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Crystal structure of poly[μ-diphenyl(pyridin-4-yl)-phosphane-κ^2N:P-μ-trifluoroacetato-κ^2O:O'-silver(I)] from synchrotron data

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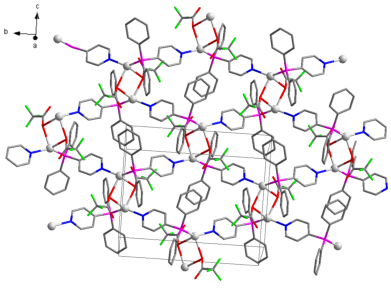
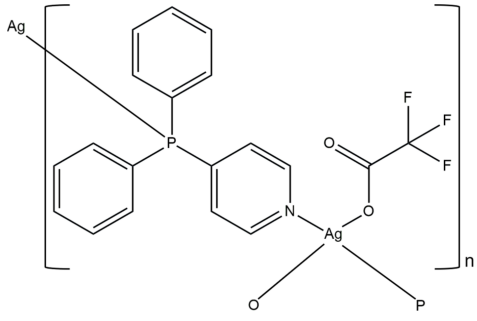
The crystal structure of the title compound, $[Ag(CF_3CO_2)(C_{17}H_{14}NP)]$ has been determined from synchrotron data (λ = 0.70000 Å). Centrosymmetric dinuclear Ag_2O_2 units, generated by inversion centers, extend into a two-dimensional coordination polymer linked by the N and P atoms of the bridged-bidentate (μ^2) ligand. The resulting two-dimensional array can be described as a 6^3 (hcb) network.

1. Chemical context

Research on the construction and packing motifs of two-dimensional coordination networks is of interest due to their many applications such as adsorption/desorption, molecular recognition, separation, energy transfer template, chemo-sensors, ion exchangers, drying agents, nuclear waste storage, crystal structure templates of liquids and heterogeneous catalysis (Kim *et al.*, 2022) *via* the stretching and sliding between layers (Kim *et al.*, 2021). Thus, new types of two-dimensional coordination frameworks have been constructed by the self-assembly of metal cations as a geometric component and designed multidonor ligands as a spacer. Furthermore, such frameworks could be rationally designed by controlling weakly coordinating (counter)anions due to the less effective electrostatic binding interactions. Anion chemistry has emerged as an active field owing to a timely interest from environmental pollution, industrial chemicals, biological process, ionic liquids, catalysis, lithium battery, and health-related perspectives (Beer & Gale, 2001). Among ligands, diphenyl-4-pyridyl phosphine spacers exhibit delicate differences in the size, lone-pair delocalization, conformational energy barrier, and donating ability (Wang *et al.*, 2004). Here, we describe the synthesis and crystal structure of the title coordination polymer $[Ag(CF_3CO_2)(C_{17}H_{14}NP)]_n$, (**I**).





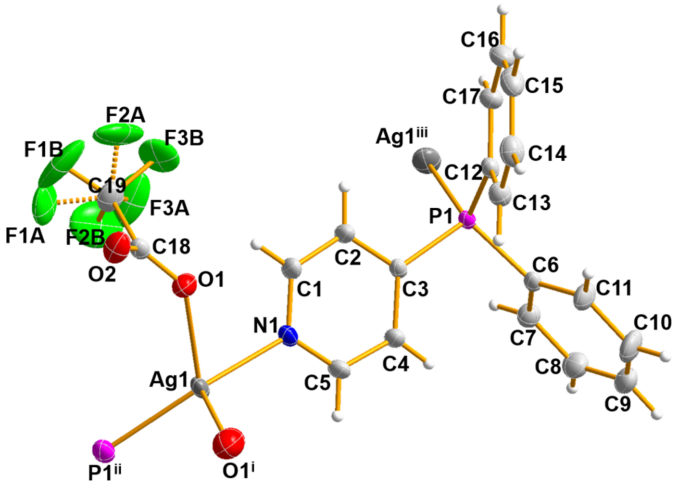


Figure 1
The asymmetric unit of (**I**) expanded to show the complete silver-ion coordination sphere with displacement ellipsoids drawn at the 30% probability level. [Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.]

2. Structural commentary

The metal ion in (**I**) is coordinated by the N and P atoms of two diphenyl-4-pyridyl phosphine (*L*) ligands (one symmetry generated) and two O-atom donors of the bridging trifluoroacetate anions (one symmetry generated) and selected bond lengths and angles are listed in Table 1. This results in a very distorted AgO_2NP tetrahedral arrangement (Fig. 1) with the N—Ag—P bond angle being 142.00 (6)°. The O atoms bridge to an adjacent silver atom, which thus forms a centrosymmetric dinuclear unit generated by an inversion center at (1/2, 1, 1/2) for the asymmetric atoms. A short Ag1···O2($1-x, 2-y, 1-z$) contact of 2.764 (2) Å arises within the dimer.

The $C_{17}H_{14}NP$ (*L*) spacer ligand connects two silver(I) ions to give a single polymeric strand propagating in the [010] direction. The two O-atom donors of the trifluoroacetate anion bridge the single strands in an 'up and down' mode and the resulting extended structure is a two-dimensional coordination polymer propagating in the (100) plane, as shown in Fig. 2, with an hcb 6^3 topology (O'Keeffe *et al.*, 2008). The double-bridge *via* the two acetate O atoms induces an Ag···Ag($1-x, 2-y, 1-z$) distance of 3.7495 (8) Å, which is probably too long to be regarded as an argentophilic (Pyykkö, 1997) silver–silver 'bond'. A weak C—H···O interaction (Table 2, Fig. 3) may help to consolidate the sheets. No directional interactions could be identified in the inter-layer packing.

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.242 (2)	Ag1—O1	2.463 (2)
Ag1—P1^{i}	2.3594 (7)	Ag1—O1^{ii}	2.588 (2)
N1—Ag1—P1^{i}	142.00 (6)	N1—Ag1—O1^{ii}	113.05 (7)
N1—Ag1—O1	87.13 (8)	P1^{i}—Ag1—O1^{ii}	98.45 (5)
P1^{i}—Ag1—O1	117.84 (6)	O1—Ag1—O1^{ii}	84.18 (7)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1	0.94	2.53	3.160 (3)	124

3. Database survey

A search of the Cambridge Structural Database (CSD, version 6.00 with updates through April 2025; Groom *et al.*, 2016) using ConQuest was made for metal complexes containing diphenyl(4-pyridyl)phosphane ligands. Among the 18 hits retrieved, no closely related structure to the title complex was found.

4. Synthesis and crystallization

A solution was prepared by dissolving $AgCF_3CO_2$ (4.42 mg, 0.020 mmol) in acetone, and another solution was prepared by dissolving diphenyl-4-pyridyl phosphine (5.27 mg,

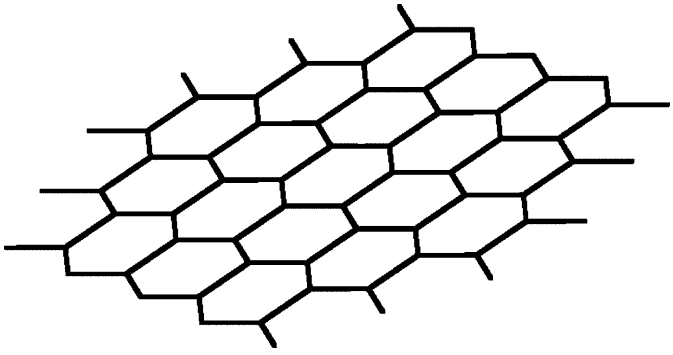


Figure 2
Schematic representation of the hcb (6^3) topology of title compound. Topological analysis was performed with a metal-centered simplification, treating the central point between the Ag_2 pairs as nodes and the diphenyl(4-pyridyl)phosphane ligands as linkers.

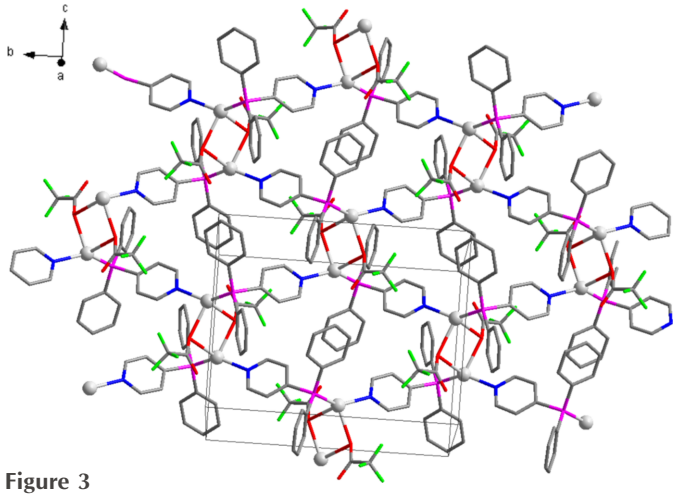


Figure 3
The two-dimensional network structure of title compound. For clarity, H atoms have been omitted.

0.020 mmol) in ethanol. Slow diffusion of the two solutions over several days afforded colorless block-shaped crystals of (**I**) suitable for X-ray diffraction. Yield: 8.33 mg (86%).

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.94 Å and U_{iso}(H) = 1.2 U_{eq}(carrier). The F atoms of the –CF_3 group are disordered over two sets of sites in a 0.510 (13):0.490 (13) ratio.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Ag(C_2F_3O_2)(C_{17}H_{14}NP)]$
M_r	484.15
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	223
a, b, c (Å)	11.683 (2), 14.843 (3), 11.335 (2)
β (°)	103.68 (3)
V (Å^3)	1909.8 (7)
Z	4
Radiation type	Synchrotron, λ = 0.700 Å
μ (mm^{-1})	1.12
Crystal size (mm)	0.30 × 0.22 × 0.12
Data collection	
Diffractometer	Rayonix MX225HS CCD area detector
Absorption correction	Multi-scan (*HKL3000sm SCALEPACK*; Otwinowski *et al.*, 2003)
T_{min}, T_{max}	0.939, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19907, 5327, 4578
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.704
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.116, 1.11
No. of reflections	5327
No. of parameters	272
No. of restraints	36
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −1.86

Computer programs: *PAL BL2D-SMDC Program* (Shin *et al.*, 2016), *HKL3000sm* (Otwinowski & Minor, 2003), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *DIAMOND* (Putz & Brandenburg, 2014) and *publCIF* (Westrip, 2010).

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Jiyeong Song, Young-A Lee and Dongwon Kim

Computing details

Poly[μ-diphenyl(pyridin-4-yl)phosphane-κ^2N:P-μ-trifluoroacetato-κ^2O:O'-silver(I)]

Crystal data

$[Ag(C_2F_3O_2)(C_{17}H_{14}NP)]$
M_r = 484.15
Monoclinic, $P2_1/c$
a = 11.683 (2) Å
b = 14.843 (3) Å
c = 11.335 (2) Å
β = 103.68 (3)°
V = 1909.8 (7) Å^3
Z = 4
$F(000)$ = 960
D_x = 1.684 Mg m^{-3}
Synchrotron radiation, λ = 0.700 Å
Cell parameters from 40378 reflections
θ = 0.4–29.5°
μ = 1.12 mm^{-1}
T = 223 K
Block, colorless
0.30 × 0.22 × 0.12 mm

Data collection

Rayonix MX225HS CCD area detector diffractometer
Radiation source: PLSII 2D bending magnet
ω scan
Absorption correction: multi-scan (*HKL3000sm Scalepack*; Otwinowski *et al.*, 2003)
T_{min} = 0.939, T_{max} = 1.000
19907 measured reflections
5327 independent reflections
4578 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 29.5°, θ_{min} = 2.2°
h = −16→16
k = −20→20
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.11
5327 reflections
272 parameters
36 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.3483P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −1.86 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C3	0.35805 (18)	0.68394 (14)	0.2085 (2)	0.0271 (4)	
C4	0.3393 (2)	0.76130 (16)	0.1382 (2)	0.0355 (5)	
H4	0.288044	0.760537	0.060553	0.043*	
C5	0.3975 (2)	0.83998 (17)	0.1840 (2)	0.0391 (5)	
H5	0.383473	0.892220	0.135841	0.047*	
C6	0.17431 (19)	0.60043 (15)	0.0265 (2)	0.0299 (4)	
C7	0.2086 (2)	0.6029 (2)	−0.0828 (2)	0.0411 (5)	
H7	0.287002	0.589939	−0.084168	0.049*	
C8	0.1274 (3)	0.6243 (2)	−0.1901 (3)	0.0525 (7)	
H8	0.151235	0.626321	−0.263645	0.063*	
C9	0.0125 (3)	0.6427 (2)	−0.1886 (3)	0.0567 (8)	
H9	−0.042092	0.657774	−0.260991	0.068*	
C10	−0.0227 (3)	0.6389 (3)	−0.0807 (3)	0.0659 (10)	
H10	−0.101556	0.650869	−0.080129	0.079*	
C11	0.0577 (2)	0.6176 (2)	0.0269 (3)	0.0487 (7)	
H11	0.033058	0.614747	0.099967	0.058*	
C12	0.2249 (2)	0.54983 (16)	0.2876 (2)	0.0315 (4)	
C13	0.1573 (2)	0.61216 (19)	0.3340 (2)	0.0404 (5)	
H13	0.138279	0.668030	0.295284	0.048*	
C14	0.1185 (3)	0.5915 (2)	0.4371 (3)	0.0511 (7)	
H14	0.071082	0.632748	0.466804	0.061*	
C15	0.1493 (3)	0.5101 (3)	0.4971 (3)	0.0549 (8)	
H15	0.124504	0.497203	0.568348	0.066*	
C16	0.2158 (3)	0.4488 (2)	0.4520 (3)	0.0525 (7)	
H16	0.235659	0.393394	0.491779	0.063*	
C17	0.2539 (3)	0.46832 (17)	0.3475 (3)	0.0402 (6)	
H17	0.299643	0.426076	0.317180	0.048*	
C18	0.6472 (2)	0.90684 (16)	0.6714 (2)	0.0352 (5)	
C19	0.7446 (3)	0.8351 (2)	0.6859 (3)	0.0579 (8)	
Ag1	0.56236 (2)	0.97380 (2)	0.36594 (2)	0.03465 (8)	
P1	0.28994 (5)	0.57488 (4)	0.16075 (5)	0.02680 (12)	
O1	0.57810 (19)	0.91102 (15)	0.57033 (18)	0.0479 (5)	
N1	0.47206 (18)	0.84526 (14)	0.29276 (19)	0.0352 (4)	
C1	0.4910 (2)	0.76980 (18)	0.3596 (2)	0.0418 (6)	
H1	0.543413	0.772179	0.436476	0.050*	
O2	0.6465 (2)	0.95088 (17)	0.7623 (2)	0.0584 (6)	
C2	0.4374 (2)	0.68904 (17)	0.3208 (2)	0.0402 (6)	
H2	0.454302	0.637557	0.370122	0.048*	
F1A	0.8505 (5)	0.8723 (6)	0.7220 (11)	0.098 (3)	0.490 (13)
F2A	0.7521 (10)	0.7926 (10)	0.5923 (11)	0.131 (6)	0.490 (13)
F3A	0.7391 (10)	0.7777 (8)	0.7754 (14)	0.129 (5)	0.490 (13)
F1B	0.8208 (12)	0.8362 (9)	0.7831 (10)	0.152 (6)	0.510 (13)
F2B	0.7951 (11)	0.8384 (10)	0.5961 (13)	0.148 (6)	0.510 (13)
F3B	0.6952 (7)	0.7545 (3)	0.6669 (13)	0.108 (4)	0.510 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.0229 (9)	0.0185 (9)	0.0390 (10)	0.0001 (7)	0.0055 (7)	−0.0020 (7)
C4	0.0365 (12)	0.0235 (11)	0.0417 (12)	−0.0019 (9)	−0.0003 (9)	0.0029 (9)
C5	0.0459 (14)	0.0225 (11)	0.0447 (13)	−0.0042 (10)	0.0027 (10)	0.0045 (9)
C6	0.0252 (10)	0.0194 (10)	0.0420 (11)	−0.0016 (7)	0.0018 (8)	−0.0020 (8)
C7	0.0388 (13)	0.0396 (14)	0.0434 (13)	0.0016 (11)	0.0068 (10)	−0.0017 (10)
C8	0.0578 (18)	0.0528 (19)	0.0421 (15)	−0.0041 (15)	0.0022 (12)	−0.0001 (12)
C9	0.0498 (17)	0.0537 (19)	0.0544 (16)	−0.0058 (14)	−0.0120 (13)	0.0035 (14)
C10	0.0270 (13)	0.091 (3)	0.072 (2)	0.0048 (15)	−0.0051 (13)	0.0061 (19)
C11	0.0266 (12)	0.064 (2)	0.0531 (15)	0.0044 (12)	0.0051 (10)	0.0049 (13)
C12	0.0280 (10)	0.0255 (10)	0.0399 (12)	−0.0044 (8)	0.0060 (8)	−0.0012 (8)
C13	0.0358 (12)	0.0356 (13)	0.0508 (14)	0.0005 (10)	0.0123 (10)	−0.0028 (10)
C14	0.0417 (15)	0.060 (2)	0.0552 (17)	−0.0048 (13)	0.0178 (12)	−0.0128 (14)
C15	0.0527 (18)	0.069 (2)	0.0431 (16)	−0.0190 (16)	0.0112 (12)	0.0012 (14)
C16	0.0583 (19)	0.0473 (17)	0.0491 (16)	−0.0135 (15)	0.0070 (13)	0.0109 (13)
C17	0.0433 (15)	0.0316 (13)	0.0442 (14)	−0.0018 (10)	0.0074 (11)	0.0046 (9)
C18	0.0306 (11)	0.0244 (11)	0.0487 (13)	−0.0007 (8)	0.0057 (9)	0.0011 (9)
C19	0.0427 (16)	0.0526 (19)	0.074 (2)	0.0143 (14)	0.0052 (14)	−0.0084 (16)
Ag1	0.03016 (12)	0.02218 (11)	0.05095 (14)	−0.00674 (6)	0.00828 (8)	−0.00112 (6)
P1	0.0226 (3)	0.0167 (2)	0.0394 (3)	−0.00019 (18)	0.0039 (2)	−0.00062 (19)
O1	0.0444 (11)	0.0526 (13)	0.0439 (10)	0.0006 (9)	0.0047 (8)	0.0074 (9)
N1	0.0329 (10)	0.0222 (9)	0.0482 (11)	−0.0044 (7)	0.0049 (8)	−0.0011 (8)
C1	0.0403 (13)	0.0287 (12)	0.0480 (13)	−0.0061 (10)	−0.0063 (10)	0.0012 (10)
O2	0.0557 (14)	0.0507 (13)	0.0622 (14)	0.0094 (11)	0.0011 (10)	−0.0211 (11)
C2	0.0399 (13)	0.0248 (11)	0.0470 (13)	−0.0042 (10)	−0.0075 (10)	0.0056 (9)
F1A	0.026 (2)	0.112 (6)	0.148 (8)	0.003 (3)	0.006 (3)	−0.011 (5)
F2A	0.091 (6)	0.148 (9)	0.134 (8)	0.060 (6)	−0.009 (5)	−0.092 (7)
F3A	0.107 (6)	0.100 (7)	0.194 (11)	0.066 (5)	0.063 (7)	0.099 (7)
F1B	0.135 (9)	0.154 (10)	0.115 (7)	0.104 (8)	−0.074 (6)	−0.052 (6)
F2B	0.121 (8)	0.169 (10)	0.199 (11)	0.054 (7)	0.124 (8)	0.026 (8)
F3B	0.102 (5)	0.028 (2)	0.194 (10)	0.025 (3)	0.035 (6)	0.003 (4)

Geometric parameters (Å, º)

C3—C4	1.385 (3)	C15—C16	1.372 (6)
C3—C2	1.388 (3)	C16—C17	1.391 (4)
C3—P1	1.828 (2)	C18—O2	1.222 (3)
C4—C5	1.389 (3)	C18—O1	1.238 (3)
C5—N1	1.333 (3)	C18—C19	1.538 (4)
C6—C11	1.387 (3)	C19—F1B	1.242 (7)
C6—C7	1.390 (3)	C19—F2A	1.255 (8)
C6—P1	1.820 (2)	C19—F2B	1.292 (9)
C7—C8	1.391 (4)	C19—F3B	1.325 (7)
C8—C9	1.374 (5)	C19—F1A	1.327 (7)
C9—C10	1.380 (5)	C19—F3A	1.339 (8)
C10—C11	1.389 (4)	Ag1—N1	2.242 (2)

C12—C17	1.390 (3)	Ag1—P1^{i}	2.3594 (7)
C12—C13	1.397 (4)	Ag1—O1	2.463 (2)
C12—P1	1.816 (2)	Ag1—O1^{ii}	2.588 (2)
C13—C14	1.384 (4)	N1—C1	1.341 (3)
C14—C15	1.391 (5)	C1—C2	1.376 (3)
C4—C3—C2	117.6 (2)	F2A—C19—F3A	110.2 (8)
C4—C3—P1	124.43 (17)	F1A—C19—F3A	103.9 (6)
C2—C3—P1	117.97 (17)	F1B—C19—C18	116.3 (5)
C3—C4—C5	119.0 (2)	F2A—C19—C18	117.1 (5)
N1—C5—C4	123.4 (2)	F2B—C19—C18	110.7 (6)
C11—C6—C7	119.2 (2)	F3B—C19—C18	108.9 (4)
C11—C6—P1	124.9 (2)	F1A—C19—C18	110.9 (5)
C7—C6—P1	115.93 (18)	F3A—C19—C18	110.8 (4)
C6—C7—C8	120.3 (3)	N1—Ag1—P1^{i}	142.00 (6)
C9—C8—C7	120.0 (3)	N1—Ag1—O1	87.13 (8)
C8—C9—C10	120.0 (3)	P1^{i}—Ag1—O1	117.84 (6)
C9—C10—C11	120.4 (3)	N1—Ag1—O1^{ii}	113.05 (7)
C6—C11—C10	120.0 (3)	P1^{i}—Ag1—O1^{ii}	98.45 (5)
C17—C12—C13	119.1 (2)	O1—Ag1—O1^{ii}	84.18 (7)
C17—C12—P1	117.8 (2)	C12—P1—C6	109.72 (11)
C13—C12—P1	122.8 (2)	C12—P1—C3	100.46 (10)
C14—C13—C12	119.9 (3)	C6—P1—C3	104.35 (10)
C13—C14—C15	120.5 (3)	C12—P1—Ag1^{iii}	115.27 (8)
C16—C15—C14	119.9 (3)	C6—P1—Ag1^{iii}	116.50 (8)
C15—C16—C17	120.2 (3)	C3—P1—Ag1^{iii}	108.70 (7)
C12—C17—C16	120.5 (3)	C18—O1—Ag1	140.99 (19)
O2—C18—O1	128.4 (3)	C18—O1—Ag1^{ii}	95.32 (17)
O2—C18—C19	115.6 (3)	Ag1—O1—Ag1^{ii}	95.82 (7)
O1—C18—C19	116.0 (2)	C5—N1—C1	117.2 (2)
F1B—C19—F2B	109.5 (9)	C5—N1—Ag1	122.66 (17)
F1B—C19—F3B	110.4 (7)	C1—N1—Ag1	120.10 (16)
F2B—C19—F3B	99.7 (7)	N1—C1—C2	123.1 (2)
F2A—C19—F1A	102.9 (7)	C1—C2—C3	119.7 (2)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*+1, −*y*+2, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1	0.94	2.53	3.160 (3)	124