Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

catena-Poly[[(tetrafluoroborato-κF)silver(I)]-μ-triphenylphosphine-$\kappa^2 P$:C^3]

Simon K. Brayshaw,* Eduardo Molinos and Andrew S. Weller

Department of Chemistry, University of Bath, Bath BA2 7AY, England

Correspondence e-mail: sb285@bath.ac.uk

Received 29 November 2006
Accepted 15 December 2006

The title compound, $[Ag(BF_4)(C_{18}H_{15}P)]_n$, crystallizes from dichloromethane–pentane as a one-dimensional coordination polymer in which the Ag atom is bound to a phosphine P atom, one F atom of tetrafluoroborate and one C atom of a neighbouring triphenylphosphine ligand.

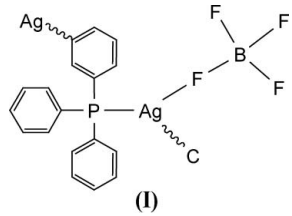
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
wR factor = 0.072
Data-to-parameter ratio = 22.6

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

Comment

Complexes of silver in which close metal–arene interactions are present in the solid state are not uncommon, with the first example reported by Smith & Rundle (1958). Typically, in such complexes, the silver is partnered with weakly or non-coordinating anions such as trifluoromethanesulfonate or perchlorate. On the other hand, there have been few reports of solid state structures of silver complexes which contain bound tetrafluoroborate.



(I)

We have previously described (tertiary phosphine)silver complexes of functionalized 1-*closo*-carborane anions (Patmore *et al.*, 2002; Clarke *et al.*, 2004). Whilst attempting to prepare one such complex from silver tetrafluoroborate and $[(PPh_3)_2Rh(nbd)]\cdot CB_{11}H_7Et_5$ (Molinos *et al.*, 2005), colourless single crystals suitable for an X-ray diffraction experiment were obtained. The crystals were determined to be the title complex, (I), and the results of the diffraction study are described below.

In (I) (Fig. 1), the coordination of the silver is quasi-trigonal, the silver bonding to P, F1 and C3^{i} [symmetry code: (i) $\frac{3}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$], with the silver having only slight deviation from the P—F—C ligand plane [0.0672 (7) Å]. The Ag—C3^{i} and Ag—F1 distances are long (Table 1), but are consistent with bonding interactions, and the coordination of C3^{i} results in a one-dimensional coordination polymer. As expected, the coordination of F1 results in a B—F1 distance greater than the other B—F distances.

There are two other Ag···F contacts within van der Waals radii. An Ag···F2 contact is accommodated by a small Ag—F1—B—F2 torsion angle and a reduced F1—B—F2 angle. The effect of this close contact is also seen in an increased P—Ag—F1 angle relative to P—Ag—C3^{i} and F1—Ag—C3^{i}. Finally, an Ag···F contact occurs between Ag and F3^{ii} [symmetry code:

(ii) $1-x, -y, -z$] in a pairwise manner, with a matching contact between the symmetry-related Ag^{ii} and F3 (Fig. 2).

Experimental

A solution containing equimolar quantities of silver tetrafluoroborate and $[(PPh_3)_2Rh(nbd)]\cdot CB_{11}H_7Et_5$ (Molinos *et al.*, 2005) in dichloromethane was layered with pentanes and held at 278 K for one week to crystallize. A crystal of (I) suitable for a single-crystal X-ray diffraction study was selected directly from the sample.

Crystal data

$[Ag(BF_4)(C_{18}H_{15}P)]$
$M_r = 456.95$
Monoclinic, $P2_1/n$
$a = 12.0606$ (1) Å
$b = 11.2379$ (1) Å
$c = 12.9254$ (1) Å
$\beta = 90.0093$ (7)°
$V = 1751.85$ (3) Å^3
$Z = 4$
$D_x = 1.733$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.28$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.33 × 0.25 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.678$, $T_{max} = 0.803$
31041 measured reflections
5109 independent reflections
4717 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.073$
$S = 1.04$
5109 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 1.6142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.01$ e Å^{-3}
$\Delta\rho_{min} = -1.37$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag—P	2.3903 (4)	B—F2	1.372 (3)
Ag—F1	2.4242 (13)	B—F3	1.380 (2)
Ag—C3^{i}	2.5706 (18)	B—F1	1.411 (3)
B—F4	1.367 (3)		
Ag···F3^{ii}	2.6912 (14)	Ag···F2	2.913 (2)
P—Ag—F1	148.19 (4)	F4—B—F1	109.54 (19)
P—Ag—C3^{i}	129.96 (5)	F2—B—F1	107.37 (18)
F1—Ag—C3^{i}	81.56 (6)	F3—B—F1	108.32 (17)
F4—B—F3	109.72 (17)	B—F1—Ag	112.70 (12)
F2—B—F3	110.87 (19)		
Ag—F1—B—F2	−5.7 (2)		

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

H atoms were located in difference Fourier maps and placed in idealized positions, with C—H = 0.95 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. The largest peak and deepest hole in the final difference map are located 0.75 and 0.60 Å from the Ag atom, respectively.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

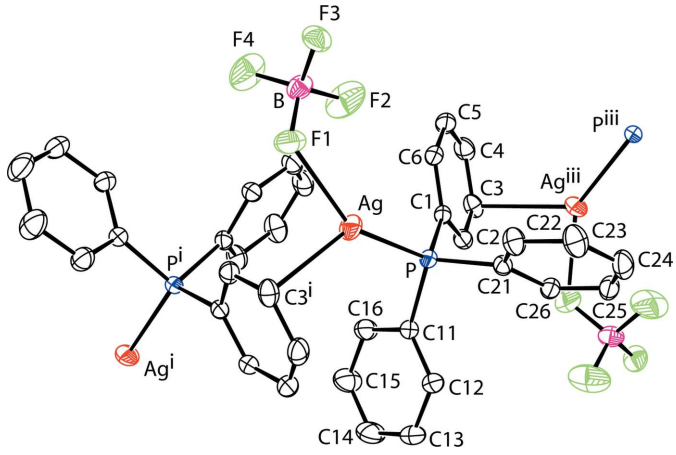


Figure 1
Part of the polymeric structure of (I), showing its polymeric nature. Displacement ellipsoids are shown at the 50% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

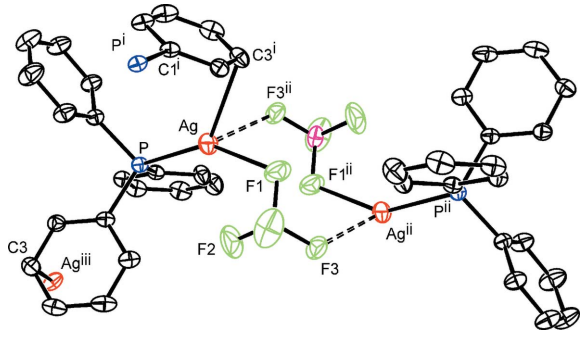


Figure 2
Two asymmetric units of (I), together with neighbouring Ag^{iii} and $C_6H_5P^{ii}$ groups, showing the pairwise packing. Displacement ellipsoids are shown at the 50% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $1-x,-y,-z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

The EPSCR and Royal Society are thanked for financial support.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Clarke, A. J., Ingleson, M. J., Kociok-Köhn, G., Mahon, M. F., Patmore, N. J., Rourke, J. P., Ruggiero, G. D. & Weller, A. S. (2004). *J. Am. Chem. Soc.* **126**, 1503–1517.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Molinos, E., Kociok-Köhn, G. & Weller, A. S. (2005). *Chem. Commun.* pp. 3609–3611.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Patmore, N. J., Hague, C., Cotgreave, J. H., Mahon, M. F., Frost, C. G. & Weller, A. S. (2002). *Chem. Eur. J.* **8**, 2088–2098.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Smith, H. G. & Rundle, R. E. (1958). *J. Am. Chem. Soc.* **80**, 5075–5080.

supporting information

Acta Cryst. (2007). E**63**, m302–m303 [https://doi.org/10.1107/S1600536806054511]

catena-Poly[[(tetrafluoroborato-κF)silver(I)]-μ-triphenylphosphine-$\kappa^2 P$:C^3]

Simon K. Brayshaw, Eduardo Molinos and Andrew S. Weller

***catena*-Poly[[(tetrafluoroborato-κF)silver(I)]-μ-triphenylphosphine- $\kappa^2 P$:C^3]**

Crystal data

$[Ag(BF_4)(C_{18}H_{15}P)]$
$M_r = 456.95$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.0606$ (1) Å
$b = 11.2379$ (1) Å
$c = 12.9254$ (1) Å
$\beta = 90.0093$ (7)°
$V = 1751.85$ (3) Å^3
$Z = 4$
$F(000) = 904$
$D_x = 1.733$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 38097 reflections
$\theta = 2.9$–30.0°
$\mu = 1.28$ mm^{-1}
$T = 150$ K
Block, colourless
0.33 × 0.25 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
652 1.0° images with φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.678$, $T_{max} = 0.803$
31041 measured reflections
5109 independent reflections
4717 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 30.0°$, $\theta_{min} = 3.8°$
$h = -16 \rightarrow 16$
$k = -15 \rightarrow 15$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.073$
$S = 1.04$
5109 reflections
226 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 1.6142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.02$ e Å^{-3}
$\Delta\rho_{min} = -1.37$ e Å^{-3}

Special details

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag	0.611584 (13)	−0.042004 (13)	0.164743 (11)	0.03024 (6)
P	0.69044 (3)	0.09718 (4)	0.28367 (3)	0.01861 (8)
C1	0.82162 (14)	0.16844 (14)	0.25136 (13)	0.0200 (3)
C2	0.89562 (14)	0.20668 (15)	0.32828 (13)	0.0229 (3)
H2	0.8786	0.1939	0.3992	0.028*
C3	0.99410 (15)	0.26337 (15)	0.30070 (15)	0.0266 (3)
H3	1.0445	0.2889	0.3526	0.032*
C4	1.01831 (16)	0.28241 (17)	0.19599 (17)	0.0316 (4)
H4	1.0856	0.3204	0.1769	0.038*
C5	0.94475 (18)	0.24616 (16)	0.12056 (16)	0.0316 (4)
H5	0.9612	0.2603	0.0497	0.038*
C6	0.84627 (16)	0.18891 (15)	0.14766 (14)	0.0253 (3)
H6	0.7961	0.1639	0.0954	0.030*
C11	0.71924 (14)	0.02228 (14)	0.40527 (13)	0.0209 (3)
C12	0.64926 (17)	0.03004 (16)	0.49078 (14)	0.0271 (4)
H12	0.5866	0.0812	0.4889	0.032*
C13	0.6713 (2)	−0.03743 (18)	0.57922 (16)	0.0352 (4)
H13	0.6234	−0.0320	0.6374	0.042*
C14	0.7621 (2)	−0.1117 (2)	0.58266 (18)	0.0431 (5)
H14	0.7772	−0.1566	0.6433	0.052*
C15	0.8318 (2)	−0.1210 (2)	0.49674 (18)	0.0423 (5)
H15	0.8940	−0.1728	0.4987	0.051*
C16	0.81032 (17)	−0.05452 (17)	0.40873 (16)	0.0307 (4)
H16	0.8578	−0.0612	0.3504	0.037*
C21	0.60075 (14)	0.22346 (14)	0.31065 (13)	0.0206 (3)
C22	0.50795 (16)	0.24133 (16)	0.24879 (16)	0.0290 (4)
H22	0.4890	0.1843	0.1975	0.035*
C23	0.44245 (17)	0.34282 (19)	0.26175 (19)	0.0368 (4)
H23	0.3788	0.3545	0.2196	0.044*
C24	0.47023 (17)	0.42631 (18)	0.33594 (18)	0.0344 (4)
H24	0.4259	0.4955	0.3444	0.041*
C25	0.56295 (16)	0.40925 (16)	0.39822 (15)	0.0294 (4)
H25	0.5818	0.4669	0.4490	0.035*
C26	0.62809 (15)	0.30801 (15)	0.38628 (13)	0.0231 (3)
H26	0.6910	0.2962	0.4293	0.028*
B	0.66670 (19)	−0.0528 (2)	−0.08054 (17)	0.0296 (4)
F1	0.59061 (13)	−0.10780 (13)	−0.01281 (10)	0.0465 (3)
F2	0.72340 (16)	0.03121 (18)	−0.02458 (14)	0.0662 (5)
F3	0.60793 (12)	−0.00064 (12)	−0.16008 (10)	0.0379 (3)
F4	0.73747 (15)	−0.13653 (18)	−0.11968 (13)	0.0648 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag	0.03680 (9)	0.02773 (8)	0.02618 (8)	−0.00542 (5)	−0.00191 (6)	−0.00851 (5)
P	0.02229 (19)	0.01627 (17)	0.01727 (18)	−0.00097 (14)	0.00198 (14)	−0.00160 (13)
C1	0.0230 (7)	0.0157 (6)	0.0212 (7)	0.0002 (5)	0.0045 (6)	−0.0010 (5)
C2	0.0238 (8)	0.0204 (7)	0.0247 (8)	0.0007 (6)	0.0007 (6)	0.0018 (6)
C3	0.0218 (8)	0.0209 (7)	0.0370 (9)	0.0011 (6)	−0.0014 (7)	0.0036 (7)
C4	0.0288 (9)	0.0237 (8)	0.0424 (11)	−0.0015 (7)	0.0120 (8)	0.0042 (7)
C5	0.0407 (10)	0.0230 (8)	0.0309 (9)	−0.0038 (7)	0.0158 (8)	−0.0005 (7)
C6	0.0343 (9)	0.0191 (7)	0.0223 (8)	−0.0012 (6)	0.0065 (6)	−0.0025 (6)
C11	0.0251 (8)	0.0184 (7)	0.0193 (7)	−0.0001 (6)	0.0021 (6)	−0.0002 (5)
C12	0.0319 (9)	0.0261 (8)	0.0231 (8)	0.0036 (7)	0.0061 (7)	0.0019 (6)
C13	0.0457 (12)	0.0359 (10)	0.0241 (9)	0.0037 (8)	0.0094 (8)	0.0059 (7)
C14	0.0528 (13)	0.0456 (12)	0.0310 (10)	0.0101 (10)	0.0033 (9)	0.0159 (9)
C15	0.0415 (11)	0.0459 (12)	0.0396 (11)	0.0171 (10)	0.0053 (9)	0.0150 (9)
C16	0.0325 (9)	0.0307 (9)	0.0288 (9)	0.0076 (7)	0.0068 (7)	0.0060 (7)
C21	0.0217 (7)	0.0175 (7)	0.0226 (7)	−0.0010 (5)	0.0030 (6)	0.0001 (6)
C22	0.0269 (8)	0.0238 (8)	0.0361 (10)	−0.0010 (6)	−0.0062 (7)	−0.0020 (7)
C23	0.0280 (9)	0.0303 (9)	0.0520 (13)	0.0046 (7)	−0.0056 (8)	0.0014 (9)
C24	0.0303 (9)	0.0241 (8)	0.0489 (12)	0.0064 (7)	0.0087 (8)	0.0003 (8)
C25	0.0333 (9)	0.0216 (8)	0.0333 (9)	0.0006 (7)	0.0095 (7)	−0.0040 (7)
C26	0.0257 (8)	0.0206 (7)	0.0230 (7)	−0.0007 (6)	0.0031 (6)	−0.0023 (6)
B	0.0291 (10)	0.0369 (11)	0.0229 (9)	0.0034 (8)	−0.0060 (7)	−0.0042 (8)
F1	0.0651 (9)	0.0474 (8)	0.0270 (6)	−0.0102 (7)	0.0058 (6)	0.0015 (5)
F2	0.0600 (10)	0.0889 (14)	0.0497 (9)	−0.0271 (9)	−0.0101 (8)	−0.0274 (9)
F3	0.0452 (7)	0.0356 (6)	0.0328 (6)	0.0025 (5)	−0.0092 (5)	0.0080 (5)
F4	0.0595 (10)	0.0807 (12)	0.0543 (9)	0.0422 (9)	−0.0040 (8)	−0.0117 (8)

Geometric parameters (Å, º)

Ag—P	2.3903 (4)	C13—C14	1.378 (3)
Ag—F1	2.4242 (13)	C13—H13	0.9500
Ag—C3^{i}	2.5706 (18)	C14—C15	1.397 (3)
P—C11	1.8163 (17)	C14—H14	0.9500
P—C21	1.8182 (17)	C15—C16	1.385 (3)
P—C1	1.8219 (17)	C15—H15	0.9500
C1—C6	1.392 (2)	C16—H16	0.9500
C1—C2	1.403 (2)	C21—C22	1.390 (2)
C2—C3	1.394 (2)	C21—C26	1.402 (2)
C2—H2	0.9500	C22—C23	1.398 (3)
C3—C4	1.401 (3)	C22—H22	0.9500
C3—Ag^{ii}	2.5706 (18)	C23—C24	1.383 (3)
C3—H3	0.9500	C23—H23	0.9500
C4—C5	1.380 (3)	C24—C25	1.391 (3)
C4—H4	0.9500	C24—H24	0.9500
C5—C6	1.396 (3)	C25—C26	1.391 (2)
C5—H5	0.9500	C25—H25	0.9500

C6—H6	0.9500	C26—H26	0.9500
C11—C12	1.393 (2)	B—F4	1.367 (3)
C11—C16	1.398 (2)	B—F2	1.372 (3)
C12—C13	1.397 (3)	B—F3	1.380 (2)
C12—H12	0.9500	B—F1	1.411 (3)
Ag···F3^{iii}	2.6912 (14)	Ag···F2	2.913 (2)
P—Ag—F1	148.19 (4)	C14—C13—H13	119.8
P—Ag—C3^{i}	129.96 (5)	C12—C13—H13	119.8
F1—Ag—C3^{i}	81.56 (6)	C13—C14—C15	119.86 (19)
C11—P—C21	108.03 (8)	C13—C14—H14	120.1
C11—P—C1	103.66 (8)	C15—C14—H14	120.1
C21—P—C1	102.57 (7)	C16—C15—C14	120.0 (2)
C11—P—Ag	109.22 (5)	C16—C15—H15	120.0
C21—P—Ag	113.41 (6)	C14—C15—H15	120.0
C1—P—Ag	119.06 (5)	C15—C16—C11	120.41 (18)
C6—C1—C2	119.71 (15)	C15—C16—H16	119.8
C6—C1—P	118.61 (13)	C11—C16—H16	119.8
C2—C1—P	121.63 (12)	C22—C21—C26	119.49 (16)
C3—C2—C1	120.04 (16)	C22—C21—P	118.78 (13)
C3—C2—H2	120.0	C26—C21—P	121.51 (13)
C1—C2—H2	120.0	C21—C22—C23	120.27 (18)
C2—C3—C4	119.63 (18)	C21—C22—H22	119.9
C2—C3—Ag^{ii}	85.52 (11)	C23—C22—H22	119.9
C4—C3—Ag^{ii}	98.12 (12)	C24—C23—C22	120.00 (19)
C2—C3—H3	120.2	C24—C23—H23	120.0
C4—C3—H3	120.2	C22—C23—H23	120.0
Ag^{ii}—C3—H3	86.4	C23—C24—C25	120.17 (18)
C5—C4—C3	120.24 (17)	C23—C24—H24	119.9
C5—C4—H4	119.9	C25—C24—H24	119.9
C3—C4—H4	119.9	C24—C25—C26	120.16 (18)
C4—C5—C6	120.39 (17)	C24—C25—H25	119.9
C4—C5—H5	119.8	C26—C25—H25	119.9
C6—C5—H5	119.8	C25—C26—C21	119.92 (17)
C1—C6—C5	119.98 (18)	C25—C26—H26	120.0
C1—C6—H6	120.0	C21—C26—H26	120.0
C5—C6—H6	120.0	F4—B—F2	110.9 (2)
C12—C11—C16	119.30 (16)	F4—B—F3	109.72 (17)
C12—C11—P	122.79 (14)	F2—B—F3	110.87 (19)
C16—C11—P	117.64 (13)	F4—B—F1	109.54 (19)
C11—C12—C13	119.98 (18)	F2—B—F1	107.37 (18)
C11—C12—H12	120.0	F3—B—F1	108.32 (17)
C13—C12—H12	120.0	B—F1—Ag	112.70 (12)
C14—C13—C12	120.43 (19)		

Ag—F1—B—F2	−5.7 (2)

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*, −*z*.