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

U_{iso} for C12–C19; $U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	U_{eq}
Sn	0	0.34721 (5)	1/4	0.0192 (2)
S	−0.08497 (14)	0.3195 (2)	0.49334 (9)	0.0596 (5)
N1	−0.0316 (3)	0.3479 (4)	0.3620 (2)	0.0276 (10)
C1	−0.0538 (3)	0.3369 (5)	0.4164 (3)	0.0253 (12)
C2	0	0.1315 (7)	1/4	0.021 (2)
C3	0.0138 (3)	0.0571 (5)	0.3125 (3)	0.0267 (12)
C4	0.0150 (3)	−0.0819 (6)	0.3130 (3)	0.0346 (13)
C5	0	−0.1512 (9)	1/4	0.043 (2)
C6	0.1190 (3)	0.4556 (5)	0.2838 (2)	0.0196 (10)
C7	0.1208 (3)	0.5559 (5)	0.3343 (2)	0.0251 (11)
C8	0.1997 (3)	0.6266 (5)	0.3569 (3)	0.0319 (13)
C9	0.2754 (3)	0.5959 (6)	0.3289 (3)	0.0337 (13)
C10	0.2740 (3)	0.4967 (6)	0.2784 (3)	0.0364 (14)
C11	0.1962 (3)	0.4284 (6)	0.2556 (3)	0.0344 (13)
N2	−1/4	3/4	1/2	0.038 (2)
C12†	−0.2432 (7)	0.7474 (10)	0.5806 (3)	0.045 (3)
C13†	−0.2986 (15)	0.860 (2)	0.6051 (8)	0.116 (9)
C14†	−0.2461 (7)	0.8920 (7)	0.4796 (6)	0.051 (3)
C15†	−0.1710 (9)	0.9723 (11)	0.5179 (7)	0.056 (4)
C16†	−0.3392 (5)	0.7006 (10)	0.4731 (6)	0.050 (3)
C17†	−0.3657 (9)	0.5682 (13)	0.5027 (8)	0.065 (4)
C18†	−0.1801 (7)	0.6679 (13)	0.4816 (6)	0.080 (5)
C19†	−0.1677 (13)	0.682 (2)	0.4028 (7)	0.086 (6)

† Partial occupancy (see *Comment*).

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

Sn—C6	2.135 (4)	S—C1	1.622 (6)
Sn—C2	2.142 (7)	N1—C1	1.147 (7)
Sn—N1	2.268 (5)		
C6—Sn—C6[i]	119.4 (3)	C2—Sn—N1	90.19 (11)
C6—Sn—C2	120.28 (13)	N1[i]—Sn—N1	179.6 (2)
C6—Sn—N1[i]	90.2 (2)	C1—N1—Sn	172.5 (4)
C6—Sn—N1	89.6 (2)	N1—C1—S	179.3 (5)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Data were corrected for Lorentz and polarization effects but not for absorption. The diffractometer was fitted with an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986). Absence of crystal decay in the X-ray beam was confirmed by checking equivalent reflections at the beginning and end of the data collection period of *ca* 8 h. All non-H atoms were refined with anisotropic displacement parameters, except the C atoms of the disordered ethyl substituents in the Et_4^+ cation which were refined isotropically with C—N and C—C bond lengths restrained to 1.47 (1) and 1.52 (1) Å, respectively.

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994).

The use of the EPSRC X-ray Crystallographic Service at the University of Wales, Cardiff, is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1025). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chloro(ethylenediamine)(6-phenylimidazo-[2,1-*b*]thiazole-N^7)platinum(II) Nitrate

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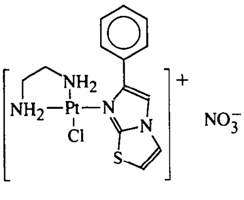
Abstract

Platination of 6-phenylimidazo[2,1-*b*]thiazole at the imidazole N atom to give chloro(ethylenediamine)(6-phenylimidazo[2,1-*b*]thiazole)platinum(II) nitrate, $[PtCl(C_2H_8N_2)(C_{11}H_8N_2S)]NO_3$, is accompanied by a rotation of 49.3 (8)° of the phenyl ring and a loss of extended conjugation in the normally planar 6-phenylimidazo[2,1-*b*]thiazole molecule.

Comment

Cisplatin, *cis*-$[Pt(NH_3)_2Cl_2]$, has long been used in the treatment of various forms of cancer (Loehrer & Einhorn, 1984). The reported activities of both platinum triamine complexes (Hollis, Amundsen & Stern, 1989) and platinum imidazole and thiazole compounds (van Beusichem & Farrell, 1992) in preliminary antitumor screens imply that additional structure–activity relationships for platinum-based therapeutic agents should be established. Therefore, we prepared a series of platinum triamine imidazothiazole complexes as part of a program to map out the relationships between structure and antitumor activity for this new class of potential chemotherapeutic agents (Arvanitis, Berardini, Parkinson & Schneider, 1993). The title compound, (I), is a member of that series.



(I)

Compound (I) is a square-planar Pt triamine complex of 6-phenylimidazo[2,1-*b*]thiazole. A displacement ellipsoid plot is given in Fig. 1 and a packing diagram in Fig. 2. The bond distances and angles at the Pt^{II} atom are normal, accounting for slight distortions to accommodate the chelating ethylenediamine moiety (Iball & Scrimgeour, 1974; Lippert, Lock & Speranzini, 1981). The imidazo[2,1-*b*]thiazole ring is planar and the dihedral angle between the Pt-coordination plane and the imidazothiazole plane is 65.4°. The Pt atom is positioned 0.30 Å out of the imidazothiazole best plane.

The most interesting structural feature is the orientation of the phenyl substituent. In the free ligand, the phenyl and imidazothiazole rings are coplanar due to conjugation effects (Cavalca, Domiano & Musatti, 1972). This coplanarity is compromised in compound (I) in favor of minimizing the Pt to *ortho*-H nonbonded contacts. The result is a substantial increase in the phenyl–imidazothiazole torsion angle [−0.8° in the free ligand *versus* 49.3 (8)° in compound (I)] and a slight lengthening of the phenyl–imidazothiazole bond [1.450 (7) *versus* 1.483 (7) Å]. The latter approaches that of the platinum complex with the saturated derivative 6-phenyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazole (Arvanitis *et al.*, 1993). Both observations are consistent with a slight loss of conjugation between the phenyl and imidazothiazole rings upon complexation. Biological assays and structural studies on additional platinum triamine imidazothiazole derivatives are in progress to correlate these spatial and electronic changes with variations in anticancer activity.

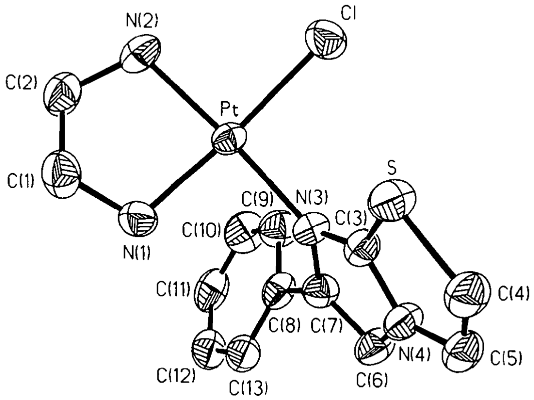


Fig. 1. Structure and labeling scheme for $[PtCl(C_2H_8N_2)(C_{11}H_8N_2S)]NO_3$ showing 50% probability displacement ellipsoids.

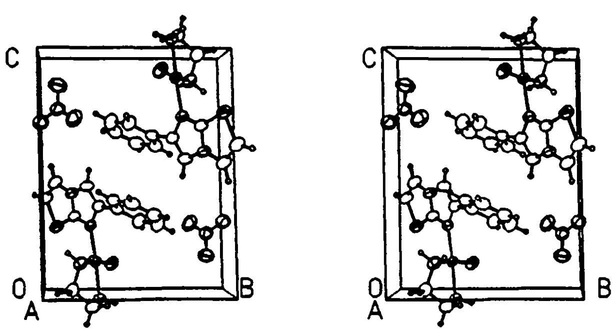


Fig. 2. Contents of the unit cell.

Experimental

The procedure used for synthesis is a modification of the methods of Lippert (Lippert *et al.*, 1981) and Hollis (Hollis *et al.*, 1989). 300 mg (1 mmol) of $PtCl_2(C_2H_8N_2)$ was dissolved in 20 ml of dimethylformamide (DMF). 169 mg (1 mmol) of $AgNO_3$ was added and the mixture was stirred overnight in the dark. The mixture was filtered to remove AgCl and 200 mg (1 mmol) of 6-phenylimidazo[2,1-*b*]thiazole in 10 ml of DMF was added to the filtrate. After 12 h the solvent was removed under reduced pressure and the resulting product was washed with CH_2Cl_2 and recrystallized from water. The product was then dissolved in a minimum amount of hot water, loaded onto a Waters Sep-Pak cartridge (C18) and eluted with 10 ml of water, 5 ml of a 10% methanol solution, and 5 ml of a 20% methanol solution. The eluate was collected in 1 ml increments. The solvent was allowed to evaporate from the fractions. An increment eluted with the 10% methanol solution yielded colorless needle-like crystals. The crystal density D_m was obtained by suspension in a CCl_4–$CHBr_3$ mixture.

Crystal data

$[PtCl(C_2H_8N_2)(C_{11}H_8N_2S)]$-$NO_3$
$M_r = 552.91$
Triclinic
$P\bar{1}$
$a = 6.872 (1)$ Å
$b = 9.960 (2)$ Å
$c = 12.782 (2)$ Å
$\alpha = 91.72 (1)°$
$\beta = 92.14 (1)°$
$\gamma = 94.31 (1)°$
$V = 871.3 (2)$ Å^3
$Z = 2$
$D_x = 2.108$ Mg m^{-3}
$D_m = 2.055$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15.0$–$18.5°$
$\mu = 8.347$ mm^{-1}
$T = 296 (2)$ K
Needle
$0.25 \times 0.15 \times 0.10$ mm
Colorless

Data collection

Siemens *P*4 diffractometer
ω scans

$R_{int} = 0.0168$
$\theta_{max} = 25.05°$

Absorption correction: ψ scan
T_{min} = 0.0898, T_{max} = 0.1387
3360 measured reflections
3078 independent reflections
2773 observed reflections [$I > 2\sigma(I)$]
$h = 0 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0230$
$wR(F^2) = 0.0502$
$S = 1.077$
3078 reflections
217 parameters
H atoms refined as riding
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.638$ e Å^{-3}
$\Delta\rho_{min} = -0.829$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Pt	0.05135 (3)	0.28862 (2)	0.11701 (1)	0.03267 (7)
Cl	−0.2444 (2)	0.3823 (1)	0.0999 (1)	0.0500 (3)
N1	0.3091 (6)	0.2016 (4)	0.1199 (3)	0.045 (1)
C1	0.3444 (9)	0.1550 (6)	0.0108 (4)	0.054 (1)
C2	0.2940 (9)	0.2631 (6)	−0.0622 (4)	0.053 (1)
N2	0.0962 (7)	0.3028 (4)	−0.0382 (3)	0.045 (1)
N3	0.0199 (6)	0.2627 (4)	0.2715 (3)	0.0359 (9)
C3	−0.1110 (7)	0.1745 (5)	0.3104 (4)	0.038 (1)
S	−0.3066 (2)	0.0710 (1)	0.2618 (1)	0.0462 (3)
C4	−0.3393 (8)	0.0127 (6)	0.3878 (4)	0.051 (1)
C5	−0.2107 (8)	0.0695 (5)	0.4594 (4)	0.047 (1)
N4	−0.0799 (6)	0.1630 (4)	0.4143 (3)	0.041 (1)
C6	0.0814 (8)	0.2482 (5)	0.4449 (4)	0.045 (1)
C7	0.1421 (7)	0.3087 (5)	0.3566 (4)	0.038 (1)
C8	0.3113 (8)	0.4080 (5)	0.3452 (4)	0.041 (1)
C9	0.2855 (9)	0.5273 (5)	0.2945 (4)	0.053 (1)
C10	0.4442 (9)	0.6204 (6)	0.2854 (5)	0.061 (2)
C11	0.6243 (9)	0.5949 (6)	0.3246 (4)	0.056 (2)
C12	0.6504 (8)	0.4776 (6)	0.3744 (4)	0.053 (1)
C13	0.4927 (8)	0.3836 (5)	0.3866 (4)	0.045 (1)
N5	0.1489 (7)	−0.0980 (5)	0.2393 (4)	0.048 (1)
O1	0.2435 (6)	−0.0079 (4)	0.2920 (3)	0.064 (1)
O2	0.0363 (6)	−0.1817 (4)	0.2801 (4)	0.071 (1)
O3	0.1696 (9)	−0.1018 (5)	0.1440 (4)	0.094 (2)

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

Pt—N3	2.017 (4)	C3—N4	1.347 (6)
Pt—N2	2.026 (4)	C3—S	1.716 (5)
Pt—N1	2.029 (4)	S—C4	1.745 (5)
Pt—Cl	2.305 (1)	C4—C5	1.331 (7)
N1—C1	1.491 (7)	C5—N4	1.397 (6)
C1—C2	1.497 (8)	N4—C6	1.382 (6)
C2—N2	1.484 (7)	C6—C7	1.360 (7)
N3—C3	1.330 (6)	C7—C8	1.483 (7)
N3—C7	1.393 (6)		
N3—Pt—N2	175.6 (2)	N3—C3—N4	111.4 (4)
N3—Pt—N1	92.0 (2)	N3—C3—S	136.3 (4)
N2—Pt—N1	84.0 (2)	N4—C3—S	112.3 (3)
N3—Pt—Cl	92.0 (1)	C3—S—C4	88.8 (2)
N2—Pt—Cl	91.9 (1)	C5—C4—S	113.8 (4)
N1—Pt—Cl	175.5 (1)	C4—C5—N4	111.0 (5)
C1—N1—Pt	107.6 (3)	C3—N4—C6	107.6 (4)
N1—C1—C2	108.5 (4)	C3—N4—C5	114.0 (4)
N2—C2—C1	108.1 (4)	C6—N4—C5	138.3 (4)
C2—N2—Pt	110.4 (3)	C7—C6—N4	106.0 (4)
C3—N3—C7	105.3 (4)	C6—C7—N3	109.7 (4)
C3—N3—Pt	123.9 (3)	C6—C7—C8	128.3 (5)
C7—N3—Pt	129.8 (3)	N3—C7—C8	122.0 (4)

Data collection: Siemens *P*3VAX system. Cell refinement: Siemens *P*3VAX system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dipyridiniomethane *cis*- and *trans*-Difluorotetrachloroosmate(IV), *cis*- and *trans*-$[(C_5H_5N)_2CH_2][OsCl_4F_2]$

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Abstract

In the structures of *N*,*N*′-methylenedipyridinium *cis*-difluorotetrachloroosmate(IV), (I), and *N*,*N*′-methylenedipyridinium *trans*-difluorotetrachloroosmate(IV),