ON THE STRUCTURE OF COMPLEX SILVER QUINOLINE PERCHLORATE

T. RATHO AND MRS. M. KRISHNASWAMY

REGIONAL ENGINEERING COLLEGE, ROURKELA

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ABSTRACT. The Debye Scherrer pattern of Silver Quinoline Per Chlorate has been studied and analysed. The unit cell dimensions are found to be $a = 15.15$ Å, $b = 11.91$ Å, 13.08 Å and $\beta = 97°.47'$. There are four molecules in the unit cell. The space group $P2_1/n$ C_{2h}^2 can be assigned to the crystal.

INTRODUCTION

Silver Quinoline perchlorate $Ag[C_9H_7N]_2ClO_4$ crystals are obtainable in a microcrystalline form, white in colour. The study of crystal structure of these complex ligants throws light on their diamagnetic behaviour. It is also possible to know something about the stretching force constants between the various groups of atoms in these molecules. As it is not possible to grow large crystals we have tried to analyse the crystal by the wellknown powder methods.

EXPERIMENTAL

Copper radiation from a Machlett A-2 X-ray diffraction tube was made monochromatic by using a Ni filter. The apparatus was running at 20 m.A. and 30 K.V. The finely powdered specimen was contained in a Lindemann glass capillary of 0.01 mm. wall thickness and of 0.5 mm diameter. The Debye Scherrer photograph was obtained in a Rigaku camera of 9 cm. diameter. The time of exposure was 4 hours. In the usual manner the interplanar distances were calculated accurately. Methods applicable to the cubic, tetragonal and hexagonal systems were applied and the experimental data did not fit into any one of them. Therefore the crystal does not belong to the higher symmetry class. The Lipson's method (1949), when tried, did not give a good number of constant differences. Therefore the most general method of Ito (1950) applicable to crystals of lower symmetry, namely, the monoclinic and the triclinic was applied.

The experimental data relating to the d values and the corresponding Q values ($1/d_{hkl}^2 = Q_{hkl}$) are also given in the table. All the six parameters were found by using the Ito's method and the powder pattern was indexed after Azaroff and Bourger (1958).

We have after the above method of Ito,

$$Q_{hkl} = h^2a^{*2}+k^2b^{*2}+l^2c^{*2}+2klb^*c^* \cos\alpha^*+2hlc^*a^* \cos\beta^*+2hka^*b^* \cos\gamma^*$$

where $\alpha^*, \beta^*, \gamma^*$ and a^*, b^* and c^* are the reciprocal angles and axes respectively.

The first two reflections of very weak intensity, after some trials, could be taken as Q_{100}, Q_{001} and the seventh reflection was taken as Q_{020}. Using the above formula it was possible to find out the reciprocal cell dimensions. They are

$$a^* = 0.06664$$

$$b^* = 0.08397$$

$$c^* = 0.07714$$

It then became necessary to examine the (*hko*), (*hol*) and (*okl*) reflections to select the reciprocal cell angles α^*, β^* and γ^*. If α^* and γ^* were taken to be 90 then (*hko*) and (*okl*) reflections could be detected. Therefore β^* was calculated after studying pairs of (*hol*) and ($h\bar{o}l$) reflections after the equation

$$\cos \beta^* = \frac{Q_{hol} - Q_{h\bar{o}l}}{4hla^*c^*}$$

The value of β^* so found was 82°13′. The direct cell dimensions are therefore obtained from these six reciprocal parameters as

$$a = 15.15 \text{ A.U.}$$

$$b = 11.91 \text{ A.U.}$$

$$c = 13.08 \text{ A.U.}$$

$$\alpha = 90^\circ$$

$$\beta = 97°47'$$

$$\gamma = 90^\circ$$

The above dimensions were found to be the reduced ones after applying the Buerger test (1958). The above data speaks about the monoclinic structure of the crystal. After indexing all the powder lines in the light of the above formula the following conditions for the appearance of the lines were found.

hkl—no condition

hol—no condition

oko—$k = 2n$

The possible space group, therefore, is $P2_1/m - C_{2h}^2$. The observed density is 1.383 gms/cc. and the calculated density comes out to be 1.323 gms/cc. This gives four molecules per unit cell.

TABLE

No. of lines	Intensity	d Å	$Q_{hkl} = 1/d^2$ observed	Q_{hkl} computed	Indices
1.	vw	14.99	0.00444	0.00444	100
2.	vw	12.97	0.00595	0.00595	001
3.	s	8.024	0.0155	0.0160	$11\bar{1}$
4.	m	7.463	0.0180	0.0178	200
				0.0188	111
5.	s	6.977	0.0205	0.0209	$20\bar{1}$
6.	vvw	6.511	0.0236	0.0238	002
7.	vvw	5.979	0.0280	0.0282	020
				0.0280	$21\bar{1}$
8.	s	5.537	0.0326	0.0326	120
				0.0325	$11\bar{2}$
9.	s	5.241	0.0364	0.0360	$20\bar{2}$
				0.0372	$12\bar{1}$
10.	m	5.021	0.0397	0.0399	300
11.	s	4.809	0.0432	0.0430	$21\bar{2}$
12.	vvw	4.603	0.0472	0.0470	310
				0.0471	202
13.	vw	4.314	0.0537	0.0536	003
				0.0538	$10\bar{3}$
				0.0537	$12\bar{2}$
				0.0541	212
14.	s	4.131	0.0586	0.0592	122
15.	m	3.918	0.0651	0.0612	$22\bar{2}$
16.	vw	3.649	0.0751	0.0754	131
				0.0753	222
17.	w	3.457	0.0837	0.0837	$40\bar{2}$
				0.0813	$23\bar{1}$
				0.0836	$32\bar{2}$
18.	m	3.318	0.0908	0.0904	123
				0.0912	$22\bar{3}$
				0.0908	$41\bar{2}$
19.	w	3.237	0.0954	0.0952	004

TABLE—contd.

No. of lines	Intensity	d Å	$Q_{hkl}=1/d^2$ observed	Q_{hkl} computed	Indices
20.	m	3.077	0.1057	0.1052	104
				0.1060	303
				0.1052	402
				0.1052	$33\bar{1}$
21.	w	2.975	0.1130	0.1128	040
				0.1123	114
				0.1135	331
				0.1131	313
				0.1130	412
22.	m	2.799	0.1276	0.1279	$5\bar{1}\bar{2}$
23.	vw	2.663	0.1410	0.1413	403
24	vvw	2.581	0.1501	0.1507	$51\bar{3}$
				0.1510	$41\bar{4}$
25.	vw	2.519	0.1576	0.1574	$60\bar{1}$
				0.1575	$13\bar{4}$
26.	m	2.436	0.1685	0.1678	$30\bar{5}$
				0.1687	134
				0.1682	$34\bar{2}$
27.	m	2.322	0.1854	0.1854	503
				0.1852	$15\bar{1}$
				0.1849	342
				0.1855	$51\bar{4}$
				0.1856	$62\bar{1}$
28.	w	2.214	0.2041	0.2047	433
29.	w	2.142	0.2180	0.2180	144
				0.2180	$35\bar{1}$
				0.2188	343
				0.2187	442
30.	m	2.087	0.2295	0.2298	053
				0.2291	$30\bar{6}$
31.	w	2.034	0.2418	0.2424	026
				0.2411	514
				0.2419	$53\bar{4}$
32.	w	1.962	0.2597	0.2598	061
				0.2600	$45\bar{2}$
				0.2589	$41\bar{6}$
33.	w	1.909	0.2746	0.2747	$26\bar{1}$
				0.2749	$61\bar{5}$

TABLE—contd.

No. of lines	Intensity	d Å	$Q_{hkl}=1/d^2$ observed	Q_{hkl} computed	Indices
34.	vw	1.844	0.2942	0.2938	360
				0.2946	505
				0.2948	$35\bar{4}$
35.	vw	1.791	0.3118	0 3121	236
				0 3111	435
				0.3116	$52\bar{6}$
36.	vw	1.729	0.3342	0.3335	263
				0 3348	$36\bar{3}$
				0.3337	$65\bar{1}$
				0.3344	$64\bar{4}$
37	w	1.676	0.3562	0.3556	$26\bar{4}$
				0.3567	255
38.	vw	1.628	0.3775	0.3779	264
				0 3777	561, 562
39.	vw	1.589	0 3958	0.3951	463
				0.3952	526
				0 3962	$54\bar{6}$
40.	w	1.559	0.4116	0 4112	$66\bar{1}$
41.	vw	1.513	0 4372	0 4367	$64\bar{6}$
42.	vw	1.469	0.4633	0.4634	365
				0.4632	645

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