

Solution stability and thermal degradation of some cyclic dithiocarbamates

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Abstract : Although cyclic dithiocarbamates (DTC) present better solution and solid state stability when compared to aliphatic analogues, relatively few thermo-analytical and solution stability studies have so far been reported which concern these important substances. In solution, the degradation of dithiocarbamates undergoes via a protonated intermediate and is very fast in acidic media. A spectrophotometric method based on diode array detection has been proposed for a fast and accurate determination of pK_a and degradation kinetic constants of these cyclic DTC's and is discussed here.

Thermal analytical data were also collected regarding ammonium, sodium, potassium salts as well as for zinc, cadmium, lead, cobalt, nickel, copper, mercury, silver, iron and manganese complexes of pyrrolidinedithiocarbamate (pyr), piperidinedithiocarbamate (pip), morpholinedithiocarbamate (mor) and hexamethyleneiminedithiocarbamate (hex) and are summarized in this article. Differences in the decomposition intermediates permitted to conclude that structural differences make the decomposition to occur by specific pathways, depending on the number and nature of members of the aminic ring.

Keywords : Dithiocarbamates, solution stability, thermal analysis, pK_a .

Yttrium and lanthanide perchlorate complexes of 2,3-dimethyl-4-formyl-[2'-(aminomethyl)pyridine]-1-phenyl-3-pyrazoline-5-one and 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one

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Abstract : Two series of complexes of two Schiff bases, 2,3-dimethyl-4-formyl-[2'-(aminomethyl)pyridine]-1-phenyl-3-pyrazolin-5-one (L_1) and 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one (L_2), with the general compositions $[Ln(L_1)_2(ClO_4)_2](ClO_4)$ and $[Ln(L_2)_2(ClO_4)](ClO_4)_2$ (where $Ln = Y, La, Pr, Nd, Sm, Eu, Gd, Dy, Ho$ or Er) have been synthesized and characterized. Both L_1 and L_2 are neutral ligands coordinating through the carbonyl oxygen(s) and the azomethine nitrogen in these complexes. Two perchlorate ions are bidentately coordinated in the complexes of L_1 and one perchlorate ion is bidentately coordinated in the complexes of L_2 . A coordination number of eight is assigned to the metal in both series of complexes.

Keywords : Rare earth perchlorate complexes, 2,3-dimethyl-4-formyl-[2'-(aminomethyl)pyridine]-1-phenyl-3-pyrazoline-5-one, 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazoline-5-one.

Electrochemical reactions at sacrificial electrode. Part-XVIII : Synthesis of coordination compounds of zinc(II) alkoxides

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Abstract : Electrochemical oxidation of sacrificial zinc anode in alcohols containing a ligand (1,10-phenanthroline or 2,2'-bipyridyl) yields coordination compounds of zinc(II) alkoxides. The products are identified by elemental analysis and infrared spectral studies. These are polymeric in nature like their parent zinc(II) alkoxides. Current efficiencies of most of these systems are quite high.

Keywords : Zinc(II) alkoxides, synthesis, electrochemical reactions, sacrificial electrodes.

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Volumetric, compressibility and transport behaviour of some α -amino acids in water and aqueous glycerine at 303.15 K

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Abstract : Density (ρ), viscosity (η) and ultrasonic velocity (U) have been measured for L-glutamine, L-asparagine and L-lysine in water and aqueous glycerine (0, 0.5 and 1 mol dm⁻³) at 303.15 K. These measurements have been performed to evaluate some important parameters viz. adiabatic compressibility (β), molar hydration number (n_H), apparent molar compressibility (ϕ_K), apparent molar volume (ϕ_v), limiting apparent molar compressibility (ϕ_K^0), limiting apparent molar volume (ϕ_v^0) and their constants (S_K, S_v), transfer volume ($\Delta\phi_v^0$) and viscosity A and B coefficients of Jones-Dole equation. The results have been discussed in terms of solute-co-solute and ion-solvent interaction.

Keywords : Ultrasonic velocity, apparent molar compressibility, apparent molar volume, hydration number, transfer volume.

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Non-equilibrium growth patterns and oscillations during electrochemical deposition of Zn-Cu binary system in batch and flow reactors

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Abstract : Growth patterns of Zn-Cu alloys in the η and β phases have been developed and examined. Patterns become more compact and dense on increasing the copper content and field intensity. The compactness is more in the β phase than those in the η phase. Fractal dimension D was computed in each case by box counting method. Weight of aggregates at different concentrations of CuSO₄ was determined. Electrodeposited aggregates were characterized by X-ray diffraction (XRD) study. Electrodeposition of metals under the diffusion limited conditions shows spontaneous potential oscillations. Cathode potential was measured as a function of time during electrochemical deposition of Zn, Cu and Zn-Cu alloys in batch and in a Continuously Stirred Tank Reactors (CSTR). The morphology, amplitude and frequency of oscillation were found to depend on the copper content. A

transition from biperiodicity to periodic oscillation was observed when copper sulphate was introduced in ZnSO₄ solution.

Keywords : Electrochemical deposition, growth patterns, electrochemical oscillation, batch and flow reactors, Zn-Cu binary system.

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Ultrasonic investigations in ternary liquid mixtures of substituted benzenes with acetophenone at different temperatures

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Abstract : The ultrasonic velocity, density and viscosity have been measured for the ternary mixtures of substituted benzenes such as chlorobenzene, bromobenzene and nitrobenzene with acetophenone in 1-butanol at 303, 308 and 313 K. The experimental data have been used to calculate the acoustical parameters namely adiabatic compressibility (β), free length (L_f), free volume (V_f), internal pressure (π_i), acoustic impedance (Z) and molar volume (V_m). Some of the above excess parameters have been evaluated and fitted to Redlich-Kister polynomials. It is eventually observed that molecular association between acetophenone and 1-butanol is through hydrogen bonding and substituted benzenes through strong dipole-dipole interactions. The formation of donor-acceptor complexes also observed in the present study.

Keywords : Adiabatic compressibility, dipole-dipole interaction, hydrogen bonding.

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Synthesis and characterization of a novel class of molecular sieves namely organophosphate

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Abstract : A novel class of molecular sieves namely organophosphates are reported for the first time. The molecular sieves are similar to zeolites in many properties. One among them was *n*-propylamino phosphate and it is similar to ZSM-5 (MFI) zeolite. Characterization by various instrumental techniques shows interesting properties. Attempt to synthesize organosulphate results in less stable materials. Secondary and tertiary amines did not produce solid product. A plausible mechanism on its synthesis is also proposed. It is soluble in water but insoluble in most of the organic solvents.

Keywords : *n*-Propylaminophosphate, MFI, basic catalyst.

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Ruthenium(III) mediated oxidation of succinic acid by cerium(IV) in aqueous sulphuric acid medium – A kinetic and mechanistic approach

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Abstract : The oxidation of succinic acid by cerium(IV) has been studied spectrophotometrically in aqueous sulphuric acid medium at 298 K at constant ionic strength of 1.6 mol dm⁻³. A minute amount of ruthenium(III) (10⁻⁶ mol dm⁻³) is sufficient to enhance the slow reaction between succinic acid and cerium(IV). The main reaction products are cerium(III) and oxaloacetic acid. The stoichiometry is 1 : 4, that is [suc. acid] : [Ce^{IV}] = 1 : 4. The reaction is first order in both cerium(IV) and ruthenium(III) concentrations. The order with respect to suc. acid concentration varies from first order to zero order, as the succinic acid concentration increases. Increase in sulphuric acid concentration decreases the reaction rate. The added sulphate and bisulphate decreases the rate of reaction. The added product cerium(III) retards the reaction rate. The active species of oxidant and catalyst are Ce(SO₄)₂ and [Ru(H₂O)₆]³⁺ respectively. Possible mechanism is proposed. The activation parameters were determined with respect to slow step and reaction constants involved have been determined.

Keywords : Kinetics, oxidation, succinic acid, cerium(IV), ruthenium(III).

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Conversion of solar energy to chemical energy

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Abstract : The rate of evolution of hydrogen from water by photochemical process using solar energy has been investigated employing fourteen metal complexes as catalysts, ten electron relays, three electron donors and two co-catalysts in different permutation and combinations. The effect of varying reaction conditions like temperature, concentration and pH have also been investigated for the optimum production of hydrogen by the photochemical cleavage of water molecules.

Keywords : Solar energy, chemical energy, photosensitizer.

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Kinetics and mechanism of periodate oxidation of *o*-anisidine in acetone-water medium

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Abstract : Main reaction product of the periodate oxidation of *o*-anisidine (OMA) in acetone-water medium is methoxy-1,4-benzoquinone. It was isolated and characterized. The kinetics of the reaction

has been followed by monitoring the increase in the absorbance of reaction intermediate, C. Results under pseudo-first order conditions, $[o\text{-anisidine}] \gg [\text{IO}_4^-]$, are in agreement with the rate law :

$$d[\text{C}]/dt = kK_w [\text{S}]_0 [\text{IO}_4^-] [\text{H}^+] / \{K_2 K_w + (K_w + K_b K_2) [\text{H}^+] + K_b [\text{H}^+]^2\}$$

where kK is the empirical composite rate constant, K_w is ionic product of water, K_2 is acid dissociation constant of H_4IO_6^- , K_b is base dissociation constant of OMA and $[\text{S}]_0$ represents the concentration of OMA that has been taken in excess. In agreement with the rate law the $1/k_2$ versus $[\text{H}^+]$ profile passes through the minimum. Increase in ionic strength increases the rate of reaction. Free radical scavengers do not affect the reaction rate. Thermodynamic parameters evaluated are : $E_a = 3.1 \text{ kcal mol}^{-1}$, $A = 25.9 \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$, $\Delta S^\ddagger = -54.1 \text{ cal mol}^{-1}$, $\Delta G^\ddagger = 19.5 \text{ kcal mol}^{-1}$ and $\Delta H^\ddagger = 2.4 \text{ kcal mol}^{-1}$.

Keywords : Kinetics, periodate oxidation, *o*-anisidine, methoxy-1,4-benzoquinone.

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Synthesis of 3-amino-4,5-dihydro(5*H*)4-oxothieno[3,2-*c*]quinoline-2-carboxylic acids and their alkyl esters

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Abstract : 4-Hydroxyquinolin-2(*H*)ones are converted to 4-chloro-3-cyanoquinolin-2(*H*)ones and are cyclised with thioglycolic acid and its alkyl esters to furnish the title compounds viz. 3-amino-4,5-dihydro(5*H*)4-oxothieno[3,2-*c*]quinoline-2-carboxylic acids and their corresponding alkyl esters, which are characterised by elemental analysis and spectral data.

Keywords : Quinolone antibacterials, 4-chloro-3-cyanoquinolin-2(*H*)ones, oxothieno[3,2-*c*]quinolones, thioglycolic acid.

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Thermal behaviour of co-precipitated magnesium-aluminium hydroxides gel

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Abstract : Thermal behaviour of co-precipitated Mg-Al hydroxides gel towards the formation of composite powders of periclase and MgAl_2O_4 spinel at low temperature was investigated. The co-precipitated Mg-Al hydroxy compounds were synthesized at two different temperatures and at pH ranges in between 8.56 and 9.20 from chloride salts of respective ions with molar ratio for MgO : Al_2O_3 as 2 : 1 using various combinations of ammonium hydroxides, triethanol amine (TEA) and ammonium carbonate mixture as basic media for co-precipitation. The co-precipitated hydroxide gel dried at 110 °C and heated at different temperatures up to 1000 °C were characterised by DTA, TGA and XRD studies. In all cases the co-precipitated Mg-Al hydroxy phases collapsed at 400 °C with the conversion to transient amorphous phases. Further heat treatment at higher temperatures led to the formation of MgAl_2O_4 spinel and minor periclase phases at temperature as low as 600 °C. The co-precipitated Mg-Al hydroxides gel derived from TEA- NH_4OH basic media exhibited promising results

so far as transformation to composite powders of MgAl_2O_4 spinel and periclase at relatively low temperature is concerned.

Keywords : Co-precipitation, basic media, Mg-Al hydroxide gel, MgAl_2O_4 spinel.

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Dimorphosim of a dimethoxy bridged oxovanadium(V)-hydrazone complex

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Abstract : A monoclinic variety with $C2/c$ space group of the title complex $[\text{V}^{\text{VO}}(\text{L})(\text{OCH}_3)_2]$, incorporating the doubly deprotonated benzoyl hydrazone of 2-hydroxy-5-methylacetophenone (H_2L) was synthesized from the decomposition of $[\text{V}^{\text{IVO}}(\text{L})(\text{bipy})]$ (where bipy representing the 2,2'-bipyridine) in methanol which has been reported very recently from our laboratory. In this paper we report another monoclinic variety of this complex with $P2_1/n$ space group that showed some differences in bonding patterns in the solid state (but in solution they are almost identical) prepared by different synthetic method viz. from the equimolar reaction of $[\text{V}^{\text{IVO}}(\text{acac})_2]$, H_2L and imidazole in CH_3OH .

Keywords : Oxovanadium(V) complex, hydrazone complex, crystal structure, dimorphism.

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Theoretical evaluation of crystalline state properties of chalcogenide crystals with sodium chloride structure

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Abstract : Hellmann and Ali-Hasan forms of repulsive potential functions have been applied to compute the values of cohesive energy, atomization energy, force constant, IR absorption frequency, Debye temperature, Grüneisen parameter, Anderson-Grüneisen parameter and Moelwyn-Hughes parameter for 45 chalcogenide crystals of NaCl-structure. The results are reported here. Calculations are also performed for the estimation of first order volume dependence of Grüneisen parameter commonly known as second Grüneisen parameter using expressions of higher order derivatives of interaction potential within the frame-work of Dugdale and MacDonald theory. The high pressure behaviour of these crystals have also been studied.

Keywords : Crystalline state properties, Grüneisen parameter, second Grüneisen parameter.

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Hydrogen bond interaction study of methanol in non-polar solute : A theoretical dielectric approach

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Abstract : The dielectric permittivity of methanol in chlorobenzene and 1,2-dichloroethane mixtures for various concentration and temperatures have been studied using hydrogen bonded model suggested by Luzar. Luzar has proposed a more realistic hydrogen bonded model, to determine the correlation factor g_1 and g_2 from single value of the dielectric constant. The interaction of the chloro group molecules with methanol as hydrogen bonded liquid is discussed.

Keywords : Dielectric permittivity, Kirkwood correlation factor.

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Non-specific solvent effects on proton transfer equilibrium between toluic acids and carbinol base of crystal violet

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Abstract : Acid-base reactions in apolar aprotic media provide a sufficiently close measure of intrinsic acid/base strengths. A study on reaction equilibria between *o*- and *m*-methyl benzoic (toluic) acids with the carbinol base of crystal violet in five apolar aprotic solvents of similar Kirkwood polarity function $(\epsilon - 1)/(2\epsilon + 1)$ and significantly different Dimroth-Reichardt's $E_T(30)$ and Kamlet-Taft's polarity-polarisability π^* parameter indicates strongly the overall non-specific solvent effects are contributed by both electrostatic and dispersion interactions, the latter having a more dominant role therein.

Keywords : Apolar, aprotic, crystal violet, carbinol base.

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Synthesis of various pharma based pyrido[2,3-*d*]pyrimidine as well as pyrido[1,2-*a*]pyrimidine and their antimicrobial activities

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Abstract : 4-Amino-7-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-5-(substitutedphenyl)-1*H*-pyrido[2,3-*d*]pyrimidine-2-one (4a-j), 4-amino-7-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-5-(substitutedphenyl)-1*H*-pyrido[2,3-*d*]pyrimidine-2-thione (5a-j), 4-thioureido-7-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-5-(substitutedphenyl)-1*H*-pyrido[2,3-*d*]pyrimidine-2-thione (6a-

j), 2-methyl-7-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-5-(substitutedphenyl)-3H-pyrido[2,3-d]pyrimidine-4-one (7a-j) and 4-methyl-5-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-2-oxo-7-(substitutedphenyl)-2H-pyrido[1,2-a]pyrimidine-8-carbonitrile (8a-j) have been synthesized by the reaction of 2-amino-6-{4'-[(4''-methylpiperazinyl)diazanyl]phenyl}-4-(substitutedphenyl)pyridine-3-carbonitrile (3) with urea, thiourea, ammonium thiocyanate, acetic anhydride and ethylacetoacetate respectively. These compounds have been screened for their antibacterial and antifungal activities against different microorganisms. The structures of novel synthesized compounds have been established on the basis of elemental analysis, ¹H NMR and IR spectral data.
Keywords : Pyrimidine, synthesis, antimicrobial activities.

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Some new furanones : Synthesis, characterisation and antimicrobial activity

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Abstract : The synthesis of some new furanones starting from three γ -keto acids, β -(3,4-dimethylbenzoyl)acrylic acid, β -(2,4-dimethylbenzoyl)acrylic acid and β -(2,5-dimethylbenzoyl)acrylic acid is described. Each acid is condensed with various phenols using catalytic quantity of sulphuric acid as condensing agent to get the furanones. The structures of synthesized compounds have been elucidated by elemental analysis, spectral data and chemical reactions. Some of the synthesized compounds have been evaluated for their antimicrobial activities also.

Keywords : Furanones, antimicrobial activity, phenols.

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Excellent chemoselective synthesis of α,β -unsaturated ester moiety in introducing side chain in coumarin

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Abstract : Organomagnesium reagents failed to divulge any degree of chemoselectivity towards on 7-methoxy-4-methyl-8-coumarinyl methyl ketone 1 and 7-methoxy-4-methyl-8-formylcoumarin 2 but the carbanion triethyl phosphonoester shows excellent chemoselectivity together with high stereoselectivity during the synthesis of β -coumarinylacrylates 13, 14 and β -coumarinylcrotonates 15 and 16.

Keywords : Coumarin, chemoselective synthesis, unsaturated ester, side chain.

Sorption and separation studies of thorium(IV) in L-valine medium using poly[dibenzo-18-crown-6]

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Abstract : A simple method has been developed for the separation of thorium(IV) in L-valine medium using poly[dibenzo-18-crown-6] and column chromatography. The sorption of thorium(IV) was quantitative from 1×10^{-4} to 1×10^{-1} M L-valine. Amongst the various eluents 0.5–8.0 M hydrobromic acid, perchloric acid, hydrochloric acid and 0.5–5.0 M sulfuric acid were found to be efficient eluents for thorium(IV). The capacity of poly[dibenzo-18-crown-6] for thorium(IV) was 0.258 mmol/g of crown polymer. Thorium(IV) was separated from number of cations and anions in binary mixtures. Most of the cations and anions showed very high tolerance limit. Selective separation of thorium(IV) was possible from uranium(VI), cerium(III) and lanthanum(III). The method was extended to determine thorium(IV) from geological and industrial samples.

Keywords : Sorption, separation, thorium(IV), L-valine, poly[dibenzo-18-crown-6].

Improvement of bioaccumulation of cadmium by *Aspergillus niger* as a function of complex nutrient source

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Abstract : The present study indicated that complex nutrients enhanced bioaccumulation of cadmium by *Aspergillus niger* AB₁₀. Amongst them, commercial yeast extract showed most prominent effect. Highly significant increment of cadmium accumulation of about 31.2% was observed from a chemically defined medium containing 4 µg/ml of cadmium when the culture medium was supplemented with yeast extract at a concentration of 7.5 mg/ml. Specific composition of a complex nutrient was also found to influence cadmium accumulation. A commercial peptone preparation enriched with tryptophan showed 19.4% increase in bioaccumulation of cadmium, in comparison to peptone without any fortification of tryptophan (9.4% increase). Tryptophan, when supplemented separately in the culture medium, was found to 6.7% increase in cadmium accumulation. The amino acid arginine (8.03% increase) and water-soluble vitamins like ascorbic acid (11.34% increase) and pyridoxine (13.43% increase) were also found to stimulate cadmium uptake in similar cultural conditions.

Keywords : Bioaccumulation, *Aspergillus niger*, cadmium, amino acids, vitamins.