Table S-17. Anisotropic Displacement Parameters for **8** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(35)	0.13(2)	0.033(10)	0.046(12)	0.000(9)	-0.022(13)	0.043(12)
C(36)	0.063(11)	0.034(9)	0.036(11)	0.009(7)	-0.004(8)	0.013(8)
C(41)	0.041(9)	0.030(8)	0.024(8)	0.000(6)	0.007(7)	0.009(7)
C(42)	0.034(9)	0.052(10)	0.035(10)	0.010(8)	0.005(7)	-0.011(7)
C(43)	0.048(11)	0.062(12)	0.034(11)	0.008(9)	0.000(8)	0.002(9)
C(44)	0.065(12)	0.049(10)	0.028(10)	0.015(8)	-0.017(9)	0.010(9)
C(45)	0.047(10)	0.070(12)	0.044(13)	-0.008(9)	-0.015(9)	0.032(9)
C(46)	0.028(9)	0.078(13)	0.030(10)	0.001(8)	-0.003(7)	0.004(8)
C(51)	0.022(7)	0.032(8)	0.026(9)	0.004(6)	-0.001(6)	0.004(6)
C(52)	0.051(10)	0.026(8)	0.028(9)	-0.012(6)	-0.007(7)	0.010(7)
C(53)	0.048(11)	0.043(10)	0.052(12)	-0.029(9)	-0.023(9)	0.009(8)
C(54)	0.034(9)	0.040(10)	0.078(15)	-0.017(10)	-0.014(9)	-0.008(8)
C(55)	0.034(9)	0.055(11)	0.049(11)	-0.005(9)	0.000(8)	-0.023(8)
C(56)	0.040(9)	0.029(8)	0.054(11)	-0.001(8)	-0.006(8)	-0.004(7)
C(61)	0.018(7)	0.018(7)	0.033(9)	0.000(6)	-0.005(6)	0.001(5)
C(62)	0.034(8)	0.043(9)	0.025(9)	0.001(7)	-0.005(6)	0.011(7)
C(63)	0.030(9)	0.052(11)	0.056(12)	0.000(9)	-0.007(8)	0.015(8)
C(64)	0.078(14)	0.040(10)	0.046(12)	0.006(8)	-0.022(10)	0.019(10)
C(65)	0.053(10)	0.033(9)	0.032(9)	0.011(7)	-0.004(8)	-0.005(8)
C(66)	0.026(8)	0.029(8)	0.056(11)	-0.009(7)	-0.003(7)	-0.003(6)
C(71)	0.018(6)	0.015(6)	0.023(8)	-0.006(5)	0.002(5)	0.000(5)
C(72)	0.028(8)	0.028(8)	0.031(9)	0.001(6)	-0.008(6)	0.002(6)
C(73)	0.034(9)	0.035(9)	0.047(11)	-0.017(8)	-0.019(7)	0.005(7)
C(74)	0.045(10)	0.021(8)	0.062(12)	-0.010(8)	0.012(8)	-0.007(7)
C(75)	0.060(11)	0.017(7)	0.041(10)	0.005(7)	0.007(8)	0.012(7)
C(76)	0.030(7)	0.029(7)	0.028(8)	-0.010(6)	0.012(6)	0.001(6)
C(81)	0.037(8)	0.014(7)	0.029(9)	-0.003(6)	0.001(6)	-0.002(6)
C(82)	0.040(9)	0.050(10)	0.018(8)	0.004(7)	0.007(7)	-0.010(7)
C(83)	0.062(12)	0.091(14)	0.013(9)	-0.005(9)	-0.009(8)	-0.013(11)
C(84)	0.044(10)	0.070(12)	0.014(8)	-0.017(8)	0.008(7)	-0.010(8)
C(85)	0.044(10)	0.060(11)	0.056(13)	-0.029(9)	0.036(9)	-0.002(8)
C(86)	0.028(8)	0.047(9)	0.027(9)	0.008(7)	0.015(7)	0.008(7)
Cl(1S)	0.096(5)	0.125(6)	0.094(6)	-0.010(4)	0.013(4)	-0.006(4)
Cl(2S)	0.110(5)	0.110(5)	0.076(5)	0.019(4)	0.004(4)	0.030(4)
C(1S)	0.064(15)	0.17(3)	0.064(17)	0.008(17)	-0.002(12)	0.008(17)
C(2S)	0.091(19)	0.11(2)	0.13(3)	-0.025(19)	0.015(18)	0.054(17)

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-18. Derived Coordinates and Displacement Parameters for Hydrogen Atoms of **8**

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(3A)	0.1700	0.5662	0.0554	0.024
H(3B)	0.2108	0.5555	0.1275	0.024
H(7A)	0.2790	0.6657	0.1292	0.091
H(7B)	0.2714	0.6729	0.0047	0.091
H(7C)	0.3103	0.6440	0.0445	0.091
H(9A)	0.2494	0.5655	-0.4432	0.097
H(9B)	0.2781	0.5271	-0.3996	0.097
H(9C)	0.2876	0.5750	-0.3620	0.097
H(10A)	0.0605	0.5382	-0.0615	0.028
H(10B)	0.0673	0.4901	-0.1009	0.028
H(12)	0.0727	0.4707	0.2614	0.054
H(13)	0.0351	0.4095	0.3213	0.058
H(14)	0.0046	0.3635	0.2020	0.061
H(15)	0.0100	0.3761	0.0221	0.047
H(16)	0.0520	0.4309	-0.0404	0.041
H(20A)	0.2902	0.4689	-0.0741	0.024
H(20B)	0.2912	0.4206	-0.0320	0.024
H(22)	0.1379	0.5619	0.2174	0.051
H(23)	0.1100	0.6168	0.3161	0.062
H(24)	0.0379	0.6338	0.3066	0.079
H(25)	-0.0081	0.5917	0.2053	0.088
H(26)	0.0210	0.5344	0.1063	0.052
H(32)	0.1842	0.5965	-0.2301	0.046
H(33)	0.1973	0.6677	-0.1917	0.069
H(34)	0.1588	0.7032	-0.0637	0.082
H(35)	0.1045	0.6711	0.0229	0.085
H(36)	0.0859	0.5980	-0.0143	0.053
H(42)	0.1584	0.5390	-0.3631	0.048
H(43)	0.1335	0.5401	-0.5340	0.058
H(44)	0.0598	0.5372	-0.5657	0.057
H(45)	0.0140	0.5324	-0.4223	0.065
H(46)	0.0403	0.5293	-0.2505	0.055
H(52)	0.2545	0.5081	0.2759	0.042
H(53)	0.2938	0.5673	0.3381	0.057
H(54)	0.3475	0.5953	0.2421	0.061
H(55)	0.3642	0.5686	0.0739	0.055
H(56)	0.3237	0.5123	0.0018	0.049
H(62)	0.3279	0.4287	0.1558	0.040
H(63)	0.3485	0.3727	0.2633	0.055
H(64)	0.2993	0.3336	0.3583	0.065

Table S-18. Derived Parameters for Hydrogen Atoms of **8** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(65)	0.2266	0.3446	0.3326	0.047
H(66)	0.2037	0.4015	0.2214	0.044
H(72)	0.1876	0.3681	-0.2602	0.035
H(73)	0.1779	0.2939	-0.2503	0.047
H(74)	0.1998	0.2574	-0.1007	0.051
H(75)	0.2415	0.2912	0.0296	0.047
H(76)	0.2532	0.3658	0.0159	0.035
H(82)	0.2065	0.4481	-0.3526	0.043
H(83)	0.2392	0.4482	-0.5194	0.066
H(84)	0.3094	0.4254	-0.5394	0.051
H(85)	0.3490	0.4092	-0.3896	0.064
H(86)	0.3181	0.4123	-0.2206	0.041
H(1SA)	0.0712	0.1128	-0.1079	0.120
H(1SB)	0.0498	0.0970	0.0005	0.120
H(2SA)	0.1059	0.1271	0.0991	0.132
H(2SB)	0.0820	0.1625	0.0305	0.132
H(3SA)	0.2206	0.6475	0.2127	0.215
H(3SB)	0.1705	0.6370	0.2150	0.215
H(4SA)[a]	0.2040	0.7091	0.3303	0.215
H(4SB)[a]	0.1548	0.7009	0.3014	0.215
H(4SC)[a]	0.1607	0.7004	0.3267	0.215
H(4SD)[a]	0.1757	0.7116	0.2080	0.215

[a]Included with an occupancy factor of 0.5.

Table S-19. Crystallographic Experimental Details for **15**

A. Crystal Data	
formula	$C_{65}H_{64}BF_4O_8OsP_4Rh$
formula weight	1476.96
crystal dimensions (mm)	0.33 × 0.10 × 0.07
crystal system	monoclinic
space group	$P2_1/n$ (an alternate setting of $P2_1/c$ [No. 14])
unit cell parameters[a]	
a (Å)	12.6248 (10)
b (Å)	28.170 (2)
c (Å)	17.3062 (13)
β (deg)	91.0780 (15)
V (Å^3)	6153.6 (8)
Z	4
ρ_{calcd} (g cm^{-3})	1.594
μ (mm^{-1})	2.502
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°) (40 s exposures)
data collection 2θ limit (deg)	52.84
total data collected	46114 (-15 ≤ h ≤ 15, -35 ≤ k ≤ 35, -21 ≤ l ≤ 21)
independent reflections	12592 (R_{int} = 0.0685)
number of observed reflections (*NO*)	9450 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	direct methods (*SHELXS–86*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	multi-scan (*SADABS*)
range of transmission factors	0.8443–0.4923
data/restraints/parameters	12592 [$F_o^2 \geq -3\sigma(F_o^2)$] / 0 / 757
goodness-of-fit (S)[e]	1.113 [$F_o^2 \geq -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0433
wR_2 [$F_o^2 \geq -3\sigma(F_o^2)$]	0.0998
largest difference peak and hole	1.123 and –0.861 e Å^{-3}

[a]Obtained from least-squares refinement of 7215 reflections with 4.34° < 2θ < 52.34°.

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

(continued)

Table S-19. Crystallographic Experimental Details for **15** (continued)

[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; $w = [\sigma^2(F_o^2) + (0.0443P)^2 + 1.9039P]^{-1}$ 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-20. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **15**

(a) 'inner-core' atoms of [RhOs(CO)$_3${η^2–CH_2CH_2C(CO_2Me)C(CO_2Me)}(dppm)$_2$]$^+$

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Os	0.110634(15)	0.154859(7)	0.401684(11)	0.02452(7)*
Rh	0.27995(3)	0.090473(14)	0.43258(2)	0.02764(10)*
P(1)	0.03359(10)	0.10394(5)	0.30310(8)	0.0305(3)*
P(2)	0.20862(11)	0.03423(5)	0.34700(8)	0.0297(3)*
P(3)	0.16774(10)	0.20703(5)	0.50333(7)	0.0255(3)*
P(4)	0.34406(10)	0.13602(5)	0.53525(8)	0.0270(3)*
O(1)	0.3167(3)	0.16669(13)	0.3084(2)	0.0330(9)*
O(2)	0.0842(3)	0.07558(14)	0.5207(2)	0.0403(9)*
O(3)	0.4896(3)	0.04035(17)	0.4349(3)	0.0634(14)*
O(90)	-0.1220(4)	0.26801(17)	0.2489(3)	0.0604(13)*
O(91)	-0.1917(4)	0.2832(2)	0.3628(3)	0.0829(18)*
O(92)	0.1544(3)	0.23798(14)	0.2437(2)	0.0461(10)*
O(93)	0.0851(3)	0.29348(14)	0.3198(2)	0.0453(10)*
C(1)	0.2444(4)	0.15955(18)	0.3465(3)	0.0277(11)*
C(2)	0.1135(4)	0.10170(19)	0.4735(3)	0.0307(12)*
C(3)	0.4091(5)	0.0581(2)	0.4358(3)	0.0405(14)*
C(4)	-0.0476(4)	0.15557(19)	0.4517(3)	0.0303(11)*
C(5)	-0.1194(4)	0.1952(2)	0.4201(3)	0.0383(14)*
C(6)	-0.0635(4)	0.22340(19)	0.3613(3)	0.0312(12)*
C(7)	0.0384(4)	0.21455(19)	0.3466(3)	0.0306(12)*
C(8)	0.0661(4)	0.04233(19)	0.3287(3)	0.0339(13)*
C(9)	0.2417(4)	0.17322(18)	0.5765(3)	0.0293(12)*
C(90)	-0.1257(5)	0.2606(2)	0.3171(4)	0.0435(15)*
C(91)	-0.2595(8)	0.3187(4)	0.3262(6)	0.129(5)*
C(92)	0.0977(4)	0.2481(2)	0.2961(3)	0.0351(13)*
C(93)	0.1393(6)	0.3286(2)	0.2731(4)	0.0601(19)*

(b) dppm phenyl carbons

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C(11)	-0.1114(4)	0.1052(2)	0.2924(3)	0.0395(14)*
C(12)	-0.1551(5)	0.1426(3)	0.2522(4)	0.0494(17)*
C(13)	-0.2654(5)	0.1469(3)	0.2446(4)	0.066(2)*
C(14)	-0.3295(6)	0.1124(4)	0.2756(5)	0.076(3)*
C(15)	-0.2860(6)	0.0748(3)	0.3142(5)	0.075(2)*
C(16)	-0.1761(5)	0.0707(3)	0.3226(4)	0.0588(19)*
C(21)	0.0674(4)	0.1051(2)	0.2001(3)	0.0334(12)*
C(22)	0.1389(4)	0.1364(2)	0.1701(3)	0.0393(14)*
C(23)	0.1628(5)	0.1343(3)	0.0917(4)	0.0515(17)*
C(24)	0.1161(5)	0.1004(3)	0.0450(4)	0.0516(17)*

Table S-20. Atomic Coordinates and Displacement Parameters for **15** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C(25)	0.0438(6)	0.0698(2)	0.0749(4)	0.0553(18)*
C(26)	0.0176(5)	0.0716(2)	0.1525(4)	0.0494(16)*
C(31)	0.2710(4)	0.03172(19)	0.2527(3)	0.0331(12)*
C(32)	0.3618(4)	0.0579(2)	0.2402(3)	0.0366(13)*
C(33)	0.4134(5)	0.0551(2)	0.1709(4)	0.0448(15)*
C(34)	0.3773(5)	0.0267(2)	0.1125(4)	0.0477(16)*
C(35)	0.2850(6)	0.0009(3)	0.1246(4)	0.065(2)*
C(36)	0.2318(5)	0.0039(2)	0.1937(4)	0.0528(17)*
C(41)	0.2138(4)	-0.0265(2)	0.3845(3)	0.0381(13)*
C(42)	0.2679(5)	-0.0375(3)	0.4521(4)	0.0565(18)*
C(43)	0.2804(6)	-0.0860(3)	0.4774(4)	0.068(2)*
C(44)	0.2414(5)	-0.1220(2)	0.4309(5)	0.061(2)*
C(45)	0.1792(7)	-0.1104(2)	0.3653(5)	0.070(2)*
C(46)	0.1703(6)	-0.0636(2)	0.3419(4)	0.064(2)*
C(51)	0.0638(4)	0.23532(19)	0.5591(3)	0.0314(12)*
C(52)	0.0176(5)	0.2765(2)	0.5305(4)	0.0469(15)*
C(53)	-0.0635(6)	0.2985(2)	0.5701(4)	0.063(2)*
C(54)	-0.0990(6)	0.2794(3)	0.6375(4)	0.069(2)*
C(55)	-0.0534(6)	0.2388(3)	0.6670(4)	0.063(2)*
C(56)	0.0280(5)	0.2162(2)	0.6281(3)	0.0481(16)*
C(61)	0.2565(4)	0.25682(18)	0.4851(3)	0.0292(12)*
C(62)	0.2971(4)	0.26669(19)	0.4131(3)	0.0328(12)*
C(63)	0.3666(5)	0.3048(2)	0.4032(4)	0.0416(14)*
C(64)	0.3946(5)	0.3326(2)	0.4647(4)	0.0459(16)*
C(65)	0.3543(5)	0.3232(2)	0.5371(4)	0.0473(16)*
C(66)	0.2857(5)	0.28585(19)	0.5474(3)	0.0395(14)*
C(71)	0.4559(4)	0.17472(19)	0.5182(3)	0.0300(12)*
C(72)	0.5025(4)	0.1755(2)	0.4464(3)	0.0321(12)*
C(73)	0.5846(4)	0.2063(2)	0.4323(3)	0.0402(14)*
C(74)	0.6208(5)	0.2371(2)	0.4895(4)	0.0426(14)*
C(75)	0.5754(5)	0.2363(2)	0.5616(4)	0.0439(15)*
C(76)	0.4935(4)	0.2050(2)	0.5760(3)	0.0363(13)*
C(81)	0.3810(4)	0.09813(19)	0.6166(3)	0.0332(12)*
C(82)	0.4860(4)	0.0894(2)	0.6357(3)	0.0420(14)*
C(83)	0.5125(5)	0.0592(2)	0.6955(4)	0.0554(18)*
C(84)	0.4346(7)	0.0402(3)	0.7412(5)	0.078(2)*
C(85)	0.3254(7)	0.0492(3)	0.7228(5)	0.073(2)*
C(86)	0.3018(5)	0.0762(2)	0.6588(4)	0.0584(19)*

Table S-20. Atomic Coordinates and Displacement Parameters for **15** (continued)

(c) tetrafluoroborate ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
F(1)	0.0551(4)	0.04053(15)	-0.2343(3)	0.0924(16)*
F(2)	0.1237(5)	0.08851(18)	-0.1444(3)	0.1058(19)*
F(3)	0.0617(6)	0.1183(2)	-0.2529(4)	0.148(3)*
F(4)	-0.0491(6)	0.0871(3)	-0.1724(5)	0.207(5)*
B	0.0479(8)	0.0834(4)	-0.1986(6)	0.072(3)*

(d) solvent diethyl ether atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O(1S)	0.3809(5)	-0.0606(2)	-0.0231(3)	0.0833(17)*
C(1S)	0.2828(8)	-0.0597(4)	-0.0657(6)	0.096(3)*
C(2S)	0.2645(9)	-0.0129(4)	-0.0945(6)	0.136(5)*
C(3S)	0.3989(10)	-0.1028(3)	0.0136(8)	0.124(4)*
C(4S)	0.5055(9)	-0.0992(3)	0.0586(6)	0.116(4)*

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-21. Selected Interatomic Distances (Å) for **15**

(a) involving 'inner-core' atoms of $[RhOs(CO)_3\{\eta^2\text{–}\underline{C}H_2CH_2C(CO_2Me)\underline{C}(CO_2Me)\}(dppm)_2]^+$

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Os	Rh	2.8464(5)	P(3)	C(9)	1.827(5)
Os	P(1)	2.4190(13)	P(4)	C(9)	1.821(5)
Os	P(3)	2.3929(13)	O(1)	C(1)	1.153(6)
Os	C(1)	1.960(5)	O(2)	C(2)	1.165(6)
Os	C(2)	1.946(6)	O(3)	C(3)	1.133(6)
Os	C(4)	2.192(5)	O(90)	C(90)	1.200(7)
Os	C(7)	2.129(5)	O(91)	C(90)	1.322(7)
Rh	P(2)	2.3377(14)	O(91)	C(91)	1.454(8)
Rh	P(4)	2.3247(14)	O(92)	C(92)	1.200(6)
Rh	C(1)	2.487(5)	O(93)	C(92)	1.353(7)
Rh	C(2)	2.252(5)	O(93)	C(93)	1.456(7)
Rh	C(3)	1.868(6)	C(4)	C(5)	1.533(7)
P(1)	P(2)	3.042(2)†	C(5)	C(6)	1.481(7)
P(1)	C(8)	1.836(6)	C(6)	C(7)	1.340(7)
P(2)	C(8)	1.836(5)	C(6)	C(90)	1.509(8)
P(3)	P(4)	3.0357(18)†	C(7)	C(92)	1.497(7)

†Nonbonded distance.

(b) involving dppm phenyl carbons

Atom1	Atom2	Distance	Atom1	Atom2	Distance
P(1)	C(11)	1.837(5)	C(25)	C(26)	1.390(9)
P(1)	C(21)	1.841(5)	C(31)	C(32)	1.383(7)
P(2)	C(31)	1.827(5)	C(31)	C(36)	1.373(8)
P(2)	C(41)	1.829(6)	C(32)	C(33)	1.378(8)
P(3)	C(51)	1.826(5)	C(33)	C(34)	1.360(8)
P(3)	C(61)	1.827(5)	C(34)	C(35)	1.393(9)
P(4)	C(71)	1.813(5)	C(35)	C(36)	1.384(9)
P(4)	C(81)	1.820(5)	C(41)	C(42)	1.379(8)
C(11)	C(12)	1.373(9)	C(41)	C(46)	1.387(8)
C(11)	C(16)	1.377(9)	C(42)	C(43)	1.444(10)
C(12)	C(13)	1.401(9)	C(43)	C(44)	1.380(10)
C(13)	C(14)	1.379(11)	C(44)	C(45)	1.407(11)
C(14)	C(15)	1.361(12)	C(45)	C(46)	1.383(9)
C(15)	C(16)	1.398(9)	C(51)	C(52)	1.385(8)
C(21)	C(22)	1.370(8)	C(51)	C(56)	1.394(8)
C(21)	C(26)	1.395(8)	C(52)	C(53)	1.389(8)
C(22)	C(23)	1.398(8)	C(53)	C(54)	1.367(10)
C(23)	C(24)	1.376(9)	C(54)	C(55)	1.374(10)
C(24)	C(25)	1.365(9)	C(55)	C(56)	1.393(8)

Table S-21. Selected Interatomic Distances for **15** (continued)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C(61)	C(62)	1.384(7)	C(73)	C(74)	1.386(8)
C(61)	C(66)	1.398(7)	C(74)	C(75)	1.382(8)
C(62)	C(63)	1.398(7)	C(75)	C(76)	1.384(7)
C(63)	C(64)	1.362(8)	C(81)	C(82)	1.383(7)
C(64)	C(65)	1.388(9)	C(81)	C(86)	1.394(8)
C(65)	C(66)	1.376(8)	C(82)	C(83)	1.377(8)
C(71)	C(72)	1.385(7)	C(83)	C(84)	1.381(10)
C(71)	C(76)	1.392(7)	C(84)	C(85)	1.432(11)
C(72)	C(73)	1.377(7)	C(85)	C(86)	1.372(9)

(c) within the tetrafluoroborate ion

Atom1	Atom2	Distance	Atom1	Atom2	Distance
F(1)	B	1.360(10)	F(3)	B	1.373(11)
F(2)	B	1.336(10)	F(4)	B	1.319(11)

(d) within the solvent diethyl ether molecule

Atom1	Atom2	Distance	Atom1	Atom2	Distance
O(1S)	C(1S)	1.430(10)	C(1S)	C(2S)	1.427(13)
O(1S)	C(3S)	1.363(11)	C(3S)	C(4S)	1.546(13)

Table S-22. Selected Interatomic Angles (deg) for **15**

(a) involving 'inner-core' atoms of $[RhOs(CO)_3\{\eta^2\text{-}\underline{C}H_2CH_2C(CO_2Me)\underline{C}(CO_2Me)\}(dppm)_2]^+$

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Rh	Os	P(1)	92.60(3)	C(1)	Rh	C(2)	85.39(19)
Rh	Os	P(3)	92.20(3)	C(1)	Rh	C(3)	123.2(2)
Rh	Os	C(1)	58.92(15)	C(2)	Rh	C(3)	151.4(2)
Rh	Os	C(2)	52.03(15)	Os	P(1)	C(8)	107.77(17)
Rh	Os	C(4)	128.19(14)	Rh	P(2)	C(8)	112.94(18)
Rh	Os	C(7)	154.28(14)	Os	P(3)	C(9)	109.42(17)
P(1)	Os	P(3)	173.82(4)	Rh	P(4)	C(9)	112.22(17)
P(1)	Os	C(1)	92.00(14)	C(90)	O(91)	C(91)	116.3(6)
P(1)	Os	C(2)	89.81(15)	C(92)	O(93)	C(93)	114.4(5)
P(1)	Os	C(4)	85.81(14)	Os	C(1)	Rh	78.61(18)
P(1)	Os	C(7)	89.39(14)	Os	C(1)	O(1)	171.3(4)
P(3)	Os	C(1)	93.81(14)	Rh	C(1)	O(1)	110.0(4)
P(3)	Os	C(2)	90.08(15)	Os	C(2)	Rh	85.0(2)
P(3)	Os	C(4)	88.17(14)	Os	C(2)	O(2)	158.7(4)
P(3)	Os	C(7)	87.97(14)	Rh	C(2)	O(2)	116.3(4)
C(1)	Os	C(2)	110.9(2)	Rh	C(3)	O(3)	176.1(6)
C(1)	Os	C(4)	172.6(2)	Os	C(4)	C(5)	113.8(3)
C(1)	Os	C(7)	95.4(2)	C(4)	C(5)	C(6)	110.5(4)
C(2)	Os	C(4)	76.2(2)	C(5)	C(6)	C(7)	120.2(5)
C(2)	Os	C(7)	153.7(2)	C(5)	C(6)	C(90)	118.0(5)
C(4)	Os	C(7)	77.53(19)	C(7)	C(6)	C(90)	121.7(5)
Os	Rh	P(2)	91.94(4)	Os	C(7)	C(6)	117.8(4)
Os	Rh	P(4)	92.37(4)	Os	C(7)	C(92)	123.0(4)
Os	Rh	C(1)	42.47(12)	C(6)	C(7)	C(92)	119.1(5)
Os	Rh	C(2)	42.93(14)	P(1)	C(8)	P(2)	111.9(3)
Os	Rh	C(3)	165.6(2)	P(3)	C(9)	P(4)	112.7(3)
P(2)	Rh	P(4)	169.34(5)	O(90)	C(90)	O(91)	122.7(6)
P(2)	Rh	C(1)	95.03(12)	O(90)	C(90)	C(6)	126.1(6)
P(2)	Rh	C(2)	86.78(13)	O(91)	C(90)	C(6)	111.0(5)
P(2)	Rh	C(3)	90.78(17)	O(92)	C(92)	O(93)	121.9(5)
P(4)	Rh	C(1)	94.73(12)	O(92)	C(92)	C(7)	127.1(5)
P(4)	Rh	C(2)	89.75(13)	O(93)	C(92)	C(7)	110.9(5)
P(4)	Rh	C(3)	87.45(17)				

(b) involving dppm phenyl carbons

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Os	P(1)	C(11)	116.53(17)	C(8)	P(1)	C(21)	101.3(2)
Os	P(1)	C(21)	125.08(19)	C(11)	P(1)	C(21)	98.7(2)
C(8)	P(1)	C(11)	105.1(3)	Rh	P(2)	C(31)	115.14(18)

Table S-22. Selected Interatomic Angles for **15** (continued)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
Rh	P(2)	C(41)	113.46(19)	P(2)	C(41)	C(46)	120.4(5)
C(8)	P(2)	C(31)	106.8(2)	C(42)	C(41)	C(46)	117.8(6)
C(8)	P(2)	C(41)	101.9(3)	C(41)	C(42)	C(43)	121.3(7)
C(31)	P(2)	C(41)	105.5(2)	C(42)	C(43)	C(44)	118.9(6)
Os	P(3)	C(51)	116.55(18)	C(43)	C(44)	C(45)	119.1(6)
Os	P(3)	C(61)	121.45(17)	C(44)	C(45)	C(46)	119.9(7)
C(9)	P(3)	C(51)	102.9(2)	C(41)	C(46)	C(45)	122.3(7)
C(9)	P(3)	C(61)	102.3(2)	P(3)	C(51)	C(52)	118.6(4)
C(51)	P(3)	C(61)	101.9(2)	P(3)	C(51)	C(56)	122.1(4)
Rh	P(4)	C(71)	117.93(18)	C(52)	C(51)	C(56)	119.3(5)
Rh	P(4)	C(81)	110.35(18)	C(51)	C(52)	C(53)	120.5(6)
C(9)	P(4)	C(71)	106.2(2)	C(52)	C(53)	C(54)	120.0(6)
C(9)	P(4)	C(81)	102.0(2)	C(53)	C(54)	C(55)	120.2(6)
C(71)	P(4)	C(81)	106.8(2)	C(54)	C(55)	C(56)	120.6(6)
P(1)	C(11)	C(12)	117.1(5)	C(51)	C(56)	C(55)	119.4(6)
P(1)	C(11)	C(16)	123.0(5)	P(3)	C(61)	C(62)	123.3(4)
C(12)	C(11)	C(16)	119.9(6)	P(3)	C(61)	C(66)	118.0(4)
C(11)	C(12)	C(13)	120.1(7)	C(62)	C(61)	C(66)	118.7(5)
C(12)	C(13)	C(14)	119.5(8)	C(61)	C(62)	C(63)	120.6(5)
C(13)	C(14)	C(15)	120.3(7)	C(62)	C(63)	C(64)	120.0(6)
C(14)	C(15)	C(16)	120.4(8)	C(63)	C(64)	C(65)	120.1(5)
C(11)	C(16)	C(15)	119.8(8)	C(64)	C(65)	C(66)	120.3(6)
P(1)	C(21)	C(22)	123.0(4)	C(61)	C(66)	C(65)	120.3(5)
P(1)	C(21)	C(26)	116.7(5)	P(4)	C(71)	C(72)	120.1(4)
C(22)	C(21)	C(26)	120.3(5)	P(4)	C(71)	C(76)	120.5(4)
C(21)	C(22)	C(23)	119.8(6)	C(72)	C(71)	C(76)	119.4(5)
C(22)	C(23)	C(24)	120.0(6)	C(71)	C(72)	C(73)	120.1(5)
C(23)	C(24)	C(25)	119.9(6)	C(72)	C(73)	C(74)	120.5(5)
C(24)	C(25)	C(26)	121.1(6)	C(73)	C(74)	C(75)	119.8(6)
C(21)	C(26)	C(25)	118.9(6)	C(74)	C(75)	C(76)	119.8(6)
P(2)	C(31)	C(32)	119.4(4)	C(71)	C(76)	C(75)	120.4(5)
P(2)	C(31)	C(36)	122.1(4)	P(4)	C(81)	C(82)	121.4(4)
C(32)	C(31)	C(36)	118.5(5)	P(4)	C(81)	C(86)	119.3(4)
C(31)	C(32)	C(33)	120.8(5)	C(82)	C(81)	C(86)	119.3(5)
C(32)	C(33)	C(34)	121.5(6)	C(81)	C(82)	C(83)	120.6(6)
C(33)	C(34)	C(35)	117.7(6)	C(82)	C(83)	C(84)	120.3(6)
C(34)	C(35)	C(36)	121.3(6)	C(83)	C(84)	C(85)	119.8(7)
C(31)	C(36)	C(35)	120.2(6)	C(84)	C(85)	C(86)	118.1(7)
P(2)	C(41)	C(42)	121.6(5)	C(81)	C(86)	C(85)	121.5(6)

Table S-22. Selected Interatomic Angles for **15** (continued)

(c) within the tetrafluoroborate ion

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
F(1)	B	F(2)	111.3(9)	F(2)	B	F(3)	107.8(7)
F(1)	B	F(3)	108.3(8)	F(2)	B	F(4)	114.0(9)
F(1)	B	F(4)	107.3(7)	F(3)	B	F(4)	108.0(10)

(d) within the solvent diethyl ether molecule

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C(1S)	O(1S)	C(3S)	113.0(8)	O(1S)	C(3S)	C(4S)	108.3(8)
O(1S)	C(1S)	C(2S)	109.2(8)				

Table S-23. Anisotropic Displacement Parameters (U_{ij}, Å^2) for **15**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Os	0.02166(10)	0.03025(11)	0.02173(10)	-0.00304(9)	0.00289(7)	-0.00029(9)
Rh	0.0247(2)	0.0304(2)	0.0279(2)	-0.00726(18)	0.00119(16)	0.00050(17)
P(1)	0.0225(7)	0.0388(8)	0.0303(7)	-0.0080(6)	0.0010(6)	-0.0026(6)
P(2)	0.0287(7)	0.0303(7)	0.0303(7)	-0.0055(6)	0.0038(6)	-0.0030(6)
P(3)	0.0276(7)	0.0268(7)	0.0221(7)	-0.0014(5)	0.0029(5)	0.0019(5)
P(4)	0.0267(7)	0.0294(7)	0.0248(7)	-0.0029(6)	-0.0003(5)	0.0004(6)
O(1)	0.028(2)	0.042(2)	0.029(2)	0.0009(16)	0.0058(17)	-0.0017(16)
O(2)	0.037(2)	0.047(2)	0.038(2)	0.009(2)	0.0118(18)	-0.0014(18)
O(3)	0.040(3)	0.073(3)	0.077(3)	-0.028(3)	-0.010(2)	0.020(2)
O(90)	0.058(3)	0.084(4)	0.039(3)	0.021(2)	0.002(2)	0.023(3)
O(91)	0.099(4)	0.092(4)	0.059(3)	0.029(3)	0.028(3)	0.069(3)
O(92)	0.054(3)	0.051(3)	0.034(2)	0.007(2)	0.019(2)	0.004(2)
O(93)	0.056(3)	0.038(2)	0.043(2)	0.0051(19)	0.003(2)	-0.001(2)
C(1)	0.030(3)	0.029(3)	0.024(3)	-0.005(2)	-0.003(2)	0.001(2)
C(2)	0.027(3)	0.032(3)	0.034(3)	-0.009(2)	0.000(2)	0.001(2)
C(3)	0.040(4)	0.043(3)	0.038(3)	-0.018(3)	-0.003(3)	0.004(3)
C(4)	0.025(3)	0.039(3)	0.027(3)	-0.001(2)	0.004(2)	-0.001(2)
C(5)	0.027(3)	0.053(4)	0.035(3)	0.001(3)	0.007(2)	0.008(3)
C(6)	0.032(3)	0.037(3)	0.024(3)	0.001(2)	0.002(2)	0.005(2)
C(7)	0.027(3)	0.039(3)	0.026(3)	-0.001(2)	0.002(2)	-0.001(2)
C(8)	0.030(3)	0.042(3)	0.030(3)	-0.008(2)	0.003(2)	-0.009(2)
C(9)	0.032(3)	0.030(3)	0.026(3)	-0.002(2)	0.000(2)	0.006(2)
C(11)	0.024(3)	0.059(4)	0.035(3)	-0.024(3)	-0.001(2)	-0.001(3)
C(12)	0.030(3)	0.080(5)	0.037(3)	-0.021(3)	-0.007(3)	0.006(3)
C(13)	0.042(4)	0.108(6)	0.046(4)	-0.027(4)	-0.012(3)	0.022(4)
C(14)	0.030(4)	0.125(8)	0.073(6)	-0.051(6)	0.000(4)	-0.009(5)
C(15)	0.037(4)	0.087(6)	0.101(7)	-0.035(5)	0.006(4)	-0.016(4)
C(16)	0.029(3)	0.072(5)	0.075(5)	-0.027(4)	0.003(3)	-0.013(3)
C(21)	0.032(3)	0.042(3)	0.027(3)	-0.006(2)	-0.001(2)	0.007(2)
C(22)	0.034(3)	0.050(4)	0.034(3)	-0.009(3)	-0.003(3)	-0.001(3)
C(23)	0.046(4)	0.070(5)	0.039(4)	0.006(3)	0.007(3)	0.005(3)
C(24)	0.056(4)	0.071(5)	0.029(3)	-0.007(3)	0.002(3)	0.023(4)
C(25)	0.076(5)	0.052(4)	0.037(4)	-0.011(3)	-0.011(3)	0.006(4)
C(26)	0.062(4)	0.049(4)	0.037(4)	-0.010(3)	-0.011(3)	-0.008(3)
C(31)	0.037(3)	0.031(3)	0.031(3)	-0.005(2)	0.003(2)	0.003(2)
C(32)	0.039(3)	0.036(3)	0.035(3)	-0.005(3)	0.005(3)	-0.002(3)
C(33)	0.039(3)	0.050(4)	0.045(4)	0.009(3)	0.013(3)	0.000(3)
C(34)	0.041(4)	0.067(4)	0.036(4)	-0.011(3)	0.011(3)	0.005(3)
C(35)	0.068(5)	0.086(6)	0.043(4)	-0.028(4)	0.016(4)	-0.011(4)
C(36)	0.049(4)	0.070(5)	0.040(4)	-0.023(3)	0.013(3)	-0.016(3)

Table S-23. Anisotropic Displacement Parameters for **15** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(41)	0.042(3)	0.040(3)	0.032(3)	0.003(3)	0.006(3)	-0.003(3)
C(42)	0.060(4)	0.063(4)	0.046(4)	0.010(3)	-0.001(3)	-0.014(4)
C(43)	0.065(5)	0.089(6)	0.048(4)	0.029(4)	0.000(4)	-0.006(4)
C(44)	0.051(4)	0.038(4)	0.097(6)	0.032(4)	0.039(4)	0.007(3)
C(45)	0.100(6)	0.038(4)	0.072(5)	0.009(4)	0.016(5)	-0.010(4)
C(46)	0.087(6)	0.042(4)	0.063(5)	0.006(4)	-0.018(4)	-0.015(4)
C(51)	0.033(3)	0.035(3)	0.026(3)	-0.002(2)	0.001(2)	0.006(2)
C(52)	0.056(4)	0.047(4)	0.038(3)	0.003(3)	0.013(3)	0.012(3)
C(53)	0.067(5)	0.052(4)	0.072(5)	0.005(4)	0.024(4)	0.028(4)
C(54)	0.071(5)	0.084(6)	0.054(5)	-0.006(4)	0.026(4)	0.034(4)
C(55)	0.065(5)	0.084(5)	0.041(4)	0.008(4)	0.025(3)	0.016(4)
C(56)	0.053(4)	0.052(4)	0.040(4)	0.008(3)	0.017(3)	0.011(3)
C(61)	0.030(3)	0.029(3)	0.029(3)	-0.005(2)	-0.002(2)	0.004(2)
C(62)	0.036(3)	0.034(3)	0.028(3)	-0.004(2)	0.001(2)	-0.006(2)
C(63)	0.045(4)	0.042(3)	0.038(3)	0.000(3)	0.004(3)	-0.009(3)
C(64)	0.045(4)	0.037(3)	0.055(4)	0.008(3)	-0.005(3)	-0.015(3)
C(65)	0.063(4)	0.033(3)	0.045(4)	-0.006(3)	-0.008(3)	-0.015(3)
C(66)	0.062(4)	0.032(3)	0.025(3)	-0.005(2)	0.001(3)	-0.008(3)
C(71)	0.025(3)	0.031(3)	0.033(3)	-0.001(2)	-0.002(2)	0.002(2)
C(72)	0.030(3)	0.041(3)	0.026(3)	-0.002(2)	-0.002(2)	0.000(2)
C(73)	0.032(3)	0.053(4)	0.036(3)	0.000(3)	0.008(2)	0.001(3)
C(74)	0.036(3)	0.040(3)	0.052(4)	0.000(3)	0.006(3)	-0.002(3)
C(75)	0.041(3)	0.043(3)	0.048(4)	-0.011(3)	0.004(3)	-0.007(3)
C(76)	0.038(3)	0.041(3)	0.030(3)	-0.007(3)	0.006(2)	-0.006(3)
C(81)	0.036(3)	0.029(3)	0.034(3)	-0.001(2)	-0.003(2)	0.004(2)
C(82)	0.034(3)	0.049(4)	0.043(4)	0.002(3)	0.000(3)	0.007(3)
C(83)	0.046(4)	0.056(4)	0.065(5)	0.015(4)	-0.007(3)	0.012(3)
C(84)	0.096(6)	0.071(5)	0.066(5)	0.025(4)	-0.005(5)	0.027(5)
C(85)	0.084(6)	0.060(5)	0.076(6)	0.039(4)	0.024(4)	0.013(4)
C(86)	0.036(4)	0.064(4)	0.075(5)	0.026(4)	-0.003(3)	0.003(3)
C(90)	0.036(3)	0.050(4)	0.045(4)	0.007(3)	0.004(3)	0.010(3)
C(91)	0.147(9)	0.148(9)	0.093(7)	0.069(7)	0.050(7)	0.113(8)
C(92)	0.037(3)	0.039(3)	0.029(3)	0.003(3)	-0.004(3)	0.003(3)
C(93)	0.079(5)	0.037(4)	0.064(5)	0.004(3)	-0.005(4)	-0.010(3)
F(1)	0.105(4)	0.061(3)	0.110(4)	-0.033(3)	-0.038(3)	-0.012(3)
F(2)	0.162(5)	0.097(4)	0.057(3)	-0.005(3)	-0.031(3)	-0.065(4)
F(3)	0.181(7)	0.096(4)	0.163(6)	0.027(4)	-0.086(5)	-0.040(4)
F(4)	0.119(5)	0.270(10)	0.233(9)	-0.191(8)	0.071(6)	-0.065(6)
B	0.079(7)	0.073(7)	0.064(6)	-0.021(5)	0.006(5)	-0.032(5)
O(1S)	0.115(5)	0.070(4)	0.066(4)	-0.015(3)	0.021(3)	-0.015(3)

Table S-23. Anisotropic Displacement Parameters for **15** (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1S)	0.118(8)	0.103(8)	0.066(6)	-0.042(6)	0.006(6)	-0.024(6)
C(2S)	0.154(11)	0.165(12)	0.088(8)	0.061(8)	-0.040(7)	-0.059(9)
C(3S)	0.172(12)	0.047(6)	0.154(11)	0.005(6)	0.023(9)	-0.007(7)
C(4S)	0.156(11)	0.072(7)	0.120(9)	0.013(6)	0.003(8)	0.000(7)

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-24. Derived Coordinates and Displacement Parameters for Hydrogen Atoms of **15**

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(4A)	-0.0821	0.1246	0.4415	0.036
H(4B)	-0.0401	0.1593	0.5085	0.036
H(5A)	-0.1843	0.1811	0.3966	0.046
H(5B)	-0.1409	0.2162	0.4629	0.046
H(8A)	0.0270	0.0332	0.3755	0.041
H(8B)	0.0428	0.0211	0.2861	0.041
H(9A)	0.2749	0.1956	0.6139	0.035
H(9B)	0.1918	0.1529	0.6051	0.035
H(12)	-0.1104	0.1657	0.2294	0.059
H(13)	-0.2958	0.1733	0.2184	0.079
H(14)	-0.4042	0.1148	0.2699	0.091
H(15)	-0.3307	0.0513	0.3355	0.090
H(16)	-0.1460	0.0443	0.3491	0.071
H(22)	0.1721	0.1594	0.2026	0.047
H(23)	0.2113	0.1564	0.0706	0.062
H(24)	0.1342	0.0983	-0.0079	0.062
H(25)	0.0109	0.0469	0.0422	0.066
H(26)	-0.0335	0.0504	0.1728	0.059
H(32)	0.3890	0.0780	0.2800	0.044
H(33)	0.4755	0.0734	0.1637	0.054
H(34)	0.4138	0.0246	0.0651	0.057
H(35)	0.2580	-0.0191	0.0847	0.078
H(36)	0.1680	-0.0134	0.2002	0.063
H(42)	0.2975	-0.0126	0.4826	0.068
H(43)	0.3150	-0.0930	0.5253	0.081
H(44)	0.2563	-0.1543	0.4431	0.074
H(45)	0.1433	-0.1347	0.3371	0.084
H(46)	0.1332	-0.0567	0.2950	0.077
H(52)	0.0415	0.2897	0.4835	0.056
H(53)	-0.0944	0.3269	0.5503	0.076
H(54)	-0.1553	0.2942	0.6639	0.083
H(55)	-0.0776	0.2261	0.7143	0.076
H(56)	0.0588	0.1879	0.6485	0.058
H(62)	0.2777	0.2474	0.3701	0.039
H(63)	0.3941	0.3113	0.3536	0.050
H(64)	0.4419	0.3584	0.4579	0.055
H(65)	0.3741	0.3426	0.5798	0.057
H(66)	0.2582	0.2798	0.5972	0.047
H(72)	0.4778	0.1548	0.4067	0.039
H(73)	0.6168	0.2065	0.3831	0.048

Table S-24. Derived Parameters for Hydrogen Atoms of **15** (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H(74)	0.6766	0.2587	0.4792	0.051
H(75)	0.6003	0.2571	0.6011	0.053
H(76)	0.4627	0.2043	0.6256	0.044
H(82)	0.5404	0.1044	0.6073	0.050
H(83)	0.5847	0.0513	0.7054	0.066
H(84)	0.4534	0.0211	0.7846	0.093
H(85)	0.2708	0.0369	0.7541	0.088
H(86)	0.2298	0.0800	0.6429	0.070
H(91A)	-0.3088	0.3313	0.3642	0.154
H(91B)	-0.2995	0.3042	0.2833	0.154
H(91C)	-0.2157	0.3446	0.3066	0.154
H(93A)	0.1246	0.3604	0.2928	0.072
H(93B)	0.1141	0.3262	0.2193	0.072
H(93C)	0.2158	0.3227	0.2756	0.072
H(1SA)	0.2856	-0.0824	-0.1093	0.115
H(1SB)	0.2241	-0.0692	-0.0319	0.115
H(2SA)	0.1974	-0.0121	-0.1238	0.163
H(2SB)	0.3225	-0.0037	-0.1283	0.163
H(2SC)	0.2612	0.0094	-0.0511	0.163
H(3SA)	0.4019	-0.1288	-0.0247	0.149
H(3SB)	0.3409	-0.1096	0.0496	0.149
H(4SA)	0.5191	-0.1290	0.0864	0.139
H(4SB)	0.5023	-0.0730	0.0958	0.139
H(4SC)	0.5627	-0.0933	0.0224	0.139

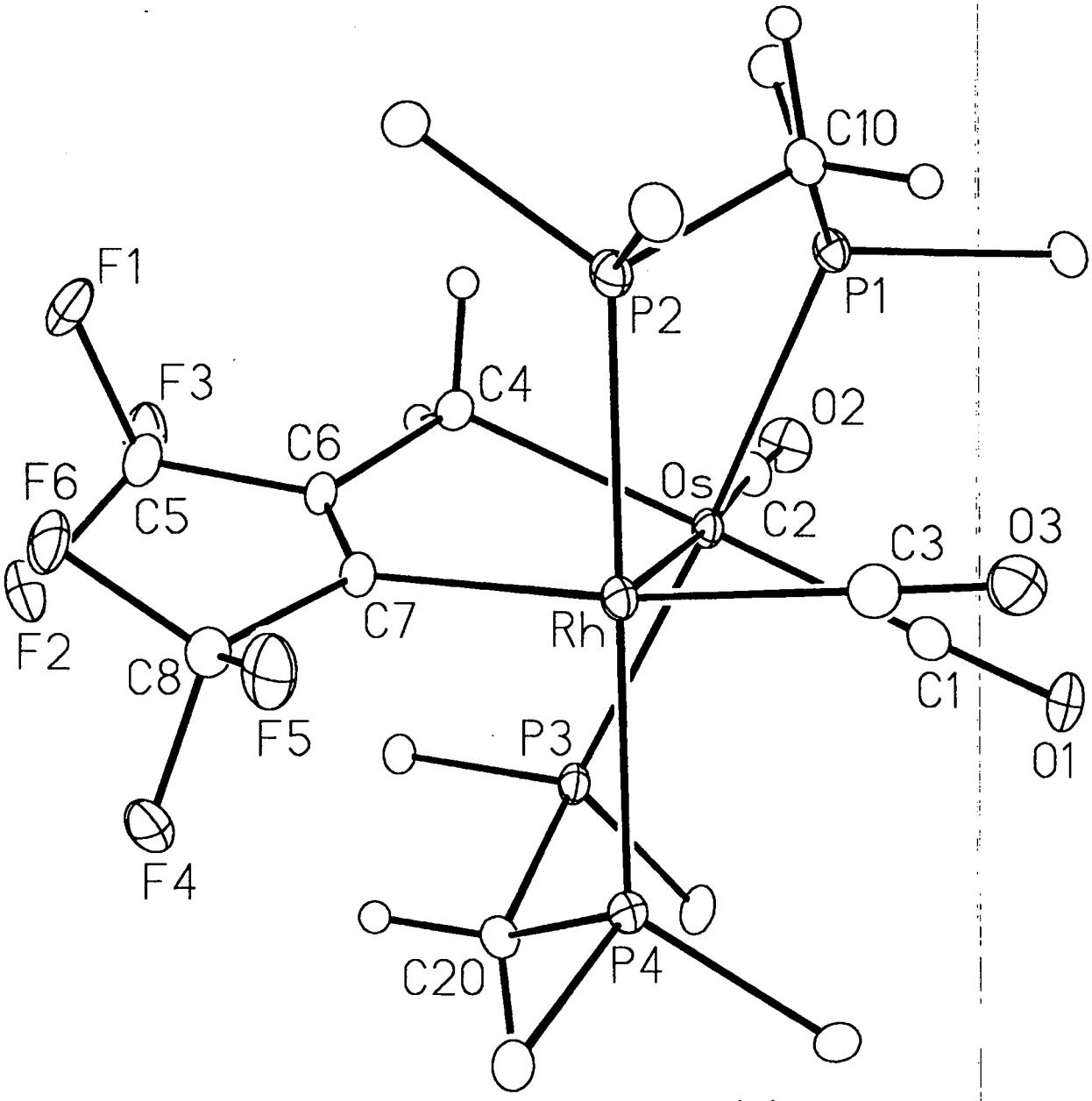

Figure S1. Perspective view of the cation of $[RhOs(CO)_3(\mu\text{-}\eta^1\text{:}\eta^1\text{-}(CF_3)C{=}C(CF_3)CH_2)\text{-}(dppm)_2][CF_3SO_6]$ (**6**). Thermal ellipsoids at the 20% level except for hydrogen atoms, which are shown artificially small. Only the ipso carbons of the dppm phenyl groups are shown.

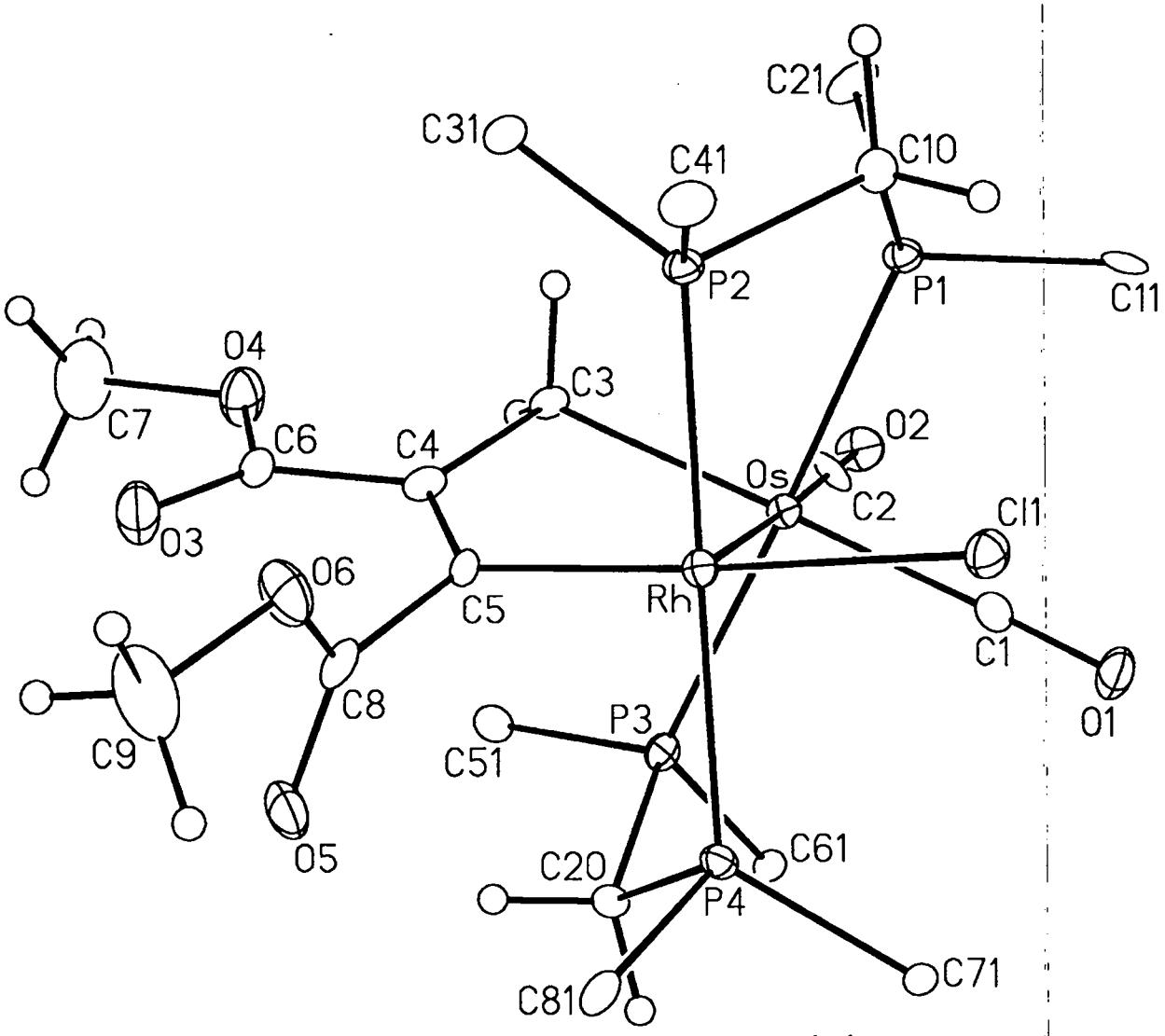

Figure S2. Perspective view of the cation of $[RhOsCl(CO)_2(\mu\text{-}\eta^1{:}\eta^1\text{-}(CO_2Me)C{=}C(CO_2Me))CH_2)(dppm)_2]$ (**8**). Thermal ellipsoids at the 20% level except for hydrogen atoms, which are shown artificially small. Only the ipso carbons of the dppm phenyl groups are shown.