Table S-1. Crystallographic Experimental Details for **4**

A. Crystal Data	
formula	$C_{61}H_{63}BCl_2F_4O_3OsP_5Rh$
formula weight	1449.78
crystal dimensions (mm)	0.45 × 0.16 × 0.08
crystal system	monoclinic
space group	$P2_1/c$ (No. 14)
unit cell parameters[a]	
a (Å)	22.0870 (11)
b (Å)	12.0207 (6)
c (Å)	23.9857 (12)
β (deg)	93.4093 (9)
V (Å^3)	6357.0 (5)
Z	4
ρ_{calcd} (g cm^{-3})	1.515
μ (mm^{-1})	2.520
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/SMART 1000 CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.2°) (20 s exposures)
data collection 2θ limit (deg)	52.92
total data collected	43363 (-27 ≤ h ≤ 27, -15 ≤ k ≤ 15, -30 ≤ l ≤ 30)
independent reflections	13085 (R_{int} = 0.0389)
number of observed reflections (*NO*)	11341 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	Patterson search/structure expansion (*DIRDIF–99*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	multi-scan (*SADABS*)
range of transmission factors	0.8238–0.3968
data/restraints/parameters	13085 [$F_o^2 \geq -3\sigma(F_o^2)$] / 0 / 720
goodness-of-fit (S)[e]	1.129 [$F_o^2 \geq -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0457
wR_2 [$F_o^2 \geq -3\sigma(F_o^2)$]	0.1372
largest difference peak and hole	3.301 and –0.986 e Å^{-3}

[a]Obtained from least-squares refinement of 6899 reflections with 4.87° < 2θ < 52.78°.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table S-1. Crystallographic Experimental Details for **4** (continued)

[c]Beurskens, P. T.; Beurskens, G.; de Gelder, R.; Garcia-Granda, S.; Israel, R.; Gould, R. O.; Smits, J. M. M. (1999). The *DIRDIF-99* program system. Crystallography Laboratory, University of Nijmegen, The Netherlands.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted *R*-factors wR_2 and all goodnesses of fit *S* are based on F_o^2; conventional *R*-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. *R*-factors based on F_o^2 are statistically about twice as large as those based on F_o, and *R*-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0657P)^2 + 33.5252P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table S-2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **4**

(a) 'inner-core' atoms of $[RhOs(CO)_3(PEt_3)(\mu\text{–}CH_2)(dppm)_2]^+$

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Os	0.332139(10)	-0.147778(18)	0.294715(9)	0.01920(8)*
Rh	0.233753(19)	0.01045(4)	0.272119(18)	0.01776(11)*
P(1)	0.34751(7)	-0.17631(12)	0.19825(6)	0.0199(3)*
P(2)	0.27178(7)	0.02294(12)	0.18379(6)	0.0190(3)*
P(3)	0.30335(7)	-0.18450(12)	0.38625(6)	0.0209(3)*
P(4)	0.22342(7)	0.01375(12)	0.36895(6)	0.0204(3)*
P(5)	0.12757(7)	-0.00817(14)	0.23825(7)	0.0252(3)*
O(1)	0.4024(3)	-0.3602(4)	0.3151(2)	0.0458(14)*
O(2)	0.4396(2)	0.0028(4)	0.3309(2)	0.0369(11)*
O(3)	0.2479(3)	0.2595(4)	0.2786(2)	0.0417(12)*
C(1)	0.3752(3)	-0.2795(6)	0.3058(3)	0.0305(14)*
C(2)	0.4000(3)	-0.0541(6)	0.3165(3)	0.0270(13)*
C(3)	0.2432(3)	0.1670(5)	0.2767(3)	0.0254(13)*
C(4)	0.2398(3)	-0.1686(5)	0.2712(3)	0.0208(12)*
C(5)	0.2890(3)	-0.1104(5)	0.1518(2)	0.0213(12)*
C(6)	0.2310(3)	-0.1220(5)	0.4029(3)	0.0240(12)*

(b) dppm phenyl carbons

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C(11)	0.3448(3)	-0.3254(5)	0.1823(3)	0.0246(12)*
C(12)	0.3984(3)	-0.3866(5)	0.1860(3)	0.0286(13)*
C(13)	0.3970(3)	-0.5012(5)	0.1808(3)	0.0314(14)*
C(14)	0.3428(4)	-0.5562(5)	0.1715(3)	0.0428(18)*
C(15)	0.2896(4)	-0.4958(7)	0.1667(4)	0.053(2)*
C(16)	0.2897(3)	-0.3809(6)	0.1718(3)	0.0382(17)*
C(21)	0.4179(3)	-0.1370(5)	0.1663(3)	0.0246(13)*
C(22)	0.4682(3)	-0.1009(5)	0.1978(3)	0.0305(14)*
C(23)	0.5215(3)	-0.0737(6)	0.1732(3)	0.0371(16)*
C(24)	0.5245(3)	-0.0822(6)	0.1162(4)	0.0430(18)*
C(25)	0.4750(4)	-0.1200(6)	0.0841(3)	0.0422(18)*
C(26)	0.4222(3)	-0.1488(6)	0.1088(3)	0.0332(15)*
C(31)	0.3435(3)	0.1012(5)	0.1790(2)	0.0216(12)*
C(32)	0.3745(3)	0.1428(5)	0.2260(3)	0.0266(13)*
C(33)	0.4294(3)	0.1982(6)	0.2219(3)	0.0336(15)*
C(34)	0.4532(3)	0.2113(6)	0.1706(3)	0.0383(16)*
C(35)	0.4225(3)	0.1705(6)	0.1234(3)	0.0357(16)*
C(36)	0.3676(3)	0.1158(6)	0.1271(3)	0.0308(14)*
C(41)	0.2261(3)	0.0953(5)	0.1281(2)	0.0230(12)*
C(42)	0.2199(3)	0.2106(6)	0.1322(3)	0.0314(14)*

Table S-2. Atomic Coordinates and Displacement Parameters for **4** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C(43)	0.1865(3)	0.2691(6)	0.0910(3)	0.0399(17)*
C(44)	0.1600(4)	0.2149(7)	0.0462(3)	0.048(2)*
C(45)	0.1659(4)	0.1008(7)	0.0414(3)	0.049(2)*
C(46)	0.1993(3)	0.0409(7)	0.0823(3)	0.0369(16)*
C(51)	0.2896(3)	-0.3337(5)	0.3984(3)	0.0285(14)*
C(52)	0.2656(4)	-0.4005(6)	0.3558(3)	0.0466(19)*
C(53)	0.2546(5)	-0.5115(7)	0.3648(4)	0.060(2)*
C(54)	0.2675(4)	-0.5590(6)	0.4157(4)	0.054(2)*
C(55)	0.2906(5)	-0.4935(7)	0.4583(4)	0.063(3)*
C(56)	0.3013(4)	-0.3816(6)	0.4504(3)	0.048(2)*
C(61)	0.3581(3)	-0.1485(5)	0.4436(3)	0.0259(13)*
C(62)	0.4193(3)	-0.1663(6)	0.4365(3)	0.0332(15)*
C(63)	0.4627(3)	-0.1430(7)	0.4789(3)	0.0408(17)*
C(64)	0.4451(4)	-0.1013(7)	0.5292(3)	0.0420(18)*
C(65)	0.3847(4)	-0.0850(6)	0.5371(3)	0.0401(17)*
C(66)	0.3410(3)	-0.1081(6)	0.4952(3)	0.0324(15)*
C(71)	0.2779(3)	0.0990(5)	0.4112(2)	0.0223(12)*
C(72)	0.3321(3)	0.1342(5)	0.3899(3)	0.0275(13)*
C(73)	0.3748(3)	0.1928(6)	0.4232(3)	0.0392(17)*
C(74)	0.3635(3)	0.2171(7)	0.4773(3)	0.0408(17)*
C(75)	0.3096(3)	0.1832(6)	0.4993(3)	0.0369(16)*
C(76)	0.2673(3)	0.1239(5)	0.4668(3)	0.0294(14)*
C(81)	0.1531(3)	0.0678(5)	0.3949(2)	0.0264(13)*
C(82)	0.1376(3)	0.1782(6)	0.3833(3)	0.0337(15)*
C(83)	0.0840(3)	0.2224(7)	0.4017(3)	0.046(2)*
C(84)	0.0463(4)	0.1581(8)	0.4318(3)	0.051(2)*
C(85)	0.0619(3)	0.0500(8)	0.4435(3)	0.0467(19)*
C(86)	0.1149(3)	0.0054(7)	0.4257(3)	0.0360(16)*
C(91)	0.0688(3)	-0.0460(6)	0.2857(3)	0.0344(15)*
C(92)	0.0749(4)	-0.1635(7)	0.3105(4)	0.048(2)*
C(93)	0.1171(3)	-0.1133(6)	0.1835(3)	0.0358(16)*
C(94)	0.0540(4)	-0.1285(8)	0.1565(4)	0.055(2)*
C(95)	0.0919(3)	0.1147(6)	0.2045(3)	0.0382(16)*
C(96)	0.0855(4)	0.2150(7)	0.2409(4)	0.059(2)*

(c) tetrafluoroborate ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
F(1)	0.1944(3)	-0.4163(5)	0.0436(2)	0.0695(16)*
F(2)	0.1052(3)	-0.3249(6)	0.0350(3)	0.0796(18)*
F(3)	0.1731(3)	-0.2775(7)	0.1024(3)	0.094(2)*

Table S-2. Atomic Coordinates and Displacement Parameters for **4** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
F(4)	0.1919(5)	-0.2400(8)	0.0154(5)	0.149(4)*
B	0.1664(5)	-0.3152(10)	0.0509(4)	0.055(3)*

(d) solvent dichloromethane atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Cl(1S)[a]	0.0707(2)	0.4424(4)	0.1329(2)	0.0647(13)*
Cl(2S)[a]	0.1149(4)	0.5505(5)	0.2347(3)	0.103(2)*
C(1S)[a]	0.0842(11)	0.559(2)	0.1666(10)	0.080(6)
Cl(3S)[a]	-0.0081(2)	0.6602(7)	0.4408(3)	0.101(3)*
Cl(4S)[a]	0.0972(3)	0.5721(5)	0.3964(3)	0.0849(19)*
C(2S)[a]	0.0633(11)	0.627(2)	0.4474(10)	0.078(6)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$. [a]Refined with an occupancy factor of 0.5.

Table S-3. Selected Interatomic Distances (Å) for **4**

(a) involving 'inner-core' atoms of $[RhOs(CO)_3(PEt_3)(\mu\text{–}CH_2)(dppm)_2]^+$

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Os	Rh	2.9143(5)	Rh	C(4)	2.156(5)
Os	P(1)	2.3832(15)	P(1)	P(2)	2.930(2)†
Os	P(3)	2.3635(15)	P(1)	C(5)	1.835(6)
Os	C(1)	1.858(7)	P(2)	C(5)	1.827(6)
Os	C(2)	1.922(6)	P(3)	P(4)	2.980(2)†
Os	C(4)	2.099(6)	P(3)	C(6)	1.831(6)
Rh	P(2)	2.3298(15)	P(4)	C(6)	1.826(6)
Rh	P(4)	2.3476(15)	O(1)	C(1)	1.155(8)
Rh	P(5)	2.4462(16)	O(2)	C(2)	1.148(8)
Rh	C(3)	1.896(6)	O(3)	C(3)	1.117(8)

†Nonbonded distance.

(b) involving dppm phenyl carbons

Atom1	Atom2	Distance	Atom1	Atom2	Distance
P(1)	C(11)	1.833(6)	C(32)	C(33)	1.391(9)
P(1)	C(21)	1.835(6)	C(33)	C(34)	1.375(10)
P(2)	C(31)	1.853(6)	C(34)	C(35)	1.376(11)
P(2)	C(41)	1.844(6)	C(35)	C(36)	1.388(10)
P(3)	C(51)	1.845(6)	C(41)	C(42)	1.397(9)
P(3)	C(61)	1.829(6)	C(41)	C(46)	1.381(9)
P(4)	C(71)	1.839(6)	C(42)	C(43)	1.388(9)
P(4)	C(81)	1.827(6)	C(43)	C(44)	1.358(11)
P(5)	C(91)	1.834(7)	C(44)	C(45)	1.384(12)
P(5)	C(93)	1.827(7)	C(45)	C(46)	1.393(10)
P(5)	C(95)	1.839(7)	C(51)	C(52)	1.380(10)
C(11)	C(12)	1.392(9)	C(51)	C(56)	1.384(10)
C(11)	C(16)	1.399(9)	C(52)	C(53)	1.376(11)
C(12)	C(13)	1.383(9)	C(53)	C(54)	1.362(13)
C(13)	C(14)	1.374(10)	C(54)	C(55)	1.365(13)
C(14)	C(15)	1.379(11)	C(55)	C(56)	1.380(11)
C(15)	C(16)	1.386(10)	C(61)	C(62)	1.389(9)
C(21)	C(22)	1.377(9)	C(61)	C(66)	1.402(9)
C(21)	C(26)	1.397(9)	C(62)	C(63)	1.383(10)
C(22)	C(23)	1.385(9)	C(63)	C(64)	1.383(11)
C(23)	C(24)	1.377(11)	C(64)	C(65)	1.373(11)
C(24)	C(25)	1.376(12)	C(65)	C(66)	1.380(10)
C(25)	C(26)	1.383(10)	C(71)	C(72)	1.395(9)
C(31)	C(32)	1.378(9)	C(71)	C(76)	1.399(9)
C(31)	C(36)	1.394(9)	C(72)	C(73)	1.391(9)

Table S-3. Selected Interatomic Distances for **4** (continued)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C(73)	C(74)	1.369(11)	C(83)	C(84)	1.373(13)
C(74)	C(75)	1.393(11)	C(84)	C(85)	1.368(12)
C(75)	C(76)	1.380(9)	C(85)	C(86)	1.378(10)
C(81)	C(82)	1.394(9)	C(91)	C(92)	1.536(11)
C(81)	C(86)	1.377(10)	C(93)	C(94)	1.513(10)
C(82)	C(83)	1.394(10)	C(95)	C(96)	1.501(11)

(c) within the tetrafluoroborate ion

Atom1	Atom2	Distance	Atom1	Atom2	Distance
F(1)	B	1.379(12)	F(3)	B	1.317(11)
F(2)	B	1.386(12)	F(4)	B	1.385(14)

(d) within the solvent dichloromethane molecules

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Cl(1S)	C(1S)	1.64(2)	Cl(3S)	C(2S)	1.63(2)
Cl(2S)	C(1S)	1.73(2)	Cl(4S)	C(2S)	1.61(2)

Table S-4. Selected Interatomic Angles (deg) for **4**

(a) involving 'inner-core' atoms of $[RhOs(CO)_3(PEt_3)(\mu\text{-}CH_2)(dppm)_2]^+$

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Rh	Os	P(1)	93.50(4)	P(2)	Rh	P(4)	163.76(6)
Rh	Os	P(3)	93.20(4)	P(2)	Rh	P(5)	95.41(5)
Rh	Os	C(1)	162.2(2)	P(2)	Rh	C(3)	86.80(19)
Rh	Os	C(2)	103.22(19)	P(2)	Rh	C(4)	91.67(16)
Rh	Os	C(4)	47.60(15)	P(4)	Rh	P(5)	100.51(6)
P(1)	Os	P(3)	159.51(5)	P(4)	Rh	C(3)	86.74(19)
P(1)	Os	C(1)	85.1(2)	P(4)	Rh	C(4)	92.11(16)
P(1)	Os	C(2)	101.18(19)	P(5)	Rh	C(3)	102.1(2)
P(1)	Os	C(4)	85.08(17)	P(5)	Rh	C(4)	87.91(16)
P(3)	Os	C(1)	82.6(2)	C(3)	Rh	C(4)	170.0(2)
P(3)	Os	C(2)	96.08(19)	Os	P(1)	C(5)	113.1(2)
P(3)	Os	C(4)	85.22(17)	Rh	P(2)	C(5)	114.9(2)
C(1)	Os	C(2)	94.4(3)	Os	P(3)	C(6)	114.3(2)
C(1)	Os	C(4)	114.7(3)	Rh	P(4)	C(6)	114.4(2)
C(2)	Os	C(4)	150.7(2)	Os	C(1)	O(1)	177.1(6)
Os	Rh	P(2)	84.61(4)	Os	C(2)	O(2)	177.9(6)
Os	Rh	P(4)	86.69(4)	Rh	C(3)	O(3)	178.8(7)
Os	Rh	P(5)	133.76(4)	Os	C(4)	Rh	86.4(2)
Os	Rh	C(3)	124.0(2)	P(1)	C(5)	P(2)	106.3(3)
Os	Rh	C(4)	45.97(15)	P(3)	C(6)	P(4)	109.1(3)

(b) involving dppm phenyl carbons

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Os	P(1)	C(11)	109.8(2)	Rh	P(4)	C(81)	118.5(2)
Os	P(1)	C(21)	122.7(2)	C(6)	P(4)	C(71)	102.3(3)
C(5)	P(1)	C(11)	106.4(3)	C(6)	P(4)	C(81)	102.9(3)
C(5)	P(1)	C(21)	102.6(3)	C(71)	P(4)	C(81)	98.9(3)
C(11)	P(1)	C(21)	100.5(3)	Rh	P(5)	C(91)	121.1(2)
Rh	P(2)	C(31)	116.4(2)	Rh	P(5)	C(93)	112.3(2)
Rh	P(2)	C(41)	118.6(2)	Rh	P(5)	C(95)	116.8(2)
C(5)	P(2)	C(31)	102.6(3)	C(91)	P(5)	C(93)	102.2(3)
C(5)	P(2)	C(41)	103.2(3)	C(91)	P(5)	C(95)	99.9(3)
C(31)	P(2)	C(41)	98.5(3)	C(93)	P(5)	C(95)	101.9(4)
Os	P(3)	C(51)	112.6(2)	P(1)	C(11)	C(12)	119.1(5)
Os	P(3)	C(61)	116.9(2)	P(1)	C(11)	C(16)	121.4(5)
C(6)	P(3)	C(51)	102.2(3)	C(12)	C(11)	C(16)	119.2(6)
C(6)	P(3)	C(61)	106.6(3)	C(11)	C(12)	C(13)	120.4(6)
C(51)	P(3)	C(61)	102.6(3)	C(12)	C(13)	C(14)	120.6(7)
Rh	P(4)	C(71)	117.1(2)	C(13)	C(14)	C(15)	119.3(6)

Table S-4. Selected Interatomic Angles for **4** (continued)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C(14)	C(15)	C(16)	121.4(7)	C(53)	C(54)	C(55)	118.6(8)
C(11)	C(16)	C(15)	119.1(7)	C(54)	C(55)	C(56)	121.2(8)
P(1)	C(21)	C(22)	121.9(5)	C(51)	C(56)	C(55)	120.4(8)
P(1)	C(21)	C(26)	119.7(5)	P(3)	C(61)	C(62)	118.4(5)
C(22)	C(21)	C(26)	118.3(6)	P(3)	C(61)	C(66)	123.1(5)
C(21)	C(22)	C(23)	121.2(7)	C(62)	C(61)	C(66)	118.5(6)
C(22)	C(23)	C(24)	119.9(7)	C(61)	C(62)	C(63)	121.0(7)
C(23)	C(24)	C(25)	119.8(7)	C(62)	C(63)	C(64)	119.7(7)
C(24)	C(25)	C(26)	120.3(7)	C(63)	C(64)	C(65)	119.9(7)
C(21)	C(26)	C(25)	120.5(7)	C(64)	C(65)	C(66)	120.9(7)
P(2)	C(31)	C(32)	121.2(5)	C(61)	C(66)	C(65)	119.9(7)
P(2)	C(31)	C(36)	119.5(5)	P(4)	C(71)	C(72)	121.0(5)
C(32)	C(31)	C(36)	119.3(6)	P(4)	C(71)	C(76)	120.1(5)
C(31)	C(32)	C(33)	120.4(6)	C(72)	C(71)	C(76)	118.8(6)
C(32)	C(33)	C(34)	120.1(7)	C(71)	C(72)	C(73)	120.7(6)
C(33)	C(34)	C(35)	119.9(6)	C(72)	C(73)	C(74)	119.8(7)
C(34)	C(35)	C(36)	120.4(7)	C(73)	C(74)	C(75)	120.3(7)
C(31)	C(36)	C(35)	119.8(6)	C(74)	C(75)	C(76)	120.2(7)
P(2)	C(41)	C(42)	118.0(5)	C(71)	C(76)	C(75)	120.2(7)
P(2)	C(41)	C(46)	122.9(5)	P(4)	C(81)	C(82)	118.3(5)
C(42)	C(41)	C(46)	119.1(6)	P(4)	C(81)	C(86)	123.3(5)
C(41)	C(42)	C(43)	120.2(6)	C(82)	C(81)	C(86)	118.4(6)
C(42)	C(43)	C(44)	120.4(7)	C(81)	C(82)	C(83)	120.2(7)
C(43)	C(44)	C(45)	120.2(7)	C(82)	C(83)	C(84)	120.3(8)
C(44)	C(45)	C(46)	120.1(7)	C(83)	C(84)	C(85)	119.3(7)
C(41)	C(46)	C(45)	120.0(7)	C(84)	C(85)	C(86)	120.9(8)
P(3)	C(51)	C(52)	120.6(5)	C(81)	C(86)	C(85)	120.9(7)
P(3)	C(51)	C(56)	121.5(5)	P(5)	C(91)	C(92)	114.8(5)
C(52)	C(51)	C(56)	117.9(6)	P(5)	C(93)	C(94)	117.7(6)
C(51)	C(52)	C(53)	120.8(8)	P(5)	C(95)	C(96)	116.2(6)
C(52)	C(53)	C(54)	121.2(9)				

(c) within the tetrafluoroborate ion

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
F(1)	B	F(2)	109.2(8)	F(2)	B	F(3)	109.9(8)
F(1)	B	F(3)	113.2(10)	F(2)	B	F(4)	108.2(10)
F(1)	B	F(4)	107.2(8)	F(3)	B	F(4)	109.0(10)

(d) within the solvent dichloromethane molecules

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Cl(1S)	C(1S)	Cl(2S)	117.6(15)	Cl(3S)	C(2S)	Cl(4S)	121.1(15)

Table S-5. Anisotropic Displacement Parameters (U_{ij}, Å^2) for **4**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Os	0.02157(12)	0.01778(12)	0.01809(12)	-0.00121(9)	-0.00026(8)	0.00176(8)
Rh	0.0207(2)	0.0157(2)	0.0168(2)	-0.00066(16)	0.00025(16)	0.00037(16)
P(1)	0.0230(7)	0.0160(7)	0.0208(7)	-0.0023(6)	0.0018(6)	0.0003(6)
P(2)	0.0230(7)	0.0176(7)	0.0163(7)	-0.0003(5)	0.0002(5)	-0.0004(6)
P(3)	0.0262(7)	0.0175(7)	0.0190(7)	0.0009(6)	0.0006(6)	0.0019(6)
P(4)	0.0232(7)	0.0200(7)	0.0181(7)	-0.0013(6)	0.0017(6)	0.0015(6)
P(5)	0.0224(7)	0.0286(8)	0.0244(8)	-0.0006(6)	-0.0013(6)	0.0004(6)
O(1)	0.059(3)	0.038(3)	0.040(3)	0.001(2)	-0.001(3)	0.026(3)
O(2)	0.030(2)	0.038(3)	0.042(3)	-0.007(2)	-0.002(2)	-0.010(2)
O(3)	0.064(3)	0.016(2)	0.046(3)	-0.001(2)	0.010(3)	0.006(2)
C(1)	0.035(3)	0.034(4)	0.021(3)	-0.007(3)	-0.002(3)	0.009(3)
C(2)	0.026(3)	0.032(3)	0.023(3)	-0.005(3)	0.000(2)	0.004(3)
C(3)	0.034(3)	0.018(3)	0.024(3)	0.000(2)	0.004(3)	0.003(2)
C(4)	0.026(3)	0.013(2)	0.023(3)	0.003(2)	0.003(2)	0.004(2)
C(5)	0.027(3)	0.018(3)	0.019(3)	-0.001(2)	-0.001(2)	0.000(2)
C(6)	0.026(3)	0.024(3)	0.022(3)	0.002(2)	0.006(2)	0.001(2)
C(11)	0.031(3)	0.021(3)	0.021(3)	0.001(2)	0.002(2)	0.002(2)
C(12)	0.033(3)	0.026(3)	0.027(3)	0.000(3)	-0.004(3)	-0.002(3)
C(13)	0.044(4)	0.022(3)	0.029(3)	0.004(3)	0.001(3)	0.003(3)
C(14)	0.059(5)	0.012(3)	0.057(5)	-0.005(3)	-0.002(4)	0.000(3)
C(15)	0.042(4)	0.033(4)	0.082(7)	-0.008(4)	-0.002(4)	-0.007(3)
C(16)	0.033(4)	0.020(3)	0.062(5)	-0.006(3)	0.000(3)	0.001(3)
C(21)	0.028(3)	0.018(3)	0.030(3)	0.000(2)	0.010(2)	0.000(2)
C(22)	0.034(3)	0.023(3)	0.035(4)	-0.002(3)	0.003(3)	-0.002(3)
C(23)	0.034(4)	0.027(3)	0.051(5)	0.000(3)	0.006(3)	-0.005(3)
C(24)	0.039(4)	0.031(4)	0.061(5)	0.000(4)	0.025(4)	-0.001(3)
C(25)	0.055(5)	0.036(4)	0.037(4)	-0.003(3)	0.019(4)	0.003(3)
C(26)	0.035(4)	0.034(4)	0.031(4)	-0.005(3)	0.007(3)	0.001(3)
C(31)	0.024(3)	0.016(3)	0.025(3)	0.002(2)	0.002(2)	0.003(2)
C(32)	0.030(3)	0.024(3)	0.026(3)	0.001(3)	0.001(2)	-0.003(2)
C(33)	0.030(3)	0.030(3)	0.040(4)	-0.001(3)	-0.004(3)	-0.003(3)
C(34)	0.027(3)	0.033(4)	0.055(5)	0.008(3)	0.006(3)	-0.002(3)
C(35)	0.035(4)	0.031(4)	0.042(4)	0.012(3)	0.014(3)	-0.001(3)
C(36)	0.037(4)	0.028(3)	0.028(3)	0.004(3)	0.004(3)	0.003(3)
C(41)	0.025(3)	0.024(3)	0.021(3)	0.005(2)	0.002(2)	0.001(2)
C(42)	0.034(3)	0.031(3)	0.029(3)	0.003(3)	0.000(3)	0.005(3)
C(43)	0.047(4)	0.031(4)	0.041(4)	0.015(3)	0.002(3)	0.006(3)
C(44)	0.044(4)	0.055(5)	0.044(4)	0.021(4)	-0.012(3)	0.003(4)
C(45)	0.056(5)	0.055(5)	0.032(4)	0.003(4)	-0.017(3)	-0.001(4)
C(46)	0.041(4)	0.047(4)	0.022(3)	0.001(3)	-0.004(3)	0.001(3)
C(51)	0.037(3)	0.021(3)	0.029(3)	0.004(3)	0.005(3)	0.004(3)
C(52)	0.071(6)	0.027(4)	0.040(4)	0.003(3)	-0.009(4)	-0.006(4)
C(53)	0.080(7)	0.032(4)	0.065(6)	0.001(4)	-0.013(5)	-0.003(4)

Table S-5. Anisotropic Displacement Parameters for **4** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(54)	0.075(6)	0.020(4)	0.068(6)	0.009(4)	0.006(5)	-0.003(4)
C(55)	0.102(8)	0.040(5)	0.047(5)	0.021(4)	0.004(5)	-0.003(5)
C(56)	0.085(6)	0.030(4)	0.029(4)	0.006(3)	0.000(4)	-0.008(4)
C(61)	0.030(3)	0.026(3)	0.021(3)	0.003(2)	0.000(2)	0.000(3)
C(62)	0.033(3)	0.041(4)	0.025(3)	0.001(3)	0.002(3)	0.006(3)
C(63)	0.031(4)	0.053(5)	0.038(4)	0.006(3)	-0.007(3)	0.002(3)
C(64)	0.051(5)	0.042(4)	0.031(4)	0.002(3)	-0.017(3)	-0.004(4)
C(65)	0.058(5)	0.038(4)	0.023(3)	-0.001(3)	-0.006(3)	0.004(3)
C(66)	0.045(4)	0.030(3)	0.023(3)	0.001(3)	-0.001(3)	0.004(3)
C(71)	0.028(3)	0.017(3)	0.021(3)	-0.002(2)	0.001(2)	0.005(2)
C(72)	0.029(3)	0.026(3)	0.027(3)	-0.003(3)	0.000(3)	0.000(3)
C(73)	0.032(4)	0.043(4)	0.042(4)	-0.006(3)	-0.003(3)	-0.008(3)
C(74)	0.041(4)	0.042(4)	0.038(4)	-0.013(3)	-0.010(3)	-0.002(3)
C(75)	0.049(4)	0.036(4)	0.025(3)	-0.011(3)	-0.006(3)	0.004(3)
C(76)	0.040(4)	0.028(3)	0.020(3)	-0.001(3)	0.000(3)	0.002(3)
C(81)	0.027(3)	0.032(3)	0.020(3)	-0.005(3)	0.000(2)	0.007(3)
C(82)	0.038(4)	0.035(4)	0.027(3)	-0.002(3)	-0.002(3)	0.009(3)
C(83)	0.043(4)	0.053(5)	0.042(4)	-0.012(4)	-0.005(3)	0.024(4)
C(84)	0.039(4)	0.079(6)	0.035(4)	-0.006(4)	0.007(3)	0.026(4)
C(85)	0.036(4)	0.065(5)	0.040(4)	0.010(4)	0.012(3)	0.006(4)
C(86)	0.030(3)	0.048(4)	0.030(4)	0.002(3)	0.006(3)	0.003(3)
C(91)	0.025(3)	0.047(4)	0.031(4)	0.002(3)	0.000(3)	-0.002(3)
C(92)	0.039(4)	0.056(5)	0.050(5)	0.009(4)	0.008(4)	-0.008(4)
C(93)	0.031(3)	0.041(4)	0.035(4)	-0.012(3)	-0.002(3)	-0.005(3)
C(94)	0.038(4)	0.067(6)	0.058(5)	-0.024(5)	-0.018(4)	-0.002(4)
C(95)	0.029(3)	0.044(4)	0.041(4)	0.002(3)	-0.003(3)	0.006(3)
C(96)	0.060(5)	0.042(5)	0.073(6)	-0.007(4)	-0.014(5)	0.019(4)
F(1)	0.065(3)	0.068(4)	0.075(4)	-0.025(3)	0.006(3)	0.013(3)
F(2)	0.057(3)	0.093(5)	0.087(4)	-0.019(4)	-0.018(3)	0.007(3)
F(3)	0.077(4)	0.131(6)	0.071(4)	-0.059(4)	-0.012(3)	0.021(4)
F(4)	0.173(9)	0.109(7)	0.177(9)	0.014(6)	0.109(8)	-0.008(6)
B	0.049(5)	0.066(7)	0.050(6)	-0.027(5)	0.014(4)	0.001(5)
Cl(1S)	0.051(2)	0.065(3)	0.080(3)	0.024(3)	0.024(2)	0.011(2)
Cl(2S)	0.127(6)	0.056(3)	0.126(6)	-0.010(4)	0.005(5)	-0.008(3)
Cl(3S)	0.050(3)	0.165(7)	0.087(4)	-0.065(4)	-0.010(3)	0.031(3)
Cl(4S)	0.088(4)	0.066(3)	0.107(5)	0.014(3)	0.054(3)	0.028(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table S-6. Derived Coordinates and Displacement Parameters for Hydrogen Atoms of **4**

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(4A)	0.2162	-0.2060	0.2996	0.025
H(4B)	0.2317	-0.2023	0.2338	0.025
H(5A)	0.3042	-0.0989	0.1142	0.026
H(5B)	0.2523	-0.1576	0.1482	0.026
H(6A)	0.1971	-0.1709	0.3895	0.029
H(6B)	0.2295	-0.1133	0.4438	0.029
H(12)	0.4362	-0.3495	0.1920	0.034
H(13)	0.4338	-0.5423	0.1838	0.038
H(14)	0.3419	-0.6350	0.1683	0.051
H(15)	0.2522	-0.5337	0.1597	0.063
H(16)	0.2527	-0.3404	0.1683	0.046
H(22)	0.4664	-0.0945	0.2372	0.037
H(23)	0.5558	-0.0492	0.1957	0.045
H(24)	0.5606	-0.0621	0.0991	0.052
H(25)	0.4771	-0.1263	0.0447	0.051
H(26)	0.3887	-0.1769	0.0864	0.040
H(32)	0.3583	0.1336	0.2616	0.032
H(33)	0.4503	0.2269	0.2544	0.040
H(34)	0.4909	0.2485	0.1679	0.046
H(35)	0.4390	0.1799	0.0880	0.043
H(36)	0.3464	0.0883	0.0943	0.037
H(42)	0.2385	0.2491	0.1632	0.038
H(43)	0.1821	0.3474	0.0942	0.048
H(44)	0.1374	0.2556	0.0181	0.058
H(45)	0.1470	0.0632	0.0101	0.058
H(46)	0.2036	-0.0374	0.0787	0.044
H(52)	0.2566	-0.3695	0.3198	0.056
H(53)	0.2378	-0.5558	0.3350	0.072
H(54)	0.2606	-0.6360	0.4214	0.065
H(55)	0.2995	-0.5255	0.4941	0.075
H(56)	0.3168	-0.3373	0.4808	0.058
H(62)	0.4315	-0.1949	0.4020	0.040
H(63)	0.5044	-0.1555	0.4734	0.049
H(64)	0.4748	-0.0840	0.5582	0.050
H(65)	0.3729	-0.0574	0.5719	0.048
H(66)	0.2993	-0.0966	0.5013	0.039
H(72)	0.3399	0.1179	0.3522	0.033
H(73)	0.4117	0.2160	0.4083	0.047
H(74)	0.3927	0.2572	0.5000	0.049
H(75)	0.3018	0.2010	0.5368	0.044

Table S-6. Derived Parameters for Hydrogen Atoms of **4** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(76)	0.2308	0.0999	0.4821	0.035
H(82)	0.1637	0.2233	0.3628	0.040
H(83)	0.0734	0.2974	0.3933	0.055
H(84)	0.0098	0.1883	0.4445	0.061
H(85)	0.0358	0.0051	0.4642	0.056
H(86)	0.1251	-0.0695	0.4347	0.043
H(91A)	0.0698	0.0084	0.3168	0.041
H(91B)	0.0287	-0.0397	0.2652	0.041
H(92A)	0.0425	-0.1760	0.3361	0.058
H(92B)	0.1144	-0.1710	0.3310	0.058
H(92C)	0.0715	-0.2185	0.2804	0.058
H(93A)	0.1446	-0.0947	0.1537	0.043
H(93B)	0.1306	-0.1857	0.1995	0.043
H(94A)	0.0548	-0.1863	0.1277	0.066
H(94B)	0.0401	-0.0583	0.1393	0.066
H(94C)	0.0263	-0.1507	0.1848	0.066
H(95A)	0.1160	0.1362	0.1727	0.046
H(95B)	0.0510	0.0932	0.1890	0.046
H(96A)	0.0639	0.2737	0.2195	0.071
H(96B)	0.1258	0.2418	0.2539	0.071
H(96C)	0.0626	0.1949	0.2732	0.071
H(1SA)[a]	0.0456	0.6008	0.1672	0.096
H(1SB)[a]	0.1121	0.6040	0.1449	0.096
H(2SA)[a]	0.0859	0.6959	0.4582	0.093
H(2SB)[a]	0.0684	0.5755	0.4794	0.093

[a]Included with an occupancy factor of 0.5.

Table S-7. Crystallographic Experimental Details for **5**

A. Crystal Data	
formula	$C_{69}H_{61}Cl_2F_3O_6OsP_5RhS$
formula weight	1594.10
crystal dimensions (mm)	0.47 × 0.19 × 0.09
crystal system	triclinic
space group	$P\bar{1}$ (No. 2)
unit cell parameters[a]	
a (Å)	11.4718 (4)
b (Å)	12.7929 (5)
c (Å)	22.1408 (8)
α (deg)	99.5651 (6)
β (deg)	94.9797 (7)
γ (deg)	92.6553 (7)
V (Å^3)	3186.0 (2)
Z	2
ρ_{calcd} (g cm^{-3})	1.662
μ (mm^{-1})	2.556
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/SMART 1000 CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.2°) (20 s exposures)
data collection 2θ limit (deg)	52.74
total data collected	20691 ($-14 \le h \le 12, -15 \le k \le 15, -27 \le l \le 27$)
independent reflections	12785 (R_{int} = 0.0201)
number of observed reflections (*NO*)	11831 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	direct methods (*SHELXS–86*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.8026–0.3797
data/restraints/parameters	12785 [$F_o^2 \ge -3\sigma(F_o^2)$] / 0 / 794
goodness-of-fit (S)[e]	1.045 [$F_o^2 \ge -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0289
wR_2 [$F_o^2 \ge -3\sigma(F_o^2)$]	0.0722
largest difference peak and hole	1.961 and –0.694 e Å^{-3}

[a]Obtained from least-squares refinement of 7957 reflections with 4.52° < 2θ < 52.68°.

(continued)

Table S-7. Crystallographic Experimental Details for **5** (continued)

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

[c]Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467–473.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and all goodnesses of fit S are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0340P)^2 + 4.5177P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table S-8. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **5**

(a) 'inner-core' atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Os	0.259362(10)	0.090884(9)	0.176296(5)	0.01593(4)*
Rh	0.28864(2)	0.094927(18)	0.307335(11)	0.01656(6)*
P1	0.14487(7)	0.24041(6)	0.18655(4)	0.01714(16)*
P2	0.27372(7)	0.27767(6)	0.31054(4)	0.01755(16)*
P3	0.28446(7)	-0.09362(6)	0.15371(4)	0.01867(16)*
P4	0.35223(7)	-0.07791(6)	0.28833(4)	0.01876(16)*
P5	0.13867(7)	0.06087(7)	0.37491(4)	0.02031(17)*
O1	0.2079(2)	0.0770(2)	0.03904(11)	0.0349(6)*
O2	0.5050(2)	0.1887(2)	0.17087(13)	0.0352(6)*
O3	0.5139(2)	0.1431(2)	0.39178(13)	0.0381(6)*
C1	0.2251(3)	0.0812(3)	0.09133(15)	0.0219(7)*
C2	0.4131(3)	0.1519(3)	0.17232(15)	0.0216(7)*
C3	0.4289(3)	0.1275(3)	0.35948(15)	0.0250(7)*
C4	0.1427(3)	0.0490(2)	0.23710(14)	0.0187(6)*
C10	0.1406(3)	0.3056(2)	0.26669(14)	0.0198(6)*
C20	0.2774(3)	-0.1622(2)	0.21979(15)	0.0207(6)*
C91	0.0161(3)	0.1489(3)	0.38498(16)	0.0241(7)*
C92	-0.0714(3)	0.1466(3)	0.33654(17)	0.0293(8)*
C93	-0.1668(3)	0.2088(3)	0.34301(19)	0.0364(9)*
C94	-0.1776(4)	0.2741(3)	0.3980(2)	0.0394(9)*
C95	-0.0930(4)	0.2771(3)	0.4471(2)	0.0390(9)*
C96	0.0029(3)	0.2148(3)	0.44031(18)	0.0316(8)*
C101	0.1942(3)	0.0598(3)	0.45442(15)	0.0228(7)*
C102	0.2663(3)	0.1458(3)	0.48521(17)	0.0314(8)*
C103	0.3064(4)	0.1524(3)	0.54664(18)	0.0381(9)*
C104	0.2774(4)	0.0711(4)	0.57740(18)	0.0401(10)*
C105	0.2088(4)	-0.0150(4)	0.54740(19)	0.0408(10)*
C106	0.1652(3)	-0.0207(3)	0.48645(17)	0.0319(8)*
C111	0.0532(3)	-0.0664(3)	0.35214(17)	0.0273(7)*

(b) dppm phenyl carbons

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C11	0.1789(3)	0.3489(2)	0.14481(15)	0.0210(7)*
C12	0.1059(3)	0.4332(3)	0.14553(17)	0.0303(8)*
C13	0.1323(4)	0.5158(3)	0.1148(2)	0.0406(10)*
C14	0.2291(4)	0.5146(3)	0.0824(2)	0.0401(10)*
C15	0.3025(4)	0.4321(3)	0.08120(19)	0.0366(9)*
C16	0.2771(3)	0.3489(3)	0.11213(16)	0.0274(7)*
C21	-0.0110(3)	0.2044(3)	0.16239(15)	0.0216(7)*

Table S-8. Atomic Coordinates and Displacement Parameters for **5** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C22	-0.0983(3)	0.2654(3)	0.18710(17)	0.0272(7)*
C23	-0.2159(3)	0.2365(3)	0.16733(18)	0.0322(8)*
C24	-0.2474(3)	0.1482(3)	0.12342(18)	0.0346(9)*
C25	-0.1610(3)	0.0859(3)	0.09932(18)	0.0366(9)*
C26	-0.0442(3)	0.1139(3)	0.11866(16)	0.0299(8)*
C31	0.2671(3)	0.3561(2)	0.38757(15)	0.0215(7)*
C32	0.3683(3)	0.3709(3)	0.42843(16)	0.0286(8)*
C33	0.3656(4)	0.4220(3)	0.48858(18)	0.0374(9)*
C34	0.2626(4)	0.4622(3)	0.50774(19)	0.0427(11)*
C35	0.1637(4)	0.4520(3)	0.4669(2)	0.0413(10)*
C36	0.1652(3)	0.3990(3)	0.40658(17)	0.0292(8)*
C41	0.3915(3)	0.3589(2)	0.28641(15)	0.0209(6)*
C42	0.3663(3)	0.4547(3)	0.26735(17)	0.0298(8)*
C43	0.4559(4)	0.5210(3)	0.2542(2)	0.0398(10)*
C44	0.5710(4)	0.4963(3)	0.2629(2)	0.0403(10)*
C45	0.5976(3)	0.4030(3)	0.28278(19)	0.0363(9)*
C46	0.5075(3)	0.3330(3)	0.29306(17)	0.0294(8)*
C51	0.4140(3)	-0.1401(3)	0.11657(15)	0.0245(7)*
C52	0.4721(3)	-0.0740(3)	0.08398(19)	0.0358(9)*
C53	0.5641(4)	-0.1099(4)	0.0504(2)	0.0453(11)*
C54	0.5979(4)	-0.2111(4)	0.0497(2)	0.0491(12)*
C55	0.5403(5)	-0.2780(4)	0.0811(2)	0.0568(13)*
C56	0.4483(4)	-0.2429(3)	0.11473(19)	0.0407(10)*
C61	0.1649(3)	-0.1631(3)	0.09943(15)	0.0233(7)*
C62	0.1827(3)	-0.2136(3)	0.04120(17)	0.0345(8)*
C63	0.0889(4)	-0.2631(4)	0.00073(19)	0.0440(10)*
C64	-0.0226(4)	-0.2616(3)	0.0180(2)	0.0407(10)*
C65	-0.0414(4)	-0.2114(4)	0.0752(2)	0.0548(13)*
C66	0.0514(4)	-0.1626(4)	0.1159(2)	0.0507(12)*
C71	0.5061(3)	-0.0920(3)	0.27266(15)	0.0210(6)*
C72	0.5711(3)	-0.0108(3)	0.25389(16)	0.0278(7)*
C73	0.6825(3)	-0.0278(3)	0.23550(18)	0.0350(9)*
C74	0.7307(3)	-0.1240(3)	0.23780(19)	0.0385(9)*
C75	0.6683(3)	-0.2040(3)	0.25743(19)	0.0355(9)*
C76	0.5569(3)	-0.1887(3)	0.27528(17)	0.0288(8)*
C81	0.3416(3)	-0.1580(3)	0.34899(15)	0.0232(7)*
C82	0.3989(3)	-0.1179(3)	0.40652(17)	0.0325(8)*
C83	0.3973(4)	-0.1762(4)	0.45398(19)	0.0409(10)*
C84	0.3385(4)	-0.2751(3)	0.4443(2)	0.0391(10)*

Table S-8. Atomic Coordinates and Displacement Parameters for **5** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C85	0.2813(4)	-0.3149(3)	0.3881(2)	0.0404(10)*
C86	0.2813(3)	-0.2577(3)	0.33976(18)	0.0318(8)*

(c) triflate ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
S	-0.03882(9)	0.60093(8)	0.27816(5)	0.0359(2)*
F1T	-0.0097(3)	0.6885(3)	0.39499(13)	0.0727(9)*
F2T	-0.0865(3)	0.5309(3)	0.37807(17)	0.0828(11)*
F3T	-0.1894(3)	0.6571(3)	0.35858(15)	0.0788(10)*
O1T	-0.1305(3)	0.5303(3)	0.24478(17)	0.0650(10)*
O2T	-0.0332(4)	0.7048(3)	0.26303(16)	0.0599(9)*
O3T	0.0721(3)	0.5550(2)	0.28548(15)	0.0467(7)*
C1T	-0.0836(4)	0.6216(4)	0.3561(2)	0.0463(11)*

(d) solvent dichloromethane atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Cl1S	0.80918(16)	0.55938(17)	0.08497(9)	0.0938(6)*
Cl2S	0.62408(15)	0.39578(16)	0.06415(9)	0.0902(5)*
C1S	0.7203(7)	0.4782(5)	0.1182(3)	0.085(2)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table S-9. Selected Interatomic Distances (Å) for **5**

(a) involving 'inner-core' atoms

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Os	Rh	2.8831(3)	P4	C20	1.829(3)
Os	P1	2.3613(8)	P4	C71	1.840(3)
Os	P3	2.3664(8)	P4	C81	1.828(3)
Os	C1	1.870(3)	P5	C91	1.847(3)
Os	C2	1.910(3)	P5	C101	1.822(3)
Os	C4	2.092(3)	P5	C111	1.833(3)
Rh	P2	2.3418(8)	O1	C1	1.149(4)
Rh	P4	2.3434(8)	O2	C2	1.140(4)
Rh	P5	2.4458(9)	O3	C3	1.147(4)
Rh	C3	1.885(4)	C91	C92	1.400(5)
Rh	C4	2.172(3)	C91	C96	1.391(5)
P1	C10	1.836(3)	C92	C93	1.387(5)
P1	C11	1.836(3)	C93	C94	1.376(6)
P1	C21	1.837(3)	C94	C95	1.387(6)
P1	P2	2.9568(11)†	C95	C96	1.392(5)
P2	C10	1.819(3)	C101	C102	1.389(5)
P2	C31	1.839(3)	C101	C106	1.388(5)
P2	C41	1.836(3)	C102	C103	1.385(5)
P3	C20	1.832(3)	C103	C104	1.378(6)
P3	C51	1.836(3)	C104	C105	1.363(6)
P3	C61	1.834(3)	C105	C106	1.387(5)
P3	P4	2.9855(11)†			

†Nonbonded distance.

(b) involving dppm phenyl carbons

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C11	C12	1.394(5)	C31	C36	1.383(5)
C11	C16	1.392(5)	C32	C33	1.386(5)
C12	C13	1.385(5)	C33	C34	1.383(6)
C13	C14	1.373(6)	C34	C35	1.376(6)
C14	C15	1.378(6)	C35	C36	1.395(5)
C15	C16	1.391(5)	C41	C42	1.395(5)
C21	C22	1.391(5)	C41	C46	1.388(5)
C21	C26	1.395(5)	C42	C43	1.384(5)
C22	C23	1.395(5)	C43	C44	1.377(6)
C23	C24	1.374(6)	C44	C45	1.376(6)
C24	C25	1.389(5)	C45	C46	1.394(5)
C25	C26	1.382(5)	C51	C52	1.383(5)
C31	C32	1.394(5)	C51	C56	1.386(5)

Table S-9. Selected Interatomic Distances for **5** (continued)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C52	C53	1.394(5)	C71	C76	1.399(5)
C53	C54	1.367(7)	C72	C73	1.390(5)
C54	C55	1.371(7)	C73	C74	1.379(6)
C55	C56	1.392(6)	C74	C75	1.372(6)
C61	C62	1.380(5)	C75	C76	1.384(5)
C61	C66	1.382(5)	C81	C82	1.387(5)
C62	C63	1.394(5)	C81	C86	1.398(5)
C63	C64	1.366(6)	C82	C83	1.387(5)
C64	C65	1.362(6)	C83	C84	1.381(6)
C65	C66	1.385(6)	C84	C85	1.362(6)
C71	C72	1.390(5)	C85	C86	1.393(5)

(c) within the triflate ion

Atom1	Atom2	Distance	Atom1	Atom2	Distance
S	O1T	1.423(3)	F1T	C1T	1.325(6)
S	O2T	1.423(3)	F2T	C1T	1.331(5)
S	O3T	1.434(3)	F3T	C1T	1.319(5)
S	C1T	1.823(5)			

(d) within the solvent dichloromethane molecules

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Cl1S	C1S	1.716(7)	Cl2S	C1S	1.733(7)

Table S-10. Selected Interatomic Angles (deg) for **5**

(a) involving 'inner-core' atoms

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Rh	Os	P1	92.61(2)	C10	P2	C31	104.36(15)
Rh	Os	P3	92.99(2)	C10	P2	C41	105.27(15)
Rh	Os	C1	174.08(10)	C31	P2	C41	97.49(15)
Rh	Os	C2	94.75(10)	Os	P3	C20	114.22(11)
Rh	Os	C4	48.65(8)	Os	P3	C51	119.29(12)
P1	Os	P3	153.26(3)	Os	P3	C61	111.49(11)
P1	Os	C1	86.44(10)	C20	P3	C51	105.78(15)
P1	Os	C2	102.78(10)	C20	P3	C61	102.38(15)
P1	Os	C4	79.54(9)	C51	P3	C61	101.70(16)
P3	Os	C1	85.32(10)	Rh	P4	C20	114.48(10)
P3	Os	C2	102.77(10)	Rh	P4	C71	117.11(11)
P3	Os	C4	84.68(9)	Rh	P4	C81	117.31(11)
C1	Os	C2	91.16(14)	C20	P4	C71	100.54(15)
C1	Os	C4	125.47(13)	C20	P4	C81	104.00(15)
C2	Os	C4	143.28(13)	C71	P4	C81	101.02(15)
Os	Rh	P2	82.74(2)	Rh	P5	C91	120.24(11)
Os	Rh	P4	88.65(2)	Rh	P5	C101	114.75(11)
Os	Rh	P5	127.97(2)	Rh	P5	C111	115.25(12)
Os	Rh	C3	126.76(11)	C91	P5	C101	101.65(16)
Os	Rh	C4	46.29(8)	C91	P5	C111	98.69(16)
P2	Rh	P4	162.78(3)	C101	P5	C111	103.67(16)
P2	Rh	P5	99.83(3)	Os	C1	O1	177.6(3)
P2	Rh	C3	87.88(10)	Os	C2	O2	179.0(3)
P2	Rh	C4	94.76(9)	Rh	C3	O3	177.1(3)
P4	Rh	P5	97.23(3)	Os	C4	Rh	85.06(11)
P4	Rh	C3	85.37(10)	P1	C10	P2	107.97(16)
P4	Rh	C4	89.94(9)	P3	C20	P4	109.23(16)
P5	Rh	C3	105.27(11)	P5	C91	C92	119.7(3)
P5	Rh	C4	81.91(8)	P5	C91	C96	122.8(3)
C3	Rh	C4	171.85(14)	C92	C91	C96	117.5(3)
Os	P1	C10	113.43(10)	C91	C92	C93	121.4(4)
Os	P1	C11	119.14(11)	C92	C93	C94	120.1(4)
Os	P1	C21	112.22(11)	C93	C94	C95	119.8(4)
C10	P1	C11	104.75(15)	C94	C95	C96	119.8(4)
C10	P1	C21	101.89(15)	C91	C96	C95	121.3(4)
C11	P1	C21	103.56(15)	P5	C101	C102	118.2(3)
Rh	P2	C10	111.53(11)	P5	C101	C106	123.5(3)
Rh	P2	C31	115.38(10)	C102	C101	C106	118.3(3)
Rh	P2	C41	120.79(11)	C101	C102	C103	121.0(3)

Table S-10. Selected Interatomic Angles for **5** (continued)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C102	C103	C104	119.7(4)	C104	C105	C106	120.7(4)
C103	C104	C105	119.9(4)	C101	C106	C105	120.3(4)

(b) involving dppm phenyl carbons

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
P1	C11	C12	120.1(3)	P3	C51	C52	118.9(3)
P1	C11	C16	121.0(2)	P3	C51	C56	122.1(3)
C12	C11	C16	118.8(3)	C52	C51	C56	118.7(3)
C11	C12	C13	120.2(4)	C51	C52	C53	120.6(4)
C12	C13	C14	120.4(4)	C52	C53	C54	120.0(4)
C13	C14	C15	120.3(3)	C53	C54	C55	120.1(4)
C14	C15	C16	119.8(4)	C54	C55	C56	120.3(4)
C11	C16	C15	120.5(3)	C51	C56	C55	120.3(4)
P1	C21	C22	121.4(3)	P3	C61	C62	122.5(3)
P1	C21	C26	120.0(3)	P3	C61	C66	119.6(3)
C22	C21	C26	118.5(3)	C62	C61	C66	117.9(3)
C21	C22	C23	120.0(3)	C61	C62	C63	120.7(4)
C22	C23	C24	120.9(3)	C62	C63	C64	120.5(4)
C23	C24	C25	119.4(3)	C63	C64	C65	119.4(4)
C24	C25	C26	120.0(4)	C64	C65	C66	120.6(4)
C21	C26	C25	121.1(3)	C61	C66	C65	121.0(4)
P2	C31	C32	118.4(3)	P4	C71	C72	121.3(3)
P2	C31	C36	122.4(3)	P4	C71	C76	119.7(3)
C32	C31	C36	119.1(3)	C72	C71	C76	118.9(3)
C31	C32	C33	120.7(4)	C71	C72	C73	120.1(3)
C32	C33	C34	119.8(4)	C72	C73	C74	120.2(4)
C33	C34	C35	119.8(4)	C73	C74	C75	120.2(4)
C34	C35	C36	120.7(4)	C74	C75	C76	120.3(4)
C31	C36	C35	119.8(4)	C71	C76	C75	120.3(3)
P2	C41	C42	119.5(3)	P4	C81	C82	117.7(3)
P2	C41	C46	121.3(3)	P4	C81	C86	123.3(3)
C42	C41	C46	118.8(3)	C82	C81	C86	119.0(3)
C41	C42	C43	120.0(4)	C81	C82	C83	120.5(4)
C42	C43	C44	120.7(4)	C82	C83	C84	120.2(4)
C43	C44	C45	120.0(4)	C83	C84	C85	119.8(4)
C44	C45	C46	119.8(4)	C84	C85	C86	121.2(4)
C41	C46	C45	120.6(3)	C81	C86	C85	119.4(4)

Table S-10. Selected Interatomic Angles for **5** (continued)

(c) within the triflate ion

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
O1T	S	O2T	115.4(2)	S	C1T	F1T	112.3(3)
O1T	S	O3T	114.7(2)	S	C1T	F2T	110.8(3)
O1T	S	C1T	102.6(2)	S	C1T	F3T	112.4(3)
O2T	S	O3T	115.0(2)	F1T	C1T	F2T	106.2(4)
O2T	S	C1T	103.8(2)	F1T	C1T	F3T	108.3(4)
O3T	S	C1T	102.8(2)	F2T	C1T	F3T	106.5(4)

(d) within the solvent dichloromethane molecules

Atom1	Atom2	Atom3	Angle
Cl1S	C1S	Cl2S	112.2(4)

Table S-11. Anisotropic Displacement Parameters (U_{ij}, Å^2) for **5**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Os	0.01637(7)	0.01645(7)	0.01490(7)	0.00158(4)	0.00256(4)	0.00232(4)
Rh	0.01903(12)	0.01541(12)	0.01462(12)	0.00147(9)	0.00016(9)	0.00109(9)
P1	0.0171(4)	0.0177(4)	0.0167(4)	0.0027(3)	0.0020(3)	0.0019(3)
P2	0.0192(4)	0.0162(4)	0.0167(4)	0.0008(3)	0.0024(3)	0.0008(3)
P3	0.0186(4)	0.0176(4)	0.0186(4)	-0.0002(3)	0.0004(3)	0.0023(3)
P4	0.0204(4)	0.0167(4)	0.0189(4)	0.0033(3)	0.0002(3)	0.0014(3)
P5	0.0212(4)	0.0199(4)	0.0196(4)	0.0030(3)	0.0027(3)	-0.0009(3)
O1	0.0405(16)	0.0449(16)	0.0201(14)	0.0059(11)	0.0048(11)	0.0067(12)
O2	0.0229(14)	0.0378(15)	0.0446(16)	0.0044(12)	0.0073(11)	-0.0010(11)
O3	0.0347(15)	0.0401(15)	0.0351(15)	0.0055(12)	-0.0153(12)	-0.0035(12)
C1	0.0207(16)	0.0231(16)	0.0217(18)	0.0015(13)	0.0039(13)	0.0018(13)
C2	0.0172(16)	0.0244(16)	0.0225(17)	-0.0002(13)	0.0042(12)	0.0030(13)
C3	0.035(2)	0.0190(16)	0.0210(17)	0.0034(13)	0.0012(14)	0.0024(14)
C4	0.0208(16)	0.0195(15)	0.0147(15)	0.0010(12)	0.0007(12)	-0.0003(12)
C10	0.0209(16)	0.0190(15)	0.0190(16)	0.0014(12)	0.0034(12)	0.0017(12)
C11	0.0253(17)	0.0192(15)	0.0190(16)	0.0054(12)	0.0008(13)	0.0018(13)
C12	0.036(2)	0.0263(18)	0.032(2)	0.0087(15)	0.0075(15)	0.0088(15)
C13	0.051(3)	0.0270(19)	0.049(3)	0.0153(18)	0.010(2)	0.0141(18)
C14	0.050(3)	0.030(2)	0.044(2)	0.0190(18)	0.0070(19)	-0.0003(18)
C15	0.034(2)	0.038(2)	0.043(2)	0.0165(18)	0.0136(17)	0.0004(17)
C16	0.0275(18)	0.0281(18)	0.0290(19)	0.0101(15)	0.0057(14)	0.0029(14)
C20	0.0223(16)	0.0160(14)	0.0227(17)	0.0021(12)	-0.0003(13)	0.0004(12)
C21	0.0175(16)	0.0260(16)	0.0220(17)	0.0065(13)	0.0021(12)	0.0010(13)
C22	0.0227(17)	0.0265(17)	0.0319(19)	0.0037(15)	0.0015(14)	0.0035(14)
C23	0.0212(18)	0.039(2)	0.036(2)	0.0031(17)	0.0056(15)	0.0055(15)
C24	0.0158(17)	0.048(2)	0.038(2)	0.0038(18)	0.0031(15)	-0.0026(16)
C25	0.030(2)	0.047(2)	0.027(2)	-0.0100(17)	0.0005(15)	-0.0034(17)
C26	0.0231(18)	0.039(2)	0.0243(18)	-0.0050(15)	0.0024(14)	0.0012(15)
C31	0.0304(18)	0.0150(14)	0.0183(16)	0.0003(12)	0.0058(13)	-0.0028(13)
C32	0.0322(19)	0.0258(17)	0.0251(18)	-0.0023(14)	0.0023(14)	-0.0030(15)
C33	0.050(2)	0.034(2)	0.0241(19)	-0.0002(16)	0.0023(17)	-0.0107(18)
C34	0.066(3)	0.033(2)	0.025(2)	-0.0086(16)	0.0160(19)	-0.015(2)
C35	0.046(2)	0.035(2)	0.040(2)	-0.0084(18)	0.0218(19)	-0.0007(18)
C36	0.034(2)	0.0240(17)	0.0287(19)	-0.0011(14)	0.0087(15)	-0.0004(15)
C41	0.0239(17)	0.0195(15)	0.0180(16)	0.0002(12)	0.0033(12)	-0.0026(13)
C42	0.0317(19)	0.0231(17)	0.034(2)	0.0040(15)	0.0011(15)	-0.0008(15)
C43	0.048(3)	0.0255(19)	0.046(2)	0.0110(17)	0.0035(19)	-0.0075(17)
C44	0.037(2)	0.037(2)	0.045(2)	0.0022(18)	0.0117(18)	-0.0165(18)
C45	0.0249(19)	0.037(2)	0.046(2)	0.0028(18)	0.0109(16)	-0.0037(16)
C46	0.0282(19)	0.0278(18)	0.032(2)	0.0045(15)	0.0044(15)	0.0015(15)

Table S-11. Anisotropic Displacement Parameters for **5** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C51	0.0212(17)	0.0281(17)	0.0210(17)	-0.0057(13)	-0.0002(13)	0.0069(14)
C52	0.031(2)	0.032(2)	0.041(2)	-0.0082(17)	0.0124(17)	0.0024(16)
C53	0.034(2)	0.052(3)	0.046(3)	-0.009(2)	0.0164(19)	-0.0034(19)
C54	0.033(2)	0.070(3)	0.041(3)	-0.009(2)	0.0094(19)	0.018(2)
C55	0.063(3)	0.059(3)	0.052(3)	0.006(2)	0.014(2)	0.040(3)
C56	0.047(2)	0.040(2)	0.037(2)	0.0059(18)	0.0091(18)	0.0214(19)
C61	0.0262(17)	0.0185(15)	0.0232(17)	0.0014(13)	-0.0047(13)	0.0006(13)
C62	0.032(2)	0.042(2)	0.0264(19)	0.0011(16)	-0.0006(15)	-0.0021(17)
C63	0.050(3)	0.054(3)	0.021(2)	-0.0017(18)	-0.0081(17)	-0.008(2)
C64	0.040(2)	0.036(2)	0.041(2)	0.0032(18)	-0.0185(18)	-0.0047(18)
C65	0.024(2)	0.067(3)	0.061(3)	-0.019(2)	-0.006(2)	-0.006(2)
C66	0.026(2)	0.066(3)	0.048(3)	-0.025(2)	0.0008(18)	-0.001(2)
C71	0.0187(16)	0.0241(16)	0.0186(16)	0.0006(13)	-0.0013(12)	0.0028(13)
C72	0.0270(18)	0.0278(18)	0.0287(19)	0.0065(15)	-0.0003(14)	0.0026(14)
C73	0.0263(19)	0.044(2)	0.036(2)	0.0100(18)	0.0051(16)	-0.0031(16)
C74	0.0243(19)	0.051(2)	0.038(2)	0.0031(19)	0.0036(16)	0.0038(17)
C75	0.029(2)	0.0310(19)	0.044(2)	-0.0001(17)	-0.0006(17)	0.0111(16)
C76	0.0271(18)	0.0237(17)	0.034(2)	0.0018(15)	-0.0018(15)	0.0018(14)
C81	0.0245(17)	0.0239(16)	0.0238(17)	0.0083(13)	0.0052(13)	0.0069(13)
C82	0.036(2)	0.0322(19)	0.031(2)	0.0107(16)	0.0002(16)	-0.0004(16)
C83	0.045(2)	0.054(3)	0.026(2)	0.0147(19)	-0.0001(17)	0.008(2)
C84	0.036(2)	0.050(2)	0.041(2)	0.028(2)	0.0134(18)	0.0088(18)
C85	0.044(2)	0.034(2)	0.049(3)	0.0215(19)	0.0127(19)	0.0016(18)
C86	0.034(2)	0.0269(18)	0.037(2)	0.0107(16)	0.0058(16)	0.0042(15)
C91	0.0242(17)	0.0220(16)	0.0282(18)	0.0081(14)	0.0069(14)	-0.0002(13)
C92	0.0270(18)	0.038(2)	0.0246(18)	0.0086(15)	0.0077(14)	0.0044(15)
C93	0.032(2)	0.046(2)	0.037(2)	0.0192(18)	0.0063(16)	0.0101(17)
C94	0.035(2)	0.034(2)	0.053(3)	0.0107(19)	0.0143(19)	0.0133(17)
C95	0.040(2)	0.0265(19)	0.048(3)	-0.0043(17)	0.0098(19)	0.0064(17)
C96	0.0311(19)	0.0264(18)	0.035(2)	0.0000(15)	0.0031(15)	0.0004(15)
C101	0.0210(16)	0.0267(17)	0.0218(17)	0.0045(13)	0.0062(13)	0.0040(13)
C102	0.034(2)	0.0326(19)	0.0276(19)	0.0065(15)	0.0015(15)	-0.0050(16)
C103	0.038(2)	0.044(2)	0.028(2)	0.0001(17)	-0.0032(16)	-0.0061(18)
C104	0.039(2)	0.059(3)	0.0228(19)	0.0091(18)	0.0018(16)	0.002(2)
C105	0.045(2)	0.049(2)	0.032(2)	0.0205(19)	0.0049(18)	-0.003(2)
C106	0.032(2)	0.034(2)	0.030(2)	0.0094(16)	0.0020(15)	-0.0049(16)
C111	0.0251(18)	0.0241(17)	0.0318(19)	0.0028(14)	0.0040(14)	-0.0042(14)
S	0.0401(5)	0.0303(5)	0.0339(5)	-0.0038(4)	-0.0004(4)	0.0068(4)
F1T	0.078(2)	0.092(2)	0.0373(16)	-0.0148(15)	-0.0026(15)	-0.0017(18)
F2T	0.096(3)	0.084(2)	0.087(3)	0.045(2)	0.043(2)	0.025(2)

Table S-11. Anisotropic Displacement Parameters for **5** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F3T	0.0576(19)	0.111(3)	0.068(2)	0.0002(19)	0.0161(16)	0.0436(19)
O1T	0.050(2)	0.071(2)	0.061(2)	-0.0193(19)	-0.0040(17)	-0.0062(18)
O2T	0.089(3)	0.0436(18)	0.050(2)	0.0125(15)	0.0058(18)	0.0188(18)
O3T	0.0439(18)	0.0414(16)	0.0531(19)	-0.0018(14)	0.0076(14)	0.0128(14)
C1T	0.043(3)	0.053(3)	0.044(3)	0.006(2)	0.006(2)	0.015(2)
Cl1S	0.0802(11)	0.1151(14)	0.0983(13)	0.0530(12)	0.0081(9)	0.0102(10)
Cl2S	0.0630(9)	0.1143(14)	0.0876(12)	0.0076(10)	-0.0104(8)	0.0148(9)
C1S	0.112(6)	0.086(5)	0.058(4)	0.028(3)	-0.008(4)	-0.004(4)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table S-12. Derived Coordinates and Displacement Parameters for Hydrogen Atoms of **5**

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H4A	0.1173	-0.0274	0.2301	0.022
H4B	0.0753	0.0943	0.2417	0.022
H10A	0.1361	0.3832	0.2688	0.024
H10B	0.0709	0.2780	0.2838	0.024
H12	0.0380	0.4339	0.1672	0.036
H13	0.0830	0.5736	0.1161	0.049
H14	0.2455	0.5708	0.0606	0.048
H15	0.3702	0.4321	0.0593	0.044
H16	0.3273	0.2917	0.1109	0.033
H20A	0.1945	-0.1776	0.2264	0.025
H20B	0.3154	-0.2304	0.2118	0.025
H22	-0.0778	0.3267	0.2175	0.033
H23	-0.2750	0.2785	0.1845	0.039
H24	-0.3277	0.1299	0.1097	0.041
H25	-0.1820	0.0239	0.0695	0.044
H26	0.0144	0.0709	0.1019	0.036
H32	0.4399	0.3457	0.4149	0.034
H33	0.4343	0.4293	0.5165	0.045
H34	0.2602	0.4968	0.5490	0.051
H35	0.0936	0.4813	0.4798	0.050
H36	0.0965	0.3924	0.3787	0.035
H42	0.2875	0.4744	0.2634	0.036
H43	0.4379	0.5841	0.2391	0.048
H44	0.6321	0.5436	0.2552	0.048
H45	0.6770	0.3864	0.2895	0.044
H46	0.5257	0.2670	0.3047	0.035
H52	0.4491	-0.0035	0.0845	0.043
H53	0.6032	-0.0640	0.0281	0.054
H54	0.6614	-0.2351	0.0273	0.059
H55	0.5632	-0.3487	0.0799	0.068
H56	0.4090	-0.2896	0.1365	0.049
H62	0.2598	-0.2148	0.0286	0.041
H63	0.1025	-0.2980	-0.0391	0.053
H64	-0.0864	-0.2953	-0.0097	0.049
H65	-0.1188	-0.2098	0.0873	0.066
H66	0.0368	-0.1284	0.1558	0.061
H72	0.5393	0.0564	0.2536	0.033
H73	0.7255	0.0270	0.2213	0.042
H74	0.8073	-0.1349	0.2258	0.046
H75	0.7018	-0.2703	0.2588	0.043

Table S-12. Derived Parameters for Hydrogen Atoms of **5** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H76	0.5146	-0.2441	0.2894	0.035
H82	0.4396	-0.0499	0.4135	0.039
H83	0.4367	-0.1480	0.4932	0.049
H84	0.3380	-0.3153	0.4768	0.047
H85	0.2406	-0.3828	0.3818	0.049
H86	0.2407	-0.2863	0.3008	0.038
H92	-0.0653	0.1015	0.2984	0.035
H93	-0.2249	0.2064	0.3094	0.044
H94	-0.2427	0.3171	0.4023	0.047
H95	-0.1005	0.3215	0.4853	0.047
H96	0.0604	0.2173	0.4742	0.038
H102	0.2885	0.2010	0.4638	0.038
H103	0.3537	0.2127	0.5675	0.046
H104	0.3052	0.0750	0.6195	0.048
H105	0.1905	-0.0716	0.5685	0.049
H106	0.1154	-0.0800	0.4665	0.038
H11A	-0.0083	-0.0707	0.3798	0.033
H11B	0.1050	-0.1247	0.3546	0.033
H11C	0.0174	-0.0719	0.3098	0.033
H1SA	0.6749	0.5224	0.1476	0.102
H1SB	0.7695	0.4341	0.1415	0.102

Table S-13. Crystallographic Experimental Details for **8**

A. Crystal Data	
Formula[a]	$C_{64.25}H_{69.5}Cl_{4.5}F_3O_{10}OsP_5RhS$
formula weight	1698.24
crystal dimensions (mm)	0.37 × 0.36 × 0.19
crystal system	triclinic
space group	$P\bar{1}$ (No. 2)
unit cell parameters[b]	
a (Å)	17.7754 (12)
b (Å)	18.5573 (12)
c (Å)	24.0598 (16)
α (deg)	109.2759 (11)
β (deg)	102.0736 (11)
γ (deg)	104.4936 (12)
V (Å^3)	6873.5 (8)
Z	4
ρ_{calcd} (g cm^{-3})	1.641
μ (mm^{-1})	2.472
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/SMART 1000 CCD[c]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.2°) (20 s exposures)
data collection 2θ limit (deg)	52.78
total data collected	48707 ($-22 \le h \le 22$, $-23 \le k \le 23$, $-30 \le l \le 30$)
independent reflections	27682 (R_{int} = 0.0302)
number of observed reflections (*NO*)	22158 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	direct methods (*SIR97*[d])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[e])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.6509–0.4615
data/restraints/parameters	27682 [$F_o^2 \ge -3\sigma(F_o^2)$] / 76[f] / 1361
goodness-of-fit (*S*)[g]	1.057 [$F_o^2 \ge -3\sigma(F_o^2)$]
final R indices[h]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0477
wR_2 [$F_o^2 \ge -3\sigma(F_o^2)$]	0.1369
largest difference peak and hole	4.272 and –2.104 e Å^{-3}

(continued)

Table S-13. Crystallographic Experimental Details for **8** (continued)

[a]The SQUEEZE routine (Sluis, P. van der; Spek, A. L. *Acta Crystallogr.* **1990**, *A46*, 194–201) of the program *PLATON* (Spek, A. L. *Acta Crystogr.* **1990**, *A46*, C-34) was implemented to remove the contributions of the disordered solvent dichloromethane and diethylether molecules to the observed structure factors. A 1512 Å^3 void centred at ($^1/_2$, 0 $^1/_2$) with scattering associated with a total density equivalent to 555 electrons was found. On the basis of the structural modelling of the disordered solvent molecules prior to implementing SQUEEZE, the recovered electron density was atttributed to nine equivalents of dichloromethane and four equivalents of diethylether per unit cell volume.

[b]Obtained from least-squares refinement of 7722 reflections with $4.74° < 2\theta < 52.72°$.

[c]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

[d]Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *J. Appl. Cryst.* **1999**, *32*, 115–119.

[e]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and all goodnesses of fit S are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[f]The following distance restraints were applied to the disordered triflate anions: S–C, 1.800(2) Å; S–O, 1.450(2) Å; C–F 1.350(2) Å; O···O, 2.370(2) Å; F···F, 2.200(2) Å; O···F, 3.040(2) Å.

[g]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0892P)^2]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[h]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table S-14. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **8**

(a) 'inner-core' atoms of molecule A

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Os	-0.050665(12)	-0.068998(12)	0.264715(8)	0.02626(6)*
Rh	-0.14606(2)	-0.10219(2)	0.141078(16)	0.02472(9)*
P1	-0.11262(8)	0.02382(8)	0.30908(5)	0.0267(3)*
P2	-0.19792(8)	0.00083(8)	0.18058(6)	0.0259(3)*
P3	0.01660(8)	-0.15974(8)	0.22663(6)	0.0293(3)*
P4	-0.08593(8)	-0.19566(8)	0.09709(6)	0.0291(3)*
P5	-0.25211(9)	-0.20646(8)	0.13946(6)	0.0336(3)*
O1	0.0933(3)	0.0207(3)	0.38250(18)	0.0592(13)*
O2	-0.1505(3)	-0.1935(3)	0.3040(2)	0.0544(12)*
O3	-0.1810(3)	-0.0793(3)	0.02049(18)	0.0507(11)*
O5	-0.3222(2)	-0.2692(2)	0.07503(19)	0.0474(10)*
O6	-0.3028(2)	-0.1767(2)	0.1841(2)	0.0469(10)*
O7	-0.2211(2)	-0.2713(2)	0.15807(18)	0.0413(9)*
C1	0.0382(3)	-0.0143(3)	0.3375(2)	0.0364(12)*
C2	-0.1153(3)	-0.1475(3)	0.2894(2)	0.0331(12)*
C3	-0.1688(3)	-0.0885(3)	0.0658(2)	0.0358(12)*
C4	-0.0254(3)	-0.0165(3)	0.2025(2)	0.0270(10)*
C5	-0.3633(4)	-0.2421(4)	0.0326(3)	0.0539(17)*
C6	-0.3801(4)	-0.2263(5)	0.1823(4)	0.065(2)*
C7	-0.2696(5)	-0.3523(4)	0.1469(4)	0.065(2)*
C10	-0.2086(3)	0.0141(3)	0.2564(2)	0.0282(10)*
C20	-0.0414(3)	-0.2375(3)	0.1484(2)	0.0331(11)*

(b) dppm phenyl carbons of molecule A

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C11	-0.1416(3)	0.0109(3)	0.3745(2)	0.0286(11)*
C12	-0.2199(4)	-0.0288(3)	0.3711(2)	0.0367(12)*
C13	-0.2368(4)	-0.0398(3)	0.4224(3)	0.0388(13)*
C14	-0.1749(4)	-0.0105(4)	0.4775(3)	0.0463(15)*
C15	-0.0954(4)	0.0295(4)	0.4821(3)	0.0495(16)*
C16	-0.0790(4)	0.0399(4)	0.4318(3)	0.0484(15)*
C21	-0.0523(3)	0.1327(3)	0.3434(2)	0.0304(11)*
C22	-0.0829(4)	0.1880(4)	0.3769(3)	0.0462(14)*
C23	-0.0371(4)	0.2706(4)	0.4032(3)	0.0569(18)*
C24	0.0346(5)	0.2970(4)	0.3941(3)	0.0603(19)*
C25	0.0657(4)	0.2437(4)	0.3601(3)	0.0573(18)*
C26	0.0218(4)	0.1606(4)	0.3353(3)	0.0404(13)*
C31	-0.1411(3)	0.1028(3)	0.1888(2)	0.0315(11)*
C32	-0.0941(4)	0.1139(3)	0.1515(2)	0.0382(13)*

Table S-14. Atomic Coordinates and Displacement Parameters for **8** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C33	-0.0573(4)	0.1918(4)	0.1549(3)	0.0542(17)*
C34	-0.0654(5)	0.2583(4)	0.1968(3)	0.0610(19)*
C35	-0.1113(5)	0.2491(4)	0.2350(3)	0.0543(17)*
C36	-0.1504(4)	0.1710(3)	0.2300(3)	0.0419(13)*
C41	-0.3003(3)	-0.0162(3)	0.1315(2)	0.0307(11)*
C42	-0.3080(4)	-0.0011(4)	0.0782(3)	0.0399(13)*
C43	-0.3848(4)	-0.0200(4)	0.0372(3)	0.0499(16)*
C44	-0.4546(4)	-0.0528(4)	0.0501(3)	0.0484(16)*
C45	-0.4480(4)	-0.0658(4)	0.1041(3)	0.0441(14)*
C46	-0.3709(3)	-0.0477(3)	0.1446(3)	0.0367(12)*
C51	0.0304(4)	-0.2202(4)	0.2721(3)	0.0399(13)*
C52	-0.0110(4)	-0.3015(4)	0.2494(3)	0.0537(17)*
C53	-0.0006(5)	-0.3445(4)	0.2868(3)	0.067(2)*
C54	0.0483(6)	-0.3058(5)	0.3460(3)	0.075(3)*
C55	0.0910(8)	-0.2259(6)	0.3674(4)	0.131(5)*
C56	0.0841(6)	-0.1820(5)	0.3306(3)	0.089(3)*
C61	0.1197(3)	-0.1200(4)	0.2230(3)	0.0391(13)*
C62	0.1604(4)	-0.1738(5)	0.2037(3)	0.0579(17)*
C63	0.2384(5)	-0.1457(6)	0.2005(4)	0.068(2)*
C64	0.2777(4)	-0.0650(6)	0.2167(4)	0.071(2)*
C65	0.2381(4)	-0.0100(5)	0.2371(4)	0.066(2)*
C66	0.1593(4)	-0.0374(4)	0.2414(3)	0.0467(15)*
C71	-0.0065(4)	-0.1627(4)	0.0641(2)	0.0392(13)*
C72	0.0280(4)	-0.2173(4)	0.0360(3)	0.0567(17)*
C73	0.0883(5)	-0.1947(7)	0.0095(4)	0.081(3)*
C74	0.1112(5)	-0.1185(6)	0.0099(3)	0.083(3)*
C75	0.0762(4)	-0.0624(5)	0.0372(3)	0.066(2)*
C76	0.0180(4)	-0.0855(4)	0.0645(3)	0.0495(16)*
C81	-0.1570(3)	-0.2869(3)	0.0301(2)	0.0345(12)*
C82	-0.1915(4)	-0.2788(4)	-0.0242(3)	0.0533(17)*
C83	-0.2476(5)	-0.3447(5)	-0.0758(3)	0.078(3)*
C84	-0.2700(5)	-0.4206(5)	-0.0737(4)	0.085(3)*
C85	-0.2338(6)	-0.4314(5)	-0.0217(4)	0.086(3)*
C86	-0.1781(5)	-0.3640(4)	0.0305(3)	0.0573(18)*

Table S-14. Atomic Coordinates and Displacement Parameters for **8** (continued)

(c) 'inner-core' atoms of molecule B

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Os	-0.306150(11)	0.419147(11)	0.379308(8)	0.02393(6)*
Rh	-0.43778(2)	0.38089(2)	0.270348(16)	0.02494(9)*
P1	-0.38347(8)	0.31448(8)	0.40116(5)	0.0258(3)*
P2	-0.52076(8)	0.27749(8)	0.28802(6)	0.0269(3)*
P3	-0.22022(8)	0.52117(8)	0.35990(6)	0.0275(3)*
P4	-0.36437(8)	0.49240(8)	0.25514(6)	0.0285(3)*
P5	-0.39515(9)	0.28780(8)	0.20496(6)	0.0314(3)*
O1	-0.2090(3)	0.5119(2)	0.51578(17)	0.0489(11)*
O2	-0.2069(3)	0.3093(3)	0.3376(2)	0.0503(11)*
O3	-0.5931(3)	0.3908(3)	0.1970(2)	0.0657(14)*
O5	-0.4562(3)	0.2249(2)	0.13704(17)	0.0479(11)*
O6	-0.3788(3)	0.2222(2)	0.23017(17)	0.0397(9)*
O7	-0.3139(3)	0.3282(2)	0.19241(19)	0.0470(10)*
C1	-0.2450(3)	0.4750(3)	0.4633(2)	0.0324(11)*
C2	-0.2424(3)	0.3506(3)	0.3528(2)	0.0317(11)*
C3	-0.5337(4)	0.3863(4)	0.2238(3)	0.0414(14)*
C4	-0.4042(3)	0.4617(3)	0.36791(19)	0.0268(10)*
C5	-0.5011(4)	0.2509(4)	0.0955(3)	0.0510(16)*
C6	-0.3748(5)	0.1444(4)	0.1942(3)	0.0551(17)*
C7	-0.2853(5)	0.2863(4)	0.1429(4)	0.064(2)*
C10	-0.4634(3)	0.2346(3)	0.3326(2)	0.0304(11)*
C20	-0.2540(3)	0.5150(3)	0.2804(2)	0.0319(11)*

(d) dppm phenyl carbons of molecule B

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C11	-0.3222(3)	0.2590(3)	0.4279(2)	0.0309(11)*
C12	-0.3407(4)	0.1780(4)	0.3974(3)	0.0566(18)*
C13	-0.2914(5)	0.1380(4)	0.4189(4)	0.069(2)*
C14	-0.2255(4)	0.1800(4)	0.4704(3)	0.0550(17)*
C15	-0.2070(6)	0.2605(5)	0.5005(4)	0.095(4)*
C16	-0.2566(5)	0.3022(5)	0.4819(4)	0.083(3)*
C21	-0.4364(3)	0.3364(3)	0.4584(2)	0.0290(11)*
C22	-0.4358(4)	0.4135(4)	0.4897(2)	0.0372(12)*
C23	-0.4832(4)	0.4258(4)	0.5287(3)	0.0480(15)*
C24	-0.5295(4)	0.3622(5)	0.5382(3)	0.0560(17)*
C25	-0.5273(5)	0.2844(4)	0.5088(3)	0.0575(18)*
C26	-0.4808(4)	0.2725(4)	0.4702(3)	0.0486(15)*
C31	-0.5954(3)	0.3026(4)	0.3270(2)	0.0380(13)*
C32	-0.6399(4)	0.2440(4)	0.3451(3)	0.0515(16)*

Table S-14. Atomic Coordinates and Displacement Parameters for **8** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C33	-0.6966(5)	0.2608(6)	0.3742(3)	0.068(2)*
C34	-0.7090(5)	0.3325(6)	0.3878(3)	0.071(2)*
C35	-0.6671(4)	0.3905(5)	0.3677(3)	0.0619(19)*
C36	-0.6103(4)	0.3734(4)	0.3384(3)	0.0445(14)*
C41	-0.5880(3)	0.1879(3)	0.2170(2)	0.0327(11)*
C42	-0.5725(4)	0.1152(4)	0.1997(3)	0.0442(14)*
C43	-0.6219(4)	0.0513(4)	0.1431(3)	0.0587(19)*
C44	-0.6857(4)	0.0592(4)	0.1058(3)	0.0592(19)*
C45	-0.7019(4)	0.1311(5)	0.1230(3)	0.0594(19)*
C46	-0.6540(4)	0.1942(4)	0.1798(3)	0.0497(16)*
C51	-0.1174(3)	0.5160(3)	0.3688(2)	0.0340(12)*
C52	-0.0626(4)	0.5447(4)	0.4293(3)	0.0479(15)*
C53	0.0131(4)	0.5387(4)	0.4379(3)	0.0523(17)*
C54	0.0389(4)	0.5050(4)	0.3893(3)	0.0509(16)*
C55	-0.0143(4)	0.4755(4)	0.3288(3)	0.0451(14)*
C56	-0.0909(3)	0.4813(3)	0.3190(3)	0.0389(13)*
C61	-0.1981(3)	0.6275(3)	0.4091(2)	0.0332(12)*
C62	-0.1375(4)	0.6883(4)	0.4050(3)	0.0469(15)*
C63	-0.1196(5)	0.7693(4)	0.4404(3)	0.061(2)*
C64	-0.1634(5)	0.7910(4)	0.4808(3)	0.063(2)*
C65	-0.2237(4)	0.7323(4)	0.4864(3)	0.0501(16)*
C66	-0.2412(4)	0.6498(3)	0.4498(2)	0.0376(13)*
C71	-0.3796(4)	0.5891(3)	0.2917(2)	0.0354(12)*
C72	-0.4524(4)	0.5908(4)	0.3038(3)	0.0418(13)*
C73	-0.4652(5)	0.6640(4)	0.3289(3)	0.0586(19)*
C74	-0.4048(5)	0.7355(5)	0.3418(4)	0.067(2)*
C75	-0.3328(5)	0.7351(4)	0.3302(3)	0.0610(19)*
C76	-0.3195(4)	0.6621(3)	0.3036(3)	0.0454(14)*
C81	-0.3902(4)	0.4793(3)	0.1737(2)	0.0360(12)*
C82	-0.4628(4)	0.4874(4)	0.1465(3)	0.0477(15)*
C83	-0.4871(5)	0.4723(4)	0.0837(3)	0.062(2)*
C84	-0.4386(5)	0.4494(4)	0.0482(3)	0.060(2)*
C85	-0.3649(5)	0.4436(4)	0.0750(3)	0.0531(17)*
C86	-0.3396(4)	0.4581(3)	0.1378(2)	0.0403(13)*

Table S-14. Atomic Coordinates and Displacement Parameters for **8** (continued)

(e) triflate ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
S1A	-0.4864(3)	-0.0364(2)	0.26932(19)	0.0696(5)[a]
F91A	-0.4579(3)	-0.0553(3)	0.37339(18)	0.0696(5)[a]
F92A	-0.5826(3)	-0.1258(3)	0.3078(3)	0.0696(5)[a]
F93A	-0.4790(4)	-0.1633(3)	0.2902(3)	0.0696(5)[a]
O91A	-0.5353(4)	-0.0841(4)	0.20494(18)	0.0696(5)[a]
O92A	-0.4004(3)	-0.0078(4)	0.2758(3)	0.0696(5)[a]
O93A	-0.5123(4)	0.0324(3)	0.2946(3)	0.0696(5)[a]
C90A	-0.5025(3)	-0.0984(3)	0.3126(2)	0.0696(5)[a]
S1B	-0.4812(3)	-0.0496(2)	0.26631(19)	0.0696(5)[a]
F91B	-0.4632(3)	-0.0144(3)	0.38360(17)	0.0696(5)[a]
F92B	-0.5903(3)	-0.0785(4)	0.3213(3)	0.0696(5)[a]
F93B	-0.5045(4)	-0.1451(3)	0.3247(3)	0.0696(5)[a]
O91B	-0.5345(4)	-0.1137(3)	0.20715(17)	0.0696(5)[a]
O92B	-0.3971(3)	-0.0449(4)	0.2745(3)	0.0696(5)[a]
O93B	-0.4898(4)	0.0273(3)	0.2707(3)	0.0696(5)[a]
C90B	-0.5114(3)	-0.0731(3)	0.32706(19)	0.0696(5)[a]
S2A	-0.1301(3)	-0.5269(3)	0.1170(2)	0.0812(6)[b]
F94A	-0.0270(4)	-0.5838(4)	0.0703(3)	0.0812(6)[b]
F95A	0.0134(3)	-0.5250(4)	0.1723(3)	0.0812(6)[b]
F96A	-0.0823(4)	-0.6461(3)	0.1229(3)	0.0812(6)[b]
O94A	-0.1550(4)	-0.5156(4)	0.1718(3)	0.0812(6)[b]
O95A	-0.1990(3)	-0.5795(4)	0.0616(3)	0.0812(6)[b]
O96A	-0.0959(4)	-0.4488(3)	0.1146(3)	0.0812(6)[b]
C91A	-0.0521(3)	-0.5728(3)	0.1209(2)	0.0812(6)[b]
S2B	-0.1228(2)	-0.5409(3)	0.1178(2)	0.0812(6)[b]
F94B	-0.0081(3)	-0.5425(4)	0.0662(3)	0.0812(6)[b]
F95B	0.0358(3)	-0.4746(4)	0.1676(3)	0.0812(6)[b]
F96B	-0.0114(3)	-0.6085(4)	0.1271(3)	0.0812(6)[b]
O94B	-0.1344(4)	-0.5402(4)	0.1757(3)	0.0812(6)[b]
O95B	-0.1818(3)	-0.6133(3)	0.0663(3)	0.0812(6)[b]
O96B	-0.1308(4)	-0.4689(3)	0.1101(3)	0.0812(6)[b]
C91B	-0.0210(3)	-0.5417(3)	0.1198(2)	0.0812(6)[b]

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$. [a]Refined with an occupancy of 0.5 and a common isotropic displacement parameter. [b]Refined with an occupancy of 0.5 and a common isotropic displacement parameter.

Table S-15. Selected Interatomic Distances (Å) for **8**

(a) involving 'inner-core' atoms of molecule A

Atom1	Atom2	Distance
Os	Rh	2.8741(4)
Os	P1	2.3550(13)
Os	P3	2.3563(14)
Os	C1	1.860(5)
Os	C2	1.953(6)
Os	C4	2.101(5)
Rh	P2	2.3361(13)
Rh	P4	2.3366(14)
Rh	P5	2.3223(14)
Rh	C3	1.886(5)
Rh	C4	2.174(4)
P1	C10	1.827(5)
P1	C11	1.824(5)
P1	C21	1.832(5)
P1	P2	2.9805(16)†
P2	C10	1.816(5)
P2	C31	1.831(5)
P2	C41	1.837(5)
P3	C20	1.827(5)
P3	C51	1.832(5)
P3	C61	1.828(6)
P3	P4	3.0205(17)†
P4	C20	1.812(5)
P4	C71	1.832(6)
P4	C81	1.827(5)
P5	O5	1.608(4)
P5	O6	1.591(4)
P5	O7	1.592(4)
O1	C1	1.152(6)
O2	C2	1.126(7)
O3	C3	1.143(6)
O5	C5	1.428(7)
O6	C6	1.433(7)
O7	C7	1.437(7)

(b) involving 'inner-core' atoms of molecule B

Atom1	Atom2	Distance
Os	Rh	2.8679(4)
Os	P1	2.3618(13)
Os	P3	2.3601(13)
Os	C1	1.864(5)
Os	C2	1.949(6)
Os	C4	2.090(5)
Rh	P2	2.3287(14)
Rh	P4	2.3380(14)
Rh	P5	2.3223(14)
Rh	C3	1.879(5)
Rh	C4	2.181(4)
P1	C10	1.814(5)
P1	C11	1.834(5)
P1	C21	1.819(5)
P1	P2	2.9976(17)†
P2	C10	1.826(5)
P2	C31	1.843(6)
P2	C41	1.840(5)
P3	C20	1.837(5)
P3	C51	1.826(6)
P3	C61	1.823(5)
P3	P4	2.9880(17)†
P4	C20	1.821(5)
P4	C71	1.835(5)
P4	C81	1.835(5)
P5	O5	1.605(4)
P5	O6	1.590(4)
P5	O7	1.590(4)
O1	C1	1.160(6)
O2	C2	1.127(7)
O3	C3	1.155(7)
O5	C5	1.430(7)
O6	C6	1.445(7)
O7	C7	1.457(7)

†Nonbonded distance.

Table S-15. Selected Interatomic Distances for **8** (continued)

(c) involving dppm phenyl carbons of molecule A

Atom1	Atom2	Distance
C11	C12	1.373(8)
C11	C16	1.414(7)
C12	C13	1.396(7)
C13	C14	1.370(8)
C14	C15	1.385(9)
C15	C16	1.363(8)
C21	C22	1.383(8)
C21	C26	1.368(8)
C22	C23	1.391(9)
C23	C24	1.335(10)
C24	C25	1.373(11)
C25	C26	1.391(8)
C31	C32	1.377(8)
C31	C36	1.398(8)
C32	C33	1.396(8)
C33	C34	1.374(10)
C34	C35	1.374(10)
C35	C36	1.394(8)
C41	C42	1.387(7)
C41	C46	1.390(8)
C42	C43	1.389(8)
C43	C44	1.383(10)
C44	C45	1.385(9)
C45	C46	1.391(7)
C51	C52	1.358(9)
C51	C56	1.355(8)
C52	C53	1.401(8)
C53	C54	1.339(10)
C54	C55	1.346(12)
C55	C56	1.394(10)
C61	C62	1.390(9)
C61	C66	1.386(8)
C62	C63	1.381(10)
C63	C64	1.363(11)
C64	C65	1.397(12)
C65	C66	1.403(9)
C71	C72	1.376(9)
C71	C76	1.384(9)
C72	C73	1.403(10)

(d) dppm phenyl carbons of molecule B

Atom1	Atom2	Distance
C11	C12	1.352(8)
C11	C16	1.379(8)
C12	C13	1.406(8)
C13	C14	1.348(10)
C14	C15	1.343(10)
C15	C16	1.407(10)
C21	C22	1.376(8)
C21	C26	1.400(8)
C22	C23	1.388(8)
C23	C24	1.377(10)
C24	C25	1.397(10)
C25	C26	1.368(9)
C31	C32	1.422(9)
C31	C36	1.356(9)
C32	C33	1.383(10)
C33	C34	1.347(12)
C34	C35	1.428(12)
C35	C36	1.389(9)
C41	C42	1.392(8)
C41	C46	1.374(8)
C42	C43	1.394(8)
C43	C44	1.363(10)
C44	C45	1.382(11)
C45	C46	1.386(8)
C51	C52	1.409(7)
C51	C56	1.396(8)
C52	C53	1.358(9)
C53	C54	1.362(10)
C54	C55	1.396(9)
C55	C56	1.370(8)
C61	C62	1.397(8)
C61	C66	1.386(8)
C62	C63	1.375(9)
C63	C64	1.390(11)
C64	C65	1.387(10)
C65	C66	1.404(8)
C71	C72	1.392(8)
C71	C76	1.396(8)
C72	C73	1.389(8)

Table S-15. Selected Interatomic Distances for **8** (continued)

(c) involving dppm phenyl carbons of molecule A

Atom1	Atom2	Distance
C73	C74	1.367(13)
C74	C75	1.395(13)
C75	C76	1.391(9)
C81	C82	1.394(8)
C81	C86	1.390(8)
C82	C83	1.378(9)
C83	C84	1.386(12)
C84	C85	1.383(12)
C85	C86	1.395(9)

(d) dppm phenyl carbons of molecule B

Atom1	Atom2	Distance
C73	C74	1.374(11)
C74	C75	1.367(11)
C75	C76	1.397(9)
C81	C82	1.387(8)
C81	C86	1.408(8)
C82	C83	1.392(8)
C83	C84	1.380(11)
C84	C85	1.382(10)
C85	C86	1.395(8)

(e) within the triflate ions

Atom1	Atom2	Distance†
S1A	O91A	1.444(2)
S1A	O92A	1.442(2)
S1A	O93A	1.454(2)
S1A	C90A	1.802(2)
F91A	C90A	1.353(2)
F92A	C90A	1.352(2)
F93A	C90A	1.354(2)
S1B	O91B	1.446(2)
S1B	O92B	1.442(2)
S1B	O93B	1.444(2)
S1B	C90B	1.802(2)
F91B	C90B	1.356(2)
F92B	C90B	1.354(2)
F93B	C90B	1.356(2)

Atom1	Atom2	Distance†
S2A	O94A	1.445(2)
S2A	O95A	1.445(2)
S2A	O96A	1.450(2)
S2A	C91A	1.802(2)
F94A	C91A	1.353(2)
F95A	C91A	1.353(2)
F96A	C91A	1.353(2)
S2B	O94B	1.448(2)
S2B	O95B	1.446(2)
S2B	O96B	1.444(2)
S2B	C91B	1.804(2)
F94B	C91B	1.353(2)
F95B	C91B	1.353(2)
F96B	C91B	1.353(2)

†All distances for disordered triflate anions were restrained during refinement.

Table S-16. Selected Interatomic Angles (deg) for **8**

(a) involving 'inner-core' atoms of molecule A

Atom1	Atom2	Atom3	Angle
Rh	Os	P1	91.74(3)
Rh	Os	P3	91.82(3)
Rh	Os	C1	155.98(18)
Rh	Os	C2	105.79(14)
Rh	Os	C4	48.85(12)
P1	Os	P3	176.43(4)
P1	Os	C1	87.70(17)
P1	Os	C2	89.35(16)
P1	Os	C4	93.21(15)
P3	Os	C1	88.95(17)
P3	Os	C2	89.91(16)
P3	Os	C4	88.96(15)
C1	Os	C2	98.2(2)
C1	Os	C4	107.2(2)
C2	Os	C4	154.55(19)
Os	Rh	P2	90.53(3)
Os	Rh	P4	91.74(3)
Os	Rh	P5	92.56(3)
Os	Rh	C3	155.93(17)
Os	Rh	C4	46.68(12)
P2	Rh	P4	173.46(5)
P2	Rh	P5	95.76(5)
P2	Rh	C3	87.58(17)
P2	Rh	C4	87.35(14)
P4	Rh	P5	90.27(5)
P4	Rh	C3	87.85(17)
P4	Rh	C4	89.75(14)
P5	Rh	C3	111.51(17)
P5	Rh	C4	139.21(13)
C3	Rh	C4	109.3(2)
Os	P1	C10	114.34(17)
Os	P1	C11	112.71(16)
Os	P1	C21	118.27(18)
C10	P1	C11	103.5(2)
C10	P1	C21	104.8(2)
C11	P1	C21	101.4(2)
Rh	P2	C10	113.43(17)
Rh	P2	C31	117.11(18)
Rh	P2	C41	113.79(17)

(b) involving atoms of molecule B

Atom1	Atom2	Atom3	Angle
Rh	Os	P1	91.86(3)
Rh	Os	P3	91.49(3)
Rh	Os	C1	154.42(16)
Rh	Os	C2	106.21(15)
Rh	Os	C4	49.18(12)
P1	Os	P3	175.71(4)
P1	Os	C1	89.74(17)
P1	Os	C2	87.59(15)
P1	Os	C4	90.67(15)
P3	Os	C1	88.40(17)
P3	Os	C2	88.90(16)
P3	Os	C4	93.55(15)
C1	Os	C2	99.4(2)
C1	Os	C4	105.3(2)
C2	Os	C4	155.28(19)
Os	Rh	P2	91.30(3)
Os	Rh	P4	91.04(3)
Os	Rh	P5	94.16(4)
Os	Rh	C3	156.70(19)
Os	Rh	C4	46.49(12)
P2	Rh	P4	173.69(5)
P2	Rh	P5	90.55(5)
P2	Rh	C3	88.06(18)
P2	Rh	C4	89.68(14)
P4	Rh	P5	95.12(5)
P4	Rh	C3	87.47(18)
P4	Rh	C4	87.68(14)
P5	Rh	C3	109.14(19)
P5	Rh	C4	140.63(13)
C3	Rh	C4	110.2(2)
Os	P1	C10	112.59(17)
Os	P1	C11	112.73(18)
Os	P1	C21	121.12(18)
C10	P1	C11	102.5(2)
C10	P1	C21	103.4(2)
C11	P1	C21	102.4(2)
Rh	P2	C10	113.66(18)
Rh	P2	C31	116.8(2)
Rh	P2	C41	114.37(17)

Table S-16. Selected Interatomic Angles for **8** (continued)

(a) involving 'inner-core' atoms of molecule A

Atom1	Atom2	Atom3	Angle
C10	P2	C31	105.2(2)
C10	P2	C41	104.8(2)
C31	P2	C41	101.0(2)
Os	P3	C20	113.40(18)
Os	P3	C51	112.7(2)
Os	P3	C61	119.6(2)
C20	P3	C51	102.7(3)
C20	P3	C61	104.2(3)
C51	P3	C61	102.4(3)
Rh	P4	C20	114.74(18)
Rh	P4	C71	117.6(2)
Rh	P4	C81	114.40(19)
C20	P4	C71	105.2(3)
C20	P4	C81	102.5(2)
C71	P4	C81	100.3(3)
Rh	P5	O5	120.27(17)
Rh	P5	O6	114.29(16)
Rh	P5	O7	112.59(16)
O5	P5	O6	103.4(2)
O5	P5	O7	97.7(2)
O6	P5	O7	106.6(2)
P5	O5	C5	121.9(4)
P5	O6	C6	124.8(4)
P5	O7	C7	127.4(4)
Os	C1	O1	179.0(5)
Os	C2	O2	177.7(5)
Rh	C3	O3	178.6(6)
Os	C4	Rh	84.47(18)
P1	C10	P2	109.8(3)
P3	C20	P4	112.2(3)

(b) involving atoms of molecule B

Atom1	Atom2	Atom3	Angle
C10	P2	C31	105.9(2)
C10	P2	C41	103.2(2)
C31	P2	C41	101.3(3)
Os	P3	C20	114.80(17)
Os	P3	C51	112.38(17)
Os	P3	C61	118.70(19)
C20	P3	C51	103.0(2)
C20	P3	C61	104.2(2)
C51	P3	C61	101.9(3)
Rh	P4	C20	113.15(17)
Rh	P4	C71	117.0(2)
Rh	P4	C81	113.99(18)
C20	P4	C71	105.7(3)
C20	P4	C81	104.6(3)
C71	P4	C81	101.0(2)
Rh	P5	O5	120.30(18)
Rh	P5	O6	112.95(15)
Rh	P5	O7	113.88(16)
O5	P5	O6	97.1(2)
O5	P5	O7	103.9(2)
O6	P5	O7	106.7(2)
P5	O5	C5	121.8(4)
P5	O6	C6	126.4(4)
P5	O7	C7	124.3(4)
Os	C1	O1	177.0(5)
Os	C2	O2	178.1(5)
Rh	C3	O3	177.8(6)
Os	C4	Rh	84.33(17)
P1	C10	P2	110.9(3)
P3	C20	P4	109.5(3)

(c) involving dppm phenyl carbons of molecule A

Atom1	Atom2	Atom3	Angle
P1	C11	C12	124.3(4)
P1	C11	C16	118.1(4)
C12	C11	C16	117.5(5)
C11	C12	C13	121.1(5)
C12	C13	C14	120.1(6)
C13	C14	C15	119.9(5)

(d) involving atoms of molecule B

Atom1	Atom2	Atom3	Angle
P1	C11	C12	122.3(4)
P1	C11	C16	117.8(4)
C12	C11	C16	119.9(5)
C11	C12	C13	120.5(6)
C12	C13	C14	120.2(6)
C13	C14	C15	119.2(6)