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Synthesis and spectroscopic study of new ligand 3-(acetylthioureido)propanoic acid with their metal complexes

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***Abstract*---**A new ligand 3-(3-acetylthioureido)propanoic acid (ATA) was prepared through the reaction of acetyl chloride, ammonium thiocyanate and β- alanine. For this study the following methods were used: FT-IR, ^{1}H-NMR ^{13}C-NMR spectra and the Elemental analysis. complexesis of M^{+2}=Mn,Cu,Ni,Co,Zn,Cd and Hg derived from3-(3-acetylthioureido) propanoic acid have been synthesized and characterized by elemental analyses, molar conductance, magnetic suscpility, FT-IR, UV Vis spectra, The results have shown complexation with β-alanine derivative with metal ion in a 1:2 ratio. The spectral characterization confirms the formation of ligand and complexes. The evidences from the above results suggest the ligand to be abi dentate ion.

***Keywords*---**beta alanine, acetyl isothiocyanate, metal complexes.

Introduction

β-Alanine (3-aminopropionic acid) is the only naturally occurring β-type amino acid. Although it is not incorporated into proteins, it has important physiological functions in the metabolism of animals, plants and microorganisms[27]. Amino acids, their derivatives and products of their cyclization are important in the synthesis of biologically active compounds, for example pharmaceuticals, protecting agents of field plants and plant regulators. N Substituted B-alanine have been widely studied for many different purposes because of their biological activity, for example as growth stimulators of agricultural crops [1], [2][8], Amino acids play central roles both as building blocks of proteins and as intermediates

in metabolism. They are classified as essential and non-essential amino acids, which is an amino acid in which the amino group is attached to the β-carbon (the carbon two atoms away from the carboxylate group) instead of the more usual α-carbon for alanine (α-alanine). Amolecule that helps to buffer acid in muscles, increasing physical performance. In addition to the above key factor, β-alanine also exhibits neurotransmitter activity, by activating glycine and GABA (gamma-aminobutyric acid) receptors [3], and promotes anti-glycation effects because it increases carnosine levels in the body.. β - alanine plays a protective role involving preservation of enzyme structure and function. It suppresses heat-induced LDH inactivation, prevented LDH (Lactate dehydrogenase) aggregation, and reactivated thermally denatured LDH[4].. It can also act like an osmolyte, and may be able to help with proper protein folding. Carnosine appears to be an anti-oxidant and anti-aging complexesis. Recently amino acid Schiff base complexes are largely studied for DNA cleavage property as they can be used for cancer therapy. [9] [7] Zn(II) and Cd(II) coordination with β-Alanine diamondoid frameworks possessing second-order nonlinear optics propertiesChunying. Also Synthesis, crystal growth and characterization of novel semiorganic nonlinear optical crystal: Dichloro(beta-alanine)cadmium(II) [5],also study to investigate complexation of L-carnosine and its constituent amino acids β-alanine and L-histidine with copper (II)[6] The aim of this work is prepare new ligand 3-(3-acetylthioureido)propanoic acid (ATA) and it's metal complexes with Mn^{+2}, Co^{+2}, Ni^{+2}, Cu^{+2}, Zn^{+2}, Cd^{+2}, and Hg^{+2}.

Materials and Methods

(Acetyl chloride), (Beta alanine) (Fluka), Manganese chloride tetrahydrate ($MnCl_2.4H_2O$), Cobalt chloride hexahydrate ($CoCl_2.6H_2O$), Nickel chloride hexahydrate ($NiCl_2.6H_2O$), Copper chloride dehydrate ($CuCl_2.2H_2O$), Zinc chloride ($ZnCl_2$ Cadmium chloride hydrate ($CdCl_2.H_2O$) and Mercury chloride ($HgCl_2$). All reagents were annular or chemical pure grade by BDH, Merck and Fluka.

Instruments

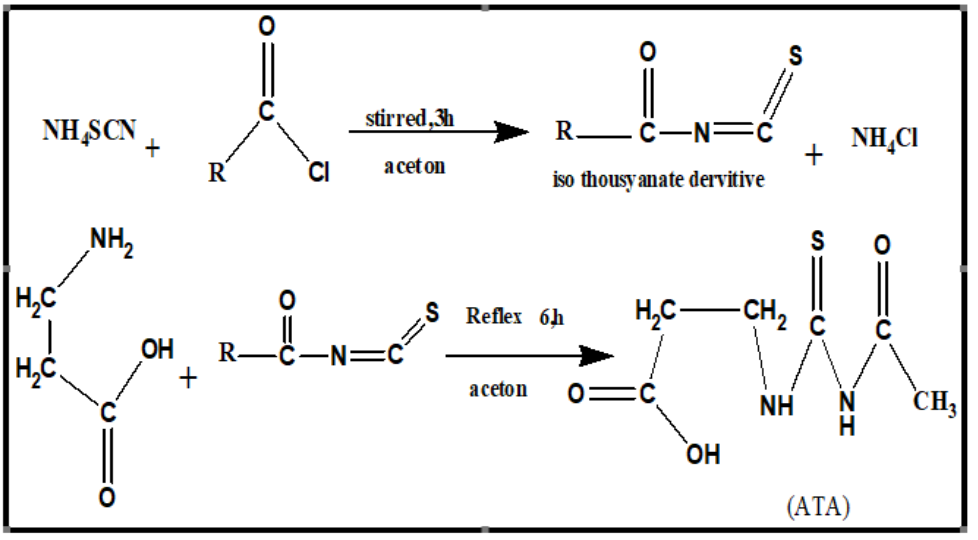
^1H and ^{13}C-NMR was recorded using Varian-Inova 500 MHz USA at Tehran University / Central Laboratory of the College of Science and using DMSO-d_6 and TMS as aggregates to determine the zero point . Melting point was recorded by using Stuart- melting point apparatus. FT-IR spectra were recorded as KBr disc using 3800 Shimadzu in the range of 4000-400 cm^{-1}. Electronic spectra were obtained using UV-160 Shimadzu spectra photometer at 25 °C in ($1x10^{-3}$) M DMSO. Conductivity was measured by using Philips Pw. Digital. Elemental analysis (C.H.N.S) have been determined for prepared ligand, also a few of their complexes at Tehran University / Iran utilizing Elementary Germany Eager 300EA1112 device. Magnetic susceptibility measurements were obtained by balance magnetic susceptibility by model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique by using Shimadz (AA680G).

Preparation of (ATA)

1. Preparation of the (Acetyl isothiocyanate)

Mixture of acetyl chloride (1.86 ml, 1mmol) and ammonium thiocyanate (2g, 1mmol) in (25ml) of acetone was stirred under refluxed for 3 hours and then filtered, the filtrate was used for further reaction[10].

2. Preparation of 3[3-(acetylthioureido)propanoic acid(ATA)
   Dissolved (2.344g, 26 mmoll) of the B-alanine in (25 ml) acetone, then filtered the former solution above B-alanine ,refluxed the mixture for(6 hours) and let it to dray. the resulting solid was collected, washed with acetone and re crystallization from ethanol. Scheme (1),Yield (60%), (m.p =185-187 °C),.(Scheme(1) . The product is Yellow, it was recycled with ethanol , C % found (37.76) calc.(37.886), H % found (5.26) calc.(5.26),N% found (14.48) calc.(14.73), S % found (16.47) calc.(16.838) see scheme (1).



Scheme (1): Preparation of ligand (ATA)

Synthesis of complexes with (ATA) ligand :
Dissolved (0.12g, 2mmol) of KOH in (25) ml aceton, then added (0.385g, 2mmol) of (ATA) ligand to former solution and setting the PH between (7-8), then adding metallic salt solution (1mmol in 25 ml aceton), and mixed them and stirred to 3 hours, then filtering the product washed with mixture (1:1) of (water:ethanol) the solution of following metal salts $MnCl_2.4H_2O$ (0.2g, 1mmole), $CoCl_2.6H_2O$ (0.24g, 1mmole), $NiCl_2.6H_2O$ (0.24g,1mmole), $CuCl_2.2H_2O$ (0.2g, 1mmole), $ZnCl_2$ $CdCl_2.H_2O$ (0.2g,1mmole), and $HgCl_2$ (0.3g,1mmole) in aceton, were added dropwise to the solution of(ATA^-K^+). The precipitate formed immediately after stirring the mixture at room temperature for 3hours. The precipitate was collected by filtration, washed with distilled water and ethanol and dried under vacuum. Physical properties were given in Table (1).

Results and Discussion

The solid complexes were soluble in some common solvent such as dimethyl formamide, dimethyl sulphoxide, and relatively thermally stable. The molar

conductivity of all complexes in DMSO were found to be non-electrolyte, Table (1) includes the physical properties for (ATA) and its metal complexes.

Table (1): Physical properties of (ATA) and its metal complexes

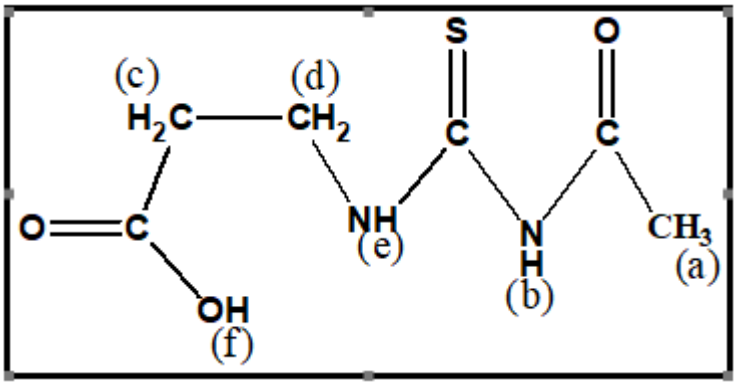
Compund	M.wt (gm/mol)	Color	M.p °C or dec.	M% Calculation (Found)	Molar condu. Ohm^{-1}Cm^{2}mol^{-1}	μ_{eff} (B.M)
(ATA)$C_6H_{10}N_2O_3S$	190.04	Yellow	185-187	-	1.50	-
[Mn(ATA)$_2$]	433.018	brown	250 d	12. 470 (12.42)	116.91	5.87
[Co(ATA)$_2$]	437.01	Blue	230 d	13.50 (13.8)	33.55	4.95
[Ni(ATA)$_2$]	436.773	Yellow	225 d	13.44 (12.61)	118.64	3.29
[Cu(ATA)$_2$]	441.62	brawn	285 d	14.40 (14.70)	112.32	1.75
[Zn(ATA)$_2$]	443.45	Yellow	260 d	14.74 (13.65)	115.44	Dia
[Cd(ATA)$_2$]	490.48	yellow	225 d	22.92 (22.55)	111.21	Dia
[Hg(ATA)$_2$]	578.68	brown	280 d	34.67	114.77	Dia

^{1}H -NMR Spectral of ligand (ATA):-

^{1}H- NMR Spectral of ligand (ATA) Fig (1) in (DMSO-d_6) showed the following signals , doublet peak at δ (1.88) ppm for (3H , CH_3 , Methyl) and singal peak at δ (2.04) ppm for (NH Amine) , pent let peak at δ (2.43) ppm as (3.74) ppm for (2H , CH_2 , Methylene) , singlet peak at δ (2.51 - 2.50) ppm for (DMSO) , singlet peak at δ (7.92) ppm for (NH , Amide) , singlet peak at δ (11.14) ppm for (OH , COOH)[11] table(2).

Table (2) (^{1}H-NMR) Spectral data for ligand (ATA)

Compound	Functional croup	δ ppm
Ligand (ATA)	CH_3 , Methyl	1.88
	NH , Amine	2.04
	CH_2 , Methylene	2.43
	CH_2 , Methylene	3.74
	NH Amide	7.92
	COOH	11.14



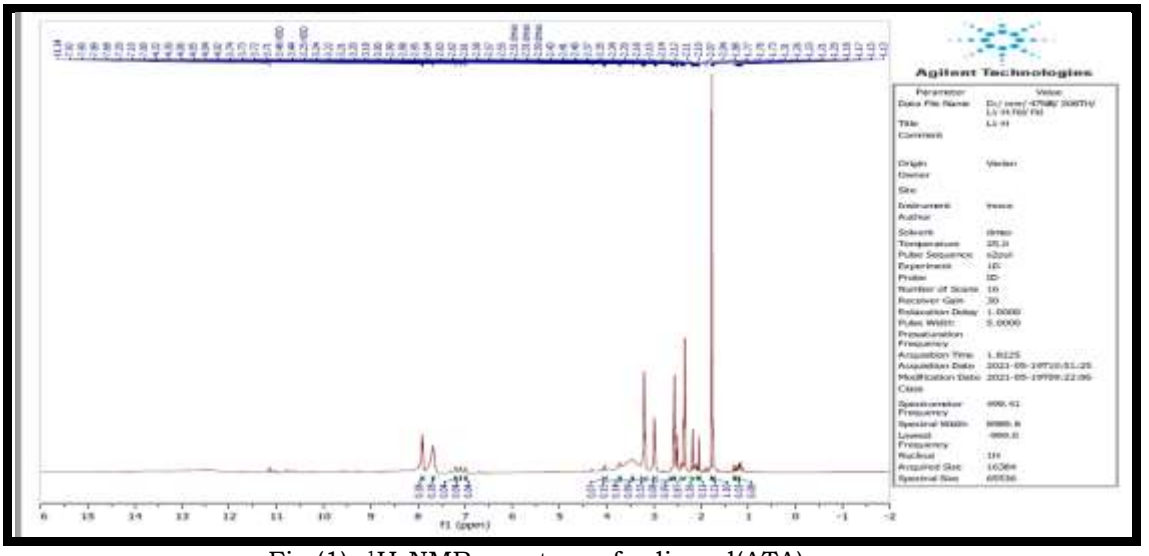


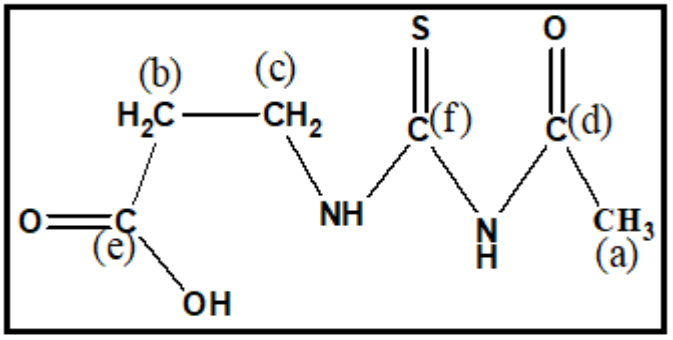
Fig (1): ^{1}H-NMR spectrum for ligand(ATA).

^{13}C- NMR Spectral of ligand (ATA)

^{13}C- NMR Spectral of ligand (ATA) Fig (2) in (DMSO - d6) showed the following signals , singal peak at δ (22.95) ppm for (CH_3),singal peak at δ(34,32) ppm for (CH_2) for (Carbon in CH_2 Aliphatic) and also singal peak at δ (40.95) ppm for (Carbon in CH_2 Aliphatic),singal peak at δ (172.35) ppm for (C=O) , singal peak at δ (172.40) ppm for (COOH) and singal peak at δ (173.36) ppm for (C=S)[14] table(3):

Table (3) C^{13}NMR Spectral data for ligand (ATA)

Compound	Functional croup	δ ppm
Ligand (ATA)	CH_3 Methyl	22.95
	CH_2 Methylene	34.32
	CH_2 , Methylene	40.37
	C=O , Amide	172.35
	COOH	172.40
	C=S	173.36



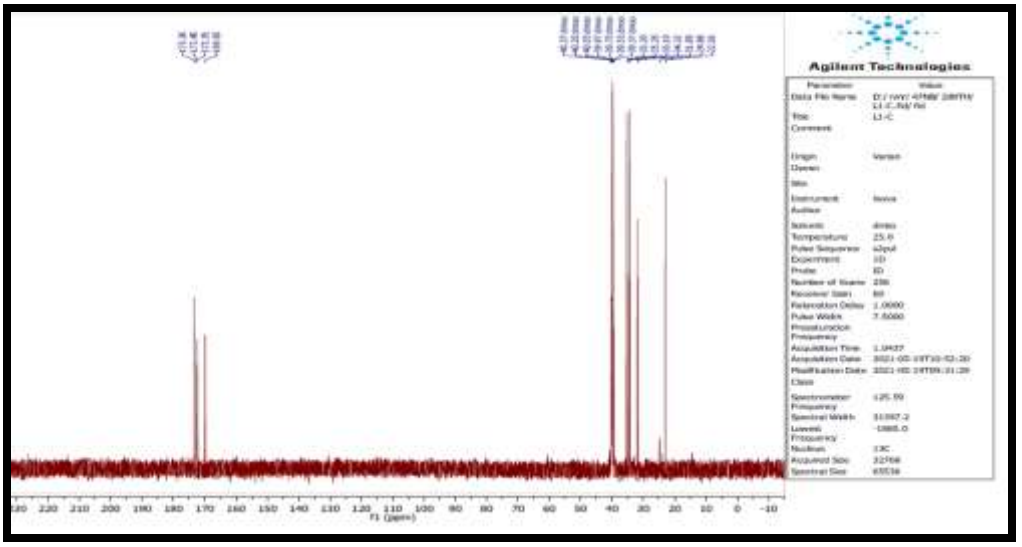


Fig (2): ^{13}C-NMR spectrum for ligand(ATA)

FT-IR spectral for ligand (ATA)

The important infrared spectra data of ligand (ATA) Fig(3) and it's complexes are given in table (4), FT-IR spectrum of the free ligand (ATA) showed bands due to ν(O-H) and ν(N-H) which absorbed (3005) $cm^{-1}$and (3101) cm^{-1}, $\nu(COO^-)_{asym}$, $\nu(COO^-)_{sym}$, ν(C=O amide) and ν(C=S) which absorbed at (1716)cm^{-1}, (1373)cm^{-1}, (1627)cm^{-1} and (1226)cm^{-1}[19] respectively.

FT-IR spectral of ligand (ATA) complexes

The FT-IR spectra of all prepared complexes exhibited marked difference between bands belonging to the stretching vibration of $\nu(COO^-)_{asym}$ in the range between ν(1554-1582)cm^{-1} shifted to lower frequencies by (162-134)cm^{-1} and $\nu(COO^-)_{sym}$ (1411-1404)cm^{-1} suggesting shifted to higher frequencies by (38-31) cm^{-1}[12][13] of the possibility of the coordination of ligand through the oxygen atom at the carbonyl group, while the band caused by ν(N-H) appeared between (3414-3336.85)cm^{-1} shifted to lower frequencies by (313-235.85)cm^{-1} which indicates to the coordination of ligand through the nitrogen atom at ν(N-H)group .The stretching vibration band ῡ(C=O amide) show change little frequencies (1612-

1639)cm^{-1} and ν(C=S) show between frequencies (1203-1261)cm^{-1}[18] there for indicating do not coordinate to the metal ion. Metal-oxygen and metal-nitrogen bonds are confirmed by the presence of the stretching vibration of ν(M-O) and ν(M-N) around (501-451)cm^{-1} and (470-405)cm^{-1} respectively[17].Table (4) describes the important bands and assignment for free ligand (ATA) and its complexes.

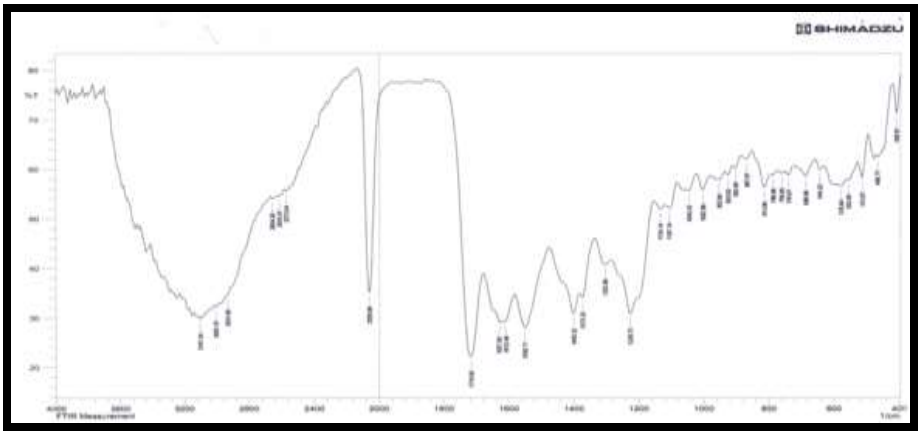


Fig (3): FT-IR for(ATA)

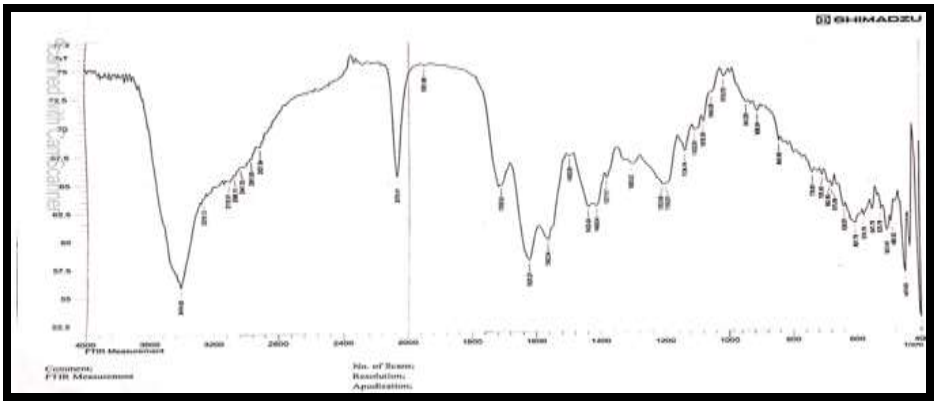


Fig (4): FT-IR for complex[Mn $(ATA)_2$]

Table (4): The characteristic infrared band for free ligand (ATA) and its metal complexes

Compound	ν(N-H)	N(Coo) Asym.	N(Coo) Sym.	Δν (COO)	ν(C=O) Amide	ν(C=S)	ν(M-O)	ν(M-N)
ATA	3101(m)	1716	1373	---------	1627	1226	---------	----------
Mn[$(ATA)_2$]	3414(m)	1582(m)	1408	174	1620(s)	1203(m)	501	447
Co[$(ATA)_2$]	3379.29	1570	1404	166	1639	1261(m)	474	443
Ni[$(ATA)_2$]	3414(m)	1558(m)	1404	154	1624(s)	1377(m)	501	470
Cu[$(ATA)_2$]	3398(m)	1554(m)	1404(m)	150	1627	1215(m)	486	432
Zn[$(ATA)_2$]	3336.85(m)	1558	1404	154	1616(s)	1226(m)	497	405
Cd [$(ATA)_2$]	3383(w)	1558	1411	147	1612	1234	451	408

$Hg[(ATA)_2]$	3398(w)	1558(m)	1404(m)	154	1624	1222(m)	489	435

b = browed, w = weak, s = strong, m = medium

Magnetic properties for the metal complexes

Magnetic moment (m_{eff}) with regard to complexes related to $Mn^{+2}(d^5)$, also $Co^{+2}(d^7)$ have been indicated (5.87)B.M, also (4.95)B.M, that in anticipated spin-only values Higher value related to m_{eff} of $Ni^{+2}(d^8)$ complex (3.29) B.M because of orbital contributions.Magnetic moment m_{eff} related to $Cu^{+2}(d^9)$complex indicated (1.75)B.M in expected value to one electro[20], all data are indicated in the Table(1).

Spectral studies

1-Ligand (ATA)

The UV-Visible spectra of yellow (ATA) and its complexes recorded in Table (5), the solution of (ATA) in 10^{-3}M (DMSO) exhibited peak at (36231) cm^{-1} Fig (5) which are attributed to$\pi \rightarrow \pi^*$transition.

2-ligand (ATA) complexes:

-[Mn $(ATA)_2$] complex:

The brown complex of Mn(II), shows bands at (36496) cm^{-1}, (28735)cm^{-1}, (14367)cm^{-1} and 12738 cm^{-1} which are caused by the electronic transfer L.F,$^6A_1 \rightarrow {}^2E_{(G)}$,$^6A_1 \rightarrow {}^4T_{2(G)}$ and $^6A_1 \rightarrow {}^4T_{1(G)}$ respectively[22].

-[Co$(ATA)_2$] complex:

The blue complex of Co (II),shows bands at (36496)cm^{-1},(2739)cm^{-1},(12857)cm^{-1} and (12195)cm^{-1} retunes to intra-ligand L.F,C.T mix with $^4A_2 \rightarrow {}^4T_{1(p)}$,$^4A_2 \rightarrow {}^4T_{1(F)}$ and $^4A_2 \rightarrow {}^4T_{2(F)}$ respectively, the repulsion parameter (B^-) value is found to be (577.9)cm^{-1},while β is equal to (0.595) [23].

-[Ni$(ATA)_2$] complex:

The yellow complex of Ni(II), shows bands at (37593)cm^{-1}, (28169)cm^{-1}, (17391)cm^{-1} and (12500)cm^{-1} retunes to reveals the following electronic transfer transition L.F, C.T,mix with $^3T_{1(F)} \rightarrow {}^3T_{1(P)}$, $^3T_1 \rightarrow {}^3A_{2(F)}$ and $^3T_1 \rightarrow {}^3T_{2(F)}$ respectively, the repulsion parameter (B^-) value is found to be (537.3)cm^{-1},while β is equal to (0.52) [24].

-[Cu$(ATA)_2$] complex:

The brown complex of Cu(II), Fig(6) shows bands at (36900)cm^{-1}, (27173)cm^{-1} , and (11312)cm^{-1}, retunes to L.F, $^2B_1g \rightarrow {}^2A_1g$ and $^2B_1g \rightarrow {}^2B_2g$ respectively[23][19].The complexes of [Zn$(ATA)_2$],[Cd$(ATA)_2$] and [Hg$(ATA)_2$] shows bonds are retunes to electronic transition L.F and C.T respectively. (36363,28985) cm^{-1}, ((36496,28968) $cm^{-1}$and (36764,28735)) cm^{-1} respectively [25] All transition with their assignments are summarized in Table (5).

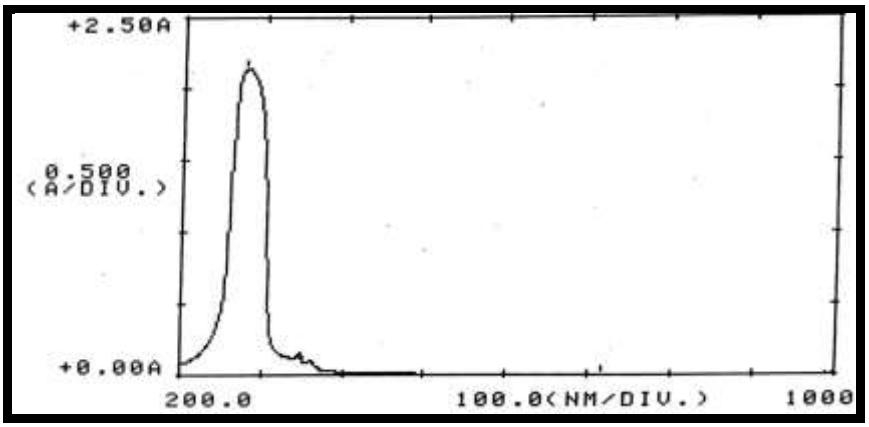


Fig. (5): Electronic spectrum of ligand(ATA)

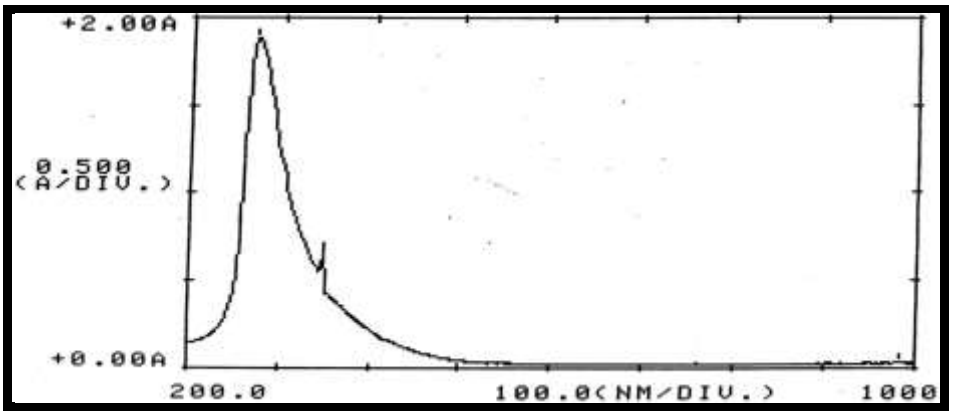


Fig. (6): Electronic spectrum of $[Cu(ATA)_2]$

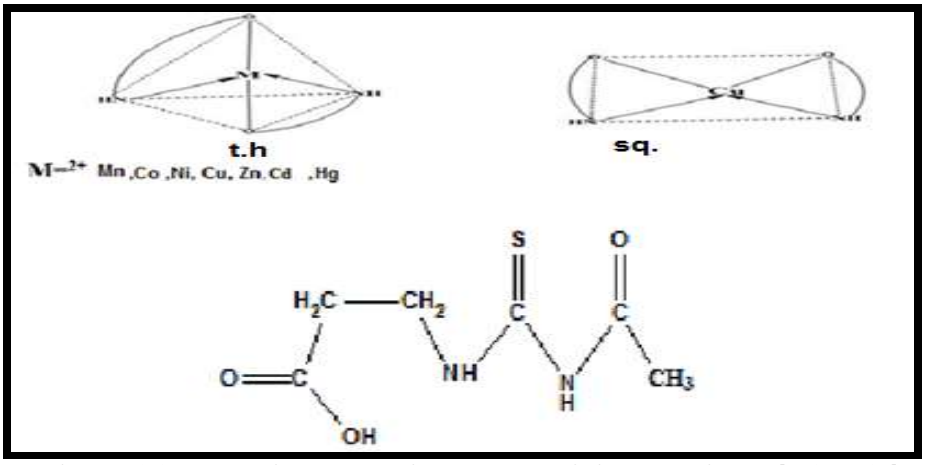
Table (5): UV-Vis Spectral Data (nm) for ligand (ATA)

Compouns	□(n)	ABC	ν-(cm)$^{1-}$	Emax	Transitions
ATA	216	2.144	36231	2144	π-π*
[Mn(AT)2]	274 348 696 785	2.069 0.659 0.025 0.080	36496 28735 14367 12738	2069 659 25 20	L.F $^6A_1 \rightarrow {}^4T2(G)$ $^6A_1 \rightarrow {}^4T_1(G)$ $^6A_1 \rightarrow {}^4T_1(G)$
[Co(ATA)2]	274 365 560 820	2.179 0.558 0.035 0.020	36496 27397 12857 12195	2179 858 35 20	L.F C.T $^4A_2 \rightarrow {}^4T_1(P)$ $^4A_2 \rightarrow {}^4T_1(F)$ $^4A_2 \rightarrow {}^4T_2(F)$
[Ni(ATA)2]	266 355 575 800	1.342 0.125 0.025 0.018	37593 28169 17391 12500	1342 125 25 18	L.F C.T $^3T_1 \rightarrow {}^3T_1$ (P) $^3T_1 \rightarrow {}^3A_2(F)$

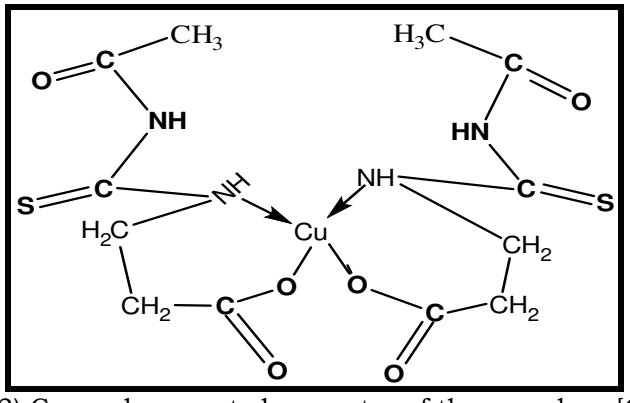
					$^3T_1 \rightarrow {}^3T_2(F)$
[Cu(ATA)2]	271 368 884	1.885 0.510 0.031	36900 27173 11312	1885 510 31	L.F $^2B_1g \rightarrow {}^2A_1g$ $^2B_1g \rightarrow {}^2B_2g$
[Zn(ATA)2]	275 345	2.182 1.128	36363 28985	2182 1128	L.F C.T
[Cd(ATA)2]	274 332	2.108 0.458	36496 28968	2108 458	L.F C.T
[Hg(ATA)2]	272 348	2.079 1.018	36764 28735	279 1018	L.F C.T

C.T = Charge transfer L.F = Ligand field

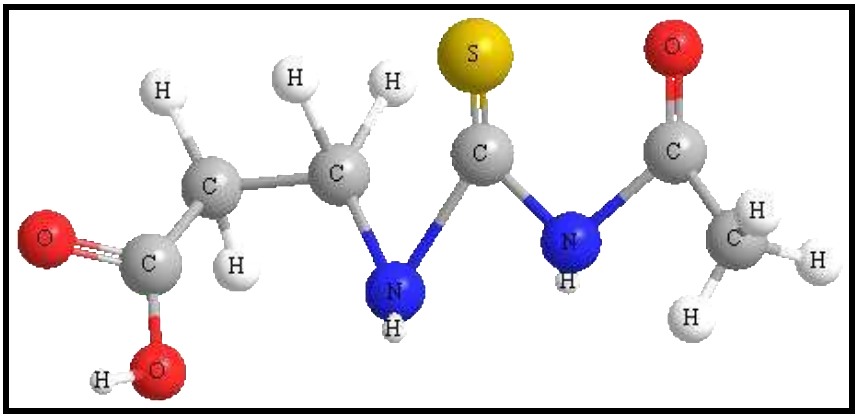
According to spectral data as well as those obtained from elemental analyses, the chemical structure of the complexes may be suggested as tetrahedral for $[M(ATA)_2]$ where M^{2+} = (Mn, Co, Ni, Zn, Cd and Hg), sch. (2) while Copper complex s has square planer[21].



Scheme (2):General suggested geometry of the complexes $[M(ATA)_2]$



Scheme (3):General suggested geometry of the complexe $[Cu(ATA)_2]$



Fig(7)3D of ligand (ATA)

Conclusion

The new ligand in the presented study was prepared through reaction from the Acetyl isothiocyanate with beta alanine, ligand has been characterized through elemental micro analysis C.H.N.S., FT-IR, UV Vis also ^{1}H,^{13}C-NMR spectra. Ligand's metal complexes have been prepared, also characterized through FTIR, UV Vis spectra, magnetic measurements, conductivity measurements, the suggested geometrical structure with regard to complexes have been tetrahedral geometryexcept for the copper complex that has square planer scheme(2),(3)showing such geometry.

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