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X-Ray Diffraction Studies of Some Novel Di-Oxomolybdenum (VI) Complexes Derived From Nicotinoyl Hydrazones

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ABSTRACT

Some novel dioxo-molybdenum (VI) hydrazone complexes of Schiff base ligands having the general formula cis-[MoO_2(L)(solv)] (where LH_2 = Schiff base ligand and solv=ethanol) are synthesized, characterized by elemental, spectral analysis and structure and symmetry properties is determined using XRD X-ray powder diffractograms of the dioxo-molybdenum (VI) complexes of Schiff base derived from salicylaldehyde(sal) and substituted salicylaldehyde such as o-hydroxyacetophenone (hap); o-hydroxypropiophenone (hpp); o-hydroxybenzophenone (hbp) with nicotinic acid hydrazide (NAH) have been studied and all were found crystalline. It is observed that the structures of all the complexes (**1-4**) belong to triclinic system. The lattice parameters and Miller's indices were computed. The indexing and calculation of unit cell parameters were performed with the help of Powder-X Software. The 2θ values, the relative intensity, the inter-planar distance along with Miller's indices for corresponding angles are calculated for the complexes. After indexing the X-ray powder patterns and unit cell refinements, it is found that the complexes (**1-4**) are crystalline and adopt triclinic crystal system with P type lattice. This is expected for distorted octahedral complexes.

Keywords: Dioxo-molybdenum (VI) Hydrazone complexes, Schiff base ligands, XRD, Triclinic.

Introduction

Coordination chemistry of molybdenum(VI) has attracted considerable attention due to its biochemical significance[1]as well as for the efficient catalytic properties in several organic synthesis procedures.[2] Schiff bases are a kind of interesting ligands in coordination chemistry.[3] In recent years, a number of molybdenum(VI) complexes with Schiff bases derived from salicylaldehyde and primary amines have been reported.[4] Hydrazones, bearing –C(O)–NH–N=CH– groups, are a kind of special Schiff bases, which are of particular interest in coordination chemistry and biological applications. Schiff base complexes of molybdenum have been used in applications related to catalytic, enzymological and oxygen transfer reactions [5]. Tridentate dibasic Schiff base complexes such as cis-MoO_2L(S) (S = solvent) are good substrates for redox reactions because of the ability of S replacement with other solvent [6]. The availability of such a labile site imparts catalytic property to these complexes. Dibasic tridentate Schiff base ligands around octahedral molybdenum(VI) provide suitable geometry with one vacant site for substrate binding [7-9].We have been interested in five co-ordinated octahedral *cis*-MoO_2 complexes with non-symmetrical Schiff base having mixed sets of donor atoms in which the sixth co-ordination site is occupied by a solvent molecule. . X-ray powder diffractometry deals exclusively with crystalline

materials, the diffraction pattern being used to determine the degree of crystallinity, (e.g. the dimensions of the crystalline region in otherwise amorphous substance).

In view of this, we report the synthesis and and X-ray diffraction analysis of some *cis*-dioxomolybdenum(VI) complexes with Schiff bases derived from salicylaldehyde(sal), o-hydroxy-acetophenone(hap) and o-hydroxy-propiophenone (hpp) with nicotinic acid hydrazide (NAH). These Schiff bases form mononuclear dioxomolybdenum(VI) complexes having the general formula $MoO_2(L)(S)$ (where LH_2= Schiff base represented as H_2sal-NAH, H_2hap-NAH and H_2hpp-NAH). The ligands and the complexes are characterized by elemental analysis, molar conductance and spectroscopic (IR, ^{1}HNMR and UV-Vis), thermogravimetric (TGA) and X-ray diffraction analysis.

Physical Measurements

Microanalysis of the Schiff base ligands and complexes were performed on a Perkin-Elmer(USA) 2400 Series II, elemental analyzer. The solutions of both ligands and complexes were prepared in HPLC grade DMF and electrical conductance measurements were performed using a Toshniwal Conductivity Bridge and a dip type cell calibrated with potassium chloride solutions. IR spectra for ligands and complexes were recorded in the range 400-4000cm^{-1} on a Nicolet Magna IR 550 series II spectrophotometer using KBr pellets. ^{1}H NMR spectra were recorded in DMSO-d^6 on a Bruker DRX-300 instrument, using TMS as an internal standard. Electronic spectra were recorded for solutions of ligands and complexes in DMF on a Shimadzu UV 3101 PC spectrophotometer. The thermogravimetric analysis is done on Mettler Toledo (Star Switzerland SDTA/TGA 851) Instrument, to determine the decomposition temperature of complexes. The X ray diffraction patterns have been recorded in 2θ range from 13 to 64° on Philips (Holland) automated X-ray powder diffractometer. The operating target voltage was 35 kV, and the tube current was 20 mA. The scanning speed was 0.5 2θ/min. Radiation used was Cu-k wavelength 1.54056 A^o using monochromater for filtering β – radiations and reducing noise due to white radiations and also to increase resolution. The values of interplaner spacing (d) corresponding to Bragg reflections (2θ) were obtained and indexing and calculation of unit cell parameters were performed with the help of Powder-X Software [11-14].

Materials & Methods

Ammonium molybdate(VI) tetrahydrate was obtained from Sisco Research Laboratory,(Mumbai,India).**S**alicylaldehyde**,** o-hydroxyacetophenone, o-hydroxypropiophenone, o-hydroxybenzophenone and benzoic acid hydrazide were procured from Lancaster synthesis Ltd**.** (UK). Ethanol and acetone used as solvent for synthesis were of high purity.

SYNTHESIS OF SCHIFF BASE LIGANDS (1a-4a): The Schiff base ligand was synthesised by refluxing an ethanolic solution of ketone/aldehyde such as o-hydroxyacetophenone (hap); o-hydroxypropiophenone (hpp); o-hydroxybenzophenone (hbp) and salicyldehyde (sal) with nicotinic acid hydrazide(NAH) in 1: 1 molar ratio.

SYNTHESIS OF COMPLEXES (1b-4b): To the hot ethanolic solution of the appropriate Schiff base ligand (1 mmol,) was added an ethanolic solution of [*cis*-$MoO_2(acac)_2$] (1 mmol) with vigorous stirring.Bis(acetylacetonato)dioxomolybdenum(VI) [*cis*-($MoO_2(acac)_2$] undergoes ligand exchange with the Schiff bases in a suitable solvent and complexes of the type $MoO_2(L)(S)$ (where LH_2 = Schiff base) are formed as follows:

$$MoO_2(acac)_2 + LH_2 \xrightarrow{EtOH} \textit{cis}\text{-}MoO_2(L)(S) + 2\ acacH$$

Where, S is a solvent

RESULTS AND DISCUSSIONS

Bis(acetylacetonato) dioxomolybdenum(VI) [*cis*-$MoO_2(acac)_2$], undergoes ligand exchange reaction with the Schiff bases (1a-4a) and complexes of the type [MoO_2 (L)(**S**)] (where LH_2= Schiff base) are formed as follows:

$$\textit{cis}\text{-}MoO_2(acac)_2 + LH_2 \xrightarrow[EtOH]{\Delta,\ 6hrs} \textbf{\textit{cis}}\text{-}[MoO_2(L)(\textbf{S})] + 2\ acacH$$

The Schiff bases behave as dibasic, tridentate ligands, hence complete replacement of the bidentate acetylactone occurs under the reaction conditions and the solvent molecule occupies the sixth coordination position[15].The analytical data further support the formulation of the complexes as *cis*-[MoO_2 (L)(S)]. The structures 1a-4a for ligands and 1b-4b for complexes are shown in Fig 1.

X-ray powder diffraction analysis: The single crystals of complexes in DMF could not be obtained; hence, XRD patterns of the same are studied and reported. All the complexes were found to be crystalline and their X-ray powder diffractograms were collected. The lattice parameters and Miller's indices were computed. Various lattice parameters for complexes are tabulated in Table 1. The indexing and calculation of unit cell parameters are performed with the help of Powder-X Software. The calculated and the observed 2θ value, the relative intensity, the interplanar distance along with Miller's indices for corresponding angles are tabulated for the complexes (Tables 2-4).

On the basis of X-ray powder patterns and unit cell refinements, it is found that all the complexes adopt triclinic crystal system with P type lattice space group. The lattice constants were calculated, complex **(1b)** - a = 7.2Å ; b = 10.45 Å ; c = 10.1 Å and α = 79.6° , β = 78.4° γ = 78.0° ; **(2b)** - a = 7.65 Å ; b = 10.0 Å ; c = 10.3 Å and α =78.2° ; β= 80.1°; γ= 79.1°; (**3b**) - a = 7.9 Å b = 10.1 Å ; c = 10.35 Å and α = 80°, β = 79°, γ = 76°. **(4b)** - a = 7.59 Å b = 10.1 Å ; c = 10.42 Å and α = 81°, β = 80°, γ = 79°. We have earlier reported the single crystal structure of cis-MoO_2(L)(Solv), where L = salicylaldehyde salicyloyl hydrazide) which was found to be triclinic with P type space group[10].

CONCLUSIONS:

It is evident from the above data, the Schiff base ligands behave as dibasic tridentate ligands and co-ordinate through phenolic oxygen, azomethine nitrogen and enolic oxygen atoms. The complexes are found to be monomers, non-electrolytes, diamagnetic and six co-ordinated**.** The sixth site in the complex is occupied by an

ethanol/water group, which allows the binding and displacement of several substrate molecules during their use as a catalyst in the oxidation reactions. The use of the complexes of this type as catalyst for epoxidation of olefins is studied and reported elsewhere [7-9]. After indexing the X-ray powder patterns and unit cell refinements, it is found that the complexes (1b-4b)are crystalline and adopt triclinic crystal system with P type lattice. This is expected for distorted octahedral complexes.

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TABLES AND FIGURES:

Table 1

S.No	Complex	Lattice Parameters (unit cell dimensions)			Lattice Parameters		
		a	b	c	Alpha	Beta	Gamma
1.	MoO_2(hap-NAH)(C_2H_5OH) **(1)**	7.2 °A	10.45 °A	10.1 °A	79.6°	78.4°	78°
2.	MoO_2(hpp-NAH)(C_2H_5OH) **(2)**	7.65 °A	10 °A	10.3 °A	78.2°	80.1°	79.1°
3.	MoO_2(hbp-NAH) (C_2H_5OH) **(3)**	7.9 °A	10.1 °A	10.35 °A	80 °	79 °	76 °
4.	MoO_2 (sal-NAH)(C_2H_5OH) **(4)**	7.59 °A	10.1 °A	10.42 °A	81 °	80 °	79°

Table 2 : X-ray Diffraction Data of Complex MoO_2(hap-NAH)(C_2H_5OH) (1b)

h	k	l	2 θ (Exp.)	2 θ (Calc.)	2 θ (Diff.)	d (Exp.)	d (Calc.)	Intensity (Exp.)
-1	0	0	12.463	12.745	-0.282	7.09655	6.94016	2105.94
-1	-1	0	14.221	14.121	0.100	6.22305	6.26686	138.44
-1	1	0	16.658	16.717	-0.059	5.31756	5.29905	260.90
0	2	0	17.421	17.523	-0.103	5.08652	5.05698	63.06
0	0	2	18.262	18.109	0.154	4.85392	4.89474	218.47
1	-1	1	18.758	18.590	0.168	4.72683	4.76907	124.95
0	-1	2	21.748	21.263	0.485	4.08315	4.17525	72.87
0	2	2	23.200	23.373	-0.173	3.83083	3.80293	53.38
-1	2	1	24.631	25.105	-0.474	3.61146	3.54428	104.08
-1	2	1	25.138	25.105	0.033	3.53971	3.54428	165.13
0	3	1	26.559	26.669	-0.110	3.35349	3.33993	70.31
0	-4	1	38.139	37.990	0.149	2.35772	2.36660	76.59

Crystal system: Triclinic Lattice Type: P
Lattice Parameters: a = 7.2 °A
(Unit cell dimensions) b = 10.45 °A
c = 10.1 °A
Lattice Parameters: Alpha = 79.6°
Beta = 78.4°
Gamma = 78°

Table 3 : X-ray Diffraction Data of Complex MoO_2(hpp-NAH)(C_2H_5OH) (2b)

H	k	l	2Theta (Exp.)	2Theta (Calc.)	2Theta (Diff.)	d (Exp.)	d (Calc.)	Intensity (Exp.)
0	1	1	11.584	11.547	0.038	7.63274	7.65746	26.29
-1	-1	0	13.523	13.804	-0.281	6.54270	6.41019	114.59
-1	-1	0	13.802	13.804	0.001	6.41088	6.41019	155.78
1	1	1	14.359	14.529	0.171	6.16367	6.09161	64.92
-1	0	1	15.962	15.798	0.163	5.54799	5.60497	39.52
-1	1	0	16.608	16.141	0.467	5.33348	5.48664	50.56
0	2	0	18.377	18.348	0.030	4.82382	4.83157	102.66
1	2	1	20.026	20.026	0.000	4.43020	4.43024	63.18

1	2	0	20.549	20.238	0.311	4.31872	4.38431	67.82
0	-1	2	20.950	21.419	-0.469	4.23689	4.14511	224.40
0	-2	1	22.065	21.796	0.269	4.02525	4.07440	64.92
0	2	2	23.269	23.213	0.056	3.81962	3.82873	42.87
-2	0	0	23.944	23.903	0.041	3.71347	3.71972	91.78
2	0	1	24.408	24.330	0.078	3.64397	3.65543	60.34
-1	2	1	24.602	24.551	0.052	3.61557	3.62308	46.44
-2	-1	1	27.453	27.510	0.057	3.24625	3.23963	21.07
-2	1	1	28.927	29.003	0.076	3.08408	3.07619	32.97
1	2	3	29.470	29.464	0.006	3.02853	3.02913	26.51
-2	-2	1	31.172	31.244	-0.072	2.86695	2.86050	28.87
-1	-3	1	31.678	31.730	-0.052	2.82229	2.81776	29.87
2	1	3	32.365	32.367	-0.002	2.76391	2.76375	19.21
-1	2	3	34.573	34.613	-0.040	2.59228	2.58937	27.28
0	-3	2	35.695	35.731	-0.036	2.51332	2.51088	24.47
1	4	1	36.267	36.262	0.005	2.47499	2.47530	19.19
2	-2	2	37.338	37.352	-0.014	2.40643	2.40558	19.80
-3	0	1	38.514	38.572	-0.058	2.33561	2.33225	25.88
-2	1	3	39.515	39.610	-0.095	2.27871	2.27345	21.91
-2	-1	3	40.058	40.072	-0.014	2.24909	2.24833	22.85
2	4	2	40.703	40.699	0.004	2.21491	2.21512	24.35
2	-2	3	42.610	42.649	-0.038	2.12009	2.11827	21.11
-2	2	3	42.749	42.706	0.044	2.11350	2.11557	22.33
0	-2	4	43.652	43.637	0.016	2.07185	2.07256	22.70

Crystal system : Triclinic Lattice Type: P

Lattice Parameters : a = 7.65 °A

(unit cell dimensions) b = 10 °A

c = 10.3 °A

Lattice Parameter : Alpha = 78.2°

Beta = 80.1°

Gamma = 79.1°

Table 4: X-ray Diffraction Data of Complex MoO_2(hbp-NAH) (C_2H_5OH) (3b)

H	k	l	2θ (Exp.)	2θ (Calc.)	2θ (Diff.)	d (Exp.)	d (Calc.)	Intensity (Exp.)
-1	0	0	11.357	11.678	-0.321	7.78530	7.57183	101.42
0	1	1	11.796	11.774	0.022	7.49612	7.51027	99.76
0	-1	1	13.426	13.477	-0.051	6.58947	6.56468	327.81
1	1	1	13.835	14.051	-0.216	6.39554	6.29791	161.00
1	1	1	14.456	14.051	0.405	6.12245	6.29791	87.27
-1	0	1	15.520	15.688	-0.168	5.70475	5.64406	115.84
-1	0	1	15.930	15.688	0.242	5.55902	5.64406	121.18
0	0	2	17.767	17.603	0.164	4.98823	5.03421	163.40
0	2	0	18.087	18.255	-0.168	4.90053	4.85590	176.50
0	1	2	18.629	18.722	-0.093	4.75936	4.73593	113.65
1	0	2	19.548	19.581	-0.033	4.53756	4.53004	185.45
1	0	2	19.936	19.581	0.356	4.45005	4.53004	170.05

0	-1	2	20.763	20.918	-0.154	4.27456	4.24339	286.22
0	-2	1	21.225	21.335	-0.110	4.18263	4.16128	238.64
-1	0	2	22.251	22.664	-0.413	3.99196	3.92019	189.83
1	2	2	23.235	23.252	-0.016	3.82513	3.82247	110.25
-1	-1	2	24.305	24.402	-0.097	3.65917	3.64482	115.68
-1	2	1	25.013	25.180	-0.167	3.55713	3.53390	196.93
2	2	1	25.844	25.941	-0.097	3.44459	3.43199	134.99
2	2	0	26.482	26.522	-0.041	3.36307	3.35802	111.12
0	3	0	27.520	27.531	-0.010	3.23847	3.23727	140.91
2	2	2	28.120	28.319	-0.199	3.17075	3.14895	113.76
2	2	2	28.492	28.319	0.173	3.13019	3.14895	122.16
-1	-2	2	28.818	29.144	-0.326	3.09553	3.06163	107.83
1	3	2	29.557	29.618	-0.061	3.01976	3.01371	135.30
-2	-2	1	29.856	29.904	-0.048	2.99025	2.98552	123.59
-1	-3	1	30.641	30.608	0.033	2.91543	2.91850	91.81
2	1	3	31.575	31.566	0.009	2.83124	2.83206	105.09
-2	-1	2	32.229	32.237	-0.008	2.77523	2.77460	81.91
2	0	3	32.800	32.761	0.039	2.72823	2.73140	95.55
2	3	2	33.403	33.086	0.317	2.68036	2.70531	95.70
3	1	1	34.118	34.093	0.025	2.62582	2.62767	94.95
3	1	0	34.764	34.710	0.054	2.57849	2.58234	108.33
3	2	1	35.496	35.458	0.038	2.52698	2.52960	80.00
-3	-1	1	37.714	37.576	0.138	2.38328	2.39171	101.91
2	1	4	38.380	38.460	-0.080	2.34346	2.33875	90.36
-1	1	4	40.296	40.000	0.297	2.23632	2.25222	79.43

Crystal system : Triclinic Lattice Type: P

Lattice Parameters : a = 7.9 °A
(unit cell dimensions) b = 10.1 °A
c = 10.35 °A

Lattice Parameter : Alpha = 80°
Beta = 79°
Gamma = 76°

Table 5 : X-ray Diffraction Data of Complex MoO_2 (sal-NAH)(C_2H_5OH) (4b)

h	k	l	2 θ (Exp.)	2 θ (Calc.)	2 θ (Diff.)	d (Exp.)	d (Calc.)	Intensity (Exp.)
0	1	1	11.522	11.679	-0.157	7.67403	7.57091	61.00
0	-1	1	12.799	13.283	-0.484	6.91080	6.66017	259.21
1	0	1	13.762	13.741	0.021	6.42940	6.43920	126.15
1	1	1	14.893	14.615	0.278	5.94369	6.05599	78.91
1	1	1	15.105	14.615	0.490	5.86055	6.05599	61.47
-1	1	0	16.280	16.187	0.093	5.44021	5.47132	154.08
0	2	0	17.972	18.027	-0.055	4.93158	4.91673	72.10
0	2	1	19.017	19.003	0.014	4.66307	4.66647	177.85
-1	-2	0	19.877	19.936	-0.059	4.46306	4.45003	128.66
-1	0	2	22.251	22.642	-0.391	3.99201	3.92399	89.54

-1	2	0	23.218	23.361	-0.142	3.82786	3.80489	28.93
-1	-1	2	24.498	24.478	0.020	3.63072	3.63360	53.75
1	-2	1	25.012	25.147	-0.135	3.55724	3.53841	71.07
1	-2	1	25.617	25.147	0.470	3.47455	3.53841	116.41
0	0	3	26.039	26.247	-0.208	3.41923	3.39263	24.37
2	0	2	27.728	27.685	0.043	3.21468	3.21960	45.38
2	-1	1	28.319	27.850	0.469	3.14895	3.20092	34.50
1	-1	3	30.284	30.429	-0.145	2.94897	2.93526	29.45
2	-1	2	31.196	31.111	0.085	2.86481	2.87245	23.25
-1	3	1	32.471	32.427	0.044	2.75518	2.75880	23.01
-2	-1	2	32.896	32.790	0.106	2.72051	2.72908	45.17
1	-3	1	33.271	33.351	-0.080	2.69068	2.68443	56.75
-1	2	3	34.900	35.016	-0.116	2.56872	2.56049	20.86
-2	0	3	38.630	38.621	0.010	2.32884	2.32940	24.00
2	2	4	40.290	40.184	0.105	2.23667	2.24229	24.32
0	-3	3	40.585	40.605	-0.019	2.22107	2.22006	22.07
-1	2	4	42.259	42.256	0.004	2.13688	2.13705	24.72
0	-4	2	42.765	42.810	-0.045	2.11279	2.11067	25.66
-1	4	2	43.210	43.221	-0.011	2.09205	2.09154	20.23
1	5	1	45.461	44.989	0.472	1.99355	2.01336	22.04

Crystal system : Triclinic Lattice Type: P

Lattice Parameters : a = 7.59 °A

(unit cell dimensions) b = 10.1 °A

c = 10.42 °A

Lattice Parameter : Alpha = 81°

Beta = 80°

Gamma = 79°

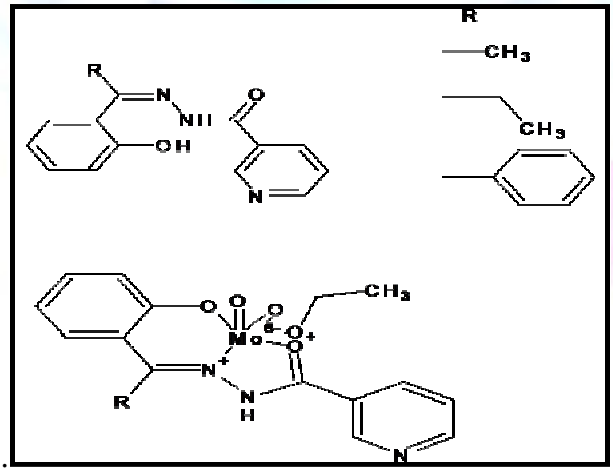

Fig1. The structures 1a-4a for ligands and 1b-4b for complexes

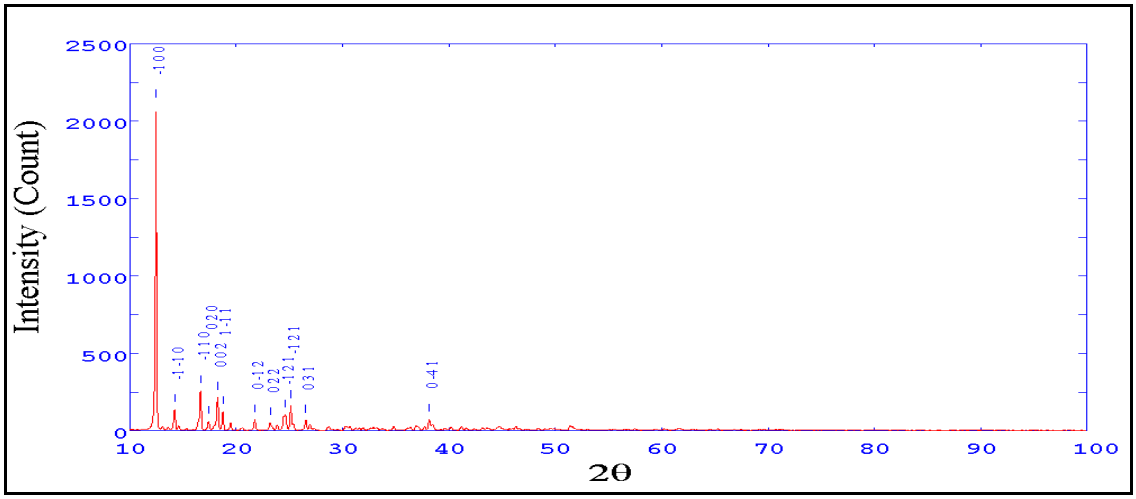

Fig. 2 : XRD Pattern for Complex MoO_2 (hap-NAH)(C_2H_5OH) (1b)

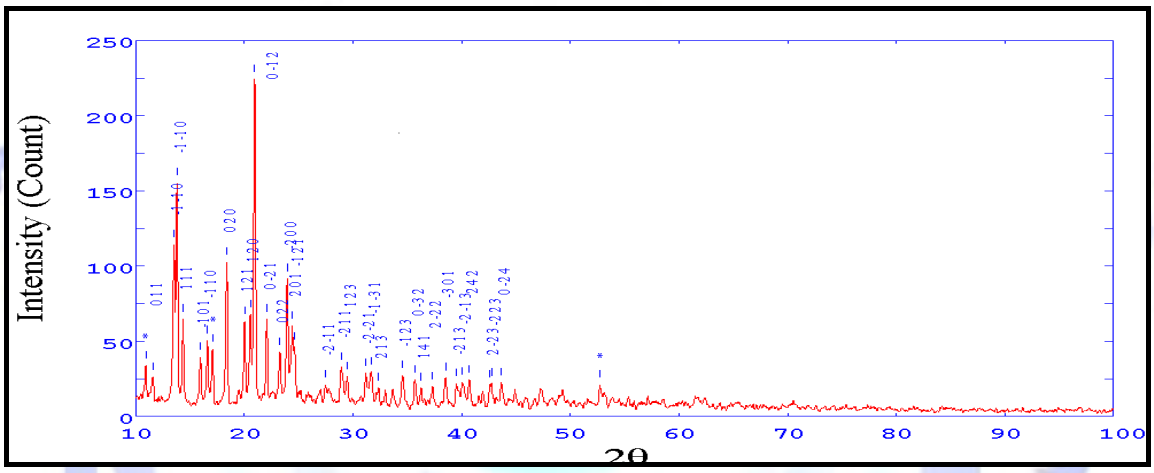

Fig. 3 : XRD Pattern for Complex MoO_2 (hpp-NAH)(C_2H_5OH) (2b)

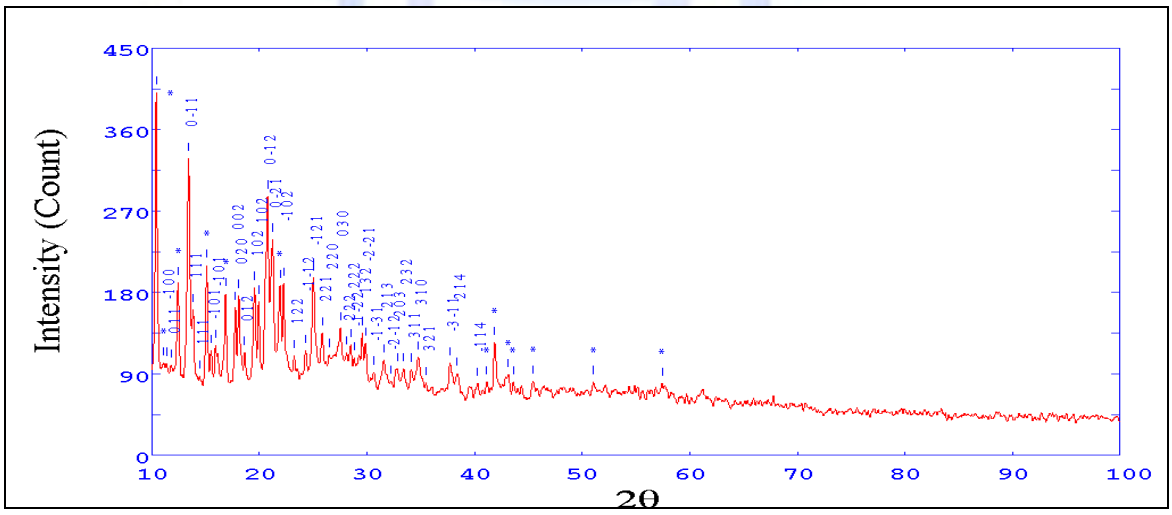

Fig. 4 : XRD Pattern for Complex MoO_2(hbp-NAH) (C_2H_5OH) (3b)

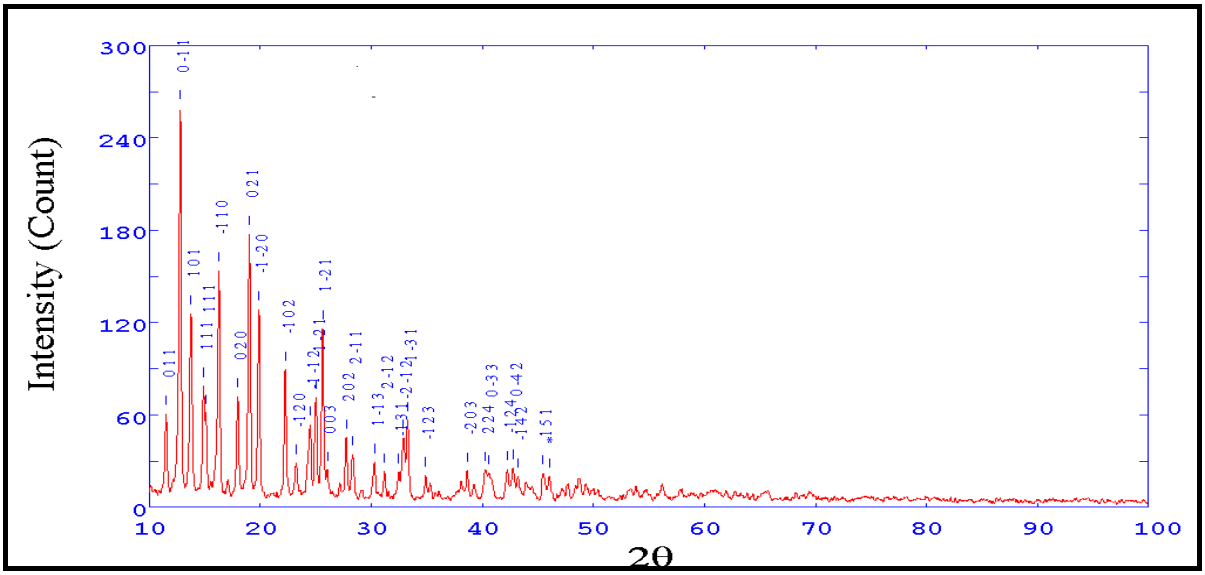

Fig. 5 : XRD Pattern for Complex MoO_2 (sal-NAH)(C_2H_5OH) (4b)