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trans-Bis{2-chloro-6-[(3-hydroxybenzyl)amino]-9-isopropylpurine-κN^7}platinum(II) dimethylformamide disolvate

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Key indicators

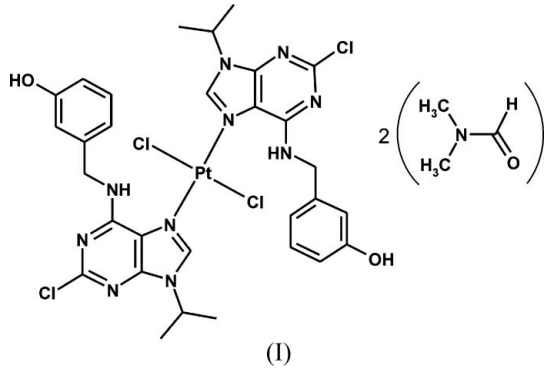
Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.007 Å
R factor = 0.034
wR factor = 0.086
Data-to-parameter ratio = 13.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, *trans*-$[Pt^{II}Cl_2(C_{15}H_{16}ClN_5O)_2]\cdot 2C_3H_7NO$, is centrosymmetric, with square-planar coordination of the Pt atom within a *trans*-Cl_2N_2 donor set. The complex is connected through O—H···O hydrogen bonds to two dimethylformamide solvent molecules. The complex is the first structural example of a Pt^{II} complex involving two coordinated cyclin-dependent kinase inhibitors, *viz.* 2-chloro-6-[(3-hydroxybenzyl)amino]-9-isopropylpurine.

Comment

The development of anti-cancer Pt^{II} or generally antineoplastic transition metal complexes originated in the 1960s after the discovery of the unexpected biological activity of *cis*-$Pt(NH_3)_2Cl_2$ (Cisplatin) (Rosenberg *et al.*, 1965). In our opinion, the antitumour action of metal-based complexes might be improved by the utilization of potentially active molecules, such as *N*-donor ligands. Thus, cyclin-dependent kinase (CDK) inhibitors derived from 6-benzylaminopurine might be successfully employed as such ligands, as we have recently demonstrated (*e.g.* Trávníček *et al.*, 2003, 2005, 2006; Szüčová *et al.*, 2006). To date, only one structure of a Pt complex with a 6-benzylaminopurine CDK inhibitor has been determined (Trávníček *et al.*, 2003). In this paper, we report the structural characterization of the first platinum complex, (I), containing two CDK inhibitors, *viz.* two 2-chloro-6-[(3-hydroxybenzyl)amino]-9-isopropylpurine molecules (*L*), as ligands.



(I)

The structure of (I) (Fig. 1 and Table 1) comprises a centrosymmetric *trans*-$[PtCl_2L_2]$ complex connected through O—H···O hydrogen bonds to two dimethylformamide molecules. The Pt^{II} atom, lying on a centre of symmetry, is coordinated by two Cl atoms and two *L* ligands through the N7 atoms of the adenine units, forming a *trans*-square-planar coordination around the central atom. The Pt—N and Pt—Cl bond distances are comparable with the average bond lengths

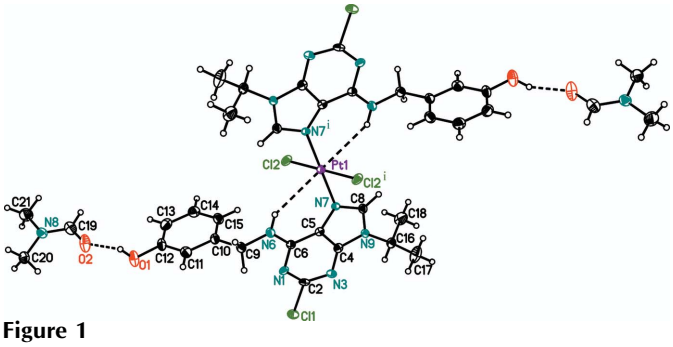


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds and the intermolecular N—H···Pt interactions. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

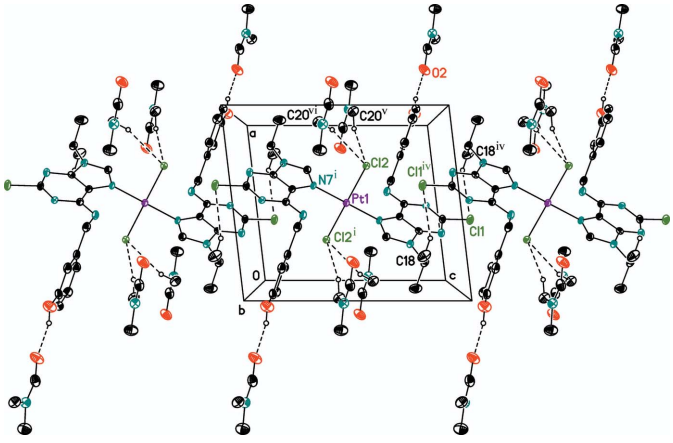


Figure 2
A view of the crystal packing of (I), showing C—H···Cl interactions between neighbouring complexes. Dashed lines indicate intermolecular hydrogen bonds. [Symmetry codes: (i) $1-x, 1-y, 1-z$; (iv) $1-x, 1-y, 2-z$; (v) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (vi) $\frac{5}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$.]

of 2.02 and 2.29 Å, respectively, found in related $PtCl_2N_2$ complexes reported in the Cambridge Structural Database (Version 5.27.2; Allen, 2002).

The Pt and N6 atoms both deviate significantly from the planarity of the purine ring system, with out-of-plane deviations of 0.1996 (1) and 0.135 (4) Å, respectively, and the almost planar six-membered (pyrimidine) and five-membered (imidazole) rings of purine form an angle of 3.5 (1)°.

The relatively short intermolecular N6—H···Pt distance of 2.76 Å, in comparison with distances in the range 2.88–2.90 Å observed in *trans*-(dichloro)-bis(creatinine)platinum(II) (Matos Beja *et al.*, 1991; Ramos Silva *et al.*, 1996) and *trans*-bis(2-amino-1-methyl-1,5-dihydro-4*H*-imidazol-4-one-*N*′)-(dichloro)platinum(II) (Lynch & Duckhouse, 2001), indicates a moderate N—H···Pt interaction operating in (I). The least-squares planes of the hydroxybenzyl group and purine systems form a dihedral angle of 88.11 (12)°.

The secondary structure of (I) is stabilized by intermolecular O—H···O hydrogen bonds involving both hydroxybenzyl- and dimethylformamide-O atoms, and by intermolecular C—H···Cl interactions, as detailed in Table 2 and illustrated in Fig. 2.

Experimental

2-Chloro-6-[(3-hydroxybenzyl)amino]-9-isopropylpurine (*L*) was prepared by the method described in the literature (Kryštof *et al.*, 2002). The title complex, (I), was synthesized as follows. The organic ligand *L* (1.0 mmol) was dissolved in EtOH (20 ml) and then added to a mixture of K_2PtCl_4 (0.5 mmol) in EtOH (15 ml). The reaction mixture was heated to 343 K and stirred for 72 h. The solution was then filtered and left to stand at room temperature for three weeks. The solid which formed was filtered off and single crystals of (I) suitable for X-ray analysis were obtained by recrystallization of the sample from a solution in dimethylformamide.

Crystal data

$[PtCl_2(C_{15}H_{16}ClN_5O)_2]\cdot 2C_3H_7NO$
$M_r = 1047.74$
Monoclinic, $P2_1/n$
$a = 9.4136$ (4) Å
$b = 20.0628$ (10) Å
$c = 10.9064$ (5) Å
$\beta = 96.723$ (5)°
$V = 2045.65$ (16) Å^3
$Z = 2$
$D_x = 1.701$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.75$ mm^{-1}
$T = 105$ (2) K
Prism, yellow
0.5 × 0.45 × 0.3 mm

Data collection

Oxford Xcalibur2 diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.151$, $T_{max} = 0.321$
12305 measured reflections
3588 independent reflections
3395 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.086$
$S = 1.28$
3588 reflections
264 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.00$ e Å^{-3}
$\Delta\rho_{min} = -0.74$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—N7	2.017 (4)	Pt1—Cl2	2.3052 (12)
N7—Pt1—Cl2	88.94 (12)	N7—Pt1—Cl2^{i}	91.06 (12)
C9—N6—C6—C5	−174.4 (4)	C6—N6—C9—C10	−89.7 (6)
Cl2—Pt1—N7—C5	−59.3 (4)	N6—C9—C10—C11	−179.5 (4)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.84	1.84	2.637 (6)	159
C20—H20C···Cl2^{ii}	0.98	2.74	3.660 (6)	156
C20—H20A···Cl2^{iii}	0.98	2.83	3.706 (6)	149
C18—H18A···Cl1^{iv}	0.98	2.90	3.755 (6)	146

Symmetry codes: (ii) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $-x+1, -y+1, -z+2$.

H atoms were included in the riding-model approximation, with C—H = 0.95–1.00 Å, N—H = 0.88 Å and O—H = 0.84 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N,O)$. The highest unassigned difference Fourier peak of 1.00 e Å^{-3} is located 1.04 Å from the Pt atom.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Johnson & Burnett, 1996); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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