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Research Article

X-RAY DIFFRACTION ANALYSIS OFTi(IV), Zr(IV), Cd(II) and Hg(II) Chelates with 4-CHLORO-2-(2-OXO-1, 2-DIHYDRO-INDOL-3-YLIDENE AMINO)-BENZOIC ACID

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ABSTRACT

4-Chloro-2-(2-oxo-1, 2-dihydro-indol-3-ylidene amino)-benzoic acid withused to synthesize Ti(IV), Zr(IV), Cd(II) and Hg(II) chelates. Metal chelates were characterized by elemental analysis,UV-Visible, Infra-red, Nmr and TGA/DSC.The crystal structure of chelates were further determined by X-ray diffraction method. The XRD data was used to index the compound for monoclinic and orthorhombic system.

Keywords: Crystal structure, (ACBAI), Ti(IV),Zr(IV),Cd(II) and Hg(II)complexes.

INTRODUCTION

Schiff bases are widely employed in synthetic organic and inorganic chemistry, they wereereported to show diverse biological activity,and have many applications as ligands incoordination chemistry of transition metals[1].Schiff bases obtained by the condensation of aromatic amines with isatin are powerful anticonvulsant, antiviral, antibacterial and antifungal agents[2,3]Containing different donor atoms, the Schiff bases are an important class of ligands in the coordination chemistry, widely reported[4-6].The chemistry of transition metal complexes with multi dentate ligands have attracted attention because these metal ions can exhibit several oxidation states, metal complexes with Schiff base ligands.These have been studied for their application in biological, clinical, analytical and pharmacological areas[7-9].

Reduced Schiff base have recently gained considerable attention, because of the flexibility of Schiff base ligands can be improved by hydrogenation of their C=N bonds, they should thus co-ordinate metal ions move easily[10-11].Isatin, an endogenous indole and its derivatives exhibit a wide range of biological activities[12]. Metal–isatin binary complexes wereadvantageous over simple isatin in chemotherapy and found to act as anticancer agents, especially Schiff-basetransition metal complexes derived from isatin[13].

EXPERIMENTAL

All the chemicals used for the preparation of chelates are of Merck and AR grade mark. Metal chelates are synthesized by adding metal salt solution in appropriate solvent to the solution of the ligand. The P^H of reaction mixture is adjusted about 6.9 to 7.3 by adding alcoholic ammonia drop wise. The mixture was refluxed for five to six hours. Then the precipitate of metal chelates was obtained. It is filtered, washed & dried in vacuum desiccators.Preparation method was given in details in published papers.

RESULT AND DISCUSSION

X-ray diffractograms of the metal chelates were recorded in the 2θ range from 10-90^o at a wave length of 1.5405 A^{o}and using Cu Kα radiation source. Results of miller indices, lattice parameters and unit cell volume are computed from programme. Data has been summarized in the following tables.

[Ti(ACBAI)$H_2O.Cl_2$]Cl
Crystal system: Monoclinic Lattice Type: P
Radiation: CuWavelength: 1.540598 A^0
Lattice Parameter:a= 4.9168 b= 4.9168 c= 5.4089 A^0
Lattice Parameter: Alpha= 90 Beta= 90 Gama=120^0
2Theta Start= 10 2Theta End= 89.98

Table 1 : Powder XRD data of [Ti(ACBAI)$H_2O.Cl_2$]Cl Complex

h	k	l	2θ	2θ	d	d	Intensity
			(Exp.)	(Calc.)	(Exp.)	(Calc.)	(Exp.)
0	0	1	16.349	16.375	5.41759	5.40890	59.82
-1	1	2	39.401	39.440	2.28508	2.28291	60.50
-1	2	1	40.214	40.263	2.24071	2.23807	64.95
-2	0	1	45.707	45.763	1.98340	1.98109	62.51
1	1	2	50.170	50.104	1.81691	1.81913	61.46
0	0	3	50.627	50.585	1.80158	1.80297	62.93
0	2	2	54.833	54.835	1.67292	1.67286	61.44
0	1	3	55.347	55.286	1.65858	1.66027	62.18
-2	3	0	57.225	57.191	1.60854	1.60940	60.48
1	2	1	59.931	59.916	1.54221	1.54256	60.61
-3	0	0	65.695	65.737	1.42016	1.41936	61.52
-3	1	2	67.722	67.692	1.38250	1.38303	58.90
0	0	4	69.388	69.452	1.35333	1.35222	63.83
-1	0	4	73.371	73.409	1.28937	1.28880	63.78
-3	3	2	75.659	75.601	1.25597	1.25679	59.29
-4	2	0	77.671	77.609	1.22838	1.22920	61.08
-3	2	3	79.868	79.818	1.20001	1.20064	62.21

The cell data and crystal parameters of [Ti(ACBAI)$H_2O.Cl_2$]Cl complex is given in the tables indicates that complex have monoclinic crystal system[14].

[Zr(ACBAI)$H_2O.Cl_2$]Cl
Crystal system: Monoclinic Lattice Type: P
Radiation: Cu Wavelength: 1.540598 A^0
Lattice Parameter: a= 4.9168 b= 4.9168 c= 5.4089 A^0
Lattice Parameter: Alpha= 90 Beta= 90 Gama=120^0
2Theta Start= 10 2Theta End= 89.98

Table 2 : Powder XRD data of [Zr(ACBAI)$H_2O.Cl_2$]Cl Complex

h	k	l	2θ	2θ	d	d	Intensity
			(Exp.)	(Calc.)	(Exp.)	(Calc.)	(Exp.)
0	0	1	16.373	16.375	5.40949	5.40890	55.43
-1	1	0	20.769	20.845	4.27336	4.25807	51.68
-1	1	1	26.610	26.622	3.34721	3.34573	43.48
-2	1	0	36.540	36.520	2.45716	2.45840	48.54
-1	1	2	39.356	39.440	2.28754	2.28291	42.58
-1	2	1	40.248	40.263	2.23892	2.23807	45.50
-2	0	1	45.832	45.763	1.97826	1.98109	37.22
1	1	2	50.051	50.104	1.82095	1.81913	35.66
0	0	3	50.601	50.585	1.80244	1.80297	34.88
0	2	2	54.863	54.835	1.67206	1.67286	31.93
1	2	1	59.903	59.916	1.54285	1.54256	32.33
-3	0	0	65.739	65.737	1.41930	1.41936	33.62
0	3	1	68.198	68.262	1.37400	1.37288	29.68

Cell data and crystal lattice parameters of [Zr(ACBAI)$H_2O.Cl_2$]Cl complex attributed to monoclinic crystal system[15]. Cell data and crystal cell parameters of Zr(IV) complex is given in the table 2.

[Cd(ACBAI)H_2O]Cl
Crystal system: Orthorombic Lattice Type: P
Radiation: Cu Wavelength: 1.540598 A^0
Lattice Parameter: a= 4.9168 b= 4.9168 c= 5.4089 A^0
Lattice Parameter: Alpha= 90 Beta= 90 Gama=90^0
2Theta Start= 10 2Theta End= 89.98

Table 3: Powder XRD data of [Cd(ACBAI)H_2O]Cl Complex

h	k	l	2θ	2θ	d	d	Intensity
			(Exp.)	(Calc.)	(Exp.)	(Calc.)	(Exp.)
0	0	1	16.399	16.375	5.40117	5.40890	53.38
0	1	0	18.060	18.027	4.90798	4.91680	48.78
0	1	1	24.418	24.447	3.64245	3.63826	41.54
0	0	2	33.097	33.097	2.70442	2.70445	55.43
0	2	0	36.468	36.520	2.46182	2.45840	46.84
0	2	1	40.332	40.263	2.23442	2.23807	36.80
1	1	2	42.330	42.305	2.13345	2.13466	64.07
1	2	1	44.476	44.439	2.03540	2.03697	30.02
0	0	3	50.533	50.585	1.80469	1.80297	31.47
2	2	0	52.530	52.606	1.74071	1.73835	27.59
2	1	2	53.649	53.679	1.70699	1.70610	26.56
2	2	1	55.494	55.478	1.65454	1.65498	30.62
1	1	3	57.512	57.537	1.60118	1.60055	24.95
0	3	1	58.876	58.826	1.56731	1.56851	29.43
0	2	3	64.039	63.987	1.45282	1.45388	24.89
0	3	2	66.746	66.675	1.40031	1.40164	22.21
1	2	3	67.013	67.077	1.39540	1.39421	30.75
2	2	3	75.946	75.982	1.25193	1.25142	24.44
0	4	1	80.036	79.979	1.19793	1.19864	20.67

Cell data and crystal lattice parameters of [Cd(ACBAI)H_2O]Cl complex indicates that complex have Orthorhombic crystal system[16].

[Hg(ACBAI)H_2O]Cl
Crystal system: Orthorombic Lattice Type: P
Radiation: Cu Wavelength: 1.540598 A^0
Lattice Parameter: a= 4.9168 b= 4.9168 c= 5.4089 A^0
Lattice Parameter: Alpha= 90 Beta= 90 Gama=90^0
2Theta Start= 10 2Theta End= 89.98

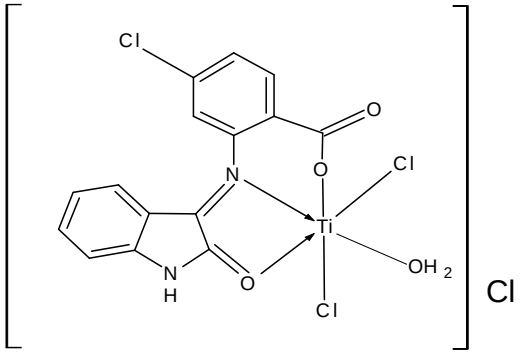
Table 4: Powder XRD data of [Hg(ACBAI)H_2O]Cl Complex

h	k	l	2θ	2θ	d	d	Intensity
			(Exp.)	(Calc.)	(Exp.)	(Calc.)	(Exp.)
0	1	0	18.068	18.027	4.90583	4.91680	53.24
1	1	0	25.554	25.601	3.48300	3.47670	45.76
1	1	1	30.606	30.542	2.91861	2.92464	40.56
0	2	1	40.237	40.263	2.23950	2.23807	38.89
1	2	0	40.989	41.013	2.20014	2.19886	39.22
0	2	2	50.140	50.104	1.81794	1.81913	50.52
2	2	0	52.590	52.606	1.73884	1.73835	37.47
2	1	2	53.602	53.679	1.70839	1.70610	33.41
1	0	3	54.199	54.137	1.69098	1.69275	33.05
0	3	1	58.781	58.826	1.56961	1.56851	37.53
2	2	2	63.535	63.574	1.46312	1.46232	29.44
3	1	2	69.684	69.705	1.34829	1.34794	32.65
1	1	4	75.357	75.356	1.26024	1.26026	28.67
3	2	2	78.444	78.487	1.21820	1.21764	33.70
0	4	1	79.968	79.979	1.19877	1.19864	28.94
1	4	0	80.440	80.474	1.19293	1.19250	29.58
1	3	3	81.780	81.718	1.17673	1.17747	30.60
1	4	1	82.848	82.824	1.16425	1.16453	29.93
3	3	0	83.241	83.316	1.15975	1.15890	25.80

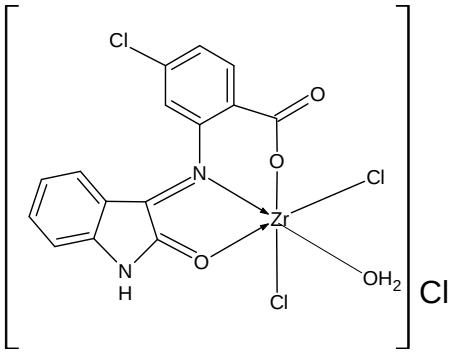
Cell data and crystal lattice parameters of [Hg(ACBAI)H_2O]Cl complex indicates that complex have Orthorhombic crystal system[17] with lattice type-P.

CONCLUSION

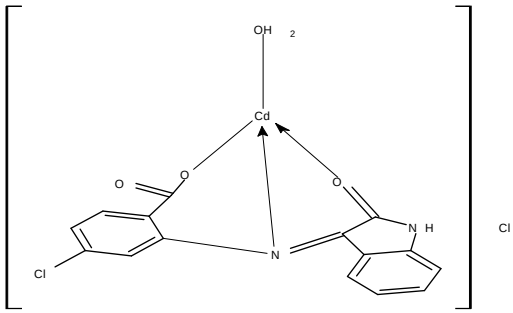
Powder X-ray diffraction studies of the complexes revealed monoclinic test for $[Ti(ACBAI)H_2O.Cl_2]Cl$and $[Zr(ACBAI)H_2O.Cl_2]Cl$where as orthorhombic test for $[Cd(ACBAI)H_2O]Cl$, $[Hg(ACBAI)H_2O]Cl$ with P type lattice. From above observations it is observed that the precursor of heterocyclic ligand has undergone structural rearrangements to acquire stability and coordinated to other metals through donor groups. Following structures are proposed to the metal chelates of ligand ACBAI.



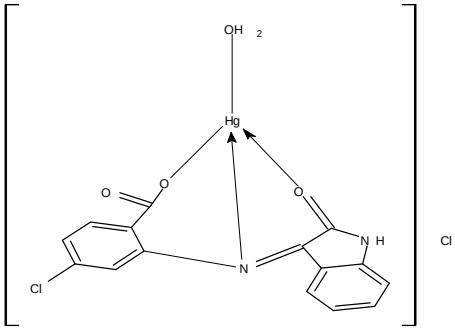
Proposed Structure of Complex $[Ti(ACBAI)H_2O.Cl_2]Cl$



Proposed Structure of Complex $[Zr(ACBAI)H_2O.Cl_2]Cl$



Proposed Structure of Complex $[Cd(ACBAI)H_2O]Cl$



Proposed Structure of Complex $[Hg(ACBAI)H_2O]Cl$

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