Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and hydrogen-bond lengths* (Å)

Cu(1)—O(1)	1.976 (4)	N(4)—C(9)	1.480 (8)
Cu(1)—O(*W*1)	2.521 (7)	N(4)—C(10)	1.442 (9)
Cu(1)—N(1)	2.040 (5)	N(5)—C(15)	1.72 (3)
Cu(1)—N(7)	1.917 (5)	N(5)—C(15′)	1.40 (2)
Cu(1)—N(8)	2.020 (6)	N(5)—C(16)	1.483 (9)
Cu(2)—O(3)	1.999 (4)	N(6)—C(17)	1.303 (8)
Cu(2)—O(*W*2)	2.342 (7)	N(6)—C(18)	1.467 (9)
Cu(2)—N(3)	1.929 (5)	N(7)—C(19)	1.468 (9)
Cu(2)—N(4)	2.029 (6)	N(7)—C(20)	1.313 (9)
Cu(2)—N(5)	2.033 (6)	N(8)—C(21)	1.481 (9)
O(1)—C(5)	1.264 (7)	N(8)—C(22)	1.46 (1)
O(2)—C(8)	1.271 (7)	C(4)—C(5)	1.49 (1)
O(3)—C(17)	1.265 (7)	C(6)—C(7)	1.49 (1)
O(4)—C(20)	1.266 (8)	C(8)—C(9)	1.472 (9)
N(1)—C(3)	1.435 (9)	C(16)—C(17)	1.500 (9)
N(1)—C(4)	1.488 (9)	C(18)—C(19)	1.49 (1)
N(2)—C(5)	1.307 (9)	C(20)—C(21)	1.50 (1)
N(2)—C(6)	1.465 (9)	C(22)—C(23)	1.52 (1)
N(3)—C(7)	1.476 (9)	C(23)—C(24)	1.51 (2)
N(3)—C(8)	1.316 (8)		
O(1)—Cu(1)—O(*W*1)	89.2 (2)	O(1)—C(5)—N(2)	122.8 (6)
O(1)—Cu(1)—N(1)	82.7 (2)	O(1)—C(5)—C(4)	119.6 (6)
O(1)—Cu(1)—N(7)	94.2 (2)	N(2)—C(5)—C(4)	117.6 (6)
O(1)—Cu(1)—N(8)	177.3 (2)	N(2)—C(6)—C(7)	111.0 (7)
O(*W*1)—Cu(1)—N(1)	86.5 (2)	N(3)—C(7)—C(6)	108.4 (6)
O(*W*1)—Cu(1)—N(7)	104.4 (3)	O(2)—C(8)—N(3)	123.7 (6)
O(*W*1)—Cu(1)—N(8)	89.2 (3)	O(2)—C(8)—C(9)	118.9 (6)
N(1)—Cu(1)—N(7)	168.6 (3)	N(3)—C(8)—C(9)	117.2 (5)
N(1)—Cu(1)—N(8)	99.4 (2)	N(4)—C(9)—C(8)	112.8 (5)
N(7)—Cu(1)—N(8)	84.0 (2)	Cu(1)—O(1)—C(5)	112.9 (4)
O(3)—Cu(2)—O(*W*2)	88.3 (3)	Cu(2)—O(3)—C(17)	112.1 (4)
O(3)—Cu(2)—N(3)	93.7 (2)	Cu(1)—N(1)—C(3)	122.3 (5)
O(3)—Cu(2)—N(4)	172.9 (3)	Cu(1)—N(1)—C(4)	105.1 (4)
O(3)—Cu(2)—N(5)	81.9 (2)	C(3)—N(1)—C(4)	113.2 (6)
O(*W*2)—Cu(2)—N(3)	98.0 (3)	C(5)—N(2)—C(6)	125.4 (5)
O(*W*2)—Cu(2)—N(4)	98.8 (3)	Cu(2)—N(3)—C(7)	125.2 (4)
O(*W*2)—Cu(2)—N(5)	88.8 (3)	Cu(2)—N(3)—C(8)	115.5 (4)
N(3)—Cu(2)—N(4)	84.9{2)	C(7)—N(3)—C(8)	119.2 (5)
N(3)—Cu(2)—N(5)	171.8 (3)	Cu(2)—N(4)—C(9)	108.0 (4)
N(4)—Cu(2)—N(5)	98.6 (2)	Cu(2)—N(4)—C(10)	120.6 (4)
C(9)—N(4)—C(10)	114.0 (6)	N(5)—C(16)—C(17)	110.4 (5)
Cu(2)—N(5)—C(16)	106.5 (4)	O(3)—C(17)—N(6)	122.6 (6)
C(17)—N(6)—C(18)	124.1 (5)	O(3)—C(17)—C(16)	118.8 (6)
Cu(1)—N(7)—C(19)	126.0 (4)	N(6)—C(17)—C(16)	118.6 (5)
Cu(1)—N(7)—C(20)	114.6 (4)	N(6)—C(18)—C(19)	110.3 (6)
C(19)—N(7)—C(20)	119.3 (5)	N(7)—C(19)—C(18)	109.6 (6)
Cu(1)—N(8)—C(21)	105.3 (4)	O(4)—C(20)—N(7)	126.2 (7)
Cu(1)—N(8)—C(22)	109.9 (5)	O(4)—C(20)—C(21)	117.6 (6)
C(21)—N(8)—C(22)	113.2 (6)	N(7)—C(20)—C(21)	116.2 (6)
N(1)—C(4)—C(5)	109.4 (5)	N(8)—C(21)—C(20)	110.1 (6)

$D—H\cdots A$	$D\cdots A$	$D—H\cdots A$	$D\cdots A$
O(*W*1)—H(*W*11)···O(13^{i})	3.03 (2)	N(2)—H(N2)···O(4^{v})	2.736 (7)
O(*W*1)—H(*W*12)···O(14)	2.89 (1)	N(4)—H(N4)···O(21)	3.06 (1)
O(*W*2)—H(*W*22)···O(2^{ii})	2.663 (8)	N(6)—H(N6)···O(2^{vi})	2.813 (7)
O(*W*2)—H(*W*21)···O(*W*2^{iii})	3.10 (1)	N(8)—H(N8)···O(12)	3.18 (1)
N(1)—H(N1)···O(23^{iv})	2.99 (1)		

Symmetry codes: (i) $2-x, -y, 2-z$; (ii) $1-x, -y, 1-z$; (iii) $2-x, -y, 1-z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $x-1, y, z$; (vi) $1+x, y, z$.

The structure was solved by direct and Fourier methods and refined by full-matrix least squares. H atoms were located by difference Fourier methods and theoretical calculation. The *NRCVAX* program (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71490 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1044]

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Tris(2-pyridinethiolato)(*p*-tolyl)tin(IV), $[Sn(C_5H_4NS)_3(C_7H_7)]$

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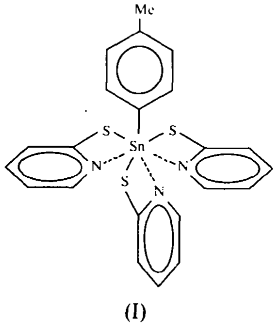
Abstract

In the title compound three bidentate 2-pyridinethiolato ligands (2-SPy) together with *p*-tolyl form a distorted pentagonal bipyramid around Sn. Two S atoms and three N atoms form the pentagonal plane. One S and the C(*p*-tolyl) atom are in the axial positions. The distortion of the molecule is explicated by the axial S—Sn—C angle of 156.0 (3)° and by the distance of 0.635 (7) Å of one of the N atoms from the pentagonal plane. The Sn—S distances in the plane are longer [2.571 (3) Å] than the axial Sn—S bond [2.486 (3) Å]. The Sn—N distances are in the range 2.468 (8) ± 0.024 Å. Intermolecular distances shorter than the sum of the van der Waals radii do not exist.

Comment

In $Ph_2Sn(2\text{-}SPy)_2$ (Schmiedgen, Huber & Preut, 1993) as well as in $Sn(2\text{-}SPy)_4.HSPy$ (Damude, Dean, Manivannan, Srivastava & Vittal, 1990), tin is six-coordinated. In the latter compound two 2-SPy ligands are bonded bidentately *via* S and N atoms, while two 2-SPy ligands are bonded virtually unidentately *via* an S atom; the Sn—N distance (3.0 Å) of only one of these ligands is less than the sum of the van der Waals radii. In the course of our studies on the coordination of organotin compounds by ligands with S and N donor centres, it seemed worthwhile to find out whether Sn in mono-organotin compounds, such as RSn(2-SPy)$_3$, would be also six-coordinated or if it would bind all three 2-SPy ligands bidentately to attain the coordination number seven.

The title compound (I) was prepared from (p-tolyl)SnCl$_3$ in CHCl$_3$ and an equivalent amount of NaOMe in MeOH. After evaporating the solvents, the residue was redissolved in CHCl$_3$, the solution filtered and the light yellow product (m. p. 434 K) precipitated by adding petroleum ether (b. p. 313–333 K).



(I)

The molecular structure and atomic numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. All three 2-SPy ligands are bidentately bonded to Sn; two S and three N atoms form a pentagonal plane containing Sn. Two S and two N atoms belong to two 2-SPy ligands, which lie in the plane; one N atom is contributed by the third ligand which is approximately vertically arranged with respect to the equatorial plane. The remaining S atom and the C(p-tolyl) atom are arranged axially. They complete a pentagonal bipyramidal polyhedron around Sn. The axial S(1)—Sn(1)—C(1) angle of 156.0 (3)° and the deviations of the equatorial atoms from the least-squares plane through these atoms [Sn(1) 0.031 (1), S(2) −0.053 (3), S(3) −0.101 (3), N(11) −0.635 (7), N(21) −0.188 (7), N(31) 0.052 (7) Å] demonstrate the distortion of the molecule. The least-squares planes through the atoms of the 2-SPy ligands show that these are approximately planar. The Sn—N distances [2.444 (8)–2.487 (7) Å] are not significantly different. They are considerably shorter than the sum of the appropriate van der Waals radii [3.75 Å (Bondi, 1964)] and indicate the bidentate chelating nature of the 2-SPy ligands. In the inorganic compound $Sn(SPy)_4.HSPy$, the Sn—N distances are remarkably shorter [2.324 (5)–2.332 (5) Å]. In other comparable organotin compounds the Sn—N distances are longer, *e.g.* in ^{n}BuSn(SPym)$_3$ (SPym = 2-pyrimidinethiolato) [2.517 (6)–2.633 (9) Å (Schmiedgen, Huber & Schürmann, 1994)], $Ph_2Sn(SPy)_2$ [2.667 (4) Å (Schmiedgen, Huber & Preut, 1993)] and ^{n}Bu$_2$Sn(2-SPy-5-NO$_2$)$_2$ [2.77 (1) Å (Domazetis, James, Mackay & Magee, 1979)]. In the analogous methyltin compound MeSn(SPy)$_3$, the Sn—N distances are similar [2.4159 (5)–2.5781 (4) Å (Schmiedgen, 1992)].

The covalent Sn—S bond distances [2.486 (3)–2.571 (3) Å] are essentially the same as in Sn(SPy$_4$).HSPy [2.463 (2)–2.542 (2) Å], ^{n}Bu$_2$Sn(2-SPy-5-NO$_2$)$_2$ [2,477 (1) Å], MeSn(SPy)$_3$ [2.4793 (5)–2.5857 (4) Å], PhSn(ptc)$_3$ (ptc = 1-pyrrolthiocarboxylato) [2.448 (1)–2.538 (1) Å (Srivastava, Gupta, Nöth & Rattay, 1988)] and ^{n}BuSn(S$_2$CNEt$_2$)$_3$ [2.491 (7)–2.820 (7) Å (Morris & Schlemper, 1979)].

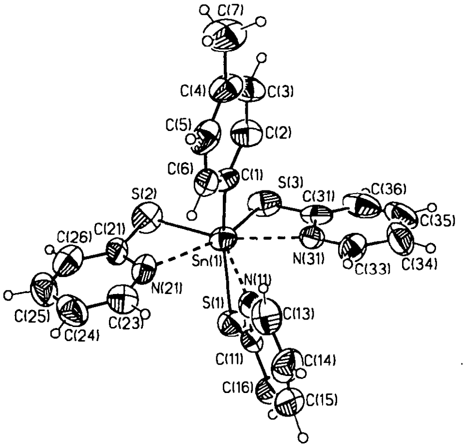


Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of (p-tolyl)Sn(SPy)$_3$, showing the atom-numbering scheme.

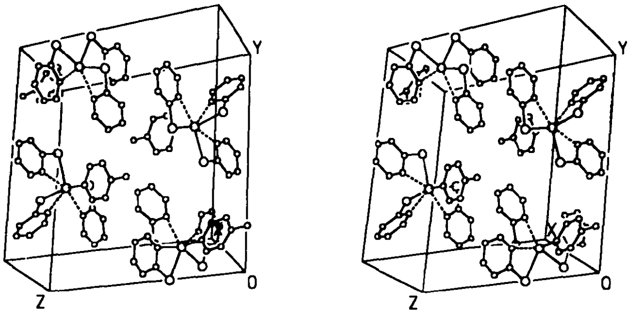


Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of the unit cell of (p-tolyl)Sn(SPy)$_3$ (H atoms are omitted for clarity).

The S—Sn—N bite angles [61.9 (2)–63.3 (2)°] are similar to those in Ph$_2$Sn(SPy)$_2$ [60.7 (8)°] and in ^{n}BuSn(SPym)$_3$ [60.9 (4)–63.3 (4)°]; they tend to be smaller than those in Sn(SPy$_4$).HSPy[64.2 (2)–65.1 (2)°].

Experimental

Crystal data

[Sn(C$_5$H$_4$NS)$_3$(C$_7$H$_7$)]
M_r = 540.28
Monoclinic
$P2_1/n$
a = 8.848 (3) Å
b = 16.139 (3) Å
c = 16.465 (4) Å
β = 103.64 (2)°
V = 2285 Å^3
Z = 4
D_x = 1.571 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 50 reflections
θ = 7.5–12.8°
μ = 1.406 mm^{-1}
T = 291 (1) K
Prism
0.16 × 0.14 × 0.14 mm
Light yellow

Data collection

Nicolet *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min}$ = 0.90, $T_{\max}$ = 0.97
7070 measured reflections
4047 independent reflections
1887 observed reflections [$F \geq 2\sigma(F)$]

$R_{\rm int}$ = 0.018
$\theta_{\max}$ = 25°
$h = -10 \rightarrow 4$
$k = -1 \rightarrow 19$
$l = -19 \rightarrow 19$
6 standard reflections frequency: 150 min intensity variation: <3.8%

Refinement

Refinement on F
R = 0.0485
wR = 0.0393
S = 0.6
1887 reflections
264 parameters
Only H-atom U's refined
$w = \sigma^2(F) + 0.0003F^2$

$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.66 e Å^{-3}
$\Delta\rho_{\min}$ = −0.44 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The programs used were *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PLATON* (Spek, 1990) and *MISSYM* (Le Page, 1987). All H atoms were placed in geometrically calculated positions (C—H 0.96 Å) and refined with common isotropic temperature factors for different C—H types.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn(1)	0.12796 (8)	0.12104 (4)	0.29360 (4)	0.0497
S(1)	0.1001 (3)	0.1595 (2)	0.4355 (1)	0.0575
S(2)	−0.1513 (4)	0.0691 (2)	0.2351 (2)	0.0718
S(3)	0.1927 (4)	−0.0312 (2)	0.3330 (2)	0.0704
C(1)	0.203 (1)	0.1354 (6)	0.1815 (5)	0.0455
C(2)	0.264 (1)	0.0682 (6)	0.1465 (6)	0.0583
C(3)	0.308 (1)	0.0737 (7)	0.0719 (6)	0.0677
C(4)	0.294 (1)	0.1481 (7)	0.0271 (6)	0.0622
C(5)	0.239 (1)	0.2158 (7)	0.0627 (6)	0.0647
C(6)	0.195 (1)	0.2087 (6)	0.1394 (5)	0.0538
C(7)	0.337 (1)	0.1524 (7)	−0.0565 (6)	0.0924
C(11)	0.218 (1)	0.2438 (6)	0.4264 (5)	0.0481
N(11)	0.2519 (9)	0.2530 (5)	0.3528 (4)	0.0434
C(13)	0.341 (1)	0.3162 (7)	0.3415 (6)	0.0637
C(14)	0.402 (1)	0.3737 (7)	0.4024 (7)	0.0722
C(15)	0.369 (1)	0.3633 (7)	0.4791 (7)	0.0760
C(16)	0.275 (1)	0.3001 (7)	0.4924 (6)	0.0652
C(21)	−0.197 (1)	0.1726 (7)	0.2250 (5)	0.0526
N(21)	−0.0784 (9)	0.2250 (6)	0.2460 (4)	0.0515
C(23)	−0.102 (1)	0.3079 (7)	0.2405 (6)	0.0588
C(24)	−0.247 (2)	0.3403 (7)	0.2133 (6)	0.0719
C(25)	−0.372 (1)	0.2870 (9)	0.1906 (6)	0.0745
C(26)	−0.347 (1)	0.2031 (8)	0.1981 (6)	0.0671
C(31)	0.381 (1)	0.0036 (7)	0.3688 (5)	0.0576
N(31)	0.397 (1)	0.0866 (5)	0.3620 (4)	0.0478
C(33)	0.537 (1)	0.1209 (7)	0.3840 (5)	0.0598
C(34)	0.668 (1)	0.0783 (8)	0.4194 (6)	0.0767
C(35)	0.650 (2)	−0.0080 (9)	0.4269 (7)	0.0867
C(36)	0.512 (2)	−0.0430 (8)	0.4039 (7)	0.0809

Table 2. *Selected geometric parameters* (Å, °)

Sn(1)—S(1)	2.486 (3)	S(2)—C(21)	1.72 (1)
Sn(1)—S(2)	2.571 (3)	S(3)—C(31)	1.72 (1)
Sn(1)—S(3)	2.571 (3)	C(11)—N(11)	1.32 (1)
Sn(1)—C(1)	2.119 (9)	N(11)—C(13)	1.33 (1)
Sn(1)—N(11)	2.487 (7)	C(21)—N(21)	1.33 (1)
Sn(1)—N(21)	2.468 (8)	N(21)—C(23)	1.35 (1)
Sn(1)—N(31)	2.444 (8)	C(31)—N(31)	1.36 (1)
S(1)—C(11)	1.74 (1)	N(31)—C(33)	1.33 (1)
N(21)—Sn(1)—N(31)	150.2 (3)	S(1)—Sn(1)—N(21)	84.2 (2)
N(11)—Sn(1)—N(31)	73.9 (3)	S(1)—Sn(1)—N(11)	63.3 (2)
N(11)—Sn(1)—N(21)	76.5 (3)	S(1)—Sn(1)—C(1)	156.0 (3)
C(1)—Sn(1)—N(31)	87.4 (3)	S(1)—Sn(1)—S(3)	93.86 (9)
C(1)—Sn(1)—N(21)	90.6 (3)	S(1)—Sn(1)—S(2)	97.7 (1)
C(1)—Sn(1)—N(11)	92.7 (3)	Sn(1)—S(1)—C(11)	85.4 (3)
S(3)—Sn(1)—N(31)	62.1 (2)	Sn(1)—S(2)—C(21)	84.5 (4)
S(3)—Sn(1)—N(21)	146.5 (2)	Sn(1)—S(3)—C(31)	85.1 (4)
S(3)—Sn(1)—N(11)	131.9 (2)	Sn(1)—C(1)—C(6)	123.1 (7)
S(3)—Sn(1)—C(1)	103.1 (3)	Sn(1)—C(1)—C(2)	120.4 (6)
S(2)—Sn(1)—N(31)	147.5 (2)	Sn(1)—N(11)—C(11)	95.1 (5)
S(2)—Sn(1)—N(21)	61.9 (2)	Sn(1)—N(11)—C(13)	145.4 (6)
S(2)—Sn(1)—N(11)	136.2 (2)	Sn(1)—N(21)—C(21)	97.5 (7)
S(2)—Sn(1)—C(1)	100.5 (2)	Sn(1)—N(21)—C(23)	141.5 (7)
S(2)—Sn(1)—S(3)	85.42 (9)	Sn(1)—N(31)—C(31)	98.7 (6)
S(1)—Sn(1)—N(31)	85.7 (2)	Sn(1)—N(31)—C(33)	140.9 (7)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71485 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1055]

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Acta Cryst. (1994). **C50**, 209–211

Synthèse et Structure du Tétrachloroplatinate(II) de Pyrido[2,3-*h*]pyrrolo[1,2-*a*]quinoxalinium Monohydrate, $[C_{14}H_{11}N_3][PtCl_4].H_2O$

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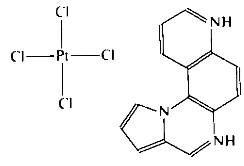
Abstract

The structure of the title compound, $5H^+,8H^+$-pyrido[2,3-*h*]pyrrolo[1,2-*a*]quinoxalinium tetrachloroplatinate monohydrate, consists of a square planar $(PtCl_4)^{2-}$ anion and a $PPQH_2^{2+}$ cation, where $PPQH_2^+$ is the protonated organic ligand. The protonation on the N(5) and N(8) atoms gives rise to an opening of the angles C(4)—N(5)—C(14) and C(9)—N(8)—C(15) when compared with the homologous values in the neutral PPQ form. Although each ring of the $PPQH_2^{2+}$ ion is almost planar to within experimental error, the ion as a whole is not, as shown by the value of the dihedral angle [18.8 (2)°] between the mean planes of the pyrrole (*P*1) and the pyridine (*P*4). A hydrogen bond between the water O atom and a $PPQH_2^{2+}$ N atom exists.

Commentaire

La découverte par Abrams *et al.* (1986) de propriétés antitumorales de sels d'anions complexes du platine(II) avec la rhodamine 123 nous a incités à synthétiser des sels d'anions complexes de tétrachloroplatinate(II) avec des molécules polyhétérocycliques à activité antitumorale potentielle, en particulier le pyrido[2,3-*h*]pyrrolo[1,2-*a*]quinoxaline ou PPQ.

La réaction du tétrachloroplatinate(II) de potassium (10^{-4} mol) avec une solution chlorhydrique (0,2 mol l^{-1}) de PPQ dans un rapport K_2PtCl_4/PPQ = 1/4 conduit à la formation d'un sel d'anion complexe: le tétrachloroplatinate(II) de pyrido[2,3-*h*]pyrrolo[1,2-*a*]quinoxalinium $(PPQH_2)^{2+}$, (I). L'analyse centésimale a révélé la présence d'une molécule d'eau. La cristallisation a été obtenue par évaporation lente de la solution chlorhydrique. Les cristaux obtenus sont de couleur jaune pâle.



(I)

Les atomes de chlore liés au platine forment un quadrilatère qui, compte-tenu des incertitudes, est assimilable à un carré. En effet, les distances Pt—Cl sont comprises entre 2,288 (1) et 2,312 (1) Å et l'angle varie de 89,11 (6) à 91,07 (5)°. On peut noter que ces valeurs sont comparables à celles observées dans le tétrachloroplatinate(II) de *trans*-dichlorobis(propanediamine-1,3)-platinate(IV) (Delafontaine, Toffoli, Khodadad & Rodier, 1988).

Dans l'ion $PPQH_2^{2+}$, les conformations respectives des cycles pyrrolique (*A*, plan moyen *P*1), pyrazinique (*B*, plan *P*2), benzénique (*C*, plan *P*3) et pyridinique (*D*, plan *P*4) ne diffèrent pas de façon significative de celles décrites dans le complexe neutre PPQ-TCNQ (TCNQ désigne le 7,7,8,8-tétracyanoquinodiméthane) (Viossat, Nguyen-Huy Dung, Daran, Lancelot & Robba, 1988). La protonation au niveau des atomes d'azote N(5) et N(8) induit une ouverture plus grande des angles C(4)—N(5)—C(14) et C(9)—N(8)—C(15) (environ 7°, Tableau 2) par rapport aux valeurs homologues dans le complexe PPQ-TCNQ [115,8 (3) et 117,4 (3)°, respectivement]. Les valeurs des autres angles et distances sont très comparables à l'exception d'un raccourcissement

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