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The Crystal Structures of Compounds Analogous to Magnus' Green Salt

BY M. ELISABETH CRADWICK, D. HALL* AND R. K. PHILLIPS

Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada

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Tetra(methylamine)platinum(II) chloroplatinate(II) and tetra(ethylamine)platinum(II) bromoplatinate(II) are confirmed as being isostructural with Magnus' green salt, $\text{Pt}(\text{NH}_3)_4\text{PtCl}_4$. The dimensions of the tetragonal cells are $a=10.32$, $c=6.58$, and $a=12.27$, $c=6.71$ Å respectively; the space group is $P4/mnc$. Tetra(ethylamine)platinum(II) chloroplatinate(II) forms triclinic crystals, space group $P\bar{1}$, with $a=7.56$, $b=9.78$, $c=7.24$ Å, $\alpha=98.3^\circ$, $\beta=113.1^\circ$, $\gamma=103.4^\circ$; it has a similar structure in that cation and anion alternate in chains, but the cation is non-planar and orbital overlap between adjacent platinum atoms is inhibited. The differing optical properties of this compound are thus explained. The variation in structure is ascribed to packing effects.

The crystal structure of Magnus' green salt, $\text{Pt}(\text{NH}_3)_4\text{PtCl}_4$ and hereafter MGS, comprises chains of square-planar cations and anions stacked directly over one another, with a Pt-Pt separation of 3.26 Å (Atoji, Richardson & Rundle, 1957). Both the green colour and the dichroism exhibited by these crystals are distinct from those characteristic of the constituent ions (Yamada, 1951), and have been attributed to weak metal-metal bonding. The compound $\text{Pt}(\text{NH}_2\text{CH}_3)_4\text{PtCl}_4$ is also green and similar in dichroism to MGS, but the analogues with higher amines are pink, and show only the dichroism of the chloroplatinate(II) ion. The methylamine compound is similar in powder diffraction pattern to MGS, whereas the ethylamine compound is not (Miller, 1961), and it has been assumed that steric effects inhibit the chain structure and thus prevent the metal-metal bond interaction. Subsequently it has been shown (Yamada, 1962, 1965) that crystals of $\text{Pt}(\text{amine})_4\text{PtBr}_4$ and $\text{Pt}(\text{amine})_4\text{PtI}_4$ are all abnormally coloured and dichroic, whether amine be ammonia, methylamine, ethylamine or various higher amines. This clearly indicated that the difference in structure of the higher amine chloroplatinates(II) is not caused by intra-chain steric interference, and Yamada (1965) suggested that it may be due to a weakening influence of the chlorine ligand on the metal-metal bond. This view is not supported

by reflectance spectra studies (Miller, 1965), nor indeed by Yamada's own single-crystal spectra, from which it is apparent that the band shifts are less and not greater in bromoplatinates(II) *vis-à-vis* chloroplatinates(II). Further, the compounds $\text{Pt}(\text{NH}_3)_4\text{PtCl}_4$, $\text{Pt}(\text{NH}_3)_4\text{PdCl}_4$, $\text{Pd}(\text{NH}_3)_4\text{PtCl}_4$ and $\text{Pd}(\text{NH}_3)_4\text{PdCl}_4$ show varying spectral shifts, yet all have the MGS structure, with the same metal-metal separation (Miller, 1961, 1965). It appears that chemical variations affect the optical phenomena far more than the crystal structure, and the difference in behaviour of tetra(ethylamine)platinum(II) chloroplatinate(II) remains anomalous. We have examined crystals of several of these compounds in an attempt to elucidate this problem.

In each case, compounds were prepared by mixing solutions containing the constituent ions.

Tetra(methylamine)platinum(II) chloroplatinate(II)

Green tetragonal needles gave $a=10.32 \pm 0.02$, $c=6.58 \pm 0.02$ Å, $\rho_{\text{obs}}=3.12$, $\rho_{\text{calc}}=3.11$ g.cm⁻³ for two molecules of $\text{Pt}(\text{NH}_2\text{CH}_3)_4\text{PtCl}_4$ per unit cell. Reflexions were systematically absent for $0kl$ with $k+l$ odd, and for hhl with l odd, and were systematically weak for hkl either with l odd or with l even, $h+k$ odd. These observations give the space group as either $P4/mnc$ or $P4nc$, and indicate that the platinum atoms occupy the positions of twofold multiplicity $0,0,0; \frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ and $0,0, \frac{1}{2}; \frac{1}{2}, \frac{1}{2}, 0$. The structure was thus confirmed as essentially similar to that of MGS, for which the pattern

* Present address: Chemistry Department, University of Auckland, Private Bag, Auckland, New Zealand.

of absent and weak reflexions is the same, and for which $a=9.03$, $c=6.49$ Å.

Intensities of the layers $hk0$ – $hk2$ were measured visually from Weissenberg photographs taken, using Cu $K\alpha$ radiation and a square needle of side 0.015 mm. No absorption corrections were applied. The chlorine atom was located from a platinum-phased density synthesis, and after refinement the carbon and nitrogen atoms were clearly visible on a difference synthesis. The centrosymmetric space group $P4/mnc$ was assumed throughout and nothing in the analysis, or the final structure, gave cause to doubt it. Atom coordinates, isotropic temperature factors and individual layer scale factors were refined by least-squares to an R index of 0.085. Scattering factors were as in *International Tables for X-ray Crystallography* (1962), with a real dispersion correction of $5.0e^-$ for platinum. Atom coordinates are listed in Table 1 and observed and calculated structure factors in Table 2. Bond lengths and angles are as follows:

Pt–Cl, 2.29; Pt–N, 1.92; N–C, 1.7 Å; Pt–N–C, 123°. The angles Cl–Pt–Cl and N–Pt–N are 90° by symmetry. The structure may be described as chains of planar, parallel, $\text{Pt}(\text{NH}_2\text{CH}_3)_2^+$ and PtCl_4^- ions, stacked directly over one another with Pt–Pt separation of 3.29 Å.

Table 1. Atom coordinates for $\text{Pt}(\text{CH}_3\text{NH}_2)_4\text{PtCl}_4$

Standard deviations are 0.014 Å in Cl coordinates, 0.07 Å in N and C coordinates.

	x/a	y/b	z/c
Pt(1)	0	0	0
Pt(2)	$\frac{1}{2}$	$\frac{1}{2}$	0
Cl	0.1003	0.1981	0
N	0.398	0.344	0
C	0.461	0.195	0

Table 2. Observed and calculated structure amplitudes ($\times 10$) for tetra(methylamine)platinum(II) chloroplatinate(II)

h	k	F_o	F_c	h	k	F_o	F_c	h	k	F_o	F_c	h	k	F_o	F_c
$hk0$				hkl				$hk2$							
0	4	1802	2046	3	6	206	205	0	3	406	374	0	2	2205	2297
0	6	1997	1988	3	7	1903	1943	0	4	91	100	0	4	2105	1934
0	8	1535	1512	3	8	414	348	0	6	189	169	0	6	1999	1868
0	10	1757	1827	3	9	1363	1275	1	3	512	531	0	8	1465	1445
0	12	972	1031	3	11	1046	1020	1	4	57	81	0	10	1837	1793
1	3	1713	2111	3	12	177	213	1	5	97	99	0	12	1007	995
1	4	317	333	4	4	2157	2123	1	7	275	245	1	2	170	150
1	5	237	2283	4	6	1785	1698	2	4	370	356	1	4	2056	1972
1	6	494	474	4	8	1960	1720	2	5	241	295	1	4	309	303
1	7	1795	1742	4	10	1407	1221	2	6	296	270	1	5	2267	2141
1	9	1566	1656	4	11	193	186	3	7	95	74	1	6	447	429
1	11	1448	1378	5	5	1362	1180	3	9	163	164	1	7	1773	1643
2	2	1856	2128	5	7	1846	1722	3	10	107	122	1	9	1629	1592
2	3	536	493	5	9	1724	1550	3	11	124	131	1	11	1403	1319
2	4	2447	2693	5	11	1085	1211	4	5	186	212	2	2	2022	1994
2	6	2202	2350	6	6	1610	1426	4	6	145	98	2	3	554	425
2	7	1719	1737	6	11	1795	1598	4	8	167	153	2	5	1572	1435
2	8	1476	1498	6	9	250	240	4	12	94	106	2	6	2021	2198
2	9	189	175	6	10	1208	1123	5	6	125	161	2	7	215	210
2	10	1393	1337	6	11	244	212	5	8	166	163	2	8	1572	1435
2	12	1010	1053	7	7	1746	1518	6	8	166	163	2	10	1350	1281
3	3	2509	2948	7	8	225	244	7	9	119	104	2	12	1041	1017
3	4	321	278	7	9	1214	1094	7	3	282	2667	8	8	1090	1105
3	5	2197	2349	8	8	1519	1148	8	4	265	248	8	10	752	935
												3	5	2081	2189
												9	9	817	1051

Tetra(ethylamine)platinum(II) bromoplatinate(II)

Green tetragonal needles gave $a=12.27 \pm 0.02$, $c=6.71 \pm 0.02$ Å, $\rho_{\text{calc}}=2.93$, two molecules of $\text{Pt}(\text{NH}_2\text{C}_2\text{H}_5)_4\text{PtBr}_4$ per unit cell. The systematic absences and pattern of systematically weaker reflexions were exactly as for MGS and $\text{Pt}(\text{NH}_2\text{CH}_3)_4\text{PtCl}_4$, and

the crystals obviously have the same structure, with a Pt–Pt separation of 3.35 Å.

Tetra(ethylamine)platinum(II) chloroplatinate(II)

Fine pink needles elongated along c were analysed chemically (found: C, 13.8; H, 4.2; N, 8.17; calculated: C, 13.5; H, 3.9; N, 7.9). The crystals were not only very small, but were multiple and deformed. After examining many crystals from different preparations and crystallizations, only one single-crystal was found, a fragment of approximate dimensions $0.035 \times 0.035 \times 0.085$ mm. This was mounted about the a axis of the B -centred cell as in Table 3, and the layers $0kl$ – $3kl$ collected by Weissenberg photography, using Cu $K\alpha$ radiation. The crystal was lost before its uniqueness was appreciated and as no other even approaching suitability was found, the above represents the total data which could be collected. Intensities were measured visually, and no correction for absorption was attempted.

Table 3. Unit-cell dimensions

B -centred cell	Reduced cell
$a = 13.90$ Å	7.56 Å
$b = 9.78$	9.78
$c = 7.24$	7.24
$\alpha = 98.3^\circ$	98.3°
$\beta = 90.1$	113.1
$\gamma = 108.3$	103.4
$V = 923.2$ Å ³	461.6 Å ³
2 molecules per cell	1 molecule per cell

The coordinates in Table 4 may be transformed to those of the reduced cell by

$$\begin{pmatrix} 2 & 0 & 0 \\ 0 & 1 & 0 \\ 1 & 0 & 1 \end{pmatrix}.$$

This crystal was triclinic and was initially described in terms of a primitive cell, identical with the B -centred cell in Table 3, but with a and c halved. Long-exposure rotation photographs indicated, however, that a should be doubled, and data for the true weak 1st layer were collected. The resulting cell is B -centred, but as the layers of data had to be scaled separately it was convenient to retain this cell, and all results are given with respect to it. The reduced cell parameters are given in Table 3.

The apparent subcell corresponding to the strong reflexions contained one platinum atom only, which was assumed to be at 0,0,0. The isotropic temperature factor and the scale factors for the various layers were refined by least-squares, and a difference synthesis then calculated. The platinum atom was surrounded by two sets of peaks in square-planar arrays, rotated by 45° with respect to each other, which were the chlorine and nitrogen atoms, apparently superimposed. The carbon atoms were located, and on reference to the B -centred cell and assuming one complex ion at 0,0,0 and the other at $\frac{1}{2}, 0, 0$, refinement proceeded to an R index of 0.078. The R index for the 36 weak $1kl$ reflexions was

Table 4. Atom coordinates, with respect to the *B*-centred cell

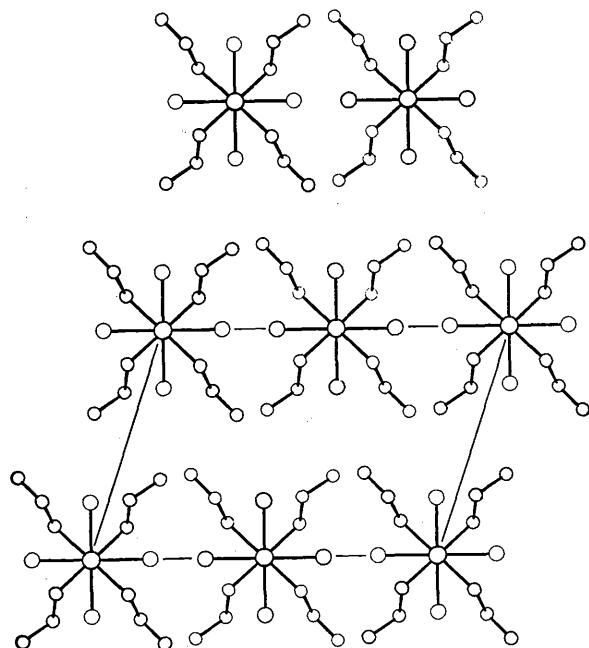
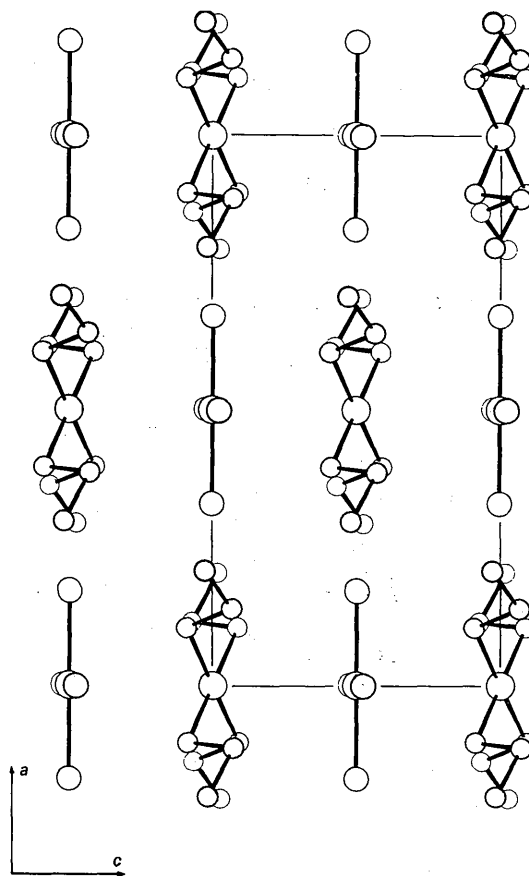
	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i>
Pt(1)	0	0	0	1.6 (1) Å ²
Pt(2)	0.5	0	0	0.9 (1)
Cl(1)	0.555 (3)	0.253 (2)	0.072 (2)	3.4 (3)
Cl(2)	0.674 (3)	0.001 (1)	0.002 (2)	3.8 (3)
N(1)	0.136 (8)	0.152 (5)	-0.063 (7)	4.0 (1)
N(2)	0.072 (8)	-0.146 (5)	0.056 (6)	4.0 (1)
C(1)	0.193 (11)	0.264 (7)	0.119 (10)	5.0 (2)
C(2)	0.067 (11)	-0.266 (7)	-0.119 (9)	5.0 (2)
C(3)	0.288 (17)	0.366 (9)	0.048 (12)	6.0 (2)
C(4)	0.125 (16)	-0.362 (9)	-0.054 (11)	6.0 (2)

0.116. Scattering factors were as in *International Tables for X-ray Crystallography* (1962), with a real dispersion correction for platinum. Isotropic temperature factors were assumed for all atoms, and a final difference synthesis indicated major residual error associated with the platinum atoms, but in view of the uncertainties in the data no corrective action was taken. The atom coordinates are listed in Table 4, bond lengths and angles in Table 5, and observed and calculated structure factors in Table 6. Projections of the structure are shown in Figs. 1 and 2.

The crystal structure of tetra(methylamine)platinum(II) chloroplatinate(II) may be described as chains parallel to *c*, of alternate cations and anions, the mean planes of which are very nearly perpendicular to *c*. It differs from that of MGS in that the tetra(ethylamine)platinum cation is not a truly planar unit, and as a consequence the Pt-Pt separation in the present structure is somewhat longer, at 3.62 Å. More significantly perhaps, although the folding of the amine ligands is such that the overall mean plane of the cation is nearly parallel to that of the PtCl₂⁻ anion, the plane of the platinum-nitrogen coordination square is inclined by 29° to that of the anion. The overlap of the axial orbitals

Table 5. Bond lengths and angles

Pt(2)-Cl(1)	2.33 (1) Å	Cl(1)-Pt(2)-Cl(2)	90 (1)°
Pt(2)-Cl(2)	2.41 (4)	N(1)-Pt(1)-N(2)	93 (3)
Pt(1)-N(1)	2.11 (8)	Pt(1)-N(1)-C(1)	112 (5)
Pt(1)-N(2)	2.06 (8)	Pt(1)-N(2)-C(2)	112 (5)
N(1)-C(1)	1.61 (9)	N(1)-C(1)-C(3)	105 (6)
N(2)-C(2)	1.58 (9)	N(2)-C(2)-C(4)	105 (7)
C(1)-C(3)	1.53 (20)		
C(2)-C(4)	1.53 (20)		

Fig. 1. Projection of the structure of Pt(NH₂-C₂H₅)₄PtCl₄ on (001) of the *B*-centred cell.Fig. 2. Projection of the structure of Pt(NH₂-C₂H₅)₄PtCl₄ on (010) of the *B*-centred cell.

tals on the platinum atoms is then greatly reduced, and the interaction which gives rise to abnormal light absorption is prevented.

The bond lengths and angles listed in Table 5 are all normal, within error. The six closest inter-ion approaches are between nitrogen and chlorine, and range from 3.27 to 3.48 Å. The shorter approaches are less than those in MGS (minimum 3.43 Å), but are still long for nitrogen-chlorine hydrogen bonds (Nakamoto, Margoshes & Rundl, 1955). The infrared spectrum shows four bands between 3135 and 3240 cm⁻¹ in the N-H stretching region, these frequencies being normal for coordinated amines (Svatos, Curran & Quagliano, 1955; Penland, Mizushima, Curran & Quagliano, 1957), and similar to those observed for MGS, *viz* 3182 and 3277 cm⁻¹ (Atoji, Richardson & Rundl, 1957). There is then no reason to suppose that such hydrogen

bonding has any major influence on the structure, although it may in part be responsible for the specific conformation adopted by the ethylamine ligands.

It is apparent from Figs. 1 and 2 that a consequence of the folding of ethylamine ligands is that the cation-anion chain maintains a more or less constant diameter, and thus the chains stack efficiently without mutual interference. If the cation were planar its greater diameter might prevent this, and as intra-chain repulsions must make it unlikely that cation and anion could adopt any other conformation than respective rotation of 45°, any such inter-chain repulsion may be sufficient to destabilize the structure. If the anion were larger, however, as in the bromo- and iodo-platinates(II), a somewhat larger cation could be tolerated, as is observed. It is then possible to rationalize the different structure observed for tetra(ethylamine)platinum(II)

Table 6. Observed and calculated structure factors ($\times 10$) for tetra(ethylamine)platinum(II) chloroplatinate(II)

K L PO PC				K L PO PC				K L PO PC				K L PO PC				K L PO PC				K L PO PC															
H = 0				H = 0				H = 0				H = 0				H = 0				H = 0															
0	4	1164	1059	5	4	670	704	-10	4	524	524	4	-6	609	603	-9	-4	515	427	1	-6	793	737	4	-6	609	603	-9	-4	515	427	1	-6	691	635
0	8	699	673	-5	4	998	1092	-11	0	652	503	-4	3	131	-118	-2	-6	757	714	-4	-6	538	552	7	2	749	690	-9	-6	389	449	1	8	487	492
0	8	533	492	5	6	715	716	-11	2	533	473	-4	-3	232	-241	-2	-6	744	736	-4	-6	581	554	7	-2	700	691	10	0	463	433	1	-8	547	523
1	4	1055	965	-5	6	643	737	-11	4	407	426	-5	1	152	-164	-2	-6	737	666	-4	-8	356	423	-7	2	610	675	-10	0	529	481	-1	-8	841	431
-1	4	1370	1253	-5	8	466	486					-5	-1	140	-145	-2	8	478	456	-4	-8	420	454	-7	-2	773	753	10	2	474	425	2	0	1104	1162
1	6	778	767	-6	0	934	908					-5	3	169	-147	-2	-8	534	541	-4	-8	394	385	7	4	649	555	-10	2	397	428	2	2	1021	1037
-1	4	782	792	6	2	919	855	0	3	83	-63					-2	-8	501	497	-5	0	701	705	7	-4	535	570	-10	2	532	516	2	-2	1129	1083
1	8	652	548	-6	2	877	974	0	-3	231	-193	0	2	1144	1145	-2	-8	501	497	-5	0	899	1038	-7	4	504	597	-10	-2	508	643	-2	2	1165	1099
-1	8	582	527	6	4	769	727	1	-1	195	-158	0	4	871	800	3	0	384	1123	-7	-4	794	647	-10	-4	382	419	-2	-2	918	796				
2	2	1302	1386	-6	4	800	943	-1	1	144	-137	0	-4	1006	895	3	-2	983	1006	5	-2	145	818	7	6	446	384	-10	4	484	515	2	4	850	864
-2	2	1067	999	-6	6	734	543	1	3	225	146	0	6	636	582	-1	2	734	723	-5	-2	773	783	-7	6	457	529	-13	6	391	450	-2	4	1047	1050
-2	4	9102	1043	-6	8	636	742	1	-3	293	-276	0	-6	669	617	-3	-2	968	950	5	4	565	572	-7	-6	596	480	11	0	518	429	-2	-4	795	641
-2	4	934	965	-6	8	403	504	-1	3	290	-270	0	8	451	450	4	8	836	835	5	-4	278	810	-7	8	381	493	-11	0	435	434	2	6	700	621
-2	4	824	842	-7	0	995	934	-1	-3	91	64	0	-8	444	438	3	-4	694	674	-5	-4	831	907	8	0	695	636	11	-2	411	407	2	-6	630	590
2	8	636	546	7	2	1034	904	1	-5	251	-246	-1	-2	1132	1121	3	-4	694	674	-5	-4	831	907	8	0	695	636	11	-2	411	407	2	-6	630	590
-2	8	632	569	-7	2	717	765	-1	5	203	-231	1	4	852	786	3	-4	798	742	-5	-4	592	582	8	0	763	762	-11	2	390	424	-2	6	843	780
-3	2	1068	1049	7	4	772	688	2	-1	114	-37	1	-4	1104	1021	3	-6	490	922	5	6	524	500	8	2	627	555	-11	2	483	426	-2	-6	604	575
-3	4	1155	1066	-7	4	547	640	-2	1	164	-33	-1	4	975	902	3	6	606	567	5	-6	668	660	8	-2	700	657	-11	4	325	412	2	8	395	404
-3	4	926	907	-7	6	621	476	2	-3	130	-101	-1	-4	747	804	3	-6	546	533	-5	6	536	629	8	2	672	696	-12	0	477	431	-2	-8	493	479
3	6	718	709	-7	6	413	569	-2	3	238	-210	1	6	674	649	-3	-6	698	677	-5	8	381	425	8	4	487	426	1	-4	-2	518	497			
-3	6	749	788	-7	8	437	461	2	-5	158	-125	1	-6	833	744	3	0	415	387	6	0	734	713	8	-4	540	576	3	2	822	872	0	10	1003	1116
3	8	545	469	8	2	890	749	-2	5	225	-217	-1	-6	635	613	3	5	376	461	-6	0	699	710	-8	4	516	560	-12	2	1219	1143	3	2	1015	1044
-3	8	620	540	-8	2	936	836	1	-1	236	-210	-1	-6	637	607	3	5	523	538	6	2	776	727	-8	-4	639	531	0	4	724	666	3	-2	1025	1003
-4	0	1158	1369	8	4	712	555	3	-1	199	-213	1	8	509	498	-3	-8	476	452	-6	-2	629	652	8	-6	393	451	0	4	1128	1049	-3	2	1040	1000
4	2	922	1024	-8	4	527	629	-2	1	145	-147	1	8	482	493	-3	-8	476	452	-6	-2	629	652	8	-6	393	451	0	4	1128	1049				
-4	2	1111	1307	-8	6	438	476	3	-1	149	-159	-1	8	423	427	4	0	967	1092	-6	-2	679	828	-8	6	379	446	0	6	582	564				
-4	4	758	753	-9	0	580	615	3	3	227	-240	-1	-8	466	472	4	0	1203	1148	-6	-2	654	638	-8	-6	430	373	0	6	815	754				
-4	4	800	911	9	2	628	530	3	-3	84	-55	2	2	830	798	4	2	800	824	6	4	700	659	9	0	561	498	0	8	461	480	3	-4	808	776
4	6	628	509	-9	2	708	698	-3	-3	171	-175	-2	2	1132	1131	4	-2	836	999	-6	4	699	793	-9	0	703	620	0	-8	513	471				
-4	6	683	717	9	4	617	464	4	1	248	-233	-2	-2	1203	925	4	-2	894	1092	-6	-4	638	596	-9	0	703	620	0	-8	513	471				
-4	6	556	441	8	2	890	661	4	-1	240	-269	2	4	998	877	4	4	619	592	6	6	528	481	-3	2	636	640	1	-2	1072	1027				
-4	8	508	528	-9	4	504	482	-1	4	290	-315	2	-4	908	808	4	4	896	877	6	-6	518	554	-3	-2	623	528	-1	2	1066	1173				
-5	0	1285	1113	-10	0	518	484	-1	1	283	-327	-2	4	1204	915	-1	-4	685	701	-6	6	620	653	3	4	468	396	1	4	761	735				
-5	2	881	835	10	2	633	490	4	3	147	-148	-2	-4	836	801	-4	-4	801	818	-6	-6	587	514	7	-4	531	531	1	-4	964	836				
-5	2	1100	1316	-10	2	550	497	4	-3	172	-153	2	6	132	683	4	-6	472	463	-6	8	422	459	-9	4	532	557	1	6	672	659				

chloroplatinate(II) in terms of packing arguments alone, and the optical phenomena follow as a consequence.

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Structure Cristalline à -140, 20, 120 °C et Dilatation Thermique de l'Azélamide

PAR MICHEL HOSPITAL

Laboratoire de Cristallographie et de Physique Cristalline associé au C.N.R.S., Faculté des Sciences de Bordeaux, 351 cours de la Libération, 33 Talence, France

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The crystal structure of azelamide has been determined at three temperatures: -140, 20 and 120 °C. The intensities were measured with a two-circle semi-automated diffractometer with Cu K α radiation. The space group is *C2/c* with 4 molecules in the unit cell. The respective *R* values are: *R* = 0.106 at -140; *R* = 0.077 at 20; *R* = 0.141 at 120 °C. The study of the molecular conformation, crystal cohesion and atomic thermal motion leads to an explanation of the mechanism of thermal expansion and Poisson contraction which is a characteristic temperature effect in only two dimensions.

Dans le cadre d'une étude qui a pour but d'expliquer les caractères de la dilatation thermique, à partir des données de structure cristallographique des cristaux organiques, nous nous sommes intéressés au cristal d'azélamide. La structure moléculaire a été déterminée pour les températures: -140, 20 et 120 °C et la dilatation thermique a été mesurée de -140 à 100 °C.

Les cristaux utilisables pour la technique de diffraction des rayons X sont obtenus par très lente évaporation d'une solution dans un mélange d'acide formique et d'acide acétique en quantité égale. Une plaquette est clivée pour obtenir une baguette allongée suivant l'axe *c* (section du cristal: 0,15 × 0,14 mm²).

Les paramètres de la maille sont mesurés sur des clichés obtenus à la chambre de Weissenberg et calculés

par une méthode de moindres carrés après indexation des taches:

$$\begin{aligned} a &= 5,782 \pm 0,006 \text{ \AA} \\ b &= 8,641 \pm 0,009 \\ c &= 27,687 \pm 0,015 \\ \beta &= 131^\circ 45' \pm 20' \end{aligned}$$

Le groupe spatial est *C2/c*.

La densité mesurée est 1,20; la densité calculée est 1,196 g.cm⁻³ avec 4 molécules par maille.

Des mesures de paramètres de maille faites tous les 40 °C à partir de -140 °C ont permis d'établir les variations de ces paramètres en fonction de la température.

Nous fournissons ici les résultats sous forme de courbes (Fig. 1) pour *a*, *b*, *c*, β en fonction de la température

Tableau 1. Coordonnées atomiques et leurs déviations standard

	<i>x</i>	$\sigma(x)$	<i>y</i>	$\sigma(y)$	<i>z</i>	$\sigma(z)$
C(1)	0,0000	—	0,1098	0,0006	0,2500	—
	0,0000	—	0,1181	0,0004	0,2500	—
	0,0000	—	0,1347	0,0014	0,2500	—
C(2)	0,0236	0,0008	0,2069	0,0004	0,2071	0,0002
	0,0216	0,0006	0,2120	0,0002	0,2067	0,0001
	0,0027	0,0022	0,2269	0,0008	0,2069	0,0006