Asian Journal of Chemistry Vol. 16, Nos. 3–4 (2004), 1975–1977

NOTE

X-ray Powder Diffraction of the Complex of $S_3N_3Cl_3$ with Zn(II) Compound

SHUBHA RANI and S.P. SINGH JADON*
Department of Chemistry, S.V. College, Aligarh-202 001, India
E-mail: ad-sp@yahoo.com

On the basis of X-ray powder diffractometric studies, the complex of $S_3N_3Cl_3$ with ZnO, formulated as $(S_3N_3Cl_3)_3ZnO$ is found to be a tridentate co-ordinate complex with distorted tetrahedral geometrical structure.

Key Words: Complex, $S_3N_3Cl_3$, Zn, XRD.

In $S_3N_3Cl_3$ all the sulphur, nitrogen and chlorine atoms have lone electron pairs in spare to donate electrons and to form coordinated complex. The polar and non-polar complexes of $S_3N_3Cl_3$ with some metals have been reported[1-5]. In the present work, we describe the XRD of zinc(II) complex of $S_3N_3Cl_3$.

On the basis of the analytical data, molecular weight and its mass spectrum, the molecular composition of the complex of $S_3N_3Cl_3$ with ZnO has been reported[6] as $(S_3N_3Cl_3)_3ZnO$. The complex has also shown the paramagnetic character, which has been explained due to $4sp^3$ hybridisation in Zn^{2+} having distorted tetrahedral structure (*loc. cit.*). Further, to illustrate the structure of the complex, $(S_3N_3Cl_3)_3ZnO$, its XRD powder diffraction pattern, recorded in 2θ range from 2–70°, is analyzed and it is found that the peak at 25.7° having 100% intensity is for ZnO while the triplet in a 2θ range 28–31° is for $S_3N_3Cl_3$ ring which repeats in lower and higher 2θ region, indicating the presence of other $S_3N_3Cl_3$ rings as expressed by its molecular formula. From the XRD pattern, the Miller indices 'hkl' and interplanar distance 'd' are calculated. The observed values of 'd' are in close resemblance to the theoretical ones (Table-1). The gradation in intensity ratio suggests the crystallinity of the complex . The average values of axial distance $a_0 = 8.0946$ Å, $b_0 = 9.3427$ Å and $c_0 = 10.4508$ Å and axial angles $\alpha = +133.92°$, $\beta = 72.75°$ and $\gamma = 121.17°$ also express the distorted tetrahedral structure of $(S_3N_3Cl_3)_3ZnO$ as reported (*loc. cit.*), expounding the repulsion of $S_3N_3Cl_3$ rings from each other due to $4s^2$ electron pairs of Zn^{2+} ion, present as non-bonding and having different planes than the $(S_3N_3Cl_3)_3ZnO$ molecule packed in partial crystalline powder form. The axial ratios and axial angles (Table-2) determined from Table-1 for the molecular packing have six sets according to the atoms of $S_3N_3Cl_3$ ring, showing a triclinic packing of each $(S_3N_3Cl_3)_3Zn^{2+}$ tetrahedral in space geometry in powder form. Thus the distorted tetrahedral structure of $(S_3N_3Cl_3)_3ZnO$ is confirmed as reported (*loc. cit.*).

TABLE-1
X-RAY PATTERN OF COMPLEX $(S_3N_3Cl_3)_3ZnO$

S.No.	2θ	Hrl	d (Å)	Obs. (Theoretical)	I/I_0
1.	10.8	100	8.1926	(8.1847)	52.93
2.	17.1	110	5.1878	(5.1808)	60.77
3.	18.8	111	4.7208	(4.7160)	38.22
4.	20.5	200	4.3334	(4.3286)	83.80
5.	23.1	210	3.8507	(3.8470)	49.50
6.	25.7	211	3.4663	(3.4684)	100.00
7.	27.2	211	3.2791	(3.2757)	47.00
8.	28.7	211	3.1110	(3.1078)	59.79
9.	29.4	220	3.0387	(3.0354)	81.84
10.	30.0	220	2.9788	(2.9760)	82.33
11.	31.5	220	2.8405	(2.8376)	57.34
12.	32.2	300	2.7800	(2.7775)	44.10
13.	34.0	310	2.6365	(2.6345)	38.71
14.	35.8	310	2.5086	(2.5060)	31.85
15.	36.5	311	2.4622	(2.4596)	31.85
16.	37.8	222	2.3801	(2.3779)	27.93
17.	39.0	222	2.3095	(2.3075)	46.06
18.	41.8	320	2.1612	(2.1591)	44.10
19.	43.5	400	2.0807	(2.0786)	30.38
20.	46.2	410	1.9651	(1.9632)	29.40
21.	48.9	411	1.8630	(1.8610)	33.32
22.	51.7	331	1.7681	(1.7666)	27.44
23.	52.5	332	1.7433	(1.7415)	33.81
24.	56.0	422	1.6582	(1.6407)	30.87
25.	59.2	500	1.5608	(1.5594)	24.99
26.	61.0	511	1.5190	(1.5176)	24.50
27.	63.2	520	1.4715	(1.4700)	24.50

TABLE-2
AXIAL RATIOS AND AXIAL ANGLES OF THE COMPLEX $(S_3N_3Cl_3)_3ZnO$

S.No.	Axial ratios (Å)			Axial angles (°)		
	a_0	b_0	c_0	α	β	γ
1.	7.3452	5.9967	4.8957	95.84	125.44	138.47
2.	15.9998	14.8125	13.7132	111.89	120.79	127.32
3.	33.2862	32.3554	31.4507	117.01	120.12	122.77
4.	24.5256	25.4996	26.5144	123.78	120.20	116.02
5.	39.0181	37.6676	36.3639	116.42	120.17	123.42
6.	39.6110	27.5305	19.1344	65.02	140.95	154.03

Doubly distilled Aldrich and AnalaR grade chemicals were used. S_4N_4 was prepared by Goehring's method[7] by passing dry NH_3 gas into S_2Cl_2 dissolved in CCl_4 (1 : 10). $S_3N_3Cl_3$ (trithiazyl trichloride) was prepared by Nelson's method[8] by chlorination of S_4N_4 in CS_2 kept in ice-bath for 6–8 h. The $S_3N_3Cl_3$ and ZnO were dissolved in DMF separately and were mixed in equimolar quantities to reflux the mixture for about 24 h. The colour changed to pale yellow which indicates the formation of complex. The product was separated, washed successively with DMF, ethanol and ether , dried and stored in a vacuum desiccator over fused $CaCl_2$.

XRD pattern of the complex was recorded on ISO Deveflux 2002 X-ray powder diffractometer (German) using Cu filament as source of radiation ($\lambda = 1.5418$Å).

ACKNOWLEDGEMENTS

We express our thanks to Director, IIT Kanpur and to the Principal, S.V. College, Aligarh for providing instrumental and library facilities during the present work.

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(*Received*: 7 April 2004; *Accepted*: 10 June 2004) AJC-3486