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Crystal and Molecular Structure of Trichloro(pent-4-enylammonium)platinum(II) in Two Different Crystalline Forms

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Trichloro(pent-4-enylammonium)platinum(II), [PtCl₃($C_5H_{12}N$)], crystallizes in two monoclinic forms: an orange one, a = 10.504, b = 10.627, c = 9.608 Å, $\beta = 106.9^{\circ}$, and a yellow one, a = 6.435, b = 9.372, c = 16.737 Å, $\beta = 100.6^{\circ}$. Both forms belong to the space group P_{2_1}/c . The two structures have been determined from three-dimensional X-ray data collected at room temperature by counter methods. They have been refined by least-squares techniques to conventional R values of 6.0 and 6.4% for the orange and the yellow forms respectively. In both forms the [PtCl₃($C_5H_{12}N$)] zwitterion is a π -complex and the platinum atom is square coordinated by the three chlorine atoms and the double bond of the pent-4-enylammonium cation. The dimensions and the geometry of the molecule in the two structures are very similar; the only significant difference concerns the conformation of part of the olefinic chain. The two structures are stabilized in the solid state by intermolecular N-H···Cl hydrogen bonds.

As part of a programme to investigate the stability of π -complexes of platinum(II) with functionally substituted olefins, we have carried out structure determinations of several typical examples (Spagna, Venanzi & Zambonelli, 1970*a* and *b*; Colapietro & Zambonelli, 1971; Spagna & Zambonelli, 1971).

We now report the study of the platinum-olefin complex trichloro (pent-4-enylammonium) platinum (II), formed in acid solution by the action of pent-4enylamine on $[PtCl_4]^{2-}$ (Claridge & Venanzi, 1964). Since this complex crystallizes in two different forms, an orange and a yellow one, we have carried out an X-ray analysis of the complex in both forms in order to ascertain possible structural differences.

Experimental

(with techical assistance from F. D'APRILE and A. TAMBURRINI)

Orange and yellow crystals of trichloro(pent-4-enylammonium)platinum(II) were obtained by the procedure described by Claridge & Venanzi (1964). Both forms are stable in air and to X-rays.

Crystal data

A summary of the crystal data for each form is given in Table 1. Approximate unit-cell parameters, Laue symmetry, and systematic extinctions were obtained from zero and upper level Weissenberg photographs. Precise lattice constants and their estimated standard deviations were determined from a leastsquares refinement of the setting angles of a number of X-ray reflections centered on a Siemens AED automatic diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) at 22 ± 2 °C. The densities were measured by flotation in a mixture of chloroform and bromoform.

 Table 1. Crystal data for trichloro(pent-4-enylammoinium)platinum(II) in the two crystalline forms

The cell parameters of the orange and yellow forms were refined using 14 and 19 observed 2θ values respectively. Numbers in parentheses are standard deviations in the least significant digit.

-	Orange crystals	Yellow crystals
Formula	$[PtCl_3(C_5H_{12}N)]$	$[PtCl_3(C_5H_{12}N)]$
M.W.	387.61	387.61
Space group	$P2_1/c$	$P2_1/c$
a	10·504 (7) Å	6·435 (5) Å
Ь	10.627 (10)	9.372 (11)
с	9.608 (8)	16.737 (13)
β	106·9 (1)°	100-6 (1)°
Volume	1026-5 Å ³	992·1 Å ³
Ζ	4	4
Dc	2.51 g.cm ⁻³	2.64 g.cm ⁻³
D_m	2.53 (1) g.cm ⁻³	2.67 (2) g.cm ⁻³
μ(Mo <i>K</i> α)	150.9 cm ⁻¹	158.7 cm ⁻¹
F(000)	705.6	705.6
No. of independent		
reflexions	1245	799

Intensity measurements

Mo $K\alpha$ radiation filtered through 0.05 mm Zr foil was used. The crystals ($0.06 \times 0.15 \times 0.25$ mm for the orange and $0.05 \times 0.10 \times 0.12$ mm for the yellow form) were mounted on a Siemens AED automatic single crystal diffractometer equipped with a scintillation counter and pulse-height analyser. For both crystals the unique axis b (the longer dimension of the crystal) was coincident with the polar φ axis of the goniostat. The ω -scan technique was employed, and the procedure was similar to that previously described (Cerrini, Colapietro, Spagna & Zambonelli, 1971). A fixed symmetrical scan range of 0.90° from the computed Mo K α peak was used. At each end of the scan stationarycrystal stationary-counter background counts were

taken for a time equal to the scan time. Intensity data were collected for the independent reflexions up to θ (Mo $K\alpha$) $\leq 28^{\circ}$ for the orange crystal, and to θ (Mo $K\alpha$) $\leq 25^{\circ}$ for the yellow crystal. Past these points very few intensities were above background. For both crystals, three standard reflexions were measured at regular intervals to monitor crystal stability and crystal orientation. Their intensities remained essentially constant throughout the runs. The data were processed as previously described (Cerrini et al., 1971) (the value of p = 0.06 was chosen for both sets of data). 1245 (orange form) and 799 (yellow form) independent reflexions, with $F_o^2 > 3\sigma(F_o^2)$, were used for the analyses. The shape anisotropy of the crystals measured at $\chi = 90^{\circ}$ indicated a 58 and 60 % variation in intensities over the φ range used in the collection of data, for the orange and yellow crystal respectively. In order to correct for the shape anisotropy, the variation in intensity of conveniently intense axial reflexions (020 and 060 for the orange crystal and 020 for the yellow crystal) were measured as a function of φ . For this correction the method described in the literature (Furnas, 1957; North, Phillips & Scott Matthews, 1968) was used, with a procedure similar to that previously described (Spagna & Zambonelli, 1971). No extinction correction was applied.

Determination and refinement of the structures

Both structures were solved by the usual combination of Patterson and Fourier methods.

Orange form

The isotropic full-matrix least-squares refinement lowered the *R* index from 0.120 to 0.073. Refinement was continued allowing all atoms to vibrate anisotropically; convergence was reached at R=0.061. At this stage in no region of the difference Fourier synthesis did the electron density exceed $\pm 3\sigma(\varrho) [\sigma(\varrho)=0.35 \text{ e.Å}^{-3}]$.

The positions of the hydrogen atoms bound to the sp^3 hybridized carbon and nitrogen atoms were idealized to make the C-H and N-H bond lengths equal to 1.00 Å and to complete the tetrahedra.* The positions

* For the $-NH_3^+$ group a staggered conformation was assumed.

of the hydrogen atoms of the $C(5)H_2=C(4)H_-$ group were chosen taking into account that the olefinic carbon atoms take some alkyl-type character upon coordination to platinum (Hamilton, 1969; Spagna & Zambonelli, 1971, and references quoted therein). Accordingly the hydrogen atoms were positioned assuming a displacement, from the ideal plane of the doublebond, consistent with that observed for the C(3) carbon atom. The ideal plane of the double-bond was defined as the plane through C(4) and C(5) and normal to the Pt, C(4) and C(5) plane.

The fixed contributions of the hydrogen atoms to F_c were included in subsequent cycles of refinement, the *B* value for each hydrogen atom being equal to that which the nearest atom had at the end of the isotropic refinement. The final *R* index is 0.060 ($R_w = 0.070$). In Table 2 the positional and thermal parameters for the non-hydrogen atoms are reported; the idealized positions of the hydrogen atoms are given in Table 3.

Table 3. Ia	lealized	coordinat	tes and	isotro	pic tempe	rature
factors j	for the <i>k</i>	hydrogen	atoms i	in the	orange fo	orm

	Bonded to	x	У	z	<i>B</i> (Å ²)
H (1)	N	0.346	0.071	0.885	4.6
H(2)	N	0.420	0.011	0.806	4.6
H(3)	N	0.377	0.145	0.749	4.6
H(4)	C(1)	0.244	-0.093	0.728	4.9
H(5)	C(1)	0.171	0.041	0.672	4.9
H(6)	C(2)	0.347	-0.094	0.554	5.5
H(7)	C(2)	0.311	0.054	0.513	5.5
H(8)	C(3)	0.108	-0.139	0.473	4.7
H(9)	C(3)	0.084	0.002	0.414	4.7
H(10)	C(4)	0.212	-0.190	0.303	4.7
H(11)	C(5)	-0.017	-0.032	0.157	5.4
H(12)	C(5)	0.044	-0.143	0.075	5.4

The final values of observed and calculated structure amplitudes (in electrons \times 10) for the reflexions used in the analysis are presented in Table 4.

Yellow form

The full-matrix least-squares refinement converged at R=0.113, with isotropic temperature factors, and at R=0.063 ($R_w=0.070$), with anisotropic temperature factors. During the refinement the calculated C(2)-C(3) bond distance had values always higher than expected. The same distance had a much more reasonable value

Table 2. Positional ($\times 10^4$) and thermal parameters for the non-hydrogen atoms in the orange form

Estimated standard deviations are given in parentheses. Coefficients b_{ij} are defined by the expression $T = \exp \left[-10^{-4}(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{13}hl + b_{23}kl)\right].$

	x	У	Ζ	b11	b12	b13	b22	b23	b33
Pt	2336 (1)	327 (1)	1695 (1)	88 (1)	-2(1)	45 (1)	91 (1)	5(1)	118 (1)
Cl(1)	3238 (5)	-1288(5)	673 (6)	137 (6)	-25(8)	113 (11)	98 (5)	-54(10)	194 (8)
Cl(2)	3939 (5)	1685 (4)	1269 (6)	136 (6)	- 26 (8)	149 (11)	101 (5)	-14(10)	174(0)
Cl(3)	1345 (6)	1974 (5)	2512 (7)	148 (7)	46 (10)	142 (13)	116 (6)	-18(11)	218 (10)
N	3663 (18)	601 (14)	7902 (17)	123 (20)	8 (27)	36 (34)	106 (18)	13 (29)	134 (22)
C(1)	2543 (22)	-86(19)	6860 (23)	125 (27)	-43(31)	87 (46)	104 (21)	2 (36)	159 (30)
C(2)	2785 (23)	-269(21)	5453 (21)	118 (24)	-69 (41)	31 (38)	148 (25)	10 (44)	115 (24)
C(3)	1466 (22)	-677 (18)	4316 (23)	119 (23)	-46(33)	76 (41)	115 (22)	-9(38)	146 (28)
C(4)	1625 (23)	-1083(18)	2883 (24)	163 (30)	- 56 (37)	156 (51)	86 (20)	17 (40)	177 (31)
C(5)	650 (21)	-802(21)	1561 (23)	102(22)	-28(35)	79 (39)	146 (25)	-37(43)	145 (20)

when calculated from the positions given by the electron density map. Nevertheless the coordinates of the atoms N, C(1), C(2), C(3) obtained from the electron density map differed significantly from those given by the least-squares refinement. Without finding a plausible explanation for such discrepancies, refinement was repeated in the following manner.

The coordinates of the atoms N, C(1), C(2) and C(3) were refined by a series of difference Fourier syntheses each of which was calculated after two cycles of full-

matrix least-squares refinement in which the atoms N, C(1), C(2) and C(3) were included at their current locations. For these atoms the isotropic thermal parameters only were refined by least-squares calculations. The atoms Pt, Cl(1), Cl(2) and Cl(3) were allowed to vibrate anisotropically. Convergence was reached at R=0.065.

The contribution of the hydrogen atoms was included in the structure factor calculation as for the orange form. The final R value is 0.064 ($R_w = 0.072$).

Table 4. Observed and calculated structure amp	litudes (in electrons $ imes$ 10) of the orange form
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Table 5. Positional ($\times 10^4$) and thermal parameters for the non-hydrogen atoms in the yellow form

Estimated standard deviations are given in parentheses. Coefficients b_{ij} are defined by the expression $T = \exp \left[-10^{-4} (b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl) \right].$

	x	У	Z	<i>B</i> (Ų)	<i>b</i> ₁₁	<i>b</i> ₁₂	b13	b22	b23	b33
Pt	1489 (2)	2024 (1)	-1411 (1)		231 (3)	-4(5)	23 (1)	180 (2)	7 (2)	28 (0)
Cl(1)	-1227(13)	407 (10)	-1380(6)		352 (20)	-71 (29)	24 (15)	212 (14)	-2(13)	54 (4)
Cl(2)	2168 (12)	840 (11)	-2563(5)		361 (24)	-65(34)	55 (15)	239 (17)	-35 (13)	42 (4)
Cl(3)	4145 (11)	3658 (10)	-1501 (5)		276 (24)	-91 (35)	53 (15)	187 (18)	- 26 (14)	56 (4)
N	6484	2659	2040	5.8 (6)		- /				
C(1)	7029	2533	1154	4.6 (7)						
C(2)	5206	1985	554	4·9 (6)						
C(3)	3302	3044	373	6.0 (7)						
C(4)	1375 (43)	2600 (32)	- 149 (18)	4.4 (6)						
C(5)	- 26 (39)	3470 (30)	-682 (16)	3.8 (6)						

In Table 5 the positional and thermal parameters for the non-hydrogen atoms are reported and the idealized positions of the hydrogen atoms are given in Table 6.



Fig. 1. Perspective views of the complex trichloro(pent-4enylammonium)platinum(II) in the two crystalline forms: (a) orange, (b) yellow. The 50% probability thermal vibration ellipsoids are included. The two drawings were produced by Johnson's (1965) ORTEP program.

 Table 6. Idealized coordinates and isotropic temperature factors for the hydrogen atoms in the yellow form

	Bonded to	х	у	Z	$B(Å^2)$
H(1)	N	0.774	0.303	0.242	5.8
H(2)	Ν	0.609	0.170	0.223	5.8
H(3)	N	0.527	0.333	0.203	5.8
H(4)	C(1)	0.825	0.187	0.117	4.6
H(5)	C(1)	0.742	0.350	0.098	4.6
H(6)	C(2)	0.470	0.108	0.077	4.9
H(7)	C(2)	0.570	0.178	0.003	4.9
H(8)	C(3)	0.293	0.330	0.091	6.0
H(9)	C(3)	0.381	0.391	0.012	6.0
H(10)	C(4)	0.099	0.185	0.022	4.4
H(11)	C(5)	0.028	0.451	-0.072	3.8
H(12)	C(5)	-0.159	0.336	-0.072	3.8

The final values of observed and calculated structure amplitudes (in electrons \times 10) for the reflexions used in the analysis are presented in Table 7.

For both forms the function minimized during the least-squares refinement was $\sum w(|F_o| - |F_c|)^2$, with weights $w = 4F_o^2/\sigma(F_o^2)$. Atomic form factors given by Cromer & Mann (1968) for platinum, chlorine, nitrogen and carbon, and by Hanson, Herman, Lea & Skillman (1964) for hydrogen, were used. The effects of anomalous dispersion were included in the calculated structure factors (Ibers & Hamilton, 1964); the values of $\Delta f'$ and $\Delta f''$ for Pt and Cl were those computed by Cromer (1965).

Results and discussion

Trichloro(pent-4-enylammonium)platinum(II), in the orange form, I, as well as in the yellow form, II, is a zwitterionic platinum-olefin complex.

Perspective views of the molecule, as found in the two crystalline forms, are shown in Fig. 1. Bond lengths and angles are reported in Table 8.

Table 7. Observed and calculated structure amplitudes (in electrons $\times 10$) of the yellow form

 Table 8. Bond lengths and angles in trichloro(pent-4enylammonium)platinum(II) in the two crystalline forms

Estimated standard deviations are given in parentheses. MP defines the midpoint of the olefinic double bond.

(a) Bond lengths

(,	Orange form	Yellow form
Pt-Cl(1)	2·313 (6) Å	2·321 (9) Å
Pt-Cl(2)	2.342 (6)	2.335 (9)
Pt-Cl(3)	2.288(6)	2.321 (9)
Pt-MP	2.01	2.07
Pt-C(4)	2.145 (23)	2·193 (30)
Pt-C(5)	2.112 (22)	2·170 (28)
N-C(1)	1.496 (25)	1.59
C(1) - C(2)	1.459 (34)	1.49
C(2) - C(3)	1.557 (28)	1.56
C(3)-C(4)	1.498 (34)	1.441 (26)
C(4) - C(5)	1.412 (27)	1.406 (37)
(b) Bond angles		
Cl(1)-Pt-Cl(2)	88·8 (2)°	87·9 (3)°
Cl(1) - Pt - Cl(3)	175.2 (2)	177.5 (3)
Cl(1)-Pt-MP	88.4	88.7
Cl(2)-Pt-Cl(3)	90.6 (2)	90.4 (3)
Cl(2)-Pt-MP	173.3	176.1
Cl(3)-Pt-MP	92 ·7	92.8
C(4) - Pt - C(5)	38.8 (8)	37.6 (10)
N - C(1) - C(2)	111.8 (20)	111.9
C(1)-C(2)-C(3)	109.2 (20)	113.8
C(2)-C(3)-C(4)	114.3 (20)	119.4 (12)
C(3)-C(4)-C(5)	121.3(21)	126.7 (24)

In both I and II platinum is four-coordinated in a square-planar configuration by three chlorine atoms and the olefinic double bond of the pentenylammonium cation. Pt, Cl(1), Cl(2) and Cl(3) lie almost exactly in a plane and the best plane through them

0.332x - 0.209y + 0.784z - 1.955 = 0 in I,

0.550x - 0.592y + 0.478z + 1.751 = 0 in II,

(in terms of monoclinic coordinates) was assumed as coordination plane.

The centre of the double bond is on the coordination plane in II, but is 0.33 Å out of the coordination plane in I. The double bond makes angles of 81.7° in I and 87.5° in II with the coordination plane.

The plane defined by the platinum atom and the carbon atoms of the coordinated double bond, makes dihedral angles of 84.3 and 89.4° with the coordination plane in I and II respectively.

The dimensions and the geometry around the metal atom of trichloro(pent-4-enylammonium)platinum(II) compare reasonable well in I and II, and show trends (*i.e. trans* bond lengthening effect, lengthening of the coordinated double bond) consistent with the structural results observed in other platinum(II)-olefin complexes (Spagna & Zambonelli, 1971, and references quoted therein).

The only large structural difference in the molecule of trichloro(pent-4-enylammonium)platinum(II) observed in the two forms concerns the conformation of the coordinated pentenylammonium cation, namely the rotational angle around the C(1)-C(2) bond, as can be seen from Table 9.

Table 9. Internal rotation angles of the pent-4-enylammonium cation in the two crystals

The convention of Klyne & Prelog (1960) is adopted.

	Orange crystal	Yellow crystal
N - C(1) - C(2) - C(3)	- 166°	66°
C(1)-C(2)-C(3)-C(4)	- 171	-175
C(2)-C(3)-C(4)-C(5)	- 143	- 147

In both crystals the nitrogen atom of the pentenylammonium cation has short contacts with chlorine atoms: six in I and five in II. The distances of these contacts (listed in Table 10) are all shorter than the

Table 10. Short intermolecular contacts between nitrogen and chlorine atoms

Orange	form		
$N \cdots C$	l(1 ⁱ)	3	•23 Å
$N \cdots C$	l(2 ⁱⁱ)	3	.34
$N \cdots C$	l(2 ⁱⁱⁱ)	3	.37
N···C	l(2 ⁱ)	3	•42
N···C	l(1 ⁱⁱⁱ)	3	•47
$N \cdots C$	l(3 ¹¹)	3	•49
Yellow	form		
$N \cdots C$	l(2 ⁱⁱ)	3	·29 Å
$N \cdots C$	$l(1^{iv})$	3	·32
$N \cdots C$	l(3 ⁱⁱ)	3	.33
$N \cdots C$	l(2 ^v)	3	•46
$N \cdots C$	l(3 ^{vi})	3	-57
Superscr	ipts refer	to aton	ns at:
i	1 - x,	-y,	1 - z
ii	х,	$\frac{1}{2} - y$,	$\frac{1}{2} + z$
iii	х,	у,	1 + z
iv	1 + x,	$\frac{1}{2} - y$,	$\frac{1}{2} + z$
v	1 - x,	-y,	-z
vi	1-x,	1 - y,	-z



Fig. 2. Packing of the molecules in the orange form: the hydrogen-bond network is shown by broken lines.



Fig. 3. Packing of the molecules in the yellow form: the hydrogen-bond network is shown by broken lines.

sum of ionic radii (3.62 Å). Probably all the hydrogen atoms of the $-NH_3^+$ group take part in a $N-H\cdots$ Cl hydrogen bond network, some of the hydrogen bonds probably being bifurcated (Hamilton & Ibers, 1968).

The packing of the molecules in the two crystals is shown in Figs. 2 and 3.

Calculations were carried out on the UNIVAC 1108 computer of Rome University and were performed with the system of programs developed in this laboratory (Domenicano, Spagna & Vaciago, 1969), with the extensive contribution of J. R. Carruthers.

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