

Synthesis and Spectral Identification of Some Transition Metal Complexes with New Ligand of Dithiocarbamate

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Abstract

New dithiocarbamate ligand Ammonium(2,4-dichloro phenyl-N-dithio carbamate (DMePDTC) was prepared by reaction of between 2,4-dichloro aniline with carbon disulfide (CS₂) in the presence of ammonium hydroxide solution. Five new metal complexes of chloride salts of Fe(III),Co(II),Ni(II),Cu(II) and Zn(II) ions were prepared. The structure of dithiocarbamate ligand and resulting complexes were investigated using spectral and analytical means available such as elemental analysis (C.H.N.S), determination of metal content M%, molar conductivity measurements, magnetic moments measurements, infrared and electronic spectra. The potential structures of complexes have been identified by finding the molar ratio of the relation (metal:ligand) which the study indicates as 1:2 [M:L] for all complexes expected Zn(II)-complex is 1:1[M:L] where study. Depending on the results which have been reached at we can propose the interstitial forms of the prepared complexes where the Co(II) and Ni(II) complexes take an octahedral geometry with sp³d² hybridization and suggested square planar geometry of Cu(II)-complex with dsp² hybridization and suggested tetrahedral geometry of Zn(II)-complex with sp³ hybridization while square pyramidal geometry of Fe(III)-Complex with sp³d hybridization.

Keywords: Dithiocarbamate, metal chelate complexes, characterization.

Introduction

The compounds which consist of sulfur atom especially dithiocarbamate ligands their complexes with transition elements have their distinctive commercial value in addition to their biological activity vividness and this attracts the researcher to study them chemically and investigate their properties⁽¹⁻⁴⁾. Many of the complexes of the transition elements prepared with dithiocarbamate ligands have wide application in different fields in medicine, they are used for making antifungal drug⁽⁵⁻⁷⁾ and in the agricultural field, they are used as pesticides for agricultural pests, weeds, jungles and insects⁽⁸⁾. The dithiocarbamate ions (R₁R₂NCS₂) are considered as soft base and it is one of the important ligands in the coordination chemistry⁽⁷⁾. Mostly the ligands of such compounds behave as bidentate and they may be symmetric or asymmetric (i.e. unequal bond participation for the two sulfur atoms of the same group)⁽⁹⁾. In addition to such behavior a bidentate ligand of dithiocarbamate ligands as monodentate is also specified and the reason behind such behavior is the steric selective disability around the central metal atom^(10,11). The dithiocarbamate ligands are considered very important from a chemistry point of view because of the variety of its uses and because they work on installing a wide range of oxidation states for different transition elements and they increase the stability of the resulting complexes^(12,13). In this work we describe the synthesis and spectral characterization study of some transition metal complexes with a new ligand (DMePDTC) derived from dithiocarbamate.

Experimental

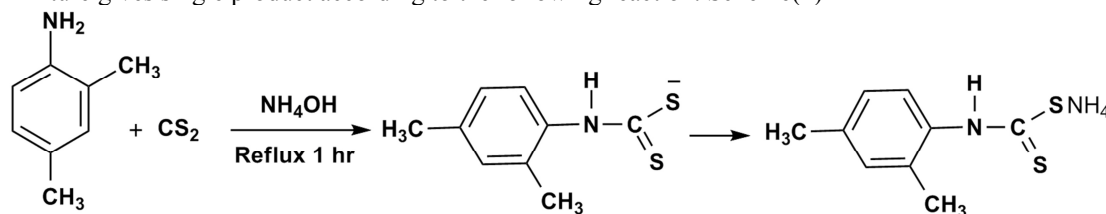
Materials and measurements

All chemicals and solvents were obtained from commercial sources and used as received without further purification. Melting points were determined by open capillary tube method and are reported in °C by using a Stuart melting point 9300. The elemental analysis of the dithiocarbamate ligand and their complexes were carried out on C.H.N.O.S EA 300 elemental analyzer by the central laboratory of the University of Kufa. IR spectra were recorded as KBr discs by using Shimadzu FTIR 8400 spectrophotometer in the range (4000-400) cm⁻¹. The electronic spectra of the prepared ligand and their complexes were recorded in the region (200-1100) nm by using absolute ethanol as solvent (10⁻³ M) at 25°C using Shimadzu UV-vis. 1700 spectrophotometer with 1.000±0.001 cm⁻¹ match quartz cell. The metal contents of the complexes were measured by using atomic absorption by Shimadzu AA-6300 spectrophotometer. Magnetic susceptibility measurements of complexes were determined by Faraday method at room temperature using Balance magnetic (MSB-Mki) apparatus and diamagnetic corrections were made by Pascal's constants. Molar conductivity of the chelate complexes were determined at room temperature in DMSO (10⁻³ M) using a 31 A digital conductivity meter. The pH measurements were carried out using a Philips PW 9421 pH meter ±0.001. Experiments and preparation of solutions and washing all glassware were using distilled water to twice in doubly distilled water.

Preparation of the dithiocarbamate ligand (DMePDTC)

The dithiocarbamate ligand (DMePDTC) was prepared through the reaction of 2,4-dimethyl aniline 1.23

gm,(0.01 mol) with carbon disulfide(CS_2)with existance of ammonium hydroxide below 8°C .The resulting mixture was left under reflux for 1 hour and allowed to stand overning and concentrated (rotary evaporator).The formed crystals product was separated by filtration and washed several times by ether and it left to be dried purified by recrystallized from hot absolute ethanol and dried over anhydrous CaCl_2 .The reaction mixture gives single product according to the following reaction. Scheme(1)



Scheme 1;-preparation of the DMePDTC ligand

Preparation of complexes

The metal complexes were prepared by adding 0.479 gm,(0.002 mole)of ligand (DMePDTC) dissolved in hot ethanol(30ml) and added dropwise with stirring to the a ppropriate amount of 1:2 [M:L] molar ratio(0.001mol) for Fe(III),Co(II),Ni(II) and Cu(II) hydrate metal chloride and anhydrous Zn(II)(1:1) M:L chloried salt dissolved in the same solvent 30 ml .The rusltng mixture was refluxed for 1 hour .After refluxing with stirring at least for 30 min,colored precipitates formed at room temperatuer, the rusltng solids were filtered off washed with 10 ml aqueous alcohol mixture to remove any traces of the unreacted starting materials and air dried recrystallized from ethanol and dried in the oven at 70°C for several hrs.

Results and Discussion

The new dithiocarbamate ligand (DMePDTC) is crystals but the prepared metal complexes of this ligand vary in colour depending of the metal ions .The complexes are stable in air and unsoluble in water but its soluble in most organic solvents and giving stable solutions at room temperature.Some physical and analytical data are listed in Table 1.In all complexes the molar ratio are 1:2[M:L]exception the Zn(II)-complexes 1:1[M:L].All complexes are non-electrolytes in solution .The purity of ligand were tested by TLC techniquis and C.H.N.Selemental analysis.

Table(1): Some physical properties and elemental analysis for the prepared ligand (DMePTC) and their complexes .

Compound	m.p $^\circ\text{C}$	pH	Color	Yield (%)	Molecular formula (Mol.Wt)	Found (Calc.) %				
						C	H	N	S	M
LNH_4 =ligand (DMePDTC)	160	6.0	Orange	84	$\text{C}_9\text{H}_{14}\text{N}_2\text{S}_2$ (215.3)	50.42 (50.21)	6.57 (6.55)	13.06 (13.27)	29.91 (30.10)	-----
$[\text{Fe}(\text{L})_2\text{Cl}]$	198	6.5	Green	67	$\text{C}_{18}\text{H}_{20}\text{N}_2\text{S}_4\text{ClFe}$ (483.89)	44.67 (44.48)	4.16 (4.05)	5.78 (5.97)	26.50 (26.68)	11.54 (11.30)
$[\text{Co}(\text{L})_2(\text{H}_2\text{O})_2]$	189	7.5	Greenish blue	72	$\text{C}_{18}\text{H}_{24}\text{N}_2\text{S}_4\text{O}_2\text{Co}$ (487.57)	44.33 (44.09)	4.95 (4.82)	5.74 (5.98)	26.30 (26.12)	12.08 (11.85)
$[\text{Ni}(\text{L})_2(\text{H}_2\text{O})_2]$	191	7.0	Green	79	$\text{C}_{18}\text{H}_{24}\text{N}_2\text{S}_4\text{O}_2\text{Ni}$ (487.35)	44.35 (44.53)	4.95 (4.86)	5.74 (5.98)	26.31 (26.18)	12.08 (12.32)
$[\text{Cu}(\text{L})_2]$	187	6.5	Purple	64	$\text{C}_{18}\text{H}_{20}\text{N}_2\text{S}_4\text{Cu}$ (456.16)	47.39 (47.51)	4.41 (4.32)	6.14 (6.38)	28.11 (27.90)	13.93 (13.67)
$[\text{Zn}(\text{L})(\text{H}_2\text{O})\text{Cl}]$	208	7.0	Dark purple	81	$\text{C}_9\text{H}_{12}\text{NS}_2\text{OClZn}$ (315.17)	34.29 (34.06)	3.83 (3.74)	4.44 (4.61)	20.34 (20.59)	20.75 (20.43)

Metal:Ligandratio

The metal:ligand ratios[M:L] of chelats complexes were determined by the method molar ratio at the wavelength of maximum absorption (λ_{max}).The solutions of prepared complexes increase the intensity of the colours as approach point of at in constant of solution^(14,15).A 1:2[M:L] mole ratio suggested the formation of $[\text{Fe}(\text{DMePDTC})_2\text{Cl}]$ and $[\text{M}(\text{DMePDTC})_2(\text{H}_2\text{O})_2]$ where M=Co(II) and Ni(II) but $[\text{Cu}(\text{DMePDTC})_2]$ of Cu(II)-complexes while 1:1[M:L] mole ratio of $[\text{Zn}(\text{DMePDTC})(\text{H}_2\text{O})\text{Cl}]$.

Infrared spectra

The important of infrared bands of free ligand and their complexes with the some transition elements are given in Table2.The strong band in the spectrum of free ligand at 3325 cm^{-1} due to $\nu(\text{N-H})$ remained unaffected after complexation⁽¹⁶⁾.Two absorption bands (medium and weak) hadbeen observed at 3132 cm^{-1} and 2954 cm^{-1} in ligand spectrum which are due to $\nu(\text{C-H})$ aromatic and aliphatic respectively .These bands are stable in position and intensity for both free ligand and metal complexes .The ligand shows its characteristic $\nu(\text{C-N})$ band at 1527 cm^{-1} .The position of this band remained at nearly the same frequency in spectra of the metal complexes which

may be explained by nonparticipation in complex⁽¹⁷⁾. The spectrum of free ligand shows two absorption bands at 1226cm⁻¹ and 1126cm⁻¹ due to $\nu(\text{C}=\text{S})$, $\nu(\text{C}-\text{S})$ respectively⁽¹⁸⁾. These bands are a little change in shape and shifted to lower frequencies^(1,19) in the prepared complexes spectra. These differences suggest the linkage of metal ion with sulfur atom. The spectra of chelate complexes showed new bands in the region (520-470)cm⁻¹ these bands did not present in the spectrum of ligand may be attributed to vibration $\nu(\text{M}-\text{S})$ provide evidences concerning the bonding of sulfur to the metal ions^(20,21).

Table (2):-Selected IR bands of the dithiocarbamate ligand (DMePDTC) and their complexes (cm⁻¹)

Compound	$\nu(\text{N}-\text{H})$	$\nu(\text{C}-\text{H})$ or.	$\nu(\text{C}-\text{H})$ al.	$\nu(\text{C}-\text{N})$	$\nu(\text{C}=\text{S})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{S})$
LNH ₄ =ligand (DMePDTC)	3325 s.	3132 m.	2954 w.	1527 s.	1226 m.	-----	-----
[Fe(L) ₂ Cl]	3339 m.	3118 m.	2942 m.	1539 s.	1232 s.	598 w.	511 vw.
[Co(L) ₂ (H ₂ O) ₂]	3353 m.	3099 m.	2950 m.	1545 s.	1245 m.	592 w.	501 w.
[Ni(L) ₂ (H ₂ O) ₂]	3341 m.	3061 s.	2960 w.	1540 vs.	1241 m.	589 w.	509 w.
[Cu(L) ₂]	3338 m.	3109 m.	2969 m.	1537 s.	1236 s.	576 w.	520 w.
[Zn(L)(H ₂ O)Cl]	3347 br.	3050 w.	2980 w.	1538 s.	1237 m.	575 w.	513 vw.

W=weak , S=stronge ,m= medium ,Vs=very strong,br=broad

Electronic absorption spectra and magnetic moments studies

Solution electronic spectra of the ligand and their complexes were recorded in absolute ethanol solution. The magnetic moment values measured at room temperature. The electronic spectral bands and magnetic moments of the complexes are listed in Table 3 and Figures 4,5,6 and 7. The free ligand (DMePDTC) spectrum gives two bands at 369 nm (27100cm⁻¹) and 299 nm (33445cm⁻¹) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition in the N-C-S and S-C-S chromophores of the dithiocarbamate moiety

Fe(III)-complex

The electronic spectrum of this complex show two bands at 610 nm (16393 cm⁻¹) and 463 nm (21598 cm⁻¹), which suggests the existence of ${}^6\text{A}_{1g} \rightarrow {}^6\text{T}_{1g(p)}(v_1)$ and ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g(G)}(v_2)$ transitions with a square-based pyramidal spatial configuration⁽²²⁾. The magnetic moment value of Fe(III)-complex is 5.79 B.M this value refers to the fact that the complex has a high spin octahedral structure due to a presence of five electrons unpaired ($t_{2g}^3 e_g^2$) and hybridization $sp^3 d$ ^(22,23).

Cobalt (II)-Complex

The electronic absorption spectrum of Co(II)-complex show three absorption bands at 895 nm (11173cm⁻¹), 605 nm (16583 cm⁻¹) and 478 nm (20920cm⁻¹) there are assigned to ${}^4\text{T}_{1g(F)} \rightarrow {}^4\text{T}_{2g(F)}(v_1)$; ${}^4\text{T}_{1g(F)} \rightarrow {}^4\text{A}_{2g(F)}(v_2)$ and ${}^4\text{T}_{1g(F)} \rightarrow {}^4\text{T}_{1g(p)}(v_3)$ transitions respectively. The magnetic moment values for the Co(II)-complex have been used as criterion to determine the type of coordination around the metal ion. The magnetic moment value of this complex equal 4.58 B.M which correspond to three unpaired electrons which may suggest a distorted octahedral geometry (Z-out) and hybridization $sp^3 d^2 (t_{2g}^5 e_g^2)$ for the Co(II)-complex in the high-spin state⁽¹⁵⁾.

Nikel(II)-complex

The electronic spectrum of Ni(II)-complex were recorded in absolute ethanol (10⁻³M). This complex show three absorption bands at 928 nm (10775cm⁻¹), 650 nm (15384 cm⁻¹) and 590 nm (16949 cm⁻¹) which are assigned to ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{2g(F)}(v_1)$, ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g(F)}(v_2)$ and ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g(p)}(v_3)$ transitions respectively with an octahedral spatial configuration. The Ni(II)-complex gave a magnetic moment value of 3.08 B.M because of presence of two unpaired electrons which may suggest high-spin irregular octahedral geometry ($t_{2g}^6 e_g^2$) and hybridization $sp^3 d^2$ ^(14,22).

Copper(II)-complex

The visible spectrum of the Cu(II)-complex exhibit one broad band at 543nm (18416 cm⁻¹) which may be assigned to ${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}$ transition corresponding to square planar geometry around the Cu(II) ion. The magnetic moment value of Cu(II)-complex found to be 1.78 B.M due to one unpaired electron which offers the possibility of square plane geometry ($t_{2g}^6 e_g^3$) and dsp^2 hybridization⁽²⁴⁾.

Zn(II)-complex

The electronic spectrum of Zn(II)-complex do not show any d-d transition. The band at 519nm (19268 cm⁻¹) which could be attributed to the presence of a charge transfer (M \rightarrow L, CT). The magnetic moment for this complex found to be zero and electronic configuration $d^{10} (t_{2g}^6 e_g^4)$ to be diamagnetic character ($\mu_{\text{eff}}=0.00$ B.M) and a tetrahedral geometry and hybridization sp^3 ^(25,26). The obtained magnetic moment values show the presence of a paramagnetic phenomena for both Fe(III), Co(II), Ni(II) and Cu(II) complexes and diamagnetic character for Zn(II)-complex.

Table(3):- Electronic Spectra (in nm and cm^{-1}),magnetic moments, geometry and hybridization

Complexes	λ_{max} (nm)	Absorption bands (cm^{-1})	Transition	μ_{eff} (B.M)	Geometry	Hybrdization
LNH ₄ =ligand (DMePDTC)	369	27100	$n \rightarrow \pi^*$	-----	-----	-----
	299	33445	$\pi \rightarrow \pi^*$			
[Fe(L) ₂ Cl]	610	16393	${}^6A_{1g} \rightarrow {}^6T_{1g(g)}(v_1)$	5.79	Square based pyramidal	sp^3d (high spin)
	463	21598	${}^6A_{1g} \rightarrow {}^4T_{1g(g)}(v_2)$			
Co(L) ₂ ((H ₂ O) ₂)	895	11173	${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}(v_1)$	4.58	Octahedral (distorted,Z-out)	sp^3d^2 (high spin)
	603	16583	${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}(v_2)$			
	478	20920	${}^4T_{1g(F)} \rightarrow {}^4T_{1g(F)}(v_3)$			
[Ni(L) ₂ (H ₂ O) ₂]	928	10775	${}^3A_{2g} \rightarrow {}^3T_{2g(F)}(v_1)$	3.08	Octahedral (regular)	sp^3d^2 (high spin)
	650	15384	${}^3A_{2g} \rightarrow {}^3T_{1g(F)}(v_2)$			
	590	16949	${}^3A_{2g} \rightarrow {}^3T_{2g(g)}(v_3)$			
[Cu(L) ₂]	543	18416	${}^2E_g \rightarrow {}^2T_{2g}$	1.78	Square planer	dsp^2
[Zn(L) (H ₂ O) Cl]	519	19268	M→L,CT	Dia	teterahedral	sp^3

L =(DMePDTC)

Calculation of the metal complexes stability constant

The calculation of stability constants of complexes in solution are determined spectrophotometrically by measuring the absorbance of mixture solution of both metal ion and ligand at fixed wavelength(λ_{max}) and optimum pH values .The degree of formation of the complexes is calculated base on the relationship^(27,28), $\beta=(1-\alpha/4\alpha^3C^2)$ for 1:2[M:L] metal complexes of Fe(III),Co(II),Ni(II) and Cu(II) ions , and $\beta=1-\alpha/\alpha^2C$ for 1:1[M:L] for Zn(II)-complex and $\alpha=(A_m-A_s/A_m)$,where A_s and A_m are the absorbance of the partially and fully formed complexes respectively at obtimum concentration .The calculated β and $\log\beta$ values for prepared complexes are listed in Table 3.

Table(3):-Stability constant values (β and Log β),molar conductivity, optimal concentration ,maximum wavelength(λ_{max}) and molar absorptivity(ϵ) of chelate complexes.

Ligand	Metal ion	Optimal molar conc. x 10 ⁻⁴ M	Optimal wave length λ_{max} (nm)	Molar absorptivity (ϵ)x10 ³ L.mol ⁻¹ .cm ⁻¹	Moler Conductivity (S.cm ² .mol ⁻¹)	Stability constant β (L ² .mol ⁻²)	Log β
LNH ₄ =DMePDTC $\lambda_{\text{max}}=369\text{nm}$ $\epsilon=6.6 \times 10^3 \text{L.mol}^{-1} .\text{cm}^{-1}$ Conc.=1.50x10 ⁻⁴ M	Fe(III)	1.75	610	5.71	6.01	1.07x10 ⁹	9.02
	Co(II)	2.00	603	3.26	8.62	1.10x10 ¹⁰	10.04
	Ni(II)	1.50	590	10.66	10.22	1.05x10 ¹⁰	10.02
	Cu(II)	2.25	543	1.35	12.47	1.06x10 ⁹	9.05
	Zn(II)	2.00	519	2.56	9.13	1.00x10 ⁹	9.00

Conductivity measurement

All chelate complexes prepared in this work showed conductivity values ranged between (6.01-12.47)S.mol⁻¹.cm² in DMSO at room temperature.The results proved in the Table 3, this study has shown the values of molar conductivity of all metal complexes that non-electrolyte and no conductive species exist^(29,30) According to there results and discussed through different techniques suggest below the proposed the structural formula of chelate complexes prepared and shown in Figures 1,2,3 and 4.

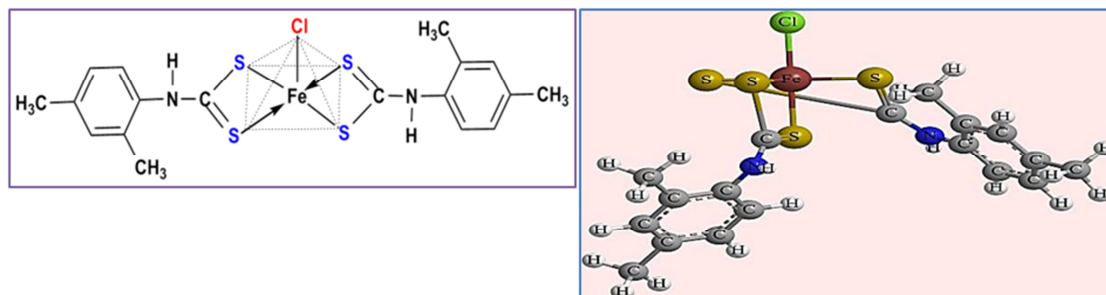
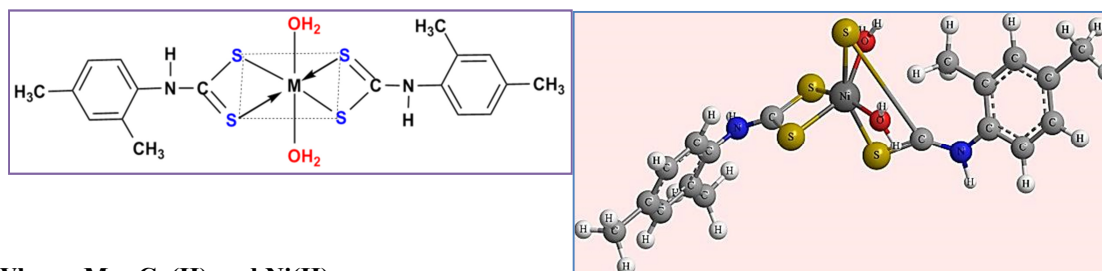


Figure (1): The proposed structural formula of Fe(III) complex.



When $M = \text{Co(II)}$ and Ni(II)

Figure (2): The proposed structural formula of Co(II) and Cu(II) complexes.

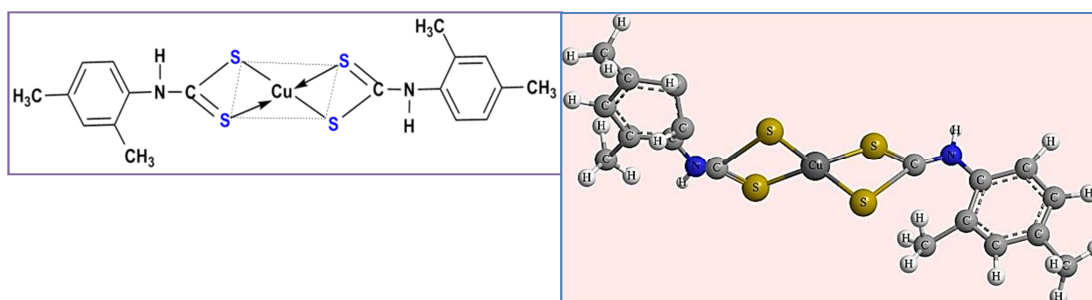


Figure (3): The proposed structural formula Cu(II) complex.

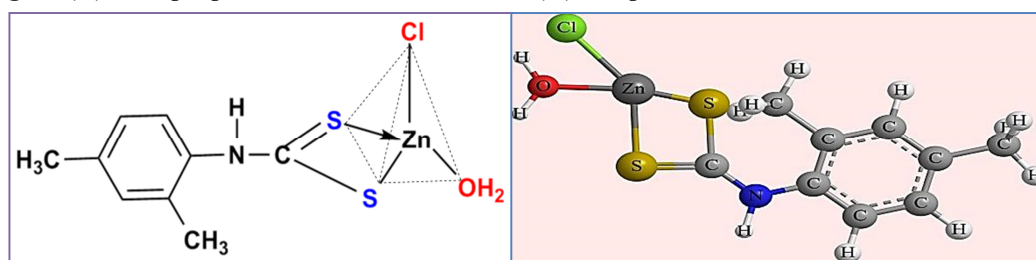


Figure (4): The proposed structural formula Zn(II) complex.

Electrostatic potential(E.P) and frontier molecular orbital

The electrostatic potential describes the interaction of energy of the molecular system with a positive point charge of the ligand were plotted as two dimensions and three dimensions contours to investigate the reactive sites of the molecular(DMePDTC).The electrostatic potential of ligand show that S atom more reactive than other atoms .Also one can interpret the stereochemistry and rates of many reactions involving soft electrophiles and nucleophiles in terms of the properties of frontier orbital(HOMO and LUMO) overlap between HOMO and LUMO is a governing factor in many reactions .The HOMO and LUMO value were plotted as three dimensions counter to get more information about these molecules .The results show that the LUMO of transition metal ion prefers to react with HOMO of donor atoms in the prepared ligand,Figure5.

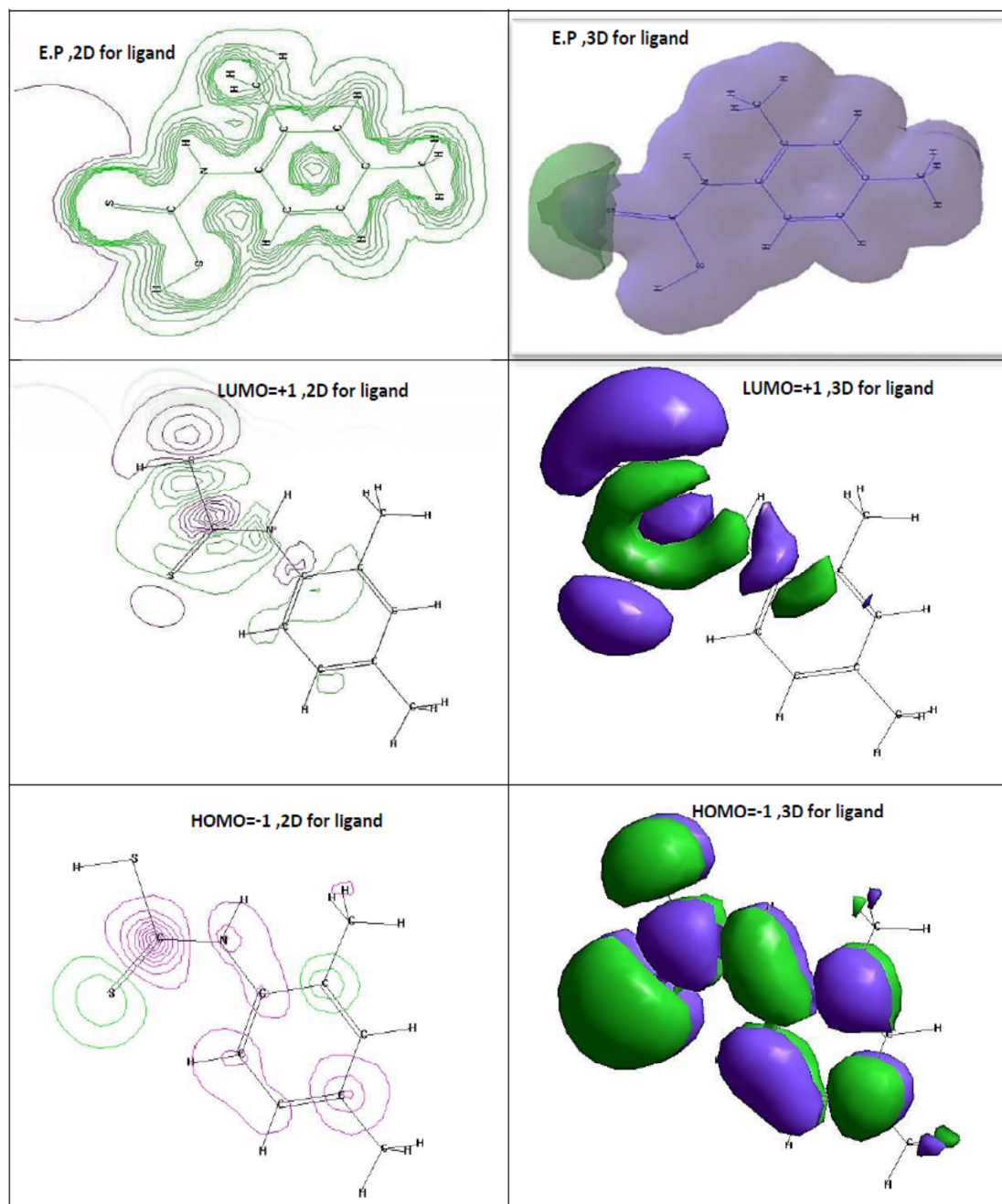


Fig.(5):- LUMO,HOMO and electrostatic potential as 2&3D counters for ligand

Conclusion

In this paper we reported the preparation and spectral identification of dithiocarbamat ligand (DMePDTC) and their metal complexes with Fe(II),Co(II),Ni(II),Cu(II) and Zn(II) ions .The ligand (DMePDTC) exhibit a bidentate coordination mode in the Fe(II),Co(II),Ni(II) and Cu(II) but monodentate coordination mode in the Zn(II) – complex .All complexes characterized solid metal complexes not effect by air ,light and moisture suggesting high stability .The geometry is proposed for Co(II) and Ni(II) are octahedral stereo chemistry and square –based pyramidal of Fe(III)-complex while Cu(II)-complex is square planer geometry with the dithiocarbamate ligands (DMePDTC)and tetrahedral stereo chemistry of Zn(II) –complex .All metal complexes are non-electrolyte and non-ionic .

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