

catena-Poly[[[bis(cyclohexyldiphenylphosphine- κP)silver(I)]- μ -cyano- $\kappa^2 N:C$ -silver(I)- μ -cyano- $\kappa^2 C:N$] dichloromethane solvate]

Xiacong Xie, Bayrammurad Saparov and Glenn P. A. Yap*

Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716, USA

Correspondence e-mail: gpyap@udel.edu

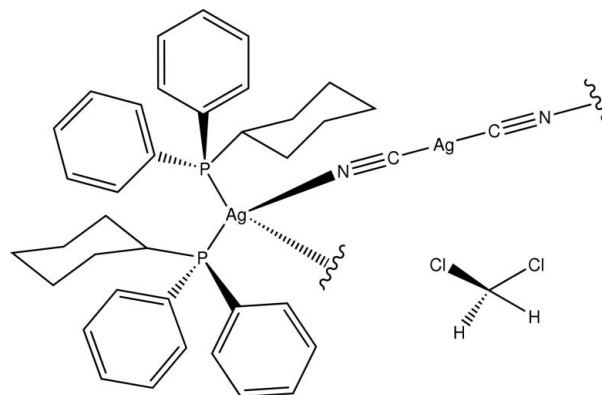
Received 4 June 2007; accepted 11 June 2007

Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(C-C) = 0.004$ Å; disorder in solvent or counterion; R factor = 0.025; wR factor = 0.058; data-to-parameter ratio = 21.3.

The 1:1 AgCN–cyclohexyldiphenylphosphine adduct was synthesized to explore the structural possibilities of C- or N-atom coordination available to the cyanide ligand in the presence of cyclohexyldiphenylphosphine (PCyPh₂), which would exert a steric influence intermediate between PCy₃ and PPh₃. The title compound, {[Ag₂(CN)₂(C₁₆H₂₁P)₂]-CH₂Cl₂}_n, is an inorganic polymer with monomeric units consisting of a linear bis(cyano)silver complex (formally a –1 anion) coordinated *via* the C atoms alternating with a tetrahedral silver complex having two phosphine ligands (formally a +1 cation). The tetrahedral coordination of the bis(phosphine)-silver fragment is completed by dative bonds through the N-atom lone pairs of two bis(cyano)silver fragments. For each disilver monomeric unit, one molecule of dichloromethane solvent is found, disordered over two positions with relative occupancies 0.88:0.12. The polymer is propagated by a twofold screw causing each polymer strand to be chiral. Crystallization in a noncentrosymmetric space group implies spontaneous resolution from the solution, which could be achiral if the solvated species is different from the solid state or racemic if the polymers persist in solution. Even at a twofold excess of the phosphine, only the 1:1 polymer is observed, suggesting that the PCyPh₂ ligand has a structural behaviour more like PPh₃ than PCy₃.

Related literature

Background information on monodentate phosphine–AgCN adducts can be found in Bowmaker, Effendy, Reid *et al.* (1998), Bowmaker, Effendy, Junk & White (1998), Bowmaker *et al.* (1996), Lin *et al.* (2005) and Herberhold *et al.* (2006).



Experimental

Crystal data

[Ag₂(CN)₂(C₁₆H₂₁P)₂]-CH₂Cl₂
 $M_r = 889.34$
 Monoclinic, $P2_1$
 $a = 10.0007$ (12) Å
 $b = 14.7062$ (18) Å
 $c = 13.5338$ (17) Å
 $\beta = 98.285$ (2)°

$V = 1969.7$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.24$ mm⁻¹
 $T = 120$ (2) K
 0.24 × 0.11 × 0.09 mm

Data collection

Bruker APEX diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2002)
 $T_{\min} = 0.784$, $T_{\max} = 0.899$

22828 measured reflections
 9243 independent reflections
 8980 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.058$
 $S = 1.07$
 9243 reflections
 434 parameters
 9 restraints

H-atom parameters constrained
 $\Delta\rho_{\max} = 0.87$ e Å⁻³
 $\Delta\rho_{\min} = -0.53$ e Å⁻³
 Absolute structure: Flack (1983),
 4318 Friedel pairs
 Flack parameter: 0.087 (16)

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 2002); software used to prepare material for publication: *SHELXTL*.

We thank Professor Klaus H. Theopold (Director) and John F. Young of the Center for Catalytic Science and Technology for synthetic assistance and the Department of Chemistry and Biochemistry for the purchase of reagents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2046).

References

- Bowmaker, G. A., Effendy, Harvey, P. J., Healey, P. C., Skelton, B. W. & White, A. H. (1996). *J. Chem. Soc. Dalton Trans.* pp. 2449–2457.
 Bowmaker, G. A., Effendy, Junk, P. C. & White, A. H. (1998). *J. Chem. Soc. Dalton Trans.* pp. 2131–2138.
 Bowmaker, G. A., Effendy, Reid, J. C., Rickard, C. E. F., Skelton, B. W. & White, A. H. (1998). *J. Chem. Soc. Dalton Trans.* pp. 2139–2146.

Bruker (2002). *SMART* (Version 5.62), *SAINTE* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.

Herberhold, M., Milius, W. & Akkus, N. (2006). *Z. Anorg. Allg. Chem.* **632**, 97–100.

Lin, Y., Lai, S., Che, C., Fu, W., Zhou, Z. & Zhu, N. (2005). *Inorg. Chem.* **44**, 1511–1524.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E63, m1902-m1903 [doi:10.1107/S1600536807028607]

***catena*-Poly[[[bis(cyclohexyldiphenylphosphine- κP)silver(I)]- μ -cyano- $\kappa^2 N:C$ -silver(I)- μ -cyano- $\kappa^2 C:N$] dichloromethane solvate]**

X. Xie, B. Saparov and G. P. A. Yap

Comment

The 1:1 adduct of AgCN and triphenylphosphine (PPh₃) is known to yield polymer chains (Bowmaker, Effendy, Reid *et al.*, 1998) since the cyanide ligand can coordinate through either the C or N atoms. In comparison, the 1:1 adduct of AgCN and the more sterically demanding tricyclohexylphosphine (PCy₃) can yield, in addition to the polymer (Bowmaker, Effendy, Junk & White, 1998), a di-silver bis-phosphine monomeric complex (Lin *et al.*, 2005).

Indeed when the even bulkier tri(1-cyclohepta-2,4,6-trienyl)phosphine is used the N atom coordination of the cyanide ligand is blocked and only a linear, monomeric 1:1 silver phosphine cyanide is observed (Herberhold *et al.*, 2006). In comparison, the mono-Ag phosphine cyano adducts reported are 1:2 AgCN:PCy₃ (Bowmaker *et al.*, 1996) and 1:3 AgCN:PPh₃ (Bowmaker, Effendy, Reid *et al.*, 1998).

In order to generate polymers with bigger phosphines, a second much smaller ligand may be employed to complete the tetrahedral coordination on the phosphine-bearing Ag as in the case of AgCN:*P(o-tolyl)*₃:pyridine (2:1:1) (Bowmaker, Effendy, Reid *et al.*, 1998) or using less phosphine and changing the coordination around the phosphine-bearing Ag to trigonal as in the case of the 2:1 AgCN:PCy₃ polymer (Lin *et al.*, 2005). Surprisingly, the reported synthesis of both the 2:1 and the 1:1 AgCN:PCy₃ polymers require a 1:1 molar ratio of the reactants!

We have decided to use cyclohexyldiphenylphosphine (PCyPh₂) in order to explore the structural chemistry in the steric regime intermediate between PCy₃ and PPh₃. We have been able to synthesize the title compound which is a polymer similar to that reported for the PPh₃ case. The monomeric unit can be described as linear bis-cyano Ag complex (formally a -1 anion) coordinated *via* the C atoms, and a tetrahedral Ag complex with two phosphines (formally a +1 cation). The tetrahedral coordination sphere is completed by dative bonds through the N lone pairs of two bis-cyano Ag fragments. The polymer is propagated by a twofold screw causing each polymer strand to be chiral. Crystallization in a noncentrosymmetric space group implies spontaneous resolution from the solution which could be achiral if the solvated species is different from the solid-state or racemic if the polymers persist in solution. In contrast, the enantiomeric polymers with PPh₃ crystallized in centrosymmetric, and therefore racemic, crystals. Even at a twofold excess of the phosphine, only the 1:1 polymer is observed suggesting that the PCyPh₂ phosphine has structural behaviour more similar to PPh₃ than to PCy₃.

Experimental

The title compound can be synthesized quantitatively using literature methods (Herberhold *et al.*, 2006) modified with the appropriate phosphine and with tetrahydrofuran reaction solvent. X-ray quality crystals were generated from a saturated solution in methylene chloride layered with hexanes.

Refinement

The cocrystallized methylene chloride molecule was located disordered in two positions. The C—Cl and Cl⋯Cl distances were restrained to be similar in the disordered contributions. Atomic displacement parameters were constrained to be equal in the chemically equivalent atomic positions. The solvent molecule was restrained from close contact to the polymer. The site occupancies refined to 88:12. H atoms were assigned calculated positions with U_{iso} restrained to be $0.2U_{eq}$ of the bonded C atom and a C—H distance of 0.95–0.99 Å.

Figures

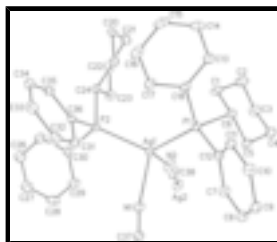


Fig. 1. Molecular diagram of a monomeric unit of the title compound with ellipsoids at 30% probability. CocrySTALLIZED solvent molecule, and hydrogen atoms omitted for clarity.



Fig. 2. Packing diagram and strand segment of the title compound along the b axis. Minor disordered contribution of the solvent molecule omitted for clarity.

catena-Poly[[[bis(cyclohexyldiphenylphosphine- κ P)silver(I)]- μ -cyano- κ^2 N:C-silver(I)- μ -cyano- κ^2 C:N] dichloromethane solvate]

Crystal data

$[\text{Ag}_2(\text{CN})_2(\text{C}_{16}\text{H}_{21}\text{P}_1)_2] \cdot \text{CH}_2\text{Cl}_2$

$M_r = 889.34$

Monoclinic, $P2_1$

Hall symbol: $p\ 2yb$

$a = 10.0007$ (12) Å

$b = 14.7062$ (18) Å

$c = 13.5338$ (17) Å

$\beta = 98.285$ (2)°

$V = 1969.7$ (4) Å³

$Z = 2$

$F_{000} = 900$

$D_x = 1.500$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 7412 reflections

$\theta = 2.5$ – 28.3 °

$\mu = 1.24$ mm⁻¹

$T = 120$ (2) K

Needle, colourless

$0.24 \times 0.11 \times 0.09$ mm

Data collection

Bruker APEX
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 836.6 pixels mm⁻¹

9243 independent reflections

8980 reflections with $I > 2\sigma(I)$

$R_{int} = 0.021$

$\theta_{max} = 28.3$ °

$T = 120(2)$ K $\theta_{\min} = 2.1^\circ$
 ω scans $h = -13 \rightarrow 13$
 Absorption correction: multi-scan (SADABS; Bruker, 2002) $k = -18 \rightarrow 19$
 $T_{\min} = 0.784$, $T_{\max} = 0.899$ $l = -17 \rightarrow 17$
 22828 measured reflections

Refinement

Refinement on F^2 Hydrogen site location: inferred from neighbouring sites
 Least-squares matrix: full H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.025$ $w = 1/[\sigma^2(F_o^2) + (0.0222P)^2 + 1.1832P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.058$ $(\Delta/\sigma)_{\max} = 0.001$
 $S = 1.07$ $\Delta\rho_{\max} = 0.87 \text{ e } \text{\AA}^{-3}$
 9243 reflections $\Delta\rho_{\min} = -0.53 \text{ e } \text{\AA}^{-3}$
 434 parameters Extinction correction: none
 9 restraints Absolute structure: Flack (1983), 4318 Friedel pairs
 Primary atom site location: structure-invariant direct methods Flack parameter: 0.087 (16)
 Secondary atom site location: difference Fourier map

Special details

Experimental. Data collection is performed with four batch runs at $\varphi = 0.00^\circ$ (600 frames), at $\varphi = 90.00^\circ$ (600 frames), at $\varphi = 180^\circ$ (600 frames) and at $\varphi = 270^\circ$ (600 frames). Frame width = 0.30 \AA and ω . Data is merged, corrected for decay, and treated with multi-scan absorption corrections.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ag1	-0.151855 (18)	0.265568 (13)	1.178117 (14)	0.02008 (5)	
Ag2	-0.54560 (2)	0.012640 (17)	1.06267 (2)	0.03267 (6)	
P1	-0.13755 (7)	0.32740 (5)	1.34766 (5)	0.01945 (14)	
P2	0.03650 (7)	0.20108 (5)	1.10216 (5)	0.01898 (14)	
N1	-0.2515 (3)	0.37361 (19)	1.0630 (2)	0.0306 (6)	
N2	-0.3297 (3)	0.1636 (2)	1.1481 (2)	0.0381 (7)	
C1	-0.1683 (3)	0.1585 (2)	1.4299 (3)	0.0292 (7)	

supplementary materials

H1A	-0.1765	0.1357	1.3605	0.035*
H1B	-0.0712	0.1601	1.4575	0.035*
C2	-0.2420 (3)	0.0939 (2)	1.4921 (3)	0.0383 (8)
H2A	-0.2041	0.0319	1.4892	0.046*
H2B	-0.2277	0.1139	1.5627	0.046*
C3	-0.3929 (3)	0.0917 (2)	1.4538 (3)	0.0353 (7)
H3A	-0.4079	0.0668	1.3851	0.042*
H3B	-0.4391	0.0515	1.4968	0.042*
C4	-0.4520 (3)	0.1871 (2)	1.4540 (3)	0.0338 (7)
H4A	-0.4441	0.2094	1.5236	0.041*
H4B	-0.5492	0.1849	1.4265	0.041*
C5	-0.3802 (3)	0.2533 (2)	1.3925 (2)	0.0284 (6)
H5A	-0.4177	0.3151	1.3977	0.034*
H5B	-0.3964	0.2349	1.3213	0.034*
C6	-0.2263 (3)	0.2547 (2)	1.4294 (2)	0.0229 (6)
H6	-0.2104	0.2796	1.4989	0.027*
C7	-0.3316 (3)	0.4613 (2)	1.2994 (2)	0.0259 (6)
H7	-0.3734	0.4171	1.2538	0.031*
C8	-0.3922 (3)	0.5458 (2)	1.3058 (2)	0.0293 (7)
H8	-0.4765	0.5583	1.2660	0.035*
C9	-0.3306 (3)	0.6116 (2)	1.3697 (2)	0.0268 (6)
H9	-0.3717	0.6694	1.3736	0.032*
C10	-0.2078 (3)	0.5923 (2)	1.4283 (2)	0.0285 (6)
H10	-0.1646	0.6376	1.4717	0.034*
C11	-0.1483 (3)	0.5080 (2)	1.4240 (2)	0.0243 (5)
H11	-0.0651	0.4956	1.4651	0.029*
C12	-0.2093 (3)	0.44075 (19)	1.3594 (2)	0.0214 (5)
C13	0.0679 (3)	0.3287 (2)	1.5178 (2)	0.0265 (6)
H13	-0.0006	0.3168	1.5579	0.032*
C14	0.2023 (3)	0.3373 (2)	1.5623 (3)	0.0332 (7)
H14	0.2248	0.3307	1.6325	0.040*
C15	0.3022 (3)	0.3551 (2)	1.5052 (3)	0.0353 (8)
H15	0.3933	0.3608	1.5360	0.042*
C16	0.2703 (3)	0.3647 (2)	1.4030 (3)	0.0364 (8)
H16	0.3393	0.3773	1.3635	0.044*
C17	0.1368 (3)	0.3560 (2)	1.3580 (2)	0.0282 (6)
H17	0.1154	0.3626	1.2877	0.034*
C18	0.0341 (3)	0.33775 (19)	1.4142 (2)	0.0225 (6)
C19	0.1764 (3)	0.1125 (2)	1.2704 (2)	0.0253 (6)
H19A	0.1028	0.1288	1.3089	0.030*
H19B	0.2425	0.1630	1.2764	0.030*
C20	0.2459 (3)	0.0248 (2)	1.3127 (2)	0.0295 (7)
H20A	0.3219	0.0104	1.2759	0.035*
H20B	0.2831	0.0342	1.3838	0.035*
C21	0.1478 (4)	-0.0542 (2)	1.3039 (3)	0.0370 (8)
H21A	0.0756	-0.0420	1.3451	0.044*
H21B	0.1958	-0.1102	1.3294	0.044*
C22	0.0849 (4)	-0.0682 (2)	1.1950 (3)	0.0343 (7)
H22A	0.1562	-0.0869	1.1554	0.041*

H22B	0.0173	-0.1177	1.1913	0.041*	
C23	0.0167 (3)	0.0185 (2)	1.1501 (2)	0.0290 (6)	
H23A	-0.0610	0.0338	1.1849	0.035*	
H23B	-0.0179	0.0085	1.0787	0.035*	
C24	0.1184 (3)	0.09822 (18)	1.1606 (2)	0.0206 (5)	
H24	0.1947	0.0817	1.1237	0.025*	
C25	0.0990 (4)	0.1231 (3)	0.9238 (2)	0.0396 (8)	
H25	0.1816	0.1047	0.9623	0.047*	
C26	0.0742 (4)	0.1020 (3)	0.8223 (3)	0.0461 (10)	
H26	0.1388	0.0685	0.7921	0.055*	
C27	-0.0444 (4)	0.1299 (3)	0.7663 (3)	0.0423 (9)	
H27	-0.0616	0.1156	0.6971	0.051*	
C28	-0.1379 (4)	0.1781 (3)	0.8097 (3)	0.0391 (8)	
H28	-0.2186	0.1981	0.7699	0.047*	
C29	-0.1158 (3)	0.1980 (2)	0.9115 (2)	0.0307 (7)	
H29	-0.1823	0.2299	0.9413	0.037*	
C30	0.0041 (3)	0.1710 (2)	0.9694 (2)	0.0237 (6)	
C31	0.1320 (3)	0.3762 (2)	1.0905 (2)	0.0247 (6)	
H31	0.0388	0.3915	1.0792	0.030*	
C32	0.2284 (3)	0.4443 (2)	1.0936 (2)	0.0280 (6)	
H32	0.2010	0.5060	1.0844	0.034*	
C33	0.3647 (3)	0.4227 (2)	1.1101 (2)	0.0287 (6)	
H33	0.4306	0.4695	1.1133	0.034*	
C34	0.4048 (3)	0.3323 (2)	1.1220 (2)	0.0271 (6)	
H34	0.4982	0.3173	1.1327	0.033*	
C35	0.3083 (3)	0.2643 (3)	1.11829 (19)	0.0238 (5)	
H35	0.3362	0.2026	1.1256	0.029*	
C36	0.1709 (3)	0.28510 (18)	1.1039 (2)	0.0198 (5)	
C37	-0.3189 (3)	0.4236 (2)	1.0136 (2)	0.0283 (6)	
C38	-0.4078 (3)	0.1074 (2)	1.1226 (3)	0.0354 (8)	
C39	-0.5746 (6)	0.2741 (4)	0.8594 (5)	0.0537 (12)	0.880 (2)
H39A	-0.6066	0.2109	0.8460	0.064*	0.880 (2)
H39B	-0.5223	0.2760	0.9273	0.064*	0.880 (2)
Cl1	-0.46825 (14)	0.30626 (12)	0.77027 (10)	0.0624 (4)	0.880 (2)
Cl2	-0.71293 (14)	0.34665 (12)	0.85379 (9)	0.0662 (4)	0.880 (2)
C40	-0.617 (4)	0.292 (3)	0.852 (4)	0.0537 (12)	0.120 (2)
H40A	-0.6348	0.3350	0.9051	0.064*	0.120 (2)
H40B	-0.7044	0.2710	0.8154	0.064*	0.120 (2)
Cl3	-0.5162 (11)	0.3441 (9)	0.7695 (8)	0.0624 (4)	0.120 (2)
Cl4	-0.5150 (10)	0.1996 (8)	0.9019 (6)	0.0662 (4)	0.120 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.01662 (9)	0.02012 (9)	0.02306 (9)	0.00090 (8)	0.00141 (7)	-0.00285 (9)
Ag2	0.02438 (11)	0.02882 (12)	0.04481 (14)	-0.00741 (10)	0.00503 (9)	-0.01479 (11)
P1	0.0181 (3)	0.0182 (3)	0.0219 (3)	0.0007 (3)	0.0024 (3)	-0.0020 (3)
P2	0.0173 (3)	0.0189 (3)	0.0206 (3)	0.0009 (3)	0.0023 (3)	-0.0017 (3)

supplementary materials

N1	0.0286 (13)	0.0287 (14)	0.0344 (15)	0.0065 (11)	0.0046 (11)	0.0035 (11)
N2	0.0312 (15)	0.0321 (15)	0.0510 (18)	-0.0047 (12)	0.0061 (13)	-0.0140 (13)
C1	0.0214 (14)	0.0214 (15)	0.0439 (19)	0.0008 (11)	0.0015 (13)	0.0074 (13)
C2	0.0309 (17)	0.0256 (16)	0.055 (2)	-0.0002 (13)	-0.0031 (16)	0.0122 (15)
C3	0.0302 (16)	0.0249 (16)	0.051 (2)	-0.0053 (13)	0.0062 (15)	0.0072 (14)
C4	0.0278 (16)	0.0328 (18)	0.0418 (19)	-0.0034 (13)	0.0086 (14)	0.0033 (14)
C5	0.0229 (13)	0.0264 (17)	0.0363 (16)	0.0023 (12)	0.0062 (11)	0.0048 (14)
C6	0.0208 (12)	0.0220 (15)	0.0257 (13)	-0.0028 (11)	0.0030 (10)	0.0002 (12)
C7	0.0247 (14)	0.0227 (15)	0.0293 (15)	-0.0014 (11)	0.0004 (12)	-0.0034 (12)
C8	0.0258 (15)	0.0280 (16)	0.0331 (16)	0.0053 (12)	0.0009 (12)	0.0007 (12)
C9	0.0314 (16)	0.0196 (14)	0.0308 (16)	0.0053 (12)	0.0089 (12)	-0.0004 (12)
C10	0.0350 (17)	0.0182 (14)	0.0313 (16)	-0.0020 (12)	0.0009 (13)	-0.0060 (12)
C11	0.0253 (13)	0.0229 (14)	0.0243 (13)	0.0019 (12)	0.0021 (10)	0.0002 (12)
C12	0.0222 (14)	0.0180 (13)	0.0242 (13)	0.0019 (11)	0.0045 (11)	-0.0017 (11)
C13	0.0285 (15)	0.0236 (14)	0.0263 (14)	-0.0006 (12)	0.0000 (11)	-0.0019 (12)
C14	0.0339 (17)	0.0239 (16)	0.0362 (17)	0.0029 (13)	-0.0140 (14)	-0.0023 (13)
C15	0.0233 (15)	0.0263 (16)	0.052 (2)	0.0084 (12)	-0.0096 (14)	-0.0112 (15)
C16	0.0225 (15)	0.0380 (19)	0.049 (2)	0.0005 (13)	0.0064 (14)	-0.0144 (16)
C17	0.0232 (14)	0.0330 (16)	0.0283 (15)	0.0004 (12)	0.0032 (12)	-0.0105 (13)
C18	0.0187 (13)	0.0184 (13)	0.0292 (14)	0.0024 (10)	-0.0004 (11)	-0.0049 (11)
C19	0.0258 (14)	0.0258 (15)	0.0238 (14)	0.0030 (12)	0.0025 (11)	-0.0005 (12)
C20	0.0291 (15)	0.0333 (18)	0.0258 (15)	0.0095 (14)	0.0028 (12)	0.0100 (13)
C21	0.046 (2)	0.0289 (17)	0.0379 (18)	0.0059 (15)	0.0130 (15)	0.0141 (14)
C22	0.0392 (19)	0.0207 (15)	0.044 (2)	-0.0003 (13)	0.0097 (15)	0.0028 (13)
C23	0.0297 (15)	0.0231 (14)	0.0337 (16)	-0.0010 (13)	0.0027 (12)	-0.0002 (13)
C24	0.0231 (13)	0.0165 (13)	0.0222 (13)	0.0031 (10)	0.0034 (11)	0.0003 (10)
C25	0.0312 (17)	0.060 (2)	0.0263 (16)	0.0124 (16)	0.0015 (13)	-0.0088 (16)
C26	0.044 (2)	0.068 (3)	0.0253 (17)	0.0154 (19)	0.0015 (15)	-0.0127 (17)
C27	0.048 (2)	0.055 (2)	0.0224 (16)	0.0026 (18)	-0.0007 (15)	-0.0102 (16)
C28	0.040 (2)	0.0403 (19)	0.0334 (18)	0.0063 (15)	-0.0082 (15)	-0.0021 (15)
C29	0.0298 (16)	0.0277 (16)	0.0327 (16)	0.0033 (13)	-0.0014 (13)	-0.0025 (13)
C30	0.0239 (14)	0.0245 (14)	0.0217 (13)	-0.0020 (11)	0.0002 (11)	-0.0019 (11)
C31	0.0227 (14)	0.0253 (15)	0.0260 (15)	0.0001 (12)	0.0039 (11)	0.0012 (12)
C32	0.0302 (16)	0.0220 (14)	0.0322 (16)	-0.0003 (12)	0.0061 (13)	0.0051 (12)
C33	0.0288 (16)	0.0299 (16)	0.0268 (15)	-0.0081 (13)	0.0024 (12)	0.0016 (12)
C34	0.0207 (14)	0.0325 (16)	0.0279 (15)	-0.0018 (12)	0.0026 (11)	0.0009 (13)
C35	0.0214 (12)	0.0251 (12)	0.0248 (12)	0.0002 (13)	0.0024 (10)	-0.0015 (14)
C36	0.0189 (12)	0.0222 (15)	0.0184 (12)	-0.0018 (10)	0.0033 (10)	-0.0008 (10)
C37	0.0245 (15)	0.0281 (16)	0.0335 (16)	0.0029 (12)	0.0081 (13)	0.0041 (13)
C38	0.0234 (15)	0.0338 (18)	0.049 (2)	-0.0013 (13)	0.0062 (14)	-0.0148 (15)
C39	0.072 (4)	0.042 (3)	0.047 (2)	0.005 (3)	0.011 (3)	-0.001 (2)
Cl1	0.0404 (7)	0.0945 (11)	0.0531 (7)	-0.0046 (7)	0.0097 (6)	-0.0245 (7)
Cl2	0.0573 (8)	0.1012 (12)	0.0411 (6)	0.0117 (7)	0.0108 (5)	-0.0066 (7)
C40	0.072 (4)	0.042 (3)	0.047 (2)	0.005 (3)	0.011 (3)	-0.001 (2)
Cl3	0.0404 (7)	0.0945 (11)	0.0531 (7)	-0.0046 (7)	0.0097 (6)	-0.0245 (7)
Cl4	0.0573 (8)	0.1012 (12)	0.0411 (6)	0.0117 (7)	0.0108 (5)	-0.0066 (7)

Geometric parameters (Å, °)

Ag1—N2	2.317 (3)	C14—C15	1.372 (5)
Ag1—N1	2.345 (3)	C15—C16	1.382 (5)
Ag1—P1	2.4532 (8)	C16—C17	1.391 (4)
Ag1—P2	2.4623 (7)	C17—C18	1.389 (4)
Ag2—C38	2.044 (3)	C19—C24	1.529 (4)
Ag2—C37 ⁱ	2.052 (3)	C19—C20	1.536 (4)
P1—C18	1.826 (3)	C20—C21	1.514 (5)
P1—C12	1.831 (3)	C21—C22	1.531 (5)
P1—C6	1.854 (3)	C22—C23	1.531 (5)
P2—C36	1.823 (3)	C23—C24	1.545 (4)
P2—C30	1.833 (3)	C25—C30	1.395 (4)
P2—C24	1.844 (3)	C25—C26	1.395 (5)
N1—C37	1.146 (4)	C26—C27	1.375 (5)
N2—C38	1.155 (4)	C27—C28	1.371 (5)
C1—C2	1.528 (4)	C28—C29	1.395 (5)
C1—C6	1.529 (4)	C29—C30	1.393 (4)
C2—C3	1.524 (5)	C31—C32	1.387 (4)
C3—C4	1.523 (5)	C31—C36	1.399 (4)
C4—C5	1.525 (4)	C32—C33	1.386 (4)
C5—C6	1.548 (4)	C33—C34	1.391 (5)
C7—C8	1.391 (4)	C34—C35	1.386 (4)
C7—C12	1.400 (4)	C35—C36	1.394 (4)
C8—C9	1.383 (4)	C37—Ag2 ⁱⁱ	2.052 (3)
C9—C10	1.391 (4)	C39—C12	1.740 (5)
C10—C11	1.380 (4)	C39—C11	1.784 (5)
C11—C12	1.401 (4)	C40—C14	1.779 (18)
C13—C14	1.397 (4)	C40—C13	1.779 (18)
C13—C18	1.401 (4)		
N2—Ag1—N1	94.59 (11)	C14—C13—C18	120.1 (3)
N2—Ag1—P1	110.09 (8)	C15—C14—C13	120.5 (3)
N1—Ag1—P1	109.41 (7)	C14—C15—C16	120.1 (3)
N2—Ag1—P2	106.97 (8)	C15—C16—C17	119.8 (3)
N1—Ag1—P2	105.12 (7)	C18—C17—C16	121.2 (3)
P1—Ag1—P2	126.04 (2)	C17—C18—C13	118.4 (3)
C38—Ag2—C37 ⁱ	173.26 (15)	C17—C18—P1	117.6 (2)
C18—P1—C12	103.51 (13)	C13—C18—P1	124.1 (2)
C18—P1—C6	104.52 (13)	C24—C19—C20	109.5 (2)
C12—P1—C6	104.20 (13)	C21—C20—C19	111.2 (3)
C18—P1—Ag1	114.59 (10)	C20—C21—C22	110.5 (3)
C12—P1—Ag1	116.63 (10)	C23—C22—C21	111.5 (3)
C6—P1—Ag1	112.09 (10)	C22—C23—C24	110.2 (2)
C36—P2—C30	101.66 (13)	C19—C24—C23	110.2 (2)
C36—P2—C24	105.62 (13)	C19—C24—P2	112.76 (19)
C30—P2—C24	103.15 (13)	C23—C24—P2	109.7 (2)
C36—P2—Ag1	109.90 (9)	C30—C25—C26	120.9 (3)

supplementary materials

C30—P2—Ag1	117.68 (10)	C27—C26—C25	119.5 (3)
C24—P2—Ag1	117.14 (9)	C28—C27—C26	120.4 (3)
C37—N1—Ag1	168.8 (3)	C27—C28—C29	120.7 (3)
C38—N2—Ag1	169.7 (3)	C30—C29—C28	119.8 (3)
C2—C1—C6	111.4 (3)	C29—C30—C25	118.7 (3)
C3—C2—C1	111.0 (3)	C29—C30—P2	119.9 (2)
C4—C3—C2	110.2 (3)	C25—C30—P2	121.4 (2)
C3—C4—C5	111.9 (3)	C32—C31—C36	120.6 (3)
C4—C5—C6	110.9 (3)	C33—C32—C31	120.1 (3)
C1—C6—C5	110.3 (2)	C32—C33—C34	119.9 (3)
C1—C6—P1	108.8 (2)	C35—C34—C33	119.9 (3)
C5—C6—P1	110.75 (19)	C34—C35—C36	120.9 (3)
C8—C7—C12	120.6 (3)	C35—C36—C31	118.6 (3)
C9—C8—C7	120.3 (3)	C35—C36—P2	124.3 (2)
C8—C9—C10	119.4 (3)	C31—C36—P2	117.2 (2)
C11—C10—C9	120.6 (3)	N1—C37—Ag2 ⁱⁱ	173.3 (3)
C10—C11—C12	120.6 (3)	N2—C38—Ag2	174.1 (4)
C7—C12—C11	118.4 (3)	C12—C39—C11	110.8 (3)
C7—C12—P1	117.8 (2)	C14—C40—C13	102.7 (13)
C11—C12—P1	123.8 (2)		

Symmetry codes: (i) $-x-1, y-1/2, -z+2$; (ii) $-x-1, y+1/2, -z+2$.

Fig. 1

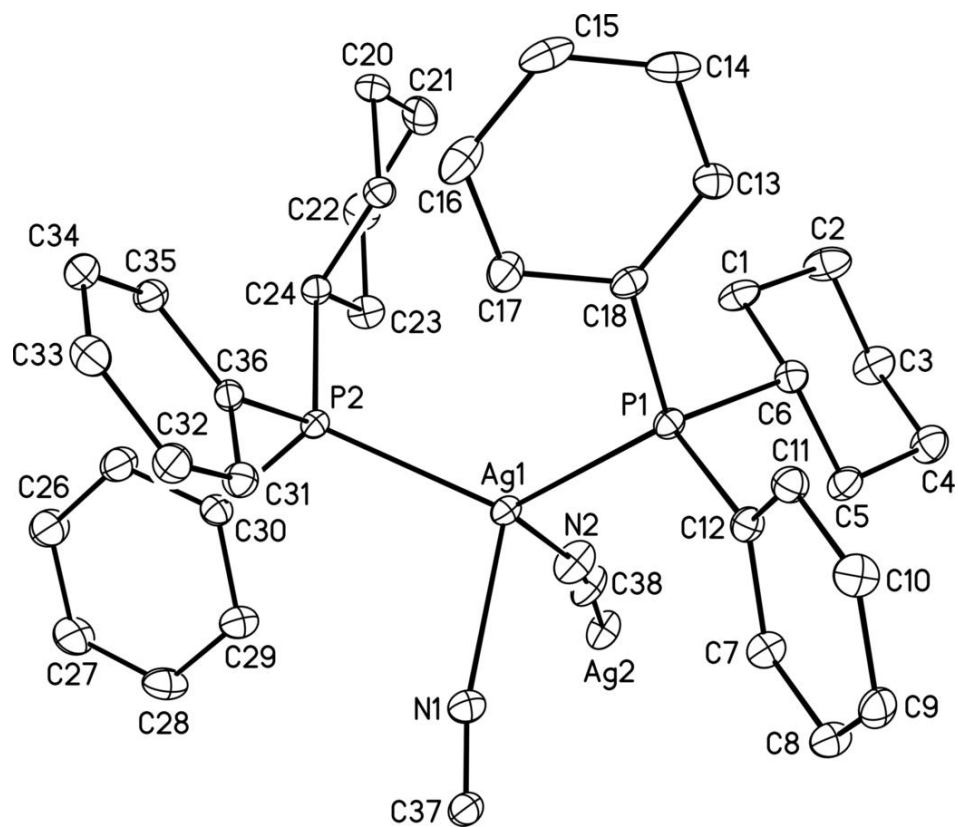


Fig. 2

