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CRYSTAL AND MOLECULAR STRUCTURES OF THE OLEFIN COMPLEXES TRICHLORO(π-ALLYLAMMONIUM)PLATINUM(II), TRICHLORO(π-BUT-3-ENYLAMMONIUM)PLATINUM(II) AND TRICHLORO(π-HEX-5-ENYLAMMONIUM)PLATINUM(II)

P. MURA, R. SPAGNA and L. ZAMBONELLI

Laboratorio di Strutturistica Chimica "Giordano Giacomello" C.N.R., Area della Ricerca, C.P. 10, 00016 Monterotondo Stazione (Roma) (Italy)

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Summary

The crystal and molecular structures of the title compounds, $[PtCl_3(C_3H_8N)]$ (I), $[PtCl_3(C_4H_{10}N)]$ (II), and $[PtCl_3(C_6H_{14}N)]$ (III) have been determined by single-crystal X-ray methods. I crystallizes in the orthorombic system (*Pbca*) with a 10.444(3), b 8.782(2), c 17.036(5) Å, Z 8; II is monoclinic ($P2_1/n$) with a 7.953(5), b 11.254(6), c 10.722(6) Å, β 111.07(4)°, Z 4; III is orthorombic ($P2_12_12_1$) with a 17.080(6), b 6.768(2), c 9.541(4) Å, Z 4. Full-matrix least-squares refinements have given final R-factors of 0.051 (1077 reflections for I), 0.043 (1325 reflections for II) and 0.041 (1123 reflections for III). The reflections were collected by counter methods and only those having $I > 3\sigma(I)$ were used in the analyses.

In all three complexes platinum is four-coordinate, having as ligands the three chlorine atoms and the double bond of the $CH_2{=}CH{-}(CH_2)_n{-}\overset{+}{N}H_3$ (n = 1, 2 or 4) cationic species. The structures are discussed, and compared with that of the analogous complex containing the pent-4-enylammonium cation, $CH_2{=}CH{-}(CH_2)_3{-}\overset{+}{N}H_3$.

Introduction

It has recently been established that unsaturated amines coordinate to platinum(II) in acid solution giving olefin complexes of the unsaturated ammonium cation species, with structures analogous to that of Zeise's salt [1]. X-ray structural studies on various platinum(II) π-complexes with unsaturated amines have been reported [2—8] which confirm that the olefinic double bonds of the ammonium cations coordinate to the metal atom according to the Dewar—Chatt—Duncanson model [9,10]. This model requires that a σ bond is formed by overlap of the π orbital of the olefinic double bond with a $5d6s6p^2$ hybrid orbital of the platinum atom, and a π back-bond is formed by overlap of a filled $5d6p$

orbital of the metal with the antibonding π orbital of the double bond. This bonding scheme is favoured when the coordinated double bond is perpendicular to the square plane surrounding the platinum [11] and such an arrangement was always found.

In order to study the effects of the electron-withdrawing ammonium group of the ligand on the olefin—metal bonding and on the molecular structure, platinum(II)—olefin complexes with the unsaturated ammonium cations of the type $CH_2{=}CH{-}(CH_2)_n{-}NH_3$ (n = 1, 2, 3, 4) have been studied by X-ray analysis. An account of the structure of the complex with the pent-4-enylammonium cation (n = 3) has been published [5] and we now present the results of the structural study of the complexes $[PtCl_3\{CH_2{=}CH{-}(CH_2)_n{-}NH_3\}]$ with n = 1, 2 and 4.

Experimental

But-3-enylamine and hex-5-enylamine were prepared from commercial but-3-enylchloride and hex-5-enylbromide, respectively, by the Gabriel phthalimide method. Commercial allylamine was used.

The zwitterionic complexes trichloro(π-allylammonium)platinum(II) (I), trichloro(π-but-3-enylammonium)platinum(II) (II), and trichloro(π-hex-5-enylammonium)platinum(II) (III), were prepared by method 1 of Claridge and Venanzi [12]. Good yellow crystals, suitable for X-ray analysis, were formed from solutions kept at room temperature.

Preliminary Weissemberg photographs revealed that the crystals of I are orthorhombic (space group *Pbca*), the crystals of II monoclinic (space group $P2_1/n$), and the crystals of III orthorombic (space group $P2_12_12_1$).

Accurate unit cell dimensions were determined using least-squares procedures from the angular positions of 15 reflections centered on a computer controlled diffractometer Syntex $P2_1$ using graphite monochromatized Mo-K_α radiation. A summary of the crystal data is presented in Table 1.

Intensity data for the three compounds were collected on the Syntex $P2_1$

TABLE 1

SUMMARY OF CRYSTAL DATA OF I—III

	I	II	III
Compound	$[PtCl_3(C_3H_8N)]$	$[PtCl_3(C_4H_{10}N)]$	$[PtCl_3(C_6H_{14}N)]$
Formula	$C_3H_8Cl_3NPt$	$C_4H_{10}Cl_3NPt$	$C_6H_{14}Cl_3NPt$
Mol. wt.	359.55	373.58	401.63
a	10.444(3) Å	7.953(5) Å	17.080(6) Å
b	8.782(2) Å	11.254(6) Å	6.768(2) Å
c	17.036(5) Å	10.722(6) Å	9.541(4) Å
β		111.07(4)°	
V	1562.5(7) Å^3	895.5(8) Å^3	1102.9(7) Å^3
Z	8	4	4
Density (calcd)	3.057 g cm^{-3}	2.760 g cm^{-3}	2.419 g cm^{-3}
Density (found) [a]	> of that of $CHBr_3$	2.75(1) g cm^{-3}	2.37(1) g cm^{-3}
Space group	D_{2h}^{15}—$Pbca$	C_{2h}^{5}—$P2_1/n$	D_2^4—$P2_12_12_1$
μ (Mo-K_α)	198.0 cm^{-1}	172.8 cm^{-1}	140.4 cm^{-1}

[a] Measured by flotation in $CHCl_3/CHBr_3$ mixtures.

TABLE 2

DATA COLLECTION DETAILS

	I	II	III
Radiation	Mo-K_α (λ 0.71069 Å)		
Monochromator	Graphite (002)		
Crystal dimensions	0.20 × 0.20 × 0.30 mm	0.07 × 0.12 × 0.29 mm	0.13 × 0.13 × 0.26 mm
Scan mode	ω	ω	ω
Scan speed	$3.0 \div 29.3^\circ$ min^{-1}	$2.0 \div 29.3^\circ$ min^{-1}	$3.0 \div 29.3^\circ$ min^{-1}
Scan range	0.80°	0.80°	0.80°
Background counts	For a time equal to the scan time at ±0.60° from the peak		
Check reflection	Three every 100 reflections: no decay		
Data collection limits	$3.0 \div 56.0^\circ$ in 2θ		
	h, k, l	$h, k, \pm l$	h, k, l
No of data	2175	2500	1552

diffractometer: relevant details of the data collection are given in Table 2. The data were processed as previously described [13] to yield values of I and $\sigma(I)$. In the estimation of $\sigma(I)$, the uncertainty factor p was set equal to 0.007 for I and to 0.01 for II and III, as indicated by the variance of the standard reflections [14].

The values of I and $\sigma(I)$ were corrected for Lorentz, polarization and shape anisotropy effects. For the latter correction the published procedure was followed [15,16,4]. 1077, 1325 and 1129 independent reflections, for I, II and III respectively, having $F_o^2 > 3\,\sigma(F_o^2)$, were used in subsequent calculations.

Solution and refinement of the structures

The three structures were solved by the usual combination of Patterson and Fourier methods, and refined by full-matrix least-squares procedures. The isotropic refinements converged at R 0.092 for I, R 0.094 for II and R 0.067 for III. Allowing all atoms to vibrate anisotropically, convergence was reached at R 0.052, 0.043 and 0.041 respectively. At this stage the positions of the hydrogen atoms were idealized in a manner previously described [5] taking into account that the olefinic carbon atoms take some alkyl-type character upon coordination [17]. The fixed contribution of the hydrogen atoms was included in the structure factor calculations, being readjusted after each cycle of refinement. Each hydrogen atom was assigned the B value which the nearest atom had at the end of the isotropic refinement. The final R is 0.051 (Rw = 0.071) for I, 0.043 (Rw = 0.062) for II and 0.041 (Rw = 0.062) for III. In each case the final difference-Fourier synthesis did not reveal any region exceeding $\pm 3\,\sigma(\rho)$ [$\sigma(\rho)$ = 0.67, 0.47 and 0.36 e Å^{-3} for I, II and III].

For the three compounds the function minimized during the refinement was $\Sigma w(|F_o|-|F_c|)^2$ with $w = (a + F_o + bF_o^2)^{-1}$ were a and b are of the order of $2\,F_o(\text{min})$ and $2/F_o(\text{max})$ respectively [18]. This function was not appreciably dependent on either θ or on F_o, indicating a correctly chosen weighting scheme.

The scattering factors for Pt, Cl, N and C were taken from [19a] and that for H from [19b]. The anomalous terms for Pt and Cl were those of Cromer and Liberman [19c] and were included in F_c [20].

TABLE 3

POSITIONAL AND THERMAL PARAMETERS [a] ($\times 10^4$) OF I, II, AND III. STANDARD DEVIATIONS ARE GIVEN IN PARENTHESES

	x	y	z	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
Compound I									
Pt	351(1)	313(1)	1204(0)	35(1)	−12(1)	−1(1)	48(1)	−7(1)	17(0)
Cl(1)	−1459(5)	223(7)	1965(3)	59(4)	14(11)	26(5)	89(7)	1(7)	27(2)
Cl(2)	−217(5)	2785(6)	832(3)	73(5)	2(10)	18(6)	52(5)	−5(6)	33(2)
Cl(3)	2158(5)	395(7)	425(3)	45(4)	−21(11)	26(5)	109(8)	−11(8)	34(2)
N	−1506(16)	−2999(20)	829(11)	51(16)	−108(37)	8(17)	88(25)	−6(22)	24(7)
C(1)	−511(18)	−3109(21)	1464(11)	63(18)	−28(34)	−6(18)	33(22)	−5(19)	20(5)
C(2)	673(18)	−2117(22)	1291(12)	46(16)	−24(30)	−5(18)	33(20)	−14(22)	28(8)
C(3)	1269(17)	−1391(17)	1895(11)	43(17)	−2(27)	−33(17)	44(16)	−18(17)	25(7)
Compound II									
Pt	5172(1)	4924(0)	2773(1)	81(1)	3(1)	60(1)	52(0)	0(1)	71(1)
Cl(1)	3571(6)	6060(4)	953(4)	174(9)	−6(9)	8(10)	79(3)	31(6)	82(4)
Cl(2)	7119(6)	6526(4)	3584(5)	127(8)	−66(8)	23(10)	73(3)	1(6)	110(5)
Cl(3)	6847(6)	3864(4)	4652(5)	146(8)	43(9)	84(11)	90(4)	72(7)	107(5)
N	49(20)	3870(17)	3397(15)	130(28)	15(36)	104(35)	142(18)	−22(27)	81(17)
C(1)	203(22)	4572(17)	2233(15)	132(31)	6(34)	11(35)	101(15)	−36(25)	49(14)
C(2)	1203(21)	3889(16)	1520(16)	118(30)	−1(34)	71(37)	93(15)	−60(25)	70(17)
C(3)	3126(20)	3545(13)	2317(15)	130(27)	−54(28)	55(33)	68(12)	−1(20)	56(14)
C(4)	4350(26)	3305(16)	1675(22)	157(36)	−53(37)	139(49)	82(15)	−32(31)	134(24)
Compound III									
Pt	1435(0)	1468(1)	1868(1)	21(0)	−2(1)	7(1)	153(1)	−23(2)	67(1)
Cl(1)	1226(3)	4031(8)	326(6)	31(2)	−18(7)	−9(5)	185(13)	8(14)	91(6)
Cl(2)	2642(3)	2832(10)	2451(7)	25(2)	−41(9)	−16(6)	250(15)	30(18)	114(6)
Cl(3)	1658(4)	−970(10)	3521(6)	41(2)	−6(9)	−7(6)	233(16)	47(14)	90(6)
N	−2312(12)	−2181(35)	−883(20)	33(7)	−35(36)	8(22)	296(58)	17(60)	76(20)
C(1)	−1477(13)	−2562(30)	−1461(19)	22(6)	−83(32)	6(21)	190(43)	−26(48)	73(18)
C(2)	−915(11)	−1242(34)	−737(19)	24(7)	−4(34)	−47(18)	208(50)	−47(58)	53(17)
C(3)	−57(15)	−1818(43)	−1229(23)	41(10)	−35(46)	−16(25)	259(70)	−25(65)	66(21)
C(4)	548(12)	−462(40)	−680(27)	21(7)	−108(36)	−2(25)	244(57)	58(82)	131(31)
C(5)	617(12)	−569(35)	956(20)	19(6)	4(32)	9(20)	194(43)	0(55)	65(20)
C(6)	230(11)	825(30)	1845(28)	21(6)	−52(27)	76(28)	132(41)	−225(75)	177(34)

[a] Coefficients b_{ij} are defined by the expression $T = \exp-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)$.

The calculations were performed, using local programmes, on the UNIVAC 1110 computer of the University of Rome [21a] and on the HP 21MX minicomputer of the CNR Research Area [21b].

The final positional and thermal parameters of I, II and III are given in Table 3.

Description of the structures and discussion

Perspective views of the three zwitterionic molecules are shown in Fig. 1. Interatomic bond lengths and angles are given in Table 4, in which also those of the complex with pent-4-enylammonium are shown for comparison.

In each complex the coordination arrangement about the Pt atom is square-planar with the three chlorine atoms and the midpoint of the olefinic double bond. The equatorial planes were calculated as the best plane through the platinum and the chlorine atoms. The equations of these planes, expressed in terms of the crystallographic axes, are reported in Table 5. Additional data about the arrangement of the olefinic ligand with respect to the $PtCl_3$ moeity are also reported for all three complexes and for that with the pent-4-enylammonium ligand.

In I and II the deviation of the double bond from the direction perpendicular to the equatorial plane is mostly due to an inclination of the double bond in the plane, while in III it appears that it is only due to a tilt around the Pt—olefin direction.

As observed in many other platinum—olefin complexes also in the present compounds the Pt—Cl bond in *trans* to the coordinated double bond is slightly but significantly longer than those in the *cis* positions. It is known that olefins

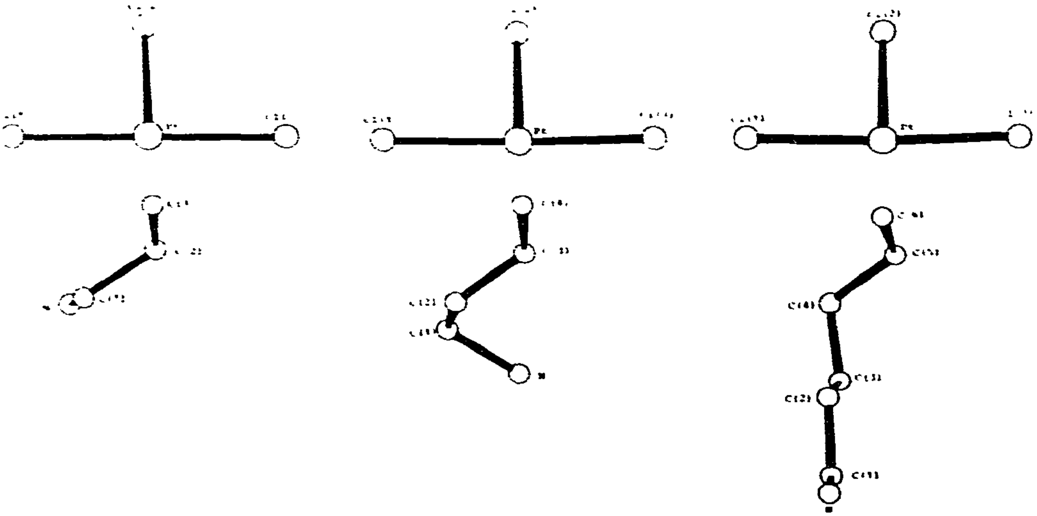

Fig. 1. Perspective views of the zwitterions [$PtCl_3$(π-allylammonium)], [$PtCl_3$(π-but-3-enylammonium)] and [$PtCl_3$ π-hex-5-enylammonium)].

TABLE 4

BOND LENGTHS (Å) AND BOND ANGLES (DEGREES). STANDARD DEVIATIONS ARE GIVEN IN PARENTHESES

	I	II	III	$[\mathrm{PtCl_3(C_5H_{12}N)}]$ [a]	
				Orange form	Yellow form
Pt—Cl(1)	2.294(5)	2.298(6)	2.302(6)	2.313(6)	2.321(9)
Pt—Cl(2)	2.337(6)	2.332(6)	2.327(6)	2.342(6)	2.335(9)
Pt—Cl(3)	2.309(5)	2.308(6)	2.314(6)	2.288(6)	2.321(9)
Pt—C(α) [b]	2.166(19)	2.173(16)	2.148(22)	2.145(23)	2.193(30)
Pt—C(β) [b]	2.131(17)	2.140(21)	2.103(20)	2.112(22)	2.170(28)
Pt—M [c]	2.04	2.04	2.00	2.01	2.07
N—C(1)	1.50(3)	1.52(2)	1.55(3)	1.50(3)	1.59
C(1)—C(2)	1.54(3)	1.50(2)	1.48(3)	1.46(3)	1.49
C(2)—C(3)	1.36(3)	1.51(3)	1.59(3)	1.56(3)	1.56
C(3)—C(4)		1.41(3)	1.48(4)	1.50(3)	1.44(3)
C(4)—C(5)			1.57(3)	1.41(3)	1.41(4)
C(5)—C(6)			1.43(3)		
Cl(1)—Pt—Cl(2)	88.6(2)	88.7(2)	89.5(2)	88.8(2)	87.9(3)
Cl(1)—Pt—Cl(3)	179.3(2)	177.3(2)	176.6(2)	175.2(2)	177.5(3)
Cl(1)—Pt—M	92.7	93.2	93.7	88.4	88.7
CL(2)—Pt—Cl(3)	91.3(2)	88.7(2)	88.5(2)	90.6(2)	90.4(3)
Cl(2)—Pt—M	174.7	172.3	176.4	173.3	176.1
Cl(3)—Pt—M	87.4	89.6	88.3	92.7	92.8
C(α)—Pt—C(β)	36.9(7)	38.0(6)	39.3(8)	38.8(8)	37.6(10)
N—C(1)—C(2)	112.4(15)	111.5(16)	109.3(16)	111.8(20)	111.9
C(1)—C(2)—C(3)	119.1(17)	117.3(17)	108.2(18)	109.2(20)	113.8
C(2)—C(3)—C(4)		120.7(18)	112.9(21)	114.3(20)	119.4(12)
C(3)—C(4)—C(5)			112.1(20)	121.3(21)	126.7(24)
C(4)—C(5)—C(6)			121.7(20)		

[a] For comparison the values relative to the structure of the complex $[\mathrm{PtCl_3\{CH_2{=}CH(CH_2)_3NH_3\}}]_n$, published in ref. 5, are also reported. This complex crystallizes in two different forms, called, according to the color of the crystals, the orange and the yellow form. Both were studied by X-ray analysis, but refined by different procedures. The yellow form was refined by least-squares and difference-Fourier methods. For this reason some of its parameters do not present standard deviations. [b] C(α) and C(β) refer to the vinylic group, —C(α)=C(β)H$_2$, for each chain. [c] M is the midpoint of the coordinated double bond.

TABLE 5

ARRANGEMENT OF THE COORDINATED DOUBLE BOND WITH RESPECT TO THE EQUATORIAL PLANE. STANDARD DEVIATIONS ARE IN THE ORDER OF MAGNITUDE OF 0.07 Å AND 2.0°

	I	II	III	$[\mathrm{PtCl_3(C_5H_{12}N)}]$ [a]	
				Orange form	Yellow form
M from ψ [b]	0.20 Å	0.28 Å	0.13 Å	0.33 Å	0.00 Å
(C=C) $\Lambda\psi$ [c]	82.9°	80.7°	80.4°	81.7°	87.5°
(PtCC) $\Lambda\psi$ [d]	88.0°	87.9°	80.4°	84.3°	89.4°

M is the midpoint of the coordinated double bond.

ψ is the equatorial plane calculated as the best plane through Pt, Cl(1), Cl(2) and Cl(3). The equations, expressed in terms of crystallographic coordinates, are:

$5.45\,x + 3.19\,y + 13.15\,z = 1.87$ for I

$6.41\,x - 3.83\,y - 7.95\,z = -0.78$ for II

$7.23\,x - 3.75\,y - 6.84\,z = -0.83$ for III

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[a] Ref. 5. [b] Deviation of M from the equatorial plane. [c] Angle between the direction of the coordinated double bond and the equatorial plane. [d] Dihedral angle between the equatorial plane and the plane through the metal atom and the carbon atoms of the coordinated double bond. This value gives a measure of the tilt of the double bond around the Pt—olefin direction.

TABLE 6

TORSION ANGLES (°) IN THE OLEFINIC LIGANDS [a]

I	II	III	$[PtCl_3(C_5H_{12}N)]$ [b]	
			Orange form	Yellow form
142.9(17)	60.9(19)	174.1(17)	166	−66
	156.8(17)	174.2(19)	171	175
		65.0(26)	143	147
		−97.2(27)		

[a] The signs of the angles have only a relative meaning. For each chain in fact they could be changed and referred to the specular configuration, as explained in the text. For a better comparison in this table the signs of the angles of the complex with pent-4-enylammonium as published in ref. 5 have been changed, so that all the angles given here refer to the C(α)*R* configuration. [b] Ref. 5.

exert a strong *trans* effect when coordinated [22], and with this high *trans* effect, which is a kinetic effect and can be explained by the accepted bonding scheme, is associated a weak *trans* influence in the solid state. This weak *trans* influence, defined as the tendency of a ligand to weaken the bond *trans* to itself, and observed in the solid state as a lengthening of the *trans* bond, is well explained by the accepted bonding scheme [23].

The conformations assumed by the olefinic chains may be deduced from the torsion angles reported in Table 6, according to the convention of Klyne and Prelog [24], together with those observed for the pent-4-enylammonium in the corresponding complex.

Chirality arises at the α-carbon atom of the vinylic group upon coordination [25], and so the zwitterionic complexes I, II and III are asymmetric. In the centrosymmetric space groups *Pbca* and $P2_1/n$, I and II are present in the racemic form, while in the acentric space group $P2_12_12_1$ III is present as an enantiomer. Coordinates reported in Table 3 refer to the C(α)*R* configuration for I and III and to the C(α)*S* configuration for II, while data of Table 6 and drawings of Fig. 1 refer for the three compounds to the C(α)*R* configuration.

There are significant N ... Cl contacts, the lengths of which are reported in Table 7. Probably all the hydrogen atoms of the NH_3 group in each complex

TABLE 7

INTERMOLECULAR CONTACTS (Å) BETWEEN NITROGEN AND CHLORINE ATOMS SHORTER THAN THE SUM OF THE IONIC RADII (3.62 Å). THE STANDARD DEVIATION IS 0.01 Å FOR ALL THE VALUES

I		II		III	
N...Cl(1)	3.43	N...Cl(1)^{i}	3.34	N...Cl(1)^{i}	3.33
Cl(1)^{i}	3.27	Cl(2)^{ii}	3.24	Cl(2)^{i}	3.30
Cl(2)^{ii}	3.36	Cl(2)^{i}	3.44	Cl(2)^{ii}	3.32
Cl(2)^{i}	3.49	Cl(3)^{iii}	3.28	Cl(3)^{iii}	3.32
Cl(3)^{ii}	3.20			Cl(3)^{ii}	3.59
Cl(3)^{iii}	3.31				
(i) $-\frac{1}{2}-x \quad -\frac{1}{2}+y \quad z$		(i) $\frac{1}{2}-x \quad -\frac{1}{2}+y \quad \frac{1}{2}-z$		(i) $-\frac{1}{2}+x \quad \frac{1}{2}-y \quad -z$	
(ii) $-x \quad -y \quad -z$		(ii) $1-x \quad 1-y \quad 1-z$		(ii) $-x \quad -\frac{1}{2}+y \quad \frac{1}{2}-z$	
(iii) $-\frac{1}{2}+x \quad -\frac{1}{2}-y \quad -z$		(iii) $-1+x \quad y \quad z$		(iii) $-\frac{1}{2}+x \quad -\frac{1}{2}-y \quad -z$	

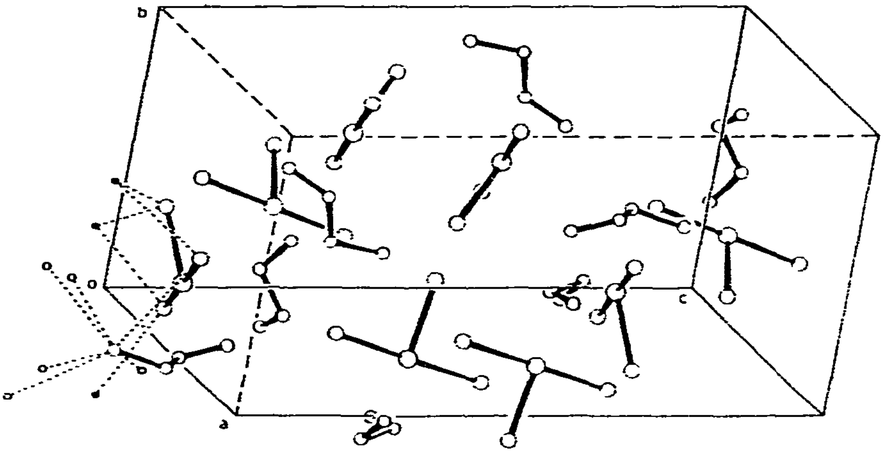

Fig. 2. Packing of the molecules in the structure of I: the hydrogen-bond network of the unit at xyz is shown by broken lines. Black and white circles refer respectively to nitrogen and chlorine atoms of surrounding units.

participate in H --- Cl hydrogen bonds, some of which bifurcated [26]. The packings are shown in Fig. 2—4 for I, II and III respectively.

No significant differences in the structures can be associated with the varying distances of the electron-withdrawing group $-\overset{+}{N}H_3$ from the coordinated double bond in the series of complexes $[PtCl_3\{CH_2{=}CH-(CH_2)_n-NH_3\}]$ with n = 1—4.

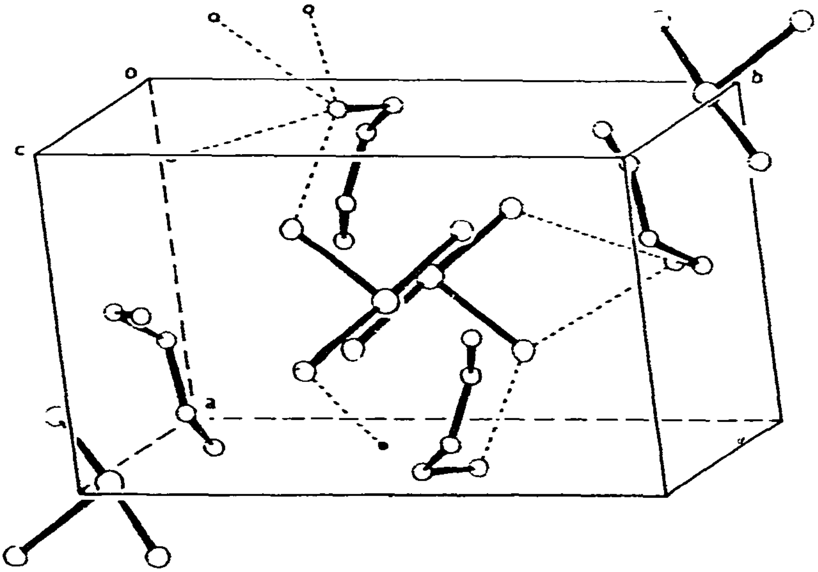

Fig. 3. Packing of the molecules in the structures of II: the hydrogen-bond network of the unit at xyz is shown by broken lines. Black and white circles refer respectively to nitrogen and chlorine atoms of surrounding units.

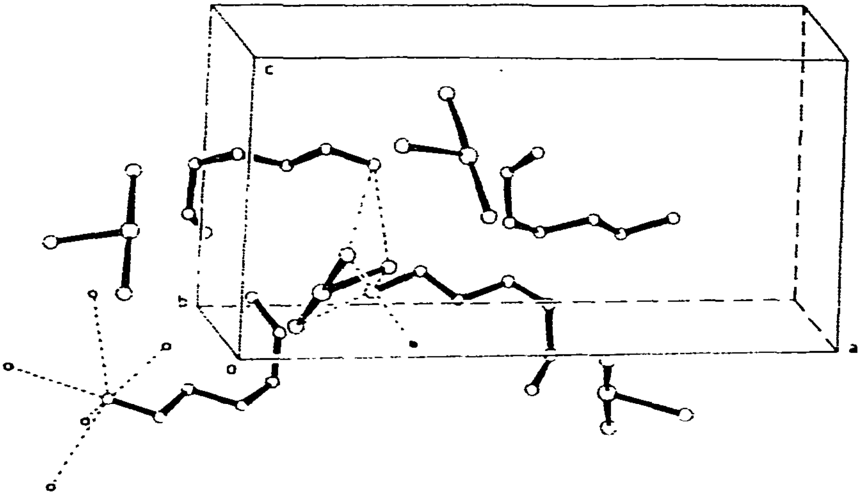

Fig. 4. Packing of the molecules in the structure of III; the hydrogen-bond network of the unit at xyz is shown by broken lines. Black and white circles refer respectively to nitrogen and chlorine atoms of surrounding units.

References

1 F.R. Hartley, Chem. Rev., 69 (1969) 799.
2 R. Spagna, L.M. Venanzi and L. Zambonelli, Inorg. Chim. Acta, 4 (1970) 283.
3 R. Spagna, L.M. Venanzi and L. Zambonelli, Inorg. Chim. Acta, 4 (1970) 475.
4 R. Spagna and L. Zambonelli, J. Chem. Soc. A, (1971) 2544.
5 R. Spagna and L. Zambonelli, Acta Crystallogr. B, 28 (1972) 2760.
6 R. Spagna, G. Ughetto and L. Zambonelli, Acta Crystallogr. B, 29 (1973) 1151.
7 P. Mura, R. Spagna, G. Ughetto and L. Zambonelli, Acta Crystallogr. B, 32 (1976) 2532.
8 P. Mura, R. Spagna, G. Ughetto and L. Zambonelli, J. Cryst. Mol. Struct., in press.
9 M.J.S. Dewar, Bull. Soc. Chim. Fr., 18 (1951) C79.
10 J. Chatt and L.A. Duncanson, J. Chem. Soc., (1953) 2939.
11 K.S. Wheelock, J.H. Nelson, L.C. Cusachs and H.B. Jonassen, J. Amer. Chem. Soc., 92 (1970) 5110.
12 D.V. Claridge and L.M. Venanzi, J. Chem. Soc., (1964) 3419.
13 F. Bachechi, L. Zambonelli and G. Marcotrigiano, J. Cryst. Mol. Struct., 7 (1977) 11.
14 L.E. McCandlish, G.H. Stout and L.C. Andrews, Acta Crystallogr., A, 31 (1975) 245.
15 T.C. Furnas, Single-Crystal Orienter Instruction Manual, General Electric Comapny, Milwaukee, 1957.
16 A.C.T. North, D.C. Phillips and F.S. Mattews, Acta Crystallogr. A, 24 (1968) 351.
17 R.A. Love, T.F. Koetzle, G.J.B. Williams, L.C. Andrews and R. Bau, Inorg. Chem., 14 (1975) 2653.
18 D.W.J. Cruickshank, In: J.S. Rollett (Ed.), Computing Methods in Crystallography, Pergamon Press, Oxford, 1965, p. 114.
19 (a) D.T. Cromer and J.T. Waber, International Tables for X-Ray Crystallography, Vol. IV, Kynoch-Press, Birmingham, England, 1974, Table 2.2A; (b) R.F. Stewart, E.R. Davidson and W.T. Simpson, ibid., Table 2.2C; (c) D.T. Cromer and D. Liberman, ibid., Table 2.3.1.
20 J.A. Ibers and W.C. Hamilton, Acta Crystallogr., 17 (1964) 781.
21 (a) J.R. Carruthers and R. Spagna, Ital. Crystallogr. Assoc., 7th Meet., Abstracts, (1975) 65; (b) S. Cerrini and R. Spagna, 4th Europ. Crystallogr. Meet., Abstracts, (1977) 7.
22 F. Basolo and R.G. Pearson, Mechanisms of Inorganic Reactions, 2nd edn., Wiley, New York, 1967, p. 355.
23 F.R. Hartley, Angew. Chem. Int. Ed., 11 (1972) 596.
24 W. Klyne and V. Prelog, Experientia, 16 (1960) 521.
25 G. Paiaro and A. Panunzi, J. Amer. Chem. Soc., 86 (1964) 5148.
26 W.C. Hamilton and J.A. Ibers, Hydrogen Bonding in solids, Benjamin, New York, 1968, p. 166.