

Separation of selected 4f and 5f metals by solid phase extraction : A review

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Abstract: This paper reviews the solid phase extraction of selected 4f and 5f metals reported by various researchers during the last decade. It is seen that major work has been carried out on uranium and thorium. This literature survey presents in a nutshell the characterization and different parameters for effectiveness of the solid phase extractants, which has been applied for separation of uranium, thorium and lanthanides in different matrices. A discussion of analytical methodologies, applications and analytical characteristics has also been included.

Keywords : Solid phase extraction, uranium, thorium, lanthanide separation, review.

Synthesis, spectral and thermal studies of tin(IV) complexes using 2-benzimidazolylmercaptoaceto hydrazone type of ligands

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Abstract: The metal complexes of Sn^{IV} with 2-benzimidazolylmercaptoaceto hydrazones of 1 : 2 metal to ligand stoichiometry have been synthesized. Molar conductance data reveal their non-electrolytic nature. The spectral studies show that the ligands react in enol form and behave as dibasic tridentate ONO donor. The thermal stabilities of the complexes have been studied by TG and their kinetic parameters calculated using Coats-Redfern and MKN methods. From these results, a coordination number six for tin ion in the complexes of the type SnL₂ have been proposed.

Keywords : Tin(IV) complexes, hydrazones, kinetic, TG.

Synthesis and spectral studies of mixed ligand complexes $\text{Cr}(\text{acac})_2\text{L}$

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Abstract: Mixed ligand complexes of the type $[\text{Cr}(\text{acac})_2\text{L}]$ (where acacH = acetylacetone and HL = salicylaldehyde, 5-bromo-(5-chloro- or 5-nitro-)salicylaldehyde, 2-hydroxyacetophenone, 2-hydroxypropiofenone, benzoylacetone or dibenzoylmethane) have been synthesized by the reactions of $\text{Cr}(\text{acac})_3$ with the corresponding carbonyl compounds in 1 : 1 molar ratios. These complexes have been characterized by IR, electronic spectroscopy and fast atom bombardment mass spectrometry. They exhibit less intense molecular ion $[\text{Cr}(\text{acac})_2\text{L}^+]$ peaks, however, very strong peak due to the fragment $\text{Cr}(\text{acac})^+$ is observed. Peaks due to other fragments e.g. $\text{Cr}(\text{acac})_2^+$, CrL_2^+ , $\text{Cr}(\text{acac})\text{L}^+$ are also observed. Oligomeric species such as $\text{Cr}_2(\text{acac})_3^+$, $\text{Cr}_2(\text{acac})_4^+$ are also formed.

Keywords : Chromium, mixed ligand complexes, FAB mass spectra, IR spectra, electronic spectra.

Cyclic voltammetric investigations of copper(II) complexes with various imidazoles in dimethylformamide

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Abstract: The cyclic voltammetric behaviour of copper(II) complexes with various imidazoles, viz. imidazole (ImH), 2-methylimidazole (2-MeIm), 1,2-dimethylimidazole (1,2-Me₂Im) and benzimidazole (BzIm) has been investigated in dimethylformamide (DMF) containing 0.1 M tetrabutyl ammonium perchlorate (TBAP) as a supporting electrolyte at a glassy carbon electrode (GCE). All these copper(II) complexes revealed a diffusion-controlled quasi-reversible one-electron transfer process ($\text{Cu}^{2+/+}$). It has been observed that the reduction peak potential becomes less positive (difficult reduction) with increasing concentration of a given ligand. The observed reduction potentials of these complexes become more positive in the order : Im \otimes 2-MeIm \otimes 1,2-Me₂Im \otimes BzIm. The deviation of reduction potentials from the expected trend based on pK_a values of the ligands is attributed to the dominance of steric effects over the electronic effects (i.e. inductive effects) in the case of 2-MeIm and 1,2-Me₂Im ligands.

Keywords : Cyclic voltammetry, electrochemistry, imidazoles, copper(II) complexes.

Synthesis and mass spectral studies of mixed ligand complexes of Co^{II} with 5-chlorosalicylaldehyde and β -diketones, hydroxyaryl aldehydes or ketones

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Abstract: A series of mixed ligand complexes of cobalt(II) having the general formula [CoLL ζ (H₂O)₂] (where HL = 5-chlorosalicylaldehyde, HL ζ = salicylaldehyde, 2-hydroxyacetophenone, 2-hydroxypropiophenone, 2-hydroxybenzophenone, 2-hydroxy-1-naphthaldehyde, pentane-2,4-dione, 1-phenylbutane-1,3-dione and 1,3-diphenylpropane-1,3-dione) have been synthesized and characterized. The complexes have distorted octahedral structures. The characterization of the complexes has been made on the basis of elemental analyses, conductances, TLC, magnetic moments and IR, electronic and FAB mass spectra.

Keywords: Mixed ligand complexes, cobalt(II), IR, electronic spectra, magnetic moments, FAB mass spectra.

Spectroscopic and biochemical studies of chromium(III) and manganese(II) complexes with *p*-vanillin containing thiosemicarbazone and semicarbazone ligands

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Abstract: A new series of chromium(III) and manganese(II) complexes with *p*-vanillin thiosemicarbazone (L¹) and *p*-vanillin semicarbazone (L²) have been synthesized and characterized. The nature of bonding and stereochemistry of the complexes have been deduced from elemental analysis, conductivity measurements, mass, magnetic susceptibility, infrared and electronic spectral studies. On the basis of these spectral studies, an octahedral geometry has been assigned for all the complexes. The antimicrobial activities of the free ligands and their metal complexes were evaluated on different species of phytopathogenic fungi and their biopotency has been discussed.

Keywords: Thiosemicarbazone, semicarbazone, spectral, antifungal studies.

Schiff bases of 3-(2-thiazolylazo)-2,4-pentanedione with aliphatic diamines and their metal complexes

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Abstract: Reaction of aliphatic diamines [1,2-diaminoethane, 1,3-diaminopropane and 1,6-diaminohexane] with 3-(2-thiazolylazo)-2,4-pentanedione yielded a new series of polydentate Schiff base ligands. The existence of these compounds predominantly in the intramolecularly hydrogen bonded keto-imine form has been well demonstrated from their analytical, IR, ¹H NMR and mass spectral data. Dibasic tetradentate N₄ coordination of the compounds in their [ML] complexes [M = Ni^{II}, Cu^{II} and Zn^{II}] has been established on the basis of analytical and spectral data.

Keywords: 3-(2-Thiazolylazo)-2,4-pentanedione, Schiff base, keto-imine, IR spectra, ¹H NMR spectra, mass spectra.

Synthesis and characterization of mixed ligand complexes of tin(II) and tin(IV)

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Abstract: Some new and novel complexes of tin(II) and tin(IV) of the type [(sb)SnCl_{n-2}(sb_ϕ)] [where sb = Schiff bases; salicylidene-4-methyl-1-aminobenzene (*p*-smab*H*), salicylidene-3-nitro-1-amino benzene (snab*H*); sb_ϕ = Schiff bases; 4-nitrobenzylidene-2-aminophenol (nbaph*H*), salicylidene-2-aminopyridine (sap*H*), salicylidene-4-aminoacetophenone (sapphe*H*) and n = 2 for tin(II), 4 for tin(IV)] have been prepared by the interaction of metal chloride with sodium salts of Schiff bases in the presence of benzene-methanol mixture. All these complexes are monomeric in nature. The complexes of tin(II) and tin(IV) are coloured solids with sharp melting points and were characterized on the basis of elemental (C, H, N, Cl and Sn) analysis and spectral [IR, NMR (¹H, ¹³C) and FAB-mass] studies.

Keywords: Tin(II) and tin(IV), Schiff bases, NMR, FAB-mass.

Effect of different organic material on lead adsorption in soils of Aligarh district

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Abstract: The effect of different organic material (FYM, sewage sludge and poultry litter) on adsorption-desorption of lead on three soil samples of Aligarh district was studied. The adsorption isotherms were of 'L' type and adsorption-desorption data conformed to Freundlich isotherm equation. The adsorption increased with the increase in organic material and followed the order : sewage sludge > FYM > poultry litter. The adsorptivity of soils was in order : Soil No. 1 > 2 > 3. The adsorption capacity was significantly positively correlated with soil organic carbon and soil-clay-content. The desorption of lead was more by CaCl₂ than water and followed the order : soil 3 > 2 > 1. The desorption of lead decreased with the addition of organic material suggesting a less availability of lead to crops grown on such soils. The complete desorption of lead did not take place.

Keywords : Adsorption, desorption, lead, soils.

Dead polyacrylamide of low polydispersity from atom transfer radical polymerization of acrylamide using CuCl/Me₆TREN as catalyst

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Abstract: Polyacrylamide with a low polydispersity index (PDI) has been obtained by atom transfer radical polymerization of acrylamide. The polymerization was carried out in dimethylformamide-water (50 to 70% DMF by volume). 2-Chloropropanamide, CuCl and tris(2-dimethylaminoethyl)amine (Me₆TREN) were used as initiator, catalyst and ligand respectively. Apart from these reagents, CuCl₂ and LiCl were used as additives with the expectation of increasing the rate of deactivation of the polymer radicals and thus lowering the PDI. The LiCl also helps to keep the polymer in solution at low temperature (*ca.* 0 °C or below). The polymerization was carried out in the temperature range -20 to 130 °C. Better yield and lower PDI of the polymers are obtained as the temperature is lowered. However, the polymer separates out of the solution at subzero temperatures even in the presence of LiCl. Kinetic study of the polymerization at 0 °C reveals that the conversion is incomplete but the molecular weights increases with increase in conversion. Polymers with PDI »1.15 can be prepared. However, all attempts of chain extension failed, which indicates end group loss.

Keywords : Polyacrylamide, atom transfer radical polymerization, polydispersity.

Uncatalysed and catalysed oxidation of dextrose by *N*-bromosuccinimide in basic medium : A kinetic and mechanistic study

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Abstract: Kinetic investigation on uncatalysed and iridium trichloride catalysed oxidation of dextrose by alkaline solution of *N*-bromosuccinimide (NBS) have been carried out in the temperature range 25–45 °C. The reaction has been found to be first order with respect to each of oxidant and substrate in both uncatalysed and catalysed reaction. The reaction follows a first order dependence on OH⁻ ion concentration. A first order dependence on [catalyst] is also observed. Negligible effect of chloride ion on its higher concentration has been observed. Addition of succinimide shows a negative effect. The increase in ionic strength of the medium increases the rate of reaction. A 1 : 1 stoichio-metry is observed in the oxidation. Effect of temperature on the rate of oxidation has been studied to show the validity of Arrhenius equation and various activation parameters have been computed.

Keywords : Kinetics, dextrose, *N*-bromosuccinidime.

A mild and efficient semi-synthesis of shikonin leucoacetates

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Abstract: A mild and efficient synthesis of shikonin triacetate is developed by acetylation of shikonin with anhydrous acetic anhydride in the presence of iodine as an extremely powerful catalyst under solvent-free condition in 94.5% yield, then a regioselective reduction of carbonyl of 1,4-naphthoquinone with sodium monoacetoxyborohydride and acetylation of shikonin triacetate can afford shikonin leucoacetates with the yield of 76.4%.

Keywords : Shikonin leucoacetates, shikonin triacetate, acetylation, monoacetoxyborohydride

Synthesis of some new arylidenes-substituted phenyl-1,3,4-thiadiazol-4-oxo-thiazolidines : Antimicrobial and diuretic activities

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Abstract: A series of new [2-(substituted aryl)-3-{5-(substituted phenyl)-1,3,4-thiadiazol}]-4-oxo-thiazolidines, 3(a-o) have been synthesized via the condensation of 5-(substituted phenyl)-2-(2-substituted benzylidene amino)-1,3,4-thiadiazoles, 2(a-o) with thioglycolic acid. The compounds 2(a-o) were synthesized from 5-(substituted phenyl)-2-amino-1,3,4-thiadiazole, 1(a-c) with various aromatic aldehydes which itself was prepared from substituted aromatic acid(s) using thiosemicarbazide. The compounds 3(a-o) on treatment with various aromatic aldehydes afforded [5-(substituted arylidenes)-2-(substituted aryl)-3-{5-(substituted phenyl)-1,3,4-thiadiazol}]-4-oxo-thiazolidines, 4(a-o). The structures of all the synthesized compounds have been determined by spectral and chemical methods. The compounds 2, 3 and 4 were screened for their antifungal and antibacterial activities against *E. coli*, *S. typhimurium* and *B. subtilis* bacteria and *R. oryzae*, *C. albicans* and *A. niger* fungi and diuretic activity on adult male rats.

Keywords: New thiazolidines, antimicrobial, diuretic activity.

Synthesis and antileishmanial activity of 1-aminomethyl-5-substituted-3-{4 ϵ -(3 $\epsilon\epsilon$ -chlorobenzoyloxy)-benzoylhydrazono}-2-indolinones

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Abstract: 5-Substituted-3-{4 ϵ -(3 $\epsilon\epsilon$ -chlorobenzoyloxy)-benzoylhydrazono}-2-indolinones (3-6) were synthesised by the condensation of 4-(3 ϵ -chlorobenzoyloxy)-benzoylhydrazine (2) and 5-substituted isatins. Mannich reaction in the presence of formaldehyde and heterocyclic secondary amines on indolinones (3-6) furnished 1-aminomethyl-5-substituted-3-{4 ϵ -(3 $\epsilon\epsilon$ -chlorobenzoyloxy)-benzoylhydrazono}-2-indolinones (7-22). The structures of the compounds have been established by means of correct elemental analysis and spectral data (IR, PMR and Mass). The compounds have been screened for their antileishmanial potential against *Leishmania donovani*.

Keywords: Isatins, Mannich reaction, antileishmanial activity.

Polarographic study of ternary complexes of [Cd^{II}-L-amino acidate-vitamin-PP] system

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Abstract: Interaction between Cd^{II} with some L-amino acids such as L-lysine, L-ornithine, L-threonine, L-serine, L-phenylglycine, L-phenylalanine, L-glutamic acid and L-aspartic acid as primary ligands and vitamin-PP (nicotinamide or niacinamide) as secondary ligand has been studied by simple DC polarography using dropping mercury electrode as an indicator electrode and calomel electrode (saturated) as reference electrode at pH = 7.3±0.01 in 1.0 M KNO₃ (supporting electrolyte) at 298 K. Schaap and McMaster method confirmed the formation of 1 : 1 : 1, 1 : 1 : 2 and 1 : 2 : 1 complexes with stability constant trend L-lysine < L-ornithine < L-threonine < L-serine < L-phenylglycine < L-phenylalanine < L-glutamic acid < L-aspartic acid can be explained on the basis of nature of amino acids, size and bonding between metal and ligands.
Keywords : Polarography, stability constant, [Cd^{II}-L-amino acidate-vitamin-PP] system.

Methylthymolblue complexon as corrosion inhibitors for aluminum in trichloroacetic acid

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Abstract: The corrosion of aluminum in trichloroacetic acid (TCA) containing methylthymolblue complexon has been studied. In plain trichloroacetic acid, the corrosion rate increases with the acid concentration. At constant acid concentration, the inhibition efficiency (IE) of methylthymolblue complexon increases with the inhibitor concentration. At constant inhibitor concentration, the IE decreases with the increase of acid concentration. As temperature increases, percentage of inhibition decreases. Plot of log (q/1 - q) versus log C results in a straight line, suggesting that the inhibitors cover both the anodic and cathodic regions through general adsorption following Langmuir isotherm. The curves show very little anodic but significant cathodic polarization.
Keywords : Corrosion, aluminum, trichloroacetic acid, methylthymolblue complexon.

Formation and stability of transition metal chelates with 2-(2-hydroxyphenyl) benzoxazole and other ligands

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Abstract: The formation equilibria of (1 : 1 and 1 : 2) binary chelates, ML and ML₂ and (1 : 1 : 1) ternary chelates, MAL where, M = Co^{II}, Ni^{II} and Zn^{II}; L = 2-(2-hydroxyphenyl)benzoxazole (HPBO) and A = glycine, alanine, proline and histidine have been studied pH-metrically in aqueous-dioxane (50% v/v) medium at 30 °C and 0.1 M (KNO₃) ionic strength. The formation constants of the complexes were evaluated. The relative stabilities of the ternary complexes were expressed in terms of the parameter $\log K$ and various factors influencing the formation and stability of complexes are discussed. The thermodynamic parameters (ΔH , ΔS and ΔG) associated with the ternary systems were also evaluated by determining the formation constants at 20, 30 and 40 °C and 0.1 M KNO₃ ionic strength. All the parameters were found to be favourable for the complexation.

Keywords: Binary chelates, ternary chelates, formation constants, stability, thermodynamic parameters.

Spectroscopy, electrochemistry and biocidal activity of amino acid Schiff base metal complexes

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Abstract: Cu^{II}, Ni^{II}, Zn^{II}, Co^{II}, Mn^{II} and VO^{II} Schiff base complexes derived from indole-3-carboxaldehyde (Ind) and amino acids viz. L-alanine (ala), L-phenylalanine (pala) and L-histidine (his) have been prepared and characterized by elemental analysis, molar conductance, magnetic susceptibility, IR, electronic absorption and EPR (at room temperature and at 77 K) spectroscopic techniques. The electrochemical properties of Cu^{II} complexes exhibit a well-defined quasireversible redox wave attributed to one electron transfer process. The Cu^{II} and Ni^{II} Schiff base complexes were screened for their biocidal activities *in vitro* on common bacteria.

Keywords: Indole-3-carboxaldehyde, cyclic voltammetry, biocidal activity.

Thermodynamic studies of ternary eutectic mixture $\text{KNO}_3 + \text{NaNO}_2 + \text{NaNO}_3$ at different temperatures

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Abstract: The internal pressure, energy of vaporization and heat of vaporization of pure components of ternary eutectic mixture $\text{KNO}_3 + \text{NaNO}_2 + \text{NaNO}_3$ were computed from the reported values of thermal expansivity and isothermal compressibility at different temperatures. These thermodynamic properties were also obtained for the eutectic mixture at two different compositions as a function of temperature.

Keywords: Eutectic mixture, thermodynamic study.

Determination of dissociation constants of salicylic acids and stability constants of complexes of these ligands with copper(II), beryllium(II) and iron(III) ions :

Correlation of dissociation and stability constants with molecular descriptors

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Abstract: Salicylic acid and its various derivatives have anti-rheumatic and antifungal action on human body. Dissociation constants of salicylic acid, *o*-/*m*-/*p*-cresotic acids and sulphosalicylic acid and stability constants of complexes of these ligands with Cu^{II} , Be^{II} ions were determined pH metrically and Fe^{III} ion were determined spectrophotometrically. A quantitative structure relationship of stability/dissociation constant of the compounds with molecular properties were considered, which include molecular weight, surface tension, index of refraction and density. The biological action of these compounds vary with the substituent present in the ring.

Keywords: Dissociation constant, stability constant, copper(II), beryllium(II), iron(III).

Kinetics and thermodynamics of fluoride ion sorption on fire clay from aqueous solution

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Abstract: The kinetics and thermodynamics of fluoride ions sorption on fire clay from aqueous solution have been investigated. The various factors that influence the defluoridation efficiency of the material were studied. Adsorption obeyed both Langmuir and Freundlich isotherm models. Various thermodynamic parameters such as DG° , DH° and DS° were calculated indicating that this system was a spontaneous one and endothermic in nature. FTIR studies showed the involvement of hydroxyl group on the surface in the adsorption interaction.

Keywords: Fluoride removal, fire clay, adsorption, Langmuir isotherm, kinetics.
