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Bis(flavonolato-κ^2O,O')dioxidoosmium(VI) dichloromethane disolvate

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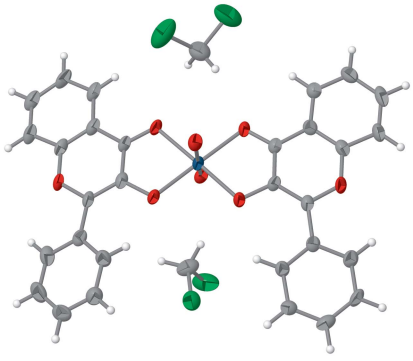
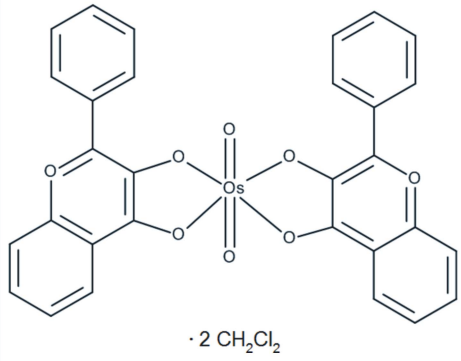
In the crystal structure of the title solvated *trans*-dioxidoosmium(VI) flavonolate (flav) complex, $[Os(C_{15}H_9O_3)_2O_2]\cdot 2CH_2Cl_2$ or $[Os(flav)_2O_2]\cdot 2CH_2Cl_2$, the two dichloromethane solvent molecules have nonclassical hydrogen-bonding contacts at or greater than 3.18 Å. The pseudo-octahedrally coordinated central metal cation is observed with all donor atoms being oxygen. The Os=O bond lengths are 1.721 (5) and 1.728 (5) Å, with a 170.4 (2)° bond angle. The O—Os bond lengths arising from the flanvonolate ligand are observed to all be slightly over 2.0 Å. The chelate bond angles arising from the flavonolate O atoms with the osmium cation are constrained by the ligand at 80.72 (18) and 80.92 (17)°.

3D view



Chemical scheme



Structure description

Quercetin 2,3-dioxygenase is a metalloprotein that catalyzes a ring-opening reaction of the polyphenolic heterocycle quercetin. Quercetin (3′,4′,5,7-tetrahydoxyflavonol) undergoes activation at a central metal cation to relase carbon monoxide and produce the corresponding depside. A great deal of attention has been focused recently on small biomimetic complexes that bind flavonol (and its derivatives) to a central metal cation (see, for example, Sun *et al.*, 2013, and references therein). We have extended some of these reports to present the first osmium flavonolate complex reported in the literature. This structure is the third osmyl (*trans*-OsO_2^{2+}) complex reported with the equatorial plane being composed of four O-atom donors.

The dioxidoosmium(VI) moiety in the title structure is completed by four O atoms from two flavonolate anions, resulting in a pseudo-octahedrally coordinated central metal cation, with all six donor atoms being oxygen. Previous structures of this type with the *trans*-disposed osmyl ion comprised of all O atoms in the equatorial plane are moderately rare (Stanislas *et al.*, 2000; Burvikova *et al.*, 2007; Struess & Preetz, 1998). These examples are also of the highly oxidized ligands malonate and oxalate, so a structure of a

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O3	0.95	2.24	2.876 (9)	124
C26—H26···O6	0.95	2.24	2.910 (8)	127
C30—H30···O2^{i}	0.95	2.49	3.393 (9)	158
C31—H31*A*···O4	0.99	2.56	3.180 (11)	121
C31—H31*B*···O7	0.99	2.47	3.221 (11)	133
C32—H32*A*···O2	0.99	2.62	3.420 (10)	138
C32—H32*A*···O6	0.99	2.55	3.489 (11)	158

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Os(C_{15}H_9O_3)_2O_2]\cdot 2CH_2Cl_2$
M_r	866.49
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	8.523 (1), 17.041 (2), 21.255 (2)
β (°)	93.565 (3)
V (Å^3)	3081 (1)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.54
Crystal size (mm)	0.24 × 0.18 × 0.16
Data collection	
Diffractometer	Rigaku XtalLab mini CCD
Absorption correction	Multi-scan (*REQAB*; Rigaku, 1998)
T_{min}, T_{max}	0.613, 0.765
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32224, 7032, 5602
R_{int}	0.116
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.116, 1.10
No. of reflections	7032
No. of parameters	406
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.85, −2.79

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2011), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2017* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

moderately oxidizable ligand, such as 3-hydroxyflavone, is noteable. The title compound (Fig. 1) crystallizes with two dichloromethane solvent molecules in the unit cell. The *trans* Os═O axial bond lengths are 1.721 (5) Å for Os1—O1 and 1.728 (5) Å for Os1—O2. The *trans*-osmyl bond angle of 170.4 (2)° for O1—Os—O2 is similar to others wherein the structure is not centrosymmetric about the Os atom (see, for example, Lynch *et al.*, 1991). In the equatorial plane lie two anions of the deprotonated 3-hydroxyflavone, which are *cis* to each other. The Os—O bond lengths found from the ketone are 2.094 (4) Å for Os1—O4 and 2.088 (4) Å for Os1—O7. These are slightly longer than those observed for the deprotonated hydroxy O atoms of 2.008 (5) Å for Os1—O3 and 2.019 (4) Å for Os1—O6. Typically, in a flavonolate–metal complex, the hydroxy(oxygen)-to-metal bond length has been found to be shorter than that for the ketone(oxygen)-to-metal bond length (Sun *et al.*, 2013). In the title compound, the corresponding Δd(Os—O) are 0.086 and 0.069 Å. The chelate bond angle arising from the flavonolate and Os central atom are 80.72 (18)° for O3—Os1—O4 and 80.92 (17)° for O6—Os1—O7. These constrained bond angles are typical for metal–flavonolate chelates.

The two dichloromethane solvent molecules interact with the complex *via* several weak nonclassical hydrogen-bonding interactions, with donor–acceptor ($D\cdots A$) distances less than or equal to 3.5 Å (Table 1). The C31—O4 interaction length is 3.180 (11) Å, whereas the corresponding C31—O7 distance is 3.221 (11) Å. This single dichloromethane (C31 centered dicholormethane) solvent molecule spans the two flavonolate ligands *via* its two H atoms. The other solvent spans the opposite side of the equatorial plane using only atom H32*A*. The C32—O2 interaction length is 3.420 (10) Å, whereas the corresponding C32—O6 is distance 3.489 (11) Å (Fig. 2).

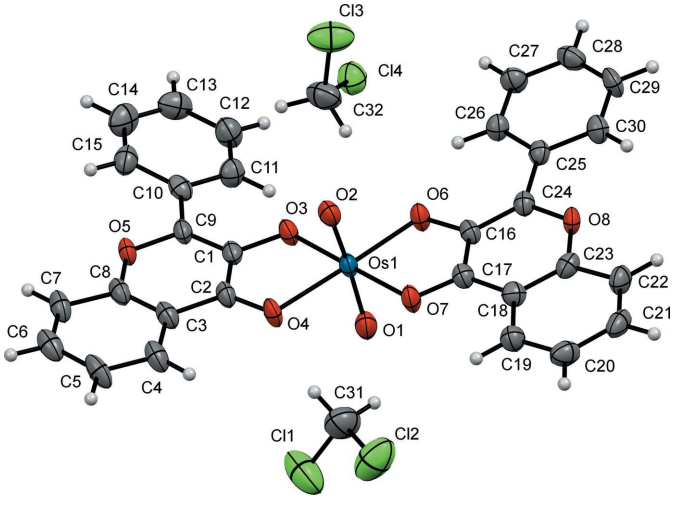


Figure 1
A view of the molecular components in the structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

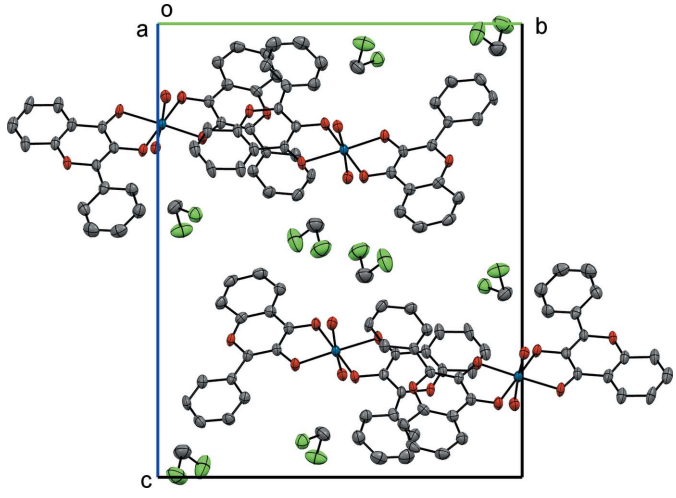


Figure 2
Crystal packing diagram of title compound viewed along the *a* axis. H atoms have been omitted for clarity.

Synthesis and crystallization

0.100 g of $K_2OsO_2(OH)_4$ (0.271 mmol) (Malin, 1980) was dissolved in approximately 20 mL of methanol under an ambient atmosphere. The solution turned royal blue as the potassium osmate dissolved. A second solution was made by dissolving 0.129 g of 3-hydroxyflavone (Hflav) (0.543 mmol) and 0.066 g of benzoic acid (0.543 mmol) in 20 mL of methanol. After dissolution, the solutions were mixed into one portion. The combined reaction mixture turned red, and a precipitate formed immediately. The solution was stirred for 10 min, filtered, washed with methanol and diethyl ether, and dried under vacuum. The yield of the crude red solid was 0.156 g. The solid product was dissolved in dichloromethane and deep-red–brown crystals were grown by slow evaporation of the solvent; the final yield of $[OsO_2(flav)_2]\cdot CH_2Cl_2$ was 0.082 g (35%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Three reflections with $(I_{obs} - I_{calc})/\sigma > 10$ were removed.

Acknowledgements

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full crystallographic data

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Bis(flavonolato-κ^2O,O')dioxidoosmium(VI) dichloromethane disolvate

Crystal data

$[Os(C_{15}H_9O_3)_2O_2]\cdot 2CH_2Cl_2$
$M_r = 866.49$
Monoclinic, $P2_1/n$
$a = 8.523$ (1) Å
$b = 17.041$ (2) Å
$c = 21.255$ (2) Å
$\beta = 93.565$ (3)°
$V = 3081$ (1) Å^3
$Z = 4$
$F(000) = 1688$
$D_x = 1.868$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7657 reflections
$\theta = 1.9$–$27.5°$
$\mu = 4.54$ mm^{-1}
$T = 173$ K
Prism, dark red-brown
0.24 × 0.18 × 0.16 mm

Data collection

Rigaku XtalLab mini CCD diffractometer
ω scans
Absorption correction: multi-scan (REQAB; Rigaku, 1998)
$T_{min} = 0.613$, $T_{max} = 0.765$
32224 measured reflections
7032 independent reflections
5602 reflections with $I > 2\sigma(I)$
$R_{int} = 0.116$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -11\rightarrow11$
$k = -22\rightarrow22$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.116$
$S = 1.10$
7032 reflections
406 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 4.729P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.85$ e Å^{-3}
$\Delta\rho_{min} = -2.79$ e Å^{-3}

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.

Refinement. All H atoms were positioned geometrically and refined as riding, with C—H = 0.95 or 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Os1	0.48652 (3)	0.51111 (2)	0.27829 (2)	0.02737 (9)
O1	0.6279 (6)	0.5216 (3)	0.3391 (2)	0.0400 (12)
O2	0.3256 (6)	0.4939 (2)	0.2266 (2)	0.0358 (11)
O3	0.6350 (5)	0.4595 (2)	0.2213 (2)	0.0314 (10)
O4	0.4613 (5)	0.3945 (2)	0.3069 (2)	0.0337 (11)
O5	0.6944 (5)	0.2514 (2)	0.1929 (2)	0.0315 (10)
O6	0.5081 (5)	0.6228 (2)	0.2482 (2)	0.0315 (10)
O7	0.3324 (5)	0.5657 (2)	0.3371 (2)	0.0336 (10)
O8	0.3221 (5)	0.7994 (2)	0.3028 (2)	0.0309 (10)
C1	0.6308 (8)	0.3796 (3)	0.2266 (3)	0.0303 (14)
C2	0.5357 (8)	0.3477 (3)	0.2719 (3)	0.0305 (14)
C3	0.5208 (8)	0.2640 (4)	0.2771 (3)	0.0360 (16)
C4	0.4271 (9)	0.2272 (4)	0.3206 (4)	0.0421 (18)
H4	0.374111	0.257647	0.350157	0.051*
C5	0.4126 (8)	0.1462 (4)	0.3201 (4)	0.0448 (19)
H5	0.348633	0.120842	0.348916	0.054*
C6	0.4938 (8)	0.1015 (4)	0.2762 (4)	0.0441 (19)
H6	0.481459	0.046136	0.275451	0.053*
C7	0.5901 (8)	0.1365 (4)	0.2348 (4)	0.0396 (17)
H7	0.647215	0.106259	0.206426	0.048*
C8	0.6005 (8)	0.2187 (4)	0.2361 (3)	0.0333 (15)
C9	0.7097 (8)	0.3311 (3)	0.1875 (3)	0.0309 (14)
C10	0.8096 (7)	0.3527 (4)	0.1375 (3)	0.0324 (15)
C11	0.8723 (8)	0.4290 (4)	0.1340 (3)	0.0378 (16)
H11	0.846246	0.467443	0.164045	0.045*
C12	0.9715 (10)	0.4486 (5)	0.0872 (4)	0.0473 (19)
H12	1.013997	0.500057	0.085599	0.057*
C13	1.0090 (9)	0.3934 (5)	0.0428 (4)	0.049 (2)
H13	1.075930	0.407190	0.010460	0.059*
C14	0.9482 (10)	0.3176 (5)	0.0456 (4)	0.052 (2)
H14	0.974602	0.279715	0.015201	0.063*
C15	0.8510 (9)	0.2972 (4)	0.0918 (4)	0.0415 (17)
H15	0.810849	0.245240	0.093262	0.050*
C16	0.4203 (7)	0.6736 (3)	0.2813 (3)	0.0270 (13)
C17	0.3310 (8)	0.6406 (4)	0.3284 (3)	0.0303 (14)
C18	0.2405 (8)	0.6910 (4)	0.3653 (3)	0.0314 (14)
C19	0.1505 (8)	0.6651 (4)	0.4145 (3)	0.0371 (16)
H19	0.147408	0.610917	0.424767	0.045*
C20	0.0658 (9)	0.7191 (5)	0.4484 (3)	0.0439 (18)
H20	0.005498	0.701891	0.481912	0.053*
C21	0.0706 (9)	0.7994 (4)	0.4324 (3)	0.0432 (19)
H21	0.013035	0.836124	0.455526	0.052*
C22	0.1561 (9)	0.8254 (4)	0.3845 (3)	0.0386 (16)
H22	0.158238	0.879587	0.374028	0.046*
C23	0.2408 (8)	0.7706 (4)	0.3511 (3)	0.0317 (14)

C24	0.4097 (8)	0.7525 (4)	0.2679 (3)	0.0293 (14)
C25	0.4792 (8)	0.7970 (4)	0.2169 (3)	0.0295 (14)
C26	0.5772 (8)	0.7613 (4)	0.1749 (3)	0.0315 (14)
H26	0.599592	0.706839	0.178319	0.038*
C27	0.6417 (8)	0.8056 (4)	0.1282 (3)	0.0375 (16)
H27	0.706811	0.780772	0.099439	0.045*
C28	0.6132 (9)	0.8850 (4)	0.1227 (3)	0.0411 (17)
H28	0.660083	0.914895	0.091133	0.049*
C29	0.5153 (9)	0.9206 (4)	0.1638 (4)	0.0398 (17)
H29	0.493774	0.975128	0.159809	0.048*
C30	0.4482 (8)	0.8779 (4)	0.2106 (3)	0.0355 (15)
H30	0.381369	0.903213	0.238503	0.043*
Cl1	0.5133 (4)	0.37694 (16)	0.47783 (17)	0.0986 (10)
Cl2	0.2072 (4)	0.44771 (18)	0.49726 (13)	0.0908 (9)
C31	0.3557 (12)	0.4323 (6)	0.4445 (4)	0.069 (3)
H31A	0.310036	0.404778	0.406660	0.082*
H31B	0.395507	0.483724	0.430934	0.082*
Cl3	0.3489 (3)	0.56545 (17)	0.04430 (11)	0.0729 (7)
Cl4	0.6777 (2)	0.60300 (12)	0.07876 (10)	0.0519 (5)
C32	0.5139 (10)	0.5474 (5)	0.0958 (4)	0.058 (2)
H32A	0.486664	0.558914	0.139420	0.070*
H32B	0.541291	0.491028	0.093663	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.03312 (16)	0.01694 (13)	0.03253 (16)	0.00219 (9)	0.00586 (11)	0.00099 (9)
O1	0.054 (3)	0.025 (2)	0.042 (3)	0.003 (2)	0.008 (2)	−0.002 (2)
O2	0.046 (3)	0.023 (2)	0.039 (3)	0.0004 (19)	0.005 (2)	0.0035 (18)
O3	0.037 (3)	0.018 (2)	0.040 (3)	0.0029 (17)	0.009 (2)	0.0050 (18)
O4	0.039 (3)	0.015 (2)	0.047 (3)	0.0003 (17)	0.010 (2)	0.0061 (18)
O5	0.036 (3)	0.015 (2)	0.043 (3)	0.0040 (17)	0.001 (2)	−0.0009 (18)
O6	0.036 (3)	0.019 (2)	0.041 (3)	0.0026 (17)	0.015 (2)	0.0006 (18)
O7	0.042 (3)	0.019 (2)	0.041 (3)	0.0053 (18)	0.012 (2)	0.0007 (18)
O8	0.037 (3)	0.016 (2)	0.039 (3)	0.0009 (17)	0.005 (2)	−0.0061 (18)
C1	0.035 (4)	0.021 (3)	0.034 (4)	0.003 (2)	−0.006 (3)	−0.001 (2)
C2	0.034 (4)	0.017 (3)	0.040 (4)	−0.001 (2)	−0.001 (3)	0.001 (3)
C3	0.039 (4)	0.022 (3)	0.047 (4)	0.001 (3)	−0.002 (3)	0.006 (3)
C4	0.041 (4)	0.024 (3)	0.061 (5)	0.004 (3)	−0.002 (4)	0.007 (3)
C5	0.036 (4)	0.024 (4)	0.074 (6)	−0.006 (3)	0.000 (4)	0.016 (3)
C6	0.037 (4)	0.027 (4)	0.067 (5)	−0.002 (3)	−0.008 (4)	0.009 (3)
C7	0.037 (4)	0.020 (3)	0.061 (5)	−0.001 (3)	−0.006 (3)	−0.001 (3)
C8	0.035 (4)	0.019 (3)	0.045 (4)	−0.003 (2)	−0.006 (3)	0.003 (3)
C9	0.038 (4)	0.018 (3)	0.036 (4)	0.004 (2)	−0.002 (3)	−0.001 (2)
C10	0.027 (3)	0.029 (3)	0.041 (4)	0.004 (2)	−0.003 (3)	0.006 (3)
C11	0.036 (4)	0.036 (4)	0.042 (4)	0.001 (3)	0.010 (3)	−0.002 (3)
C12	0.051 (5)	0.038 (4)	0.053 (5)	0.004 (3)	0.013 (4)	0.005 (3)
C13	0.038 (4)	0.066 (6)	0.043 (5)	0.015 (4)	0.012 (3)	0.009 (4)

C14	0.045 (5)	0.060 (6)	0.052 (5)	0.019 (4)	0.010 (4)	−0.009 (4)
C15	0.042 (4)	0.035 (4)	0.047 (5)	0.005 (3)	−0.004 (3)	−0.010 (3)
C16	0.026 (3)	0.020 (3)	0.035 (4)	−0.001 (2)	0.000 (3)	0.002 (2)
C17	0.031 (4)	0.025 (3)	0.035 (4)	0.002 (2)	0.001 (3)	0.001 (3)
C18	0.027 (4)	0.034 (4)	0.033 (4)	0.004 (3)	0.003 (3)	0.000 (3)
C19	0.042 (4)	0.033 (4)	0.037 (4)	0.004 (3)	0.005 (3)	0.001 (3)
C20	0.044 (5)	0.055 (5)	0.032 (4)	0.011 (3)	0.006 (3)	−0.002 (3)
C21	0.044 (5)	0.047 (4)	0.037 (4)	0.019 (3)	−0.006 (3)	−0.017 (3)
C22	0.042 (4)	0.034 (4)	0.041 (4)	0.011 (3)	0.006 (3)	−0.009 (3)
C23	0.029 (4)	0.034 (4)	0.032 (4)	0.001 (3)	0.002 (3)	−0.009 (3)
C24	0.029 (4)	0.024 (3)	0.034 (4)	0.002 (2)	0.000 (3)	−0.003 (3)
C25	0.031 (4)	0.024 (3)	0.033 (4)	−0.003 (2)	0.000 (3)	−0.004 (2)
C26	0.032 (4)	0.027 (3)	0.037 (4)	−0.001 (2)	0.007 (3)	−0.002 (3)
C27	0.041 (4)	0.038 (4)	0.035 (4)	0.002 (3)	0.011 (3)	0.004 (3)
C28	0.047 (5)	0.036 (4)	0.040 (4)	−0.007 (3)	0.000 (3)	0.013 (3)
C29	0.050 (5)	0.018 (3)	0.052 (5)	0.001 (3)	0.005 (4)	0.009 (3)
C30	0.038 (4)	0.026 (3)	0.043 (4)	−0.003 (3)	0.003 (3)	−0.001 (3)
Cl1	0.101 (2)	0.0601 (17)	0.132 (3)	0.0052 (15)	−0.013 (2)	0.0314 (17)
Cl2	0.101 (2)	0.102 (2)	0.0723 (18)	−0.0254 (17)	0.0329 (16)	−0.0250 (15)
C31	0.081 (7)	0.075 (7)	0.052 (6)	0.013 (5)	0.016 (5)	0.003 (5)
Cl3	0.0541 (14)	0.111 (2)	0.0536 (14)	−0.0061 (13)	0.0045 (11)	0.0158 (13)
Cl4	0.0532 (12)	0.0462 (11)	0.0570 (13)	0.0032 (9)	0.0097 (10)	−0.0016 (9)
C32	0.068 (6)	0.056 (5)	0.050 (5)	−0.008 (4)	−0.001 (4)	0.017 (4)

Geometric parameters (Å, º)

Os1—O1	1.721 (5)	C14—C15	1.368 (11)
Os1—O2	1.728 (5)	C14—H14	0.9500
Os1—O3	2.008 (5)	C15—H15	0.9500
Os1—O6	2.019 (4)	C16—C24	1.376 (8)
Os1—O7	2.088 (4)	C16—C17	1.411 (9)
Os1—O4	2.094 (4)	C17—C18	1.423 (9)
O3—C1	1.365 (7)	C18—C23	1.389 (9)
O4—C2	1.284 (8)	C18—C19	1.406 (10)
O5—C9	1.371 (7)	C19—C20	1.396 (10)
O5—C8	1.372 (8)	C19—H19	0.9500
O6—C16	1.368 (7)	C20—C21	1.412 (10)
O7—C17	1.289 (7)	C20—H20	0.9500
O8—C24	1.348 (8)	C21—C22	1.362 (11)
O8—C23	1.365 (8)	C21—H21	0.9500
C1—C9	1.377 (9)	C22—C23	1.401 (9)
C1—C2	1.406 (9)	C22—H22	0.9500
C2—C3	1.436 (8)	C24—C25	1.476 (9)
C3—C8	1.376 (10)	C25—C26	1.399 (9)
C3—C4	1.406 (10)	C25—C30	1.410 (9)
C4—C5	1.385 (9)	C26—C27	1.388 (9)
C4—H4	0.9500	C26—H26	0.9500
C5—C6	1.416 (11)	C27—C28	1.377 (9)

C5—H5	0.9500	C27—H27	0.9500
C6—C7	1.377 (11)	C28—C29	1.386 (10)
C6—H6	0.9500	C28—H28	0.9500
C7—C8	1.404 (8)	C29—C30	1.384 (10)
C7—H7	0.9500	C29—H29	0.9500
C9—C10	1.450 (10)	C30—H30	0.9500
C10—C11	1.410 (9)	Cl1—C31	1.753 (9)
C10—C15	1.415 (9)	Cl2—C31	1.762 (10)
C11—C12	1.386 (10)	C31—H31A	0.9900
C11—H11	0.9500	C31—H31B	0.9900
C12—C13	1.383 (11)	Cl3—C32	1.754 (8)
C12—H12	0.9500	Cl4—C32	1.743 (9)
C13—C14	1.396 (11)	C32—H32A	0.9900
C13—H13	0.9500	C32—H32B	0.9900

O1—Os1—O2	170.4 (2)	C13—C14—H14	119.7
O1—Os1—O3	93.5 (2)	C14—C15—C10	120.7 (7)
O2—Os1—O3	92.7 (2)	C14—C15—H15	119.7
O1—Os1—O6	93.8 (2)	C10—C15—H15	119.7
O2—Os1—O6	92.45 (19)	O6—C16—C24	122.9 (6)
O3—Os1—O6	98.57 (17)	O6—C16—C17	116.7 (5)
O1—Os1—O7	86.6 (2)	C24—C16—C17	120.3 (6)
O2—Os1—O7	87.3 (2)	O7—C17—C16	119.7 (6)
O3—Os1—O7	179.49 (17)	O7—C17—C18	121.3 (6)
O6—Os1—O7	80.92 (17)	C16—C17—C18	119.0 (6)
O1—Os1—O4	87.8 (2)	C23—C18—C19	118.4 (6)
O2—Os1—O4	86.08 (19)	C23—C18—C17	117.5 (6)
O3—Os1—O4	80.72 (18)	C19—C18—C17	124.1 (6)
O6—Os1—O4	178.33 (18)	C20—C19—C18	120.0 (7)
O7—Os1—O4	99.78 (18)	C20—C19—H19	120.0
C1—O3—Os1	111.5 (4)	C18—C19—H19	120.0
C2—O4—Os1	111.0 (4)	C19—C20—C21	119.4 (7)
C9—O5—C8	121.3 (5)	C19—C20—H20	120.3
C16—O6—Os1	111.7 (4)	C21—C20—H20	120.3
C17—O7—Os1	110.9 (4)	C22—C21—C20	121.3 (7)
C24—O8—C23	121.7 (5)	C22—C21—H21	119.3
O3—C1—C9	122.1 (6)	C20—C21—H21	119.3
O3—C1—C2	117.5 (6)	C21—C22—C23	118.6 (7)
C9—C1—C2	120.3 (6)	C21—C22—H22	120.7
O4—C2—C1	118.8 (5)	C23—C22—H22	120.7
O4—C2—C3	121.5 (6)	O8—C23—C18	121.5 (6)
C1—C2—C3	119.7 (6)	O8—C23—C22	116.3 (6)
C8—C3—C4	119.3 (6)	C18—C23—C22	122.2 (7)
C8—C3—C2	117.3 (7)	O8—C24—C16	119.8 (6)
C4—C3—C2	123.4 (7)	O8—C24—C25	111.2 (5)
C5—C4—C3	119.5 (7)	C16—C24—C25	129.0 (6)
C5—C4—H4	120.2	C26—C25—C30	118.7 (6)
C3—C4—H4	120.2	C26—C25—C24	121.9 (6)

C4—C5—C6	119.6 (7)	C30—C25—C24	119.4 (6)
C4—C5—H5	120.2	C27—C26—C25	120.0 (6)
C6—C5—H5	120.2	C27—C26—H26	120.0
C7—C6—C5	121.5 (7)	C25—C26—H26	120.0
C7—C6—H6	119.2	C28—C27—C26	121.3 (7)
C5—C6—H6	119.2	C28—C27—H27	119.4
C6—C7—C8	117.3 (7)	C26—C27—H27	119.4
C6—C7—H7	121.3	C27—C28—C29	119.1 (6)
C8—C7—H7	121.3	C27—C28—H28	120.4
O5—C8—C3	121.9 (6)	C29—C28—H28	120.4
O5—C8—C7	115.5 (6)	C30—C29—C28	121.0 (6)
C3—C8—C7	122.6 (7)	C30—C29—H29	119.5
O5—C9—C1	119.5 (6)	C28—C29—H29	119.5
O5—C9—C10	112.0 (5)	C29—C30—C25	119.9 (7)
C1—C9—C10	128.5 (6)	C29—C30—H30	120.0
C11—C10—C15	118.0 (7)	C25—C30—H30	120.0
C11—C10—C9	120.9 (6)	Cl1—C31—Cl2	112.7 (5)
C15—C10—C9	121.1 (6)	Cl1—C31—H31A	109.0
C12—C11—C10	120.6 (7)	Cl2—C31—H31A	109.0
C12—C11—H11	119.7	Cl1—C31—H31B	109.0
C10—C11—H11	119.7	Cl2—C31—H31B	109.0
C13—C12—C11	120.2 (8)	H31A—C31—H31B	107.8
C13—C12—H12	119.9	Cl4—C32—Cl3	113.3 (4)
C11—C12—H12	119.9	Cl4—C32—H32A	108.9
C12—C13—C14	119.9 (8)	Cl3—C32—H32A	108.9
C12—C13—H13	120.0	Cl4—C32—H32B	108.9
C14—C13—H13	120.0	Cl3—C32—H32B	108.9
C15—C14—C13	120.6 (7)	H32A—C32—H32B	107.7
C15—C14—H14	119.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O3	0.95	2.24	2.876 (9)	124
C26—H26···O6	0.95	2.24	2.910 (8)	127
C30—H30···O2^{i}	0.95	2.49	3.393 (9)	158
C31—H31*A*···O4	0.99	2.56	3.180 (11)	121
C31—H31*B*···O7	0.99	2.47	3.221 (11)	133
C32—H32*A*···O2	0.99	2.62	3.420 (10)	138
C32—H32*A*···O6	0.99	2.55	3.489 (11)	158

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+1/2.