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Synthesis ,characterization and antibacterial activity of mixed ligand complexes of some metals with 1-nitroso-2-naphthol and L-phenylalanine

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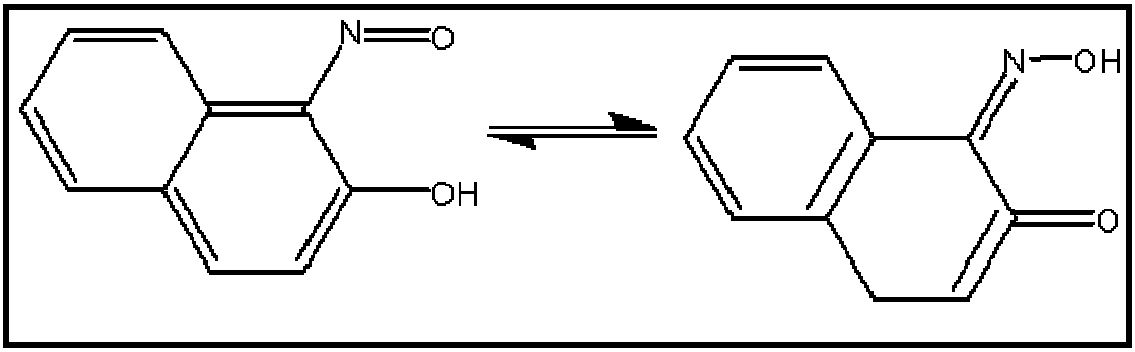
Abstract
The mixed ligand complexes of Mn(II),Fe(II),Co(II),Ni(II),Cu(II), Zn(II) and Cd (II) with 1-nitroso-2-naphthol ($C_{10}H_7NO_2$), symbolized (NNPhH)] and amino acid L-phenylalanine($C_9H_{10}NO_2$), symbolized (**phe** H), were synthesized and characterized by: Melting points, Solubility, Molar conductivity. determination the percentage of the metal in the complexes by flame(AAS), Molecular weight determined by Rast's Camphor method, susceptibility measurements, Spectroscopic Method [FT-IR and UV-Vis], And Program [Chem office– CS .Chem.– 3D pro 2006] was used for draw compounds . The results showed that the deprotonated two ligands acts as a bidentate ligand , (**phe**$^-$) was coordinated to the metal ions through the oxygen of the carboxylic group and the nitrogen of the amine and the 1-nitroso-2-naphthol ligand was coordinated to the metal ions through the oxygen and nitrogen atoms. The electronic absorption spectra and magnetic susceptibility measurements of the complexes indicate octahedral geometry for all the complexes.

Key words: phenyl alanine , amino acid) Complexes , 1-nitroso-2-naphthol ,mixed ligands and Antibacterial Activities

Introduction

1-nitroso-2-naphthol (IUPAC Name:1-nitrosonaphthalen-2-ol) ($C_{10}H_7NO_2$) is crystalline solid, sparingly soluble in water and readily soluble in alcohol ,ether and common organic solvent. Its melting point is equal to 103–106°C, Ortho-substituted nitrosonaphthols can undergo tautomerisation to give oxo-oximes. (Figur- 1) In the case of 1-nitroso-2-naphthol, the equilibrium is greatly displaced toward the keto-form and the compound has, in the solid state and in solution, a predominately quinone which is a hybrid of resonance forms of the type **[1-2]**



Figur-1:Tautomerisation in 1-nitroso-2-naphthol

It is also a sensitive and specific histochemical reagent for fluorimetric determinations of tyrosine residues in proteins and peptides. [3] Along with other phenols and naphthols, it belongs to biologically important compounds,especially because of

its cytotoxic action.4 Although there has been considerable interest in iron(II) complexes of 1-nitroso-2-naphthol,1,5 surprisingly little work was carried out on chelate complexes of the same metal ion with the isomeric 2-nitroso-1-naphthol. [4-6]

Phenylalanine (Phe H) [7-8] is aromatic essential glucogenic and ketogenic amino acid. In metabolism phenylalanine is converted into tyrosine. In metabolism homogenstic acid is formed which undergoes cleavage and form fumarate and acetoacetate. The hormones such as adrenaline, noradrenaline, thyrosine and melanin pigment formed from tyroxine.Several abnormalities observed in phenylanine metabolism such as phenylketonaria and alkaptonaria. In phenylketonaria, there is a black in hydroxylation of phenyl alanine to
form tyrosine, this leads to mental retardation. [7-8]

Biological importance of several amino acids their complexes with transition metals is well documented [9-10].

In this paper we present the synthesis and study of (II),Fe(II),Co(II),Ni(II),Cu(II), Zn(II) and Cd (II) complexes with 1-nitroso-2-naphthol as a primary ligand and amino acid
(L- Phenylalanine) as a secondary ligand have been used, respectively.

Experimental

a- Reagents and instruments:L- phenylalanin was purchased from(Merck),1-nitroso-2-naphthol a Fluka Chemise AG, metals chloride and solvents and camphor(C10H16O) from (B.D.H) and (Merck).The reagents were used without further purification .

b- Instruments: FT-I.R spectra were recorded as KBr discs using Fourier transform Infrared Spectrophotometer Shimadzu 24 FT-I.R 8400s. Electronic spectra of the prepared complexes were measured in the region (200-1100) nm for 10^{-3}M solutions in ethanol at 25ºC using shimadzu-U.V-160.AUltra Violet Visible-Spectrophotometer with 1.000 ± 0.001 cm matched quartz cell. While metal contents of the complexes were determined by Atomic Absorption (A.A)Technique using Japan A.A-67G Shimadzu. Electrical conductivity measurements of the complexes were recorded at 25ºC for 10^{-3} M solutions of the samples in ethanol using pw 9527 Digital conductivity meter (Philips).Magnetic susceptibility measurements were measured using Bruker magnet BM6. Melting points were recorded by using Stuart melting point apparatus. Molecular weight of ligands and their metal complexes were determined by Rast camphor method and chloride ion content were also evolution by (Mohr method),. The proposed molecular structure of the complexes were determined by using chem. office program, 3DX (2006).

C- **General synthesis :** **a)**

Sodium phenylalaninate (Na^+phe$^-$) : L- **phenylalanin** [0.165 gm, 1m mol] was dissolved in 10 ml ethanol and added to 10 ml of ethanolic solution containing [0.04 gm (1mmol)] of the sodium hydroxide , the solution was deprotonated according to the following reaction (scheme -1)

b) sodium 1-Nitroso-2-naphthol ate (Na^+NNPh$^-$):

1-nitroso-2-naphthol ((NNPhH) [0.346 gm(2mmol)] was dissolved in 10 ml ethanol and added to 10 ml of ethnolic solution containing[0.08 gm (2mmol)] of the sodium hydroxide,the solution was deprotonated according to the following reaction (scheme -1)

c) Synthesis of complexes: The complexes were prepared by the addition of ethnolic solutions of the (Na^+NNPh$^-$) and (Na^+**phe**$^-$) to warm stirred ethnolic solution of the respective metal (II) chloride in the stoichiometric ratio matel:ligand (2 NNPh :M: **phe**) in (30 min). The mixture was stirred for half an hour at room temperature , crystalline precipitates observed . The resulting precipitates were filtered off , recrystallized from ethanol and dried at 50C^0 . according to the following reaction (scheme -1).

Results and Discussion

All the complexes are colored, non-hygroscopic and thermally stable solids (Table1), indicating a strong metal-ligand bond. The complexes are insoluble in water but soluble in common organic solvents such as ethanol, ethyl alcohol, acetone, chloroform, benzene ,DMF and DMSO. The observed molar conductance (Table1) values measured in ethanol in 10^{-3}M solution lie in the (32-40) Ω^{-1} cm^2 mol^{-1} range, indicating their electrolytic nature with(1:1). [11-12]

The atomic absorption measurements (Table-1) for all complexes gave approximated values for theoretical values.

Molecular weight determined by Rast's Camphor method and were found in accordance with calculated value the range of metal complexes (**597-658**),

The infrared spectra of the synthesized complexes were recorded KBr disk in the range 400-4000 cm^{-1},the important group frequencies of the (N-H$_2$), u(-COO)$_{asy}$,u(-COO)$_{sym}$, (C=O), (N=O), (C=C , CH) aromatic) and(O-H) stretching vibration. In conclusion, our investigation this suggest that the ligands L- phenylalanine and 1-nitroso-2-naphthol

coordinate with M (II) forming Octahedral geometry (scheme -1)., Table (2),displays the (FT-IR) spectrum for the L-phenylalanine exhibited a band around υ (3410m) cm^{-1} that corresponds to the stretching vibration of u (N-H) $_{+}$u (O-H), while another strong absorption band at υ (3350) cm^{-1} is due to the $u(N-H_2)_{sym}$ while the bands at (1560) cm^{-1} and (1420)cm^{-1} were assigned to the u(-COO)asy and u(-COO)sym respectively. $u\Delta (-COO)_{asy-sym}$ =150 cm^{-1}. ,Table (2) Figure (1),displays the (FT-IR) spectrum for the(1-nitroso-2-naphthol) which exhibits very strong band at (1616)cm^{-1} due to υ(C=O) stretching vibration. [14-15]The band at (3425)cm^{-1} is due to the υ(O–H) stretching vibration [14]. The band at (1523)cm^{-1} is due to the υ(C=N) while the bands at (1450) and (2790)cm^{-1} were assigned to the υ(C=C) aromatic and υ(C–H) aromatic stretching respectively. The band at (3066)cm^{-1} were assigned to υ(HO---H) hydrogen bonding[15,16]. and the band at (1153) cm^{-1} is due to the υ(N-O) stretching vibration. In comparison with free amino acids, the $u(-COO)_{asy}$, shows positive shifts and $u(-COO)_{sym}$, records negative shifts, which confirm the monodenticity 13,17 of the carboxyl ate group. The complexes show band at (524-532) and (445-455) cm^{-1} rang, due to the υ (M-N) and υ (M-O) vibrations respectively. [16-18]. The electronic spectra and magnetic moment values of the mixed ligand complexes were recorded in ethanol and their assignments are given in Table-3.

The Uv-Vis spectra of the free ligand (1-nitroso-2-naphthol) in ethanol solvent appeared a high, intense absorption bands at (304nm) 32894 cm^{-1} and at (383nm) 26109cm^{-1} .These bands are attributed to (p®p*)and (n®p*) transitions respectively. The spectrum of the free ligand (pheH) in DMF solvent show a high intensity band in wavelength (271nm)36832cm^{-1} assigned to (n→ π*) [19-20]. The values of Dq and B' have been calculated from the values of u1, and u2, bands using the matrices of Tanabe and Sugano [19-22].The positions of electronic spectral bands indicate that Co(1I) and Ni(I1) possess octahedral configuration in these complexes. The Dq values of these complexes are in the range prescribed for the octahedral complexes of Co(I1) and Ni(II). [19-24]

The assignment of the electronic spectral bands, their positions, and the spectral parameters for Co(I1) 4T1(F) →4T2g(F) u1= (980nm)10200 cm^{-1} , 4T1(F) →4A2 g, u2= (526nm)19000 cm^{-1},4TI g ,(F) →4TI g ,u3= (455nm)21950 cm^{-1}, *10Dq*= 10200 cm^{-1} , *B'=950* cm- , β =0.974 cm^{-1} , (μ_{eff} =4.5 μB). The Co(II) complex under present investigation process interelectronic repulsion parameter (B') 950 cm-1. The Racha parameter (B) is less than free ion value (971) suggesting a considerable orbital overlap and delocalization of electrons on the metal ion. The nephelauxetic ratio (β) for the present Co(II) complex (0.97). This is less than one, suggesting partial covalency in the metal ligand bond. suggest the octahedral geometry for Co(II) complex [19].And The assignment of the electronic spectral bands for , Ni(II) are 3A2g →3T2,(F) u1=(944nm) 10590 cm-1, 3A2g (F) →3T1g(F) u2= (578nm)17300 cm^{-1} ,3A2g → 4T1g(P) u3= (343nm)29050 cm-1,*10Dq*=10620 cm^{-1}, *B'=966* cm^{-1}, =0.93 , (μ_{eff} = 3.18 μB).

The Light Brown colored Cu(II) complex (μ_{eff} =1.95 μB) exhibits a broad asymmetric band in the region (609nm)16395 - (820nm)12185 cm^{-1} with maxima at (819nm) 12210 cm^{-1} in an distorted octahedral geometry [21] .The broadness of the band may be due to dynamic Jahn-Teller distortion and is assigned to 2T2g→2Eg transitions.The electronic spectrum of the Fe(II)complex gave three bands,(722nm)13850 cm^{-1} (u1),(470nm)21276 cm^{-1} (u2) and (344nm)29069cm^{-1} (u3) nm suggesting an octahedral geometry around the Fe (II) ion , similar to those found for distorted octahedral complexes .The doublet is attributed to a Jahn-Teller distortion in the excited state .The room temperature magnetic moment (μ_{eff} = 4.98 μB) corresponded to octahedral symmetry[21-24] Figures(3-5)

Mn(II) complex in ethanol solution contained three bands at (13850 , 21276 and 29069) cm^{-1}, assignable to the transitions 6A1g → 4T1g, 6A1g →4T2g and charge transfer, respectively. The electronic transitions together with a magnetic moment value (μ_{eff} = 5.77 μB). which is close to the spin- -only value (5.92 μB) suggests high spin octahedral geometry for the Mn(II) complex. [19-20] The complex of Cd (II) complex exhibits two bands at (304 and 382) nm assigned to charge –transfer transitions [21-24].From the above data ,we suggested that the geometry of the all complexes are octahedral.

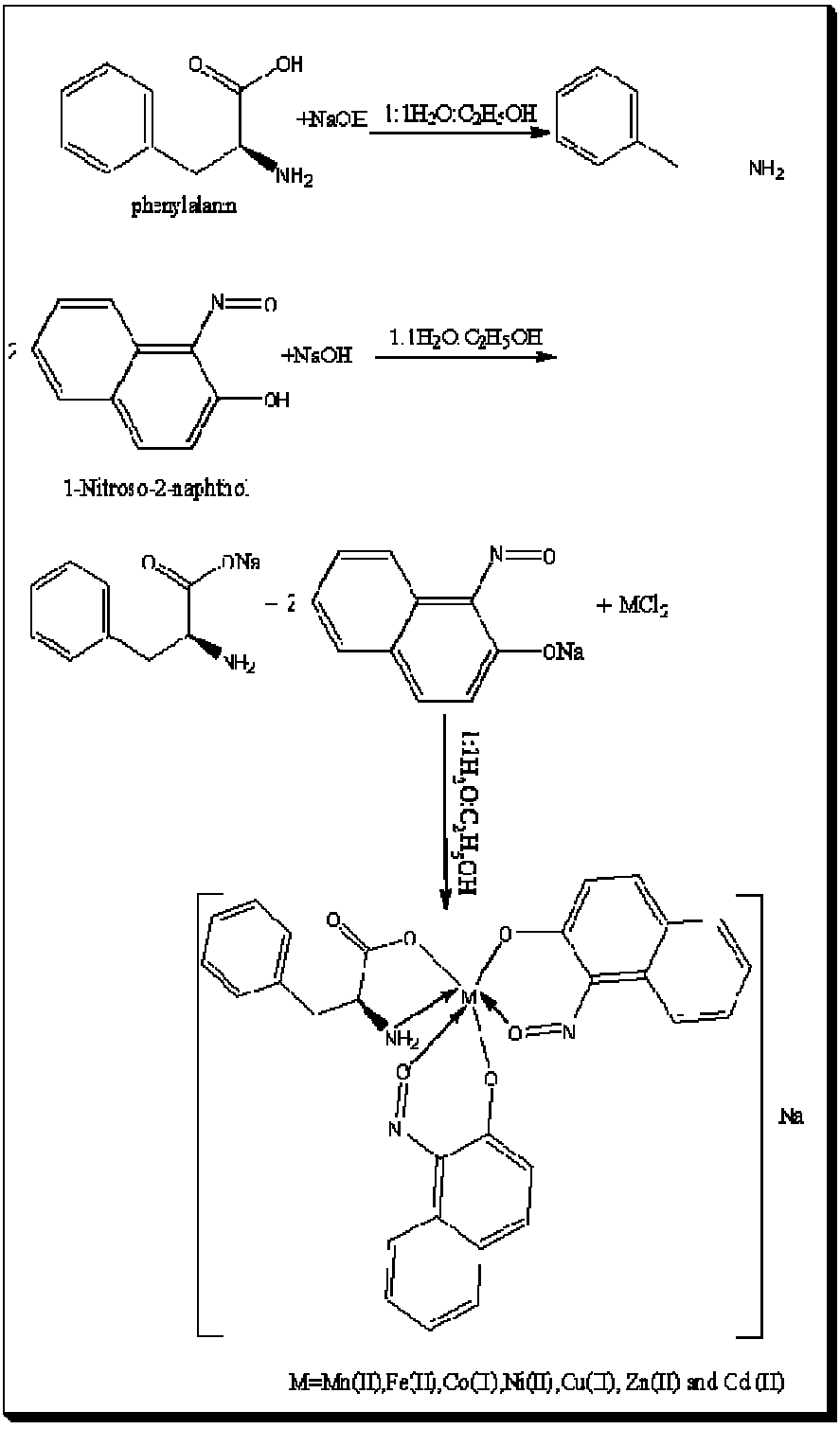
Biological activity(antibacterial activity)

The comparison of the biological activity of the ***Pseudomonas aeruginosa*** and ***Escherichia coli (G-)*** *bacteria and.* ***Staphylococcus aureus*** *and* ***Bacillis subtilis*** *(G+)* .The data are listed in Tables (4) and shown in Scheme (2)

The biological activities of metal complexes are higher than the free ligand towards the gram positive and gram negative bacteria, In addition, the biological activity of the complexes follow the order Co(II) > Ni(II) > Cu(II) > Zn(II) > Cd(II) > Mn(II) > Fe(II) against ***Staphylococcus aureus*** and ***Escherichia coli*** organisms. But with ***Bacillis subtilis*** organism, the biological activity of the binary complexes follows the order:Co(II) > Ni(II) > Cu(II) > Zn(II) > Mn(II) > Cd(II) > Fe(III).

is evident from the above data that the antibacterial activity significantly increased on coordination.

It has been suggested that the ligand with nitrogen and oxygen donor systems inhibit enzyme activity. Coordination reduces the polarity of the metal ion mainly because of the partial sharing of its positive charge with the donor groups within the chelate ring system[25, 9 , 26].



Scheme (1): Synthesis of the Na [M ($C_{29}H_{22}N_3O_6$)] complexes

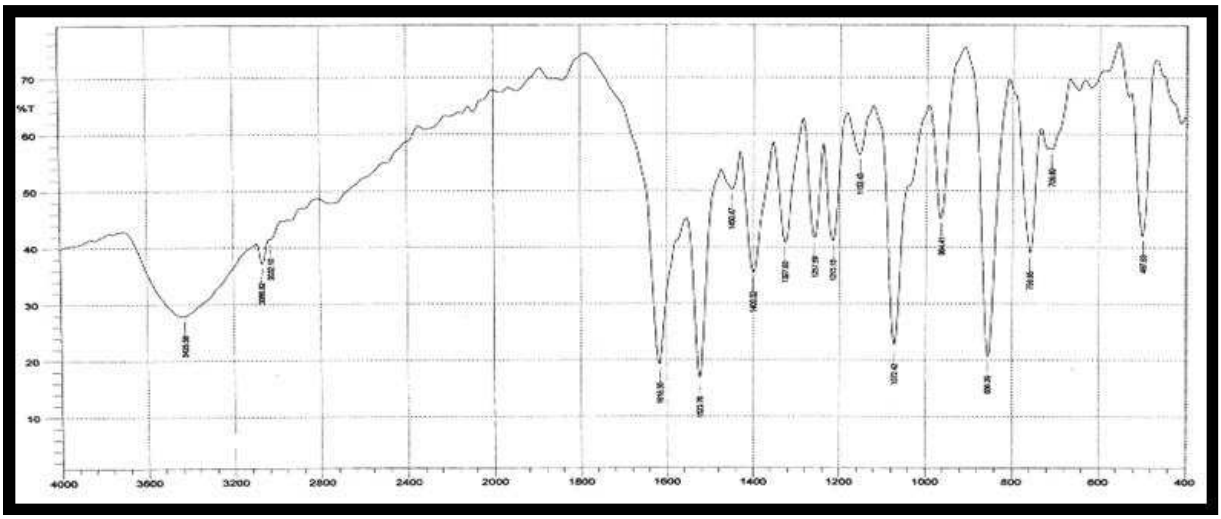


Figure .(2)FT- IR Spectrum of 1-nitroso-2-naphthol

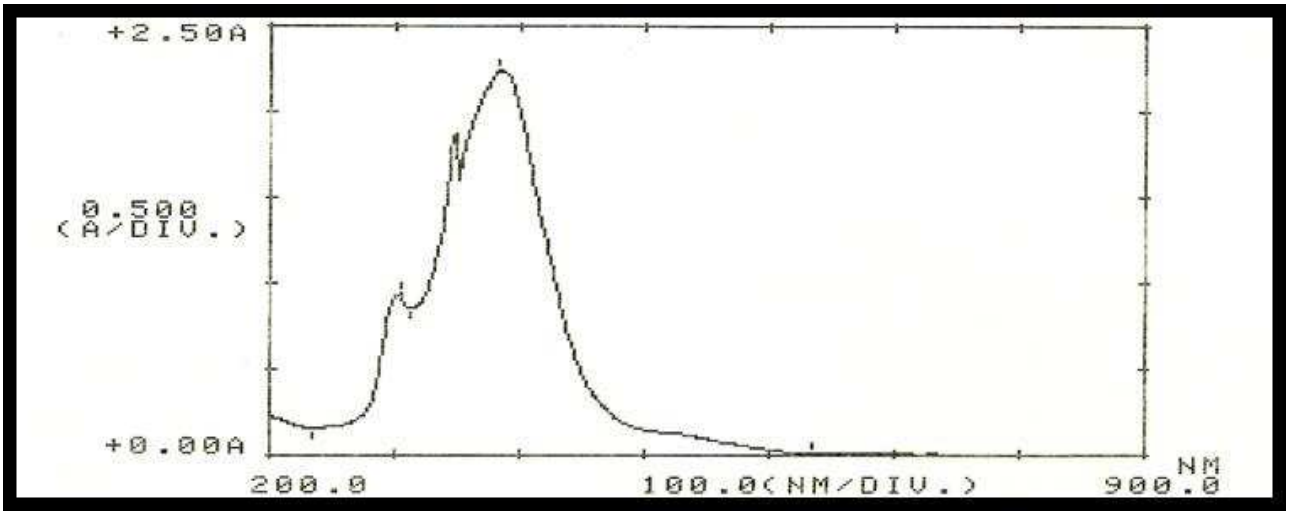


Figure .(3)The (UV-Vis) Spectra of 1-nitroso-2-naphthol

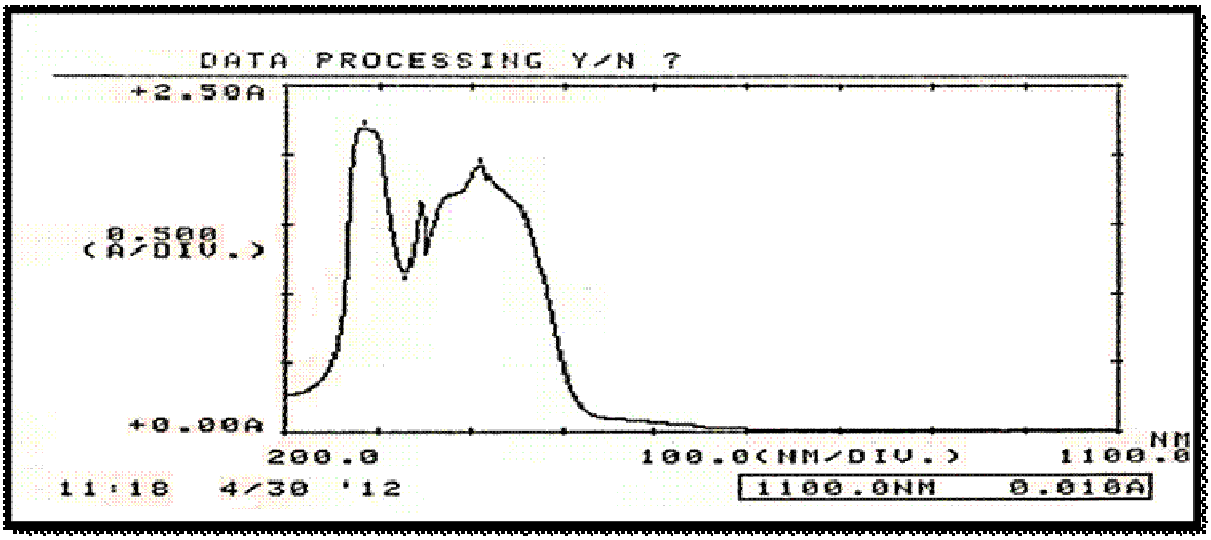


Figure .(4)The (UV-Vis) Spectra of . Na [Ni($C_{29}H_{22}N_3O_6$)]

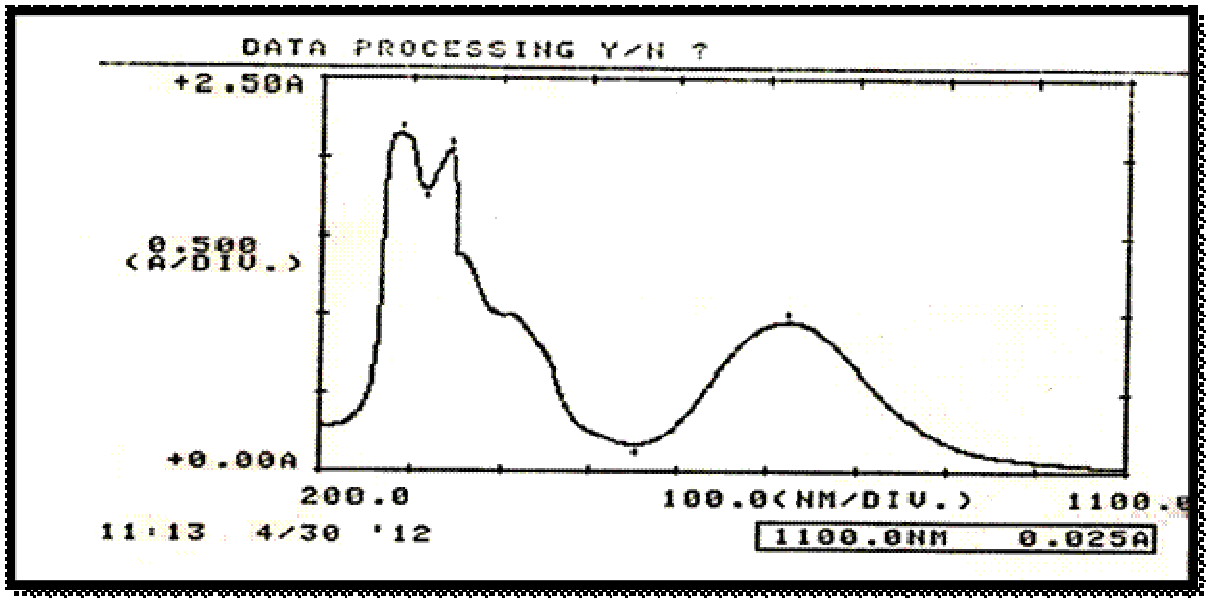


Figure .(5)The (UV-Vis) Spectra of . Na [Fe($C_{29}H_{22}N_3O_6$)]

Table 1-The physical properties of the complexes

Compound / Ligand	M.W. theory (calc .)	Color	M .p °c (de) °c	Lm µS.cm^2.Mol$^-$ in ethanol	Metal% theory	Metal% exp	C l%
1-nitroso-2-naphthol $C_{10}H_7NO_2$	**173.17**	**Dark Brown**	**103**	**1.77**	**-**	**-**	**-**
$C_9H_{11}NO_2$	**165 .23**	**White**	**291**	**6.7**			
Na [Mn ($C_{29}H_{22}N_3O_6$)]	**586.08 (598)**	**Dark Brown**	**220 de**	**37**	**9.37**	**9.30**	**Nill**
Na [Fe ($C_{29}H_{22}N_3O_6$)]	**587.08 (599)**	**Dark green**	**236 de**	**39**	**9.51**	**9.31**	**Nill**
Na [Co ($C_{29}H_{22}N_3O_6$)]	**590.07 (599)**	**Red**	**240de**	**37**	**9.98**	**9.30**	**Nill**
Na [Ni ($C_{29}H_{22}N_3O_6$)]	**589.08 (596)**	**Brown**	**262de**	**39**	**9.94**	**9.37**	**Nill**
Na [Cu ($C_{29}H_{22}N_3O_6$)]	**594.07 (598)**	**Light Brown**	**230de**	**38**	**10.68**	**10.0**	**Nill**
Na [Zn ($C_{29}H_{22}N_3O_6$)]	**596.90 (597)**	**green**	**212de**	**42**	**10.96**	**10.06**	**Nill**
Na [Cd ($C_{29}H_{22}N_3O_6$)]	**645.04 (658)**	**Dark green**	**228de**	**40**	**17.46**	**16.96**	**Nill**

Lm = Molar Conductivity de = decomposition

Table (2) FT-IR spectral data of the Ligands and there complexes

Compound	υ(N-H)+ υ (O-H)	υ (C-H)$_{ay}$ (C-H)$_{ali}$	υ(C=O)	υ(C=N)	υ(N-O)	υ(-COO)$_{asy}$	υ (-COO)$_{sym}$	(-COO)$_{sym}$	M-N	M-O
$C_9H_{11}NO_2$	3471*s* 3410 m	2962*vs* 2970*vs*	1623*vs*	-	-	1558*s*	1408 *vs*	150	-	-
1-nitroso-2-naphthol ($C_{10}H_7NO_2$)	3414*s*-br-3066*w*	2790*vw*	1616*vs*	1523*vs*	1153m	-	-	-	-	-
Na [Mn ($C_{29}H_{22}N_3O_6$)]	3479*vs* 3417 *vs*-br	3059m 3028m	1616*vs*	1500*s*	1130m	1585*vs*	1346 v *s*	239	528m	447
Na [Fe ($C_{29}H_{22}N_3O_6$)]	3475*vs* 3417 *vs*-br	3062m 3028m 2893*w*	1620*vs*	1508*vs*	1149m	1559*vs*	1357*s*	202	528m	447*s*
Na [Co ($C_{29}H_{22}N_3O_6$)]	3479*vs* 3417 *vs*-br	3062m 2879*w*	1639*vs*	1516*vs*	1157m	1589*s*	1361*s*	228	532m	455*s*
Na [Ni ($C_{29}H_{22}N_3O_6$)]	3444*vs* 3417 *vs*-br	3062m 2953*w*	1608*vs*	1531*w*	1157m	1597*vs*	1354*s*	243	532m	455m
Na [Cu ($C_{29}H_{22}N_3O_6$)]	3332*s* 3248m	3032m	1622 *vs*	1531*vs*	1157m	1593v *s*	1369 v *s*	224	524	445*w*
Na [Zn ($C_{29}H_{22}N_3O_6$)]	3334 3030*w*	3030*w*	1616*vs*	1544*vs*	1155m	1595v *s*	1379 v *s*	216	513	418
Na [Cd ($C_{29}H_{22}N_3O_6$)]	3334*s* 3248m	3038m	1623 *vs*	1535*vs*	1157m	1598v *s*	1389 v *s*	209	524	445*w*

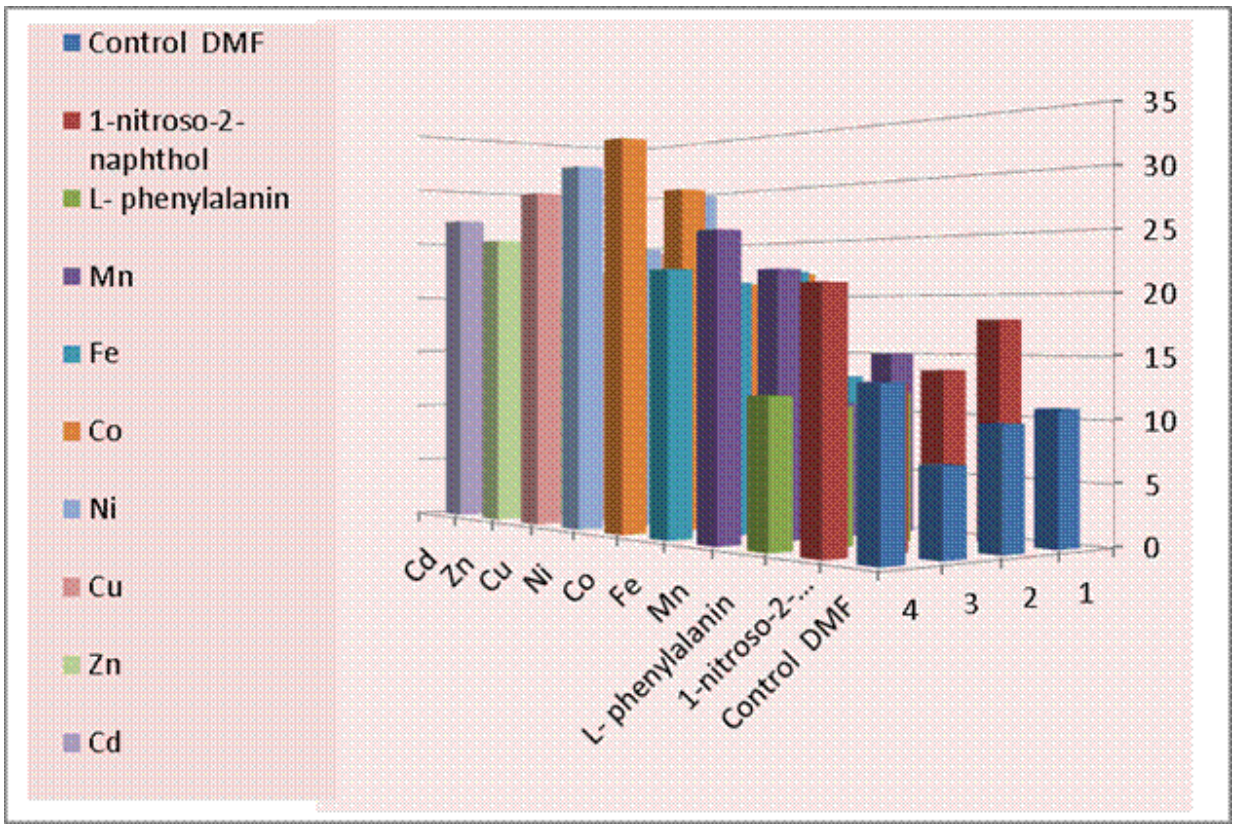
Compounds	λ (nm)	U¢(cm^{-1})	m_{eff} (µB)	Assignment
$C_9H_{11}NO_2$	371	26954	-	n → π*
1-nitroso-2-naphthol ($C_{10}H_7NO_2$)	304 383	32894 26109	-	p®p* n®p*
Na [Mn ($C_{29}H_{22}N_3O_6$)]	278 675 983	35971 14814 10172	5.77	Charge transfer $^6A_{1g}\rightarrow{}^4T_{2g}$ $^4A_{1g}\rightarrow{}^4T_{1g}$
Na [Fe ($C_{29}H_{22}N_3O_6$)]	344 470 722	29069 21276 13850	4.98	Charge transfer $^5T_{2g}\rightarrow{}^5E_g$ $^5T_{2g}\rightarrow{}^5E_g$
Na [Co ($C_{29}H_{22}N_3O_6$)]	300 343 578 944	33333 29050 17300 10590	4.50	Charge transfer 3A2g → 4T1g(P) 3A2g → 4T1g(P) 3A2g (F) →3T1g(F)
Na [Ni ($C_{29}H_{22}N_3O_6$)]	343 578 944	29050 17300 10590	3.18	3A2g → 4T1g(P) 3A2g (F) →3T1g(F) 3A2g → 3T2,(F)
Na [Cu ($C_{29}H_{22}N_3O_6$)]	397 609-820	25188 16395-12185	1.95	Charge transfer 2T2g → 2Eg
Na [Zn ($C_{29}H_{22}N_3O_6$)]	348	28735	Diamag	Charge transfer
Na [Cd ($C_{29}H_{22}N_3O_6$)]	304 385	32894 25974	Diamag	Charge transfer Charge transfer

Table 3- Electronic Spectral data, magnetic moment, of the studied Compounds

Table (4) Showed the inhibition circle diameter in millimeter for the bacteria after 24 hour incubation paid and 37°C for L- phenylalanin and 1-Nitroso-2-naphthol complexes

Compound	*Bacillis* subtilis *G (+)*	*Pseudomonas* aeruginosa (G-)	*Escherichia* coli *(G-)*	*Staphylococcus aureus(G+)*
Control DMF	**10.9**	**9.9**	**7**	**13.3**
1-nitroso-2-naphthol	**17.9**	**13.9**	**13**	**20.7**
L- **phenylalanin**	**11**	**12**	**11**	**12**
Na [Mn ($C_{29}H_{22}N_3O_6$)]	**15**	**11**	**22**	**25**
Na [Fe **($C_{29}H_{22}N_3O_6$)]**	**13**	**22**	**21**	**22**
Na [Co **($C_{29}H_{22}N_3O_6$)]**	**22**	**21**	**29**	**33**
Na [Ni **($C_{29}H_{22}N_3O_6$)]**	**21**	**29**	**24**	**31**
Na [Cu **($C_{29}H_{22}N_3O_6$)]**	**18**	**24**	**22**	**29**
Na [Zn **($C_{29}H_{22}N_3O_6$)]**	**15.5**	**22**	**20**	**25**
Na [Cd **($C_{29}H_{22}N_3O_6$)]**	**15**	**13**	**21**	**27**

1.Bacillis
3.Escherichia 2.Pseudomonas
4.Staphylococcus



Scheme (2) Inhibitory activity of the ligands and metal complexes Against *(Pseudomonas aeruginosa* and ***Escherichia coli (G-)*** *bacteria and.* ***Staphylococcus aureus*** *and* ***Bacillis subtilis*** *(G+)*

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