

SYNTHESIS AND CHARACTERIZATION OF SOME NEW METALS COMPLEXES OF 2-(3-ACETYLTHIOUREIDO)-3-PHENYLPROPANOIC ACID (APA)

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ABSTRACT : A new ligand [2- (3-acetylthioureido)-3-phenylpropanoic acid (APA) is synthesized by reaction of acetyl isothiocyanate with phenylalanine (1:1). It is characterized by micro elemental analysis (C.H.N.S.), FT-IR, (UV-Vis) and ¹H and ¹³CNMR spectra. Some metals ions complexes of this ligand were prepared and characterized by FT-IR, UV-Visible spectra, conductivity measurements, magnetic susceptibility and atomic absorption. From results obtained, the following formula [M(APA)₂] where M²⁺ = Mn, Co, Ni, Cu, Zn, Cd and Hg, the proposed molecular structure for these complexes as tetrahedral geometry, except copper complex is has square planer geometry.

Key words : Phenylalanine acetyl isothiocyanate, metal complexes.

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INTRODUCTION

L-phenylalanine (LPA) is an essential α-amino acid. It is classified as non-polar because of the hydrophobic nature of the benzyl side chain. It is an electrically neutral amino acid, one of the twenty common amino acids used to biochemically form proteins, coded for by DNA. It is used in the manufacture of food and drink products and sold as a nutritional supplement for its reputed analgesic and antidepressant effects. A non-food source of phenylalanine is the artificial sweetener aspartame. LPA is the starting compound used in the flavonoid biosynthesis (Dlnelson M M Cox *et al*, 2000). The genetic disorder phenylketonuria (PKU) is the inability to metabolize phenylalanine. Individuals with this disorder are known as “phenylketonurics” and must regulate their intake of phenylalanine. Phenylalanine uses the same active transport channel as tryptophan to cross the blood–brain barrier and, in large quantities, interferes with the production of serotonin. Individuals, who cannot metabolize phenylalanine must monitor their intake of protein to control the buildup of phenylalanine as their bodies convert protein into its component amino acids (Koley *et al*, 2010; Ali, 2020) aminoacids and their

derivatives have been used for different purposes (Deepak *et al*, 2004) and some amino acid derivatives and some of their metal complexes have been evaluated as having antibacterial, antifungal properties (Chohan *et al*, 2006).

Different Schiff bases (L) ligands derived from the condensation reaction of 4-chlorobenzaldehyde and some amino acid (DL- Alanine, DL-Phenylalanine and DL-valine) have been synthesized by using microwave irradiation. Their Ni (II), Zn (II), Cd (II), Cu (II) and Co(III) complexes have been prepared. The ligands are characterized based on elemental analysis, FT-IR, mass spectrometry and ¹HNMR. Metal complexes are characterized by elemental analysis, FTIR, ¹HNMR, molar conductance and thermogravimetric (TG/DG) analysis (Bushra K Al-salami *et al*, 2017).

The synthesis and characterization of Schiff base derived from phenylalanine and furfuraldehyde in the presence of 8-hydroxyquinoline: and its complexes (OzlenAltun *et al*, 2020). The aim of this work is preparation some new metal ions complexes of [2-(3-acetylthioureido)-3-phenylpropanoic acid] (APA).

MATERIALS AND METHODS

Acetyl chloride, Phenylalanine (Fluka), Manganese chloride tetrahydrate ($\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$), Cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), Nickel chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), Copper chloride dehydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), Zinc chloride (ZnCl_2), Cadmium chloride hydrate ($\text{CdCl}_2 \cdot \text{H}_2\text{O}$) and Mercury chloride (HgCl_2). All reagents were analar or chemical pure grade by BDH, Merck and Fluka.

Instruments

^1H and ^{13}C -NMR was recorded using Ultra Shield 300 MHz Switzerland at sharif university of techonlg. 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 $4000\text{--}400\text{ cm}^{-1}$. Electronic spectra were obtained using UV-160 Shimadzu spectra photometer at 25°C in (1×10^{-3}) M DMSO. Conductivity was measured by using Philips Pw. Digital. Micro elemental analyses (C.H.N.S) were performed using acrla Erba 1106 elemental analyzer. 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 Shimadzu (AA680G).

Preparation of (APA)

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 (Kabbani *et al*, 2005).

2. Preparation of [[2-(3-acetylthioureido)-3-phenylpropanoic acid (APA) : 4.84g, 1mmol of phenylalanine in (20ml) acetone was rapidly added to the maintain vigorous reflux. After refluxing for 6 hours, the resulting solid was collected, washed with acetone and

recrystallization from ethanol (Scheme 1), Yield (80%), (m.p = $162\text{--}164^\circ\text{C}$, C% found (54.42) while calculate (54.13), H% found (5.506), while calculate (5.30), N% found (10.08) while calculate e (10.52), S% found (12.15) while calculate (12.03).

Synthesis of metal complexes

0.532g, 2 mmole of (APA) was dissolved in 25ml of ethanol containing (0.12g, 2mmole) of KOH, then the solution of following metal salts $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (0.2g, 1mmole), $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (0.24g, 1mmole), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.24g, 1mmole), $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.2g, 1mmole), ZnCl_2 , $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ (0.2g, 1mmole), and HgCl_2 (0.3g, 1mmole) in ethanol were added dropwise to the solution of $\text{APA}^- \text{K}^+$. 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.

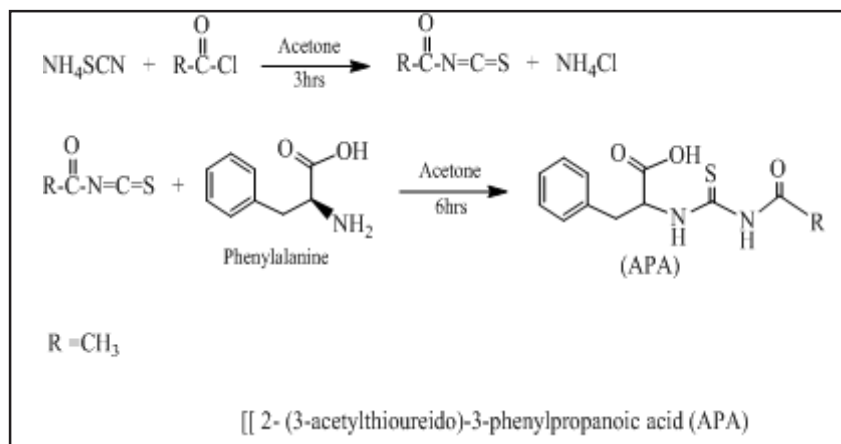
RESULTS AND DISCUSSION

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 (APA) and its metal complexes.

Spectral studies

^1H and ^{13}C -NMR spectra

1) ^1H -NMR spectrum for (APA) in DMSO as solvent, Fig. (1) showed the following signals: singlet at $\delta(1.81)\text{ppm}$ for ($3\text{H}, \text{CH}_3$), doubled at $\delta(3,10)\text{ppm}$ for ($2\text{H}, \text{CH}_2$), singlet at $\delta(2.12)\text{ppm}$ for DMSO, quartet at $\delta(3.14\text{--}3.12)\text{ppm}$ for ($1\text{H}, \text{CH}$), singlet at $\delta(7.31)\text{ppm}$ for ($1\text{H}, \text{NH sec.amine}$), multiplet at $\delta(7.13\text{--}7.19)\text{ppm}$ for (aromatic protons), singlet at $\delta(8.22)\text{ppm}$ for ($1\text{H}, \text{NH ec.amide}$), singlet at $\delta(10.91\text{--}11.35)\text{ppm}$ for ($1\text{H}, \text{COOH}$) (Gary *et al*, 2005).



Scheme 1 : Preparation of (APA).

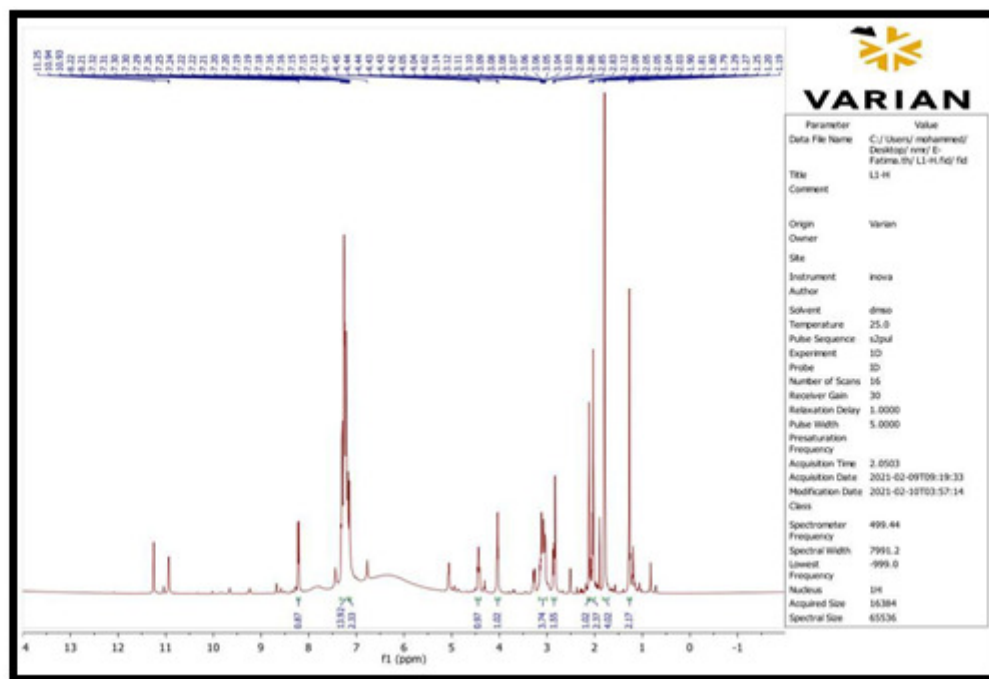

 Fig. 1 : ^1H NMR spectrum of (APA).

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

Compound	M.wt (gm/mol)	Color	M.p $^{\circ}\text{C}$ or dec.	M% calculation (Found)	Molar condu. $\text{Ohm}^{-1}\text{Cm}^2\text{mol}^{-1}$	λ_{eff} (B.M)
(APA)	266	Yellow	162-164	-	1.70	-
$[\text{Mn}(\text{APA})_2]$	584.94	Deep brown	188-190	9.23(9.11)	9.41	5.99
$[\text{Co}(\text{APA})_2]$	528.93	Deep blue	170-172	10.001(9.88)	4.6	4.82
$[\text{Ni}(\text{APA})_2]$	528.71	Deep green	173-175	9.95(9.10)	4.96	3.22
$[\text{Cu}(\text{APA})_2]$	593.54	green	178-180	10.60(10.25)	7.47	1.73
$[\text{Zn}(\text{APA})_2]$	595	Yellow	195dec	10.9(10.13)	6.27	0
$[\text{Cd}(\text{APA})_2]$	642	yellow	190-192	17.4(16.95)	6.02	0
$[\text{Hg}(\text{APA})_2]$	730.6	yellow	182-184.	27.45(26.89)	7.462	0

 Table 2 : ^1H NMR Spectral data for ligand (APA).

Compound	Functional group	$\delta(\text{ppm})$
Ligand(APA)	(3H, CH_3) s	1.81
	d(2H, CH_2)	3.10
	qu(1H,CH)	3.14-3.12
	m (aromatic protons)	7.13-7.19
	s (1H, $\text{NH}_{\text{sec.amide}}$)	7.31
	s (1H, $\text{NH}_{\text{sec.amide}}$)	8.22
	s(1H,COOH)	10.94-11.35

 Table 3 : ^{13}C NMR Spectral data for ligand (CPA).

Compound	Functional group	$\delta(\text{ppm})$
Ligand(CPA)	(CH_3) s	22.7
	s (CH_2)	36.6
	s (CH)	58.9
	s (aromatic carbons)	126-136
	s ($\text{C}=\text{O}$)	171
	s (COOH)	173
	s($\text{C}=\text{S}$)	180

2) The ^{13}C -NMR spectrum for (APA) in DMSO, Fig. (2) showed the following signals: signals at δ (22.7) ppm for CH_3 , signals at δ (36.3) ppm for (CH_2), signals at δ (39-40.11) ppm for DMSO, signal at δ (58.90) ppm for (CH), signals at δ (126-136) ppm for aromatic carbons, signal at δ (171) ppm for ($\text{C}=\text{O}$ sec.amide), signal at δ (173) ppm for (COOH), signal at δ (180) ppm for ($\text{C}=\text{S}$).

Infrared spectra

FT-IR spectrum of the free(APA), Fig. (3), showed bands due to amido $\delta(\text{NH})$, $\delta(\text{C}=\text{O})$ and $\delta(\text{C}=\text{S})$ which absorbed at $(3421) \text{ cm}^{-1}$, $(1616) \text{ cm}^{-1}$ and $(1234) \text{ cm}^{-1}$ respectively, while another absorption band appeared at $(1706) \text{ cm}^{-1}$ could be explained as $\delta(\text{COO})_{\text{asym}}$ (Silverstein *et al*, 1981; Dyer, 1965), where the $\delta(\text{OCO})_{\text{sym}}$ was

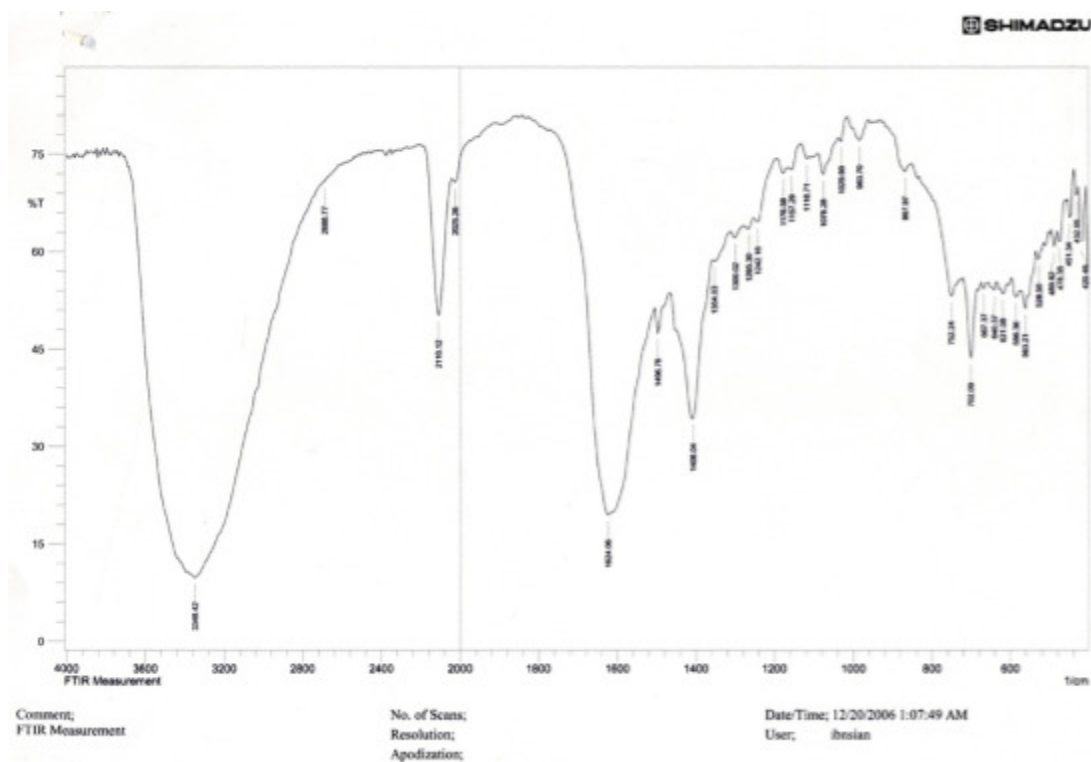


Fig. 4 : FT-IR spectrum of $[\text{Ni}(\text{APA})_2]$.

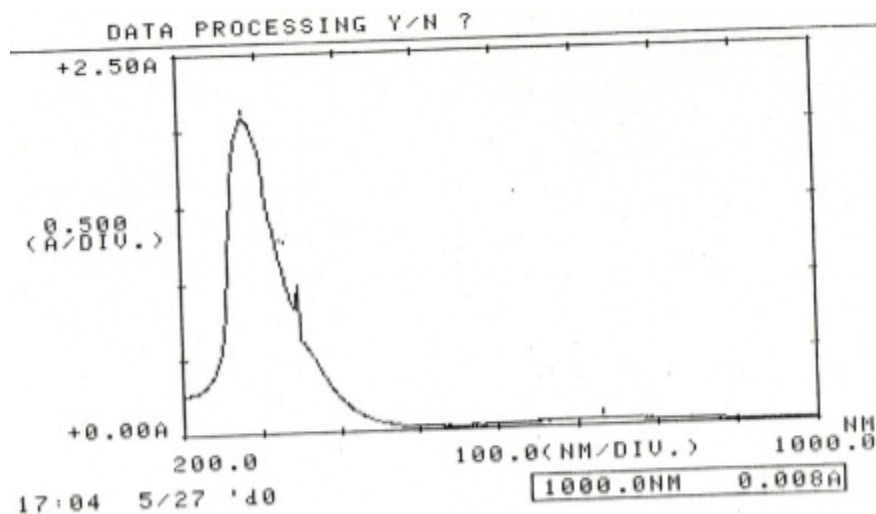


Fig. 5 : Electronic spectrum of ligand (APA).

(APA) and its complexes. The FTIR spectrum of Ni complex showed in Fig. 4.

Magnetic properties for the metal complexes

Magnetic moment (μ_{eff}) with regard to complexes related to $\text{Mn}^{+2}(\text{d}^5)$, also $\text{Co}^{+2}(\text{d}^7)$ have been indicated (5.99) B.M, also (4.82) B.M, that in anticipated spin-only values Higher value related to μ_{eff} of $\text{Ni}^{+2}(\text{d}^8)$ complex (3.22) B.M because of orbital contributions (Cotton *et al*, 1998). Magnetic moment μ_{eff} related to $\text{Cu}^{+2}(\text{d}^9)$ complex indicated (1.73) B.M in expected value to one electro (Sanja *et al*, 2004). All data are indicated in the

Table 1.

Electronic spectral

The UV-Visible spectra of (APA) and its complexes recorded in Table 3, the solution of (APA) in 10^{-3}M (DMSO) exhibited two peaks. Fig. 5 at $(35587)\text{ cm}^{-1}$ and $(28985)\text{ cm}^{-1}$ which are attributed to $\delta \rightarrow \delta^*$ and $n \rightarrow \delta^*$ transition, respectively (Nichllis, 1984).

The spectra of complexes

$[\text{Mn}(\text{APA})_2]$ d^5 : The deep brown complex of Mn (II) shows bands at $(35714)\text{ cm}^{-1}$ and (26954) due to ligand field and charge transfer and another band at

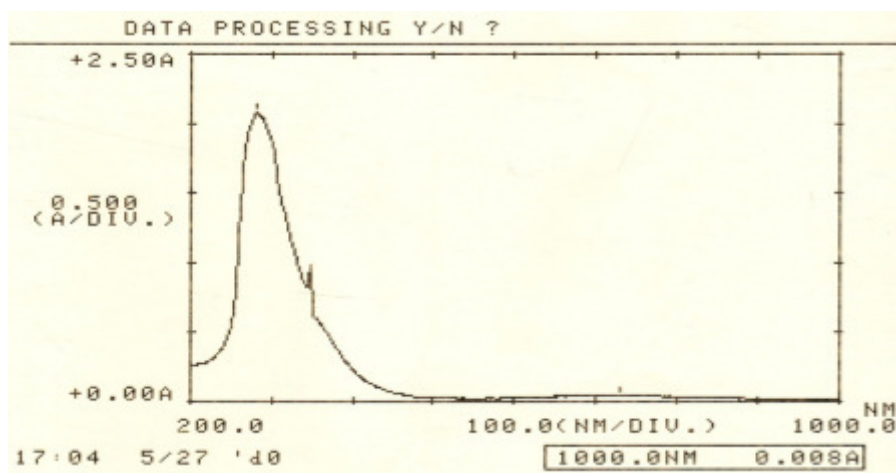


Fig. 6 : Electronic spectrum of $[\text{Cu}(\text{APA})_2]$.

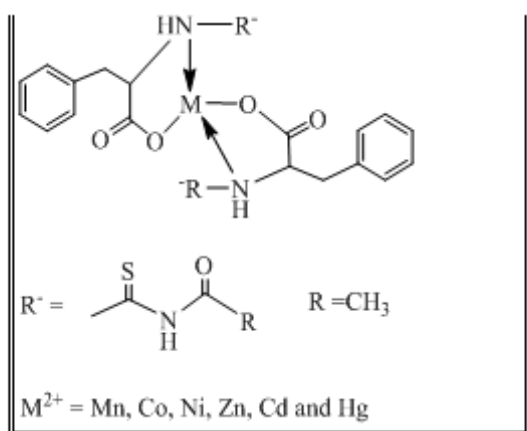


Fig. 7 : Suggested structure of complexes $[\text{M}(\text{APA})_2]$.

respectively and the rach interelectronic repulsion parameter (B^*) was found to be $(733) \text{ cm}^{-1}$, from the relation $\hat{a} = B/B_0$ was found to be a equal (0.75), these parameter are accepted to $\text{Co}(\text{II})$ tetrahedral complex (Lever, 1968).

$[\text{Ni}(\text{APA})_2] \text{ d}^8$: The spectrum of deep green complex of $\text{Ni}(\text{II})$ has revealed the following electronic transfer ligand felid (C.T), mix with ${}^3\text{T}_{1(\text{F})} \rightarrow {}^3\text{T}_{1(\text{P})}$, ${}^3\text{T}_{1(\text{F})} \rightarrow {}^3\text{A}_{2(\text{F})}$ and ${}^3\text{T}_{1(\text{F})} \rightarrow {}^3\text{T}_{2(\text{F})}$, transition at $(34722) \text{ cm}^{-1}$, $(27932) \text{ cm}^{-1}$, $(21276) \text{ cm}^{-1}$ and $(12820) \text{ cm}^{-1}$ respectively, the (B^*) value found to be $(716) \text{ cm}^{-1}$, while \hat{a} was equal to (0.687) these are the characteristics for tetrahedral complexes of $\text{Ni}(\text{II})$ (Mukhlis *et al*, 2012).

Table 4 : Some FT-IR frequencies in (cm^{-1}) for (APA) and its.

No.	Complex	$\nu(\text{COO})$ asym	$\nu(\text{COO})$ sym	$\text{COO } (\nu\Delta)$	$\nu(\text{N-H})$ $\nu(\text{O-H})$	$\nu(\text{C=O})$	$\nu(\text{C=S})$	$\nu(\text{M-N})$	$\nu(\text{M-O})$	$\nu(\text{V=O})$
1	Ligand (APA)	1706(S)	1373(M)	—	3421(M) 3232(M)	1616(M)	1234(S)	—	—	—
2	$[\text{Mn}(\text{APA})_2]$	1574(M)	1411(M)	163	3303(M)	1612(M)	1234(M)	420(w)	457(M)	—
3	$[\text{Co}(\text{APA})_2]$	1588(M)	1411(S)	177	3363(b)	1612(S)	1234(M)	447(w)	456(M)	—
4	$[\text{Ni}(\text{APA})_2]$	1577(M)	1405(S)	172	3345 (b)	1624(M)	1242(M)	420(w)	489(W)	—
5	$[\text{Cu}(\text{APA})_2]$	1581(M)	1404(M)	176	3335(S)	1627(M)	1283(M)	447(w)	496(W)	—
6	$[\text{Zn}(\text{APA})_2]$	1520(M)	1419 (S)	161	3313(M)	1604(S)	1242(S)	443 (W)	459 (w)	—
7	$[\text{Cd}(\text{APA})_2]$	1573(M)	1408 (M)	165	3371(M)	1620(W)	1230 (M)	424 (W)	489 (W)	—
8	$[\text{Hg}(\text{APA})]$	1562 (w)	1401 (M)	161	3380 (b)	1616(M)	1228 (M)	420 (w)	462 (w)	—

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

$(12106) \text{ cm}^{-1}$ which is caused by the electronic transfer ${}^6\text{A}_1 \rightarrow {}^4\text{T}_{2(\text{G})}$ (Al-hashima *et al*, 2011).

$[\text{Co}(\text{APA})_2] \text{ d}^7$: The spectrum of the deep blue complex gave four bands at $(34602) \text{ cm}^{-1}$ $(28818) \text{ cm}^{-1}$, $(13175) \text{ cm}^{-1}$ and $(10330) \text{ cm}^{-1}$ attributed to ligand felid, (C.T), ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{P})}$, ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{F})}$ and ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{2(\text{F})}$

$[\text{Cu}(\text{APA})_2] \text{ d}^9$: The spectrum of green complex of $\text{Cu}(\text{II})$, Fig. 6 shows three bands at $(35587) \text{ cm}^{-1}$, $(26246) \text{ cm}^{-1}$ and $(13679) \text{ cm}^{-1}$ caused to ligand felid, (C.T), mix with ${}^2\text{B}_{1\text{g}} \rightarrow {}^2\text{A}_{1\text{g}}$ and ${}^2\text{B}_{1\text{g}} \rightarrow {}^2\text{B}_{2\text{g}}$ transition, respectively (Arjmand *et al*, 2012).

The complexes of $[\text{Zn}(\text{APA})_2]$, $[\text{Cd}(\text{APA})_2]$ and

Table 5 : The peaks electronic transitions and structure geometries of (APA) and its complexes.

No	Complex	$\tilde{\nu}_{2\max}$ (nm)	ABC	Wave number cm^{-1}	ϵ_{\max} $\text{L.mol}^{-1}.\text{cm}^{-1}$	Transition
1	(APA)	281 345	2.085 1.735	35587 28985	2085 1735	$\delta \rightarrow \delta^*$ $n \rightarrow \delta^*$
3	[Mn(APA) ₂]	280 371 826	1.862 1.041 0.024	35714 26954 12106	1862 1041 94	L.F C.T ${}^6\text{A}_1 \rightarrow {}^4\text{T}_2(\text{G})$
4	[Co(APA) ₂]	289 347 759 968	2.201 1.390 0.035 0.030	34602 28818 13175 10330	2201 1390 35 30	L.F ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{p})}$ C.T mix ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{f})}$ ${}^4\text{A}_2 \rightarrow {}^4\text{T}_2$
5	[Ni(APA) ₂]	288 358 470 780	2.239 0.889 0.085 0.030	34722 27932 21276 12820	2239 889 85 30	L.F ${}^3\text{T}_{1(\text{f})} \rightarrow {}^3\text{T}_{1(\text{p})}$ C.T mix ³ $\text{T}_{1(\text{f})} \rightarrow {}^3\text{A}_2$ ${}^3\text{T}_{1(\text{f})} \rightarrow {}^3\text{T}_{2(\text{f})}$
6	[Cu(APA) ₂]	281 381 731	2.079 0.550 0.040	35587 26246 13679	2079 550 40	L.F C.T mix ${}^2\text{B}_{1(\text{g})} \rightarrow {}^2\text{A}_{1(\text{g})}$ ${}^2\text{B}_{1(\text{g})} \rightarrow {}^2\text{B}_{2(\text{g})}$
7	[Zn(APA) ₂]	283 345	2.243 2.246	35335 28985	2243 2246	L.F C.T
8	[Cd(APA) ₂]	281 348	2.040 1.002	35587 28985	2040 1002	L.F C.T
9	[Hg(APA) ₂]	279 358	1.791 0.525	35842 27932	1791 525	L.F C.T

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

[Hg(APA)₂] shows only ligand felid and charge transfer of (ML) at (35335, 28985) cm^{-1} , (35587, 28735) cm^{-1} and (35842, 27932) cm^{-1} respectively (Huheey, 1983). All transition with their assignments are summarized in Table 3.

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(APA)₂], where $\text{M}^{2+} = (\text{Mn}, \text{Co}, \text{Ni}, \text{Zn}, \text{Cd} \text{ and } \text{Hg})$, Fig. 6, while copper complex s has square planer.

CONCLUSION

The new ligand in the presented study was prepared through reaction from the Acetyl isothiocyanate with phenylalanine, ligand has been characterized through elemental micro analysis C.H.N.S., FTIR, UV Vis also ${}^1\text{H}$, ${}^{13}\text{C}$ -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 geometry except for the copper complex that has square planer.

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