



Unique Research Journal of Chemistry

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Research Article

CHEMICAL SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY OF 2-AMINO-2-ETHYL 1,3 PROPANEDIOL AND IT'S Cu (II), Ru (III) COMPLEXES

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Received: 24-12-2013; Revised: 22-01-2014; Accepted: 17-02-2014

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ABSTRACT

Dithiocarbamates are well known compounds to bind selectively to metal ions. Most of applications are based on complexation properties of dithiocarbamate ligands with metal ions, especially with transition metal ions. Dithiocarbamate ligands readily form chelates with all transition metal ions through its donor sulphur atoms. In this study, A new series of bidentate ligand such as dithiocarbamate of 2-Amino-2ethyl 1,3 propanediol was prepared by synthetic methods as their sodium salts. In the reaction of Copper Chloride and ruthenium Chloride with AEPDDTC the corresponding complexes were prepared. The complexes were characterized by elemental analysis, FT-IR, ^1H NMR, UV, ESR and TGA-DTA. The synthesized compounds showed an excellent anti microbial activities against clinical bacterial isolates *E. coli* and *Bacillus subtilis*.

Keywords: 2-Amino-2-ethyl 1,3 propanediol, Copper Chloride, Ruthenium Chloride, Dithiocarbamates, Synthesis, Characterization, Anti bacterial activity.

INTRODUCTION

Dithiocarbamates are organosulphur compounds and their metal complexes have drawn much research attention due to their diverse applications and interesting biological, structural, magnetic, electrochemical and thermal properties¹⁻⁷. They are used as accelerators in vulcanization, as high pressure lubricants, as fungicides and pesticides. Dithiocarbamates are often used for the synthesis of transition metal complexes⁸⁻¹². The dithiocarbamates themselves, dithiocarbamate – metal complexes are used in agriculture for controlling insects and fungi¹³. in the treatment of alcoholism etc.,. The dithiocarbamate ligands reported in only the dithiocarboxy group as the ligand group, so they behave like bidentate ligands in complexes. Dithiocarbamates have been found to act as uninegative bidentate ligands, coordinating through both sulphur atoms, and both tetra and hexa-coordinated complexes of many transition metal ions have been isolated¹⁴⁻¹⁵. The wide range of applications of dithiocarbamates¹⁶⁻²⁰ prompted the author to prepare the bidentate ligand of 2-Amino-2ethyl 1,3 propanediol as its sodium salt and to explain the detail investigation leading to the complexation of these ligand with Cu (II), Ru (III) metals.

MATERIALS AND METHODS

All materials used in this investigation were purchased from Sigma/Aldrich and AR (Merck). Solvents used were of reagent grade and purified before use by the standard methods. Cu[AEPDDTC], Ru[AEPDDTC] complexes were prepared by the procedures described in the literature. The dithiocarbamate metal complexes of Copper (II), Ruthenium(III) were soluble in dimethyl formamide (DMF). Infrared spectra of the metal complexes were recorded on a Perkin – Elmer IR 598 spectrometer (4000 – 200 cm^{-1}) using KBr Pellets. ^1H NMR spectra were recorded on av – 400 MHz NMR spectrometer in IISC, Bangalore in DMSO-d₆ solvent. The ESR spectrum of copper complex was recorded by using JEOL, JES-FA200 ESR Spectrometer, HCU, Hyderabad. Thermogravimetric analyses of the metal complexes were carried out by using the Perkin Elmer TA-System in Dept. of Chemistry, S.K. University, Anantapur. The electronic spectra were recorded on a Thermo Spectronic Heylos α Spectrophotometer.

Preparation of Sodium salt of dithiocarbamate ligands: 0.05mol of amine was dissolved in 10ml of absolute alcohol in a clean beaker which was placed in ice. To this cold solution 5ml of sodium hydroxide (10N) solution was added and then

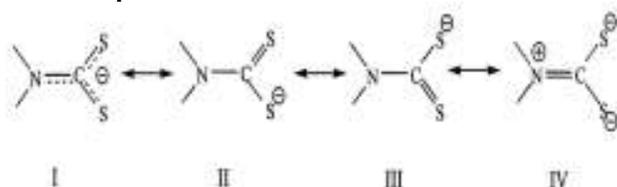
pure carbon disulphide (3.02ml, 0.05mol) was added in drop wise through separating funnel with constant stirring. The components were stirred mechanically for about 30min, sodium salt of dithiocarbamate precipitated out. It was dried and recrystallised from methanol.

Synthesis of Dithiocarbamate Metal Complexes

The aqueous solution of 0.005mol of metal salts was added with constant stirring to an aqueous solution of 0.01 sodium dithiocarbamate ligand. The reaction mixture was stirred at room temperature for 8 hours. The colored precipitate was obtained. The precipitate was filtered and washed with water and then with methanol and dried over calcium chloride in desiccators. All the complexes were prepared in 1:2 ratios of metal to ligand.

RESULTS AND DISCUSSION

Infrared spectral studies.



A single sharp band at 998.10 cm^{-1} was assigned to the stretching vibrations of the C-S bond. The band at 3418 cm^{-1} associated with the ν (N-H) and (O-H) stretching vibrations. The characteristic absorption band is appear in between the region of $1300\text{--}800\text{ cm}^{-1}$ (C-C, C-O, C-N).

I.R. Characterization of Metal Complexes

The interpretation of IR spectra of dithiocarbamate complexes of Transition metals had arisen considerable interest both diagnostically to determine the mode of co-ordination and as a mean of assessing the nature of bonding in these complexes. The Infrared spectrum of Cu (II) and Ru (III) complexes were compared the [AEPDDTC] ligand. The typical IR Spectrum of [AEPDDTC] complexes presented in Fig.1(b) and 1(c).

A strong band exhibited at 1463 cm^{-1} in the I.R spectrum of the ligand, which was assigned to the Thioureide bond was shifted to $1463\text{--}1529\text{ cm}^{-1}$ region. The presence of single band at $1016\text{--}1033\text{ cm}^{-1}$ region was assumed to ν (C-S) stretching vibrational mode and it indicates the symmetric bidentate behaviour of the ligand that means the AEPDDTC ligand was coordinated through both the sulphur atoms.

Along with these bands new bands were formed which are not observed in the spectrum of the ligand, the band in the resin $351\text{--}420\text{ cm}^{-1}$ was assigned to the ν (M-S) metal ligand bond of the complex. The appearance of a broad band at $3410\text{--}3443\text{ cm}^{-1}$ can be assigned to the ν (O-H) stretching vibrations of coordinated water molecules present in their complexes. In the spectra of both ligand and complex, no significant change were observed, for ν (N-H) stretching mode, indicating non-involvement of amino nitrogen in Co-ordination. The two weaker bands at $800\text{--}750\text{ cm}^{-1}$ and $750\text{--}720\text{ cm}^{-1}$ were assigned respectively to -OH rocking and wagging vibrations of co-ordinated water in the complex.

I.R. Analysis of the Ligand

The typical I.R spectrum of AEPDDTC dithiocarbamate was presented in the Fig.1(a). As concern the 2-Amino-2ethyl 1,3 propanediol, two main regions of the I.R. are of main interest the most significant bands recorded in the FT-IR spectra of the ligand and its metal complexes are reported in the Table 1.

First, the $(1451\text{--}1550\text{ cm}^{-1})$ region, which was primarily associated with ν (N-CSS) stretching vibrations. Second, the $950\text{--}1000\text{ cm}^{-1}$ region, which is associated with ν (C-S) stretching vibrations. The characteristic band at 1455.61 cm^{-1} , was assignable to ν (N-CSS); this band defines a carbon Nitrogen bond order between a single bond ($\nu = 1250\text{--}1350\text{ cm}^{-1}$) and a double bond ($\nu = 1640\text{--}1690\text{ cm}^{-1}$). The appearance of a band in that region indicates that, of the three possible resonance structures reported by Chart et al., characterized by a strong delocalization of electrons in the dithiocarbamate moiety.

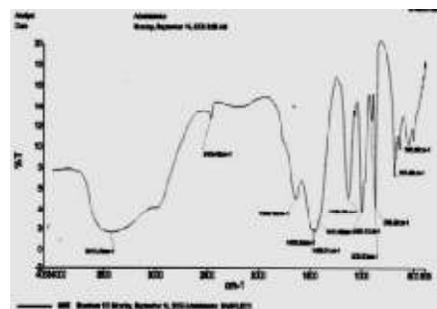


Figure 1(a): IR Spectrum of the AEPDDTC ligand

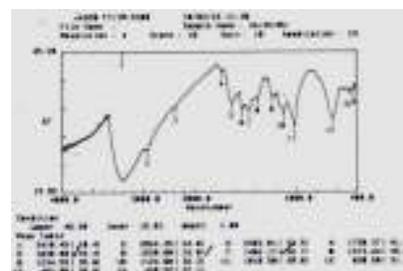


Figure 1(b): IR Spectrum of the Cu [AEPDDTC]

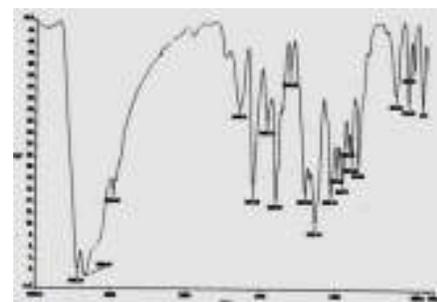


Figure 1 (c): IR Spectrum of the Ru [AEPDDTC]

¹HNMR Spectral Studies of AEPDDTC and its Metal complexes

Fig.2(a). gives the typical NMR spectrum of the ligand AEPDDTC. The important chemical shift values of the ligand and metal complexes are summarised in table 2. Methyl proton of the Ethyl group of Dithiocarbamate forms a triplet at 0.63 to 0.67 ppm, methylene protons of ethyl substituan

appears as quadrates at 1.2 to 1.33 ppm. The singlet appeared in the region 3.9 ppm was due to proton in the hydroxyl group. The singlet formed at 1.65 to 1.74 ppm was due to the (-CH₂) group in the propane maintain. The signal due to -NH in the Thioureide forms a broad singlet in the region 7.1 ppm. Fig.2.(b). to Fig.2.(c). shows the typical NMR spectrum of metal complexes. In the complexes signal due to proton bonded to Nitrogen in the Thioureide bound was observed in the 7.23 to 8.0 ppm. The down field shift of the complex may attributed to an increase of the Π - bond character and the delocalization of electron along the C-N bond contributed by the substituents and also by the bidentate nature of the

dithiocarbamate ligand . On complexation, the electron density on -NH decreases, the processional frequency of proton bonded to Nitrogen increases, hence the singal in shifted to down field regions . High NH stretching indicates as increased C-N double bond character which is due to a greater electron density on the -NCCS moiety .

The broad signal in the range 9.4 to 10.62 ppm in the case of copper Metal complex indicates the complexation of water molecule to metal ion, it was not observed in the case of free ligand.

Table 1: The Important IR bands of the Ligand and its Metal complexes

Compound	-OH (water)	Thioureide Bond	ν (C-S)	ν (M-S)
AEPDDTC	3418	1463	998	-
(AEPDDTC) ₂ Cu	3443	1551	1033	351
(AEPDDTC) ₂ Ru	3410	1529	1016	420

Table 2: ¹HNMR Spectrum of the ligands and its metal complexes in DMSO-d₆ in ppm

Compound	H-N-C (thiureide bond)	(OH) Co-ordinated water
AEPDDTC	7.25	-
(AEPDDTC) ₂ Cu	7.23	10.62
(AEPDDTC) ₂ Ru	7.9	10.4

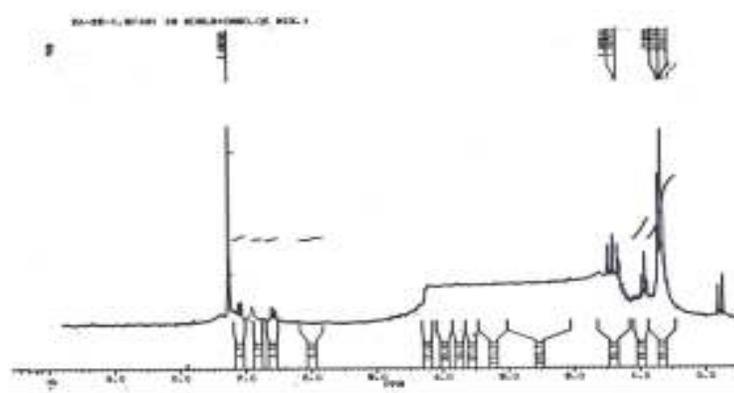


Figure 2(a): ¹HNMR Spectrum of the AEPDDTD ligand in DMSO d₆ solvent

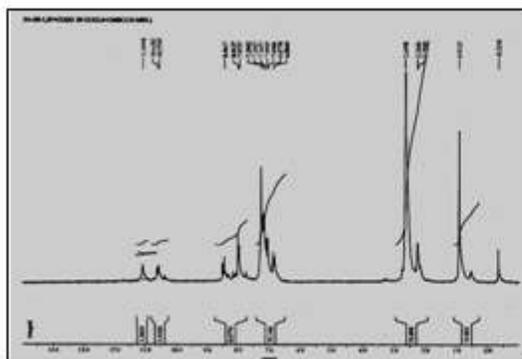


Figure.2 (b): ¹HNMR Spectrum of the Cu [AEPDDTD]

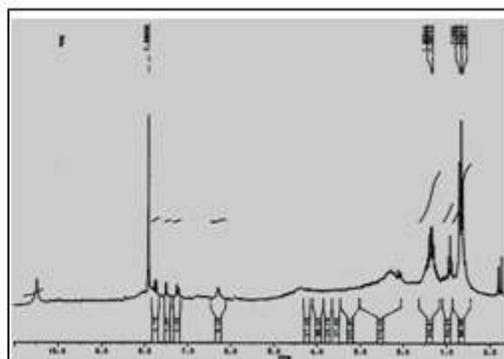


Figure 2(c): ¹HNMR Spectrum of the Ru

UV – Visible Spectral Studies:

The solution electronic spectra of the ligand and the complexes were recorded in DMF as solvent in the UV-Visible region. The electronic transition data was given in the Table.3.

Fig.3(a). shows the electronic spectra of the sodium 2-amino-2-ethyl 1,3 propanediol dithiocarbamate, it shows an intense absorption bands at 274 nm and 293 nm. These bands may be due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of N-C=S group and S-C-S. On complexation these bands were shifted. In all the complexes, bands below 300 nm were attributed to the intra ligand transitions.

The Electronic spectrum of the Cu-AEPDDTC complex was shown in the Fig.3(b). In Copper complex two intense bands observed at 267 nm and 332 nm were assigned to the intra molecular charge transfer of the ligand ($\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ in the N-C-S group). The weakest band can be attributed to an $d \rightarrow d$ metal orbitals transitions. In particular this broad band can be assigned to the $d_{x^2-y^2} \rightarrow d_{xy}$ and $d_{x^2-y^2} \rightarrow d_{xz, yz}$. The electronic configuration of Cu (II) is $[Ar] 3d^9$. In an octahedral crystal field, the corresponding ground state

electronic configuration was $t_{2g}^6 e_g^3$ which yields 2E_g term.

The excited electronic configuration, $t_{2g}^5 e_g^4$ corresponds to ${}^2T_{2g}$ term. Thus only one single electronic transition ${}^2E_g \rightarrow {}^2T_{2g}$ was expected in the octahedral crystal field thus only one single electron transition expected in an octahedral crystal field. Based on the above data the structure of the complex was tentatively observed as octahedral tendency.

The Electronic spectrum of the Ru-AEPDDTC complex was shown in the Fig.3(c). The Ruthenium complex shows two intense bands observed at 270 nm and 320 nm intramolecular charge transfer of ligand ($\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ in the N-C = S group). The complex shows two less intense broad bands in the high wave length region corresponding to intraligand and $d \rightarrow d$ metal orbitals transitions. In particular this broad band can be assigned to the $d_{xy} \rightarrow d_{x^2-y^2}$ and $d_{xz, yz} \rightarrow d_{x^2-y^2}$ transitions. A moderately intense peak observed at 352 nm may be due to the ligand-metal charge transfer [LMCT].

Table 3: UV-Vis spectral data of the ligand and its metal complexes

Compound	λ_{max}/nm	Possible assignment
L = AEPDDTC	274	$\pi \rightarrow \pi^*$
	293	$n \rightarrow \pi^*$
[Cu(L) ₂ (H ₂ O) ₂]	267, 332	$\pi \rightarrow \pi^*$
	388	LMCT
	536	d-d
[Ru(L) ₂ (H ₂ O) ₂]	270, 320	$\pi \rightarrow \pi^*$
	351	LMCT
	480, 625	d-d

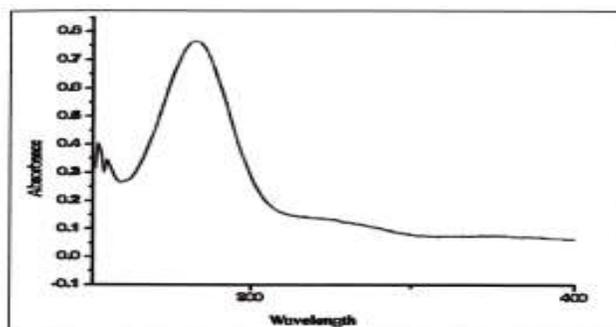


Fig 3(a): UV-Vis Spectrum of the [AEPDDTC] Ligand

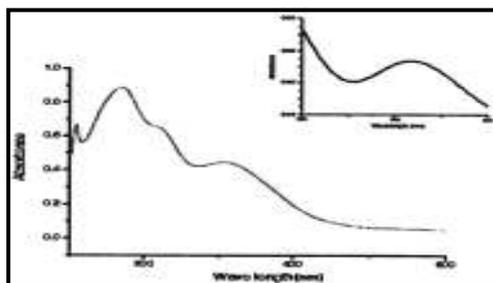


Figure 3(b): UV-Vis Spectrum of the Cu [AEPDDTC]

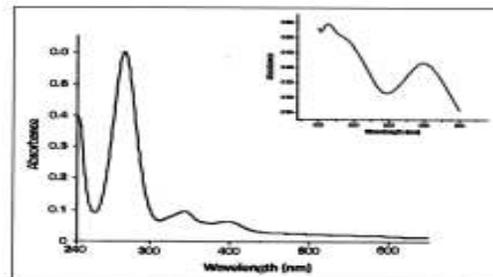


Figure 3(c): UV-Vis Spectrum of the Ru [AEPDDTC]

ESR Spectral analysis of Cu [AEPDDTC] metal complex

ESR spectra of metal complexes give useful information regarding the stereochemistry and nature of metal–ligand bonding. The ESR spectra of the complex in polycrystalline state exhibit only one broad signal, which was attributed to dipolar broadening and enhanced spin–lattice relaxation. ESR spectra obtained for copper complex in DMF at liquid nitrogen temperature and representative ESR spectrum of Cu (II) ion complex are presented in Fig.4. In this low temperature spectrum, four peaks of small intensity have been identified which are considered to originate from ($g_{||}$, g_{\perp} , $A_{||}$ and A_{\perp}) were determined from the intense peaks of the spectrum. Kivelson & Neiman have reported that $g_{||}$ value is less than 2.3 for covalent character and it is greater than 2.3 for ionic character of the metal–ligand bond in complex. Applying this criterion, the covalent bond character can be predicted to exist between the metal and the ligand for complex.

The trend $g_{||} > g_{ave} > g_{\perp} > 2.0023$ observed for the complex suggests that the unpaired electron was localized in $dx^2 - y^2$ orbital of the copper (II) complex. The lowest g value (>2.04) also consistent with a $dx^2 - y^2$ ground state. The $g_{||} / A_{||}$ quotient ranges was 111.58 cm^{-1} , evidence in support of the octahedral geometry without any distortion.

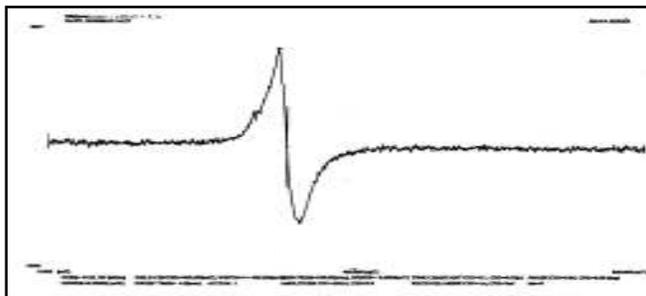
The axial symmetry parameter G values was calculated by using Kneuh's method by using the expression, $G = g_{||} - 2 / g_{\perp} - 2$ and related to the exchange interactions between copper – copper centers. According to Hathway, if the G value was greater than four, the exchange interaction was negligible indicating the monomeric nature of complex. For the copper complex the $G=4.148$ indicates the formation of monomeric complexes.

The ESR parameters $g_{||}$, g_{\perp} , $A_{||}^*$, and A_{\perp}^* of the complex and the energies of d–d transitions were used to evaluate the orbital reduction parameters ($K_{||}$, K_{\perp}). The molecular orbital coefficients or the bonding parameters α^2 (in plane σ -bonding) and β^2 (in plane π -bonding) were calculated. If the $\alpha^2 = 0.5$, it indicates a complete covalent bonding, while the value of $\alpha^2=1.0$ suggests a complete ionic bonding. The observed α^2 value for the present chelate 0.112 indicates that the complex was exhibiting some covalent character.

The dipolar interaction term (P) which takes into account the dipole–dipole interaction of the electron moment with the nuclear moment. The Fermi constant interaction term (K) indicates the interaction between the electronic and the nuclear spins given by the expression $K=A_0 / (P-\Delta g_0)$, where ($\Delta g_0 = g_e - g_0$), it represents the amount of unpaired electron density at the nucleus and K was the independent property of the central ion. The observed $K_{||} < K_{\perp}$ indicates the presence of significant in plane π -bonding. Giordano and Bereman suggested the identification of bonding groups from the values of dipolar term P , reduction of P values from the free ion value (0.036 cm^{-1}) might be attributed to the strong covalent bonding. The lower P and α^2 values for Mn and Cu [AEPDDTC] complex suggest the presence of strong in-plane π bonding in agreement with higher ligand field. The shape of ESR lines, ESR data together with the electronic spectral data suggest octahedral geometry for manganese complex.

Table 4: Spin Hamiltonian and orbital reduction parameters of Cu complex in DMF solution

Parameters	Cu (AEPDDTC) ₂
$g_{ }$	2.1796
g_{\perp}	2.0433
g_{ave}	2.088
G	4.1480
$A_{ }^*$	0.0203
A_{\perp}^*	0.0095
A_{ave}^*	0.0131
$K_{ }$	0.1068
K_{\perp}	0.1772
P^*	0.0126
α^2	0.12204



* Values are given as cm^{-1} units.

TG techniques were employed to follow the thermal behavior of complexes. According to the results obtained, the complexes were not volatile and their decomposition occurs in more than one step. The typical Thermogram of complexes were shown in the Fig.5(a) and Fig.5(b). Thermogravimetric studies of the complexes confirmed their proposed molecular formulae. The thermal decomposition of metal complexes had been followed up to 1000°. The decomposition behavior of the complexes was observed in nitrogen atmosphere.

The Copper complex shows three main decomposition stages, and the first stage was due to endothermic loss of water molecules coordinated to the metal. The Second step was the Exothermic decomposition of the ligand moiety forming stable intermediate $M(SCN)_2$ at around 140.23 – 251.2°C, the decomposition of this intermediate occurs at the third stage between 650.43–852.3°C.

The thermogram of the Mn and Ru complex exhibit, First stage of decomposition around 148 to 150°C, which indicates the presence of coordinated water molecules and this decomposition corresponds to small endothermic dehydration of the complex and giving anhydrous complex. The second decomposition stage with successive sub steps with two (or) three exothermic peaks corresponds to the degradation of ligand moiety into stable $M(SCN)_2$ intermediate around 200°-422°C. This on further decomposition forms corresponding MnS as the final decomposition product at a high temperature i.e. 730.46°– 900.42° C. All the experimental mass loss and total mass loss percentage values found were presented in the Table. 5.

Table 5: Thermal Analytical Data of metal complexes

Complex X=H ₂ O	Temperature range in °C	Probable assignment	Mass loss (%)	Total mass loss (%)
CuL ₂ X ₂ L= C ₆ H ₁₂ NO ₂ S ₂	95.26-136.23	Loss of 2H ₂ O molecules	7.75	82.13
	140.23-251.2	Decomposition of L	54.26	
	650.43-852.3	Formation of CuS	20.12	
RuL ₂ X ₂ L=C ₆ H ₁₂ NO ₂ S ₂	87.42-148.24	Loss of 2H ₂ O molecules	6.56	85.28
	200.23-422.34	Decomposition of L	52.26	
	735.46-900.42	Formation of RuS	26.46	

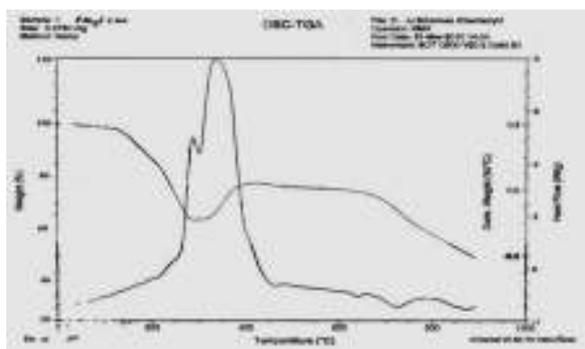


Figure 5(a): Thermogram of the Cu [AEPDDTD]

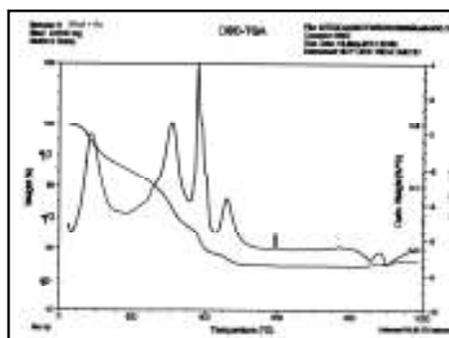


Figure 5(b): Thermogram of the Cu [AEPDDTD]

ANTIBACTERIAL ACTIVITY

Method Employed

To prove the antibacterial activity of the ligand and metal complexes on bacteria, paper disc diffusion procedure was employed.

Medium

Nutrient agar medium was employed for the testing.

Peptone (5g) was dissolved in a liter of distilled water. Beef extract (5g) was added to the peptone solution and agar (15g) was mixed. The contents were mixed thoroughly and the pH was adjusted to 7.4–7.6. The solution was sterilized in the autoclave for 10–15 minutes at 15 lbs. per sq. inch pressure, to prove the same.

Testing Equipment

Petri dishes, hot air oven, autoclave, sterilized pipettes, spreader, suitable Whatman filter paper and incubator.

Organisms selected for testing : *Escherichia coli*, *Klebsiella*, *Bacillus subtilis*

Maintenance and Sterility

All the required apparatus was sterilized before use and every reasonable precaution was taken to avoid contamination throughout the operation. Procedure of Testing The solutions of the synthesized compounds were prepared at different concentrations viz 50ppm, 100ppm concentrations DMF solvent. Few sterilized filter paper (Whatman) discs soaked in the solvent were used for preparing test solution. These sterilized paper discs were soaked in the 5ml of the solution of known concentration of the corresponding test samples, for 2–3 minutes. Sufficient time was allowed for the solvent to evaporate from the paper discs. Sterilized nutrient agar 15–20ml was poured into Petri dishes under aseptic conditions. The bacterial culture was poured over the solidified surface of the nutrient agar in Petri dishes and spread evenly for uniform distribution with spreader. Paper discs soaked with the solution of known concentration of the desired test samples were placed under aseptic conditions at a distance in each Petri plates, containing known bacterial suspension. These Petridishes were labeled with the compound number and the

incubated for 24 hours at 25–30°C in incubator. After 24 hours of incubation each Petri plate was observed for bacterial growth. Later the zone of inhibition of bacterial growth in each petri plate was measured. The discs soaked with the respective solvents of test solution were used as controls. The bacterial growth in the test Petri plates was compared with the growth in the controls. Then zone of inhibition of bacterial growth in the Petri plates under examination were measured.

The present investigation was an attempt to find out the antibacterial activity of ligand and their metal complexes against *Escherichia coli*, *Klebsiella* and *Bacillus subtilis* in the range 50–100 urn/ml. Choosing serial paper disc diffusion method. The Antibacterial activity results were given in the Table.6. the results of the biological activity of the metal complexes indicated the following facts.

The high antimicrobial activities of all the newly synthesized metal complexes surmounting that of ligands showed that complexation of the organic moiety to the metal ions substantially enhanced their activities such increased activity of metal chelates had been explained by Overtones concept and the Tweedy's chelation theory.

On chelation the polarity of the metal ion reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of metal ion with donor groups. It was further noted that the delocalization of EI–electrons over the whole chelate ring enhanced the lipophilicity of the complexes. This increased lipophilicity enhanced the penetration of the complexes into lipid membrane and blocking the metal binding sites on enzymes of microorganism thus retards the normal cell processes.

Table 6: Antibacterial activities of ligands and their transition metal complexes (Zone formation in mm)

Compound	<i>Escherichia coli</i>	<i>Klebsiella</i>	<i>Bacillus subtilis</i>
AEPDDTC	10	9	10
(AEPDDTC) ₂ Cu	12	12	13
(AEPDDTC) ₂ Ru	13	12	13

CONCLUSION

By concluding the above data 2-Amino-2ethyl 1,3 propanediol act as good complexing agent towards transition metal ions. by using all the above mentioned analytical data it was concluded that they behave as symmetric bidentate ligand during complexation. The investigations revealed that the complexes are better anti-bacterial agents than the ligands.

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Source of support: Nil, Conflict of interest: None Declared