

Chemical speciation and thermodynamic stability of quaternary mixed chelates of bio-metals involving lysine, proline and uracil

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Abstract : The metal-ligand formation constants of quaternary complexes of the type M_1M_2AB (where A = lysine/proline; B = uracil; $M_1 = M_2 = \text{Cu/Ni/Zn/Co}$) have been determined potentiometrically in biologically relevant conditions at constant ionic strength $I = 0.1 \text{ M NaNO}_3$. The overall stability constants have been evaluated using SCOGS computer program. The complexation equilibria have been derived on the basis of species distribution diagram. In all cases, amino acids and pyrimidines are found to be compatible ligands and thereby proving greater stability of ternary and quaternary complexes as compared to binary ones. The order of stability constants of quaternary systems with both the amino acids have been observed as : $\text{Cu-Ni} > \text{Cu-Zn} > \text{Cu-Co} > \text{Ni-Zn} > \text{Ni-Co} > \text{Co-Zn}$. The factors responsible for the compatibility of both the ligands have also been discussed.

Keywords : Uracil, proline, lysine, thermodynamic stability, chemical speciation.

Electrochemical investigations of oxovanadium(IV) complexes with bidentate Schiff base ligands in dimethylformamide

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Abstract : The electrochemical redox properties of bidentate Schiff base oxovanadium(IV) complexes viz. $\text{VO}(\text{sal-anl})_2$ 1, $\text{VO}(\text{sal-tod})_2$ 2, $\text{VO}(\text{hmp-and})_2$ 3 and $\text{VO}(\text{hmp-oxime})_2$ 4 have been studied in DMF/0.1 M TBAP using cyclic voltammetry at a Pt-working electrode. The electrochemistry of sal-anl and sal-tod ligands was also examined for the sake of comparison. Complexes 1 and 2 show almost similar electrochemistry. Oxidation of complex 2 is easier while reduction is difficult in comparison to 1 due to the +I inductive effect of electron-donating methyl group at the *para* position of toluidine. Complex 1 showed two irreversible anodic waves at $E_{\text{pa}1} = +0.68 \text{ V}$ and $E_{\text{pa}2} = +1.06 \text{ V}$ vs SCE and two irreversible cathodic peaks at $E_{\text{pc}1} = -0.90 \text{ V}$ and $E_{\text{pc}2} = -1.325 \text{ V}$ vs SCE followed by an irreversible anodic peak at $E_{\text{pa}c2} = -0.42 \text{ V}$ at scan rate 100 mV s^{-1} . On the basis of CV results, it is concluded that in complexes 1 and 2, the redox processes a_1 and c_2 are metal-based while the redox processes a_2 and c_1c seem to be ligand-based. The CVs of 3 and 4 are also qualitatively similar. It is noteworthy that the anodic peak potential for the process $\text{VO}^{2+/3+}$ shifted to more positive values in the sequence : $\text{VO}(\text{sal-tod})_2 \text{ @ } \text{VO}(\text{sal-anl})_2 \text{ @ } \text{VO}(\text{hmp-oxime})_2 \text{ @ } \text{VO}(\text{hmp-and})_2$.

Keywords : Cyclic voltammetry, electrochemistry, Schiff bases, vanadium complexes, biomimetic.

Mg^{II} , Ca^{II} , Sr^{II} and Ba^{II} complexes of 2,3,15,16-tetramethyl/ethyl/*p*-tolyl-1,4,14,17-tetraazacyclohexacosyl,3,14,16-tetraene

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Abstract : Hexacoordinated tetraazamacrocyclic complexes of the general formula $[MLX_2]$ (where L = N_4 macrocycle having 26-membered ring, X = Cl or NCS and M = Mg, Ca, Sr or Ba) have been synthesized by 2+2 cyclocondensation reactions of 1,9-diaminononane with α -diketones viz. 2,3-butanedione, 3,4-hexanedione or 4,4-dimethylbenzil in the presence of metal ions as templates. The synthesized complexes were characterized by elemental analyses, conductance measurements and IR and 1H NMR spectra.

Keywords : Macrocyclic complexes, alkaline earth metal complexes, conductances, IR spectra, NMR spectra.

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Electrochemical and spectral studies of binuclear monohydroxo-bridged copper(II) complexes

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Abstract : The electrochemical behavior of m-hydroxo-tetrakis(2,2'-bipyridine)dicopper(II) perchlorate, $[(bipy)_2Cu(m-OH)Cu(bipy)_2](ClO_4)_3$, 1 and m-hydroxo-tetrakis(1,10-phenanthroline)dicopper(II) perchlorate, $(phen)_2Cu(m-OH)Cu(phen)_2 (ClO_4)_3$, 2 has been studied in dimethylsulfoxide (DMSO), dimethylformamide (DMF) and acetonitrile (AN) containing 0.2 M sodium perchlorate at a glassy carbon working electrode (GCE) at 25 °C using cyclic voltammetry. Both these complexes undergo a diffusion-controlled single-electron quasi-reversible electrode process. The value of formal electrode potentials, E^{0c} for complex 1 are 80 mV and 145 mV vs Ag/AgCl in DMSO and DMF, respectively. The coulometry performed at -170 mV vs Ag/AgCl in the case of light blue solution of complex 1 in DMSO containing 0.2 M $NaClO_4$ yielded an intense red solution involving a single electron transfer per molecule of complex ($Cu^{II}Cu^{II} \rightarrow Cu^{II}Cu^I$). The cyclic voltammograms of the red coloured mixed-valent complex solution were also recorded, giving a quasi-reversible anodic couple (Cu^+/Cu^{2+}) with E^{0c} about 50 mV vs Ag/AgCl. The red solution, on keeping overnight, turned light greenish-blue in colour. The CV experiments with this light greenish-blue solution, result in a quasi-reversible cathodic couple (Cu^{2+}/Cu^+) with E^0 about 50 mV. On the basis of our present electrochemical studies it is found that the redox potentials of 1 and 2 are strongly dependent on the organic solvents. The electronic spectra of these complexes were also recorded in DMSO, DMF and AN.

Keywords : Electrochemistry, cyclic voltammetry, hydroxy-bridged binuclear copper(II) complexes, bipyridine, phenanthroline.

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Group-contribution method for the estimation of surface tension of binary liquid mixtures

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Abstract : Corresponding-states group-contributions method (CSGC), for the first time, has been extended to estimate surface tension of five binary liquid mixtures namely, 1,4-dioxane + hexane, 1,4-dioxane + heptane, 1,4-dioxane + octane, 1,4-dioxane + nonane, 1,4-dioxane + decane. Computed values of the employed method are compared with the experimental findings. The agreement between experimental and theoretical values is found to be satisfactory.

Keywords : Surface tension, group-contributions, corresponding-states, liquid mixtures, binary mixtures.

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Theoretical study of crystalline state properties of heavy metal halide with different structures

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Abstract : The crystalline state properties such as cohesive energy, atomization energy, force constant, IR absorption frequency, Debye temperature, Grüneisen parameter, Anderson Grüneisen parameter, Moelwyn-Hughes parameter of heavy metal halide are reported here. These parameters have been obtained by using the Born-Mayer, Hellmann, Varshini-Shukla and Ali-Hasan interionic short-range repulsive interactions. Calculations are also performed for the computation 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 framework of Dugdale and MacDonald theory. The high pressure behaviour of these crystals has also been studied.

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

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Spectroscopic and physicochemical studies on the charge transfer complex of dextromethorphan drug with iodine

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Abstract : The drug dextromethorphan had been shown to form a charge transfer (CT) complex of 1 : 1 stoichiometry with iodine in different solvents. The enthalpy and entropy of formation of this complex had been determined by estimating the formation constant spectrophotometrically at three different temperatures. The solvent variation study on the intermolecular CT complex formation revealed that the intensity of the interaction increases with decrease in relative permittivity of the medium. Transition energy, oscillator strength and dipole moments of the complex as well as the ionization potential of the donor have been computed from spectroscopic data and discussed. The experimentally measured ionization potential of the donor drug closely agrees with that computed theoretically using MOPAC (PM3) semi-empirical method.

Keywords : Charge transfer complex, dextromethorphan, iodine, solvent effect.

A study on hydrodynamic permeabilities of aqueous solutions of some alkali and alkaline earth metal chlorides across an inorganic membrane of aluminium oxide

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Abstract : Experimental results for the measurement of hydrodynamic permeabilities of very dilute aqueous solutions of some alkali and alkaline earth metal chlorides viz. sodium chloride, potassium chloride, magnesium chloride and calcium chloride through an inorganic membrane of aluminium oxide of known thickness are reported. The results have been analysed in terms of the methodology of non-equilibrium thermodynamics. The mechanical filtration coefficient, L_p and the rejection coefficient/membrane selectivity (ξ) have been evaluated and the effect of concentration on these parameters also discussed. To evaluate activation parameters the hydrodynamic permeabilities have been determined at different temperatures.

Keywords : Hydrodynamic permeabilities, aqueous solutions, alkali and alkaline earth metal chlorides.

Study on the spectral characteristics of 4-hydroxy-3-methoxy benzoic acid in different pH and α -cyclodextrin

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Abstract : Effect of buffer solutions of different pH and α -cyclodextrin (α -CD) on the absorption and fluorescence spectra of 4-hydroxy-3-methoxy benzoic acid (vanillic acid HMB) have been investigated. The inclusion complex of α -CD with HMB is investigated by UV-Visible and fluorimetry and Scanning Electron Microscope (SEM) methods. These studies show that HMB forms 1 : 1 inclusion complex with α -CD. There is an unusual blue shift of hydroxyl ion (dianion) in the α -CD medium. This confirms that the -OH group is present in the interior part of the α -CD cavity and -COOH group is present in the upper part of the α -CD cavity. A mechanism is proposed to explain the inclusion process.

Keywords : 4-Hydroxy-3-methoxy benzoic acid (vanillic acid HMB), α -cyclodextrin (α -CD), pH effect, inclusion complex.

A kinetic study of Ru^{III} catalyzed oxidation of galactose and cyclopentanol by potassium bromate in alkaline medium

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Abstract : The kinetics of Ru^{III} catalyzed oxidation of D(+)-galactose (galac) and cyclopentanol (cyclopen) by KBrO₃ in alkaline medium using mercuric acetate as a scavenger for Br⁻ ion (galac) as well as co-catalyst (cyclopen), have been studied in the temperature range 30–45 °C. The reactions exhibit first-order kinetics with respect to oxidant and catalyst while zero-order kinetics with respect to substrate was observed in case of both galactose and cyclopentanol. For cyclopentanol, a positive effect on the rate of reaction were observed on successive addition of [Hg(OAc)₂] and [Cl⁻] whereas [OH⁻] exhibited an inverse fractional order. In case of galactose, [OH⁻] and [Hg(OAc)₂] have no effect on the reaction velocity. A positive fractional order in acetic acid concentration (AcOH) and negligible effect of ionic strength of the medium were observed.

The reactive species of Ru^{III} in alkaline medium is [RuCl₂(H₂O)₃(OH)] (galac) and [RuCl₃(H₂O)₂OH]⁻¹ (cyclopen) under the experimental pH range. A suitable mechanism in conformity with the kinetic observation has been proposed. The various activation parameters such as energy of activation (ΔE*), Arrhenius factor (A), entropy of activation (ΔS*) were calculated from the rate measurements at 30, 35, 40 and 45 °C. A rate law has been derived on the basis of data obtained.

Keywords : Ru^{III}, galactose, cyclopentanol, oxidation, kinetics.

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1,3-Dipolar cycloadditions. Part-XV : Systematic spectroscopic investigations of C-aryl-N-methylnitrones¹

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Abstract : Several C-aryl-N-methylnitrones have been synthesised during the course of our work on 1,3-dipolar cycloadditions. Detailed systematic spectroscopic investigations of all these nitrones have been undertaken. These are reported along with some generalisations made regarding their spectroscopic properties.

Keywords : C-Aryl-N-methylnitrones, UV, IR, NMR, MS.

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Palladium catalyzed cross-coupling reactions of organomercurials[†]

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Abstract : The palladium catalyzed cross-coupling reactions of methyl mercury iodide provide a mild, selective and general method for the construction of new C-C bond i.e. for the synthesis of arenes. The reaction proceeds in DMF, in the presence of a nucleophilic catalyst (iodide ion), at room temperature under argon atmosphere. Pd⁰ which is generated in the reaction mixture catalyzes the reaction. The catalytic cycle involved in the reaction consists of a sequence of *oxidative addition*,

transmetallation and reductive elimination steps. The role played by the iodide ion as a nucleophilic catalyst is discussed. It forms an ionic complex $(Pd^{0}I)^{-}$ and therefore precipitation of palladium black is prohibited. Secondly the formation of ionic complex $(CH_3HgI_2)^{-}$ helps the heterolytic fission of C–Hg bond in transmetallation step. Thus the selectivity of the reaction towards the expected product is enhanced.

Keywords : Palladium catalyzed reactions, cross-coupling reactions, organomercurials, C–C bond formation.

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Kinetic method for the estimation of aldopentoses

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Abstract : Kinetic methods of analysis are now accepted as standard analytical procedure for various types of samples. Aldopentoses (D-xylose, D-arabinose and D-ribose) have been estimated in simulated samples by kinetic (uncatalyzed) method (D-xylose and D-arabinose [0.05–0.3 M] and D-ribose [0.0250–0.1375 M]) using cerium(IV)-aldopentose redox indicator reaction in sulphuric acid medium. The indicator reaction is well understood as electron transfer reaction. The investigations have been made from the calibration plots obtained by using pseudo first order rate constant data of the indicator reaction and also by fixed time and fixed concentration procedures. The rate data was obtained for the indicator reaction under varying kinetic experimental conditions. Results of this study indicate that estimated values for the simulated samples are consistent and reproducible within $\pm 1\%$.

Keywords : Kinetic determination, aldopentoses, estimation, redox reaction, kinetic estimation.

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Synthesis, spectral characterization and reactivity study of new Schiff base complexes of divalent manganese, copper and zinc

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Abstract : New Schiff base complexes of Mn^{II} , Cu^{II} and Zn^{II} of the type $[M(HL)CH_3COO(H_2O)].2H_2O$ ($M = Mn$), $[M(HL)CH_3COO(H_2O)]$ ($M = Cu$ or Zn), $H_2L = N$ -(4-carboxyphenyl)salicylalimine, have been synthesized from the reaction of the Schiff base ligand with the respective metal acetate in a methanolic solution maintaining the metal to ligand ratio as 1 : 1. Complexes have been characterized on the basis of elemental analyses, FT-IR, NMR, ESR, electronic spectroscopy, room temperature magnetic moment measurements and by TGA/DTA studies. The results of above studies suggest that the newly synthesized complexes are tetra coordinated and Schiff base is acting as bidentate ligand, ligated to the metal center through its phenolic oxygen and azomethine nitrogen atoms respectively. Copper(II) complex appears to be square planar while Mn^{II} and Zn^{II} complexes are essentially tetrahedral. Thermal study indicate that Cu^{II} and Zn^{II} complexes are thermally stable up to a temperature of *ca.* 150 °C, whereas manganese(II) complex undergo loss of two molecules of lattice water between 120–125 °C, followed by the loss of coordinated water molecule between 190–200 °C. Mn^{II} complex has demonstrated its catalytic potential in oxidizing the selective organic substrate viz. benzyl alcohol by H_2O_2 as the oxidant.

Keywords : Mn^{II} , Cu^{II} , Zn^{II} , Schiff base complexes, synthesis, characterization, reactivity.

Study of kinetic parameters and stability constants of [Mn-antibiotics-cefoperazone] complexes vis-a-vis kinetics of electrode reaction

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Abstract : Kinetic parameters and stability constant of complexes of Mn with doxycycline, chlortetracycline, oxytetracycline, tetracycline, monocyline, amoxicillin and chloramphenicol as primary ligands and cefoperazone as secondary ligand have been reported polarographically at pH = 7.30 ± 0.01 , $m = 1.0 M NaClO_4$ at 25 °C. Mn^{2+} formed 1 : 1 : 1, 1 : 1 : 2 and 1 : 2 : 1 complexes. The stability constant (log b) varied from 1.85 to 9.25 showed that these drugs or in combination with cefoperazone or their metal complexes could be used for Mn toxicity. The values of kinetic parameters viz. transfer coefficient (α), degree of irreversibility (l) and rate constant (k) confirmed that the electrode processes were quasireversible and the 'transition state' is a function of applied potential. A small variation in potential not only affects the rate but rate constant greatly.

Keywords : Cefoperazone, Mn, antibiotic, kinetics, stability constant.

Kinetics and mechanism of the oxidative regeneration of carbonyl compounds from oximes by benzyltrimethylammonium chlorobromate

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Abstract : The oxidative deoxygenation of several aldo- and keto-oximes by benzyltrimethylammonium chlorobromate (BTMACB) in 1 : 1 (v/v) acetic acid-water is found to be first order with respect to both oximes and BTMACB. The oxidation of ketoximes is slower than that of aldoximes. The rates of oxidation of aldoximes correlated well in terms of Pavelich-Taft dual substituent-parameter equation. The low positive value of polar reaction constant indicated a nucleophilic attack by a chlorobromate ion on the carbonyl carbon. The reaction is subjected to steric hindrance by the alkyl groups. The effect of solvent composition indicated that the rate increases with an increase in the polarity of the solvent. A mechanism involving the formation of a cyclic activated complex, in the rate-determining step, has been proposed.

Keywords : Oxime, benzyltrimethylammonium chlorobromate, carbonyl, mechanism, kinetics.

Refractive indices of formamide + alkanol/alkanediol binary mixtures at different temperatures

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Abstract : The refractive indices (n) of pure formamide (FA), ethanol, 1-propanol, 1,2-ethanediol, 1,2-propanediol and those of their forty four binary mixtures, with FA as common component, covering the whole composition range have been measured at 293.15, 298.15, 303.15, 308.15, 313.15 and 318.15 K. The deviation in refractive index (Δn) has been calculated from the experimental data. The variation of Δn with composition and temperature of the mixtures have been discussed in terms of molecular interaction in these mixtures. It is observed that the extent of deviation in Δn for these mixtures follows the sequence : ethanol > 1-propanol > 1,2-ethanediol > 1,2-propanediol, indicating the presence of strong interactions in these mixtures in the same order.

Keywords : Refractive index, formamide, alkanol, alkanediol, molecular interactions.

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Studies on mixed ligand complexes involving (*N*-methyliminodiacetic acid)-lead(II) and some bioligands : A voltammetric investigation

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Abstract : Composition and stability constants of mixed ligand complexes of Pb^{II} ion with *N*-methyliminodiacetic acid (MIDA) and some amino acids have been investigated voltammetrically. The reduction of Pb^{II} has been found to be reversible and diffusion controlled involving two electrons in each case. Only MXY_2 type of mixed ligand complexes are formed. The extended method of Schaap and McMasters has been used to evaluate the stability constants of these complexes. By using these stability constants the statistical and electrostatic effects have been discussed.

Keywords : Mixed ligand complexes, voltammetry, *N*-methyliminodiacetic acid (MIDA), amino acids.

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Determination of stability constants of metal-methionine and metal-methionine-NTA (binary and mixed) complexes by ionophoretic technique

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Abstract : Electrophoretic technique has been used for the study of Fe^{III} , Cu^{II} , Ni^{II} , Co^{II} -methionine binary and Fe^{III} , Cu^{II} , Ni^{II} , Co^{II} -methionine-NTA (nitrilotriacetic acid) ternary complexes. The stability constants of metal-methionine binary complexes are found to be $10^{6.03}$, $10^{5.23}$, $10^{5.13}$, $10^{4.23}$ and the stability constants of metal-methionine-NTA ternary complexes have been found to be $10^{6.35}$,

$10^{6.04}$, $10^{5.87}$, $10^{5.80}$ for Fe^{III} , Cu^{II} , Ni^{II} and Co^{II} complexes respectively at $m = 0.1 \text{ M}$ (HClO_4) and 25°C .

Keywords : Binary complex, ternary complex, stability constant, ionophoretic technique.

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Synthesis and biological study of oxopyrimidines and thiopyrimidines of 2-(2,4-dichlorophenyl)imidazo[1,2-*a*]pyridin-3-carbaldehyde

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Abstract : 2-(2,4-Dichlorophenyl)imidazo[1,2-*a*]pyridine-3-carbaldehyde (1) was prepared from 2-aminopyridine through multi-step reaction. Compound 1 reacts with different aryl ketones in the presence of catalytic amount of 40% KOH to give (2*E*)-3-[2-(2,4-dichlorophenyl)imidazo[1,2-*a*]pyridin-3-yl]-1-arylprop-2-en-1-ones (2a-j), which on cyclization with urea and thiourea in the presence of basic catalyst like KOH afforded 6-[2-(2,4-dichlorophenyl)imidazo[1,2-*a*]pyridine-3-yl]-4-aryl-pyrimidin-2(1*H*)-ones (3a-j) and 6-[2-(2,4-dichlorophenyl)imidazo[1,2-*a*]pyridine-3-yl]-4-aryl-pyrimidin-2(1*H*)-thiones (4a-j). The antimicrobial evaluations have been performed for their antibacterial activity and antifungal activities.

Keywords : Imidazo[1,2-*a*]pyridine, oxopyrimidines, thiopyrimidines, antibacterial activity, antifungal activity.

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Extractive spectrophotometric determination of ruthenium(III) with 4-(benzylideneimino)-5-methyl-4*H*-1,2,4-triazole-3-thiol

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Abstract : A simple, highly sensitive and selective spectrophotometric method is proposed for the extraction of microgram level concentration of ruthenium(III) with 4-(benzylideneimino)-5-methyl-4*H*-1,2,4-triazole-3-thiol (BIMTT) as an extractant at pH 5.4 in chloroform. The optimum pH range for extraction of complex is 4.5-6.5. The method obeys Beer's law in the range 10 ppm to 50 ppm, a molar absorptivity and Sandell's sensitivity of $1.52 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and 0.066 mg cm^{-2} respectively at 394 nm. The effect of reagent concentration, pH, equilibration time and various foreign ions has been investigated. The method is applicable to determine ruthenium(III) from synthetic mixture corresponding to Fissium alloys.

Keywords : Ruthenium(III), solvent extraction, chloroform, Fissium alloy.