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X-Ray Diffraction Studies on *cis*- and *trans*-[Co $en_2(CH_3COO)_2$]ClO_4

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In a previous paper[1] X-ray crystallographic data of *cis*- and *trans*-[Co en_2Cl_2]ClO_4 and of *cis*- and *trans*-[Co $en_2(C_6H_{11}COO)_2$]ClO_4 were reported. Extending now the studies to another couple of *cis*- and *trans*-isomers of Co(III) complexes having formula [Co en_2X_2]$^+$, *cis*- and *trans*- [Co $en_2(CH_3COO)_2$]ClO_4 are examined and characterized by the X-ray powder patterns by means of a Debye-Scherrer camera of diameter 114.8 mm with monochromatized Cu$K\alpha_1$ and Fe$K\alpha_1$ radiations. The salts are prepared as described by Linhard and

TABLE 1. X-RAY POWDER PATTERNS DATA OF *cis*-[Co $en_2(CH_3COO)_2$]ClO_4

Power diagram line	d_{hkl} (Å)	I/I_0 (visual inspection)	Q ($=1/d^2$) obsd.	Q computed	hkl
1	9.466	6	0.0111	0.0112	200
2	7.969	6	0.0157	0.0157	201
3	6.705	10	0.0222	0.0222	220
4	6.305	8	0.0252	0.0252	300
5	6.087	3	0.0270	0.0269	221
6	6.000	8	0.0278	0.0280	310
7	5.801	3	0.0297	0.0297	301
8	5.534	4	0.0327	0.0325	311
9	4.972	6	0.0405	0.0405	003
10	4.727	4	0.0448	0.0448	400
11	4.580	5	0.0477	0.0476	410
12	4.279	9	0.0546	0.0545	213
13	3.991	1	0.0628	0.0628	402
14	3.779	1	0.0700	0.0700	500
15	3.651	1	0.0750	0.0748	104
16	3.598	8	0.0772	0.0773	511
17	3.506	1	0.0814	0.0812	520
18	3.464	5	0.0833	0.0832	204
19	3.211	1	0.0970	0.0972	304
20	3.110	1	0.1034	0.1036	610
21	2.927	2	0.1167	0.1165	621
22	2.841	1	0.1239	0.1237	205
23	2.700	2	0.1372	0.1372	700
24	2.333	1	0.1837	0.1837	801
25	2.315	1	0.1866	0.1865	811
26	2.293	2	0.1902	0.1900	316
27	2.064	1	0.2347	0.2348	516
28	1.990	1	0.2525	0.2525	715 555
29	1.858	1	0.2897	0.2896	942

1) L. Campanella, This Bulletin, **41**, 2814 (1968).

TABLE 2. X-RAY POWDER PATTERNS DATA OF *trans*-[Co $en_2(CH_3COO)_2$]ClO_4

Power diagram line	d_{hkl} (Å)	I/I_0 (visual inspection)	Q ($=1/d^2$) obsd.	Q computed	hkl
1	8.421	8	0.0141	0.0141	201
2	7.619	8	0.0172	0.0172	211
3	7.189	10	0.0193	0.0192	202
4	6.675	3	0.0224	0.0223	212
5	6.141	8	0.0265	0.0265	221
6	5.540	9	0.0326	0.0327	311
7	5.136	3	0.0379	0.0378	312
8	4.694	8	0.0454	0.0456	105
9	4.290	3	0.0543	0.0544	411
10	4.209	1	0.0564	0.0564	402
11	3.966	10	0.0636	0.0637	421
12	3.692	6	0.0734	0.0735 0.0736	315 206
13	3.381	4	0.0875	0.0874	512
14	3.283	3	0.0928	0.0928	503
15	3.030	4	0.1089	0.1088	008
16	2.993	4	0.1116	0.1116	600
17	2.905	3	0.1185	0.1184	602
18	2.834	1	0.1245	0.1243	218
19	2.748	3	0.1324	0.1324	525
20	2.679	1	0.1393	0.1393	623
21	2.570	5	0.1514	0.1512	624
22	2.524	1	0.1570	0.1572	615
23	2.478	7	0.1629	0.1629	641
24	2.405	2	0.1729	0.1728	606
25	2.350	1	0.1811	0.1815	731
26	2.300	1	0.1890	0.1891	650
27	2.120	1	0.2225	0.2223	735
28	2.106	1	0.2255	0.2255	726
29	2.060	2	0.2356	0.2362	752
30	2.009	2	0.2478	0.2480	840
31	1.973	1	0.2569	0.2566	744
32	1.927	1	0.2693	0.2695	913
33	1.907	1	0.2750	0.2752	844
34	1.790	1	0.3121	0.3117	10,01
35	1.770	1	0.3192	0.3199	10,12
36	1.753	1	0.3254	0.3253	10,09
37	1.668	1	0.3594	0.3592	857

Stirn.[2]

Ito's method[3] is successful in indexing powder patterns. The use of an IBM computer is helpful in applying this method.

The experimental data for d_{hkl}, I/I_0 and Q of the two compounds are listed in Tables 1 and 2 and compared with the Q values computed on the basis of the indexing. Studies so far perfomed lead to the following conclusions:

a) *cis*-$[Co\,en_2(CH_3COO)_2]ClO_4$: The unit cell is tetragonal with dimensions $a=b=(18.898\pm0.007)$ Å, $c=(14.908\pm0.005)$Å. The measured density is 1.48 g cm^{-3}; the cell content is 12 formula units, giving a calculated density of 1.48 g cm^{-3}.

b) *trans*-$[Co\,en_2(CH_3COO)_2]ClO_4$: The unit cell is tetragonal with dimensions $a=b=(17.953\pm0.007)$ Å, $c=(24.213\pm0.008)$Å. The measured density is 1.34 g cm^{-3}; the cell content is 16 formula units, giving a calculated density of 1.35 g cm^{-3}.

2) V. M. Linhard and G. Stirn, *Z. Anorg. Allgem. Chem.*, **268**, 105 (1952).

3) T. Ito, "X-Ray Studies on Polymorphism," Maruzen Co., Ltd., Tokyo (1950), pp. 187—228.