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SYNTHESIS AND CHARACTERIZATION OF SOME METAL COMPLEXES WITH SOME FURANOSE RINGS AND STUDY THEIR BIOLOGICAL ACTIVITY

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Abstract:

1,2:5,6-Di-O-isopropylidene (2) as starting material have been synthesized by the reaction of D-glucose with dry acetone in presence of $Zncl_2/H_3Po_3$.3-ullos derived (3) was prepared by oxidation of (2) with DMSO/Ac_2O. Hydrolysis of (2) and (3) by 60% Ac_2O gave the corresponding derivateivies 1,2-O- isopropytidene –α – D - glucofuranose (L_1) and 1,2-isoproplyene-α-D-ribohexafuranose-3-ulos(LΠ). Coordination chemistry of $L_1$and LΠ with Co(Π), Cu(Π), Ni(Π), Pd(Π) and Pt(IV) ions were studied. The two ligands were characterized depending upon CHN analyses, ΄H NMR and FTIR. Structure of metal complexes were proposed depending on atomic absorption, CHN, Thermal analyses (TG & DTG), FTIR spectra, uv-vis and chloride contents, magnetic moments (meft) as well as conductivity measurements. From the spectral measurements, monomer structures for the complexes were proposed. The newly metal complexes were subjected to in vitro testing agains pathogenic microorganisms.

Introduction

Glucose, $C_6H_{12}O_6$ also is a simple sugar (monosaccharide) and an important carbohydrate in biology. Cells use it as the primary source of energy[(1)] and a metabolic intermediate. Glucose is one of the main products of photosynthesis and starts cellular respiration[(2)].

Depending on conditions, three major solid forms of glucose can be crystallized from water solution: α-glucopyranose, β- glucopyranose, and β- glucopyranose hydrate[(3)]. The reasons why glucose, and not another mono saccharide such as fructose, is so widely used in organisms. One reason misht be that glucose has a lower tendency, relative to other hexose sugars, to react nonspecifically with the amino groups of proteins[(3,4)]. Glucose is a common medical analyte measured in blood samples, it is also used as an energy source in most organisms, from bacteria to humans[(5)]. In Industry, glucose is used as a precursor to make vitamin C in the Reichstein process, to make citric acid, gluconic acid, bio-ethanol, polylactic acid and sorbitol[(6)].

Experimental

[(a)]Materials, [(b)]instruments, and [(c)]methods.

All chemicals used were of reagent grade (supplied by either Merch or Fluka) and used as supplied. Melting point were determined using an open-ended capillary tube method, the purity of the synthesized compounds were chcked by TLC, FT-IR recorded on a perkin-Elmen (1310IR). Spectrophotomete and schimadzu corporation chart 200-91527IR spectrophotometer in a KBr and CsI disk. ΄H-NMR spectra were recorded at 300MHZ on a FT-NMR. Elemental analysis were obtained on a EA elemental analyses (Fison Ision Instrument). Electronic spectra for compounds in the (uv-visible) and near IR region (200-1100)nm΄m different solvents were recoded on schimadzu (uv-vis)-160 spectrophotometer. The metal contants of the complexes were determined by atomic absorptionte chnique using: varian-AA775 and Thermal analyses (TG & DTG) were performed under a static air atmosphere in a Metter TA 3000 thermo balance at a heating rate of $10^{\circ}c\ min^{-1}$. Electronic conductivity

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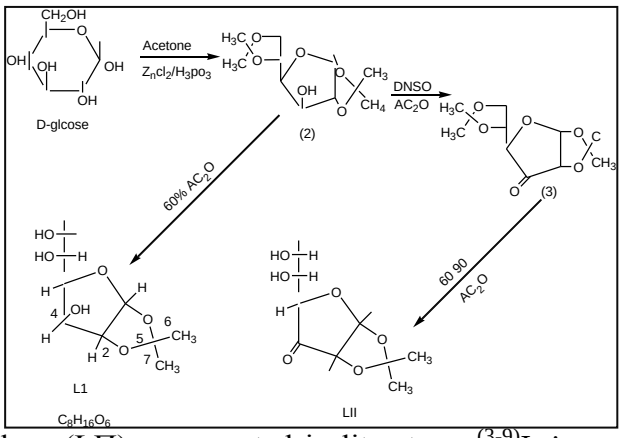
measurement for complexes in DMSO (10^{-3}M) at room temperature were carried out by using ELKta Lictfahiskeit conductivity meter (SIEMENS) and magnetic moments Meff B.M) for the prepared complexes in the solid state were calculated according to Faradays method by using Johnson mattey magnetic balance system.

A. preparation the ligandes and their metal complexes:-

1. Preparation of the ligands:-

The method used to prepare the 3-Deoxy-1,2-di-O-isopropylidene- α-D-ribohexafurannose (L_1) and 3-Deoxy-1,2,O-isopropylidene- α-D-ribohexafurannose-



3-ulose (LΠ), as reported in literature. [3-9]It is starting from D-glucose and acetone according to scheme (1), compound (2) or (3) (4.77 mmol and 6.58 mmol) respectively was dissolved in (60%) acetic acid (20ml) and stirred for 48h at room temperature. The solution was evaporated under reduced pressur and the resulting residue was coevaporated with toluene to give L_1 (yield 65%) Rf = 0.56,m.p.119-121^{0}c, IR(KBr)γcm^{-1}:3429(OH)$_1$3470-3300 (2OH), 2980 ($\upsilon_{as}CH_3$), 2875(υCH_3), 1468(δasCH3), 1189 (υc-o), ΄HNMR(DMSO) δ(ppm):82.47-2.42 (δ OH), δ3.56 (d,1-6), δ3.76 (q,΄H-5) and LΠ yield 66%as surup, R_f (1.760), IR(film)υcm^{-1} (1740)CO,3470-3300(OH). The physical properties of the product are shown in table (1), characterize by physical method and spectroscopy.

2. Preparation of complexes L_I (C_1-C_5)

To warm stirred solution of L1 (1.01 m mol) in ethanol (10ml) was added a solution of ($CoCL_2.6H_2O$, Ni $(NO_3)_2$. $2H_2O$, $CuCl_2.2H_2O$,$PdCL_2$(phCN) and $K_2ptCl_6.6H_2O$ (0.51 m mol) in water (20 ml). The resulting mixture was filtered to remove any insoluble material. The mixture was heated under refluxed for 30 min (C1, C2, C3), 1h (C4) and 3h (C5) and two days to achieve complete precipitation. the products were filtered, washed with ethanol, followed by ether and vacuum, dried[10].

3. preparation of metal complexes of (C6-C10).

To a solution of LΠ(1 m mol) in ethanol: water (1:1 V/V) was added an ethanolic solution of $Co(NO_3)_2.6H_2O$, $Ni(NO_3)_2.6H_2O$, $PdCl_2(phCN)_2$ and $K_2ptCl_6.6H_2O$, (C6-C10) respectively, with continuous stirring. The mixture was heated under refluxed for 1h (C6-C8) and 3h (C9 and C10) to achieve complete precipitation. The product was collected and washed with ethanol, ether and vacuum dried [9,10,11].

Results and Discussion:

Physical properties, Elemental analysis and metal and chloride contents of the prepared compounds are described in table (1). The results obtained are in good agreement with those calculated for the suggested formula which were supported by spectral studies, electrical conductivity measurements and magnetic moment

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determinations. The structures of some meted complexes were further confirmed by thermal analyses.

Table (1) physical properties and analytical data of L_I& L_{II} and their metal complexes

Formula (comp.no) color	Yield %	m-p (decomp) m.point	Elemental analyses% found (calculated)				
			C	H	N	M	CL
L1	65	119-120 0C	45.83 (46.15)	7.25 (7.69)	---	---	---
C1	56	232 deco	34.71 (34.05)	6.32 (6.03)	---	9.85 (10.45)	11.95 (12.57)
C2	60	177	33.78 (34.17)	6.63 (6.17)	3.02 (3.32)	6.03 (6.96)	---
C3	45	210	30.91 (31.76)	6.76 (6.28)	---	10.80 (10.51)	10.33 (11.72)
C4	42	290	23.13 (22.78)	4.43 (4.74)	---	24.63 (25.25)	16.07 (16.82)
C5		276	20.11 (20.30)	3.21 (3.72)	---	32.39 (33.01)	23.14 (23.99)
L11	660		46.11 (46.60)	6.95 (6.79)	---	---	---
C6	61	186	35.83 (36.79)	5.20 (5.79)	---	10.36 (10.04)	12.27 (12.07)
C7	57	205	32.11 (31.33)	5.91 (4.89)	4.10 (4.57)	10.04 (9.57)	---
C8	70	169	(33.01)	(5.50)	---	(10.	12.63 (12.05

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						93)	)
C9	53	230	35.44 (35.26)	5.29 (5.87)	---	(15.63)	10.89 (10.30)
C10	55	226	32.65 (32.16)	3.81 (3.35)	---	32.41 (32.68)	23.57 (23.75)

Infrared Spectra

Important characteristic stretching frequencies of the ligands and their metal complexes are described in table (2). The most important stretching modes exhibited by L_I and L_{II} are represented by cis hydroxyl groups and carbonyl group respectively. Bands related to stretching vibration of cis hydroxyl groups appeared at 3470-3300cm^{-1} in free Ligands L1 &L2[12], shifted to lower frequencies in complexes (C9,C10) as a resulted of coordination with metal ions through the lone pair electrons of oxygen atom while the spectra of complexes C9 and C10, Cpt_2 showed shift in (C=0) stretching vibration to lower frequencies in L_{II} spectra which indicate the linkage of C=0 from oxygen atom, the decreases have been attributed to metal dπ to ligand Pπ back bonding [12,13,14].

In both cases the ligands coordinated with metal ions through the oxygen atom. The spectra of complexes (C1, C3, C4, C5, C6, C8, C9, C10) exhibited additional bonds related to lattice and coordinate water vibration[14]. The spectra of the complexes (C2, C7) showed bands related to lattice ethanol appeared at 3538, 1595 and 1040 cm^{-1} attributed to γOH, δOH and γc.o respectively [15,16,17].
Nitrate ions exhibited vibrational modes related to montidentate in the complexes (C2, C7). Choride ion in the complexes (C1, C3-C6) and C8-C10) showed new bands at lower regions (305-280)cm^{-1} were assigned to (M-CL). The appearance of new bends at lower regions (550-533) cm^{-1} were assign need to (M-O) stretching vibrations[14,16], and are described in table (2).

Thermal Analysis

The TG and DTG analyses for the metal complexes of LI (C1, C2) and LΠ(C7, C8) at heating range 200-400 ^{o}C have been carried out under a static air atmosphere. The to curves of C1, C2, C7 and C8 (Fig1) show that the initial stages of thermal decomposition involve the loss of lattice solvent molecules followed by departure of uncoordinated ions and organic fragments [18,19]. There are some difference in the thermal behavior of Co(Π) and Ni(Π) or L1
with respect to Ni(Π) and Cu(Π) complexes of LΠ.

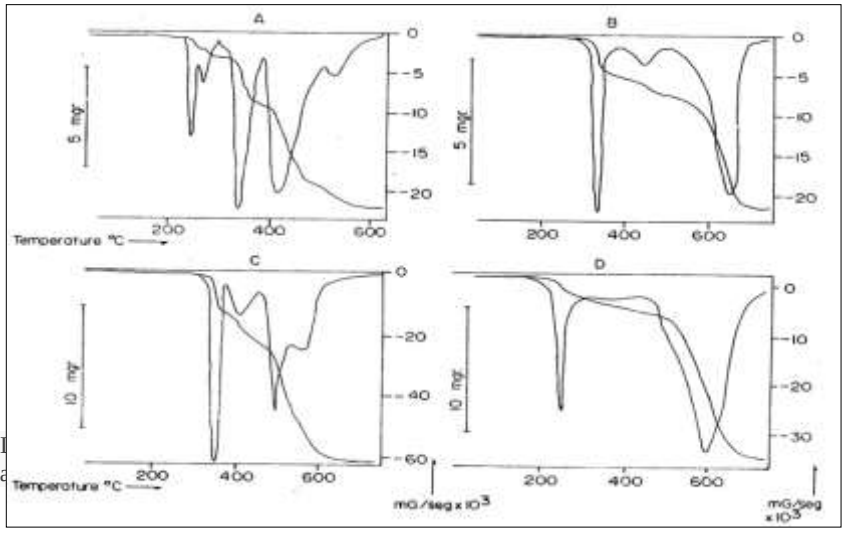

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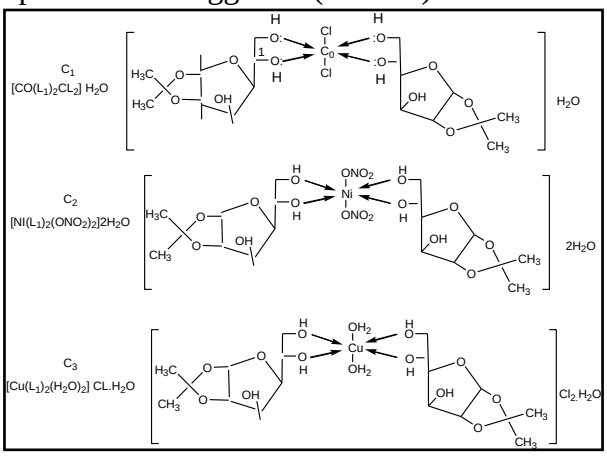
Fig (1):TG + DTG Carves of G (A), C_2 (B) C_7 (C) + c8(D) respectively.

Electronic spectra Magnatic moments and Conductivity measurement

There is no band exhibited in uv-vis regen for both LI &LII[8,10]. Complexation of L1 &LΠ with metal ions showed appearance of new bands in the visible and near i.r regions. These bands were attributed to M-L charge transfer and to ligond field transitions [20,21]. The band energy of Co(Π) and Ni(Π) complexes of both iligands were applied on Tanaba-Saugano diagrams of the two ions to obtain the values of the interelectronic repulsion parameter B^-, nepelauxetic ration (β), Dq/B^- as well as to caleulate the values of 10Dg[22]. Band assignments and values of magnetic moments referred to high spin Co(Π), Ni(Π), and Cu(II) octahedral complexes 0f LI.

The spectra of Cu(Π) complexes of both (L1 &LΠ) refer to Jahn-Teller distorted octahedral geometry[24,25]. The spectrum of diamagnetic Pd(II) complexes of LI and LII (C5&Ca respectively) showed three bands assigned to $^1A_{1g} \rightarrow {}^1A_{2g}(\upsilon_1)$, $^1A_{1g} \rightarrow {}^1B_{1g}(\upsilon_2)$ and $^1A_{1g} \rightarrow {}^1E_s$ of squar planar pd(II) complexes[22]. The spectrum of diamagnetic pt(IV) complexes of both(LI&LII), exhibited two absorption bands which were assigned to the forbidden transitions $^1A_{1g} \rightarrow {}^3T_{1g}$ and $^1A_{1g} \rightarrow {}^3T_{25}$ of octahedral pt(II) complexes[23,24].

Conductivity measurements of complexes in DMF (10^{-3}M) showed that they were electrolytes with ionic ratio of 1:2 for both Cu(Π)complexes (C3 and C6) while the complexes (C5, C6, C9, C10) were non electrolyte (25,26). From these observations, together with elemental analysis, FTIR and thermal analyses, the stereo chemical structures of the complexes were suggested (schem2).



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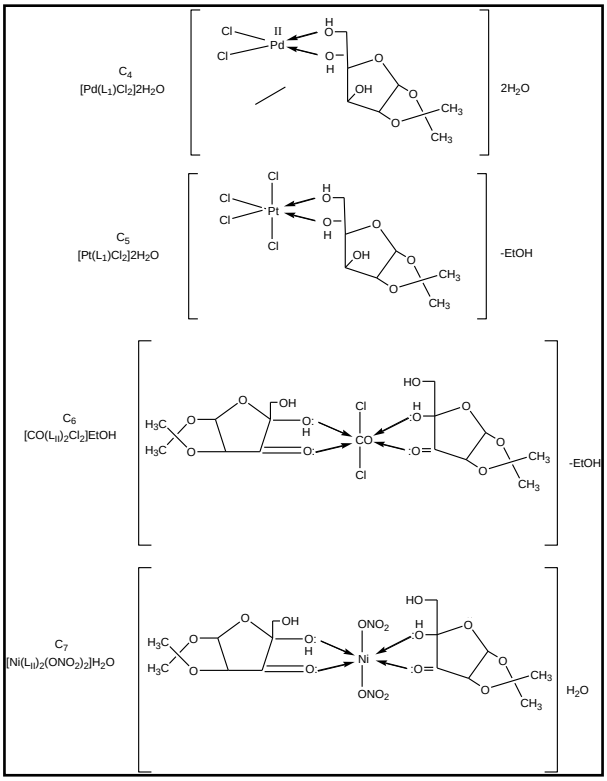

Symbol		υ O-H	υ 2OH	υ No3	H2O Coord. (Lattice)	Lattice Et OH	υ M-O	υ M-CL
L1		3429	3470 3300		---		---	---
C1 [Co(LI)Cl2]H2O		=	3225(b)	/	(3475)		445	
C2 [Ni(LI)2(ONO)2].2H2O		=	3450 (b)	1382* 1110*	(3500)		424	

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			861				
C3 [Cu(LI)2(H2O)2]Cl2.H2O	=	3360 (b)	/	3550sh (3500) 655		485	
C4 [Pd(LI)Cl2]2H2O	=	3220 (b)	/	3367		550	303
C5 [Pt(LI)Cl2]EtOH	=	3215	/	/	3540	463	308
LII	3470 3300	1740	/	---	---	---	---
C6 [Co(LII)2Cl2]EtOH	3450 (b)	1688	/	---	3408 (sh)	420	206
C7 [Ni(LII)2(ONO2)2]H2O	3270 (b)	1685	1370 1108 820	(3510)	/	460	
C8 [Cu(LII)2Cl2]2EtOH	3350 (b)	1695	/	3466	/	512	260 t 302 t
C9 [Pd(LII)2Cl2].2EtOH	3470 3305	1700	/	/	3400	545	240 200
C10 [Pt(LI)Cl2]EtOH	3469 3300	1690	/	(3530)	/	502	220

Table (2): FTIR vibration for the ligands and their metal complexes.

Symbol	Band position (cm-1)	Assignment	Dδ/B-	B- (cm-1)	β = B-B	10 Dδ (cm-1)	μeff (B.M)	Molor conductance (s-mol-1. cm2) DMF (10-3) /ionic ratio	Suggested structure
C1 Co(II)L1	υ1 5737 (Cal) υ2 12155 υ3 15894	4T1g → 4T2g 4T1g(F) → 4A2g 4T1g → 4T1g(p)	0.80	742.11	0.764	5965	5.531	7.0 non electrolyte (1:2)	Octahedral
C2 Ni(II)L1	υ110100 υ2 15295 υ3 20443	3A2g → 3T2g 3A2g → 3T1g 3A2g → 3T1g(p)	1.31	793	0.769	10105	3.95	9.0 non electrolyte (1:2)	Octahedral
C3 Cu(II)L1	υ112282 υ216500	2B1g → 2A1g 2B1g → 2B2g	---	---	---	---	2.55	175.0 electrolyte (1:2)	Oh.
C4 pd(II)L1	υ111090 υ2 15608 υ3 21053	1A1g → 1A2g 1A1g → 1B1g 1A1g → 1Eg	---	---	---	---	Diam.	5.0 non electrolyte (1:1)	Square plana
C5 Pt(IV) L1	υ116776 υ2 21221 υ3 22551	1A1g → 3T2g 1A1g → 3T2g	---	---	---	---	Diam.	12 non electrolyte (1:1)	Oh.
C6 Co(II)L2	υ16665 (cal) υ2 13250 υ3 16850	4T1g → 4T2g 4T1g(F) → 4A2g 4T1g → 4T1g(p)	0.916	825.2	0.849	7560.1	5.93	32 non electrolyte (1:2)	Oh.

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C7 Ni(II)L2	υ111102 υ2 15292 υ3 22320 (avr.)	3A2g → 3T2g 3A2g → 3T1g 3A2g → 3T1g(p)	0.128	8.54	0.828	10950	3.93	7.0 non electrolyte (1:2)	Oh.
C8 Cu(II)L2	υ113425 (ovr) υ2 14875	2B1g → 2A1g 2B1g → 2B2g					1.91	192 electrolyte (1:2)	Oh.
C9 Pd(II)L2	υ114221 υ2 15352 υ3 19725	1A1g → 1A2g 1A1g → 1B1g 1A1g → 1Eg	---	---	---	---	Diam	15.0 Non electrolyte (1:2)	Square planar
C10 Pt(II)L2	υ114757 υ2 18650	1A1g → 3T2g 1A1g → 3T2g	---	---	---	---	Diam	2.2 non electrolyte (1:1)	Octa.

Table (3) Electronic spectra, spectral `parameters molar conductance and magnetic moment with suggested structures of L1 and LII complexes.

* mono dentate
t=terminal

Antibacterial Activity:

The antibacterial action of the prepared compound in DMSO and H_2O (1000ppm) were studied following the agar diffusion method[27,28,29], the microorganisms used:staphylococcus aureus and Esacherichia coli, L1 &LII was found completely in active towards bacterial[29] action. Complexes showed various behaviors. In some cases complexation with metal ions was found to increase the biological activities of the studied ligand, which was attributed to the synergic effect of the metals and the ligands. The highest growth inhibition against the two types of bacteria was recorded by CuL1, pdL1, pdLII, and ptLII.

Table (4): Antibacterial activities of studied compounds

Compounds	Staplylocc (Cus Aureus inhibition diameter	Esacherichia coli inhibition diameter (mm) (1000ppm)
DMSO	Zero	Zero
LI	---	---
C1 [Co(LI)2Cl2]H2O	10mm	
C3 [Cu(LI)2(H2O)2]Cl2.H2O	12mm	15mm
C4 [Pd(LI)Cl2]2H2O	10mm	22mm
C5 [Pt(LI)Cl2]EtOH	20mm	18mm
LII	---	---
C7 [Ni(LII)2(ONO2)2]H2O	++	+++
C9 [Pd(LII)2Cl2]2EtOH	25mm	30mm
C10 K[Pt(LII)Cl5	30mm	35mm

Conclusion

The prepared ligands represent a new group of secures exhibiting bidentate chelating behavior with different metal ions. The presence of two hydroxyl groups led to the formation of bi and mononuclear complexes. The presence of carbonyl group led to the difference in structure between both ligands which led to difference in structures of metal complexes and biological activities.

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