

X-Ray Powder Pattern of Pd(II) Complexes Derived from Various Substituted Schiff Bases

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This is in continuation with our work on synthesis and characterisation of Pd(II) complexes derived from Schiff bases, which were diagnosed for X-ray spectral data. A powder pattern of each complex was recorded and the data were indexed using least square method. All the compounds were X-ray crystalline. The pyknometric density and the density calculated from X-ray data are in good agreement. The compounds were indexed to monoclinic crystal system with a space group $C_{2/m}$ or $P_{2/m}$.

INTRODUCTION

Many transition metal complexes have been synthesised for their analytical and commercial applications^{1,2}. Literature survey also reveals that transition metal complexes of substituted Schiff bases show variance in the structural geometry^{3,4}. Generally, these complexes are crystalline but the systematic studies on determination of lattice parameters are the shortcomings for most of these complexes. In continuation of our work for synthesising and characterising Pd(II) complexes of substituted Schiff bases, an attempt was made to evaluate lattice parameters. The X-ray powder pattern of Pd(II) complexes was indexed using least square method suggested by Ito⁵.

EXPERIMENTAL

The Pd(II) complexes were prepared by precipitation method. The Schiff base derived from 3-hydroxybenzaldehyde and 5-nitrosalicylaldehyde with few primary amines was refluxed with Pd(II) solution in equimolar proportion. The reaction mixture was refluxed for 6 h and its pH was adjusted to optimum values after cooling. The solid Pd(II) complexes were filtered, washed and dried at a constant temperature. Each Pd(II) complex was analysed for its elemental component described in the literature^{6,7}. Various spectral characteristics were obtained by recording their IR and electronic absorption spectral data. The X-ray powdered diffractograms were recorded on Shimadzu X-ray diffractograms, which were previously calibrated from silica using Ni-filtered copper radiation ($Cu\ k_{\alpha} = 1.5404 \text{ \AA}$). The relative intensities of the reflexes were calculated with respect to highest reflex. Density of each compound was determined by using toluene as a displacing solvent. The results of elemental analysis are summarised

in Table-1, while X-ray crystal data and lattice parameters are tabulated in Tables 2-7.

TABLE-1
CHEMICAL COMPOSITION OF PALLADIUM COMPLEXES

Pd(II) complexes	% Analysis, Found (Calcd.)				
	C	H	N	S	Pd
Pd(C ₁₃ H ₉ NO) ₂	61.32 (62.60)	3.87 (4.01)	5.61 (5.62)	—	21.05 (21.35)
Pd(C ₁₃ H ₉ NOS) ₂	54.42 (55.42)	3.43 (3.55)	4.47 (4.97)	11.10 (11.37)	18.74 (18.90)
Pd(C ₁₃ H ₈ N ₂ O ₃) ₂	53.49 (53.02)	3.13 (3.09)	8.97 (9.52)	—	17.95 (18.08)
Pd(C ₁₄ H ₁₀ N ₂ O ₃) ₂	53.49 (54.51)	3.34 (3.57)	8.73 (9.09)	—	16.44 (17.26)
Pd(C ₁₄ H ₁₀ N ₂ O ₃) ₂	53.91 (54.51)	3.07 (3.57)	8.98 (9.09)	—	16.80 (17.26)

TABLE-2
X-ray POWDER DIFFRACTION DATA OF Pd(C₁₃H₉NO)₂

h	k	l	d _{obs}	d _{cal}	I/I ₀
0	2	0	7.9929	7.9880	81
1	4	1	4.4269	4.4242	81
3	1	2	4.3709	4.3775	89
2	4	1	3.8870	3.8882	81
3	0	3	3.8640	3.8602	81
4	1	2	3.5480	3.5382	89
4	1	0	3.4980	3.5030	100
3	2	-2	3.4530	3.4564	92
2	3	4	3.2130	3.2202	89
2	1	-4	3.1080	3.1034	86

RESULTS AND DISCUSSION

The analytical data suggest that Pd(II) complexes of these Schiff bases be precipitated in 1 : 2 metal to ligand stoichiometry. The complexes are coloured and non-electrolytic in nature. The magnetic moment indicates that complexes are diamagnetic in nature. The electronic absorption spectra of complexes in chloroform solvent exhibit various absorption bands, which are assignable to d—d transitions. The band appearing around 2600 cm⁻¹ can be considered as charge transfer transitions. From these spectral data square-planar geometry can be assigned to these complexes.

TABLE-3
X-ray POWDER DIFFRACTION DATA OF Pd(C₁₃H₉NOS)₂

h	k	l	d _{obs}	d _{cal}	I/I ₀
0	3	0	5.6539	5.6592	100
3	0	1	4.0009	3.9911	14
3	0	2	3.9549	3.9412	14
3	1	2	3.7860	3.8392	94
1	2	3	3.6330	3.6441	55
2	5	2	2.8500	2.8440	14
2	4	4	2.4870	2.4790	14
5	0	4	2.2850	2.2712	14
4	2	-1	2.2760	2.2738	19
3	5	-1	2.2480	2.2504	14

TABLE-4
X-ray POWDER DIFFRACTION DATA OF Pd(C₁₃H₈N₂O₃)₂

h	k	l	d _{obs}	d _{cal}	I/I ₀
2	0	0	7.0079	7.0240	90
2	1	2	6.6720	3.7322	90
2	2	1	6.5440	6.5633	90
0	1	2	5.2850	5.2762	89
4	1	2	4.5210	4.2593	90
5	5	5	2.2820	2.2560	81
7	0	1	2.2590	2.2540	81
7	2	6	2.2480	2.2441	86
7	4	5	2.2340	2.2202	81
8	2	7	1.9550	1.9525	90

TABLE-5
X-ray POWDER DIFFRACTION DATA OF Pd(C₁₄H₁₀N₂O₃)₂

h	k	l	d _{obs}	d _{cal}	I/I ₀
5	1	3	6.5730	6.4968	95
5	2	3	6.6019	6.0452	50
5	2	3	6.0289	6.0451	50
5	1	4	5.7640	5.7154	35
6	1	3	5.4269	5.4812	64
7	2	5	4.2550	4.2796	90
2	3	-2	4.2309	4.2338	100
8	2	6	3.6660	3.6856	50
9	0	6	3.5809	3.6004	95
9	2	4	3.5620	3.5420	95

TABLE-6
X-ráy POWDER DIFFRACTION DATA OF Pd(C₁₄H₁₀N₂O₃)₂

h	k	l	d _{obs}	d _{cal}	I/I ₀
6	2	4	2.2960	2.2991	90
4	3	0	2.2610	2.2639	89
6	0	5	2.2530	2.2532	89
6	2	1	2.2450	2.2393	89
6	2	1	2.2370	2.2393	89
5	3	1	2.2290	2.2264	86
3	3	-1	2.2150	2.2146	85
3	2	5	1.9520	1.9489	100
2	2	-3	1.9360	1.9376	100
5	2	-1	1.9310	1.9324	100

TABLE-7
LATTICE PARAMETERS OF Pd(II) COMPLEXES

Pd(II) Complexes	A (Å)	B (Å)	C (Å)	β	V (Å ³)	d _{obs} (g/cm ³)	d _{cal} (g/cm ³)
Pd(C ₁₃ H ₉ NO) ₂	14.5972	18.6147	15.9245	139° 0'	4229	1.498	1.564
Pd(C ₁₃ H ₉ NOS) ₂	12.1099	16.9777	12.2293	109° 2'	2227	1.564	1.753
Pd(C ₁₃ H ₈ N ₂ O ₃) ₂	18.6982	18.4450	14.6583	122° 3'	3797	1.112	1.077
Pd(C ₁₄ H ₁₀ N ₂ O ₃) ₂	33.6455	28.5795	23.6880	109° 6'	14591	1.121	1.136
Pd(C ₁₄ H ₁₀ N ₂ O ₃) ₂	17.4408	9.4872	11.4025	103° 2'	1403	1.294	1.332

The IR spectra of Pd(II) complexes show characteristic bands due to azomethine stretching vibration in the range 1610–1600 cm⁻¹. The shift in these stretching vibrations suggests involvement of azomethine nitrogen in coordination with the metal ion. Two weak bands in the far infrared region pointed in the region 600 cm⁻¹ and 500 cm⁻¹ are assigned to ν(Pd—N) and ν(Pd—O) stretching vibrations respectively.

The powder diffraction patterns were indexed on a monoclinic cell by using Backcal program given by Ito⁵. The lattice parameters were refined by least square method. The Pd(II) complexes crystallise with monoclinic system having space group C_{2/m} or P_{2/m} containing Z = 4 formula factor. The structure and the crystal system of Pd(II) complexes can be compared with other reported Schiff based complexes⁸⁻¹⁰. These complexes crystallise with monoclinic system with space group C_{2/m} or P_{2/m}.

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