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Tetrakis(2,3,5,6-tetrafluorobenzenethiolato-κS)-(triphenylphosphane-κP)osmium(IV): a monoclinic polymorph

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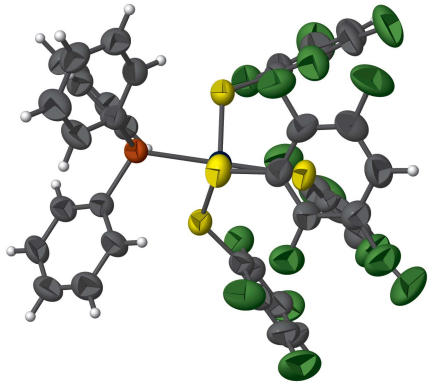
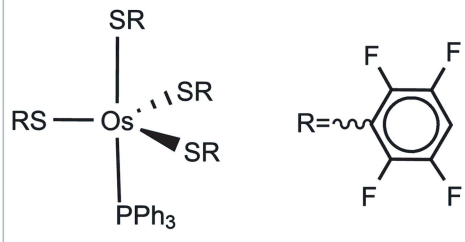
Structural data: full structural data are available from iucrdata.iucr.org

The structure of the title compound, $[Os(C_6HF_4S)_4\{P(C_6H_5)_3\}]$, has been previously reported [Arroyo *et al.* (1994). *J. Chem. Soc. Dalton Trans.* pp. 1819–1824], in the space group $P\overline{1}$. We have now obtained a monoclinic polymorph for this compound, crystallized from ethanol, while the previous form was obtained from a hexane/chloroform mixture. The molecular structure is based on a trigonal–bipyramidal Os^{IV} coordination geometry, close to that observed previously in the triclinic form.

3D view



Chemical scheme



Structure description

Within the chemistry of Os^{IV} complexes bearing polyfluorinated thiolate ligands with general formula $[Os(thiolate)_4(phosphine)]$ (Arroyo *et al.*, 1994, 2009; Mendoza *et al.*, 2006; Cerón *et al.*, 2006), we are currently interested in substitution reactions, allowing complexes to be prepared in which the thiolate ligand in an axial position is replaced by a halogen or a pseudo-halogen, such as an azide. During the recrystallization of a batch of the starting material $[Os(SC_6F_4H)_4(PPh_3)]$ from an ethanolic solution, brown crystals were obtained in space group $P2_1/c$, while this compound was previously reported in space group $P\overline{1}$ (Arroyo *et al.*, 1994). Simulated powder-diffraction patterns for both forms are clearly different, confirming that triclinic and monoclinic crystals are polymorphs. We assume that the polymorphism is triggered by the solvent used for crystallization, ethanol for the monoclinic form, and a hexane:chloroform mixture for the triclinic form. Moreover, a third form for the same compound has been reported, in which the complex crystallized as a *n*-hexane hemisolvate (Cerón *et al.*, 2006).

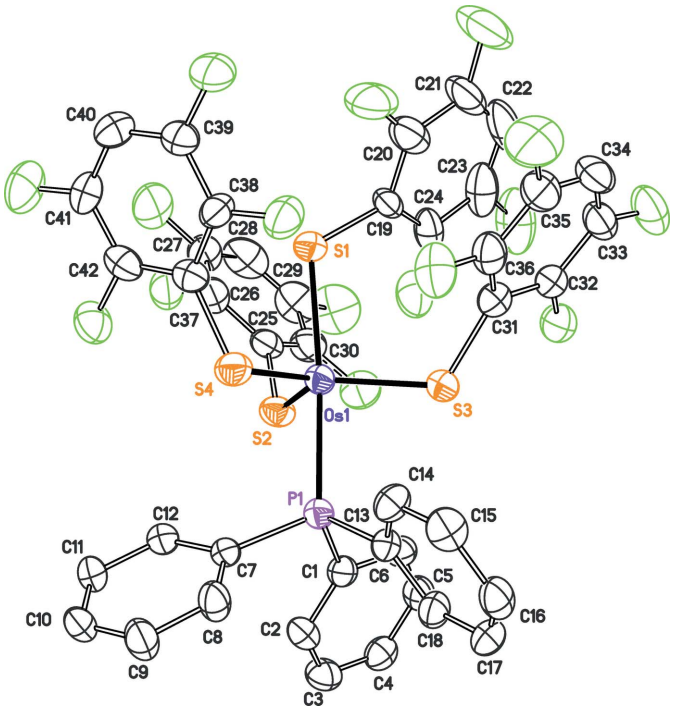


Figure 1
Molecular structure of the title compound, with displacement ellipsoids at the 30% probability level. F atoms are labelled F1 to F16 starting from the F site bonded to C20, and H atoms are omitted for clarity.

The title polymorph (Fig. 1) has the expected trigonal–bipyramidal coordination geometry, with the phosphine and one thiolate ligand in axial positions, with bond lengths Os—P = 2.4178 (19) and Os—S_{ax} = 2.421 (2) Å, while the equatorial positions are occupied by three thiolate ligands, with shorter bond lengths Os—S_{eq} = 2.207 (2)–2.225 (2) Å. A fit between the molecular geometries of the two polymorphs gives an r.m.s deviation limited to 0.179 Å (Macrae *et al.*, 2008). The single conformer found in these two polymorphs contrasts with that found in the $[Os(C_6HF_4S)_4(PPh_3)]\cdot n$-hexane hemisolvate (Cerón *et al.*, 2006): the fit between the molecular geometry of the $P2_1/c$ unsolvated complex and the *n*-hexane hemisolvate gives an r.m.s deviation of 1.016 Å.

The shortest intermolecular contact in the crystal structure is a π–π contact between two benzene rings of neighbouring molecules, with a large separation of 4.047 (5) Å. In the case of the triclinic polymorph, intermolecular π–π contacts were more efficient, with a separation between stacked rings of 3.744 Å (Arroyo *et al.*, 1994). However, this difference is probably not significant, since both polymorphs display almost the same density at room temperature. As expected, the complex crystallized with *n*-hexane in the lattice has a lower density (1.777 g cm^{-3} *versus* 1.832 g cm^{-3} for the unsolvated crystal), as well as larger separations between stacked benzene rings of 4.529 Å (Cerón *et al.*, 2006).

Synthesis and crystallization

The synthesis of the title complex was carried out using an methodology similar to that described for the triclinic polymorph (Arroyo *et al.*, 1994), by reacting 2,3,5,6-tetrafluorobenzenethiol (0.6 ml, 4 mmol), OsO_4 (0.25 g, 1 mmol) and PPh_3 (0.773 g, 3 mmol) in ethanol (15 ml) under stirring for two days at 295 K. During that time, a dark-green precipitate appeared; the solvent was distilled off under reduced pressure and the precipitate was filtered off and washed with cold ethanol (10 ml). The washed precipitate was dissolved again in ethanol and brown crystals of the title compound were obtained by slow evaporation, in 90% yield.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Os(C_6HF_4S)_4(C_{18}H_{15}P)]$
M_r	1176.98
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, *b*, *c* (Å)	12.4008 (5), 13.8892 (7), 25.2321 (11)
β (°)	100.991 (3)
V (Å^3)	4266.2 (3)
Z	4
Radiation type	Ag $K\alpha$, λ = 0.56083 Å
μ (mm^{-1})	1.80
Crystal size (mm)	0.20 × 0.06 × 0.03
Data collection	
Diffractometer	Stoe Stadivari
Absorption correction	Multi-scan (*X-AREA*; Stoe & Cie, 2018)
T_{min}, T_{max}	0.388, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	93107, 8692, 4264
R_{int}	0.186
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.092, 0.80
No. of reflections	8692
No. of parameters	577
No. of restraints	12
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.71, −1.05

Computer programs: *X-AREA* (Stoe & Cie, 2018), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2018), *XP* in *SHELXTL-Plus* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Because of an unresolved disorder affecting the benzene ring of thiol S1 (axial position), the C19–C24 ring was restrained to have a geometry similar to that of the C31–C36 ring, within standard deviations of 0.02 Å (1,2-distances) and 0.04 Å (1,3-distances; Sheldrick, 2015*b*).

Funding information

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full crystallographic data

IUCrData (2019). **4**, x191622 [https://doi.org/10.1107/S2414314619016225]

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Tetrakis(2,3,5,6-tetrafluorobenzenethiolato-κS)(triphenylphosphane-κP)osmium(IV)

Crystal data

$[Os(C_6HF_4S)_4(C_{18}H_{15}P)]$
M_r = 1176.98
Monoclinic, $P2_1/c$
a = 12.4008 (5) Å
b = 13.8892 (7) Å
c = 25.2321 (11) Å
β = 100.991 (3)°
V = 4266.2 (3) Å^3
Z = 4
$F(000)$ = 2280
D_x = 1.832 Mg m^{-3}
Ag $K\alpha$ radiation, λ = 0.56083 Å
Cell parameters from 22104 reflections
θ = 2.0–23.5°
μ = 1.80 mm^{-1}
T = 295 K
Plate, brown
0.20 × 0.06 × 0.03 mm

Data collection

Stoe Stadivari diffractometer
Radiation source: Sealed X-ray tube, Axo Astix-f Microfocus source
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (X-AREA; Stoe & Cie, 2018)
T_{min} = 0.388, T_{max} = 1.000
93107 measured reflections
8692 independent reflections
4264 reflections with $I > 2\sigma(I)$
R_{int} = 0.186
θ_{max} = 20.5°, θ_{min} = 2.0°
h = −15→15
k = −17→17
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.092
S = 0.80
8692 reflections
577 parameters
12 restraints
0 constraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −1.05 e Å^{-3}

Special details

Refinement. All H atoms were placed in idealized positions, with C—H bond lengths of 0.93 Å and U_{iso}(H) = 1.2×U_{eq}(carrier C atom).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Os1	0.26257 (2)	0.72225 (2)	0.54794 (2)	0.05251 (10)
P1	0.23237 (15)	0.63058 (14)	0.46496 (8)	0.0522 (5)
S1	0.30213 (16)	0.82548 (15)	0.62659 (8)	0.0668 (6)
S2	0.38093 (16)	0.60874 (16)	0.58113 (8)	0.0670 (6)
S3	0.08383 (15)	0.70369 (16)	0.54370 (8)	0.0656 (6)
S4	0.32211 (17)	0.83611 (16)	0.49853 (8)	0.0695 (6)
C1	0.2366 (5)	0.5002 (5)	0.4732 (3)	0.0554 (19)
C2	0.2910 (6)	0.4419 (6)	0.4430 (3)	0.065 (2)
H2	0.329275	0.469588	0.418569	0.077*
C3	0.2900 (7)	0.3425 (6)	0.4483 (4)	0.074 (2)
H3	0.326374	0.304047	0.427095	0.089*
C4	0.2349 (6)	0.3008 (6)	0.4851 (3)	0.067 (2)
H4	0.234290	0.234273	0.489147	0.080*
C5	0.1808 (6)	0.3593 (7)	0.5158 (3)	0.067 (2)
H5	0.142517	0.331698	0.540215	0.081*
C6	0.1826 (6)	0.4577 (6)	0.5106 (3)	0.059 (2)
H6	0.147568	0.496165	0.532308	0.071*
C7	0.3332 (5)	0.6512 (5)	0.4226 (3)	0.0505 (18)
C8	0.3022 (6)	0.6737 (6)	0.3679 (3)	0.071 (2)
H8	0.228602	0.685311	0.353508	0.085*
C9	0.3789 (7)	0.6791 (6)	0.3349 (3)	0.079 (3)
H9	0.355914	0.690868	0.298149	0.095*
C10	0.4878 (7)	0.6673 (6)	0.3555 (4)	0.068 (2)
H10	0.539175	0.671926	0.333165	0.081*
C11	0.5213 (6)	0.6485 (5)	0.4097 (3)	0.062 (2)
H11	0.595667	0.640958	0.423953	0.075*
C12	0.4450 (6)	0.6406 (5)	0.4432 (3)	0.060 (2)
H12	0.468844	0.628102	0.479744	0.071*
C13	0.1013 (5)	0.6534 (5)	0.4210 (3)	0.0491 (18)
C14	0.0643 (6)	0.7474 (5)	0.4116 (3)	0.062 (2)
H14	0.105932	0.798161	0.428677	0.074*
C15	−0.0352 (7)	0.7661 (7)	0.3766 (3)	0.076 (2)
H15	−0.058204	0.829321	0.369675	0.092*
C16	−0.0999 (6)	0.6912 (8)	0.3522 (3)	0.075 (3)
H16	−0.166895	0.703585	0.329645	0.090*
C17	−0.0634 (6)	0.5979 (7)	0.3619 (3)	0.071 (2)
H17	−0.105422	0.547273	0.344834	0.086*
C18	0.0359 (6)	0.5783 (6)	0.3969 (3)	0.064 (2)
H18	0.058057	0.514870	0.404097	0.077*
C19	0.2201 (6)	0.8136 (6)	0.6755 (3)	0.060 (2)
C20	0.1820 (8)	0.8938 (9)	0.6965 (4)	0.094 (3)
C21	0.1238 (10)	0.8909 (13)	0.7386 (6)	0.133 (6)
C22	0.0940 (10)	0.8069 (16)	0.7575 (5)	0.155 (9)
H22	0.047515	0.804355	0.782347	0.186*
C23	0.1340 (8)	0.7267 (12)	0.7392 (4)	0.108 (4)

C24	0.1917 (6)	0.7276 (9)	0.6974 (3)	0.081 (3)
C25	0.4434 (6)	0.6181 (6)	0.6500 (3)	0.064 (2)
C26	0.5247 (7)	0.6827 (7)	0.6702 (3)	0.073 (2)
C27	0.5792 (7)	0.6786 (9)	0.7224 (4)	0.095 (3)
C28	0.5575 (10)	0.6084 (12)	0.7576 (4)	0.117 (4)
H28	0.596547	0.604811	0.792873	0.140*
C29	0.4772 (11)	0.5456 (9)	0.7386 (5)	0.106 (3)
C30	0.4203 (8)	0.5490 (8)	0.6859 (4)	0.082 (3)
C31	0.0172 (5)	0.7771 (6)	0.5852 (3)	0.0599 (18)
C32	−0.0365 (6)	0.7385 (6)	0.6231 (3)	0.063 (2)
C33	−0.1025 (7)	0.7926 (8)	0.6505 (4)	0.079 (3)
C34	−0.1176 (7)	0.8872 (9)	0.6387 (4)	0.095 (3)
H34	−0.160757	0.924662	0.657037	0.114*
C35	−0.0691 (7)	0.9272 (7)	0.6000 (5)	0.089 (3)
C36	−0.0021 (7)	0.8743 (7)	0.5736 (4)	0.077 (3)
C37	0.3850 (6)	0.9313 (5)	0.5385 (3)	0.061 (2)
C38	0.3314 (6)	1.0133 (6)	0.5508 (3)	0.063 (2)
C39	0.3847 (7)	1.0855 (6)	0.5819 (4)	0.069 (2)
C40	0.4944 (7)	1.0825 (7)	0.5998 (3)	0.075 (2)
H40	0.529985	1.132860	0.620328	0.091*
C41	0.5525 (7)	1.0049 (7)	0.5876 (4)	0.072 (2)
C42	0.4977 (7)	0.9318 (6)	0.5572 (3)	0.069 (2)
F1	0.2049 (5)	0.9820 (5)	0.6771 (3)	0.129 (2)
F2	0.0878 (6)	0.9748 (7)	0.7556 (4)	0.205 (4)
F3	0.1098 (5)	0.6368 (7)	0.7576 (3)	0.166 (3)
F4	0.2212 (4)	0.6441 (4)	0.6780 (3)	0.110 (2)
F5	0.5539 (4)	0.7526 (4)	0.6381 (2)	0.0948 (17)
F6	0.6577 (4)	0.7465 (6)	0.7405 (2)	0.142 (3)
F7	0.4529 (6)	0.4739 (6)	0.7704 (3)	0.155 (3)
F8	0.3406 (4)	0.4832 (4)	0.6691 (2)	0.0977 (16)
F9	−0.0231 (4)	0.6436 (4)	0.6353 (2)	0.0864 (14)
F10	−0.1503 (4)	0.7474 (5)	0.6865 (2)	0.118 (2)
F11	−0.0839 (5)	1.0212 (4)	0.5886 (3)	0.141 (2)
F12	0.0411 (4)	0.9153 (4)	0.5348 (2)	0.1025 (18)
F13	0.2227 (4)	1.0214 (3)	0.5329 (2)	0.0893 (15)
F14	0.3251 (4)	1.1613 (3)	0.5948 (2)	0.0964 (16)
F15	0.6604 (4)	0.9994 (4)	0.6038 (2)	0.1128 (19)
F16	0.5587 (4)	0.8562 (3)	0.5443 (2)	0.0889 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.04872 (15)	0.06055 (18)	0.04846 (16)	−0.00044 (17)	0.00977 (11)	−0.00498 (19)
P1	0.0462 (10)	0.0602 (13)	0.0503 (13)	−0.0004 (9)	0.0095 (10)	−0.0050 (10)
S1	0.0724 (13)	0.0704 (14)	0.0611 (13)	−0.0117 (10)	0.0219 (11)	−0.0167 (11)
S2	0.0684 (13)	0.0770 (16)	0.0533 (12)	0.0080 (10)	0.0062 (10)	−0.0093 (11)
S3	0.0563 (11)	0.0772 (17)	0.0641 (13)	−0.0016 (10)	0.0139 (10)	−0.0169 (11)
S4	0.0823 (14)	0.0682 (15)	0.0600 (13)	−0.0109 (11)	0.0183 (11)	−0.0089 (11)

C1	0.044 (4)	0.062 (5)	0.058 (5)	0.004 (3)	0.003 (4)	−0.006 (4)
C2	0.066 (5)	0.073 (6)	0.059 (5)	0.005 (4)	0.023 (4)	−0.003 (5)
C3	0.083 (6)	0.060 (6)	0.082 (6)	0.007 (5)	0.023 (5)	−0.010 (5)
C4	0.059 (5)	0.061 (6)	0.075 (6)	0.002 (4)	−0.001 (4)	0.002 (5)
C5	0.065 (5)	0.080 (7)	0.058 (5)	−0.004 (4)	0.015 (4)	0.008 (5)
C6	0.061 (5)	0.054 (5)	0.063 (5)	0.000 (4)	0.011 (4)	−0.003 (4)
C7	0.043 (4)	0.057 (5)	0.052 (5)	−0.001 (3)	0.009 (3)	−0.011 (4)
C8	0.049 (5)	0.110 (7)	0.052 (5)	−0.004 (4)	0.008 (4)	0.006 (5)
C9	0.059 (5)	0.119 (8)	0.062 (6)	−0.001 (5)	0.019 (5)	0.002 (5)
C10	0.068 (6)	0.072 (6)	0.071 (6)	−0.001 (4)	0.034 (5)	−0.010 (5)
C11	0.050 (4)	0.071 (6)	0.070 (6)	−0.001 (4)	0.022 (4)	−0.003 (5)
C12	0.054 (5)	0.069 (6)	0.056 (5)	−0.004 (4)	0.011 (4)	−0.010 (4)
C13	0.046 (4)	0.049 (5)	0.053 (5)	−0.005 (3)	0.011 (3)	0.001 (4)
C14	0.055 (4)	0.061 (7)	0.063 (5)	−0.001 (3)	−0.005 (4)	0.005 (4)
C15	0.073 (5)	0.088 (7)	0.067 (5)	0.011 (5)	0.009 (4)	0.011 (5)
C16	0.048 (5)	0.117 (9)	0.058 (5)	0.010 (5)	0.005 (4)	0.002 (5)
C17	0.052 (5)	0.092 (7)	0.066 (6)	−0.011 (4)	0.001 (4)	−0.016 (5)
C18	0.048 (4)	0.077 (6)	0.065 (5)	−0.005 (4)	0.007 (4)	−0.002 (4)
C19	0.046 (4)	0.082 (7)	0.055 (5)	0.010 (4)	0.013 (4)	−0.017 (4)
C20	0.081 (7)	0.129 (10)	0.072 (7)	0.018 (7)	0.013 (5)	−0.024 (7)
C21	0.080 (9)	0.223 (19)	0.100 (11)	0.017 (9)	0.027 (7)	−0.084 (12)
C22	0.053 (7)	0.34 (3)	0.068 (8)	0.009 (11)	0.008 (6)	−0.057 (13)
C23	0.065 (6)	0.170 (12)	0.088 (8)	−0.007 (8)	0.009 (6)	0.046 (9)
C24	0.060 (5)	0.118 (9)	0.069 (6)	−0.004 (6)	0.023 (4)	0.019 (7)
C25	0.061 (5)	0.075 (6)	0.055 (5)	0.014 (4)	0.012 (4)	−0.011 (5)
C26	0.056 (5)	0.108 (8)	0.053 (5)	0.001 (5)	0.007 (4)	0.000 (5)
C27	0.063 (6)	0.158 (11)	0.060 (6)	−0.007 (6)	−0.003 (5)	−0.022 (7)
C28	0.107 (9)	0.177 (14)	0.057 (7)	0.037 (9)	−0.007 (7)	−0.004 (8)
C29	0.125 (10)	0.116 (11)	0.077 (9)	0.014 (7)	0.023 (8)	0.026 (8)
C30	0.084 (7)	0.095 (8)	0.067 (7)	0.004 (6)	0.009 (5)	−0.007 (6)
C31	0.058 (4)	0.059 (5)	0.060 (5)	0.002 (4)	0.004 (4)	0.005 (5)
C32	0.061 (5)	0.062 (6)	0.067 (5)	−0.005 (4)	0.015 (4)	−0.005 (4)
C33	0.067 (5)	0.104 (8)	0.071 (6)	0.008 (6)	0.024 (5)	−0.006 (6)
C34	0.073 (6)	0.112 (10)	0.102 (9)	0.021 (6)	0.021 (6)	−0.032 (7)
C35	0.071 (6)	0.075 (8)	0.120 (9)	0.018 (5)	0.014 (6)	−0.007 (7)
C36	0.057 (5)	0.097 (8)	0.074 (6)	0.000 (5)	0.008 (5)	0.006 (6)
C37	0.072 (5)	0.057 (5)	0.056 (5)	0.004 (4)	0.017 (4)	0.000 (4)
C38	0.057 (5)	0.052 (5)	0.075 (6)	−0.008 (4)	0.006 (4)	−0.006 (4)
C39	0.070 (6)	0.062 (6)	0.077 (6)	0.005 (4)	0.016 (5)	−0.008 (5)
C40	0.076 (6)	0.072 (7)	0.073 (6)	−0.010 (5)	−0.001 (5)	−0.010 (5)
C41	0.054 (5)	0.076 (7)	0.082 (6)	−0.012 (5)	0.004 (5)	0.011 (5)
C42	0.083 (6)	0.063 (6)	0.068 (6)	0.017 (5)	0.029 (5)	0.003 (5)
F1	0.131 (5)	0.094 (5)	0.157 (6)	0.027 (4)	0.018 (4)	−0.046 (5)
F2	0.158 (6)	0.260 (10)	0.207 (8)	0.046 (6)	0.061 (6)	−0.127 (8)
F3	0.117 (5)	0.245 (10)	0.144 (6)	−0.008 (5)	0.042 (4)	0.087 (6)
F4	0.095 (4)	0.096 (5)	0.148 (6)	0.003 (3)	0.043 (4)	0.019 (4)
F5	0.076 (3)	0.127 (5)	0.084 (3)	−0.018 (3)	0.024 (3)	−0.006 (3)
F6	0.088 (4)	0.240 (8)	0.089 (4)	−0.033 (4)	−0.007 (3)	−0.040 (5)

F7	0.203 (7)	0.158 (7)	0.101 (5)	0.005 (5)	0.018 (5)	0.048 (5)
F8	0.112 (4)	0.080 (4)	0.100 (4)	−0.002 (3)	0.016 (3)	0.007 (3)
F9	0.087 (3)	0.080 (4)	0.096 (4)	−0.011 (3)	0.030 (3)	−0.001 (3)
F10	0.094 (4)	0.167 (6)	0.105 (4)	0.010 (3)	0.054 (3)	0.003 (4)
F11	0.135 (5)	0.076 (4)	0.200 (7)	0.040 (4)	0.005 (5)	0.000 (4)
F12	0.087 (4)	0.092 (4)	0.132 (5)	0.007 (3)	0.028 (3)	0.038 (4)
F13	0.065 (3)	0.072 (3)	0.126 (4)	−0.001 (2)	0.008 (3)	−0.010 (3)
F14	0.093 (3)	0.063 (3)	0.133 (5)	0.010 (3)	0.022 (3)	−0.019 (3)
F15	0.054 (3)	0.120 (5)	0.153 (5)	−0.003 (3)	−0.008 (3)	0.009 (4)
F16	0.079 (3)	0.078 (4)	0.116 (4)	0.013 (2)	0.034 (3)	0.003 (3)

Geometric parameters (Å, º)

Os1—S2	2.207 (2)	C19—C20	1.358 (11)
Os1—S3	2.2138 (19)	C19—C24	1.391 (11)
Os1—S4	2.225 (2)	C20—F1	1.368 (11)
Os1—P1	2.4178 (19)	C20—C21	1.394 (13)
Os1—S1	2.421 (2)	C21—C22	1.339 (15)
P1—C13	1.812 (7)	C21—F2	1.346 (15)
P1—C7	1.817 (7)	C22—C23	1.336 (15)
P1—C1	1.822 (8)	C22—H22	0.9300
S1—C19	1.749 (8)	C23—C24	1.381 (11)
S2—C25	1.768 (8)	C23—F3	1.386 (13)
S3—C31	1.773 (8)	C24—F4	1.337 (11)
S4—C37	1.754 (8)	C25—C26	1.373 (11)
C1—C2	1.373 (9)	C25—C30	1.386 (12)
C1—C6	1.389 (10)	C26—F5	1.358 (9)
C2—C3	1.388 (10)	C26—C27	1.362 (12)
C2—H2	0.9300	C27—F6	1.370 (11)
C3—C4	1.380 (10)	C27—C28	1.379 (15)
C3—H3	0.9300	C28—C29	1.341 (15)
C4—C5	1.382 (10)	C28—H28	0.9300
C4—H4	0.9300	C29—F7	1.350 (12)
C5—C6	1.374 (10)	C29—C30	1.381 (13)
C5—H5	0.9300	C30—F8	1.353 (10)
C6—H6	0.9300	C31—C32	1.375 (9)
C7—C12	1.393 (9)	C31—C36	1.393 (10)
C7—C8	1.394 (9)	C32—F9	1.356 (8)
C8—C9	1.382 (10)	C32—C33	1.388 (10)
C8—H8	0.9300	C33—F10	1.332 (10)
C9—C10	1.362 (10)	C33—C34	1.353 (11)
C9—H9	0.9300	C34—C35	1.359 (12)
C10—C11	1.375 (10)	C34—H34	0.9300
C10—H10	0.9300	C35—F11	1.344 (10)
C11—C12	1.388 (9)	C35—C36	1.374 (11)
C11—H11	0.9300	C36—F12	1.329 (9)
C12—H12	0.9300	C37—C38	1.383 (10)
C13—C14	1.389 (9)	C37—C42	1.386 (10)

C13—C18	1.389 (9)	C38—F13	1.342 (8)
C14—C15	1.398 (9)	C38—C39	1.364 (10)
C14—H14	0.9300	C39—C40	1.350 (10)
C15—C16	1.384 (11)	C39—F14	1.361 (8)
C15—H15	0.9300	C40—C41	1.364 (11)
C16—C17	1.378 (11)	C40—H40	0.9300
C16—H16	0.9300	C41—F15	1.324 (8)
C17—C18	1.399 (10)	C41—C42	1.372 (11)
C17—H17	0.9300	C42—F16	1.370 (8)
C18—H18	0.9300		

S2—Os1—S3	121.49 (8)	C17—C18—H18	120.0
S2—Os1—S4	116.91 (8)	C20—C19—C24	114.5 (9)
S3—Os1—S4	119.46 (8)	C20—C19—S1	119.4 (8)
S2—Os1—P1	86.24 (7)	C24—C19—S1	126.0 (6)
S3—Os1—P1	84.75 (7)	C19—C20—F1	118.9 (9)
S4—Os1—P1	84.39 (7)	C19—C20—C21	122.9 (12)
S2—Os1—S1	95.50 (7)	F1—C20—C21	118.1 (11)
S3—Os1—S1	98.80 (7)	C22—C21—F2	120.7 (15)
S4—Os1—S1	90.08 (7)	C22—C21—C20	120.9 (14)
P1—Os1—S1	174.40 (7)	F2—C21—C20	117.9 (15)
C13—P1—C7	104.5 (3)	C23—C22—C21	117.2 (14)
C13—P1—C1	104.3 (3)	C23—C22—H22	121.4
C7—P1—C1	102.4 (3)	C21—C22—H22	121.4
C13—P1—Os1	114.2 (2)	C22—C23—C24	122.5 (13)
C7—P1—Os1	114.6 (2)	C22—C23—F3	121.0 (13)
C1—P1—Os1	115.4 (2)	C24—C23—F3	116.2 (13)
C19—S1—Os1	117.9 (3)	F4—C24—C23	119.3 (11)
C25—S2—Os1	117.1 (3)	F4—C24—C19	119.4 (7)
C31—S3—Os1	118.6 (2)	C23—C24—C19	121.2 (11)
C37—S4—Os1	111.8 (3)	C26—C25—C30	116.0 (8)
C2—C1—C6	118.5 (8)	C26—C25—S2	124.5 (7)
C2—C1—P1	122.0 (6)	C30—C25—S2	119.0 (7)
C6—C1—P1	119.5 (6)	F5—C26—C27	117.8 (9)
C1—C2—C3	121.3 (8)	F5—C26—C25	120.4 (7)
C1—C2—H2	119.3	C27—C26—C25	121.7 (10)
C3—C2—H2	119.3	C26—C27—F6	118.9 (11)
C4—C3—C2	119.7 (8)	C26—C27—C28	122.0 (11)
C4—C3—H3	120.1	F6—C27—C28	119.1 (10)
C2—C3—H3	120.1	C29—C28—C27	116.9 (10)
C3—C4—C5	119.1 (8)	C29—C28—H28	121.6
C3—C4—H4	120.5	C27—C28—H28	121.6
C5—C4—H4	120.5	C28—C29—F7	120.1 (12)
C6—C5—C4	120.9 (8)	C28—C29—C30	122.1 (11)
C6—C5—H5	119.6	F7—C29—C30	117.8 (13)
C4—C5—H5	119.6	F8—C30—C29	119.3 (11)
C5—C6—C1	120.4 (7)	F8—C30—C25	119.4 (8)
C5—C6—H6	119.8	C29—C30—C25	121.3 (10)

C1—C6—H6	119.8	C32—C31—C36	115.9 (7)
C12—C7—C8	117.4 (7)	C32—C31—S3	121.9 (7)
C12—C7—P1	120.8 (6)	C36—C31—S3	120.9 (6)
C8—C7—P1	121.8 (5)	F9—C32—C31	118.8 (7)
C9—C8—C7	121.2 (7)	F9—C32—C33	118.1 (8)
C9—C8—H8	119.4	C31—C32—C33	123.1 (9)
C7—C8—H8	119.4	F10—C33—C34	123.1 (9)
C10—C9—C8	120.6 (8)	F10—C33—C32	117.8 (10)
C10—C9—H9	119.7	C34—C33—C32	119.1 (9)
C8—C9—H9	119.7	C33—C34—C35	119.5 (9)
C9—C10—C11	119.5 (8)	C33—C34—H34	120.2
C9—C10—H10	120.2	C35—C34—H34	120.2
C11—C10—H10	120.2	F11—C35—C34	119.4 (10)
C10—C11—C12	120.6 (7)	F11—C35—C36	119.1 (10)
C10—C11—H11	119.7	C34—C35—C36	121.5 (10)
C12—C11—H11	119.7	F12—C36—C35	119.6 (10)
C11—C12—C7	120.7 (7)	F12—C36—C31	119.5 (8)
C11—C12—H12	119.7	C35—C36—C31	120.8 (9)
C7—C12—H12	119.7	C38—C37—C42	114.6 (7)
C14—C13—C18	119.0 (7)	C38—C37—S4	124.7 (6)
C14—C13—P1	119.9 (5)	C42—C37—S4	120.5 (6)
C18—C13—P1	121.1 (6)	F13—C38—C39	118.5 (7)
C13—C14—C15	120.4 (7)	F13—C38—C37	119.3 (7)
C13—C14—H14	119.8	C39—C38—C37	122.2 (8)
C15—C14—H14	119.8	C40—C39—F14	120.1 (8)
C16—C15—C14	120.5 (8)	C40—C39—C38	121.1 (8)
C16—C15—H15	119.7	F14—C39—C38	118.7 (8)
C14—C15—H15	119.7	C39—C40—C41	119.4 (8)
C17—C16—C15	119.0 (8)	C39—C40—H40	120.3
C17—C16—H16	120.5	C41—C40—H40	120.3
C15—C16—H16	120.5	F15—C41—C40	121.5 (8)
C16—C17—C18	120.9 (8)	F15—C41—C42	119.5 (9)
C16—C17—H17	119.5	C40—C41—C42	119.0 (8)
C18—C17—H17	119.5	F16—C42—C41	117.6 (8)
C13—C18—C17	120.1 (8)	F16—C42—C37	118.8 (8)
C13—C18—H18	120.0	C41—C42—C37	123.6 (8)

C13—P1—C1—C2	97.8 (6)	Os1—S2—C25—C30	113.9 (7)
C7—P1—C1—C2	−10.9 (7)	C30—C25—C26—F5	179.4 (7)
Os1—P1—C1—C2	−136.1 (5)	S2—C25—C26—F5	7.7 (11)
C13—P1—C1—C6	−81.6 (6)	C30—C25—C26—C27	0.2 (13)
C7—P1—C1—C6	169.7 (6)	S2—C25—C26—C27	−171.6 (7)
Os1—P1—C1—C6	44.5 (6)	F5—C26—C27—F6	2.2 (13)
C6—C1—C2—C3	1.9 (11)	C25—C26—C27—F6	−178.5 (8)
P1—C1—C2—C3	−177.5 (6)	F5—C26—C27—C28	−178.0 (9)
C1—C2—C3—C4	−1.0 (12)	C25—C26—C27—C28	1.3 (16)
C2—C3—C4—C5	0.6 (11)	C26—C27—C28—C29	−2.0 (18)
C3—C4—C5—C6	−1.0 (11)	F6—C27—C28—C29	177.7 (10)

C4—C5—C6—C1	1.9 (11)	C27—C28—C29—F7	178.7 (10)
C2—C1—C6—C5	−2.3 (10)	C27—C28—C29—C30	1.4 (18)
P1—C1—C6—C5	177.1 (5)	C28—C29—C30—F8	179.6 (10)
C13—P1—C7—C12	179.8 (6)	F7—C29—C30—F8	2.3 (15)
C1—P1—C7—C12	−71.7 (6)	C28—C29—C30—C25	−0.1 (17)
Os1—P1—C7—C12	54.0 (6)	F7—C29—C30—C25	−177.4 (9)
C13—P1—C7—C8	−3.3 (7)	C26—C25—C30—F8	179.6 (7)
C1—P1—C7—C8	105.2 (7)	S2—C25—C30—F8	−8.2 (11)
Os1—P1—C7—C8	−129.1 (6)	C26—C25—C30—C29	−0.7 (14)
C12—C7—C8—C9	4.1 (12)	S2—C25—C30—C29	171.5 (8)
P1—C7—C8—C9	−173.0 (6)	Os1—S3—C31—C32	−119.9 (6)
C7—C8—C9—C10	−3.4 (13)	Os1—S3—C31—C36	73.1 (6)
C8—C9—C10—C11	1.0 (13)	C36—C31—C32—F9	178.4 (6)
C9—C10—C11—C12	0.6 (12)	S3—C31—C32—F9	10.9 (9)
C10—C11—C12—C7	0.2 (11)	C36—C31—C32—C33	−2.7 (11)
C8—C7—C12—C11	−2.5 (10)	S3—C31—C32—C33	−170.3 (6)
P1—C7—C12—C11	174.5 (6)	F9—C32—C33—F10	−1.0 (11)
C7—P1—C13—C14	−81.0 (6)	C31—C32—C33—F10	−179.8 (7)
C1—P1—C13—C14	171.8 (6)	F9—C32—C33—C34	−179.6 (7)
Os1—P1—C13—C14	45.0 (6)	C31—C32—C33—C34	1.6 (13)
C7—P1—C13—C18	99.8 (6)	F10—C33—C34—C35	−177.6 (8)
C1—P1—C13—C18	−7.3 (7)	C32—C33—C34—C35	0.9 (14)
Os1—P1—C13—C18	−134.2 (5)	C33—C34—C35—F11	−179.8 (8)
C18—C13—C14—C15	−2.6 (11)	C33—C34—C35—C36	−2.1 (15)
P1—C13—C14—C15	178.2 (6)	F11—C35—C36—F12	−4.2 (13)
C13—C14—C15—C16	2.0 (12)	C34—C35—C36—F12	178.0 (8)
C14—C15—C16—C17	−1.6 (12)	F11—C35—C36—C31	178.6 (7)
C15—C16—C17—C18	1.8 (12)	C34—C35—C36—C31	0.8 (14)
C14—C13—C18—C17	2.8 (11)	C32—C31—C36—F12	−175.7 (7)
P1—C13—C18—C17	−178.0 (6)	S3—C31—C36—F12	−8.0 (10)
C16—C17—C18—C13	−2.4 (12)	C32—C31—C36—C35	1.5 (11)
Os1—S1—C19—C20	−135.1 (6)	S3—C31—C36—C35	169.2 (7)
Os1—S1—C19—C24	47.4 (8)	Os1—S4—C37—C38	−91.8 (7)
C24—C19—C20—F1	−179.4 (8)	Os1—S4—C37—C42	93.2 (6)
S1—C19—C20—F1	2.9 (12)	C42—C37—C38—F13	177.7 (7)
C24—C19—C20—C21	3.1 (13)	S4—C37—C38—F13	2.4 (11)
S1—C19—C20—C21	−174.7 (8)	C42—C37—C38—C39	−4.3 (12)
C19—C20—C21—C22	−6 (2)	S4—C37—C38—C39	−179.6 (6)
F1—C20—C21—C22	176.6 (13)	F13—C38—C39—C40	−178.2 (8)
C19—C20—C21—F2	−178.6 (9)	C37—C38—C39—C40	3.8 (13)
F1—C20—C21—F2	3.8 (15)	F13—C38—C39—F14	1.8 (12)
F2—C21—C22—C23	−179.3 (9)	C37—C38—C39—F14	−176.2 (8)
C20—C21—C22—C23	8 (2)	F14—C39—C40—C41	178.5 (8)
C21—C22—C23—C24	−8 (2)	C38—C39—C40—C41	−1.5 (14)
C21—C22—C23—F3	178.3 (11)	C39—C40—C41—F15	179.2 (8)
C22—C23—C24—F4	−174.3 (10)	C39—C40—C41—C42	0.1 (13)
F3—C23—C24—F4	−0.6 (12)	F15—C41—C42—F16	−1.1 (12)
C22—C23—C24—C19	6.1 (15)	C40—C41—C42—F16	178.2 (8)

F3—C23—C24—C19	179.7 (8)	F15—C41—C42—C37	180.0 (7)
C20—C19—C24—F4	177.2 (7)	C40—C41—C42—C37	−0.8 (13)
S1—C19—C24—F4	−5.2 (11)	C38—C37—C42—F16	−176.1 (7)
C20—C19—C24—C23	−3.1 (12)	S4—C37—C42—F16	−0.6 (10)
S1—C19—C24—C23	174.5 (6)	C38—C37—C42—C41	2.9 (12)
Os1—S2—C25—C26	−74.5 (7)	S4—C37—C42—C41	178.4 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···S2	0.93	2.98	3.742 (8)	140
C14—H14···S4	0.93	2.96	3.725 (7)	140
C14—H14···F11^{i}	0.93	2.55	3.223 (10)	129
C18—H18···F9^{ii}	0.93	2.42	3.183 (10)	139
C34—H34···F15^{iii}	0.93	2.58	3.140 (11)	119

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