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Crystal structure of bis(1,3-dimethoxyimidazolin-2-ylidene)silver(I) hexafluoridophosphate, N-heterocyclic carbene (NHC) complex

Barbara Rietzler,[a] Gerhard Laus,[a]* Volker Kahlenberg[b] and Herwig Schottenberger[a]

[a]University of Innsbruck, Faculty of Chemistry and Pharmacy, Innrain 80, 6020 Innsbruck, Austria, and [b]University of Innsbruck, Institute of Mineralogy and Petrography, Innrain 52, 6020 Innsbruck, Austria. *Correspondence e-mail: gerhard.laus@uibk.ac.at

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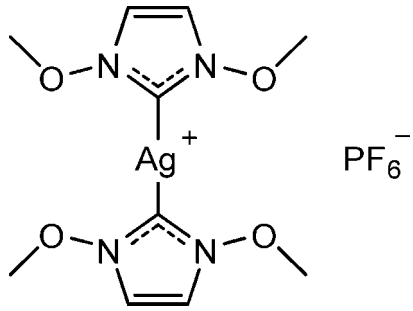
The title salt, $[Ag(C_5H_8N_2O_2)_2]PF_6$, was obtained by deprotonation and metalation of 1,3-dimethoxyimidazolium hexafluoridophosphate using silver(I) oxide in methanol. The C—Ag—C angle in the cation is 178.1 (2)°, and the N—C—N angles are 101.1 (4) and 100.5 (4)°. The methoxy groups adopt an *anti* conformation. In the crystal, anions (*A*) are sandwiched between cations (*C*) in a layered arrangement $\{C\ldots A\ldots C\}_n$ stacked along [001]. Within a $C\ldots A\ldots C$ layer, the hexafluoridophosphate anions accept several C—H···F hydrogen bonds from the cationic complex.

Keywords: crystal structure; silver(I); 1,3-dimethoxyimidazolin-2-ylidene; hexafluoridophosphate salt.

CCDC reference: 1439919

1. Related literature

For synthesis of 1,3-dimethoxyimidazolium hexafluoridophosphate, see: Laus *et al.* (2007). For related structures, see: Laus *et al.* (2008, 2010). For background to *N*-heterocyclic carbene (NHC)–silver complexes, see: Garrison & Youngs (2005); Lin *et al.* (2009); Lin & Vasam (2007); Wang & Lin (1998). For the nature of C—H···F interactions, see: D'Oria & Novoa (2008).



2. Experimental

2.1. Crystal data

$[Ag(C_5H_8N_2O_2)_2]PF_6$
M_r = 509.11
Triclinic, $P\overline{1}$
a = 7.5254 (7) Å
b = 11.7221 (12) Å
c = 11.8697 (12) Å
α = 109.481 (9)°
β = 100.698 (8)°
γ = 100.052 (8)°
V = 937.84 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.24 mm^{-1}
T = 243 K
0.25 × 0.12 × 0.05 mm

2.2. Data collection

Agilent Xcalibur (Ruby, Gemini ultra) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.770, T_{max} = 1
5767 measured reflections
3398 independent reflections
2814 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.109
S = 1.02
3398 reflections
222 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···F1	0.97	2.57	3.193 (8)	122
C8—H8···F2^{i}	0.94	2.46	3.382 (5)	165
C10—H10B···F1^{ii}	0.97	2.54	3.334 (9)	140
C9—H9C···F6^{iii}	0.97	2.58	3.516 (6)	163

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+1, -z+2$; (iii) $-x+1, -y+1, -z+3$.

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: BQ2402).

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supporting information

Acta Cryst. (2015). E**71**, m251–m252 [https://doi.org/10.1107/S2056989015023130]

Crystal structure of bis(1,3-dimethoxyimidazolin-2-ylidene)silver(I) hexafluoridophosphate, N-heterocyclic carbene (NHC) complex

Barbara Rietzler, Gerhard Laus, Volker Kahlenberg and Herwig Schottenberger

S1. Comment

N-Heterocyclic carbene (NHC)–silver complexes are valuable precursors for transmetalation to other metal NHC systems (Garrison & Youngs, 2005; Lin *et al.*, 2009; Lin & Vasam, 2007; Wang & Lin, 1998). In the crystal structure of the title compound, the central carbene–metal bonds C1—Ag and C6—Ag are 2.073 (4) and 2.070 (4) Å long, respectively, and deviate only slightly from linearity with an angle of 178.1 (2)°. The N—C—N 'carbene angles′ are 101.0 (4)° and 100.4 (4)°, significantly smaller than the mean value of 104.5° in bis(NHC)–Ag complexes from the CSD (1002 values from 344 entries), but in line with related N-alkyloxy-substituted compounds (reference codes: DOJNIA and YUWZOG), where the angles range from 100.9° to 102.0° (Laus *et al.*, 2008 and 2010). The dihedral angle between the imidazole rings is 3.0 (3)°. The methoxy groups adopt *anti* conformation. The molecular structure is shown in Figure 1. The unit cell contains two ion pairs (Figure 2). The weakly coordinating hexafluorophosphate ion accepts several C—H···F hydrogen bonds (D′Oria & Novoa, 2008) from the cationic complex (Figure 3). The hydrogen bond geometries are summarised in Table 1.

S2. Experimental

A suspension of 1,3-dimethoxyimidazolium hexafluorophosphate (1.0 g, 3.6 mmol) (Laus *et al.*, 2007) and Ag_2O (0.40 g, 1.7 mmol) in MeOH (20 ml) was stirred at room temperature for 18 h (Figure 4), until the dark Ag_2O was consumed. The desired product was filtered off (the filtrate contained the soluble $AgPF_6$), washed with MeOH and Et_2O and recrystallised from hot MeOH to yield colourless crystals (0.55 g, 62%). The PXRD (Cu *Kα* radiation) of the bulk material was identical to the one calculated from the single-crystal diffraction data (Figure 5).

Melting point: 164–166 °C. ^{1}H NMR (DMSO-d_6, 300 MHz): δ 4.16 (s, 12H), 7.91 (s, 4H) ppm ^{13}C NMR (DMSO-d_6, 75 MHz): δ 68.1 (4C), 116.6 (4C), 165.7 (2C) ppm IR (neat): ν 3172 (w), 3155 (w), 2951 (w), 1460 (w), 1440 (w), 1027 (m), 957 (m), 822 (s), 705 (m), 555 (s) cm^{-1}.

S3. Refinement

Carbon-bound H atoms were placed in calculated positions and refined riding on their respective carbon atom. Methyl hydrogens were fitted to the experimental electron density by allowing them to rotate around the C—C bond with a fixed angle (AFIX 137). Isotropic displacement parameters were constrained with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. The F atoms of the PF_6 ions were restrained with a distance of P—F = 1.57 Å.

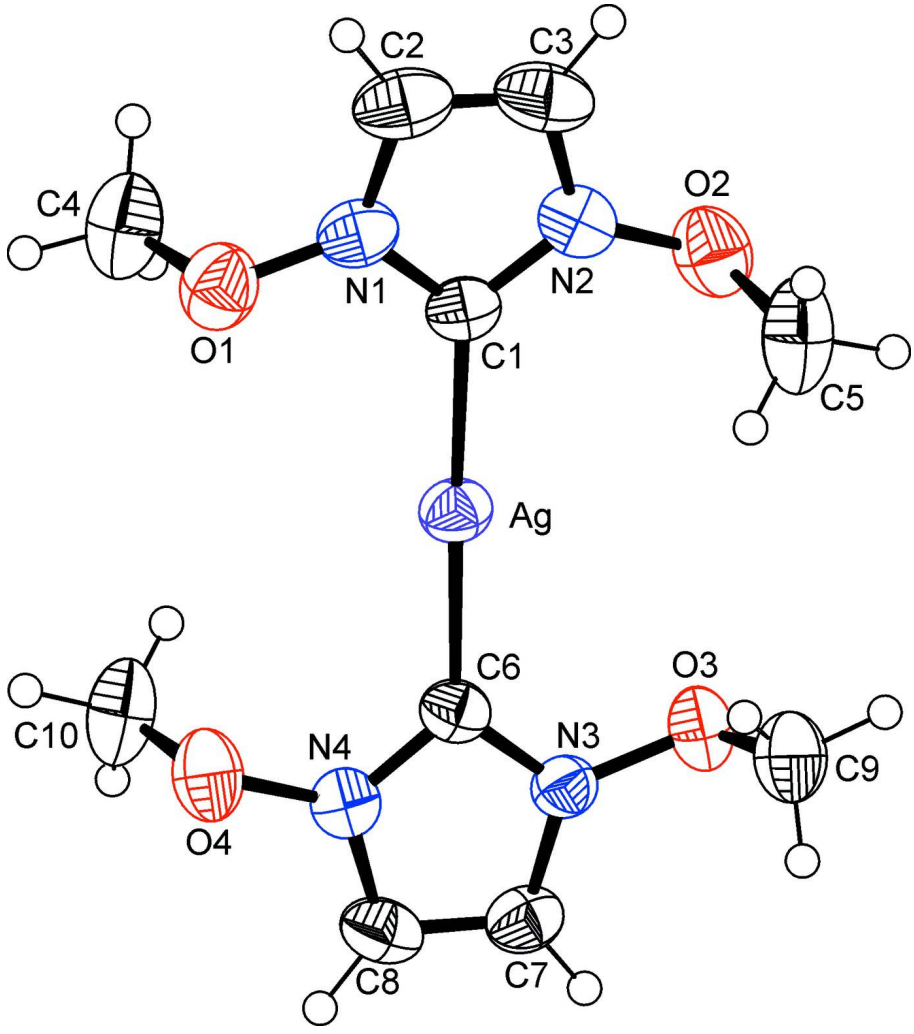

Figure 1

The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms. The hexafluoridophosphate ion is not shown.

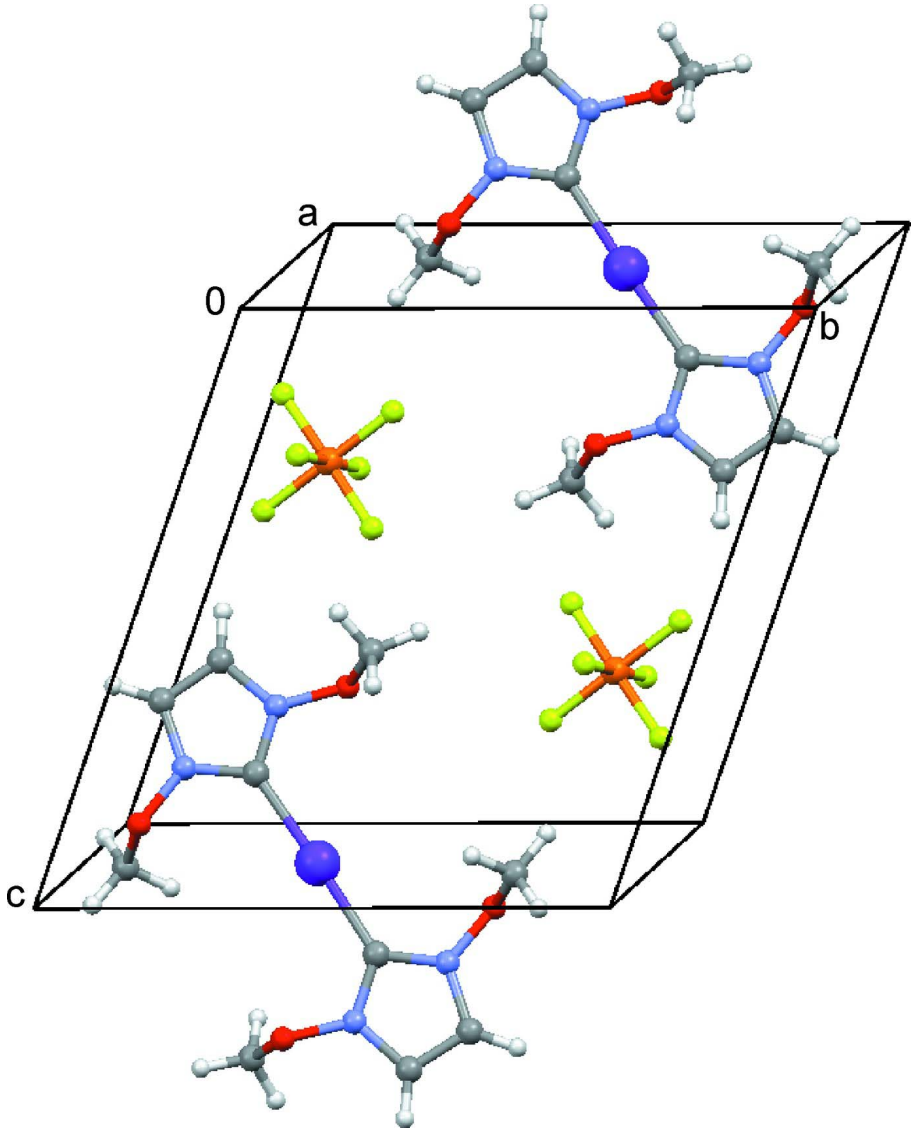

Figure 2
Unit cell of the title compound.

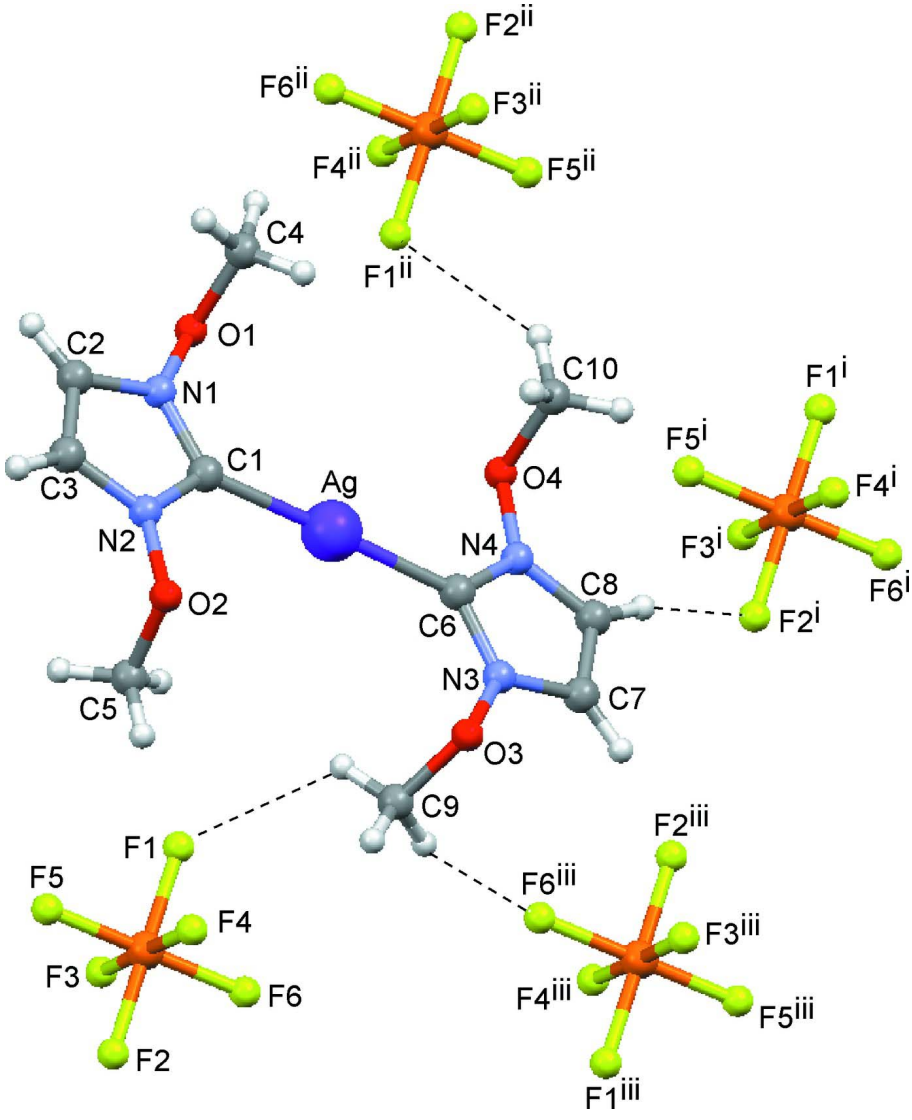

Figure 3

Interionic contacts in the crystal structure of the title compound. Symmetry codes: (i) *x*, 1 + *y*, *z*; (ii) 1 - *x*, 1 - *y*, 2 - *z*; (iii) 1 - *x*, 1 - *y*, 3 - *z*.

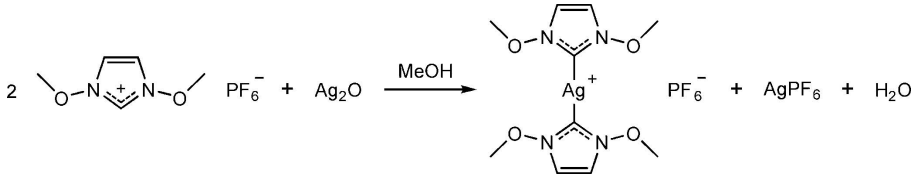

Figure 4

Reaction scheme.

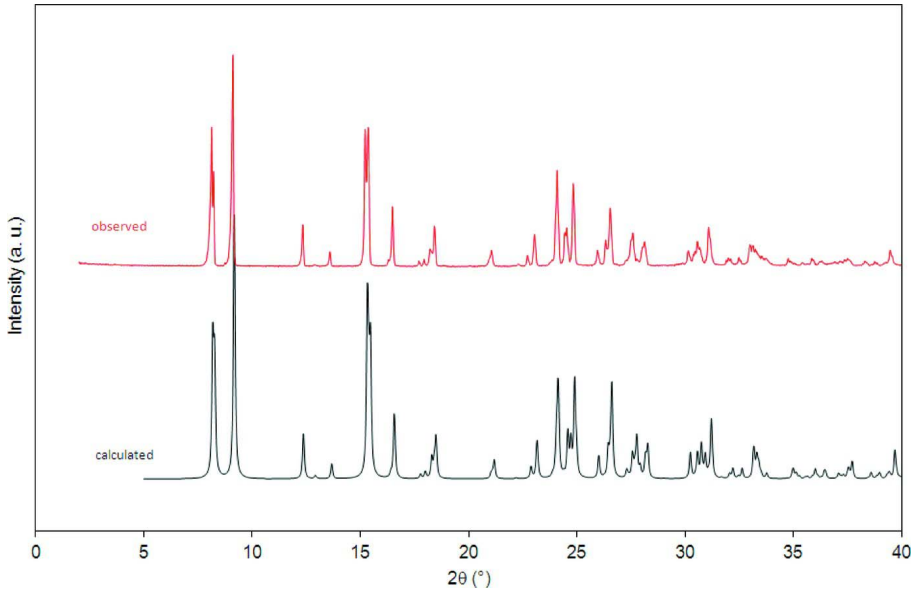

Figure 5

Observed and calculated powder X-ray diffraction data.

Bis(1,3-dimethoxyimidazolin-2-ylidene)silver(I) hexafluoridophosphate

Crystal data

$[Ag(C_5H_8N_2O_2)_2]PF_6$
M_r = 509.11
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.5254 (7) Å
b = 11.7221 (12) Å
c = 11.8697 (12) Å
α = 109.481 (9)°
β = 100.698 (8)°
γ = 100.052 (8)°
V = 937.84 (17) Å^3
Z = 2
$F(000)$ = 504
D_x = 1.803 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2189 reflections
θ = 3.1–28.5°
μ = 1.24 mm^{-1}
T = 243 K
Prismatic fragment, colourless
0.25 × 0.12 × 0.05 mm

Data collection

Agilent Xcalibur (Ruby, Gemini ultra) diffractometer
Graphite monochromator
Detector resolution: 10.3575 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.770, T_{max} = 1
5767 measured reflections
3398 independent reflections
2814 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.4°, θ_{min} = 3.2°
h = −9→7
k = −12→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.109
S = 1.02
3398 reflections
222 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 1.0584P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.75$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0166 (15)

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.36.20 (release 27-06-2012 CrysAlis171 .NET) (compiled Jul 11 2012,15:38:31) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.23186 (5)	0.43947 (3)	0.95741 (3)	0.04281 (18)
P	0.5262 (2)	0.18543 (11)	1.32827 (11)	0.0552 (4)
F1	0.5321 (7)	0.2674 (3)	1.2468 (3)	0.0968 (10)
F6	0.5226 (8)	0.3018 (3)	1.4400 (3)	0.1157 (13)
F3	0.7426 (5)	0.2188 (6)	1.3712 (6)	0.1502 (16)
F2	0.5166 (7)	0.1034 (3)	1.4097 (3)	0.0968 (10)
F5	0.5222 (8)	0.0672 (3)	1.2160 (3)	0.1157 (13)
F4	0.3055 (5)	0.1529 (6)	1.2871 (6)	0.1502 (16)
O4	0.3885 (5)	0.7554 (3)	1.0537 (4)	0.0560 (9)
O3	0.1139 (5)	0.4969 (3)	1.2341 (3)	0.0528 (9)
O2	0.0715 (5)	0.1240 (3)	0.8695 (4)	0.0611 (10)
C1	0.2102 (6)	0.2811 (4)	0.8048 (4)	0.0403 (10)
O1	0.3470 (5)	0.3732 (4)	0.6798 (3)	0.0605 (10)
N3	0.2052 (5)	0.6009 (3)	1.2185 (3)	0.0389 (8)
N2	0.1483 (6)	0.1604 (4)	0.7865 (4)	0.0471 (10)
N4	0.3151 (5)	0.7149 (3)	1.1359 (3)	0.0395 (9)
N1	0.2605 (6)	0.2710 (4)	0.7000 (4)	0.0463 (9)
C6	0.2497 (6)	0.5939 (4)	1.1125 (4)	0.0377 (10)
C10	0.2488 (9)	0.7857 (6)	0.9781 (6)	0.0709 (17)
H10A	0.1441	0.7126	0.9337	0.106*
H10B	0.301	0.812	0.9192	0.106*
H10C	0.2062	0.853	1.0298	0.106*
C7	0.2416 (7)	0.7202 (4)	1.3035 (4)	0.0450 (11)
H7	0.2208	0.7447	1.3828	0.054*
C8	0.3132 (7)	0.7948 (4)	1.2498 (5)	0.0488 (12)
H8	0.3533	0.8827	1.2831	0.059*
C5	0.2106 (9)	0.1109 (7)	0.9606 (7)	0.0790 (19)

H5A	0.2746	0.0506	0.9201	0.118*
H5B	0.1516	0.0821	1.0157	0.118*
H5C	0.3001	0.1912	1.008	0.118*
C4	0.2153 (9)	0.4083 (6)	0.6024 (6)	0.0729 (17)
H4A	0.1472	0.3355	0.5284	0.109*
H4B	0.2814	0.472	0.579	0.109*
H4C	0.1282	0.441	0.6471	0.109*
C3	0.1572 (9)	0.0797 (5)	0.6757 (5)	0.0642 (15)
H3	0.1188	−0.0081	0.645	0.077*
C9	0.2456 (9)	0.4396 (5)	1.2843 (6)	0.0704 (17)
H9A	0.3259	0.4172	1.2297	0.106*
H9B	0.1786	0.3649	1.2917	0.106*
H9C	0.3212	0.4981	1.3654	0.106*
C2	0.2309 (8)	0.1495 (5)	0.6192 (5)	0.0630 (15)
H2	0.2569	0.122	0.5415	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag	0.0481 (3)	0.0401 (2)	0.0376 (2)	0.01478 (16)	0.01134 (16)	0.00972 (16)
P	0.0909 (12)	0.0415 (7)	0.0372 (7)	0.0268 (7)	0.0195 (7)	0.0134 (6)
F1	0.174 (3)	0.0702 (16)	0.0679 (16)	0.0446 (18)	0.0437 (18)	0.0397 (14)
F6	0.229 (4)	0.0686 (16)	0.0589 (16)	0.061 (2)	0.055 (2)	0.0154 (13)
F3	0.090 (3)	0.167 (4)	0.202 (5)	0.026 (2)	0.026 (3)	0.090 (4)
F2	0.174 (3)	0.0702 (16)	0.0679 (16)	0.0446 (18)	0.0437 (18)	0.0397 (14)
F5	0.229 (4)	0.0686 (16)	0.0589 (16)	0.061 (2)	0.055 (2)	0.0154 (13)
F4	0.090 (3)	0.167 (4)	0.202 (5)	0.026 (2)	0.026 (3)	0.090 (4)
O4	0.049 (2)	0.062 (2)	0.073 (2)	0.0183 (17)	0.0291 (19)	0.037 (2)
O3	0.054 (2)	0.0500 (19)	0.063 (2)	0.0109 (16)	0.0186 (18)	0.0318 (17)
O2	0.045 (2)	0.063 (2)	0.079 (3)	0.0088 (17)	0.019 (2)	0.033 (2)
C1	0.035 (2)	0.044 (3)	0.038 (3)	0.013 (2)	0.007 (2)	0.011 (2)
O1	0.052 (2)	0.072 (2)	0.062 (2)	0.0127 (19)	0.0166 (19)	0.032 (2)
N3	0.042 (2)	0.0373 (19)	0.035 (2)	0.0112 (16)	0.0067 (17)	0.0127 (16)
N2	0.041 (2)	0.045 (2)	0.053 (2)	0.0128 (18)	0.0122 (19)	0.0145 (19)
N4	0.032 (2)	0.044 (2)	0.045 (2)	0.0132 (17)	0.0110 (17)	0.0175 (18)
N1	0.044 (2)	0.051 (2)	0.041 (2)	0.0170 (19)	0.0116 (19)	0.0103 (18)
C6	0.030 (2)	0.038 (2)	0.041 (3)	0.0091 (18)	0.0049 (19)	0.0122 (19)
C10	0.073 (4)	0.092 (4)	0.079 (4)	0.030 (4)	0.035 (3)	0.059 (4)
C7	0.048 (3)	0.046 (3)	0.039 (3)	0.021 (2)	0.010 (2)	0.011 (2)
C8	0.046 (3)	0.037 (2)	0.053 (3)	0.011 (2)	0.004 (2)	0.008 (2)
C5	0.071 (4)	0.099 (5)	0.104 (5)	0.029 (4)	0.039 (4)	0.072 (4)
C4	0.072 (4)	0.091 (4)	0.082 (4)	0.030 (4)	0.028 (4)	0.055 (4)
C3	0.068 (4)	0.044 (3)	0.063 (4)	0.018 (3)	0.012 (3)	−0.001 (3)
C9	0.092 (5)	0.057 (3)	0.071 (4)	0.022 (3)	0.012 (3)	0.038 (3)
C2	0.062 (4)	0.067 (4)	0.047 (3)	0.025 (3)	0.012 (3)	0.003 (3)

Geometric parameters (Å, º)

Ag—C6	2.070 (4)	N4—C6	1.332 (6)
Ag—C1	2.073 (4)	N4—C8	1.368 (6)
P—F3	1.550 (4)	N1—C2	1.377 (6)
P—F5	1.561 (3)	C10—H10A	0.97
P—F6	1.562 (3)	C10—H10B	0.97
P—F1	1.574 (3)	C10—H10C	0.97
P—F2	1.576 (3)	C7—C8	1.340 (7)
P—F4	1.580 (4)	C7—H7	0.94
O4—N4	1.379 (5)	C8—H8	0.94
O4—C10	1.424 (7)	C5—H5A	0.97
O3—N3	1.378 (5)	C5—H5B	0.97
O3—C9	1.438 (6)	C5—H5C	0.97
O2—N2	1.376 (5)	C4—H4A	0.97
O2—C5	1.426 (7)	C4—H4B	0.97
C1—N2	1.339 (6)	C4—H4C	0.97
C1—N1	1.341 (6)	C3—C2	1.331 (8)
O1—N1	1.375 (5)	C3—H3	0.94
O1—C4	1.433 (7)	C9—H9A	0.97
N3—C6	1.342 (6)	C9—H9B	0.97
N3—C7	1.368 (6)	C9—H9C	0.97
N2—C3	1.362 (7)	C2—H2	0.94
C6—Ag—C1	178.13 (17)	N3—C6—Ag	130.3 (3)
F3—P—F5	91.4 (3)	O4—C10—H10A	109.5
F3—P—F6	90.6 (4)	O4—C10—H10B	109.5
F5—P—F6	177.9 (3)	H10A—C10—H10B	109.5
F3—P—F1	91.7 (3)	O4—C10—H10C	109.5
F5—P—F1	91.0 (2)	H10A—C10—H10C	109.5
F6—P—F1	89.5 (2)	H10B—C10—H10C	109.5
F3—P—F2	89.3 (3)	C8—C7—N3	105.5 (4)
F5—P—F2	89.2 (2)	C8—C7—H7	127.2
F6—P—F2	90.2 (2)	N3—C7—H7	127.2
F1—P—F2	179.0 (3)	C7—C8—N4	104.8 (4)
F3—P—F4	178.9 (4)	C7—C8—H8	127.6
F5—P—F4	89.6 (3)	N4—C8—H8	127.6
F6—P—F4	88.4 (3)	O2—C5—H5A	109.5
F1—P—F4	88.8 (3)	O2—C5—H5B	109.5
F2—P—F4	90.3 (3)	H5A—C5—H5B	109.5
N4—O4—C10	110.1 (4)	O2—C5—H5C	109.5
N3—O3—C9	110.8 (4)	H5A—C5—H5C	109.5
N2—O2—C5	111.5 (4)	H5B—C5—H5C	109.5
N2—C1—N1	101.0 (4)	O1—C4—H4A	109.5
N2—C1—Ag	129.2 (3)	O1—C4—H4B	109.5
N1—C1—Ag	129.8 (3)	H4A—C4—H4B	109.5
N1—O1—C4	110.7 (4)	O1—C4—H4C	109.5
C6—N3—C7	114.2 (4)	H4A—C4—H4C	109.5

C6—N3—O3	122.1 (4)	H4B—C4—H4C	109.5
C7—N3—O3	123.4 (4)	C2—C3—N2	106.6 (5)
C1—N2—C3	113.7 (4)	C2—C3—H3	126.7
C1—N2—O2	122.1 (4)	N2—C3—H3	126.7
C3—N2—O2	124.1 (4)	O3—C9—H9A	109.5
C6—N4—C8	115.0 (4)	O3—C9—H9B	109.5
C6—N4—O4	122.1 (4)	H9A—C9—H9B	109.5
C8—N4—O4	122.8 (4)	O3—C9—H9C	109.5
C1—N1—O1	122.5 (4)	H9A—C9—H9C	109.5
C1—N1—C2	114.1 (5)	H9B—C9—H9C	109.5
O1—N1—C2	123.2 (4)	C3—C2—N1	104.6 (5)
N4—C6—N3	100.5 (4)	C3—C2—H2	127.7
N4—C6—Ag	129.2 (3)	N1—C2—H2	127.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*A*···F1	0.97	2.57	3.193 (8)	122
C8—H8···F2^{i}	0.94	2.46	3.382 (5)	165
C10—H10*B*···F1^{ii}	0.97	2.54	3.334 (9)	140
C9—H9*C*···F6^{iii}	0.97	2.58	3.516 (6)	163

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