

Novel three-dimensional coordination polymer of 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]-decan-7-yl)ethanoic acid with silver(I) tetrafluoroborate

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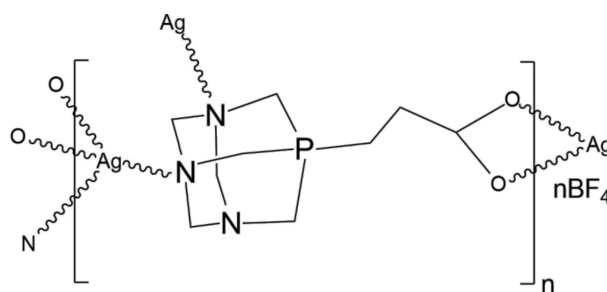
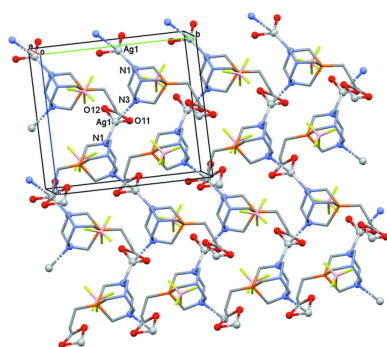
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An Ag^I-based coordination polymer (CP), namely, poly[[[μ₃-2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]-decan-7-yl)ethanoate-κ⁴N:N':O,O']silver(I)] tetrafluoroborate], {[Ag(C₉H₁₆N₃O₂P)]BF₄]_n, was synthesized in an aqueous solution of zwitterionic 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]-decan-7-yl)ethanoate (**L**) and AgBF₄ with exclusion of light at room temperature. The colourless and light-insensitive CP crystallized in the monoclinic space group *Cc*. The asymmetric unit consists of an Ag^I cation, the zwitterionic **L** ligand and a BF₄⁻ counter-ion. Each Ag^I ion is coordinated by two carboxylate oxygen atoms in a chelating coordination mode, as well as one of the nitrogen atoms of two neighbouring **L** ligands. The crystal structure of the CP was classified as a unique three-dimensional arrangement. The CP was also characterized in aqueous solutions by multinuclear NMR and HRMS spectroscopies and elemental analysis.

1. Chemical context

The architectures and antimicrobial properties of self-assembled silver-based coordination polymers (CPs) or MOFs (metal-organic frameworks), bridged by phosphatrotropines, have been widely studied (Guerriero *et al.*, 2018). According to our previous studies, the aqueous reaction of zwitterionic 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]-decan-7-yl)ethanoate (**L**) with Ag X ($X = \text{PF}_6, \text{SO}_3\text{C}_6\text{H}_4\text{CH}_3, \text{SO}_3\text{CF}_3$) yielded various 1D Ag-based coordination polymers (Udvardy *et al.*, 2021). The architectures of these Ag^I complexes depend on their counter-ions and the position of the ligand, which contains both rigid and flexible molecular moieties.



Herein, we report the crystal structure of a CP prepared by the aqueous reaction of 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]-decan-7-yl)ethanoate and AgBF₄ with the exclusion of light at 278 K (Fig. 1). The colourless crystals of

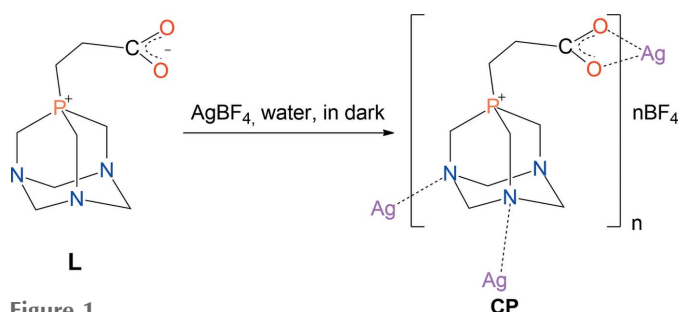


Figure 1
Schematic representation of the formation of the title compound.

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1A\cdots F2$	0.97	2.40	3.201 (13)	140

the CP were isolated by filtration, dissolved in water and characterized by ^1H -, ^{13}C - and ^{31}P -NMR spectroscopy, ESI mass spectrometry, as well as by elemental analysis.

The chemical shift of the phosphorus atom in CP ($\delta = -37.5$ ppm in D_2O) was the same as that in the free ligand. Similar to the hexafluorophosphate, tosylate (tos) and triflate (OTf) derivatives (Udvardy *et al.*, 2021), the ^1H -NMR spectrum showed differences between the $\text{P}^+-\text{CH}_2-\text{N}$ and $\text{N}-\text{CH}_2-\text{N}$ signals, which clearly indicated the coordination of the silver ions to the nitrogen donor atoms of the **L** ligand.

The most intense ESI-MS signals of the CP (aqueous solution, positive ion mode) were observed at $m/z = 252.0878$ ($[\text{L}+\text{Na}]^+$, $\text{C}_9\text{H}_{16}\text{N}_3\text{NaO}_2\text{P}$, calculated. 252.0872), 336.0026 ($[\text{L}+\text{Ag}]^+$, $\text{C}_9\text{H}_{16}\text{N}_3\text{AgO}_2\text{P}$, calculated 336.0026), and 565.1009

($[2\text{L}+\text{Ag}]^+$, $\text{C}_{18}\text{H}_{32}\text{N}_6\text{NaO}_4\text{P}_2$, calculated 565.1005). Similar ions were detected for the CP formed with AgPF_6 , $\text{AgSO}_3\text{C}_6\text{H}_4\text{CH}_3$, AgSO_3CF_3 and PTA in aqueous solutions.

2. Structural commentary

The molecular structure of the title compound is shown in Fig. 2. The CP crystallized in the monoclinic Cc space group. The asymmetric unit consists of a silver(I) cation, a zwitterionic **L** ligand and a BF_4^- counter-ion, in which the N,N',O,O' coordination mode of the silver(I) ions creates a 3D coordination architecture (Fig. 2).

In the CP, the central Ag^+ ion is coordinated by an **L** ligand via two carboxylate oxygen atoms [$\text{Ag}1^2-\text{O}11 = 2.594$ (9) Å and $\text{Ag}1^2-\text{O}12 = 2.298$ (8) Å] and two nitrogen atoms from two adjacent PTA moieties of **L** [$\text{Ag}1-\text{N}1 = 2.225$ (7) Å and $\text{Ag}1^1-\text{N}3 = 2.505$ (7) Å]. The $\text{N}1-\text{Ag}-\text{N}3^3$ and $\text{O}11^4-\text{Ag}-\text{O}12^4$ bond angles are 119.6 (3) and 52.9 (2)°, respectively. Selected bond lengths and bond angles are presented in the supporting information. The coordination geometry exhibits a distorted tetrahedral shape ($\tau_4 = 0.65$ and $\tau_4' = 0.66$; Yang *et al.*, 2007; Okuniewski *et al.*, 2015), in which the Ag^1 ion is located at the centre. The space between the 3D polymer backbones is occupied by the BF_4^- counter-ions (Fig. 3). The chemical composition was also determined by elemental analysis, which shows a good agreement with the SC-XRD results (see *Synthesis and crystallization*).

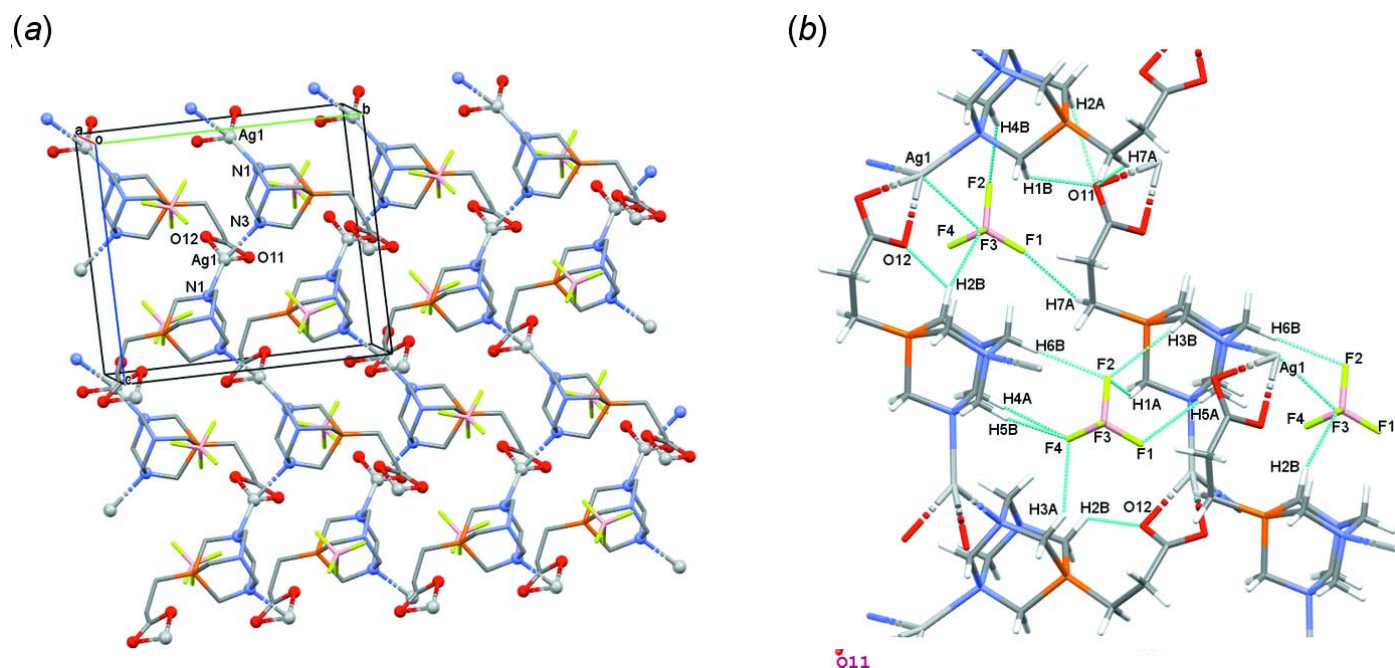


Figure 3
(a) Packing arrangement of the three-dimensional structure of the CP in the crystal viewed along the crystallographic a axis. The coordination sphere is labelled and highlighted by a ball-and-stick model. Hydrogen atoms are omitted for clarity. (b) Selected hydrogen-bond geometry in the CP showing the weak $\text{C}-\text{H}\cdots\text{F}$ and $\text{C}-\text{H}\cdots\text{O}$ secondary interactions, as well as the $\text{Ag}1\cdots\text{F}3$ interaction. For symmetry codes, see Table 1.

3. Supramolecular features

As a result of the lack of primary H-donor groups, no classical hydrogen bonds are found in the crystal structure of the title coordination polymer. The main intermolecular interactions between the molecules in the crystal are weak C—H...F and C—H...O type hydrogen bonds. The BF₄[−] anion is generally classified as a non-coordinating anion owing to its weak Lewis base properties (Grabowski, 2020). These secondary interactions play a major role in stabilizing the crystal lattice by connecting the molecular units to each other, which results in a 3D coordination polymer. All of the fluorine atoms of a BF₄[−] counter-ion are connected to at least one C—H hydrogen atom by a weak C—H...F type hydrogen bond. The shortest C—H...F distance is found for the C2—H2B...F3 interaction [C2...F3 = 3.183 (13) Å], where the F3 atom of the BF₄[−] counter-ion is also able to coordinate to the central Ag⁺ ion with a distance of 3.010 (11) Å (Fig. 3). This ionic attraction between the Ag⁺ and BF₄[−] ions is strong enough to affect the arrangement of part of the whole complex molecule and form a bent 3D structure. In comparison, the value of the longest C—H...F distance is 3.417 (14) Å (C4—H4B...F2, Fig. 3) owing to the rigid PTA cage, which is unable to change its conformation. There are numerous examples in the literature of where the C—H...F distances were investigated in the presence of BF₄[−] counter-ions [*i.e.* BIXBIT03 (Emge *et al.*, 1986) and SUXHID01 (Albinati *et al.*, 2010)]. In case of the bis[μ₂-1,1'-naphthalene-1,8-diyl-bis(1*H*-pyrazole)]tris(acetonitrile)disilver(I) bis(BF₄) acetonitrile solvate structure (OGINOI; Liddle *et al.*, 2009), it was found that the typical C...F distances are between 3.179 (2) and 3.406 (3) Å, which shows a good agreement with our results. The carboxylate oxygen atoms in the title CP are also able to form weak C—H...O type interactions with the C—H atoms of the complex molecule. Their atomic distances can also be compared to the C—H...F secondary interactions. An intramolecular hydrogen bond also helps to form a bent 3D molecular structure for the CP [C2...O12 = 2.812 (12) Å]. For selected hydrogen-bond distances and angles see Fig. 3*b* and Table 1. The considerably high calculated density (2.102 Mg m^{−3}) and KPI (Kitaigorodskii packing index) of 74.2% (Spek, 2020) indicate the tight packing arrangement of the molecules, resulting in no residual solvent-accessible voids in the crystal structure.

4. Database survey

A survey of the Cambridge Structural Database (CSD version 5.42, Sept. 2021 update; Groom *et al.*, 2016) found zwitterionic 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]decan-7-yl)ethanoate dihydrate (**L**) (SIJPOR; Tang *et al.*, 2007) and three 1D Ag-based coordination polymers containing **L**, *viz.* [Ag(μ₃-**L**-κ³N:O:O)]_n(PF₆)_n (UPUCAM; Udvardy *et al.*, 2021), [Ag(OTf)(μ₃-**L**-κ³N:O:O)]_n (UPUCIU; Udvardy *et al.*, 2021) and [Ag(tos)(μ₃-**L**-κ³N:N:O)]_n·*n*H₂O (UPUCEQ; Udvardy *et al.*, 2021). While in the cases of UPUCAM, UPUCIU and UPUCEQ only 1D polymers were obtained, in the title CP the

Table 2
Experimental details.

Crystal data	
Chemical formula	[Ag(C ₉ H ₁₆ N ₃ O ₂ P)]BF ₄
<i>M_r</i>	423.90
Crystal system, space group	Monoclinic, Cc
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.116 (5), 12.186 (5), 10.979 (5)
β (°)	98.260 (5)
<i>V</i> (Å ³)	1339.4 (11)
<i>Z</i>	4
Radiation type	Mo Kα
μ (mm ^{−1})	1.68
Crystal size (mm)	0.35 × 0.2 × 0.15
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	ψ scan (North <i>et al.</i> , 1968)
<i>T</i> _{min} , <i>T</i> _{max}	0.558, 0.755
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	1358, 1313, 1299
<i>R</i> _{int}	0.009
(sin θ/λ) _{max} (Å ^{−1})	0.605
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.048, 0.123, 1.13
No. of reflections	1313
No. of parameters	190
No. of restraints	2
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ^{−3})	1.20, −1.54
Absolute structure	Classical Flack method preferred over Parsons because s.u. lower
Absolute structure parameter	0.13 (6)

Computer programs: *MACH3/PC* (Enraf–Nonius, 1992), *PROFIT* (Streltsov & Zavodnik, 1989), *SIR97* (Burla *et al.*, 2007), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *pubCIF* (Westrip, 2010).

Ag^I complex is able to form a 3D coordination polymer owing to the relatively small size of the BF₄[−] counter-ion, which is able to occupy a smaller space compared to the PF₆[−], triflate or tosylate anions. These results show how a counter-ion can influence the packing arrangement and the coordination mode of an [(Ag**L**)*X*] type polymer.

5. Synthesis and crystallization

Water-soluble PTA (Daigle, 1998) and 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]decan-7-yl)ethanoate (**L**) (Tang *et al.*, 2007; Udvardy *et al.*, 2021) were prepared according to literature methods.

CP: With the exclusion of light, 4 mL aqueous solution containing 194.7 mg (1 mmol) AgBF₄ was added to an aqueous solution (4 mL) of **L** (100 mg, 0.44 mmol). The reaction mixture was stored at 278 K. After two days, the CP was formed as colourless crystals, which were separated by filtration and dried. Yield (based on **L**) 112 mg, 60%. ¹H NMR (360 MHz, D₂O, 298 K) δ 4.73–4.37 (*m*, 12H, ⁺P–CH₂–N, N–CH₂–N), 2.58 (*dt*, *J* = 24, 7 Hz, 2H, P⁺–CH₂–CH₂–COO), 2.44–2.22 (*m*, 2H, P⁺–CH₂–CH₂–COO) ppm. ¹³C{¹H} NMR (90 MHz, D₂O, 298 K) δ 179.5 (*s*, COO[−]), 71.5 (*d*, ³*J*_{PC} = 8 Hz, N–CH₂–N), 49.1 (*d*, ¹*J*_{PC} = 37 Hz, ⁺P–CH₂–N), 29.0 (*d*, ²*J*_{PC} = 7 Hz, P⁺–CH₂–CH₂–COO[−]), 18.5 (*d*, ¹*J*_{PC} = 35 Hz, P⁺–CH₂–CH₂–COO[−]) ppm. ³¹P{¹H} NMR (145 MHz, D₂O, 25 °C) δ

−37.5 (*s*) ppm. Elemental analysis: C₉H₁₆AgBF₄N₃O₂P (423.89): calculated C 25.05, H 3.80, N 9.91; found C 25.64, H 4.10, N 9.95.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms of the CP complex were positioned geometrically and refined using a riding model, with C–H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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supporting information

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Novel three-dimensional coordination polymer of 2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]decan-7-yl)ethanoic acid with silver(I) tetrafluoroborate

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Computing details

Data collection: *MACH3/PC* (Enraf–Nonius, 1992); cell refinement: *MACH3/PC* (Enraf–Nonius, 1992); data reduction: *PROFIT* (Streltsov & Zavodnik, 1989); program(s) used to solve structure: *SIR97* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Poly[[[μ_3 -2-(1,3,5-triaza-7-phosphoniatricyclo[3.3.1.1^{3,7}]decan-7-yl)ethanoate- κ^4 N:N':O,O']silver(I)]tetrafluoroborate]

Crystal data

[Ag(C₉H₁₆N₃O₂P)]BF₄

$M_r = 423.90$

Monoclinic, *Cc*

$a = 10.116$ (5) Å

$b = 12.186$ (5) Å

$c = 10.979$ (5) Å

$\beta = 98.260$ (5)°

$V = 1339.4$ (11) Å³

$Z = 4$

$F(000) = 840$

$D_x = 2.102$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9.1$ – 17.2 °

$\mu = 1.68$ mm⁻¹

$T = 293$ K

Prism, colourless

$0.35 \times 0.2 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

profiled $\omega/2\theta$ scans

Absorption correction: ψ scan

(North *et al.*, 1968)

$T_{\min} = 0.558$, $T_{\max} = 0.755$

1358 measured reflections

1313 independent reflections

1299 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.009$

$\theta_{\max} = 25.5$ °, $\theta_{\min} = 3.1$ °

$h = 0 \rightarrow 12$

$k = 0 \rightarrow 14$

$l = -13 \rightarrow 13$

3 standard reflections every 184 reflections

intensity decay: 2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.123$

$S = 1.13$

1313 reflections

190 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0873P)^2 + 4.2725P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.20$ e Å⁻³

$$\Delta\rho_{\min} = -1.54 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Classical Flack method
preferred over Parsons because s.u. lower
Absolute structure parameter: 0.13 (6)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	−0.0444 (9)	−0.2414 (7)	−0.2977 (8)	0.0266 (16)
H1A	0.0509	−0.2557	−0.2849	0.032*
H1B	−0.0820	−0.2737	−0.3758	0.032*
C2	−0.2914 (9)	−0.2548 (7)	−0.2148 (9)	0.0302 (19)
H2A	−0.3323	−0.2870	−0.2918	0.036*
H2B	−0.3444	−0.2743	−0.1511	0.036*
C3	−0.0546 (9)	−0.2182 (7)	−0.0445 (8)	0.0284 (17)
H3A	−0.0975	−0.2365	0.0265	0.034*
H3B	0.0408	−0.2298	−0.0232	0.034*
C4	−0.2157 (10)	−0.1011 (7)	−0.3251 (9)	0.0317 (19)
H4A	−0.2311	−0.0233	−0.3392	0.038*
H4B	−0.2532	−0.1397	−0.3994	0.038*
C5	−0.0139 (9)	−0.0704 (7)	−0.1840 (8)	0.0269 (16)
H5A	0.0798	−0.0897	−0.1646	0.032*
H5B	−0.0192	0.0087	−0.1934	0.032*
C6	−0.2256 (10)	−0.0841 (8)	−0.1135 (10)	0.035 (2)
H6A	−0.2706	−0.1113	−0.0472	0.042*
H6B	−0.2418	−0.0057	−0.1203	0.042*
C7	−0.0690 (9)	−0.4434 (7)	−0.1593 (9)	0.0311 (19)
H7A	−0.1281	−0.4868	−0.2178	0.037*
H7B	0.0201	−0.4492	−0.1815	0.037*
C8	−0.0676 (11)	−0.4934 (8)	−0.0308 (9)	0.0340 (19)
H8A	−0.0322	−0.5674	−0.0306	0.041*
H8B	−0.0084	−0.4506	0.0285	0.041*
C9	−0.2032 (10)	−0.4967 (8)	0.0078 (8)	0.0298 (18)
N1	−0.0681 (7)	−0.1216 (6)	−0.3016 (6)	0.0253 (14)
N2	−0.2837 (7)	−0.1355 (6)	−0.2263 (8)	0.0323 (17)
N3	−0.0822 (8)	−0.1022 (6)	−0.0806 (7)	0.0278 (15)
O11	−0.2377 (9)	−0.5691 (6)	0.0750 (8)	0.0444 (17)
O12	−0.2793 (8)	−0.4175 (6)	−0.0319 (7)	0.0408 (16)
P1	−0.1202 (2)	−0.30412 (17)	−0.1746 (2)	0.0234 (4)
B1	0.3222 (13)	−0.2721 (10)	−0.1875 (11)	0.039 (2)
Ag1	0.03866 (7)	−0.03737 (7)	−0.43859 (7)	0.0488 (3)
F1	0.2885 (9)	−0.1691 (6)	−0.2226 (8)	0.062 (2)
F2	0.2229 (8)	−0.3155 (7)	−0.1250 (9)	0.066 (2)
F3	0.4361 (8)	−0.2745 (9)	−0.1032 (10)	0.082 (3)

F4	0.3319 (16)	-0.3351 (7)	-0.2846 (9)	0.104 (4)
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Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.025 (4)	0.023 (4)	0.033 (4)	0.002 (3)	0.013 (3)	-0.004 (3)
C2	0.022 (4)	0.022 (4)	0.047 (5)	0.005 (3)	0.006 (4)	0.005 (4)
C3	0.029 (4)	0.027 (4)	0.029 (4)	0.007 (3)	0.003 (3)	0.001 (3)
C4	0.028 (5)	0.027 (4)	0.040 (5)	-0.001 (3)	0.001 (4)	0.008 (3)
C5	0.029 (4)	0.025 (4)	0.027 (4)	-0.004 (3)	0.008 (3)	-0.003 (3)
C6	0.031 (5)	0.028 (4)	0.049 (5)	0.010 (4)	0.019 (4)	-0.001 (4)
C7	0.026 (4)	0.029 (4)	0.042 (5)	-0.001 (3)	0.016 (4)	0.000 (4)
C8	0.040 (5)	0.031 (4)	0.030 (4)	0.004 (4)	0.001 (4)	0.009 (4)
C9	0.033 (5)	0.026 (4)	0.028 (4)	-0.001 (4)	-0.002 (3)	0.002 (4)
N1	0.023 (3)	0.025 (3)	0.027 (3)	-0.001 (3)	0.003 (3)	0.005 (3)
N2	0.023 (4)	0.027 (4)	0.048 (5)	0.001 (3)	0.008 (3)	0.005 (3)
N3	0.030 (4)	0.021 (3)	0.033 (4)	0.001 (3)	0.009 (3)	-0.002 (3)
O11	0.049 (4)	0.034 (3)	0.052 (4)	-0.002 (3)	0.013 (3)	0.016 (3)
O12	0.042 (4)	0.037 (3)	0.046 (4)	0.011 (3)	0.016 (3)	0.012 (3)
P1	0.0211 (10)	0.0193 (9)	0.0306 (10)	0.0024 (8)	0.0065 (8)	0.0009 (8)
B1	0.043 (7)	0.038 (6)	0.037 (5)	0.009 (5)	0.012 (5)	0.008 (5)
Ag1	0.0465 (4)	0.0623 (5)	0.0407 (4)	-0.0149 (4)	0.0167 (3)	0.0099 (4)
F1	0.059 (4)	0.037 (3)	0.090 (6)	0.000 (3)	0.007 (4)	0.016 (3)
F2	0.040 (4)	0.064 (5)	0.096 (6)	-0.004 (3)	0.016 (4)	0.024 (4)
F3	0.038 (4)	0.114 (8)	0.091 (6)	0.013 (5)	-0.003 (4)	0.020 (6)
F4	0.205 (14)	0.049 (4)	0.065 (5)	0.025 (6)	0.047 (7)	-0.003 (4)

Geometric parameters (Å, °)

C1—H1A	0.9700	C6—N2	1.436 (14)
C1—H1B	0.9700	C6—N3	1.460 (12)
C1—N1	1.479 (11)	C7—H7A	0.9700
C1—P1	1.815 (9)	C7—H7B	0.9700
C2—H2A	0.9700	C7—C8	1.534 (13)
C2—H2B	0.9700	C7—P1	1.775 (9)
C2—N2	1.461 (11)	C8—H8A	0.9700
C2—P1	1.826 (9)	C8—H8B	0.9700
C3—H3A	0.9700	C8—C9	1.494 (15)
C3—H3B	0.9700	C9—O11	1.233 (12)
C3—N3	1.484 (11)	C9—O12	1.272 (12)
C3—P1	1.818 (9)	N1—Ag1	2.225 (7)
C4—H4A	0.9700	N3—Ag1 ⁱ	2.505 (7)
C4—H4B	0.9700	O11—Ag1 ⁱⁱ	2.594 (9)
C4—N1	1.499 (12)	O12—Ag1 ⁱⁱ	2.298 (8)
C4—N2	1.428 (13)	B1—F1	1.343 (14)
C5—H5A	0.9700	B1—F2	1.399 (14)
C5—H5B	0.9700	B1—F3	1.370 (15)
C5—N1	1.467 (10)	B1—F4	1.328 (16)

C5—N3	1.464 (12)	Ag1—N3 ⁱⁱⁱ	2.505 (7)
C6—H6A	0.9700	Ag1—O11 ^{iv}	2.594 (9)
C6—H6B	0.9700	Ag1—O12 ^{iv}	2.298 (8)
H1A—C1—H1B	108.1	C7—C8—H8B	109.1
N1—C1—H1A	109.5	H8A—C8—H8B	107.8
N1—C1—H1B	109.5	C9—C8—C7	112.6 (8)
N1—C1—P1	110.7 (6)	C9—C8—H8A	109.1
P1—C1—H1A	109.5	C9—C8—H8B	109.1
P1—C1—H1B	109.5	O11—C9—C8	122.7 (9)
H2A—C2—H2B	108.6	O11—C9—O12	122.6 (10)
N2—C2—H2A	110.4	O12—C9—C8	114.7 (8)
N2—C2—H2B	110.4	C1—N1—C4	108.8 (7)
N2—C2—P1	106.7 (6)	C1—N1—Ag1	112.5 (5)
P1—C2—H2A	110.4	C4—N1—Ag1	112.0 (5)
P1—C2—H2B	110.4	C5—N1—C1	110.9 (6)
H3A—C3—H3B	108.5	C5—N1—C4	108.6 (7)
N3—C3—H3A	110.2	C5—N1—Ag1	104.0 (5)
N3—C3—H3B	110.2	C4—N2—C2	113.3 (7)
N3—C3—P1	107.8 (6)	C4—N2—C6	110.2 (8)
P1—C3—H3A	110.2	C6—N2—C2	112.3 (8)
P1—C3—H3B	110.2	C3—N3—Ag1 ⁱ	115.1 (5)
H4A—C4—H4B	107.7	C5—N3—C3	111.6 (7)
N1—C4—H4A	108.9	C5—N3—Ag1 ⁱ	93.4 (5)
N1—C4—H4B	108.9	C6—N3—C3	110.6 (7)
N2—C4—H4A	108.9	C6—N3—C5	109.4 (7)
N2—C4—H4B	108.9	C6—N3—Ag1 ⁱ	115.4 (5)
N2—C4—N1	113.4 (7)	C9—O11—Ag1 ⁱⁱ	85.8 (6)
H5A—C5—H5B	107.6	C9—O12—Ag1 ⁱⁱ	98.7 (6)
N1—C5—H5A	108.7	C1—P1—C2	99.7 (4)
N1—C5—H5B	108.7	C1—P1—C3	101.4 (4)
N3—C5—H5A	108.7	C3—P1—C2	103.1 (4)
N3—C5—H5B	108.7	C7—P1—C1	108.9 (4)
N3—C5—N1	114.2 (7)	C7—P1—C2	126.3 (4)
H6A—C6—H6B	107.6	C7—P1—C3	114.0 (4)
N2—C6—H6A	108.6	F1—B1—F2	108.8 (9)
N2—C6—H6B	108.6	F1—B1—F3	111.6 (11)
N2—C6—N3	114.6 (7)	F3—B1—F2	104.7 (9)
N3—C6—H6A	108.6	F4—B1—F1	110.8 (10)
N3—C6—H6B	108.6	F4—B1—F2	108.4 (11)
H7A—C7—H7B	107.5	F4—B1—F3	112.2 (12)
C8—C7—H7A	108.4	N1—Ag1—N3 ⁱⁱⁱ	119.6 (3)
C8—C7—H7B	108.4	N1—Ag1—O11 ^{iv}	134.1 (3)
C8—C7—P1	115.5 (7)	N1—Ag1—O12 ^{iv}	133.5 (3)
P1—C7—H7A	108.4	N3 ⁱⁱⁱ —Ag1—O11 ^{iv}	92.3 (3)
P1—C7—H7B	108.4	O12 ^{iv} —Ag1—N3 ⁱⁱⁱ	103.6 (3)
C7—C8—H8A	109.1	O12 ^{iv} —Ag1—O11 ^{iv}	52.9 (2)

C7—C8—C9—O11	148.3 (10)	N2—C6—N3—C5	53.5 (10)
C7—C8—C9—O12	-33.2 (12)	N2—C6—N3—Ag1 ⁱ	157.2 (6)
C8—C7—P1—C1	153.3 (7)	N3—C3—P1—C1	51.8 (7)
C8—C7—P1—C2	-88.5 (8)	N3—C3—P1—C2	-51.1 (7)
C8—C7—P1—C3	40.9 (8)	N3—C3—P1—C7	168.7 (6)
C8—C9—O11—Ag1 ⁱⁱ	177.7 (9)	N3—C5—N1—C1	-66.7 (9)
C8—C9—O12—Ag1 ⁱⁱ	-177.7 (7)	N3—C5—N1—C4	52.8 (9)
N1—C1—P1—C2	54.5 (7)	N3—C5—N1—Ag1	172.2 (6)
N1—C1—P1—C3	-51.1 (7)	N3—C6—N2—C2	71.6 (10)
N1—C1—P1—C7	-171.6 (6)	N3—C6—N2—C4	-55.7 (10)
N1—C4—N2—C2	-71.1 (10)	O11—C9—O12—Ag1 ⁱⁱ	0.8 (11)
N1—C4—N2—C6	55.6 (10)	O12—C9—O11—Ag1 ⁱⁱ	-0.7 (10)
N1—C5—N3—C3	70.1 (9)	P1—C1—N1—C4	-60.8 (8)
N1—C5—N3—C6	-52.7 (9)	P1—C1—N1—C5	58.5 (8)
N1—C5—N3—Ag1 ⁱ	-171.2 (6)	P1—C1—N1—Ag1	174.5 (4)
N2—C2—P1—C1	-53.2 (7)	P1—C2—N2—C4	64.6 (9)
N2—C2—P1—C3	51.0 (7)	P1—C2—N2—C6	-61.0 (8)
N2—C2—P1—C7	-175.5 (6)	P1—C3—N3—C5	-62.5 (8)
N2—C4—N1—C1	66.6 (10)	P1—C3—N3—C6	59.5 (8)
N2—C4—N1—C5	-54.2 (9)	P1—C3—N3—Ag1 ⁱ	-167.4 (3)
N2—C4—N1—Ag1	-168.4 (6)	P1—C7—C8—C9	62.9 (10)
N2—C6—N3—C3	-69.8 (10)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A \cdots F2	0.97	2.40	3.201 (13)	140
C1—H1B \cdots O11 ^v	0.97	2.49	3.213 (12)	131
C2—H2A \cdots O11 ^v	0.97	2.56	3.254 (12)	129
C2—H2B \cdots F3 ^{vi}	0.97	2.35	3.183 (13)	143
C2—H2B \cdots O12	0.97	2.22	2.812 (12)	118
C3—H3A \cdots F4 ⁱⁱ	0.97	2.45	3.290 (15)	145
C3—H3B \cdots F2	0.97	2.51	3.283 (12)	136
C4—H4A \cdots F4 ^{vii}	0.97	2.43	3.298 (12)	148
C4—H4B \cdots F2 ^{viii}	0.97	2.51	3.417 (14)	155
C5—H5A \cdots F1	0.97	2.49	3.370 (13)	151
C5—H5B \cdots F4 ^{vii}	0.97	2.54	3.373 (14)	144
C6—H6B \cdots F2 ^{vii}	0.97	2.34	3.314 (13)	177
C7—H7A \cdots O11 ^v	0.97	2.48	3.165 (13)	128
C7—H7A \cdots F1 ^{ix}	0.97	2.37	3.137 (12)	135

Symmetry codes: (ii) $x-1/2, -y-1/2, z+1/2$; (v) $x, -y-1, z-1/2$; (vi) $x-1, y, z$; (vii) $x-1/2, y+1/2, z$; (viii) $x-1/2, -y-1/2, z-1/2$; (ix) $x-1/2, y-1/2, z$.