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cis-Diamminechloro(2,5-dimethylbenzoxazole-N^1)platinum(II) Nitrate

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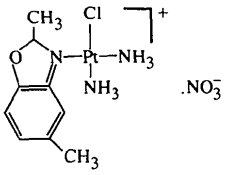
Abstract

In the title compound, $[PtCl(NH_3)_2(C_9H_9NO)]NO_3$, the complex cation features square-planar coordination around the Pt atom, with a mean deviation of 0.0007 (5) Å. The aromatic 2,5-dimethylbenzoxazole ring is planar with a mean deviation of 0.008 (8) Å. The dihedral angle between these two ring planes is 71.0 (2)°.

Comment

Triamino–Pt^{II} complexes of the form *cis*-$[PtCl(NH_3)_2L]$ with N-donor heterocyclic *L* ligands have been studied for their potential antitumor activities (Hollis, Amundsen & Stern, 1981). The ligands used for such complexes are often imidazoles, thiazoles and benzoxazoles (Gomez *et al.*, 1988). Modifications to these compounds may lead to a better understanding of the role that the ligand plays in improving the antitumor activity and water solubility of the complexes (Muir *et al.*, 1992). 2,5-Dimethylbenzoxazole (Me_2BO) is potentially an ambidentate ligand and has been reported to be coordinated through its O atom to Pt^{II} in the $[PtCl_2(Me_2BO)_2]$ complex based on IR spectroscopic data (Massacessi, Pinna & Ponticelli, 1981). We synthesized the title compound, (I), in order to clarify the coordination mode of the ligand.



(I)

The X-ray crystallographic analysis revealed that 2,5-dimethylbenzoxazole coordinates to the Pt^{II} center through the N-donor atom. The crystal lattice consists of NO_3^- anions and $[PtCl(NH_3)_2(Me_2BO)]^+$ cations linked by hydrogen bonds between the nitrate ion and coordinated ammonia molecules [N(3)···O(3) 2.917 (3) and N(2)···O(2) 3.081 (3) Å]. The $[PtCl(NH_3)_2(Me_2BO)]^+$ cation features square-planar coordination around the Pt atom, with a mean deviation of 0.0007 (5) Å (Fig. 1). Two ammonia ligands arrange themselves in a *cis* fashion. The Pt—N bond *trans* to the Cl atom exhibits a longer bond distance [2.042 (7) Å] than that *cis* to the Cl atom [2.007 (8) Å]. The aromatic Me_2BO ring is planar with a mean deviation of 0.008 (8) Å. The dihedral angle between these two planes is 71.0 (2)°. The other bond distances are in the normal range.

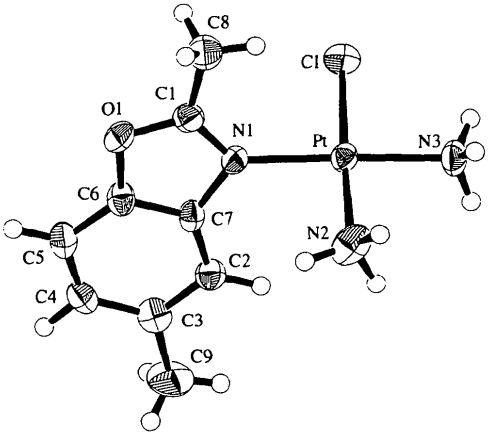


Fig. 1. An *ORTEP*II (Johnson, 1976) representation of the title compound showing 50% probability displacement ellipsoids.

Experimental

The detailed synthesis of the title compound will be published elsewhere. Slow evaporation of a dichloromethane solution of the compound at room temperature afforded pale-yellow single crystals of X-ray quality.

Crystal data

$[PtCl(NH_3)_2(C_9H_9NO)]NO_3$
M_r = 473.78

Mo $K\alpha$ radiation
λ = 0.7107 Å

Orthorhombic
$Pna2_1$
a = 7.675 (1) Å
b = 25.400 (7) Å
c = 7.270 (2) Å
V = 1417.3 (5) Å^3
Z = 4
D_x = 2.220 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 10.0–15.0°
μ = 10.064 mm^{-1}
T = 295.2 K
Platelet
0.30 × 0.30 × 0.25 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.052, T_{max} = 0.081
2811 measured reflections
2406 independent reflections

1803 reflections with $I > 3\sigma(I)$
R_{int} = 0.030
θ_{max} = 29.96°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 35$
$l = -10 \rightarrow 10$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.029
wR = 0.033
S = 1.282
1803 reflections
172 parameters
H atoms not refined
$w = 1/\sigma^2(F_o) = 1/[\sigma^2{}_c(F_o) + p^2/4F_o^2]$, where p = 0.010
$(\Delta/\sigma)_{max}$ = 0.003

$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −1.00 e Å^{-3}
Extinction correction: Zachariasen (1967) type 2, Gaussian isotropic
Extinction coefficient: 11.342 × 10^{-5}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Pt—Cl	2.294 (3)	O(1)—C(1)	1.34 (1)
Pt—N(1)	2.027 (7)	O(1)—C(6)	1.38 (1)
Pt—N(2)	2.042 (7)	N(1)—C(1)	1.30 (1)
Pt—N(3)	2.007 (8)	N(1)—C(7)	1.45 (1)
Cl—Pt—N(1)	92.3 (2)	N(2)—Pt—N(3)	88.5 (6)
Cl—Pt—N(2)	177.5 (5)	C(1)—O(1)—C(6)	106.2 (7)
Cl—Pt—N(3)	89.0 (2)	Pt—N(1)—C(1)	131.7 (7)
N(1)—Pt—N(2)	90.3 (6)	Pt—N(1)—C(7)	122.6 (6)
N(1)—Pt—N(3)	178.3 (3)	C(1)—N(1)—C(7)	105.7 (8)

The H atoms were either located from the Fourier difference electron-density maps or calculated, and included in the structure model. The largest hole in the final difference map was 4.11 Å from the N(3) atom and the largest peak was 0.90 Å from the Pt atom.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1991). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1154). Services for accessing these data are described at the back of the journal.

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Bis[μ-methylenebis(diphenylphosphine)-*P*:*P*′]disilver(I) Diperchlorate Bis(dichloromethane) Solvate

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Abstract

The title compound, $[Ag_2(C_{25}H_{22}P_2)_2](ClO_4)_2.2CH_2Cl_2$, contains eight-membered rings with crystallographic inversion symmetry, with linear coordination at silver [P—Ag—P 170.72 (4)°] and a short intra-annular Ag···Ag contact of 2.9532 (7) Å.

Comment

The use of dppm [bis(diphenylphosphino)methane] as a ligand in coinage metal chemistry often leads to complexes with central eight-membered rings of the form $[(dppm)_2M_2]$; particularly for copper and silver, additional interactions from the metal to the counterion may