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X-RAY CRYSTALLOGRAPHIC STUDIES OF SOME VANADYL COMPLEXES OF SCHIFF BASES

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Abstract—Some vanadyl complexes containing nitrogen and oxygen donors have been synthesized and characterized. They have the general formulae $[VO(C_xH_yNO)_2]$ or $[VO(C_xH_yCl_zNO)_2]$. The molecular structure of these complexes has been reported elsewhere. The present investigation reports the crystal structure determined by the X-ray powder method. All the observed reflections for every sample indicate a single phase with similar crystalline structures. The observed reflections are indexed and the unit cell dimensions are determined within an accuracy of 0.02 Å.

X-ray diffractometry of some metal complexes has been greatly studied and provided information about their crystallization and unit cell dimensions etc. The authors have also studied the vanadyl complexes by X-ray diffractometry.

EXPERIMENTAL AND DISCUSSION

The authors have prepared four vanadyl complexes of Schiff bases by refluxing vanadyl sulphate and the corresponding Schiff base in an ethanolic medium in a 1:2 ratio for 2 h. Their molecular structures are characterized[1] by studying their physical properties. In the present investigation, X-ray studies of these complexes were undertaken. X-ray diffractograms were measured[2] on a PW-1010 Philips diffractometer. The patterns obtained show a concentration of reflection with an abrupt decrease in intensities at higher angles. Similar observations have been reported by Brown and Bngaall.[3] All the observed reflections were indexed by the method described by Henry *et al.*[4] All the complexes crystallized in a primitive tetragonal unit cell with dimensions ranging from 14 to 18 Å. The crystallographic results, e.g. "*d*" in A (calculated and observed) and the "*c*/*a*" ratio, are reported in Tables 1 and 2. The observed "*c*/*a*" ratio is closer to 1.0 on both sides. The calculated and observed "*d*" values agree extremely well with each other, implying that all the reported complexes have similar crystal structures.

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Table 1. Structural results of vanadyl complexes of 2-hydroxyl-1-naphthalidene-anil (HNA) and 2-hydroxy-1-naphthalidene-2′-methylanil (HNOMA)

HNA[a]			HNOMA[b]		
"*d*" in A (observed)	"*d*" in A (calculated)	Plane *hkl*	"*d*" in A (observed)	"*d*" in A (calculated)	Plane *hkl*
8.41	8.36	002	9.54	9.54	111
7.92	7.93	200	8.97	8.97	200
7.18	7.09	210	8.00	8.02	210
6.57	6.53	211	7.27	7.25	002
5.59	5.60	220	6.34	6.34	220
4.79	4.80	311	6.01	5.98	300
4.18	4.18	004	4.98	4.97	320
3.95	3.96	400	4.68	4.70	321
3.83	3.84	303	4.38	4.35	410
3.55	3.55	420	4.26	4.23	330
3.18	3.17	500	3.64	3.62	004
2.61	2.61	610	3.41	3.42	511
			3.08	3.08	530

[a] Cell—Primitive tetragonal, $a = 15.86$, $c = 16.72$ Å and $c/a = 1.054$.
[b] Cell—Primitive tetragonal, $a = 17.94$, $c = 14.49$ Å and $c/a = 0.807$.

Table 2. Structural results of vanadyl complexes of 2-hydroxyl-1-naphthalidene-3′-methylanil (HNMMA) and 2-hydroxyl-1-naphthalidene-4′-chloro-anil (HNPCA)

HNMMA[a]			HNPCA[b]		
"*d*" in A (observed)	"*d*" in A (calculated)	Plane *hkl*	"*d*" in A (observed)	"*d*" in A (calculated)	Plane *hkl*
9.80	9.85	111	11.80	11.72	101
9.08	9.11	200	6.60	6.67	211
7.62	7.63	002	5.75	5.75	220
6.44	6.44	220	5.39	5.42	300
4.92	4.92	222	5.03	5.05	113
4.06	4.07	420	4.88	4.91	311
3.82	3.82	004	4.23	4.23	004
2.94	2.93	404	4.06	4.06	400
			3.89	3.84	303
			3.82	3.83	330
			3.64	3.64	420
			3.40	3.40	224
			2.67	2.67	610

[a] Cell—Primitive tetragonal, $a = 18.22$, $c = 15.72$ Å and $c/a = 0.838$.
[b] Cell—Primitive tetragonal, $a = 16.26$, $c = 16.90$ Å and $c/a = 1.039$.

Table 3. Results of X-ray diffraction studies of vanadyl complexes

Complex	Lattice cell dimensions "*a*" in Å	"*c*" in Å	Volume (Å^3)
HNA	15.86	16.72	4205.7
HNOMA	17.94	14.49	4668.5
HNMMA	18.22	15.27	5069.2
HNPCA	16.26	16.90	4468.1