Crystallographic Data on Ammonium Dioxovanadium(V) Bisoxalate Dihydrate by the X-Ray Powder Method

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Recently, we reported a new method for the preparation of ammonium dioxovanadium(V) bisoxalate dihydrate, $(NH_4)_3[VO_2(C_2O_4)_2]\cdot 2H_2O$, its physico-chemical properties and its structure.[1] X-Ray diffraction studies have now been carried out on the powdered crystals, and the crystal system and the unit cell parameters have been determined. The crystals of the complex were prepared in the manner reported earlier.[1] The crystals were then powdered to nearly a 300-mesh size and filled in a 0.5 mm. (inner diameter) Lindemann glass capillary. The X-ray diffraction pattern of the powdered substance was taken with a 114.6 mm. Philips Powder Camera using nickel-filtered CuK_α radiation (1.5418 Å) at 34 kV. and 18 mamp. at 25°C. The exposure time was six hours.

The complex crystallized from aqueous solutions on a microscopic slide at the laboratory temperature, when viewed under a polarising microscope, gave straight extinctions, and the optic axis figure showed the biaxial nature of the crystals. The crystal growth also clearly demonstrated that the lengths of the edges were unequal. These observations point to the possibility of an orthorhombic system for the compound. Microscopic examination further showed a prismatic type of crystals with domed faces. Five faces, (100), (010), (001), (110), and (101), appeared to be present. The powder pattern was indexed for an orthorhombic system using Hesse-Lipson's procedure;[2] the observed and calculated $\sin^2\theta$ values, together with the visually-assigned relative intensities of the lines, are given in Table I. The results show that the observed and computed $\sin^2\theta$ values for the orthorhombic system compare well. The values for the unit cell parameters are: $a=15.75$ Å, $b=11.09$ Å, and $c=8.015$ Å, as calculated from the observed $\sin^2\theta$ values of the first three lines, the volume of the unit cell being 1400 Å^3. The density of the crystals was found to be 1.665 g. ml^{-1} at 25°C, using carbon tetrachloride. Using this density, the

Table I. X-Ray powder data of $(NH_4)_3[VO_2(C_2O_4)_2]\cdot 2H_2O$

Line No.	$\sin^2\theta$ obs.	$\sin^2\theta$ calcd.	hkl	Relative intensity
1	0.00959	0.00961	2 0 0	75
2	0.01166	0.01165	1 0 1	100
3	0.01442	0.01445	2 1 0	35
4	0.01660	0.01650	1 1 1	35
5	0.01900	0.01937	0 2 0	25
6	0.02393	0.02373	2 1 1	40
7	0.02896	0.02899	2 2 0	40
8	0.03105	0.03093	1 2 1	55
9	0.03568	0.03573	3 1 1	15
10	0.03837	0.03845	4 0 0	15
11	0.04113	0.04100	3 2 0	15
12	0.04422	0.04424	1 1 2	25
13	0.05030	0.05025	3 2 1	55
14	0.05267	0.05254	4 1 1	15
15	0.05620	0.05636	0 2 2	65
16	0.06686	0.06707	4 2 1	55
17	0.07461	0.07417	5 1 1	45
18	0.07969	0.07990	1 4 0	20
19	0.08314	0.08299	1 3 2	25
20	0.08642	0.08651	6 0 0	15
21	0.09090	0.09047	1 1 3	25
22	0.09656	0.09636	2 4 1	20
23	0.1047 0.1047	0.1048 0.1050	3 0 3 1 2 3	10
24	0.1100	0.1097	3 1 3	20
25	0.1169	0.1169	1 4 2	15
26	0.1261	0.1265	4 1 3	20
27	0.1369 0.1369	0.1373 0.1364	0 7 0 2 2 3	45
28	0.1410	0.1411	4 2 3	20
29	0.1733	0.1732	8 2 0	20
30	0.1829	0.1824	3 4 3	20
31	0.1916	0.1913	4 1 4	20
32	0.2068	0.2067	1 5 3	15
33	0.2141	0.2139	2 5 3	15
34	0.2218	0.2221	4 6 1	15
35	0.2425 0.2425	0.2428 0.2422	4 5 3 3 7 3	15
36	0.2473	0.2471	3 4 4	15
37	0.2794	0.2792	3 6 3	15
38	0.2961	0.2961	5 1 5	15
39	0.3073	0.3069	3 7 4	15
40	0.3251	0.3249	1 6 4	15

$a=15.75$ Å, $b=11.09$ Å, $c=8.015$ Å;
$\alpha=\beta=\gamma=90°$

1) D. N. Sathyanarayana and C. C. Patel, This Bulletin, 37, 1736 (1964).
2) R. W. M. D'Eye and E. Wait, "X-ray Powder Photography," Butterworths Scientific Publications, London (1960), pp. 88—92.

number of formula units of the complex per unit cell was found to be $4.02 \simeq 4$.

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