

Synthesis, Spectroscopic Studies and Biologically Active of Uracil and Mixed Ligand with Aluminum (III)

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Abstract: New complexes of Al(III) such as $[Al(Ura)(Phen)(OH_2)Cl]Cl \cdot 2H_2O$, $[Al(Ura)_2(OA)(OH_2)Cl] \cdot H_2O$ and $[Al(Ura)_3Cl_3] \cdot H_2O$ type, where (Ura)=Uracil, (Phen)= 1,10-Phenanthroline monohydrate and (OA)= Oxalic acid dihydrate, were prepared. The elemental microanalysis, FT-IR, electronic spectra, and magnetic susceptibility as well as the conductivity measurements are characterized. For isolated three complexes for six coordinated of Al(III) are proposed with molecular formulas that depend on the nature of (Ura), (Phen) and (OA) present. The suggested molecular structure into all complexes for aluminum ion is octahedral geometries. The antibacterial efficacy was examined from metal salt ($AlCl_3$), ligands and metal complexes into the pathogenic bacteria efficacy has been studied.

Keywords: Mixed ligand complexes, Spectral studies, Biological activities.

1. Introduction

Uracil (Ura) according to Figure (1), is naturally happening pyrimidine derivative found at nucleic acids [1]. It is a pyrimidine base whom consists from four different binding sites, and belongs into a group to the most significant pyrimidines that played a essential role at the structure and function from enzymes and drugs. (Ura) was utilized for synthesis antibacterial [2], and antitumor agents [3]. (Ura) avails as allosteric organizer and coenzyme to reaction at the human body and at plants [4]. (Ura) structure is shown in Figure (1), It can be used to identify microbial contamination of tomatoes [5].

Mixed ligands compounds for metal ions (II) and (III) are well recognized into play a significant role at biological system [6-8]. Over the years, number about Al(III)-compounds for the association of carboxylic acid ligands relevant biologically appeared from synthetic studies implemented at aqueous solutions. These different types of Al(III) are discussed here in detach, for focus in solid state property and their solution. As a result, nuclear complexes

ate changing (contain a chang number for Al(III) ions) are analysis with chemical charecteristics as well structures are in the solution immediately contrast to the charecteristics as well structures in the solid state. In detail reckoning to the structural speciation from Al(III) for carboxylic consisting from ligands is visualized in the step set to moreover the depth of perusal with every effort toxicity types may be susceptible for displaying at vivo[9]. (Phen) ligand and its derivatives have outputed in many structures and applications in interesting biological efficiencies [10-11]. Metal compunds including diimine ligands like bipyridine as well (Phen) have obtained importance significance because to the versatile roles like constructing blocks to the synthesis from metallo-dendrimers as well like molecular scaffolds to the supramolecular synthesis and in the analytical chemistry, catalysis, electrical alchemy, open metathesis polymerization ring and biochemistry [12-13].

In this paper, the synthesis, spectral physical properties of Al(III)- complexes of (Ura) ligand and mixed ligands (Ura)(Phen) and (Ura)(OA) the current work deals with antibacterial study for these notified compounds.

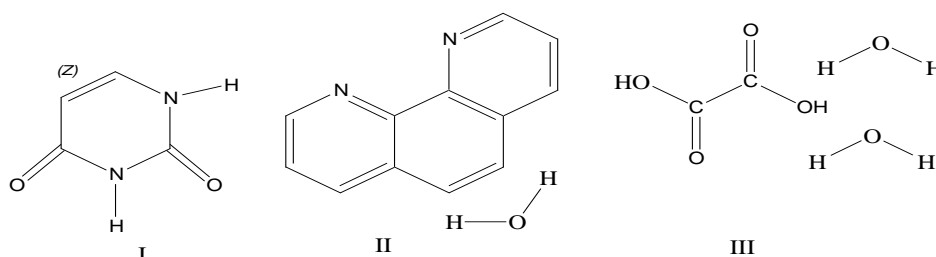


Figure 1: (I) Uracil, (II) 1,10-Phenanthroline hydrate and (III) Oxalic acid dehydrate

2. Experimental

Materials and methods

Uracil, 1,10- phenanthroline monohydrate and oxalic acid dihydrate were purchased from (Merck and B.D.H.), metals

chloride and solvents from (B.D.H., Riedel and Merck). The reagents were used without further purification.

Measure the values of molar conductance (Λ_m) at DMF solution for compounds were measured at 25°C in 10⁻³M solution at the samples utilizing PW9527 Digital

Conductivity Meter (Philips). Infrared (FT.IR) spectra were registered at 4000-400 cm^{-1} range into ligands and metal complexes at a Shimadzu IR-470 Spectrophotometer employing KBr and measured at 4000-200 cm^{-1} range for by FT.IR-8300 Shimadzu using CsI pellets. The electric spectra for complexes at DMF solution (10^{-3}) were observed in 25 $^{\circ}\text{C}$ at the 300-700 nm range with Shimadzu-U.V-160. Ultra Violet-Visible- Spectrophotometer for 1.000 ± 0.001 cm corresponded quartz cell. C, H and N content for complexes were determined through the micro analytical unit of Eurverctor EA 3000A Elemental Analyzer. Whilst the contents of metal for compounds have been specified through Atomic Absorption (A.A) Technique / Flame Emission Spectrophotometer employing AA - 680 Shimadzu. Magnetic moments

Were measured with a magnetic susceptibility balance (Sherwood balance magnetic susceptibility model MSB-MK). Melting points have been determined by employing Stuart Scientific melting point apparatus.

Preparation of ligands

a) Uracil solution: Dissolve [0.112gm, 1mmol] at 5ml warm water and added [0.04gm, 1mmol] for sodium hydroxide.

b) 1,10- Phenanthroline hydrate solution: Dissolve [0.198gm, 1mmol] at 5ml methanol.

c) Sodium oxalate dihydrate solution: Dissolve for oxalic acid dihydrate [0.126gm, 1mmol] at 5ml warm water and added [0.04gm, 1mmol] for sodium hydroxide depending on the following Figure (2).

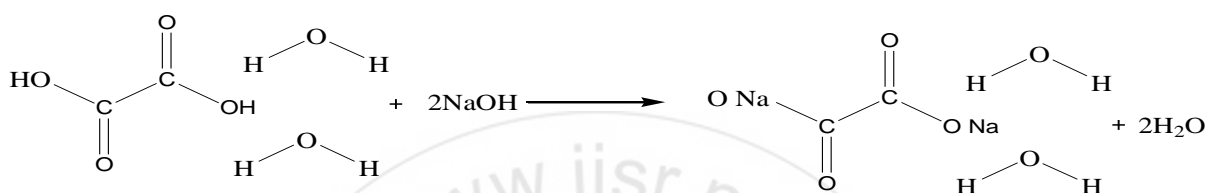


Figure 2: Sodium oxalate dihydrate

Preparation of aluminum compounds

The following generic method was adopted for synthesizing compounds:

a) Synthesis of $[\text{Al}(\text{Ura})(\text{Phen})(\text{OH}_2)\text{Cl}]\text{Cl}\cdot 2\text{H}_2\text{O}$ complex: The complex were prepared through the addition for (Phen) solution and (Ura) solution in the warm stirred water solution for respective Al(III) chloride of AlCl_3 [0.133gm, 1mmol] in the stoichiometric ratio metal : ligand (Al: Ura:Phe), (1:1:1). The mixtures were heated in the water bath into reflux about (12) hour at 70 $^{\circ}\text{C}$. The metal complex was crystalline precipitates observed. The precipitated complex then was filtered off and washed with ether followed by drying at room temperature and analyzed employing standard methods.

b) Synthesis of $[\text{Al}(\text{Ura})_2(\text{OA})(\text{OH}_2)\text{Cl}]\cdot \text{H}_2\text{O}$: The complex were prepared by the addition of the sodium oxalate solution and (Ura) solution to the warm stirred water solution for respective Al(III) chloride of AlCl_3 [0.133gm, 1mmol] to the stoichiometric ratio metal: ligand (M:Ura.:OA), (1:2:1). The mixtures were heated on a water bath to reflux about (24) hour for Al(III) complex at 70 $^{\circ}\text{C}$. The metal complex was crystalline precipitate noticed. Resulting precipitate was filtered, recrystallized of ether as well dried in room temperature.

c) Synthesis of $[\text{Al}(\text{Ura})_3\text{Cl}_3]\cdot \text{H}_2\text{O}$: The complex was prepared by the addition for (Ura) solution to the warm stirred water solution of the respective Al(III) chloride of AlCl_3 [0.133gm, 1mmol], in the stoichiometric ratio metal : ligand (M:Ura), (1:3). The mixtures were heated on a water bath to reflux for (30) hour with complex at 70 $^{\circ}\text{C}$. The product was collected through filtrations, and washed several times for the ether and dried in room temperature.

Study of biological efficacy :

The antibacterial efficacy to the metal salt (AlCl_3), ligands and metal compounds were checked by agar well- diffusion method. The antibacterial efficacy to the metal salt, ligands as well the corresponding complexes were assayed simultaneously against Gram positive bacteria (G+ve), (*Staphylococcus* and *Bacillus*) and Gram negative bacteria (G-ve), (*E. Coli* and *Pseudomonas*) activity by nutrient agar well - diffusion method. The solvent used for making test samples and standard was DMSO as well pattern of 1 to 200 $\mu\text{g}/\text{ml}$ have been utilized antibacterial activity concerning every complex were estimated through the well-diffusion manner. 1cm^3 about a 24h broth culture including 10^6 CFU/ cm^3 was putted at sterilized Petri-dishes. Molten nutritive agar (15cm^3) preserved in ca. 45 $^{\circ}\text{C}$ was thereafter poured into the Petri-dishes as well allowed into settle. Thereafter punctures about 6 mm diameters were punched accurately employing a sterilized cork auger as well these were fully filled for the check solutions. The dishes were brood at 24h in 37 $^{\circ}\text{C}$.

3. Results and Discussion

Analytical data and several physical features leads to Al-(Ura)(Phe), Al-(Ura)(OA) and Al-(Ura) compounds are illustrated in Table(1). Al(III)- compounds are colourless and all complexes soluble at DMF as well DMSO, however the complex of Al-(Ura)(Phe) soluble in water and slight soluble in CH_3Cl , CCl_4 , CH_3OH , $\text{C}_2\text{H}_5\text{OH}$ and C_6H_6 , while non soluble in ether. While, the complexes Al-(Ura)(OA) and Al-(Ura) are non soluble in most organic solvents. Molar conductance values to the compounds at DMF in 10^{-3} M condensation and type of electrolyte for the Al-(Ura)(Phen) complex, but the types of non-electrolyte for the Al-(Ura)(OA) and Al-(Ura) complexes [14] and each complex could be determined in the Table (1).

I. Infra Red Spectra:

Characteristics of frequencies to the ligands and compounds to the Al(III) ion have FT-IR spectra given Table (2) and for the three complexes. The broad band appearing in the (3437, 3425 and 3406) cm^{-1} in the Al-(Ura)(Phen) and Al-(Ura)(OA) and Al(Ura) areas on whole the compounds are assigned into OH stretching vibration to hydrated and water-coordinated molecules [15] and to the hydrogen bonds of the type (N-H---O) [16]. Moreover, The occurrence to a strong band at the rang 848 and 856 cm^{-1} at the FT-IR spectrum to the compounds Al-(Ura)(Phe) and Al-(Ura)(OA) regions, that is assignable into the OH rocking vibration, proves the existence of water-coordinated [17]. The absorption frequencies of the Al(III)- complexes with (Ura) were compared to that of the free (Ura) in the region between (1800 – 1300) cm^{-1} where the CO and NH frequency of (Ura) is located. The bending vibrations of $\delta\text{NH}_{(1)}$ and $\delta\text{NH}_{(3)}$ in 1508 cm^{-1} and 1415 cm^{-1} of free (Ura) and in the complexes remain almost constant both in intensity and position in 1508 cm^{-1} and (1415 and 1419) cm^{-1} in the Al-(Ura)(OA) and Al-(Ura) complexes, while, in the complex Al-(Ura)(Phen) appeared stay stationary package of $\delta\text{NH}_{(3)}$ in 1419 cm^{-1} with the disappearance of the package of $\delta\text{NH}_{(1)}$, $\nu(\text{C}_2=\text{O})$ band at 1735 cm^{-1} and 1716 cm^{-1} at (Ura), but the complexes of Al-(Ura)(Phen) and Al-(Ura)(OA) at (1732-1735) cm^{-1} and (1715-1716) cm^{-1} , while the Al-(Ura) complex, the package at 1665 cm^{-1} with the disappearance of the second package in the last complex. $\nu(\text{C}_4=\text{O})$ band in 1670 cm^{-1} and 1643 cm^{-1} in free (Ura), notice the emergence of a single package in the band position of the 4-keto group of Al-(Ura)(Phen) and Al-(Ura)(OA) complexes at 1662 cm^{-1} and 1666 cm^{-1} , respectively with the second package disappearance. While, the Al-(Ura) complex two packages appear at 1670 cm^{-1} and 1643 cm^{-1} without any change in the package for this complex. Therefore, it will vary Al(III) association with (Ura) in each of the three complexes. In the complex Al-(Ura)(Phen), be by package $\nu(\text{C}_4=\text{O})$ and $\delta\text{NH}_{(1)}$, which the base posses the O- and N-donor ligand in the last complex [18] according to the following Figure (3). in the solution has been deprotonated according into the subsequent reaction Figure (4).

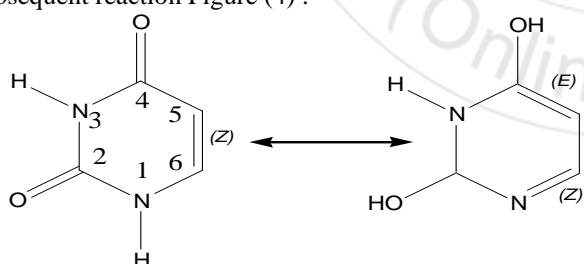


Figure 3: Tautomeric forms of uracil

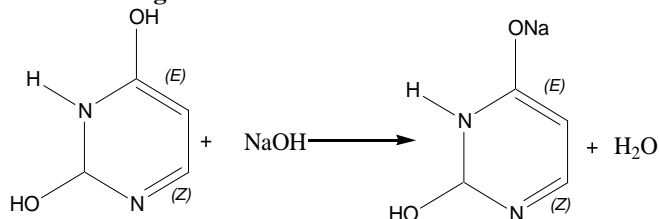


Figure 4: Sodium uracilate

While the complex Al-(Ura)(OA), be by package $\nu(\text{C}_4=\text{O})$ with Al(III). But, Al-(Ura) complex, it will link through the package $\nu(\text{C}_2=\text{O})$ with the Al(III) ion. The presence of

characteristic bands to the Al-O with (Ura) ligand in the Table (2) to Al-(Ura)(OA) and Al-(Ura) complexes, which that (Ura) is monodentate ligand and Al-(Ura)(Phen), which that (Ura) is bidentate ligand. $\nu(\text{C}=\text{N})$ and (C=C) mode noticed in 1558 cm^{-1} into the spectrum for free (Phen) ligand is found into be shifted to the lower wave number about 1523 cm^{-1} in the spectra of Al-(Ura)(Phen) complex. Negative shift at that vibrational mode at complexation appears the coordination during nitrogen donor for (Phen) with this complex, which is bidentate ligand [19]. Moreover, FT-IR spectrum display strong proof supporting the participation for carboxylate group (COO-) into coordination, at compare for free ligand (OA), a band take notice in 1697 cm^{-1} due to $\nu(\text{COO})_{\text{asy}}$ and the band at 1438 cm^{-1} in the spectrum of (OA), which was assigned to the $\nu(\text{COO})_{\text{sy}}$. On (OA) spectrum, it has been shifted into lower wave number at 1666 cm^{-1} and 1392 cm^{-1} in the spectra of mixed ligand complex with Al-(Ura)(OA) complex, which represents coordination to the carboxylic group for metal ions during the oxygen atom, that is bidentate ligand [20]. These observations were further indicated the shift at 435 cm^{-1} and 482 cm^{-1} by the appearance of U(M-O) for each of the three complexes [21]. The U(M-Cl) bands are tentatively assigned in the lower region in the (312-220) cm^{-1} for the three complexes [22].

II. UV-Visible Spectroscopy :

Electronic spectra were recorded of the ligands and compounds at DMF as indicate in Table (3) each of complexes. The Ultraviolet-Visible spectrum to the free ligand (Ura) displayed two absorption bands at 270 nm (37037 cm^{-1}) and 320 nm (31250 cm^{-1}) referred into ($\pi-\pi^*$) and ($n-\pi^*$) transition of carbonyl group, however the later to the conforming transition of N=C=O chromophore[23] as well the spectra to the free ligand (Phen) display absorption peak in 270 nm (37037 cm^{-1}), 301 nm (33222.6 cm^{-1}) and 324 nm (30864 cm^{-1}), whom indicated into ($\pi-\pi^*$), ($n-\pi^*$) and ($n-\pi^*$) transition Consecutively, which transitions of pyridine rings [23], while the (OA) ligand shows peak in 264nm (37878 cm^{-1}) was referred into ($\pi-\pi^*$) electronic transition [24]. Electronic spectra of Al(III) complexes gave absorption peaks of the Al(Ura)(Phen) complex at 325nm(30769 cm^{-1}) in the UV-Vis region caused by LMCT, while Al(Ura)(OA) at 268nm (37313 cm^{-1}) and 349nm (28653 cm^{-1}) related to L.F. and LMCT transition. But the complex Al(Ura) the packets appeared at 268 nm (37313 cm^{-1}), 340nm (29411 cm^{-1}) and 388nm (25773 cm^{-1}), these packages are due to L.F. and LMCT. Absence of absorption peaks at the visible area appeared no (d-d) electronic transition occurred in the aluminum complexes. The structure in Figure(5) is proposed for the Al(Ura)(Phen), Al-(Ura)(OA) and Al-(Ura) complexes, respectively.

III. Antimicrobial study

The results obtained for antibacterial test models studies by agar well-diffusion bioassay revealed biological activity of the ligands and Al(III)-complexes after 24h in Table (4) and Figure(6).

In case of (Phen) the antibacterial activity in *E.Coli*, *Pseudomonas* and *Staphylococcus* is higher activity as compared to (Ura) and (OA) ligands, while Al-(Ura)(Phen)

complex, is higher activity with All kinds of bacteria as compared to Al-(Ura)(OA) and Al-(Ura) complexes.

Table 1: The Physical Properties of the Complexes

Com.	M _{wt}	Color	Mp. °C	Element analysis % Calcu. (Found)				Molar conductivity In DMSO
				%C	%H	%N	%M	
(Ura)	112.09	White	335					
(Phen)	198.22	White	97					
(OA)	126.07	White	(101-102)					
[Al(Ura)(Phen)(OH ₂)Cl]Cl.2H ₂ O	443.98	White	>250d	40.08 (42.09)	3.78 (3.24)	11.69 (13.06)	5.63 (4.91)	60.20
[Al(Ura) ₂ (OA) (OH ₂)Cl].H ₂ O	410.48	White	>250d	29.26 (30.31)	1.21 (3.44)	13.65 (14.32)	6.73 (5.69)	11.90
[Al(Ura) ₃ Cl ₃].H ₂ O	487.48	White	>250d	29.57 (29.11)	2.89 (2.63)	17.24 (17.07)	5.53 (5.34)	20.22

d= decomposition temperature

Table 2: FT-IR Spectral Data of the Ligands and their Complexes

Com.	$\nu(\text{OH})_{\text{H}_2\text{O}}$	$\nu(\text{NH})$	$\nu(\text{CH})$	$\nu(\text{C}_{(2)=\text{O}})$	$\nu(\text{C}_{(4)=\text{O}})$	$\delta\text{NH}_{(1)}$	$\delta\text{NH}_{(3)}$	$\nu(\text{C}=\text{N})$ + $\nu(\text{C}=\text{C})$	$\nu_{\text{s}}(\text{COO})$ + $\nu_{\text{as}}(\text{COO})$	$\nu(\text{M}-\text{O})$	ν (M-N)
(Ura)	-	3109br.	3039w 2985w	1735w 1716w	1670w 1643w	1508s	1415s	-	-	-	-
(Phen)	3410br.	-	2993w	-	-	-	-	1558s	-	-	-
(OA)	3410br.	-	-	-	-	-	-	-	1697s 1438sho	-	-
[Al(Ura)(Phen)(OH ₂)Cl]Cl.2H ₂ O	3437br.	3109br.	3043w 2935w	1735w 1716s	1662w	-	1419s	1523s	-	3435w 482w	543m
[Al(Ura) ₂ (OA) (OH ₂)Cl].H ₂ O	3425br.	3109br.	2985w 2935w	1732w 1716w	1666br	1508s	1419s	-	1666w 1366sho	432w 482w	-
[Al(Ura) ₃ Cl ₃].H ₂ O	3406br.	3105br.	3111w 2935w	1665w	1670w 1643w	1508s	1415s	-	-	435m	-

s = sharp, br. = broad, w = weak sho=shoulder

Table 3: Electronic Spectra to the Studied Compounds and three Ligands

Com.	$\lambda(\text{nm})$	$\nu(\text{cm}^{-1})$ Wave Number	Assignment of the Transition	Suggested Structure
(Ura)	270	37037.0	$\pi \rightarrow \pi^*$	-
	320	31250.0	$n \rightarrow \pi^*$	
(Phen)	270	37037.0	$\pi \rightarrow \pi^*$	-
	301	33222.5	$n \rightarrow \pi^*$	
	324	30864.0	$n \rightarrow \pi^*$	
(OA)	264	37878.8	$\pi \rightarrow \pi^*$	-
[Al(Ura)(Phen)(OH ₂)Cl]Cl.2H ₂ O	325	30769	L.F.	Oh.
	419	23866	LMCT	
[Al(Ura) ₂ (OA) (OH ₂)Cl].H ₂ O	268	37313	L.F.	Oh.
	349	28653	LMCT	
[Al(Ura) ₃ Cl ₃].H ₂ O	268	37313	L.F.	Oh.
	340	29411	LMCT	
	388	25773	LMCT	

Table 4: Showed Inhibition Circle Dimeter at Mill miter with the Bacteria after 24 h

Compounds	<i>Bacillus</i> (G+ev)	<i>Staphylococcus</i> (G+ev)	<i>Pseudomonas</i> (G-ev)	<i>E. Coli</i> (G-ev)
Control (DMSO)	5	5	6	5
Uracil	11	14	21	20
1,10-Phen hydrate	-	24	22	27
Ocalic acid dihydrate	12	-	15	16
[Al(Ura)(Phen)OH ₂ .Cl]Cl.H ₂ O	19	15	18	23
[Al(Ura) ₂ (Ox)OH ₂ Cl].H ₂ O	17	-	15	12
[Al(Ura) ₃ Cl ₃].H ₂ O	-	-	14	14

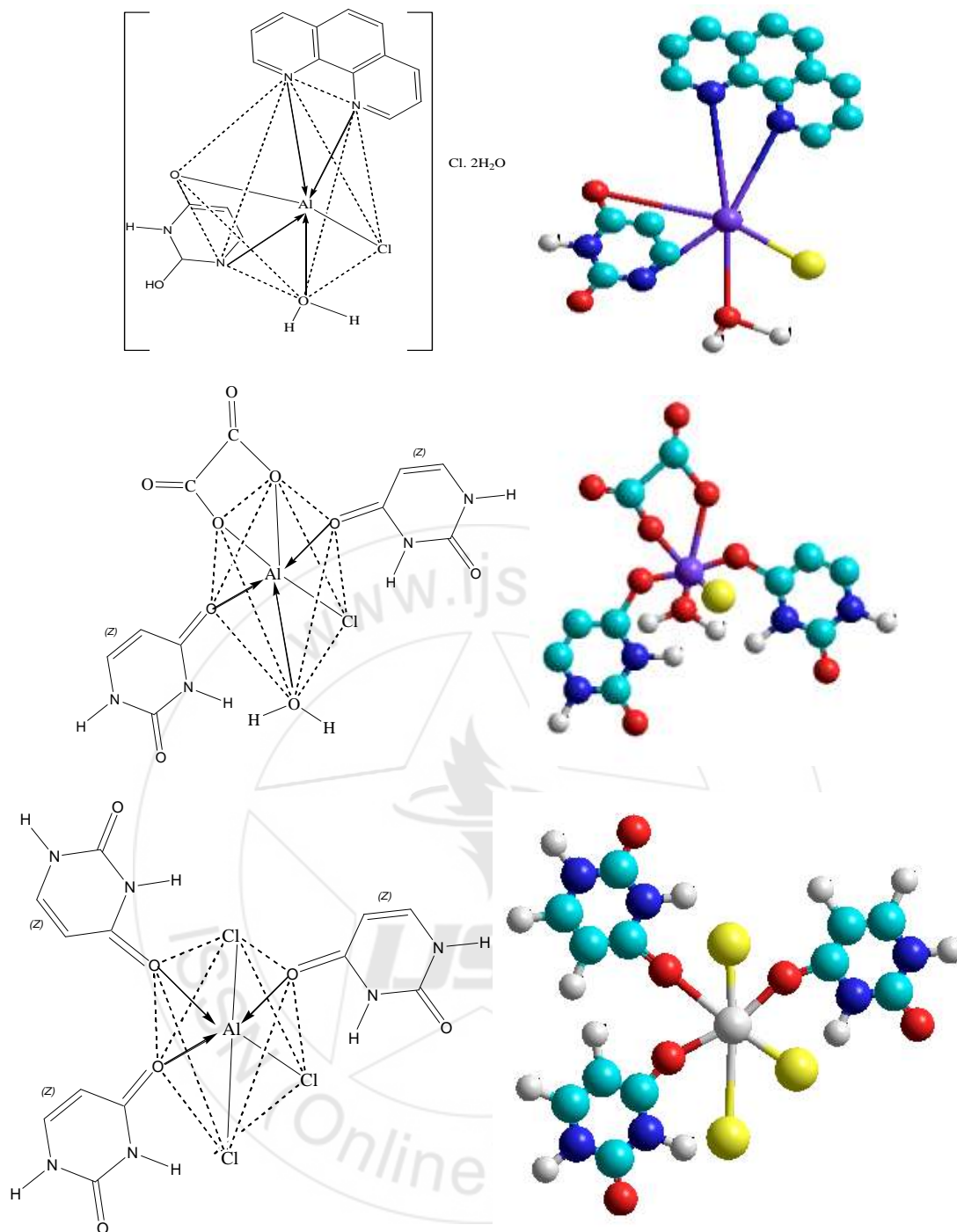


Figure 5: Proposed Structure and 3D-geometric structure to the Complex $[Al(Ura)(Phe)(OH_2)Cl]Cl \cdot 2H_2O$, $[Al(Ura)(OA)(OH_2)Cl] \cdot H_2O$ and $[Al(Ura)_3Cl_3]H_2O$, respectively

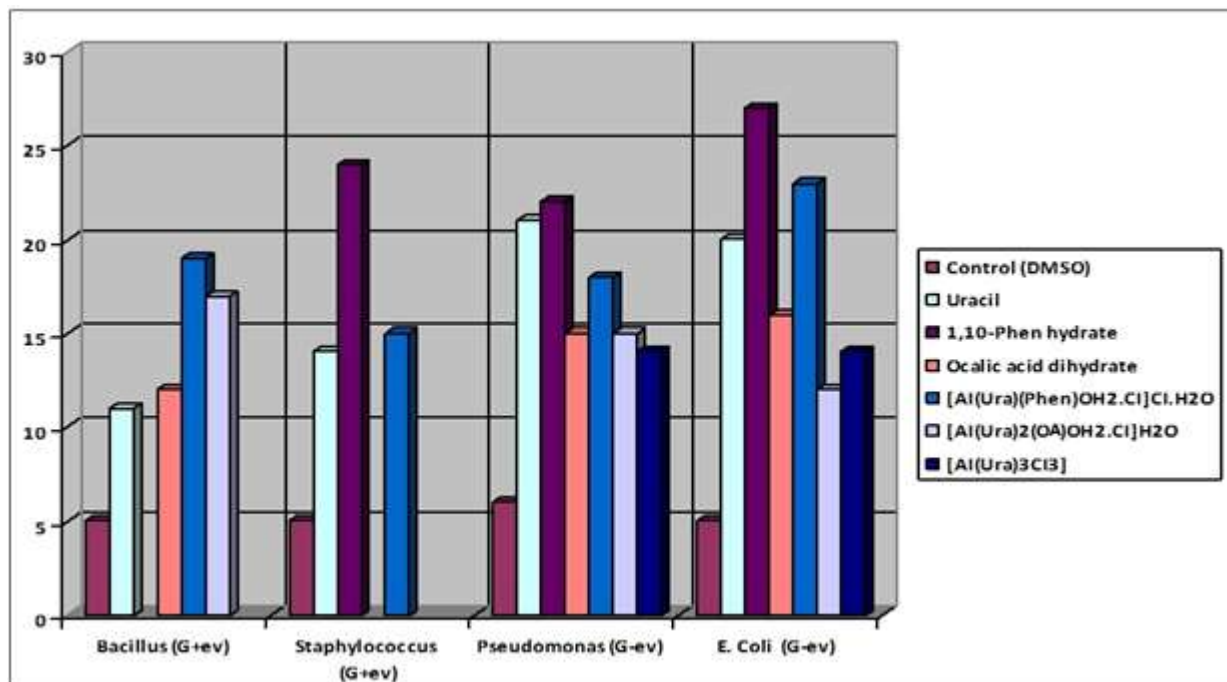


Figure 6: Antibacterial Efficacy Appears to the metal salts and the compounds Show Areas of Inhibition versus Pathogenic Bacteria (*E.coli*, *Pseudomonas*, *Streptococcus* and *Bacillus*) and the Antifungal Activity such as *Candida*(Yeast)

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