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Preparation and Characterization of Some Metal Complexes with New Heterocyclic Schiff-Azo Ligand

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Abstract

Chelation complexes of Cu(II), Hg(II), Zn(II) Ni(II) Co(II) Cd(II) were prepared with new heterocyclic Schiff azo ligand (E)-N-(١-(٤-((E)-(٤,٥-diphenyl-١H-imidazol-٢-yl) diazenyl) phenyl) ethylidene)-٤-methylaniline. This ligand was synthesized and characterized by IR spectra, U.V–visible spectra, (C.H.N) analysis and its complexes were characterized by flame atomic absorption, molar conductivity and magnetic moment. It was found that the Schiff azo ligand behaves as a neutral bidentate (N,N′) ligand forming chelates with (١: ٢) (metal: ligand) stoichiometry. Depending upon all results was proposed an octahedral geometry for the complexes.

Keywords: Imidazol, Schiff Base, Azo Complexes, Metal complexes, Schiff-Azo ligand.

Introduction

Azo Schiff base complexes contain both azo and azomethine groups. The azo group possesses excellent donor properties and is important in coordination chemistry [١,٢]. A large number of (N,N′)-doner ligands in azo imine family have been prepared in the last few years [٣-٦]. The growing interest in heterocyclic azo dye chem -istry is focused on designing new synthetic appr -oaches to these materials, theoretical calcu-lations, and applications in various industrial fields. Besides having important -applications as textile colorants [٧,٣١], some azo compoundshave been shown an antibacterial agents [٨-١٠,١٥]. This class of azo compounds possess an active (π-acidic) azo imine (─N═N─C═N─), wich function as efficient agents to stabilizes low valent metal oxidation states [١١,١٢], due to the presence of azo-centered π*-molecular orbital, for this reason a number of these azo compounds were synthesized and their abilities as chelating ligands [١٢-١٤] were investigated.

Material sand Method

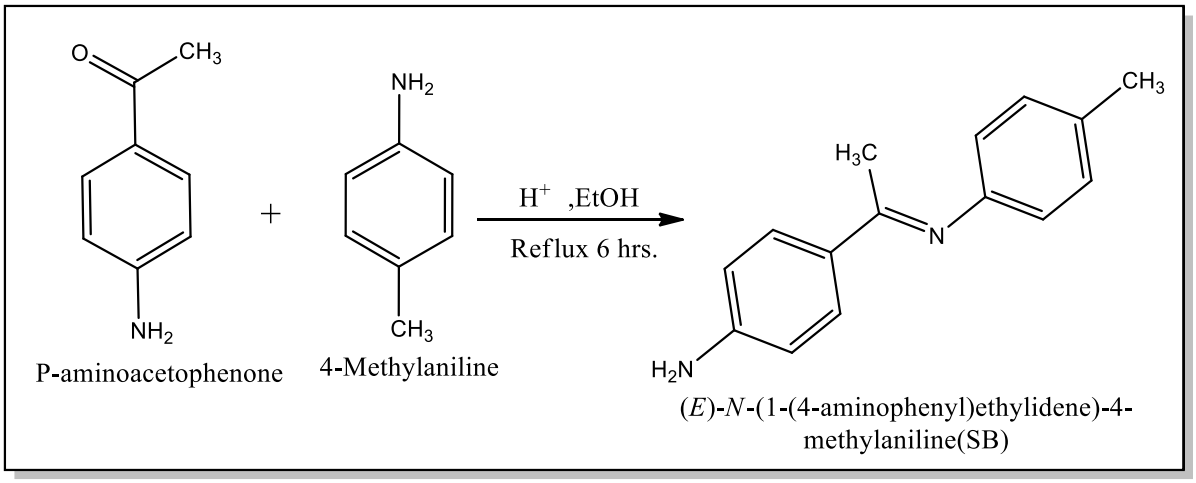
Materials and Measurements

All the used reagents and solvents had at least analytical grade, except of ٤,٥-diphenyl imidazole was prepared as reported procedure [١٥] the buffer solutions were prepared as described earlier [١٦]. Melting points were determined by open capillary tube method and were uncorrected by using a Stuart melting point (digital SMP٣٠) apparatus. The metal contents of the complexes was measured using atomic absorption technique by Shimadzu AA-٦٣٠٠. IR spectra were recorded on a Shimadzu ٨٠٠٠ FT-IR spectrophotometer in the (٤٠٠٠-٤٠٠) cm^{-1}rangeusing KBr discs. Electronic spectra were obtained on a Shimadzu ١٧٠٠UV spectrometer using ethanol as a solvent in the (٨٠٠-٢٠٠) nm range Magnetic susceptibilities were determined by Faraday method at room temperature using Balance Magnetic (MSB -MKI) apparatus, and diamagnetic corrections for the ligand were calculated using Pascal's constant [١٧]. Molar conductance of the prepared metal complexes were determined in Ethanol using conductivity meter Alpha-٨٠٠ at ٢٥°C, A concentration of the solutions was ($١٠^{-٣}$ mol. $L^{-١}$).

Preparation of [N-(١-(٤-aminophenyl) ethylidene)-٤-methylaniline](SB)

Schiff base was prepared by condensation reaction of (٤-methylaniline) with compound (٤-aminoacetophenone), by dissolving (١,٣٥g, ٠,٠١ mol) of (٤ – aminoacetophenone) in (٢٠ ml) absolute ethanol then mixed with a solution (١,٠٧g, ٠,٠١mol) of (٤-methylaniline) dissolved in (٢٠ ml) of the same solvent with the addition of four drops of Glacial acetic acid followed by reflux for (٦) hours [١٨], The solution was left to cool then poured over the ice observe the appearance of white

precipitate, It was filtered, dried and recrystallized from ethanol hot absolute to get white pure crystals of Schiff base, the yield was calculated (٧١٪) also reached a melting point (٨٠-٨٢°C), Equation (١) describes Preparation of the Schiff base (SB).



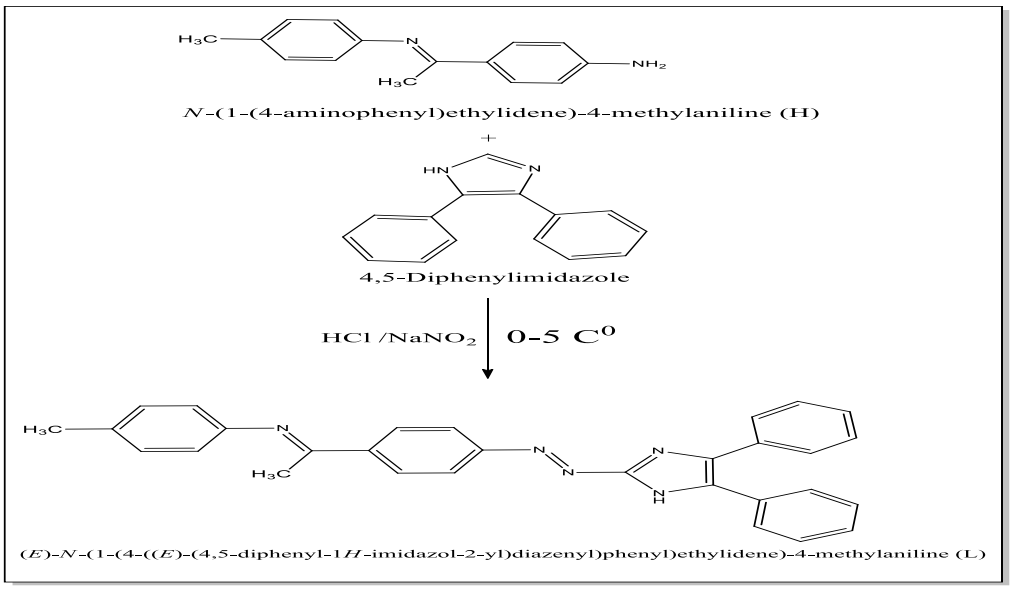
Scheme (١) Preparation of the Schiff base (SB).

Preparation of Schiff-azo ligand (E)-N-(١-(٤-((E)-(٤,٥-diphenyl-١H-imidazol-٢-yl) diazenyl) phenyl) ethylidene) -٤-methylaniline (L)

Schiff-azo ligand was prepared according to the following generalprocedure [١٩]:-

(٢,٢٤ g, ٠,٠١mol) from (SB)was dissolved in a mixture ١٠ml hydrochloric acid and ٢٠ml of distilled water cold and diazotized below ٥°C with (٠,٨٠ g, ٠,٠١١mol) of sodium nitrite dissolved in (١٠ ml) of distilled water. Then the solution was filtered. The resulting was diazonium chloride. the solution was mixed with ٤,٥-diphenyl imidazole (٢,٢ g, ١٠ mmol) dissolved in a mixture consisting of (١٥٠ ml) and ethanol (٥٠ ml) sodium hydroxide (١٠٪).

After leaving in the refrigerator for ٢٤ hrs, the mixture was acidified with dilute hydrochloric acid until pH = ٥ The precipitate was filtered off and recrystallized twice from hot ethanol and dried the yield was (٦٨٪), the melting point was (١٣٠-١٣٤) the Table (١) show physical and analytical data of the ligand and its starting materials, Equation (٢) describes Preparation of the Schiff-azo ligand.



Scheme (٢) Preparation of the Schiff-azo ligand.

Table (١)
Physical properties and analytical data of the ligand and its starting materials.

Compound Symbol	*Compound Name*	*Impirical Formula*	*M.Wt*	*Color*	*Melting Point ˚C*	*Yield Percent %*
A	٤,٥-Diphenylimidazole	$C_{١٥}H_{١٢}N_{٢}$	٢٢٠	White	٢٢٩-٢٣٠	٨٥٪
SB	N-١-(٤-aminophenl) ethylidene]-٤-methylaniline-(E)	$C_{١٥}H_{١٦}N_{٢}$	٢٢٤	White	٨٠-٨٢	٧١٪
L	(E)-N-(١-(٤-((E)-(٤,٥-diphenyl-١H-imidazol-٢-yl) diazenyl) phenyl) ethylidene)-٤-methylaniline	$C_{٣٠}H_{٢٥}N_{٥}$	٤٤٥	Red	١٣٠-١٣٤	٦٨٪

Synthesis of complexes

The chelate complexes were synthesized at optimal pH values (Table (٣)) dissolved (٠,٤٥٥ g, ١x١٠$^{-٣}$mol) of ligand (Schiff-azo)in (٥٠ ml) ethanol and then (١x١٠$^{-٣}$mol)of metal chloride (٠,١١٨٥ gm $CoCl_{٢}.٦H_{٢}O$), (٠,٠٨٥ gm $CuCl_{٢}.٢H_{٢}O$), (٠,١١٨٥ gm $NiCl_{٢}.٦H_{٢}O$), (٠,٠٦٨ gm $ZnCl_{٢}$), (٠,١٠٩٥ gm $CdCl_{٢}.٢H_{٢}O$) and (٠,١٣٥٥ gm $HgCl_{٢}$), dissolved in ٥ ml of buffer solution of ammonium acetate was added dropwise with stirring to the ligand solution. The complexes were filtered off, washed with distilled water then collects the physical properties and analytical data for these complexes are shown in Table (٢).

Table (٢)
Physical properties and analytical data of the complexes.

Complexes	*M.Wt*	*Color*	*M. p°c*	*Molar conductivity ΛM* $\Omega^{-١}cm^{٢}mol^{-١}$ *١٠$^{-٣}$M in Ethanol*	*Metal%*		M_{eff} *(B.M)*
					Calc.	*found*	
$[Cu(L)_{٢}Cl_{٢}]$	١٠٤٤	Brown	٩٨ de	٣,٩	٦,٠٣	٥,٣٥	١,٨٣
$[Co(L)_{٢}Cl_{٢}]$	١٠٣٩	Deep orang	١١٨-١٢٠	٣,٧	٥,٥	٥,٢٢	٣,٨٧
$[Ni(L)_{٢}Cl_{٢}]$	١٠٣٩	Brown	١١٥	١	٥,٥	٤,٦٥	٢,٨٩
$[Zn(L)_{٢}Cl_{٢}]$	١٠٤٦	Brown	١٠٠ de	١,٩	٦,٢	٥,٧٨	٠
$[Cd(L)_{٢}Cl_{٢}]$	١٠٩٣	Deep orang	١٠٥ De	٢,٨	١٠,٢	١٠	٠
$[Hg(L)_{٢}Cl_{٢}]$	١١٨١	Deep-orang	٩٥ De	٢	١٦,٩	١٤,٤١	٠

De = decompose, Calc. = Calculated, M.w = Molecular wight, M.p= Melting point.

Results and Discussion

Metal: ligand ratio

The (metal: ligand) ratios of complexes were determined by molar ratio method at fixed concentration and pH at wavelengths of maximum absorption. The results are given in Table (٣), at optimal conc.= ٤x١٠$^{-٤}$ for Cu(II), Co(II), Ni(II), Cd(II) and Hg (II) at optimal conc. =٠,٥x١٠$^{-٤}$ M with the ligand (Schiff-azo) the ligand was found to form (٢:١) chelates with all metal ions, these results are in agreement with the values reported for some aryl azo imidazole complexes [٢٠,٢١].

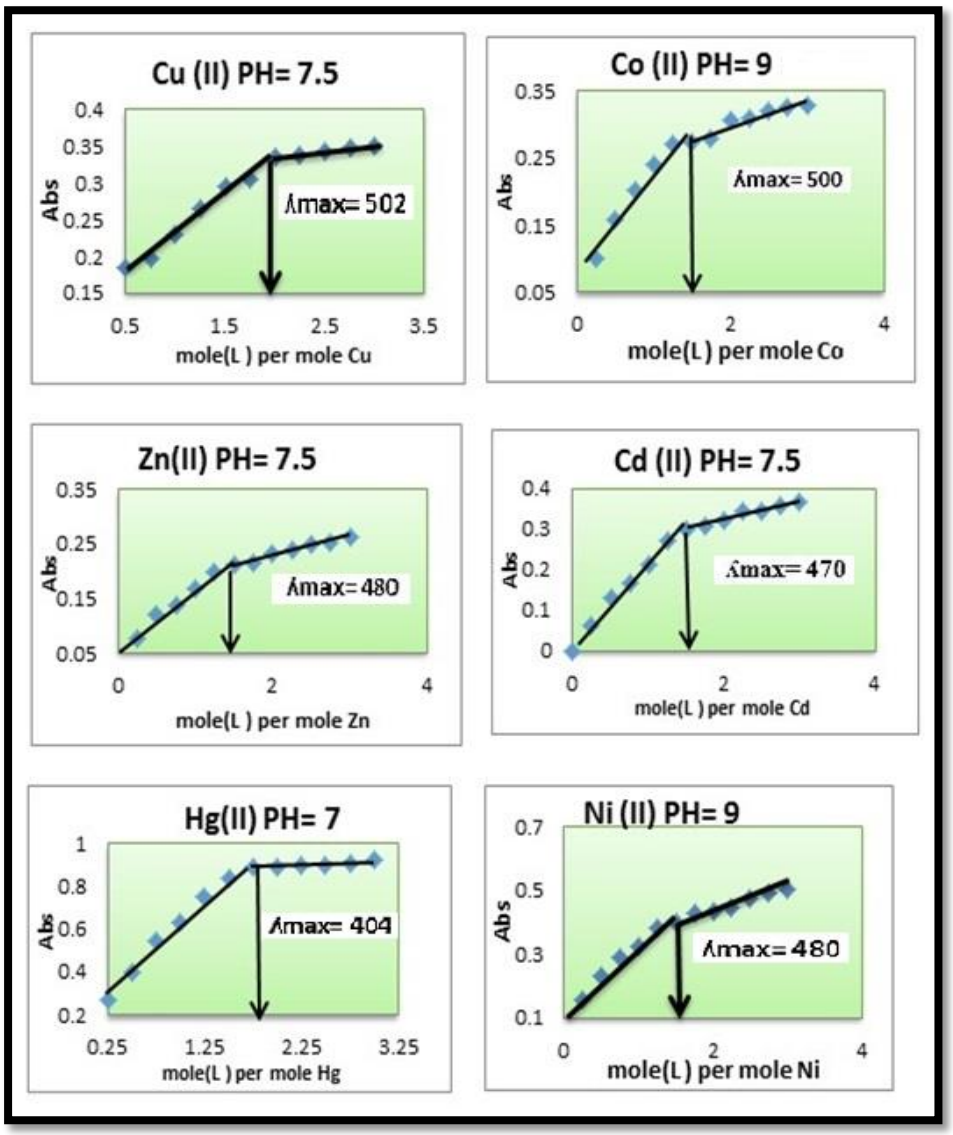


Fig. (١) The molar ratio (M:L) of metal ion Cu (II)) , Co(II) ,Zn(II), Ni(II) , Cd(II) and Hg (II) with (Schiff azo) ligand.

Absorption Spectra

The absorption spectra of ligand (Schiff-azo) and its complexes are studied. The wavelength for the maximum absorption (λ max) of the ligand was found at (٣٧٨nm). The spectra of metal complexes were recorded within wavelength range (٤٠٤– ٥٠٢) nm. The absorption maxima (λ max) of each complex shown in Table (٣).

Table (٣)
The optimal pH values, optimal molar concentration and wavelength (λ max) metal ions.

Metal Ions	*Optimal pH*	*Optimal molar conc. X ١٠$^{-٤}$ M*	*Optimal wave length (λmax)nm*
Cu(II)	٧,٥	٤	٥٠٢
Co(II)	٩	٤	٥٠٠
Ni(II)	٩	٤	٤٨٠
Zn(II),	٧,٥	٤	٤٨٠
Cd(II)	٧,٥	٤	٤٧٠
Hg(II)	٧	٠,٥	٤٠٤

IR-SPECTRA

The spectrum of the Schiff base shows two absorption bands at (٣٤٣١and ٣٣٥٤ cm^{-1}) were assigned to asymmetric and symmetric vibrations of (NH_2) group [٢٢]. The spectrum also revealed a new sharp band at (١٦٤١ cm^{-1}) related to *ν*(C=N) in Schiff compound [٢٣], while the absorption band at (٣١٨٢ cm^{-1}) assignedto (N–H) imidazole [٢٤,٢٥]. while the bands observed after the cordenation at (٣٢١٥-٣٢٤٤) cm^{-1} this shifftingrelated to the dameg of intra hydrogen bonding for N^3 of hetrocyclic ring.The band at (١٥١٨) cm^{-1} due to ν(C═N) of the N_2 imidazole nitrogen[٢٦], while the bands observed at (١٤٩٤ cm^{-1}), (١٢٥٥ cm^{-1}) and (١١٦٥ cm^{-1}) assigned to (N=N)[٢٧], (C–N=N–C) an (C=N-N=C), respectively. The spectra of complexes show achang in frequency, shape and intensity band related to (N=N) group confirm its participation in coordination with metal ions. The complexes spectra exhibited new weak bands at frequency range (٤٤٠-٥٩٠ cm^{-1}) assigned to stretching frequency of (M-N) bond [٢٨-٣٠].

Table (٤)
Characteristic IR absorption bands of the ligand and it'scomplexes in cm^{-1}.

	Ligand	*Cu(II)*	*Co(II)*	*Zn(II)*	*Ni(II)*	*Cd(II)*	*Hg(II)*
υ(N-H)	٣١٨٢	٣٢١٥	٣٢١٥	٣٢١٧	٣٢١٥	٣٢١٥	٣٢٤٤
υ(C-H)Ar	٣٠٦١	٣٠٥٧	٣٠٥٩	٣٠٥٣	٣٠٥٧	٣٠٥٩	٣٠٥٩
υ(C-H)Aliph	٢٩٩١-(٢٨٢٠)	٢٩١٤ ٢٨٦٦	٢٨٥٤ ٢٨٢١	٢٨٥٤ ٢٨٢١	٢٨٥٢ ٢٨٢٠	٢٨٥٤ ٢٨٢١	٢٨٥٤ ٢٨٢١
υ (C=N)Schiff	١٦٦٠	١٦٦٤	١٦٦٢	١٦٦٢	١٦٦٢	١٦٦٢	١٦٧٦
υ(C=C)Ar	١٥٩٩	١٥٩٥	١٥٩٩	١٥٩٩	١٥٩٩	١٥٩٩	١٥٩٧
υ (C=N)Imi.	١٥١٨	١٥٢٣	١٥٢٥	١٥٢٧	١٥٢٧	١٥٢٥	١٥٠٤
υ(N=N)	١٤٩٤	١٤٩١	١٤٩٢	١٤٩٢	١٤٩٢	١٤٩٢	---------
υ(C–N=N-C) and υ(C=N-N=C)	١٢٥٥ ١١٦٥	١٢٤٩ ١٣٥٧	١٢٤٩ ١٣٥٧	١٢٧٤ ١٢٥١ ١١٧٠	١٢٧٤ ١٢٥١ ١١٧٠	١٢٧٤ ١٢٤٩ ١١٦٨	١٢٦٧ ١١٨٤ ١١٥٣
Ph-Imi.	٧٦١	٧٦٧	٧٦١	٧٦٥	٧٦١	٧٦٣	٧٦٧
υ(M-N)	.....	٥٩٠ ٥١٥	٥٠١ ٤٤٠	٤٦٨ ٤٤٩	٥٠١ ٤٥٧	٥٠١ ٤٦٦	٥٩٠ ٤٣٨

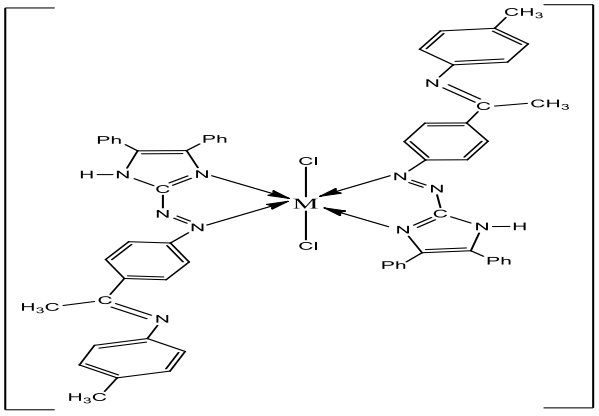
C.H.N Measurement of Schiff azo ligand

Table (٥)
Elemental analysis (C.H.N) of Schiff azo– ligand.

Compound	*C %*		*H %*		*N %*		*M.Wt*
	Calc.	Found	Calc.	Found	Calc.	Found	
$C_{٣٠}H_{٢٠}N_{٥}$	٧٩,١٢	٧٨,٥١	٥,٤٩	٥,٧٠	١٥,٣٨	١٤,١١	٤٥٥

Molecular structures of proposed complexes

Schiff azo ligand behaves as a neutral bidentate (N,N′) ligand forming chelates with (١: ٢) (metal: ligand) stoichiometry and an octahedral geometry for The complexes, Fig.(٢).



M=Cu (II), Co(II), Zn(II), Ni(II), Cd(II), Hg (II)

Fig. (٢) Complexes with Schiff azo ligand.

Conclusion

In this paper, we have explored the synthesis and coordination chemistry of Schiff azoligand (E)-N-(١-(٤-((E)-(٤,٥-diphenyl-١H-imidazol-٢-yl) diazenyl)phenyl) ethylidene)-٤-methylaniline complexes obtained from the reaction of the bidentate ligand with Cu(II), Hg(II), Zn(II) Ni(II) Co(II) Cd(II), transition metals. The mode of bonding and overall structure of the complexes was determined through physic-chemical and spectroscopic methods. Complexes formation study via molar ratio has been investigated and results were consisted to those found in the solid complexes with the ratio [M:L] as [١:٢]. Depending upon all results was proposed an octahedral geometry for the complexes.

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الخلاصة

تضمن البحث تحضير معقدات مخلبية لكل من النحاس(II) والزئبق (II) والزنك (II) والنيكل (II) والكوبلت (II) والكادميوم (II) مع ليكاند شف–ازو غير متجانس الحلقه جديد ٤– مثيل–[N-١(٤،٥–ثنائي فنيل –٢– اميدازول–٢– يل) ديازينيل (فنيل اثيليدين) انيلين). شخص الليكاند المحضر بواسطة الاشعه تحت الحمراء، الاطياف الالكترونية والتحليل الدقيق للعناصر (C.H.N). اما معقدات هذا الليكاند فقد شخصت بنفس التقنيات المستخدمة لتشخيص الليكاند يضاف لها دراسة الامتصاص الذري اللهبي، التوصيلية المولارية و الخواص المغناطيسية اذ بينت نتائج الدراسة سلوك الليكاند للمعقدات متعادل الشحنه ثنائي المخلب (N,N). اذ يرتبط مع جميع الايونات الفلزية بنسبه (١:٢) (ليكاند:فلز) وبالأعتماد على النتائج المستحصلة تم اقتراح شكل ثماني السطوح للمعقدات التي تم تحضيرها.