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(2,2′-Bipyrazine-$\kappa^2N^1,N^{1'}$)[1,2-bis(diphenylphosphanyl)methane-κP]tricarbonylrhenium(I) trifluoromethanesulfonate monohydrate

D. Paul Rillema,* Venugopal Komreddy, Nilmini K. Senaratne and David M. Eichhorn

Department of Chemistry, Wichita State University, Wichita, KS 67260, USA. *Correspondence e-mail: paul.rillema@wichita.edu

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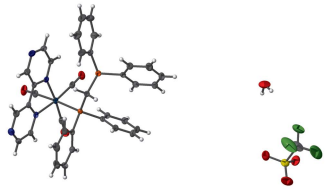
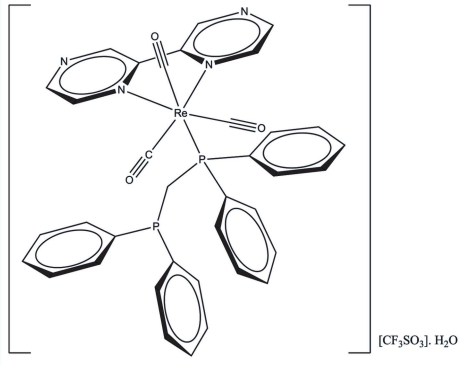
The title compound, $[Re(C_8H_6N_4)(C_{25}H_{22}P_2)(CO)_3]CF_3SO_3\cdot H_2O$, crystallizes with one $[Re(C_8N_4H_6)\{P(Ph)_2CH_2P(Ph)_2\}(CO)_3]^+$ cation, where Ph is a phenyl group, one $CF_3SO_3^-$ anion and one water molecule of hydration. The three C atoms of the *facial* oriented carbonyl groups, two N atoms from the bipyrazine ligand and one P atom from the (bis)diphenylphosphanylmethane ligand define a distorted octahedral coordination environment about the central Re^I atom. The $Re-C_{carbonyl}$ bond length *trans* to the P atom is longer than the the two $Re-C_{carbonyl}$ bond lengths in the plane with the bipyrazine ligand. Hydrogen-bonding interactions between the solvent water molecule and the cation, as well as weak $C-H\cdots O$ interactions, consolidate a three-dimensional network structure.

3D view



Chemical scheme



Structure description

We are interested in preparing Re^I complexes containing bidentate diamine ligands due to their excited state properties (Kirgan *et al.*, 2007) and determining their structures as a guide to design better photochomophores. The molecular structures of Re(diamine)-$(CO)_3X$, where X = Cl, OH_2, pyridine (py), 2,6-dimethylisocyanide (CN_x), bis(diphenylphosphinomethane (dppm) or bis(diphenylphosphinoethane (dppe), and diamine = 2,2′-bipyrazine (bpz), 2,2′-bipyridine (bpy) or 1,10-phenanthroline (phen), reveal the CO ligands lie on the face of an octahedron, the diamine ligand and two CO ligands lie in a plane and one CO ligand and the ligating atom of X are *trans* to one another (Kirgan *et al.*, 2007; Rillema *et al.*, 2007; Villegas *et al.*, 2005; Stoyanov *et al.*, 2005; Yamamoto *et al.*, 2008), as shown for the cation of the title compound (Fig. 1).

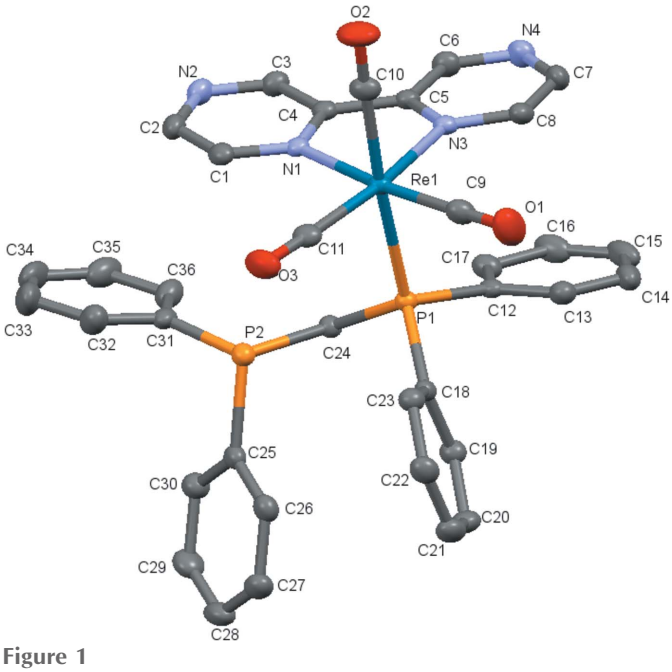


Figure 1
The molecular structure of the complex cation of the title compound with atom labels and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

The cation of the title compound is best described as a distorted octahedron. The six bond lengths for the atoms bonded to Re^{I} differ markedly and are the shortest for the C atoms of the three carbonyl groups, followed by the two N atoms of the bidentate bipyrazine ligand and the P atom of the bis(diphenylphosphinomethane) ligand (Table 1). The bond angles of the coordinating atoms *trans* to one another are less than 180°, *viz.* C9—Re1—N1 = 173.94 (8)°, C10—Re1—P1 = 175.79 (7)° and C11—Re1—N3 = 170.78 (8)°. The bond angles about P2 and the coordinating P1 atom also differ from the ideal tetrahedral angle (Table 1).

Table 1
Selected geometric parameters (Å, °).

C9—Re1	1.919 (2)	N1—Re1	2.1673 (17)
C10—Re1	1.973 (2)	N3—Re1	2.1665 (17)
C11—Re1	1.935 (2)	P1—Re1	2.4790 (6)
C12—P1—C18	102.48 (9)	C24—P1—Re1	114.39 (7)
C12—P1—C24	105.39 (9)	C25—P2—C24	102.68 (9)
C12—P1—Re1	110.43 (7)	C31—P2—C24	103.85 (10)
C18—P1—C24	103.53 (9)	C31—P2—C25	99.74 (10)
C18—P1—Re1	119.17 (7)		

The two Re—N bond lengths in the title structure are similar to those of the following molecular entities: $Re(bpz)(CO)_3Cl$ [2.150 (5), 2.151 (5) Å] and $Re(bpz)(CO)_3(py)^+$ [2.162 (2), 2.161 (2) Å; Kirgan *et al.*, 2007]; $Re(bpz)(CO)_3(OH_2)^+$ [2.167 (2) Å; Rillema *et al.*, 2007]; $Re(phen)(CO)_3(CN_x)$ [2.196 (3), 2.203 (3) Å; Villegas *et al.*, 2005], $Re(bpy)(CO)_3(CN_x)$ [2.173 (3), 2.169 (3) Å; Stoyanov *et al.*, 2005] and $Re(bpy)(CO)_3(dppe)Re(bpy)(CO)_2(dppe)Re(bpy)(CO)_3^{3+}$ [2.133 (10), 2.149 (12) Å; Yamamoto *et al.*, 2008]. The Re1—P1 bond length in the title structure compares favorably with the one for $Re(bpy)(CO)_3(dppe)Re(bpy)(CO)_2(dppe)Re(bpy)(CO)_3^{3+}$ [2.472 (4) Å] and for Re—Cl [2.484 (1) Å] in $Re(bpz)(CO)_3Cl$, but is longer than the bond length for Re—N(py) [2.203 (3) Å] in $Re(bpz)(CO)_3(py)^+$, Re—O [2.143 (3) Å] in $Re(bpz)(CO)_3(OH_2)^+$, Re—C [2.063 (4) Å] in $Re(phen)(CO)_3(CN_x)$ and Re—C [2.074 (4) Å] in $Re(bpy)(CO)_3(CN_x)$.

The crystal packing is shown in Fig. 2. Apart from Couloumbic forces between complex cations and trifluoro-

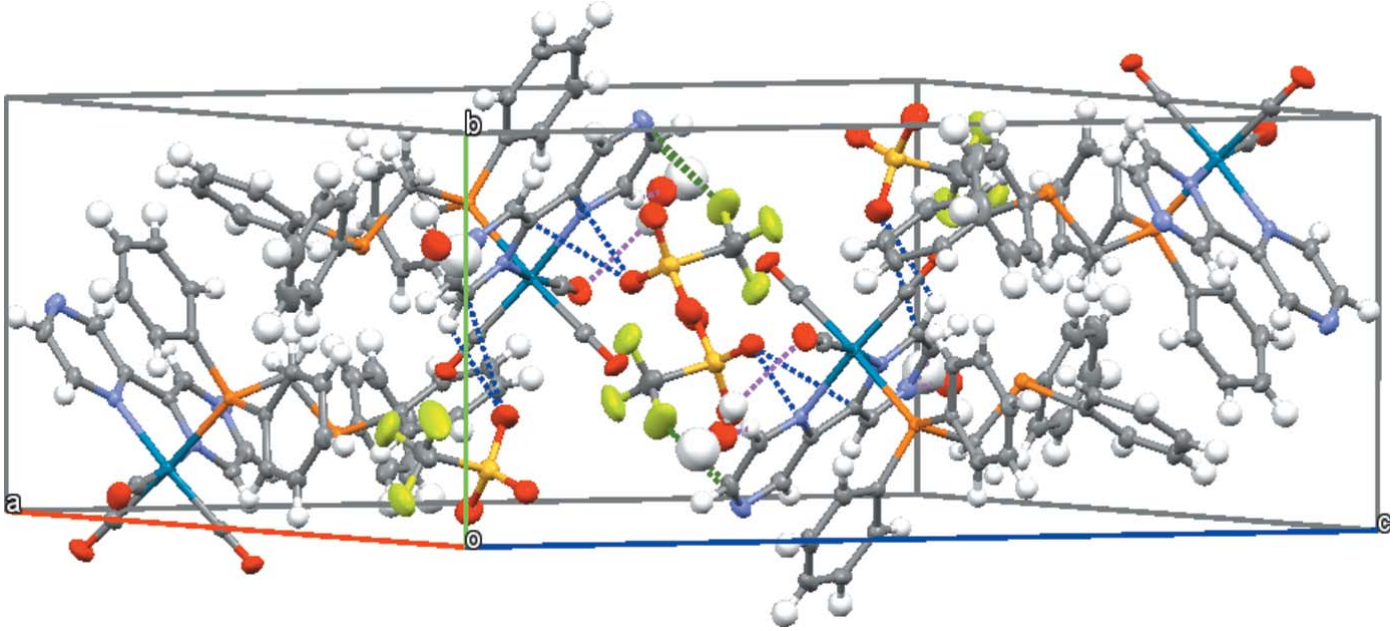


Figure 2
Packing diagram showing C···O (blue) and N···F (green) van der Waals interactions between the complex cation and the trifluoromethanesulfonate anion and C—O···H (pink) hydrogen-bonding interactions between the carbonyl groups and the water molecules.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O4^{i}	0.95	2.47	3.281 (3)	143
C6—H6···O4^{i}	0.95	2.62	3.468 (3)	148
C8—H8···O101^{ii}	0.95	2.33	3.226 (3)	156
C17—H17···O6^{i}	0.95	2.31	3.180 (3)	152
O101—H10A···N2^{iii}	0.94 (5)	2.20 (5)	2.983 (3)	141 (4)
O101—H10A···N4^{iv}	0.94 (5)	2.44 (5)	3.126 (3)	131 (4)
O101—H10B···O1^{v}	0.84 (3)	2.47 (3)	3.103 (3)	133 (3)

Symmetry codes: (i) $x+1, y, z$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x-1, y, z$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

methanesulfonate anions, C···O and N···F van der Waals interactions between the cations and the anions are present. Also O—H···O≡C and O—H···N hydrogen-bonding interactions between the water molecules and a carbonyl group and the N atoms of the diamine, respectively, are present. Weak C—H···O hydrogen bonds are also observed (Table 2).

Synthesis and crystallization

To a 100 ml round-bottom flask equipped with a stir bar, 0.20 g (0.432 mmol) of Re(bpz)$(CO)_3$Cl (Kirgan *et al.*, 2007), where bpz is 2,2′-bipyrazine, and 0.11 g (0.432 mmol) $AgCF_3SO_3$ were added along with 25 ml of absolute ethanol. This solution was allowed to reflux for 12 h under nitrogen gas. After refluxing for roughly 2 h, a gray AgCl precipitate was present. At the end of the reflux procedure, the round-bottom flask was removed from the condenser and the solution was vacuum-filtered to remove the silver chloride. The yellow filtrate was transferred to a 100 ml round-bottom flask with a stir bar and 0.166 g (0.432 mmol) of bis(diphenlyphosphanyl)methane. This solution was allowed to reflux overnight under nitrogen gas. The solution remained yellow during reflux. Then the solution was filtered to remove insoluble impurities and concentrated under rotary evaporation yielding 0.274 g (67%) of the desired title compound. A small portion of the solid was then recrystallized by slow evaporation from ethanol solution.

Refinement

Crystal data, data collection and stucture refinement details are summarized in Table 3. Reflections (002), ($\bar{2}$02), ($\bar{1}$11), (110), (200), (101), (011) and (111) were obstructed by the beam stop and were omitted from the refinement.

Acknowledgements

We are grateful for support from the National Science Foundation (EPSCoR), the Wichita State University Office of Research and the Department of Energy.

Table 3
Experimental details.

Crystal data	
Chemical formula	[Re($C_8H_6N_4$)($C_{25}H_{22}P_2$)($CO)_3$]-CF_3O_3S·H_2O
M_r	979.85
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	18.188 (4), 9.934 (2), 22.887 (5)
β (°)	110.062 (10)
V (Å^3)	3884.4 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.33
Crystal size (mm)	0.28 × 0.16 × 0.10
Data collection	
Diffractometer	Bruker SMART CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.854, 0.935
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	77073, 7628, 6856
R_{int}	0.033
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.016, 0.039, 1.04
No. of reflections	7628
No. of parameters	504
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.62, −0.30

Computer programs: *SMART* and *SAINT* (Bruker, 2004), *SIR2004* (Burla *et al.*, 2007), *SHELXL2013* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2017). **2**, x170935 [https://doi.org/10.1107/S2414314617009350X]

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Crystal data

$[Re(C_8H_6N_4)(C_{25}H_{22}P_2)(CO)_3]CF_3O_3S\cdot H_2O$
M_r = 979.85
Monoclinic, $P2_1/n$
a = 18.188 (4) Å
b = 9.934 (2) Å
c = 22.887 (5) Å
β = 110.062 (10)°
V = 3884.4 (14) Å^3
Z = 4
$F(000)$ = 1936
D_x = 1.675 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9722 reflections
θ = 2.7–27.1°
μ = 3.33 mm^{-1}
T = 150 K
Prism, yellow
0.28 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.854, T_{max} = 0.935
77073 measured reflections
7628 independent reflections
6856 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.0°, θ_{min} = 2.8°
h = −22→22
k = −12→12
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.039
S = 1.04
7628 reflections
504 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0175P)^2 + 3.0353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.12890 (13)	0.0732 (2)	0.31293 (10)	0.0215 (4)
H1	1.1000	−0.0028	0.3179	0.026*
C2	1.19548 (13)	0.1117 (2)	0.36087 (10)	0.0254 (5)
H2	1.2110	0.0609	0.3983	0.030*
C3	1.21558 (12)	0.2839 (2)	0.30326 (10)	0.0234 (5)
H3	1.2455	0.3589	0.2986	0.028*
C4	1.14895 (11)	0.2480 (2)	0.25373 (9)	0.0178 (4)
C5	1.12322 (11)	0.3146 (2)	0.19303 (9)	0.0180 (4)
C6	1.16036 (13)	0.4264 (2)	0.17868 (10)	0.0243 (5)
H6	1.2044	0.4636	0.2102	0.029*
C7	1.07519 (13)	0.4256 (2)	0.08022 (11)	0.0273 (5)
H7	1.0571	0.4624	0.0394	0.033*
C8	1.03644 (12)	0.3156 (2)	0.09239 (10)	0.0229 (5)
H8	0.9928	0.2789	0.0603	0.027*
C9	0.91755 (13)	0.0653 (2)	0.10254 (10)	0.0242 (5)
C10	1.06703 (13)	−0.0344 (2)	0.15151 (11)	0.0240 (5)
C11	0.96474 (12)	−0.0484 (2)	0.21795 (10)	0.0218 (5)
C12	0.92262 (12)	0.4244 (2)	0.16444 (9)	0.0182 (4)
C13	0.87371 (13)	0.4307 (2)	0.10223 (10)	0.0242 (5)
H13	0.8390	0.3583	0.0846	0.029*
C14	0.87538 (15)	0.5415 (3)	0.06605 (11)	0.0327 (6)
H14	0.8408	0.5465	0.0242	0.039*
C15	0.92772 (16)	0.6454 (3)	0.09115 (13)	0.0364 (6)
H15	0.9297	0.7209	0.0663	0.044*
C16	0.97686 (15)	0.6387 (2)	0.15233 (13)	0.0332 (6)
H16	1.0131	0.7094	0.1692	0.040*
C17	0.97388 (13)	0.5300 (2)	0.18945 (11)	0.0242 (5)
H17	1.0068	0.5277	0.2319	0.029*
C18	0.82138 (11)	0.2393 (2)	0.19442 (9)	0.0169 (4)
C19	0.77494 (12)	0.3460 (2)	0.20091 (10)	0.0212 (4)
H19	0.7971	0.4333	0.2105	0.025*
C20	0.69672 (13)	0.3263 (2)	0.19348 (11)	0.0261 (5)
H20	0.6657	0.3996	0.1982	0.031*
C21	0.66414 (13)	0.1990 (2)	0.17913 (10)	0.0267 (5)
H21	0.6107	0.1849	0.1742	0.032*
C22	0.70911 (13)	0.0929 (2)	0.17202 (10)	0.0246 (5)
H22	0.6865	0.0061	0.1619	0.029*
C23	0.78795 (12)	0.1126 (2)	0.17962 (10)	0.0209 (4)
H23	0.8187	0.0392	0.1747	0.025*
C24	0.96408 (12)	0.3199 (2)	0.29061 (9)	0.0180 (4)
H24A	0.9341	0.3975	0.2978	0.022*
H24B	1.0196	0.3472	0.3019	0.022*
C25	0.88188 (12)	0.2330 (2)	0.37257 (9)	0.0201 (4)
C26	0.80430 (13)	0.1956 (2)	0.34234 (10)	0.0239 (5)
H26	0.7908	0.1450	0.3049	0.029*

C27	0.74628 (13)	0.2316 (2)	0.36626 (11)	0.0288 (5)
H27	0.6934	0.2065	0.3449	0.035*
C28	0.76547 (14)	0.3035 (3)	0.42084 (11)	0.0322 (5)
H28	0.7261	0.3270	0.4375	0.039*
C29	0.84233 (15)	0.3414 (3)	0.45136 (11)	0.0322 (5)
H29	0.8554	0.3916	0.4889	0.039*
C30	0.90042 (13)	0.3066 (2)	0.42756 (10)	0.0264 (5)
H30	0.9530	0.3332	0.4489	0.032*
C31	1.04395 (12)	0.1870 (2)	0.40903 (10)	0.0235 (5)
C32	1.05876 (15)	0.0743 (3)	0.44770 (12)	0.0359 (6)
H32	1.0264	−0.0031	0.4354	0.043*
C33	1.12085 (16)	0.0746 (3)	0.50434 (13)	0.0488 (8)
H33	1.1305	−0.0020	0.5307	0.059*
C34	1.16785 (15)	0.1860 (3)	0.52169 (12)	0.0451 (7)
H34	1.2092	0.1871	0.5607	0.054*
C35	1.15567 (14)	0.2962 (3)	0.48310 (12)	0.0391 (6)
H35	1.1898	0.3714	0.4949	0.047*
C36	1.09337 (13)	0.2978 (3)	0.42682 (11)	0.0300 (5)
H36	1.0846	0.3746	0.4006	0.036*
C37	0.2103 (2)	0.6951 (3)	0.39864 (13)	0.0507 (8)
F101	0.23127 (12)	0.58796 (19)	0.43486 (8)	0.0615 (5)
F102	0.14850 (15)	0.7505 (2)	0.40888 (10)	0.0886 (7)
F103	0.26801 (15)	0.7831 (2)	0.41738 (9)	0.0949 (8)
H10A	0.352 (3)	0.188 (5)	0.432 (2)	0.120 (17)*
H10B	0.3570 (19)	0.290 (3)	0.4732 (15)	0.052 (11)*
N1	1.10464 (9)	0.14245 (17)	0.25918 (8)	0.0168 (3)
N2	1.23885 (10)	0.21675 (19)	0.35690 (8)	0.0253 (4)
N3	1.05997 (9)	0.26006 (17)	0.14938 (8)	0.0175 (4)
N4	1.13650 (11)	0.4827 (2)	0.12260 (9)	0.0287 (4)
O1	0.86691 (10)	0.0460 (2)	0.05652 (8)	0.0394 (4)
O2	1.10190 (10)	−0.10941 (17)	0.13425 (9)	0.0388 (4)
O3	0.94393 (10)	−0.13437 (16)	0.24188 (8)	0.0326 (4)
O4	0.25922 (11)	0.60542 (18)	0.31277 (9)	0.0399 (4)
O5	0.15883 (14)	0.7761 (2)	0.28536 (9)	0.0584 (6)
O6	0.12803 (10)	0.5481 (2)	0.30898 (9)	0.0434 (5)
O101	0.37966 (13)	0.2167 (3)	0.47235 (9)	0.0523 (6)
P1	0.92512 (3)	0.27010 (5)	0.20823 (2)	0.01519 (10)
P2	0.95581 (3)	0.17415 (5)	0.33975 (2)	0.01939 (11)
Re1	1.00297 (2)	0.09495 (2)	0.17872 (2)	0.01594 (3)
S101	0.18615 (4)	0.65191 (6)	0.31689 (3)	0.03235 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0222 (11)	0.0190 (11)	0.0259 (11)	0.0050 (9)	0.0117 (9)	0.0026 (9)
C2	0.0233 (11)	0.0310 (13)	0.0212 (11)	0.0091 (10)	0.0069 (9)	0.0054 (9)
C3	0.0195 (11)	0.0236 (11)	0.0264 (11)	0.0000 (9)	0.0067 (9)	−0.0013 (9)
C4	0.0163 (10)	0.0171 (10)	0.0216 (10)	0.0013 (8)	0.0088 (8)	−0.0022 (8)

C5	0.0151 (10)	0.0189 (10)	0.0220 (10)	0.0010 (8)	0.0089 (8)	−0.0006 (8)
C6	0.0207 (11)	0.0240 (12)	0.0288 (12)	−0.0033 (9)	0.0092 (9)	−0.0004 (9)
C7	0.0245 (12)	0.0353 (13)	0.0245 (11)	0.0045 (10)	0.0115 (10)	0.0100 (10)
C8	0.0180 (10)	0.0322 (12)	0.0198 (11)	0.0028 (9)	0.0083 (9)	0.0008 (9)
C9	0.0233 (11)	0.0255 (12)	0.0264 (12)	−0.0055 (9)	0.0118 (10)	−0.0043 (9)
C10	0.0216 (11)	0.0215 (11)	0.0316 (12)	−0.0042 (9)	0.0127 (10)	−0.0048 (9)
C11	0.0216 (11)	0.0180 (10)	0.0277 (11)	0.0003 (9)	0.0112 (9)	−0.0046 (9)
C12	0.0176 (10)	0.0175 (10)	0.0229 (10)	0.0030 (8)	0.0112 (8)	0.0012 (8)
C13	0.0204 (11)	0.0279 (12)	0.0259 (11)	0.0017 (9)	0.0101 (9)	0.0020 (9)
C14	0.0349 (14)	0.0400 (14)	0.0285 (12)	0.0133 (12)	0.0177 (11)	0.0127 (11)
C15	0.0490 (16)	0.0243 (12)	0.0506 (16)	0.0100 (12)	0.0359 (14)	0.0125 (12)
C16	0.0427 (15)	0.0183 (11)	0.0490 (16)	−0.0030 (11)	0.0290 (13)	−0.0013 (11)
C17	0.0267 (12)	0.0210 (11)	0.0286 (12)	−0.0025 (9)	0.0144 (10)	−0.0027 (9)
C18	0.0155 (10)	0.0210 (11)	0.0149 (9)	−0.0007 (8)	0.0060 (8)	0.0002 (8)
C19	0.0218 (11)	0.0192 (11)	0.0236 (11)	−0.0004 (9)	0.0092 (9)	−0.0016 (9)
C20	0.0203 (11)	0.0286 (12)	0.0317 (12)	0.0041 (9)	0.0118 (10)	−0.0006 (10)
C21	0.0162 (10)	0.0358 (13)	0.0282 (12)	−0.0028 (10)	0.0079 (9)	−0.0016 (10)
C22	0.0220 (11)	0.0263 (12)	0.0258 (11)	−0.0075 (9)	0.0088 (9)	−0.0030 (9)
C23	0.0198 (10)	0.0215 (11)	0.0221 (11)	−0.0004 (9)	0.0083 (9)	−0.0022 (9)
C24	0.0181 (10)	0.0190 (10)	0.0172 (10)	0.0000 (8)	0.0066 (8)	−0.0014 (8)
C25	0.0220 (11)	0.0195 (11)	0.0205 (10)	0.0032 (9)	0.0096 (9)	0.0044 (8)
C26	0.0268 (12)	0.0231 (11)	0.0230 (11)	−0.0016 (9)	0.0103 (9)	0.0025 (9)
C27	0.0223 (11)	0.0331 (13)	0.0337 (13)	0.0003 (10)	0.0130 (10)	0.0065 (10)
C28	0.0324 (13)	0.0343 (13)	0.0380 (14)	0.0073 (11)	0.0226 (11)	0.0059 (11)
C29	0.0400 (14)	0.0332 (13)	0.0285 (13)	0.0046 (11)	0.0182 (11)	−0.0040 (10)
C30	0.0253 (12)	0.0302 (13)	0.0248 (12)	0.0019 (10)	0.0100 (10)	−0.0010 (9)
C31	0.0193 (11)	0.0319 (12)	0.0207 (11)	0.0081 (9)	0.0088 (9)	0.0019 (9)
C32	0.0283 (13)	0.0432 (15)	0.0365 (14)	0.0050 (11)	0.0115 (11)	0.0130 (12)
C33	0.0367 (15)	0.073 (2)	0.0358 (15)	0.0177 (15)	0.0112 (12)	0.0255 (15)
C34	0.0250 (13)	0.078 (2)	0.0263 (13)	0.0136 (14)	0.0017 (11)	0.0043 (14)
C35	0.0262 (13)	0.0538 (17)	0.0323 (13)	0.0042 (12)	0.0036 (11)	−0.0123 (13)
C36	0.0271 (12)	0.0349 (13)	0.0246 (12)	0.0070 (10)	0.0044 (10)	−0.0022 (10)
C37	0.075 (2)	0.0435 (17)	0.0357 (15)	−0.0214 (16)	0.0211 (15)	−0.0041 (13)
F101	0.0796 (13)	0.0678 (12)	0.0340 (9)	−0.0106 (10)	0.0156 (9)	0.0112 (8)
F102	0.139 (2)	0.0839 (16)	0.0696 (14)	0.0220 (15)	0.0697 (15)	−0.0081 (12)
F103	0.145 (2)	0.0904 (16)	0.0482 (11)	−0.0793 (16)	0.0321 (13)	−0.0318 (11)
N1	0.0154 (8)	0.0159 (8)	0.0204 (9)	0.0030 (7)	0.0078 (7)	−0.0006 (7)
N2	0.0202 (9)	0.0296 (10)	0.0231 (10)	0.0032 (8)	0.0036 (8)	−0.0004 (8)
N3	0.0153 (8)	0.0193 (9)	0.0198 (9)	0.0006 (7)	0.0085 (7)	−0.0021 (7)
N4	0.0262 (10)	0.0304 (11)	0.0321 (11)	−0.0017 (9)	0.0132 (9)	0.0070 (9)
O1	0.0352 (10)	0.0501 (11)	0.0273 (9)	−0.0158 (9)	0.0035 (8)	−0.0060 (8)
O2	0.0359 (10)	0.0321 (10)	0.0574 (12)	0.0010 (8)	0.0276 (9)	−0.0134 (8)
O3	0.0384 (10)	0.0209 (8)	0.0467 (10)	−0.0036 (7)	0.0250 (9)	0.0014 (8)
O4	0.0393 (10)	0.0355 (10)	0.0508 (11)	−0.0061 (8)	0.0230 (9)	−0.0027 (8)
O5	0.0927 (17)	0.0408 (12)	0.0460 (12)	0.0211 (12)	0.0291 (12)	0.0105 (10)
O6	0.0305 (10)	0.0497 (11)	0.0463 (11)	−0.0080 (9)	0.0084 (8)	−0.0115 (9)
O101	0.0559 (14)	0.0585 (15)	0.0277 (11)	0.0001 (12)	−0.0048 (9)	0.0014 (10)
P1	0.0144 (2)	0.0153 (2)	0.0165 (2)	−0.0009 (2)	0.0060 (2)	−0.0011 (2)

P2	0.0203 (3)	0.0196 (3)	0.0189 (3)	0.0015 (2)	0.0077 (2)	0.0002 (2)
Re1	0.01489 (5)	0.01501 (5)	0.01932 (5)	−0.00169 (3)	0.00768 (3)	−0.00305 (3)
S101	0.0415 (4)	0.0285 (3)	0.0266 (3)	−0.0018 (3)	0.0111 (3)	−0.0030 (2)

Geometric parameters (Å, º)

C1—H1	0.9500	C21—C22	1.377 (3)
C1—C2	1.380 (3)	C22—H22	0.9500
C1—N1	1.345 (3)	C22—C23	1.398 (3)
C2—H2	0.9500	C23—H23	0.9500
C2—N2	1.330 (3)	C24—H24A	0.9900
C3—H3	0.9500	C24—H24B	0.9900
C3—C4	1.392 (3)	C24—P1	1.840 (2)
C3—N2	1.332 (3)	C24—P2	1.871 (2)
C4—C5	1.463 (3)	C25—C26	1.392 (3)
C4—N1	1.354 (3)	C25—C30	1.394 (3)
C5—C6	1.396 (3)	C25—P2	1.846 (2)
C5—N3	1.351 (3)	C26—H26	0.9500
C6—H6	0.9500	C26—C27	1.392 (3)
C6—N4	1.329 (3)	C27—H27	0.9500
C7—H7	0.9500	C27—C28	1.376 (3)
C7—C8	1.379 (3)	C28—H28	0.9500
C7—N4	1.328 (3)	C28—C29	1.383 (4)
C8—H8	0.9500	C29—H29	0.9500
C8—N3	1.344 (3)	C29—C30	1.388 (3)
C9—O1	1.153 (3)	C30—H30	0.9500
C9—Re1	1.919 (2)	C31—C32	1.395 (3)
C10—O2	1.134 (3)	C31—C36	1.391 (3)
C10—Re1	1.973 (2)	C31—P2	1.832 (2)
C11—O3	1.147 (3)	C32—H32	0.9500
C11—Re1	1.935 (2)	C32—C33	1.397 (4)
C12—C13	1.398 (3)	C33—H33	0.9500
C12—C17	1.390 (3)	C33—C34	1.371 (4)
C12—P1	1.823 (2)	C34—H34	0.9500
C13—H13	0.9500	C34—C35	1.376 (4)
C13—C14	1.384 (3)	C35—H35	0.9500
C14—H14	0.9500	C35—C36	1.394 (3)
C14—C15	1.387 (4)	C36—H36	0.9500
C15—H15	0.9500	C37—F101	1.322 (3)
C15—C16	1.380 (4)	C37—F102	1.342 (4)
C16—H16	0.9500	C37—F103	1.320 (3)
C16—C17	1.387 (3)	C37—S101	1.820 (3)
C17—H17	0.9500	N1—Re1	2.1673 (17)
C18—C19	1.395 (3)	N3—Re1	2.1665 (17)
C18—C23	1.388 (3)	O4—S101	1.4402 (19)
C18—P1	1.829 (2)	O5—S101	1.429 (2)
C19—H19	0.9500	O6—S101	1.443 (2)
C19—C20	1.387 (3)	O101—H10A	0.94 (5)

C20—H20	0.9500	O101—H10B	0.84 (3)
C20—C21	1.388 (3)	P1—Re1	2.4790 (6)
C21—H21	0.9500		

C2—C1—H1	119.7	C26—C27—H27	120.0
N1—C1—H1	119.7	C28—C27—C26	120.0 (2)
N1—C1—C2	120.6 (2)	C28—C27—H27	120.0
C1—C2—H2	118.6	C27—C28—H28	120.1
N2—C2—C1	122.8 (2)	C27—C28—C29	119.8 (2)
N2—C2—H2	118.6	C29—C28—H28	120.1
C4—C3—H3	118.8	C28—C29—H29	119.8
N2—C3—H3	118.8	C28—C29—C30	120.5 (2)
N2—C3—C4	122.3 (2)	C30—C29—H29	119.8
C3—C4—C5	124.25 (19)	C25—C30—H30	119.9
N1—C4—C3	120.17 (19)	C29—C30—C25	120.3 (2)
N1—C4—C5	115.54 (18)	C29—C30—H30	119.9
C6—C5—C4	124.06 (19)	C32—C31—P2	114.47 (18)
N3—C5—C4	115.75 (18)	C36—C31—C32	119.0 (2)
N3—C5—C6	120.18 (19)	C36—C31—P2	126.46 (17)
C5—C6—H6	118.7	C31—C32—H32	119.8
N4—C6—C5	122.5 (2)	C31—C32—C33	120.4 (3)
N4—C6—H6	118.7	C33—C32—H32	119.8
C8—C7—H7	118.3	C32—C33—H33	120.1
N4—C7—H7	118.3	C34—C33—C32	119.7 (3)
N4—C7—C8	123.4 (2)	C34—C33—H33	120.1
C7—C8—H8	119.8	C33—C34—H34	119.7
N3—C8—C7	120.4 (2)	C33—C34—C35	120.6 (2)
N3—C8—H8	119.8	C35—C34—H34	119.7
O1—C9—Re1	178.9 (2)	C34—C35—H35	119.9
O2—C10—Re1	177.9 (2)	C34—C35—C36	120.2 (3)
O3—C11—Re1	178.3 (2)	C36—C35—H35	119.9
C13—C12—P1	119.01 (16)	C31—C36—C35	120.0 (2)
C17—C12—C13	119.2 (2)	C31—C36—H36	120.0
C17—C12—P1	121.43 (16)	C35—C36—H36	120.0
C12—C13—H13	119.7	F101—C37—F102	107.4 (2)
C14—C13—C12	120.5 (2)	F101—C37—S101	111.8 (2)
C14—C13—H13	119.7	F102—C37—S101	110.8 (2)
C13—C14—H14	120.1	F103—C37—F101	107.7 (3)
C13—C14—C15	119.8 (2)	F103—C37—F102	106.9 (3)
C15—C14—H14	120.1	F103—C37—S101	112.0 (2)
C14—C15—H15	120.1	C1—N1—C4	117.48 (18)
C16—C15—C14	119.9 (2)	C1—N1—Re1	125.63 (14)
C16—C15—H15	120.1	C4—N1—Re1	116.84 (13)
C15—C16—H16	119.6	C2—N2—C3	116.57 (19)
C15—C16—C17	120.7 (2)	C5—N3—Re1	116.80 (13)
C17—C16—H16	119.6	C8—N3—C5	117.36 (18)
C12—C17—H17	120.1	C8—N3—Re1	125.79 (14)
C16—C17—C12	119.8 (2)	C7—N4—C6	116.1 (2)

C16—C17—H17	120.1	H10A—O101—H10B	101 (3)
C19—C18—P1	118.74 (16)	C12—P1—C18	102.48 (9)
C23—C18—C19	118.98 (19)	C12—P1—C24	105.39 (9)
C23—C18—P1	122.25 (16)	C12—P1—Re1	110.43 (7)
C18—C19—H19	119.6	C18—P1—C24	103.53 (9)
C20—C19—C18	120.8 (2)	C18—P1—Re1	119.17 (7)
C20—C19—H19	119.6	C24—P1—Re1	114.39 (7)
C19—C20—H20	120.2	C25—P2—C24	102.68 (9)
C19—C20—C21	119.6 (2)	C31—P2—C24	103.85 (10)
C21—C20—H20	120.2	C31—P2—C25	99.74 (10)
C20—C21—H21	119.9	C9—Re1—C10	89.71 (9)
C22—C21—C20	120.2 (2)	C9—Re1—C11	90.04 (9)
C22—C21—H21	119.9	C9—Re1—N1	173.94 (8)
C21—C22—H22	119.9	C9—Re1—N3	99.16 (8)
C21—C22—C23	120.2 (2)	C9—Re1—P1	89.73 (7)
C23—C22—H22	119.9	C10—Re1—N1	89.19 (8)
C18—C23—C22	120.1 (2)	C10—Re1—N3	89.88 (8)
C18—C23—H23	119.9	C10—Re1—P1	175.79 (7)
C22—C23—H23	119.9	C11—Re1—C10	90.72 (9)
H24A—C24—H24B	108.3	C11—Re1—N1	95.94 (8)
P1—C24—H24A	109.9	C11—Re1—N3	170.78 (8)
P1—C24—H24B	109.9	C11—Re1—P1	93.45 (6)
P1—C24—P2	108.99 (10)	N1—Re1—P1	90.93 (5)
P2—C24—H24A	109.9	N3—Re1—N1	74.87 (6)
P2—C24—H24B	109.9	N3—Re1—P1	86.09 (5)
C26—C25—C30	118.6 (2)	O4—S101—C37	103.91 (14)
C26—C25—P2	117.97 (16)	O4—S101—O6	114.53 (11)
C30—C25—P2	123.33 (17)	O5—S101—C37	103.60 (14)
C25—C26—H26	119.6	O5—S101—O4	114.36 (13)
C27—C26—C25	120.8 (2)	O5—S101—O6	116.14 (14)
C27—C26—H26	119.6	O6—S101—C37	101.84 (13)

C1—C2—N2—C3	−0.9 (3)	C26—C25—P2—C31	−157.87 (17)
C2—C1—N1—C4	1.6 (3)	C26—C27—C28—C29	0.9 (4)
C2—C1—N1—Re1	178.96 (15)	C27—C28—C29—C30	−0.4 (4)
C3—C4—C5—C6	−3.3 (3)	C28—C29—C30—C25	0.0 (4)
C3—C4—C5—N3	175.81 (19)	C30—C25—C26—C27	0.4 (3)
C3—C4—N1—C1	−1.9 (3)	C30—C25—P2—C24	−87.99 (19)
C3—C4—N1—Re1	−179.44 (15)	C30—C25—P2—C31	18.7 (2)
C4—C3—N2—C2	0.7 (3)	C31—C32—C33—C34	−0.6 (4)
C4—C5—C6—N4	178.5 (2)	C32—C31—C36—C35	−1.2 (3)
C4—C5—N3—C8	−177.60 (18)	C32—C31—P2—C24	−168.40 (17)
C4—C5—N3—Re1	4.6 (2)	C32—C31—P2—C25	85.84 (19)
C5—C4—N1—C1	176.05 (17)	C32—C33—C34—C35	−1.7 (4)
C5—C4—N1—Re1	−1.5 (2)	C33—C34—C35—C36	2.4 (4)
C5—C6—N4—C7	−0.7 (3)	C34—C35—C36—C31	−1.0 (4)
C6—C5—N3—C8	1.6 (3)	C36—C31—C32—C33	2.0 (4)
C6—C5—N3—Re1	−176.25 (15)	C36—C31—P2—C24	14.5 (2)

C7—C8—N3—C5	−1.2 (3)	C36—C31—P2—C25	−91.3 (2)
C7—C8—N3—Re1	176.36 (15)	F101—C37—S101—O4	−65.0 (2)
C8—C7—N4—C6	1.0 (3)	F101—C37—S101—O5	175.2 (2)
C12—C13—C14—C15	2.0 (3)	F101—C37—S101—O6	54.2 (3)
C13—C12—C17—C16	−1.1 (3)	F102—C37—S101—O4	175.2 (2)
C13—C12—P1—C18	−51.49 (18)	F102—C37—S101—O5	55.4 (2)
C13—C12—P1—C24	−159.52 (16)	F102—C37—S101—O6	−65.5 (2)
C13—C12—P1—Re1	76.46 (17)	F103—C37—S101—O4	55.9 (3)
C13—C14—C15—C16	−1.1 (4)	F103—C37—S101—O5	−63.9 (3)
C14—C15—C16—C17	−0.9 (4)	F103—C37—S101—O6	175.1 (2)
C15—C16—C17—C12	2.0 (3)	N1—C1—C2—N2	−0.2 (3)
C17—C12—C13—C14	−0.9 (3)	N1—C4—C5—C6	178.86 (19)
C17—C12—P1—C18	135.24 (17)	N1—C4—C5—N3	−2.0 (3)
C17—C12—P1—C24	27.2 (2)	N2—C3—C4—C5	−177.0 (2)
C17—C12—P1—Re1	−96.81 (17)	N2—C3—C4—N1	0.8 (3)
C18—C19—C20—C21	−0.3 (3)	N3—C5—C6—N4	−0.6 (3)
C19—C18—C23—C22	−0.6 (3)	N4—C7—C8—N3	−0.1 (3)
C19—C18—P1—C12	−46.79 (18)	P1—C12—C13—C14	−174.32 (17)
C19—C18—P1—C24	62.64 (18)	P1—C12—C17—C16	172.16 (17)
C19—C18—P1—Re1	−168.99 (13)	P1—C18—C19—C20	−177.27 (17)
C19—C20—C21—C22	−0.3 (3)	P1—C18—C23—C22	177.34 (16)
C20—C21—C22—C23	0.4 (3)	P1—C24—P2—C25	−112.46 (11)
C21—C22—C23—C18	0.0 (3)	P1—C24—P2—C31	144.00 (11)
C23—C18—C19—C20	0.8 (3)	P2—C24—P1—C12	171.97 (10)
C23—C18—P1—C12	135.25 (17)	P2—C24—P1—C18	64.71 (12)
C23—C18—P1—C24	−115.31 (18)	P2—C24—P1—Re1	−66.55 (11)
C23—C18—P1—Re1	13.1 (2)	P2—C25—C26—C27	177.11 (17)
C25—C26—C27—C28	−0.8 (3)	P2—C25—C30—C29	−176.49 (18)
C26—C25—C30—C29	0.1 (3)	P2—C31—C32—C33	−175.3 (2)
C26—C25—P2—C24	95.42 (18)	P2—C31—C36—C35	175.77 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O4^{i}	0.95	2.47	3.281 (3)	143
C6—H6···O4^{i}	0.95	2.62	3.468 (3)	148
C8—H8···O101^{ii}	0.95	2.33	3.226 (3)	156
C17—H17···O6^{i}	0.95	2.31	3.180 (3)	152
O101—H10*A*···N2^{iii}	0.94 (5)	2.20 (5)	2.983 (3)	141 (4)
O101—H10*A*···N4^{iv}	0.94 (5)	2.44 (5)	3.126 (3)	131 (4)
O101—H10*B*···O1^{v}	0.84 (3)	2.47 (3)	3.103 (3)	133 (3)

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