



Synthesis, Spectral and Biological Studies of New 2-amino-3-cyanopyridine Derivatives and Their Transition Metals Complexes

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Abstract

In this study, new substituted 2-amino-3-cyanopyridine derivatives were synthesized in excellent yields by reacting chalcone derivatives with malanonitrile in the presence of ammonium acetate, and new complexes with these ligands were synthesized and characterized by elemental analysis, UV, IR, Mass, and NMR (1H, 13C) spectroscopy, and had the general formula: $[ML_2(H_2O)_2]Cl_2$ where M= Co(II), Ni(II) and Cu(II), $[ML_2(H_2O)Cl]Cl_2$ where M= Cr (III). On the basis of analytical data and IR studies, a 1:2 metal to ligand stoichiometry has been suggested to these coordination complexes. All of these studies suggest that the ligand acted as a bi-dentate ligand when it coordinates with metal ions, and that the prepared complexes had an octahedral geometry coordinated by both the nitrogen of (cyano group) and (amino group). The ligand and its complexes were tested for antimicrobial activity against gram negative (*Pseudomonas aeruginosa*) and gram positive (*Bacillus subtilis*) bacteria. All of these complexes have been shown to be more antibacterial than their respective ligands.

Keywords: Chalcone, Cyanopyridine, malanonitrile, Transition metals complexes, Biological activity. Electron withdrawing, Antibacterial effective.

1. Introduction

Pyridine component have been interested by the researcher in the last decayed due to their wide series application in medicinal chemistry [1–3], they also exhibit antibacterial [4], antifungal [5] antimitotic [6–7] and antidepressant [8]. Some pyridine derivatives such as 2-amino-3-cyano-4,6-diaryl pyridine possess variety type of application as nonlinear optical material [9], electrical material [10] and fluorescent liquid crystal [11]. In addition, these compounds are important for preparing a wide range of heterocyclic compounds such as cyanopyridine [12]. 2-amino-3-cyanopyridine one of the most important derivatives among of these pyridine compound. In general, the compounds containing 2-amio-3-cyanopyridine moiety were used as a drug due to their pharmacological activities such as antitumor, anticancer, anti-inflammatory and used as IKK- β inhibitor [13-14]. Different methods were used to synthesis 3-cyanopyridine derivatives were described in literature [15]. However, these methods that used

to synthesis these compounds gave a low yield and impurities [16-17]. So, new route was suggested to prepare 2-amio-3-cyanopyridine derivatives by adding substituents electron with drawing group and donating group and investigated the effect of these groups in increase or decrease the reaction time and yield also a limited synthesized of their complexes. In this present work, two steps were used to prepare 2-amino-3-cyanopyridine derivatives first step to synthesis α , β -unsaturated ketone and then mixed with malononitrile to yield the title compounds which are used to synthesis a large number of their complexes with transition metals as shown in Scheme 1.

2. Experimental

All the starting material and reagent were supplied with the highest purity and used as supplied by either Merck or Fluka. To measure the melting points for all prepared compounds, the Gallen Kamp melting point was used. On the shimadzu-8300 spectrophotometer, FTIR spectra were reported in the range of (4000-

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400) Cm^{-1} . Electronic spectra of ligand and its complexes were measured by using the UV-1650 PC Shimadzu spectrophotometer at 25°C in the solution of 10^{-3} M DMF. An EM measurement was reported on the W.T.W. conductivity meter using DMF as a solvent and a concentration of 10^{-3} M at room temperature. Elemental C.H.N. analysis was performed using the normal addition method of the Shimadzu A.A-670 spectrometer. Magnetic susceptibility measurements were measured by Sherwood scientists at 25°C as a solid state using the Couy magnetic susceptibility balance. Mass spectra were recorded on Shimadzu GCMS-QP5050A Spectrometer by using direct injection probe. ^1H , ^{13}C NMR spectra were measured in DMSO- d_6 using the Bruker Spectrophotometer (300 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane (TMS).

2.1 Synthesis of an organic ligand

General procedure for preparation: (E)-1-(4-bromophenyl)-3-(4-chlorophenyl)prop-2-en-1-one LA1 [5-16]

A mixture of aldehyde derivatives (0.01 mole) and ketone derivatives (0.013 mole) in 25ml ethanol was refluxed for 12h. in the presence of NaOH (10%, 2 ml). The volume of reaction mixture was condensed to half volume and after that the solution poured into the ice-cold water (25 ml). The precipitate was formed and then collected by filtration and washing with cold ethanol and recrystallization from methanol to produce a pale yellow product with title LA1: ^1H NMR, 8.4(d, 1H, $J = 7.7\text{Hz}$, $\text{CH}=\text{ph-cl}$), 8.03 (d, 2H, $J = 6.8\text{Hz}$, Hortho-phBr), 7.8 (d, 2H, $J = 6.5\text{Hz}$, Hpara-phBr), 7.65 (d, 2H, $J = 7.62\text{Hz}$, Hortho-phCl), 7.54(d, 2H, $J = 7.7\text{Hz}$, Hpara-phCl); ^{13}C NMR, (188, 146, 120, 137, 134, 133, 132, 130, 129, 128). MS: m/z 321 [M] $^+$

Synthesis of (E)-3-(4-(dimethylamino)phenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one LA2

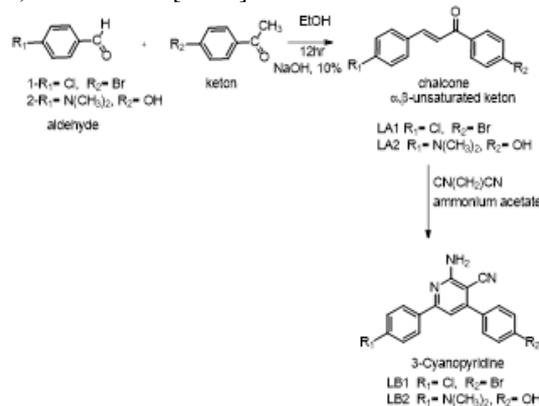
This ligand prepared by following the same procedure that used to prepare LA1. A Light yellow product was obtained with excellent yield 90%. ^1H NMR, 9.7 (s, 1H, -OH), 8.07(d, 1H, $J = 7.8\text{Hz}$, =HC-ph), 8.04 (d, 2H, $J = 6.5\text{Hz}$, Hortho-phOH), 7.65 (d, 1H, $J = 7.8\text{Hz}$, HC=), 7.56 (d, 2H, $J = 6.7\text{Hz}$, Hortho-phNCH $_3$), 7.01(d, 2H, $J = 6.05\text{Hz}$, Hpara-phOH), 6.85 (d, 2H, $J = 6.45\text{Hz}$, Hpara-phNCH $_3$), 2.4 (s, 6H, (CH $_3$) $_2\text{N}$); ^{13}C NMR (190, 165, 151, 146, 133, 131, 129, 125, 121, 117, 112, 42); MS: m/z 268 [M+H] $^+$.

General procedure for synthesis: 2-amino-4-(4-bromophenyl)-6-(4-chlorophenyl)nicotinonitrile LB1 [1]

A mixture of derivatives of Chalcone (0.002 mole) and malononitrile (0.0024 mole) and ammonium acetate (1.23 g, 0.016 mole) was dissolved in 25ml ethanol and then refluxed for 12h. Thin layer chromatography (TLC) was used to monitoring the reaction and confirmed that all starting material converted to the product. The reaction mixture was cooled and poured into the ice cold water (50 ml) and then HCl dil. solution was added to the reaction mixture to adjacent the pH. The precipitate was collected after filtration to give yellow product with title LB1 as a powder with yield 90% and melting point is (164°C); ^1H NMR, 8.34 (d, 2H, $J = 6.7\text{Hz}$, Hortho-phCl), 8.02 (s, 1H, H-py), 7.62 (d, 2H, $J = 7.70\text{Hz}$, Hpara-phCl), 7.55 (d, 2H, $J = 6.43\text{Hz}$, Hpara-phBr), 7.50 (d, 2H, $J = 6.87\text{Hz}$, Hortho-phBr), 6.70 (s, 2H, NH $_2$); ^{13}C NMR: 162, 156, 155, 137, 133, 129, 124, 122, 114, 133, 86); MS: m/z 383 [M] $^+$.

Synthesis of 2-amino-6-(4-(dimethylamino)phenyl)-4-(4-hydroxyphenyl)nicotinonitrile LB2

This ligand prepared by following the same procedure that used to prepare LB1. A reddish brown product was obtained with yield 85% Scheme 1. ^1H NMR, 9.70 (s, 1H, OH), 8.6 (s, 1H, H-py), 7.9 (d, 2H, $J = 6.06\text{Hz}$, Hortho-phNCH $_3$), 7.6(d, 2H, $J = 5.7\text{Hz}$, Hortho-phOH), 6.9 (d, 2H, $J = 7.2\text{Hz}$, Hpara-phNCH $_3$), 6.3 (d, 2H, $J = 7.6\text{Hz}$, Hpara-phOH), 6.8 (s, 2H, NH $_2$), 2.4 (s, 6H, (CH $_3$) $_2$); ^{13}C NMR: 165, 160, 158, 155, 153, 132, 131, 128, 117, 116, 112, 87, 43; MS: m/z 331[M+H] $^+$.



Scheme 1. Synthesis of LA, 2 and LB1, 2

2.2 Synthesis of metal complexes (LB1, 2-M)

A general procedure

A solution of metal ions salts (CrCl $_3$.6H $_2$ O, CoCl $_2$.6H $_2$ O, NiCl.6H $_2$ O and CuCl $_2$.2H $_2$ O) (0.01 mole) in ethanol was added to the hot solution of ligand (LB1 or LB2) (0.02 mole). Reaction mixture was refluxed for 2hr. The product was separation by filtration and washed with cold methanol for several times. The physical properties of new complexes were shown in Table 1, 2:

Table 1: Physical properties of ligand LB1 and its complexes

Compound	Color	M.P .C°	Mwt g/mol	Elemental analysis			Metal M% Found(cal.)	M:L	Suggested formula
				Found	Found	Found			
				C%	H%	N%			
LA1	Pale yellow	160	321.6	56.02 (55.97)	3.13 (3.1)			C ₁₅ H ₁₀ BrClO	
LB1	Light brown	164	384.6	56.20 (56.15)	2.88 (2.85)	10.92 (10.90)		C ₁₈ H ₁₁ BrClN ₃	
Cr-LB1	green	88	945.7	45.82 (45.6)	2.35 (2.53)	8.78 (8.88)	5.15 (5.49)	1:2	[Cr(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O)]Cl ₂
Co-LB1	green	130	935.1	46.34 (46.19)	2.59 (2.78)	9.21 (8.98)	6.27 (6.30)	1:2	[Co(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂
Ni-LB1	Light brown	106	934.8	46.35 (46.21)	2.59 (2.78)	9.04 (8.98)	6.21 (6.27)	1:2	[Ni(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂
Cu-LB1	brown	125	939.7	46.15 (45.97)	2.58 (2.76)	8.90 (8.93)	6.71 (6.76)	1:2	[Cu(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂

Table 2. Physical properties of ligand LB2 and its complexes

Compound	color	M.P .C°	Mwt g/mol	Elemental analysis %			Metal M% Found (cal.)	M:L	Suggested formula
				Found	Found	Found			
				C%	H%	N%			
LA2	Light yellow	158- 160	267	76.38 (76.31)	6.41 (6.35)	5.31 (5.23)		C ₁₇ H ₁₇ NO ₂	
LB2	Reddish brown	155	330	72.78 (72.72)	5.50 (5.45)	16.87 (16.96)		C ₂₀ H ₁₈ N ₄ O	
Cr-LB2	Dark green	175	836.5	57.53 (57.38)	4.35 (4.54)	13.36 (13.41)	6.18 (6.21)	1:2	[Cr(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O)]Cl ₂
Co-LB2	Greenish brown	300 d*	825.9	58.26 (58.11)	4.68 (4.84)	13.51 (13.56)	7.18 (7.13)	1:2	[Co(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂
Ni-LB2	Light brown	325 d*	825.6	58.28 (58.13)	4.65 (4.84)	13.48 (13.56)	7.21 (7.10)	1:2	[Ni(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂
Cu-LB2	brown	183	830.5	57.94 (57.79)	4.62 (4.81)	13.51 (13.48)	7.62 (7.65)	1:2	[Cu(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂

2. 3. Biological Study

In the current study, the agar well diffusion method was employed to examine the biological effect of investigated compound using two types of bacteria including *Escherichia coli* and *Bacillus subtilis*. In order to prepare stock solutions, testing compounds were dissolved in DMSO solvent. The first step in this process was to inoculate the agar medium with microorganisms. Then 20 µl of test solution (sample) was dispensed into well using a micropipette. The 96 well-plate was incubated at 30 °C for 72 hours. During this time, the sample was diffused. following the incubation period, OD measurements of microbial growth rate were achieved or the microbial growth rate was measured [18].

3. Results and Discussion

In this study, new complexes were prepared by using transition metals with new derivative ligands contain pyridine moiety bearing different groups, electron withdrawing (LB1) and donating group (LB2) by cyclization of Chalcone with malanitrile under basic condition according to procedure reported in the literature [1] to form new substituted according to

the molar ratio. The structure of all ligands and its metal complexes were characterization by using Elemental analysis, IR, UV, ¹H NMR and MS.

The electronic study of ligand LB1, LB2 and its complexes was carried out in ethanol which exhibited two main absorption characteristics for all complexes. The first band is assigned to the spin allowed intraligand π-π* transition of the coordinated ligand. It is generally observed at higher energies between (260-300) nm, the second broad band is metal to ligand charge transfer (MLCT) bands cyanopyridine and its metal complexes appearing between (350-450) nm and d-d transition which observed at 500-600 nm as shown in Table 3, 4. These new complexes were synthesis by mixing (2:1) ligand to metal with excellent yield.

according to literature [19]. The electronic spectra of new prepared Cr(III) complex with LB1 showed three bands at 40816, 29585 and 23310 cm⁻¹ respectively assigned to 4A_{2g} → 4T_{12g} (F), 4A_{2g} → 4T_{1g}(F) and 4A_{2g} → 4T_{1g}(P) respectively Figure 1. The magnetic moment for Cr(III) complexes was 3.50 B.M. UV/visible spectra results and magnetic moment are confirmed that the structure around Cr(III) ion has the octahedral structure which is similar to related complexes [20].

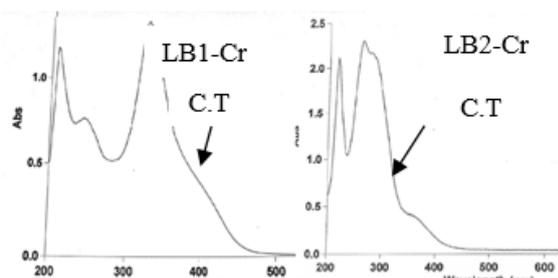


Fig. 1. UV spectra of LB1-Cr and LB2-Cr

In addition the molar conductivity was also indicated that complex was ionic as shown in Table 4 and the same transitions were exhibited for other complex with ligand LB2 [18, 20]. Ni(II) complexes show three band refer to ${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$, ${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$ and ${}^3A_{2g} \rightarrow {}^3T_{1g}(P)$ as shown in Table 3, 4. Which confirmed that the structure around Ni(II) ion is octahedral [19].

While Co(II) complexes exhibited three bands at 40160, 28169 and 15243 cm^{-1} which are attributed to the ${}^4T_{1g} \rightarrow {}^4T_{2g}(F)$, ${}^4T_{1g} \rightarrow {}^4A_{2g}(F)$ and ${}^4T_{1g} \rightarrow {}^4T_{1g}(P)$ respectively and indicated that the structure around Co(II) is octahedral, also the magnetic moment value was (4.23 B.M) confirmed that geometry for Co(III) complex is octahedral, 11764 cm^{-1} respectively attributed to the ${}^2E_g \rightarrow {}^2T_{2g}$ which indicated that the structure is octahedral around Cu(II) complexes. The value of magnetic susceptibility for all prepared complexes is consent with literature related to the same metals [21,22].

While Cu(II) complexes with LB1 and LB2 exhibited one band at 135501 and while, the elemental analysis and molar ratio shows that all metals coordination with ligand as a ratio 1:2. This result is agreement with molar conductance values which indicated that all complexes are electrolyte. Moreover, IR spectrum of ligand LA1,2 showed band at 1658 cm^{-1} assign to (C=O) group which disappeared in ligand LB1,2 and new band was observed at 2220 cm^{-1} indicate to the (C≡N) group [1] and shifted to lower frequencies in all complexes due to coordinate with metal ions, two bands at 3465, 3362 cm^{-1} in LB1 and 3315-3330 cm^{-1} in IR spectra of LB2. were attributed to NH_2 stretching vibration, these bands were shifted to higher frequencies as a result to coordination with metal ions (Table 5) [23] as shown in Figure 2, 3. A broad band was observed at in IR spectra for all complexes which are assigned to coordinate water molecule with metal ions. The new bands in the IR spectra for all metal complexes were observed in

520-540 cm^{-1} and 435-485 cm^{-1} the region are assigned to $\nu(\text{M-N})$ and $\nu(\text{M-O})$, respectively [24]

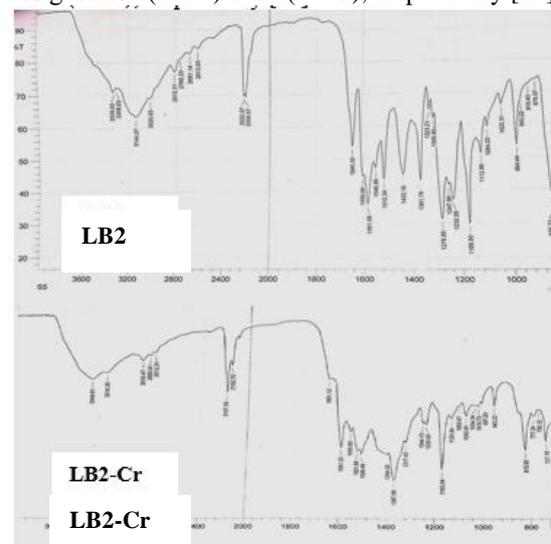
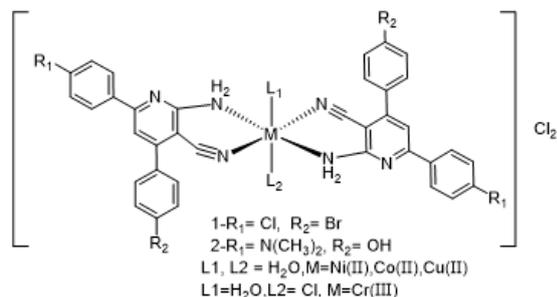


Fig. 3 The FTIR Spectrum of the ligand (LB2) and LB2-Cr

${}^1\text{H}$ NMR spectrum also confirmed the structure of ligand LA1 which exhibited doublet signal at 8.1 ppm assigned to proton of the α -olefin group and a doublet signal at 8.03 ppm attributed to the protons of aromatic ring close to ketone group. While ${}^{13}\text{C}$ NMR spectrum of LA1, LA2 show that carbon of carbonyl group was observed at 189.7 ppm. LA2 ligand shows a new two singlet signals at 9.7 ppm assigned to the proton of hydroxyl group and 2.4 ppm corresponded to the protons of alkyl group and rest of the protons are nearly similar to the LA1. A singlet signal at 8.6 ppm was observed in ${}^1\text{H}$ NMR spectrum of LB1 which attributed to the proton of pyridine ring also a doublet signal at 8.43 ppm indicated to the proton of aromatic phenyl ring (ph-Br), while a doublet signal with two integral were observed at 7.8 ppm assigned to the protons of phenyl ring (ph-Cl) in ligand LB1 and LB2.

In addition, ${}^1\text{H}$ NMR spectrum of LB2 is different to LB1 by showing peak at 2.4 ppm which attributed to alky group. Mass spectrum of LB1 showed main peak at 383 m/z indicate to $[\text{M}]^+$ and revealed the molecular formula ($\text{C}_{18}\text{H}_{11}\text{BrClN}_3$) while the mass spectrum of LB2 exhibited peak at 331 m/z assign to $[\text{M}+\text{H}]^+$ and suggested the molecular formula ($\text{C}_{20}\text{H}_{18}\text{N}_4\text{O}$), NMR spectroscopy along with mass spectrometry confirmed that the LB1 and LB2 were formed. ${}^1\text{H}$ NMR spectrum of complexes show the similar signals to the ligand with shift to down field. According to the literature reported that water molecules prefer to coordinate with metal ions as *trans*-octahedral cationic complexes due to this geometry is more stable than *cis* isomer and also the stabilization of the *cis* or *trans* geometry is driven by steric factors [25], as a result it suggests that the

structure configuration for ligand and their metals complexes as shown below:



Biological activities

The antibacterial study of the new complexes against gram positive bacteria such as (*Bacillus subtilis*) and gram negative bacteria such as (*E. coli*) was carried out in DMSO. The data was demonstrated that the ligand bearing electron withdrawing group LB1 and corresponding complexes exhibit higher active against to the both types of bacteria than the ligand substituted donating group LB2. Also, it was found that the metal ions complexes exhibit more toxic Compared to the corresponding ligand as shown in Table (7). The activity of these complexes are agreement with literature [26]

Table3. Electronic spectrum, Conductance and Magnetic moment (B.M) in (DMF) for LB1 and its Metal complexes

Compound	λ_{\max} nm	Bands cm ⁻¹	Transition	Molar Cond. $\mu\text{.cm}^{-1}$	μ_{eff} B.M	Suggested structure
C ₁₈ H ₁₁ BrClN ₃	208	48076	$\pi \rightarrow \pi^*$	-		
	338	29585	$n \rightarrow \pi^*$			
[Cr(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O)Cl]Cl ₂	245	40816	⁴ A _{2g} → ⁴ T _{2g} (F)	65.1	3.50	octahedral
	338	29585	⁴ A _{2g} → ⁴ T _{1g} (F)			
	429	23310	⁴ A _{2g} → ⁴ T _{1g} (P)			
[Co(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	249	40160	⁴ T _{1g} → ⁴ T _{2g} (F)	75.4	4.22	octahedral
	355	28169	⁴ T _{1g} → ⁴ A _{2g} (F)			
	656	15243	⁴ T _{1g} → ⁴ T _{1g} (P)			
[Ni(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	251	39840	³ A _{2g} → ³ T _{2g} (F)	67.3	2.85	octahedral
	334	29761	³ A _{2g} → ³ T _{1g} (F)			
	354	28248	³ A _{2g} → ³ T _{1g} (P)			
[Cu(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	738	135501	² E _g → ² T _{2g}	70.5	1.74	octahedral

Table 4. Electronic spectrum, Conductance and Magnetic moment (B.M) in (DMF) for LB2 and its Metal complexes

Compound	λ_{\max} nm	Bands cm ⁻¹	Transition	Molar Cond. $\mu\text{.cm}^{-1}$	μ_{eff} B.M	Suggested structure
C ₂₀ H ₁₈ N ₄ O	215	46511	$\pi \rightarrow \pi^*$			-
	341	29325	$n \rightarrow \pi^*$			
	245	40816	⁴ A _{2g} → ⁴ T _{2g} (F)			
[Cr(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O)Cl]Cl ₂	330	30303	⁴ A _{2g} → ⁴ T _{1g} (F)	63.2	3.78	octahedral
	435	22988	⁴ A _{2g} → ⁴ T _{1g} (P)			
	240	41666	⁴ T _{1g} → ⁴ T _{2g} (F)			
[Co(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	360	27777	⁴ T _{1g} → ⁴ A _{2g} (F)	72.3	4.23	octahedral
	650	15384	⁴ T _{1g} → ⁴ T _{1g} (P)			
	260	38461	³ A _{2g} → ³ T _{2g} (F)			
[Ni(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	345	28985	³ A _{2g} → ³ T _{1g} (F)	70.2	2.79	octahedral
	370	27027	³ A _{2g} → ³ T _{1g} (P)			
	850	11764	² E _g → ² T _{2g}			
[Cu(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	850	11764	² E _g → ² T _{2g}	74.7	1.78	octahedral

Table 5. IR-spectrum of ligand LB1 and its complexes

Compound	ν_{NH_2}	$\nu_{\text{C}\equiv\text{N}}$	M-N	M-O	$\nu_{\text{H}_2\text{O}}$
C ₂₀ H ₁₈ N ₄ O	3335, 3144	2222	-	-	-

[Cr(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O)Cl]Cl ₂	3338, 3210	2187	532	480	3335
[Co(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	3338, 3228	2183	530	480	3338
[Ni(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	3360, 3220	2183	530	435	3350
[Cu(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	3346, 3219	2187	535	475	3346

Table 6. IR-spectrum of ligand LB2 and its Complexes

Compound	ν_{NH_2}	$\nu_{\text{C}=\text{N}}$	M-N	M-O	$\nu_{\text{H}_2\text{O}}$
C ₁₈ H ₁₁ BrClN ₃	3460, 3262	2208	–	–	–
[Cr(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O)Cl]Cl ₂	3465, 3264	2212	530	435	3317
[Co(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	3480, 3240	2211	528	475	3315
[Ni(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	3466, 3217	2211	535	475	3333
[Cu(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	3465, 3240	2212	540	440	3308

Table 7. Antibacterial activity of ligand LB1, LB2 and its complexes

Compound	<i>Bacillus subtilis</i>	<i>E. Coli</i>
DMSO	0	0
C ₁₈ H ₁₁ BrClN ₃ (LB1)	20	10
[Cr(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O)Cl]Cl ₂	30	12
[Co(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	25	14
[Ni(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	22	12
[Cu(C ₁₈ H ₁₁ BrClN ₃) ₂ (H ₂ O) ₂]Cl ₂	28	15
C ₂₀ H ₁₈ N ₄ O (LB2)	14	8
[Cr(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O)Cl]Cl ₂	21	10
[Co(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	23	10
[Ni(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	20	12
[Cu(C ₂₀ H ₁₈ N ₄ O) ₂ (H ₂ O) ₂]Cl ₂	24	14

4. Conclusion

In conclusion new complexes were synthesis and characterized by spectroscopic and analytical method. All ligand and their corresponding complexes were tested against two types of bacteria and all were showed high antibacterial activity. The complexes bearing ligand subtitled electron withdrawing were exhibited higher activity than the other complexes. So this can be promising as antibacterial agent.

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6. References

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تحضير ودراسة طيفية وبيولوجية لمشتقات 2-أمينو-3-سيانو بيريدين ومعقداتها مع العناصر الانتقالية

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الخلاصة

تتضمن هذه الدراسة تحضير مشتقات جديدة معوضه من مشتق والذي حضر عن طريق تفاعل بين 2-amino-3-cyanopyridine بوجود اسيتات الأمونيوم malanonitrile مشتقات و chalcone ليعطي نواتج ممتازة من الليكاندات المحضرة. والمعقدات الجديدة حضرت عن طريق تفاعل هذه الليكاندات مع العناصر الانتقالية وتم تشخيصها بالطرق التحليل العناصر وأطياف الأشعة فوق البنفسجية Mass UV والأشعة تحت الحمراء IR، وأطياف NMR (1H, 13C) and وجد ان لها الصيغة العامة $[ML_2(H_2O)_2]Cl_2$, where Cu(II) = M, Co(II), Ni(II), Cr (III) = M, $[ML_2(H_2O)Cl]Cl_2$ وإشارات هذه الدراسات ان الليكاند يتناسق مع أيون الفلز كليكاند ثنائي وأيضا وجد ان المعقدات المحضرة لها شكل ثماني السطوح من خلال ارتباطه عن طريق ذرة الكربون في مجموعة السيانو وذره النتروجين في مجموعة الأمين. الفعالية المضاد للبكتريا فقد درست لكل من البليكاندات والمعقدات antimicrobial (*Bacillus subtilis*) ضد نوعين من البكتريا الموجب (البكتريا *Pseudomonas aeruginosa*) السالبة كما وجد ان هذه المعقدات ظهرت فعالية ضد البكتريا افضل من ليكانداتها.