased sharply. The rate maximum was not observed. The effect of temperature on the rate of hydrolysis has been studied. Activation parameters have been calculated. Bimolecular nature of hydrolysis has been supported by Arrhenius parameters, Bunnett-Olsen, Zucker-Hammett hypothesis etc.

Keywords : Kinetic, hydrolysis, *N*-phenylbenzohydroxamic acid.

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## Kinetics and mechanism of Ru<sup>III</sup> catalysed oxidation of 1,2,3,4-tetrahydro-naphthalene (tetralin) by Ce<sup>IV</sup> in aqueous nitric acid medium

N. Vijaya Bhaskar Rao and M. Anand Rao\*

Department of Chemistry, Osmania University, Hyderabad-500 007, India

E-mail : marao\_9@yahoo.com

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Abstract : The kinetics and mechanism of Ru<sup>III</sup> catalysed oxidation of 1,2,3,4-tetrahydronaphthalene (tetralin) by Ce<sup>IV</sup> in aqueous nitric acid to tetralone under the conditions [TL] >> [Ce<sup>IV</sup>] at different temperatures (30–50 °C) have been studied in 3.0 mol dm<sup>-3</sup> nitric acid medium. The experimentally observed rate law conforms to

$$-d[\text{Ce}^{\text{IV}}]/dt = kK[\text{Ce}^{\text{IV}}][\text{TL}][\text{Ru}^{\text{III}}]/(1 + K[\text{TL}] + K[\text{Ru}^{\text{III}}])$$

Keywords : Tetralin, Ru<sup>III</sup>, kinetics, Ce<sup>IV</sup>, oxidation, mechanism.

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## Dielectric and excess dielectric constants of binary liquid mixtures : Chlorobenzene + nitrobenzene, cyclohexanone + nitrobenzene and benzene + nitrobenzene at 303 K

Ch. V. V. Ramana and K. Malakondaiah\*

Scientific Instrumentation Laboratory, Department of Instrumentation & University Science Instrumentation Centre, Sri Krishnadevaraya University, Anantapur-515 003, Andhra Pradesh, India

**Abstract :** The nonideal behaviour of liquid mixtures are characterized by excess thermodynamic properties such as excess volumes, enthalpy of mixing, compressibilities etc. The excess dielectric constant  $\epsilon^E$  is also one such parameter that indicates the strength and nature of intermolecular interactions in binary liquid mixtures. The positive deviations from ideal behaviour ( $\epsilon^E$  being positive) are qualitatively attributed to a "build in" of components of the mixture in the structure of respective solvent. The negative deviations from ideal behaviour ( $\epsilon^E$  being negative) is explained qualitatively either due to interstitial solvation or due to breaking of aggregates. In the present study, the excess dielectric constants for the binary liquid mixtures (1) benzene + nitrobenzene, (2) chlorobenzene + nitrobenzene and (3) cyclohexanone + nitrobenzene are measured at 303 K and at different concentrations. The dielectric constants for these binary liquid mixtures are measured using a computer based system. It is based on the principle that the change in frequency of XR-2206 function generator, when the liquid forms the dielectric medium of the dielectric cell, is measured with a personal computer. The programmable interval timer 8254 available in the DIOT card is used to measure the frequency which in turn determines the capacitance of the cell and dielectric constant. The necessary software is developed in C language.

**Keywords :** Dielectric constants, excess dielectric constants.

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## Synthesis and acid catalyzed hydrolysis of di-*m*-toluidine phosphate

Pragya Awadhiya and S. A. Bhoite\*

School of Studies in Chemistry, Pt. Ravishankar Shukla University, Raipur-492 010, Chhattisgarh, India

E-mail : p\_awadhiya@rediffmail.com; sa\_bhoite@rediffmail.com

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**Abstract :** Di-*m*-toluidine phosphate was synthesized by direct phosphorylation with  $\text{POCl}_3$  of the parent compound. Kinetics of hydrolysis of di-*m*-toluidine phosphate was carried out in acid medium from 0.1 to 7.0 mol  $\text{dm}^{-3}$  in 40% dioxane-water (v/v) medium at 60 °C. Ionic strength data exhibit different contribution of neutral and conjugate acid species. Theoretical rates estimated from second empirical term of Debye-Hückel equation have been found in close agreement with experimental rates. A study of ionic strength, solvent, temperature and substrate effects have been carried out to determine the probable reaction paths. Molecularity and bond fission are also discussed in terms of Zucker-Hammett hypothesis, Bunnett and Bunnett-Olsen parameters and isokinetic relationship.

**Keywords :** Kinetics, hydrolysis, di-*m*-toluidine phosphate.

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## A facile one pot synthesis of novel $\text{N}_1$ -(2-nitrophenyl sulphenyl substituted) pyrrolidino[2,1-*c*][1,4]-benzodiazepine, thiazolidino[3,2-*c*][1,4]-benzodiazepine and thiazolidino[4,3-*c*][1,4]-benzodiazepine from the corresponding N-substituted isatoic anhydride

Sumitra Nain, Varini Gupta, Megha Arya, Reenu Sirohi and D. Kishore\*

Department of Chemistry, Banasthali University, Banasthali-304 022, Rajasthan, India

E-mail : kishoredharma@yahoo.co.in

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**Abstract :** A facile one pot synthesis of  $N_1$ -(2-nitrophenyl sulphenyl substituted) pyrrolidino[2,1-c][1,4]-benzodiazepine (6), 4-hydroxypyrrolidino[2,1-c][1,4]-benzodiazepine (7), thiazolidino[3,2-c][1,4]-benzodiazepine (8) and thiazolidino[4,3-c][1,4]-benzodiazepine (9) from the reaction of  $N_1$ -(2-nitrophenyl sulphenyl) isatoic anhydride (1) with L-proline (2), 4-hydroxy-L-proline (3), thiazolidine 2-carboxylic acid (4) and thiazolidine-3-carboxylic acid (5) respectively has been described. The formation of 6, 7, 8 and 9 from 2, 3, 4 and 5 respectively is believed to take place through the intermediate N-(2-nitrophenyl sulphenyl)-iminoketene (10).

**Keywords :**  $N_1$ -Substituted-[1,4]-benzodiazepines, pyrrolidino- and thiazolidino-[1,4]-benzodiazepines, N-(2-nitrophenyl sulphenyl)isatoic anhydride.

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## **Synthesis and antimicrobial potential of Mannich bases of 4-chloro-3-{4-(chlorobenzyloxy)-benzoylhydrazono}-indolin-2-ones**

Nisheeth Rastogi<sup>a\*</sup>, Darwin Anil Harrison<sup>a</sup>, Diwakar Tripathi<sup>b</sup> and Sarveshwar Shukla<sup>b</sup>

<sup>a</sup>Department of Chemistry, Lucknow Christian Post-Graduate College, Lucknow-226 018, Uttar Pradesh, India

*E-mail :* nisheethrastogi2003@yahoo.co.in

<sup>b</sup>Department of Chemistry, Sri Jai Narain Post-Graduate College, Lucknow-226 001, Uttar Pradesh, India

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**Abstract :** 4-Chloro-3-{4-(chlorobenzyloxy)-benzoylhydrazono}-indolin-2-ones (1-4) were synthesised by the condensation of 4-(chlorobenzyloxy)-benzoylhydrazines and 4-chloroisatin. On being subjected to aminomethylation in the presence of formaldehyde and heterocyclic secondary amines, indolinones 1-4, furnished aminomethylated indolinones (Mannich bases) 5-20. The structures of the compounds have been established by means of elemental analysis and spectral data (IR, PMR and Mass). The compounds have been screened for their antimicrobial potential against human pathogenic bacteria and fungi.

**Keywords :** Isatin, aminomethylation, Mannich bases, antimicrobial agents.

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## **Gallium(III) chloride as an efficient new catalyst for the production of Hantzsch 1,4-dihydropyridines**

Dhruva Kumar, Anil Saini, Suresh and Jagir S. Sandhu\*

Department of Chemistry, Punjabi University, Patiala-147 002, Punjab, India

*E-mail :* j\_sandhu2002@yahoo.com

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**Abstract :** Hantzsch 1,4-dihydropyridines (DHPs) are synthesized in good to excellent yields from aldehydes, 1,3-dicarbonyl compounds and ammonium acetate using gallium(III) chloride which acts as dehydrating agents and 5 mol% is enough for this protocol under solvent free conditions.

**Keywords :** Hantzsch 1,4-dihydropyridines, gallium(III) chloride, aldehydes, 1,3-dicarbonyl compound, ammonium acetate, solvent-free.