

Synthesis, spectral and antimicrobial activity of mixed ligand complexes of Co(II), Ni(II), Cu(II) and Zn(II) with *Anthranilic Acid and Tributylphosphine*

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Abstract

Mixed ligand complexes of bivalent metal ions, viz; Co(II), Ni(II), Cu(II) and Zn(II) of the composition $[M(A)_2((PBU_3)_2)]_n$ in (1:2:2)(M:A:(PBU₃)). molar ratio, (where A⁻ Anthranilate ion, (PBU₃)= tributylphosphine. M= Co(II),Ni(II),Cu(II) and Zn(II).

The prepared complexes were characterized using flame atomic absorption, by FT-IR, UV/visible spectra methods as well as magnetic susceptibility and conductivity measurements. The metal complexes were tested in vitro against three types of pathogenic bacteria microorganisms: (*Staphylococcus*, *Klebsiella SPP* and *Bacillus*) to assess their antimicrobial properties. Results. The study shows that all complexes have octahedral geometry; in addition, it has high activity against tested bacteria. Based on the reported results, it may be concluded that. The results showed that the deprotonated ligand (anthranilic acid) to anthranilate ion (A⁻) by using (KOH) coordinated to metal ions as bidentate ligand through the oxygen atom of the carboxylate group (–COO⁻), and the nitrogen atom of the amine group (-NH₂), where the Tributylphosphine coordinated as a monodentate through the phosphorus atom.

Keywords : Mixed (anthranilic acid or tributylphosphine) complexes, spectroscopy studies, mixed ligands and Antibacterial Activities

1. Introduction

Anthranilic acid (or o-amino-benzoic acid) is an organic compound with the molecular formula $C_7H_7NO_2$. The molecule consists of a benzene ring, hence is classed as aromatic, with two adjacent, or "ortho-" functional groups, a carboxylic acid and an amine. Thermodynamic and electrical functions of aminophenol and anthranilic acid complexes with Mn(II), Fe(II), Co(II), Ni(II) and Cu(II) were determined. Ga(III), Ho(III), and Ce(III), were calculated with the help of stability constant values at different temperatures. It was found that the complexing processes have an exothermic nature. The studied complexes behave like semiconductors. (1) The conduction takes place according to hopping mechanism. There have many reports on the metal-anthranilic complexes. Some transition metal anthranilic capability for aren't hydrogenation(2,3). The new substituted anthranilic acid derivatives as potent anti-inflammatory agents, the structure of these compounds have been established by IR, 1H NMR spectroscopic and elemental analysis(4). There are many reports on the metal-anthranilate complexes along with the structure of many of these compounds. Some transition metal anthranilate have capability for aren't hydrogenation.[5-8] Tributylphosphine is the organophosphorus compound with the formula $P(C_4H_9)_3$ ($C_{12}H_{27}P$). Abbreviated or PBu_3 , it is a tertiary phosphine. It is an oily liquid at room temperature, with a nauseating odor.[9]. Tributylphosphine most commonly encountered as a ligand in transition metal complexes and is also a common ligand for the preparation of complexes of transition metals in low oxidation states.[10] A series of square-planar nickel(II) hexamethylenedithiocarbamate complexes with heterogeneous coordination spheres of composition $[NiX(hmidtc)Y] \cdot nCHCl_3$ [$X = Cl, Br, I$ or NCS ; $hmi = C_6H_{12}$, $dtc = S_2CN^-$; $Y = PPh_3$ or PBu_3 , $n = 0, 1$] have been synthesized and characterized by elemental analyses, i.r. and u.v.-v.is. spectroscopy, magneto chemical and conductivity measurements, and by thermal analysis. X-ray structures of $[NiCl(hmidtc)(PPh_3)] \cdot kCHCl_3$ and $[NiBr(hmidtc)(PPh_3)] \cdot CHCl_3$ have been determined. [11]

The present paper reports the synthesis and characterization of new Co(II), Ni(II), Cu(II) and Zn(II) complexes with mixed anthranilic acids acid and tributylphosphine.

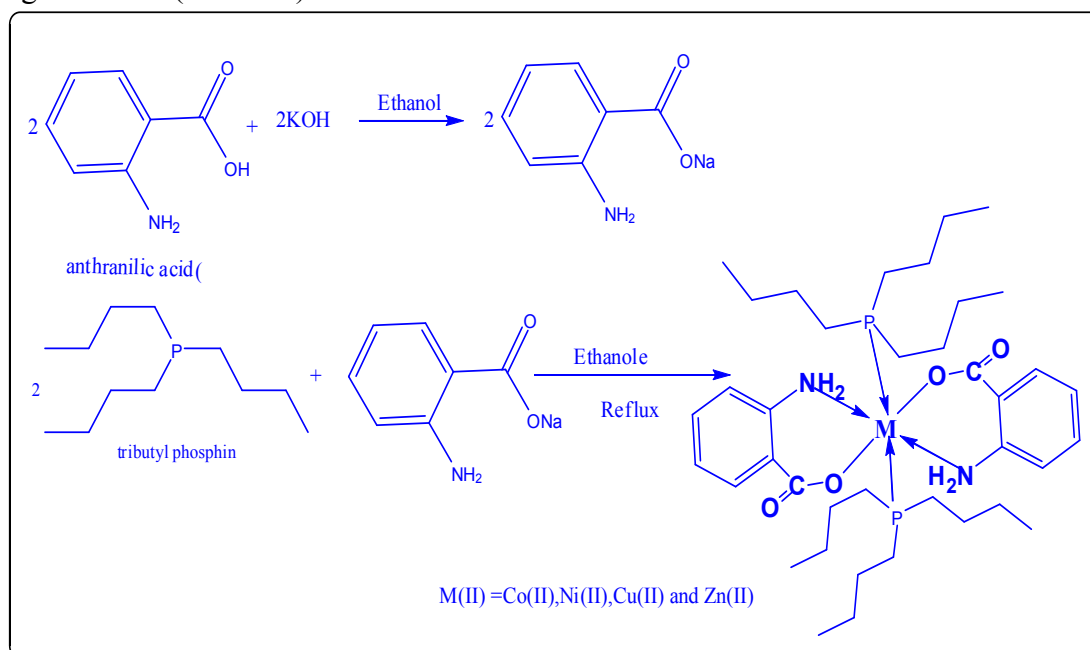
2-Experimental

2-1-Instruments: FTIR spectra were recorded as K Br discs using Fourier transform Infrared Spectrophotometer Shimadzu 24 FTIR 8400s. Electronic spectra of the prepared complexes were measured in the region (200- 1100) nm for 10^{-3} M solutions in DMSO at 25°C using shimadzu-U.V-160.A Ultra 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 room temperature for 10^{-3} M solutions of the samples in DMSO using pw9527 Digital conductivity meter (Philips). Melting points were recorded by using Stuart melting point apparatus. chloride ion content were also evolution by (Mohr method), Magnetic susceptibility measurements were measured using Bruker magnet BM6 instrument at 298°K following the Faraday's method. The proposed molecular structure of the complexes were determined by using chem. office program, 3DX (2006).

1-2-Materials: The following chemicals were used as received from suppliers; cobaltous chloride hexahydrate 98.8%, nickel chloride hexahydrate 99.9%, copper chloride dihydrate 98%, zinc chloride 98.8% (Merck), tributylphosphine and anthranilic acid (B.D.H).

2-3-Preparation of Metal Complexes (general procedure)

An aqueous solution of the metal salts containing 0.118g, 0.118g, 0.085g and 0.068g (1mmole) of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and ZnCl_2 respectively was added gradually with stirring to ethanolic KOH solution (0.264g, 2mmol) of the anthranilic acid (potassium anthranilate). (0.5ml, 2mmole) of tributylphosphine (PBu_3) was added to the mixture in each case by using stichiometric amount (1:2:2) Metal: K^+ Anth $^-$:(PBu_3) molar ratio. The mixture was refluxed with constant stirring for an hour. The mixture was cooled at room temperature pale precipitate was formed, filtered and recrystillized from ethanol and dried at room temperature according to the following reaction : (scheme1)



Scheme (1) : Schematic representation Preparation of the Complexes $[\text{M}(\text{A})_2(\text{PBu}_3)_2]$

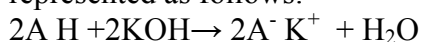
2.4. Antimicrobial activities

Antimicrobial activities of the ligands and there complexes $[\text{M}(\text{A})_2(\text{PBu}_3)_2]$ have been carried out against three types of pathogenic bacteria. One type of bacteria was gram positive which is *staphylococcus*. The second one was gram negative which is *Klebsiella* SPP. The third one was gram variability which is *Bacillus* using nutrient agar medium by disc diffusion method [8] . The test solution were prepared in DMSO and soaked in filter paper of 5 mm diameter and 1mm thickness. These discs were placed on the already seeded plates and incubated at 37°C for 24 h. The diameters (mm) of the inhibition zone around each disc were measured after 24 hours. [12-13].

3. Results and Discussion

3.1. Characterization of Metal Complexes.

Generally, the complexes were prepared by reacting the respective metal salts as chloride with the ligands using 1:2:2 mole ratio, i.e. one mole of metal chloride : two mole of tributylphosphine and two moles of potassium anthranilate. The synthesis of mixed ligand Metal complexes may be represented as follows:



(where PBu₃ is tributylphosphine and AH is anthranilic acid, M= Co(II), Ni(II), Cu(II) and Zn(II).

The formula weights and melting points, are given in (Table I). Based on the physicochemical characteristics (Table 1), it was found that all the complexes were non-hygroscopic, stable at room temperature indicating a strong metal-ligand bond. The solubility of the complexes of ligands were studied in various solvents. The complexes are soluble in dimethyl sulfoxide (DMSO) and dimethyl formamide (DMF) while insoluble in water. The observed molar conductance values measured in (DMSO) in 10⁻³ M solution lie in the (18.53-32.61)Ω⁻¹ cm²mol⁻¹ range, the conductivity measurements in DMSO indicated the non electrolyte behavior [13].

3.2. The atomic absorption measurements and chloride ion content

The atomic absorption measurements and chloride ion content (Table-1) for all complexes gave approximated values for theoretical values. In conclusion, our investigation this suggest that the ligands anthranilic acid and tributylphosphine coordinate with M (II) forming octahedral geometry.

3.3. Magnetic moment and Electronic spectra:

The magnetic moments of the complexes shown in Table (2) were calculated from the measured magnetic susceptibilities after employing diamagnetic corrections. [14-17] The magnetic moments obtained at room temperature for the complexes of Cu(II), Ni(II) and Co(II) are listed (Table 2). Cu(II) complex exhibits magnetic moment of (1.68B.M) which is less than the normal value (2.20-1.84). The lowered magnetic moment value observed for Cu(II) complex under present study is due to distorted octahedral geometry[11]The Co(II) complex shows magnetic moment of 4.73B.M. the spin free octahedral complex are reported to exhibit magnetic moment in the range of 4.46- 5.53 B.M[17]. The Ni(II) complex shows magnetic moment of 2.69 B.M. The magnetic moment of octahedral Ni(II) complex are reported to exhibit magnetic moment in the range of 2.80 – 3.40 B.M. 3.05 including spin orbital coupling contribution from 3A_{2g} and higher 3 T_{2g} states. Hence the observed magnetic moment for the Ni(II) complex suggest that it may have octahedral geometry [17]. Typical spectral data of the metal salts, ligands and complexes of Transition and Inner transition metal complexes have been investigated in DMSO and shown in table 3. The electronic spectra of the anthranilic acid (AH) displays absorption bands at 262, and 345 which is assigned to (π – π*) and (n – π*) electronic transitions respectively. The electronic spectra of the tributylphosphine displays absorption band at 297nm, which is assigned to(π → π*) electronic transition. [1-11-17]

The electronic spectrum of Co(II) complex showed peak at 269 nm due to charge transfer. Other three peaks at 612 nm, 668 nm and 926 nm were found to be caused by (d-d) electronic transition type ⁴T_{1g(F)} → ⁴T_{1g(P)}, ⁴T_{1g(F)} → ⁴A_{2g(F)} and ⁴T_{1g(F)} → ⁴T_{2g(F)} respectively [11]. The spectrum of Ni(II) complex appeared absorption peak at 278 nm was related to charge transfer, then other two peaks at 398 nm and 672 nm were assigned to electronic transition type ³A_{2g(F)} → ³T_{1g(P)} and ³A_{2g(F)} → ³T_{1g(F)} respectively [10]. The spectrum of Cu(II) complex gave absorption peak at 311 nm due to charge transfer. The peak at 433 nm was caused by electronic transition [18-11] ²E_g → ²T_{2g}. The spectrum of Zn(II) complex showed absorption peak at 273nm due to charge transfer. The absence of absorption peaks in the visible region indicated no (d-d) electronic transition happened; this is a good result for octahedral complex

3.4. Fourier transform infrared spectra:

The most important infrared spectral bands that provide conclusive structural evidence for the coordination of the ligands to the central metal ions. FT-IR Spectra The relevant vibration bands of the free ligands and the complexes are in the region 4000-400 cm⁻¹, [18 -20]. The characteristics frequencies of the ligands and mixed ligand complexes of the metals are given in (Table-3).

In order to study the binding mode of the (anthranilic acid) with the metal ions, a comparison was made for the FT.IR spectrum of the free anthranilic acid and those of the prepared complexes and the data was tabulated in (Table-3). The Fourier transform infrared spectrum of free ligand

anthranilic acid exhibited a strong band around 3325 cm^{-1} and 3238 cm^{-1} corresponds to the stretching vibration of $\nu(\text{N-H}) + \nu(\text{O-H})$, on complexation a shifting with change in shape were observed from these bands, while increasing in intensity were noticed. The spectra arising from the complexes reveals that the absorption band belonging to the stretching vibration of $\nu(\text{N-H})$ of the amine group have been found in the range between $(3304\text{-}3298)$ and $(3136\text{-}3126)\text{ cm}^{-1}$ shifted to lower frequency by $(21\text{-}11)$ and $(98\text{-}92)\text{ cm}^{-1}$ suggesting the possibility of the coordination of the ligand (A^-) through the nitrogen atom at the amine group.[13-15]

The significant may be a result of coordination with metal ion. The bands at 1664 cm^{-1} and 1589 cm^{-1} in the anthranilic acid spectrum ascribed to $\nu(\text{COO}^-)_{\text{asym}}$ and $\nu(\text{COO}^-)_{\text{symmetric}}$ stretching vibration of suffered a great change to lower frequency were also observed on complexation with metal ion. [15-16] For (FT- I.R) spectrum for the tributylphosphine a strong band around $(1114)\text{cm}^{-1}$ and $(2900)\text{cm}^{-1}$ were assigned to the $\nu(\text{C-C})$ and $\nu(\text{C-H})$ aliphatic stretching vibration respectively .

The infrared of prepared complexes have shown weak bands in the range of $(531\text{-}565)\text{cm}^{-1}$, $(430\text{-}520)\text{ cm}^{-1}$ which was attributed to the of, $\nu(\text{M-N})$, $\nu(\text{M-O})$ (Metal-Anthranilate ions) respectively and in the range of $(416\text{-}424)\text{ cm}^{-1}$ which was attributed to the of $\nu(\text{M-P})$ tributylphosphine stretching bands. According to the results obtained and spectral analysis an octahedral structure has been suggested to these complexes.

3.5. Proposed molecular structure :

Studying four complexes on bases of the above analysis , the existence of Hexa coordinated $[\text{M}(\text{C}_7\text{H}_6\text{NO}_2)_2(\text{C}_{12}\text{H}_{27}\text{P})_2]$ were, $\text{M(II)} = \text{Co(II), Ni(II), Cu(II)}$ and Zn(II) . Anthranilate ion= $(\text{C}_7\text{H}_6\text{NO}_2^-)$, tributylphosphine = $(\text{C}_{12}\text{H}_{27}\text{P})$ proposed models of the species were built with chem. 3D shows in Figure(1).

4. Antibacterial Activities:

The zone inhibition of bacterial growth were measured in mm depending upon the diameter as shown in Table (4) Figure. (2)The antibacterial activity results revealed that the ligands and there complexes Scheme (2) shown weak to good activity when compared to the Control (DMSO). [21]. The antibacterial results evidently showed that the activity of the mixed ligand complexes more pronounced when coordination to the metal ions .The activity of these substance may be due to carboxyl group . [12-12]

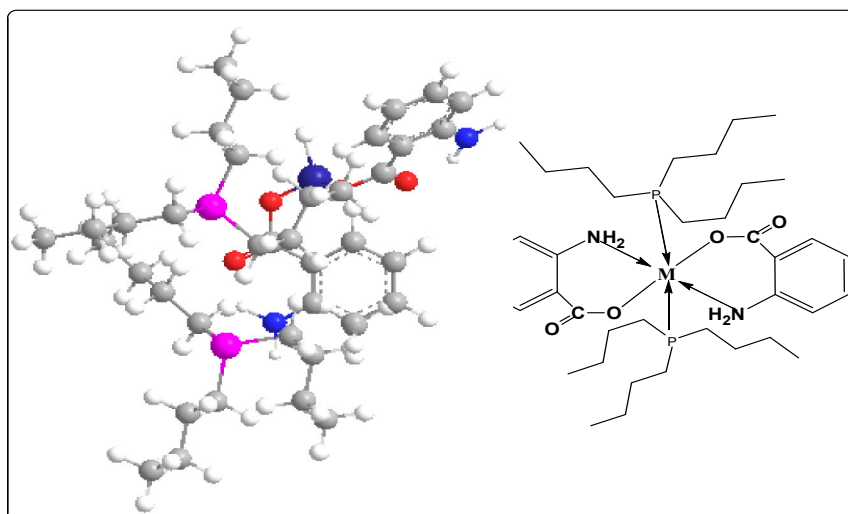


Figure (1) : The proposed structure and 3D-geometrical structure of the complexes $[\text{M}(\text{A})_2(\text{PBu}_3)_2]$, $\text{M} = \text{Co(II), Ni(II), Cu(II)}$ and Zn(II)

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Table(1): Physical Properties for metal Complexes

Compounds	Color	Melting Point (°C)	Yield%	M%	Λ_m $\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$ in DMSO(10^{-3} M)	μ_{eff} (B.M)	Cl %
[Co(A) ₂ (PBu ₃) ₂]	green	210	73	8.03 (7.63)	21.67	4.73	Nil
[Ni(A) ₂ (PBu ₃) ₂]	Pale green	260	72	7.90 (7.15)	18.53	3.05	Nil
[Cu(A) ₂ (PBu ₃) ₂]	Blue	235	75	8.64 (7.82)	32.61	1.68	Nil
[Zn(A) ₂ (PBu ₃) ₂]	Pal yellow	275	71	8.77 (7.58)	27.82	Dia	Nil

Table (2): Electronic Spectral data, magnetic moment, of the studied complexes

Compounds	λ_{max} (nm)	ν - Wave number (cm^{-1})	ϵ_{max} ($\text{L.mol}^{-1}.\text{cm}^{-1}$)	Assignment	μ_{eff} (BM) Found (Calc)
AH	262 345	38167 28985	404 1318	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-
PBu ₃	297	33670	6095	$\pi \rightarrow \pi^*$	-
[Co(A) ₂ (PBu ₃) ₂]	269 612 668 926	37174 16339 14970 10799	1530 50 61 7	charge transfer ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$, ${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$ ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$	4.73 (3.87)
[Ni(A) ₂ (PBu ₃) ₂]	278 398 672	35971 25125 14880	142 122 66	charge transfer ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(P)}$ ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(F)}$	3.0 (2.82)
[Cu(A) ₂ (PBu ₃) ₂]	311 433	32154 23094	1918 286	charge transfer ${}^2E_g \rightarrow {}^2T_{2g}$	1.68 (1.73)
[Zn(A) ₂ (PBu ₃) ₂]	273	36630	1925	charge transfer	0.00 diamagnetic

Table (3) : FTIR spectral data of the Ligands and there complexes

Compounds	$\nu(\text{NH}_2)$	$\nu_{\text{as}}(\text{COO})$	$\nu_{\text{s}}(\text{COO})$	Δ $\nu(\text{COO}^-)$ asym - - sym	$\nu(\text{M-N})$	$\nu(\text{M-O})$	$\nu(\text{M-P})$
Anthranilic acid (AH)	3325 sh. 3228 sh.	1675 s.	1550 s.	125	-	-	-
[Co(A) ₂ (PBu ₃) ₂]	3331 sh-br. 3136 sh.	1691 sh.	1546 s.	145	565 w.	516 w.	418 w.
[Ni(A) ₂ (PBu ₃) ₂]	3304 sh. 3126 sh.	1684 s.	1554 s.	130	550 w.	520 w.	424 w.
[Cu(A) ₂ (PBu ₃) ₂]	3314 sh. 3120 sh.	1695 s. sh.	1550 s.	145	531 w.	447 w.	416 w.
[Zn(A) ₂ (PBu ₃) ₂]	3298 sh. 3128 sh.	1695 s.	1539 s.	156	552 w.	430 w.	420 w.

Table (4): Showed the inhibition circle diameter in millimeter for the bacteria after 24 hour incubation paid and 37°C for complexes

Compound	<i>Bacillus</i>	<i>Klebsiella SPP</i>	<i>staphylococcus</i>
Control DMSO	10.9	9.9	13.3
(PBU ₃)	12.9	10.9	16
(AH)	17.9	13.9	20.7
[Co(A) ₂ (PBU ₃) ₂]	18.5	15.9	28.6
[Ni(A) ₂ (PBU ₃) ₂]	20.3	18.1	20.2
[Cu(A) ₂ (PBU ₃) ₂]	20.1	17.1	20.1
[Zn(A) ₂ (PBU ₃) ₂]	20.3	15.1	21.2

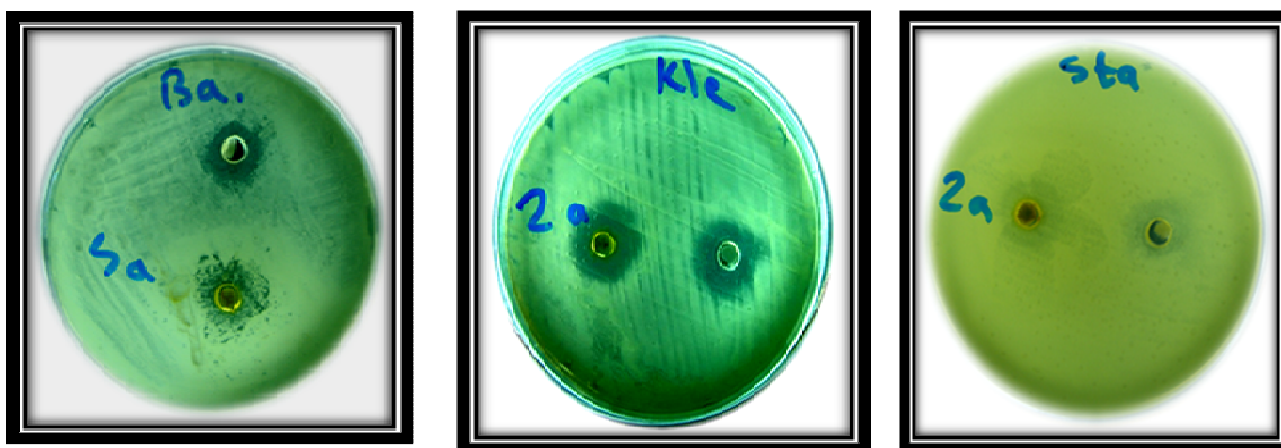
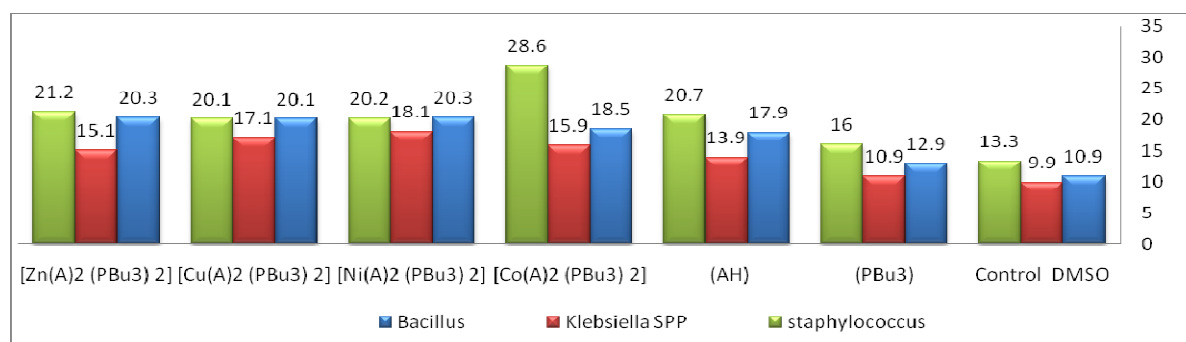


Figure.2: Shows the antimicrobial activity of (ligands Left (AH) → Right- PBU₃) appear the inhibition zones against pathogenic bacteria



Scheme (2): Inhibitory activity of the ligands and there metal complexes Against (*Staphylococcus*, *Klebsiella SPP*.and *Bacillus*)

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