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[4-(4-Methylphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole]bis(triphenylphosphine)-silver(I) perchlorate

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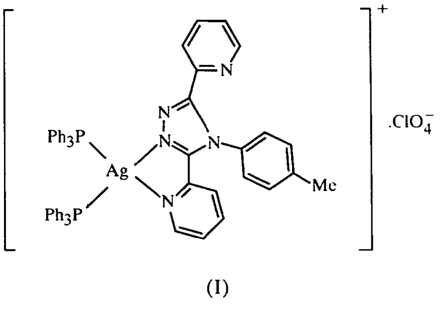
Abstract

The coordination geometry of silver(I) in the title compound, $[Ag(C_{19}H_{15}N_5)(C_{18}H_{15}P)_2]ClO_4$, is distorted tetrahedral. The pyridyl groups, the substituted benzene ring and the 1,2,4-triazole moiety do not share a common plane. The perchlorate ion is highly disordered and is involved in C—H···O hydrogen bonding.

Comment

The complexes of silver(I) and copper(I) containing phenylphosphine ligands were found to possess anticancer activity (Berners Price & Sadler, 1988; Berners Price *et al.*, 1990). As part of our ongoing study of substituted 1,2,4-triazole compounds (Fun *et al.*, 1999; Wang *et al.*, 1998; Chen *et al.*, 1998), and of their coordination behaviour and properties, we describe herein the structure of [4-(4-methylphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole]bis(triphenylphosphine)silver(I) perchlorate, (I).



(I)

The coordinating geometry of the Ag^I atom in (I) is distorted tetrahedral, with asymmetric Ag1—N1 and Ag1—N4 bond lengths of 2.308 (2) and 2.548 (2) Å, respectively. As can be seen, the Ag1—N4 bond length is greater than the Ag1—N1 bond length, but it is comparable to the value reported by Fu *et al.* (1997). The P2—Ag1—N4 and N1—Ag1—N4 bond angles of 99.90 (5) and 68.20 (7)°, respectively, deviate strikingly from the value of 109.5° expected for a normal tetrahedron. This distortion may arise from the steric hindrance of the bulky triphenylphosphine groups. The Ag—P distances of 2.438 (1) and 2.494 (1) Å in (I) are shorter than the Ag—P distances of 2.668 (5) and 2.659 (2) Å in $[Ag(PPh_3)_4]ClO_4$ (Engelhardt *et al.*, 1985).

The ligand is coordinated to Ag1 *via* the N1 atom of the triazole moiety and *via* the N4 atom of the neighbouring pyridine ring, leaving the corresponding N2 and N5 atoms uncoordinated. The pyridyl groups, the substituted benzene ring and the 1,2,4-triazole moiety do not share a common plane. The triazole ring makes dihedral angles of 17.0 (1) and 25.5 (2)° with the N4- and N5-containing pyridine rings, respectively, while the pyridine rings are inclined at an angle of 42.2 (2)° to one another. The interplanar angle between the triazole and phenyl rings is 68.3 (1)°. The perchlorate ion is highly disordered and is involved in C—H···O hydrogen bonding.

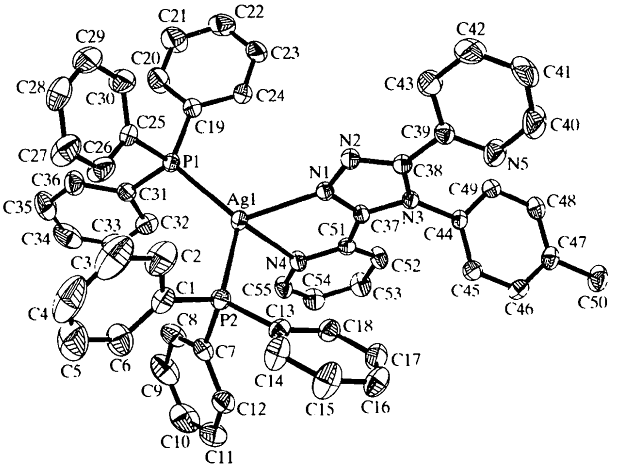


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme (the perchlorate ion and the H atoms have been omitted for clarity).

Experimental

The title compound was prepared by the reaction of equal amounts of 4-(*p*-methylphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole, $Ag(PPh_3)_2NO_3$ and $NaClO_4$ in methylene dichloride at room temperature. Single crystals suitable for X-ray analysis were obtained from acetone.

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Crystal data

$[Ag(C_{19}H_{15}N_5)(C_{18}H_{15}P)_2]$-$ClO_4$
M_r = 1045.22
Monoclinic
$P2_1/c$
a = 16.7610 (2) Å
b = 14.2287 (2) Å
c = 21.1305 (2) Å
β = 97.691 (1)°
V = 4994.02 (10) Å^3
Z = 4
D_x = 1.390 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 8192 reflections
θ = 1.23–28.36°
μ = 0.572 mm^{-1}
T = 293 (2) K
Slab
0.38 × 0.36 × 0.18 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.812, T_{max} = 0.904
33 642 measured reflections
12 270 independent reflections

8462 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 28.26°
$h = -22 \rightarrow 19$
$k = -15 \rightarrow 18$
$l = -28 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.047
12 270 reflections
641 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.330 e Å^{-3}
$\Delta\rho_{min}$ = −0.357 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

P1—C19	1.818 (3)	P2—C7	1.826 (3)
P1—C31	1.826 (3)	P2—C1	1.826 (3)
P1—C25	1.829 (3)	P2—C13	1.834 (3)
N1—Ag1—P1	120.04 (5)	P1—Ag1—P2	122.99 (2)
N1—Ag1—P2	111.87 (5)	P1—Ag1—N4	119.39 (5)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
C48—H48A···O4A^i	0.93	2.47	3.36 (1)	160
C49—H49A···O4A^{ii}	0.93	2.56	3.37 (1)	146

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

The perchlorate ion is highly disordered. The O2, O3 and O4 atoms were refined with 60 and 40% occupancies for the two conformers, while the O1 atom was refined with full occupancy.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1429). Services for accessing these data are described at the back of the journal.

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Diamminebis(2,4,6-trichlorophenolato)-copper(II)

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Abstract

The crystal structure of the title compound, $[Cu(C_6H_2Cl_3O)_2(NH_3)_2]$, has been determined by X-ray diffraction. The title monomeric centrosymmetric Cu^{II} complex