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Crystal structure of dichlorido[2-(diphenylphosphanyl)-3,4,5,6-tetrafluorobenzene-1-thiolato-κ^2P,S]gold(III)

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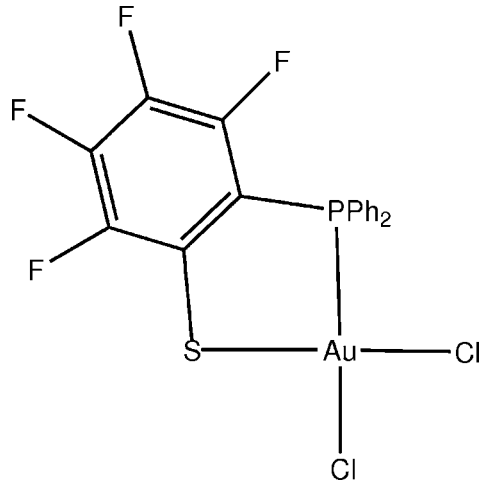
The title compound, $[Au(C_{18}H_{10}F_4PS)Cl_2]$, crystallizes as neutral molecules, with the Au^{III} atom coordinated by two Cl atoms and by the P and S atoms of the bidentate phosphanyl thiolate ligand, in a slightly distorted square-planar environment. The molecules are linked into centrosymmetric dimers *via* long axial Au—Cl bonds of 3.393 (4) Å. This axial Au—Cl distance is longer than is usually seen, although one other example has been given. Dimer formation may explain the unexpectedly low solubility of the compound in common polar solvents. There is also a separate intermolecular Au—F contact of 3.561 (6) Å, but this distance seems too long to be regarded as a bond. Two putative C—H···F hydrogen bonds appear to link the dimers into sheets parallel to (110). There is a short intermolecular F···F contact of 2.695 (10) Å between two dimers related by the twofold axis.

Keywords: crystal structure; mixed ligand; gold complex.

CCDC reference: 1422931

1. Related literature

For synthetic details, see: Eller (1971); Eller & Meek (1970). Hollis & Lippard (1983) describe a similarly long axial Au—Cl bond in a mixed-valence gold compound, although other axial Au—Cl bonds in the literature are in the 3.0–3.1 Å range, as in Elder & Watkins (1986).



2. Experimental

2.1. Crystal data

$[Au(C_{18}H_{10}F_4PS)Cl_2]$
M_r = 633.16
Monoclinic, $C2/c$
a = 18.90 (2) Å
b = 8.388 (12) Å
c = 24.15 (3) Å
β = 100.75 (3)°
V = 3761 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 8.15 mm^{-1}
T = 298 K
0.22 × 0.16 × 0.13 mm

2.2. Data collection

Picker 4-circle diffractometer
Absorption correction: gaussian (Busing & Levy, 1957)
T_{min} = 0.384, T_{max} = 0.442
4262 measured reflections
4142 independent reflections
3209 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
18 standard reflections every 500 reflections
intensity decay: −1.0(3)

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.03
4142 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Au—S	2.273 (3)	Au—Cl2	2.337 (3)
Au—P	2.258 (3)	Au—Cl1	2.305 (3)
S—Au—P	90.22 (10)	Cl2—Au—Cl1	93.88 (11)
S—Au—Cl2	87.51 (10)	S—Au—Cl2^i	88.12 (7)
P—Au—Cl2	177.69 (6)	P—Au—Cl2^i	90.45 (9)
S—Au—Cl1	176.59 (7)	Cl2—Au—Cl2^i	89.91 (9)
P—Au—Cl1	88.36 (10)	Cl1—Au—Cl2^i	94.99 (7)

Symmetry code: (i) $-x, -y, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···F3^{ii}	0.93	2.60	3.444 (7)	151
C18—H18···F4^{iii}	0.93	2.50	3.082 (7)	121

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

Data collection: Corfield (1972); cell refinement: Corfield (1972); data reduction: Corfield *et al.* (1973); program(s) used to solve structure: Corfield (1972); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL2014*.

Acknowledgements

We are grateful for the provision of a crystalline sample by Gary P. Eller and Devon W. Meek, as well as support from the National Science Foundation through equipment grant GP8534 awarded to the Ohio State University where the experimental work was carried out.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5209).

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supporting information

Acta Cryst. (2015). E**71**, m181–m182 [doi:10.1107/S2056989015016758]

Crystal structure of dichlorido[2-(diphenylphosphanyl)-3,4,5,6-tetrafluorobenzene-1-thiolato-κ^2P,S]gold(III)

Peter W. R. Corfield and Mary Bailey

S1. Synthesis and crystallization

The preparation of the compound is described by Eller (1971), and synthesis of the then novel ligand is given in Eller & Meek (1970).

S2. Refinement

To reduce the number of parameters varied, the phenyl groups C16—C21 and C22—C27 were constrained as rigid hexagons, with C—C distances of 1.385 Å. Aromatic H atoms were placed geometrically, with their U_{eq} values set 1.2 times the U_{iso} of their bonded C atoms.

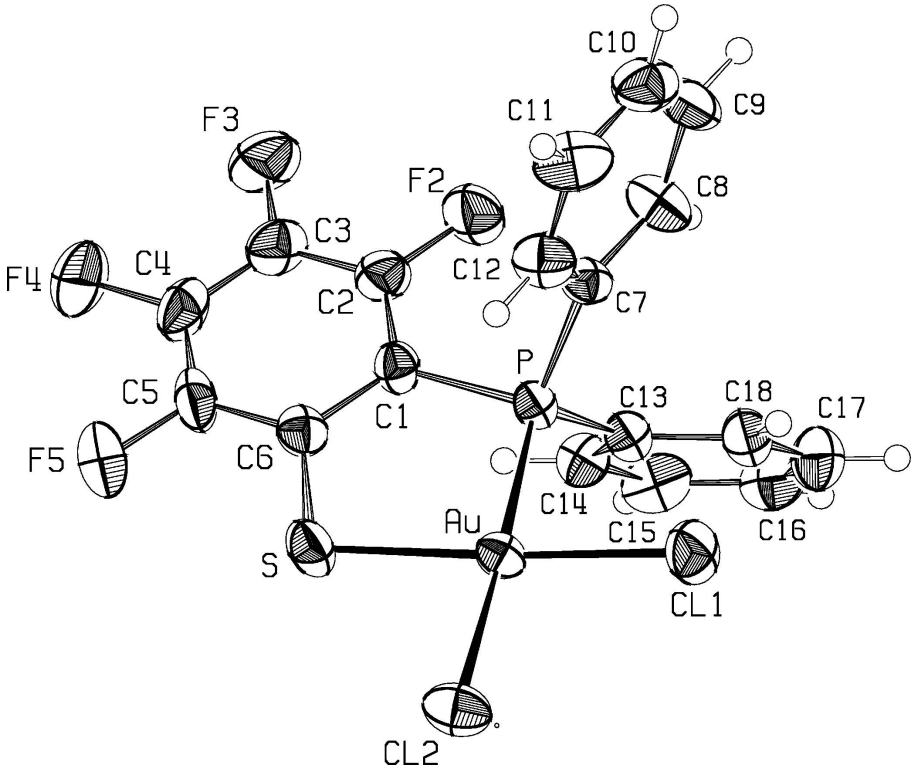

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

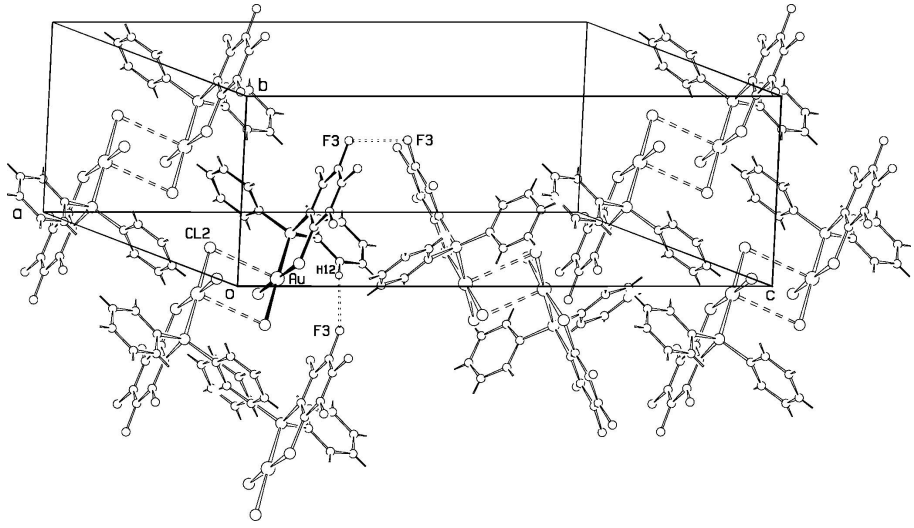

Figure 2

Packing of the title complex, viewed along a direction near to the *a* axis. The centrosymmetric dimers are shown, as well as the proximity of F3(1,*y* - 1,*z*) to a sixth coordination site for the gold atom. Long Au—Cl bonds are gived as dashed lines.

Dichlorido[2-(diphenylphosphanyl)-3,4,5,6-tetrafluorobenzene-1-thiolato-κ^2P,S]gold(III)

Crystal data

$[Au(C_{18}H_{10}F_4PS)Cl_2]$
M_r = 633.16
Monoclinic, *C*2/*c*
a = 18.90 (2) Å
b = 8.388 (12) Å
c = 24.15 (3) Å
β = 100.75 (3)°
V = 3761 (8) Å^3
Z = 8
F(000) = 2384
D_x = 2.236 Mg m^{-3}
D_m = 2.181 (3) Mg m^{-3}
D_m measured by flotation in carbon tetrachloride/bromoforom mixture. Discrepancy may be due to an uncalibrated pycnometer.
Mo *K*α radiation, λ = 0.7107 Å
Cell parameters from 24 reflections
θ = 4.2–25.1°
μ = 8.15 mm^{-1}
T = 298 K
Irregular, red
0.22 × 0.16 × 0.13 mm

Data collection

Picker 4-circle diffractometer
Radiation source: sealed X-ray tube
Oriented graphite 200 reflection monochromator
$\theta/2\theta$ scans
Absorption correction: gaussian (Busing & Levy, 1957)
T_{min} = 0.384, T_{max} = 0.442
4262 measured reflections
4142 independent reflections
3209 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°, θ_{min} = 2.5°
h = 0→24
k = 0→9
l = −31→30
18 standard reflections every 500 reflections
intensity decay: −1.0 (3)

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.086$
$S = 1.02$
4142 reflections
220 parameters
0 restraints
Primary atom site location: heavy-atom method
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.00$ e Å^{-3}
$\Delta\rho_{min} = -0.87$ e Å^{-3}

Special details

Experimental. Data reduction followed procedures in Corfield *et al.* (1973), with programs written by Corfield and by Graeme Gainsford.

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. To reduce the number of parameters varied, the phenyl groups C16—C21 and C22—C27 were constrained as rigid hexagons, with C—C distances of 1.385 Å. U_{eq} values for the aromatic H atoms were set 1.2 times the U_{iso} of their bonded C atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Au	0.03240 (2)	0.02779 (3)	0.08580 (2)	0.03238 (9)
Cl1	0.14260 (10)	−0.0955 (2)	0.09200 (9)	0.0544 (5)
Cl2	−0.03221 (10)	−0.1909 (2)	0.04280 (8)	0.0503 (4)
S	−0.07524 (9)	0.1492 (2)	0.08537 (8)	0.0438 (4)
P	0.09063 (8)	0.2433 (2)	0.12802 (7)	0.0309 (3)
F2	0.1031 (2)	0.5694 (5)	0.1812 (2)	0.0572 (12)
F3	−0.0040 (3)	0.7682 (6)	0.1937 (2)	0.0681 (13)
F4	−0.1413 (3)	0.6750 (6)	0.1616 (2)	0.0757 (15)
F5	−0.1734 (2)	0.3959 (6)	0.1090 (2)	0.0622 (12)
C1	0.0205 (3)	0.3780 (8)	0.1371 (3)	0.0343 (14)
C2	0.0351 (4)	0.5243 (9)	0.1633 (3)	0.0431 (16)
C3	−0.0190 (4)	0.6241 (9)	0.1708 (3)	0.0479 (18)
C4	−0.0886 (4)	0.5795 (9)	0.1527 (3)	0.0488 (18)
C5	−0.1042 (3)	0.4341 (10)	0.1269 (3)	0.0461 (18)
C6	−0.0507 (3)	0.3312 (8)	0.1186 (3)	0.0363 (14)
C7	0.1393 (2)	0.1964 (5)	0.19702 (13)	0.0336 (14)
C8	0.2018 (2)	0.2774 (5)	0.21989 (18)	0.0490 (18)
H8	0.2215	0.3507	0.1981	0.059*
C9	0.2350 (2)	0.2494 (6)	0.27517 (19)	0.058 (2)
H9	0.2769	0.3038	0.2905	0.070*
C10	0.2057 (3)	0.1403 (6)	0.30757 (14)	0.055 (2)
H10	0.2279	0.1215	0.3447	0.066*
C11	0.1432 (3)	0.0592 (6)	0.28470 (17)	0.060 (2)
H11	0.1235	−0.0140	0.3065	0.072*

C12	0.1100 (2)	0.0873 (5)	0.22942 (18)	0.0462 (17)
H12	0.0681	0.0329	0.2141	0.055*
C13	0.1473 (2)	0.3410 (5)	0.08744 (17)	0.0352 (14)
C14	0.12320 (19)	0.4760 (5)	0.05659 (18)	0.0400 (15)
H14	0.0771	0.5146	0.0566	0.048*
C15	0.1678 (3)	0.5534 (4)	0.02581 (18)	0.0487 (18)
H15	0.1516	0.6441	0.0051	0.058*
C16	0.2365 (2)	0.4959 (6)	0.0259 (2)	0.051 (2)
H16	0.2664	0.5480	0.0052	0.062*
C17	0.26061 (18)	0.3610 (6)	0.0567 (2)	0.0527 (19)
H17	0.3068	0.3224	0.0568	0.063*
C18	0.2160 (2)	0.2835 (5)	0.08750 (19)	0.0433 (16)
H18	0.2323	0.1929	0.1082	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au	0.03286 (13)	0.03025 (15)	0.03249 (12)	−0.00327 (11)	0.00210 (8)	−0.00023 (11)
Cl1	0.0446 (10)	0.0441 (11)	0.0717 (13)	0.0115 (8)	0.0038 (9)	−0.0094 (9)
Cl2	0.0572 (11)	0.0408 (10)	0.0499 (10)	−0.0166 (8)	0.0022 (8)	−0.0058 (8)
S	0.0279 (8)	0.0472 (11)	0.0541 (10)	−0.0065 (7)	0.0017 (7)	−0.0028 (8)
P	0.0258 (7)	0.0295 (9)	0.0361 (8)	−0.0005 (6)	0.0022 (6)	0.0001 (6)
F2	0.048 (2)	0.046 (3)	0.074 (3)	−0.006 (2)	0.000 (2)	−0.019 (2)
F3	0.082 (3)	0.051 (3)	0.071 (3)	0.010 (3)	0.012 (3)	−0.023 (2)
F4	0.066 (3)	0.073 (4)	0.088 (4)	0.037 (3)	0.015 (3)	−0.013 (3)
F5	0.034 (2)	0.072 (3)	0.080 (3)	0.009 (2)	0.008 (2)	0.005 (3)
C1	0.030 (3)	0.031 (4)	0.041 (3)	0.004 (3)	0.005 (3)	0.001 (3)
C2	0.047 (4)	0.038 (4)	0.043 (4)	0.005 (3)	0.004 (3)	−0.008 (3)
C3	0.060 (5)	0.034 (4)	0.049 (4)	0.013 (3)	0.007 (4)	−0.003 (3)
C4	0.051 (4)	0.045 (5)	0.052 (4)	0.021 (4)	0.015 (3)	0.002 (3)
C5	0.025 (3)	0.065 (5)	0.048 (4)	0.010 (3)	0.005 (3)	0.013 (4)
C6	0.036 (3)	0.041 (4)	0.032 (3)	0.003 (3)	0.005 (3)	0.007 (3)
C7	0.037 (3)	0.031 (4)	0.031 (3)	0.004 (3)	0.001 (3)	0.001 (3)
C8	0.047 (4)	0.048 (5)	0.046 (4)	−0.011 (3)	−0.006 (3)	0.006 (3)
C9	0.050 (4)	0.065 (6)	0.051 (4)	−0.001 (4)	−0.012 (4)	−0.011 (4)
C10	0.068 (5)	0.054 (5)	0.040 (4)	0.016 (4)	0.001 (4)	−0.002 (4)
C11	0.089 (6)	0.053 (5)	0.039 (4)	−0.003 (5)	0.014 (4)	0.012 (3)
C12	0.054 (4)	0.040 (4)	0.044 (4)	−0.008 (3)	0.008 (3)	0.000 (3)
C13	0.031 (3)	0.036 (4)	0.038 (3)	0.000 (3)	0.003 (3)	0.000 (3)
C14	0.042 (4)	0.039 (4)	0.040 (3)	−0.001 (3)	0.009 (3)	0.002 (3)
C15	0.070 (5)	0.031 (4)	0.047 (4)	−0.007 (4)	0.017 (4)	0.004 (3)
C16	0.059 (5)	0.051 (5)	0.047 (4)	−0.017 (4)	0.017 (4)	−0.003 (3)
C17	0.041 (4)	0.062 (5)	0.057 (5)	−0.006 (4)	0.016 (3)	−0.002 (4)
C18	0.034 (3)	0.045 (4)	0.051 (4)	0.001 (3)	0.006 (3)	−0.001 (3)

Geometric parameters (Å, º)

Au—S	2.273 (3)	C7—C12	1.3850
Au—P	2.258 (3)	C8—C9	1.3850
Au—Cl2	2.337 (3)	C8—H8	0.9300
Au—Cl1	2.305 (3)	C9—C10	1.3850
Au—Cl2^{i}	3.393 (4)	C9—H9	0.9300
Au—F3^{ii}	3.560 (6)	C10—C11	1.3850
S—C6	1.747 (7)	C10—H10	0.9300
P—C1	1.787 (6)	C11—C12	1.3850
P—C7	1.791 (4)	C11—H11	0.9300
P—C13	1.781 (4)	C12—H12	0.9300
F2—C2	1.331 (8)	C13—C14	1.3850
F3—C3	1.337 (9)	C13—C18	1.3850
F4—C4	1.328 (8)	C14—C15	1.3850
F5—C5	1.338 (8)	C14—H14	0.9300
C1—C6	1.393 (8)	C15—C16	1.3850
C1—C2	1.385 (10)	C15—H15	0.9300
C2—C3	1.360 (10)	C16—C17	1.3850
C3—C4	1.358 (10)	C16—H16	0.9300
C4—C5	1.375 (11)	C17—C18	1.3850
C5—C6	1.372 (9)	C17—H17	0.9300
C7—C8	1.3850	C18—H18	0.9300
S—Au—P	90.22 (10)	C5—C6—S	118.5 (5)
S—Au—Cl2	87.51 (10)	C1—C6—S	123.4 (5)
P—Au—Cl2	177.69 (6)	C8—C7—C12	120.0
S—Au—Cl1	176.59 (7)	C8—C7—P	121.0 (3)
P—Au—Cl1	88.36 (10)	C12—C7—P	118.7 (3)
Cl2—Au—Cl1	93.88 (11)	C7—C8—C9	120.0
S—Au—Cl2^{i}	88.12 (7)	C7—C8—H8	120.0
P—Au—Cl2^{i}	90.45 (9)	C9—C8—H8	120.0
Cl2—Au—Cl2^{i}	89.91 (9)	C10—C9—C8	120.0
Cl1—Au—Cl2^{i}	94.99 (7)	C10—C9—H9	120.0
S—Au—F3^{ii}	88.98 (11)	C8—C9—H9	120.0
P—Au—F3^{ii}	107.67 (12)	C11—C10—C9	120.0
Cl2—Au—F3^{ii}	71.87 (12)	C11—C10—H10	120.0
Cl1—Au—F3^{ii}	88.50 (11)	C9—C10—H10	120.0
Cl2^{i}—Au—F3^{ii}	161.66 (8)	C10—C11—C12	120.0
C6—S—Au	103.2 (2)	C10—C11—H11	120.0
C1—P—C7	106.8 (3)	C12—C11—H11	120.0
C1—P—C13	108.2 (3)	C11—C12—C7	120.0
C7—P—C13	110.8 (2)	C11—C12—H12	120.0
C1—P—Au	104.6 (2)	C7—C12—H12	120.0
C7—P—Au	111.53 (17)	C14—C13—C18	120.0
C13—P—Au	114.36 (18)	C14—C13—P	120.0 (2)
C6—C1—C2	119.7 (6)	C18—C13—P	120.0 (2)
C6—C1—P	118.5 (5)	C15—C14—C13	120.0

C2—C1—P	121.8 (5)	C15—C14—H14	120.0
F2—C2—C3	119.1 (6)	C13—C14—H14	120.0
F2—C2—C1	119.9 (6)	C16—C15—C14	120.0
C3—C2—C1	121.0 (7)	C16—C15—H15	120.0
C4—C3—C2	119.7 (7)	C14—C15—H15	120.0
C4—C3—F3	120.0 (7)	C17—C16—C15	120.0
C2—C3—F3	120.3 (7)	C17—C16—H16	120.0
F4—C4—C3	119.5 (7)	C15—C16—H16	120.0
F4—C4—C5	120.2 (7)	C16—C17—C18	120.0
C3—C4—C5	120.2 (6)	C16—C17—H17	120.0
C6—C5—F5	120.3 (7)	C18—C17—H17	120.0
C6—C5—C4	121.4 (6)	C17—C18—C13	120.0
F5—C5—C4	118.3 (6)	C17—C18—H18	120.0
C5—C6—C1	118.0 (6)	C13—C18—H18	120.0

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···F3^{ii}	0.93	2.60	3.444 (7)	151
C18—H18···F4^{iii}	0.93	2.50	3.082 (7)	121

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