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Experimental Details, NMR Spectroscopic and Microanalytical Data for Complexes 2-9

Complex 2: $Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)I(PMe_3)$, via $Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)I(CO)$

$Rh(\eta^5\text{-}C_5Me_5)(CO)_2$ (226 mg, 0.768 mmol) was dissolved in benzene (14 mL). Then $n\text{-}C_3F_7I$ (273 mg, 0.922 mmol) was added as a solution in benzene (1 mL). The reaction mixture was stirred at room temperature for 3 hours. The volatiles were removed under vacuum leaving behind an orange powder. The desired product recrystallized from methylene chloride and hexanes at -20°C as deep red crystals (375 mg, 87%). mp: 131-134 °C. IR (CH_2Cl_2): υ_{CO} = 2064 cm^{-1}; (C_6H_6): υ_{CO} = 2070 cm^{-1}. ^1H NMR ($CDCl_3$) δ 2.07 (15H, C_5Me_5); (C_6D_6) δ 1.44 (15H, C_5Me_5). ^{19}F NMR ($CDCl_3$) δ – 59.4 (d, J_{AB} = 252, 1F, $C_\alpha F_A$), – 70.1 (d, J_{AB} = 252, 1F, $C_\alpha F_B$),– 79.5 (t, J_{FF} = 12, 3F, CF_3), – 113.0 (d, J_{AB} = 282, 1F, $C_\beta F_A$), – 116.3 (d, J_{AB} = 282, 1F, $C_\beta F_B$); (C_6D_6) δ – 59.6 (d, J_{AB} = 253, 1F, $C_\alpha F_A$), – 69.5 (d, J_{AB} = 253, 1F, $C_\alpha F_B$), – 78.9 (t, J_{FF} = 10, 3F, CF_3), – 112.1 (d, J_{AB} = 285, 1F, $C_\beta F_A$), – 115.5 (d, J_{AB} = 285, 1F, $C_\beta F_B$). Anal. Calcd. for $C_{14}H_{15}F_7IORh$: C, 29.92; H, 2.64. Found: C, 29.82; H, 2.49.

$Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)I(CO)$ (200 mg, 0.356 mmol) was dissolved in benzene (5 mL) and PMe_3 (37 mL, 0.356 mmol) was added. The reaction was monitored by IR until the terminal carbonyl band disappeared (ca. 2 hours). The volatiles were removed under vacuum to give an orange powder which was washed with hexanes and dried again under vacuum affording the product in 85% yield (184 mg). mp: 151-153°C. ^1H NMR ($CDCl_3$) δ 1.85 (d, J_{HRh} = 2.9, 15H, C_5Me_5), 1.62 (d, J_{HP} = 10.3, 9H, PMe_3); (C_6D_6) δ 1.50 (d, J_{HRh} = 3.0, 15H, C_5Me_5), 1.22 (d, J_{HP} = 10.5, 9H, PMe_3). ^{19}F NMR ($CDCl_3$) δ – 66.8 (d, J_{AB} = 276, 1F, $C_\alpha F_A$), – 69.2 (d, J_{AB} = 276, 1F, $C_\alpha F_B$), –79.5 (t, J_{FF} = 12, CF_3), – 114.4 (d, J_{AB} =280, 1F, $C_\beta F_A$), –115.7 (d, J_{AB} = 280, 1F, $C_\beta F_B$); (C_6D_6) δ – 66.3 (d, J_{AB} = 277, 1F, $C_\alpha F_A$), – 68.7 (d, J_{AB} = 277, 1F, $C_\alpha F_B$), –78.7 (t, J_{FF} = 12, CF_3), – 113.7 (d, J_{AB} = 281, 1F, $C_\beta F_A$), –114.9 (d, J_{AB} = 281, 1F, $C_\beta F_B$. $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ 3.2 (dd, J_{PRh} = 151, J_{PF} = 15); (C_6D_6) δ 2.9 (dm, J_{PRh} = 150). Anal. Calcd. for $C_{16}H_{24}F_7IPRh$: C, 31.50; H, 3.97. Found: C, 31.70; H; 3.70.

Complex 4: $Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)(PMe_3)(OH_2)^+ BF_4^-$

$Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)I(PMe_3)$ (300 mg, 0.492 mmol) was dissolved in CH_2Cl_2 (10 mL) to give an orange colored solution and distilled H_2O (9 μL, 0.492) was added. Another flask was charged

with $AgBF_4$ (115 mg, 0.590 mmol) and CH_2Cl_2 (10 mL) was added. While the rhodium/water solution was cannula transferred (slowly over about 10 minutes) to the stirring silver mixture, a fluffy pale yellow precipitate formed and the solution became more yellow. It is essential to add the rhodium solution to the $AgBF_4$ and not the other way around otherwise the reaction does not go to completion. The reaction mixture was stirred for 20 minutes after which it was filtered via cannula. The filtrate was concentrated to about half the original volume and hexanes (10 mL) were added to precipitate the product. The last step was repeated twice more and after concentrating the last time the supernatant was removed. The yellow-orange solid was dried under vacuum giving a fluffy material (286 mg, 99%, usually ca. 90%). mp: decomposed to brown at 150°C then melted 151-159°C and turned black. IR (CH_2Cl_2) υ_{H2O} = 3600, 3352 cm^{-1}. ^1H NMR ($CDCl_3$) δ 3.49 (br s, H_2O), 1.67 (d, J_{HRh} = 2.7, C_5Me_5), 1.59 (d, J_{HP} = 11.0, PMe_3). (CD_2Cl_2) δ 3.19 (br s, H_2O, 2H), 1.65 (d, J_{HRh} = 3.0, C_5Me_5), 1.57 (d, J_{HP} = 11.0, PMe_3). ^{19}F NMR ($CDCl_3$) δ –79.7 (t, J_{FF} = 12, 3F, CF_3), –80.7 (d, J_{AB} = 265, 1F, $C_\alpha F_A$), – 90.4 (d, J_{AB} = 265, 1F, $C_\alpha F_B$), –115.5 (d, J_{AB} = 285, 1F, $C_\beta F_A$), –118.0 (d, J_{AB} = 285, 1F, $C_\beta F_B$), –150.5 (s, 4F, BF_4). (CD_2Cl_2) δ –79.5 (t, J_{FF} = 11, 3F, CF_3), –80.4 (dm, J_{AB} = 273, 1F, $C_\alpha F_A$), – 90.8 (dm, J_{AB} = 273, 1F, $C_\alpha F_B$), –114.8 (d, J_{AB} = 291, 1F, $C_\beta F_A$), –117.3 (d, J_{AB} = 291, 1F, $C_\beta F_B$), –150.3 (s, 4F, BF_4,). $^{31}P\{^1H\}$NMR ($CDCl_3$) δ 4.6 (d, J_{PRh} = 163, PMe_3). (CD_2Cl_2) δ 4.2 (dm, J_{PRh} = 153, PMe_3). Anal. Calcd. for $C_{16}H_{26}BF_{11}OPRh$: C, 32.68; H, 4.46. Found: C, 32.50; H, 4.24.

Complex 3: $Rh(\eta^5\text{-}C_5Me_5)(CF_2C_6F_5)(OH_2)(PMe_3)^+ BF_4^-$

This was prepared as described for **4.** Yield: 85%. IR (CH_2Cl_2) υ_{OH} = 3630, 3370 cm^{-1}. ^1H NMR ($CDCl_3$, –50°C) δ 3.38 (br s, 1.5H), 1.66 (d, J_{HP} = 10.7, PMe_3), 1.41 (br s, C_5Me_5); ($CDCl_3$) δ 3.32 (br s, 1.5H), 1.67 (d, J_{HP} = 11.1, PMe_3), 1.51 (br s, C_5Me_5). ^{19}F NMR ($CDCl_3$, –50°C) δ –56.3 (dm, J_{AB} = 263, 1F, $C_\alpha F_A$), – 66.8 (dm, J_{AB} = 263, 1F, $C_\alpha F_B$), –142.4 (m , 2F, ortho), –153.4 (m s, 1F, para), –151.0 (s, BF_4), –160.7 (m, 2F, meta); ($CDCl_3$) δ –55.6 (br s, 1F, CF_2), – 64.7 (br s, 1F, CF_2), –142.0 (br s , 2F, ortho), –154.3 (br s, 1F, para), –156.0 (s, BF_4), –161.9 (br s, 2F, meta). $^{31}P\{^1H\}$NMR ($CDCl_3$, –50°C) δ 12.1 (ddd, J_{PRh} = 155, J_{PF} = 66, J_{PF} =

15, PMe_3); ($CDCl_3$) δ 8.4 (dt, J_{PRh} = 162, J_{PF} = 39, PMe_3).

Complex 5: $Rh(\eta^5\text{-}C_5Me_5)(C_6F_5)(CO)(PMe_3)^+BF_4^-$

An NMR sample of $Rh(\eta^5\text{-}C_5Me_5)(CF_2C_6F_5)(OH_2(PMe_3)^+ BF_4^-$ in $CDCl_3$ was monitored over the course of 18 hours whereupon the original compound converted completely to $Rh(\eta^5\text{-}C_5Me_5)(C_6F_5)(CO)(PMe_3)^+BF_4^-$. The solution was taken to dryness under vacuum leaving a pale yellow solid: mp: 118–119°C. IR(CH_2Cl_2): υ_{CO} = 2074 cm^{-1}. ^1H NMR ($CDCl_3$) δ 1.68 (d, J_{HP} =11.5, PMe_3), 1.96 (d, J_{HRh} = 3.4, C_5Me_5). ^{19}F NMR ($CDCl_3$) δ –106.1 (m, 1F, ortho), –113.4 (m, 1F, ortho), – 152.3 (s, 4F, BF_4), –155.3 (t, J_{FF} = 20, 1F, para), –159.0 (m, 2F, meta); $^{31}P\{^1H\}$NMR ($CDCl_3$) δ 3.7 (dd, J_{PRh} = 116, J_{PF} = 14, PMe_3). Anal. Calcd. for $C_{20}H_{24}BF_9OPRh$: C, 40.30; H, 4.06. Found: C, 40.16; H, 4.19.

Complex 6: $Rh(\eta^5\text{-}C_5Me_5)(CF_2C_6F_5)(OSO_2CF_3)(PMe_3)$.

$Rh(\eta^5\text{–}C_5Me_5)(CF_2C_6F_5)(I)(PMe_3)$ (230 mg, 0.349 mmol) was dissolved in CH_2Cl_2 (5 mL) to give a deep red-orange colored solution. $AgSO_3CF_3$ (90 mg, 0.349 mmol) was added as a solid and within 30 seconds a white fluffy precipitate formed. The reaction mixture was stirred for 1 hour over which time the precipitate changed to a tan color. The reaction mixture was filtered via cannula to give a yellow solution which was concentrated to ca 0.5 mL. The solution was pipetted off and the remaining solid washed with hexanes (2 x 1 mL) and dried under vacuum to give an orange powder (144 mg, 61%). mp: 149-152°C. ^1H NMR ($CDCl_3$) δ 1.67 (d, J_{HRh} = 3.2, C_5Me_5), 1.46 (d, J_{HP} = 10.7, PMe_3); (CD_2Cl_2) δ 1.65 (d, J_{HRh} = 3.4, C_5Me_5), 1.44 (d, J_{HP} = 11.0, PMe_3); (C_6D_6) δ 1.28 (d, J_{HRh} = 3.2, C_5Me_5), 1.04 (d, J_{HP} = 11.0, PMe_3). ^{19}F NMR ($CDCl_3$) δ –50.5 (d, J_{AB} = 246, 1F, $C_\alpha F_A$), –58.7 (ddt, J_{AB} = 246, J_{FP} = 66, J_{FF} = 36, 1F, $C_\alpha F_B$), –76.3 (s, 3F, OTf), –140.4 (m, 2F, ortho), –157.8 (t, J_{FF} = 22, 1F, para), –165.1 (m, 2F, meta); (CD_2Cl_2) δ –50.6 (dtd, J_{AB} = 246, J_{FF} = 15, J_{FP} = 3, 1F, $C_\alpha F_A$), –58.8 (dddt, J_{AB} = 247, J_{FP} = 61, J_{FF} = 36, J_{FRh} = 10, 1F, $C_\alpha F_B$), –75.9 (s, 3F, OTf), –140.0 (m, 2F, ortho), –158.5 (m, 1F, para), –165.4 (m, 2F, meta); (C_6D_6) δ –50.4 (dt, J_{AB} = 246, J_{FF} = 14, 1F, $C_\alpha F_A$), –58.1 (dddt, J_{AB} =

246, J_{FP} = 58, J_{FF} = 30, J_{FRh} = 14, 1F, $C_\alpha F_B$), –75.2 (s, 3F, OTf), –140.0 (m, 2F, ortho), –157.7 (t, J_{FF} = 22, 1F, para), –164.8 (m, 2F, meta). $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ 5.5 (dd, J_{PRh} = 154, J_{PF} = 61, PMe_3); (CD_2Cl_2) δ 5.5 (ddd, J_{PRh} = 154, J_{PF} = 61, J_{PF} = 3, PMe_3); (C_6D_6) δ 4.8 (ddd, J_{PRh} = 155, J_{PF} = 60, J_{PF} = 3, PMe_3).

Complex 7: $Rh(\eta^5\text{-}C_5Me_5)(C_6F_5)(CO)(PMe_3)^+ B\{Ar_F\}_4^-$

$Rh(\eta^5\text{-}C_5Me_5)(CF_2C_6F_5)(OTf)(PMe_3)$ (200 mg, 0.294 mmol) was dissolved in CH_2Cl_2 (5 mL) and $NaB\{Ar_F\}_4$ 262 mg, 0.294 mmol) was added as a solid. Immediately the reaction mixture became a darker red in color (started orange) and precipitate formed. After 10 minutes the color faded to an orange–yellow. An additional half equivalent of $NaB\{Ar_F\}_4$ (131 mg, 0.147 mmol) was added and the color change was the same as noted above. The reaction mixture was filtered via cannula and the volatiles were removed under vacuum to give a yellow solid (332 mg, 81% yield): IR(CH_2Cl_2): υ_{CO} = 2078 cm^{-1}. ^1H NMR (CD_2Cl_2) δ 1.58 (d, J_{HP} =11.2, PMe_3), 1.89 (d, J_{HRh} = 3.4, C_5Me_5). ^{19}F NMR (CD_2Cl_2) δ –62.0 (s, 24F, $B\{Ar_F\}_4$), –106.0 (m, 1F, ortho), –113.7 (m, 1F, ortho), –154.8 (t, J_{FF} = 19, 1F, para), –158.5 (m, 2F, meta); $^{31}P\{^1H\}$NMR (CD_2Cl_2) δ 4.1 (ddd, J_{PRh} = 118, J_{PF} = 18, J_{PF} = 4, PMe_3).

Complex 8: $Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)(OSO_2CF_3)(PMe_3)$

$Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)(I)(PMe_3)$ (250 mg, 0.410 mmol) was dissolved in CH_2Cl_2 (5 mL) to give a red-orange colored solution. Then $AgSO_3CF_3$ (105 mg, 0.410 mmol) was added to a different Schlenk flask and CH_2Cl_2 (12 mL) was added. The rhodium solution was cannula transferred to the silver triflate mixture, dropwise. Additional CH_2Cl_2 (3 mL) was added to the rhodium flask and the contents were transferred to the reaction mixture via cannula. The reaction mixture was stirred for 30 minutes over which time a white precipitate was formed. The reaction mixture was transferred to a glass frit and filtered through Celite to give an orange-red solution. The Celite was rinsed with CH_2Cl_2 until the filtrate was colorless. The filtrate was concentrated to 1 mL under vacuum then hexanes (6 mL) were added to precipitate the product. The last step was repeated

once more using 3 mL hexanes and then the supernatant was filtered off. The remaining solid was dried under vacuum to give an orange powder in 94 % yield (241 mg). mp: 172-173°C. ^{1}H NMR ($CDCl_3$) δ 1.69 (d, J_{HRh} = 2.7, C_5Me_5), 1.63 (d, J_{HP} = 11.2 PMe_3). (C_6D_6) δ 1.23 (d, J_{HP} = 9.0, PMe_3), 1.22 (d, J_{HRh} = 2.9, C_5Me_5). ^{19}F NMR ($CDCl_3$) δ –78.4 (d, J_{AB} = 271, 1F, $C_\alpha F_A$), –78.6 (s, 3F, OTf), –79.9 (t, J_{FF} = 11, 3F, CF_3), –85.4 (d, J_{AB} = 271, 1F, $C_\alpha F_B$), –115.6 (d, J_{AB} = 288, 1F, $C_\beta F_A$), –118.7 (d, J_{AB} = 288, 1F, $C_\beta F_B$). (C_6D_6) δ –77.8 (s, 3F, OTf), –78.2 (d, J_{AB} = 273, 1F, $C_\alpha F_A$), –79.3 (t, J_{FF} = 11, 3F, CF_3), –84.3 (d, J_{AB} = 273, 1F, $C_\alpha F_B$), –115.4 (dt, J_{AB} = 289, $J_{FF/P}$ = 11, 1F, $C_\beta F_A$), –118.4 (d, J_{AB} = 289, 1F, $C_\beta F_B$). ^{19}F{^{31}P} NMR (C_6D_6) δ –77.8 (s, 3F, OTf), –78.2 (d, J_{AB} = 273, 1F, $C_\alpha F_A$), –79.3 (t, J_{FF} = 11, 3F, CF_3), –84.3 (d, J_{AB} = 273, 1F, $C_\alpha F_B$), –115.4 (dd, J_{AB} = 289, J_{FF} = 13, 1F, $C_\beta F_A$), –118.4 (d, J_{AB} = 289, 1F, $C_\beta F_B$). ^{31}P{^{1}H} NMR ($CDCl_3$) δ 7.6 (dm, J_{PRh} = 151, PMe_3). (C_6D_6) δ 7.6 (ddm, J_{PRh} = 155, J_{PF} = 35, PMe_3). Anal. Calcd. for $C_{17}H_{24}F_{10}O_3PRhS$: C, 32.29; H, 3.83. Found: C, 31.99; H, 3.81.

Complex 9: $Rh(\eta^5\text{-}C_5Me_5)(C_2F_5)(CO)(PMe_3)^+B\{Ar_F\}_4^-$

$Rh(\eta^5\text{-}C_5Me_5)(n\text{-}C_3F_7)(OTf)(PMe_3)$ (20 mg, 0.032 mmol) was dissolved in CH_2Cl_2 (1.5 mL) to give an orange–yellow solution. Then $NaB\{Ar_F\}_4$ (28 mg, 0.032 mmol) was added as a solid, resulting in the immediate precipitation of NaOTf. The reaction was stirred for 1 hour after which the mixture was filtered through a plug of glass wool and then dried under vacuum. ^{1}H NMR (CD_2Cl_2) δ 7.74 (br s, $B\{Ar_F\}_4$), 7.58(s, $B\{Ar_F\}_4$), 2.50 (br s, H_2O), 1.94 (d, J_{HRh} = 3.4, C_5Me_5), 1.70 (d, J_{HP} = 11.5), 1.60 (d, J_{HRh} = 3.2, C_5Me_5), 1.51 (d, J_{HP} = 10.5, PMe_3). ^{19}F NMR (CD_2Cl_2) δ –62.7 (s, $NaB\{Ar_F\}_4$), –71.8 (ddd, J_{AB} = 255, J_{FP} = 50, J_{FF} = 9, 1F, $C_\alpha F_A$), –76.9 (d, J_{AB} = 255, 1F, $C_\alpha F_B$), –82.9 (3F, CF_3).

Complex 10: $Rh(\eta^5\text{-}C_5Me_5)(C_2F_5)(CO)(PMe_3)^+BF_4^-$

$Rh(\eta^5\text{-}C_5Me_5)(C_2F_5)I(PMe_3)$ [prepared as described above for complex **2**] (200 mg, 0.357 mmol) was dissolved in methylene chloride (6 mL). Then in a separate Schlenk flask $AgBF_4$ (76 mg, 0.393 mmol) and methylene chloride (6 mL) were added. Carbon monoxide was bubbled through

the $AgBF_4$ solution and through a cannula and into the rhodium solution for 1 minute (fast flow). Then the flow was reduced (2-3 bubbles/minute) and the CO pressure was used to cannula transfer the rhodium solution into the stirring $AgBF_4$ mixture. The reaction mixture turned a milky color immediately. After all the rhodium starting material was transferred the CO flow was turned off and the reaction mixture was stirred for 10 minutes. The mixture was filtered, the volatiles were removed under vacuum, the product washed with hexanes (3 x 4 mL), and dried under vacuum to give an off white solid (168 mg, 86%). mp: gradually becomes black then melts at 201-204°C. IR (CH_2Cl_2) $\upsilon_{CO} = 2078\ cm^{-1}$. ^1H NMR ($CDCl_3$) δ 2.02 (d, $J_{HRh} = 3.4$, C_5Me_5), 1.82 (d, $J_{HP} = 12$), ^{19}F NMR ($CDCl_3$) δ –73.2 (ddd, $J_{AB} = 257$, $J_{FP} = 50$, $J_{FRh} = 9$, 1F, $C_\alpha F_A$), –80.1 (d, $J_{AB} = 257$, 1F, $C_\alpha F_B$), –82.9 (3F, CF_3). $^{31}P\{^1H\}$ ($CDCl_3$) δ 5.5 (ddd, $J_{PRh} = 124$, $J_{PF} = 52$, $J_{PF} = 11$, PMe_3). Anal. Calcd. for $C_{16}H_{24}BF_9OPRh$: C, 35.07; H, 4.41. Found: C, 35.07; H, 4.04.

Crystallographic Structural Determinations.

Crystal, data collection, and refinement parameters are given in subsequent Tables. A suitable crystal for single-crystal X-ray diffraction was selected and mounted with epoxy cement in a nitrogen-flushed, thin-walled capillary. The unit-cell parameters were obtained by the least-squares refinement of the angular settings of 24 reflections ($20° \leq 2\theta \leq 25°$).

No evidence of symmetry higher than triclinic was observed in either the photographic or diffraction data for **3**; preliminary photographic data indicated a monoclinic crystal system and the systematic absences in the diffraction data are uniquely consistent with the reported space group for **4**. E-statistics suggested the centrosymmetric space group for **3**, and P1(bar) was chosen. The space group choices yielded chemically reasonable and computationally stable results of refinement. The structures were solved by direct methods, completed by subsequent difference Fourier syntheses and refined by full-matrix least-squares procedures. Semi-empirical ellipsoid absorption corrections were applied. The pentamethylcyclopentadienyl carbon atoms and the phenyl ring in **3** were fixed as rigid planar groups to conserve data. The fluorine atoms on the

counterion in **3** were fixed as an idealized tetrahedron with an average boron-fluorine bond distance. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were treated as idealized contributions except for the hydrogen atoms on oxygen, which were ignored. None of the remaining peaks in the difference map (max = 1.68eÅ^3) for **3** and (max = 0.452 eÅ^3) for **4** were in chemically reasonable positions and were considered noise.

All software and sources of the scattering factors are contained in the SHELXTL (5.03) program library (G. Sheldrick, Siemens XRD, Madison, WI).

CRYSTALLOGRAPHIC TABLES FOR

COMPLEX 3

ORTEP of **3**

Table 1S. Crystal data and structure refinement for **3**.

Table 2S. Atomic coordinates for **3**.

Table 3S. Bond lengths and angles for **3**.

Table 4S. Anisotropic displacement coefficients for **3**.

Table 5S. Hydrogen atom coordinates for **3**.

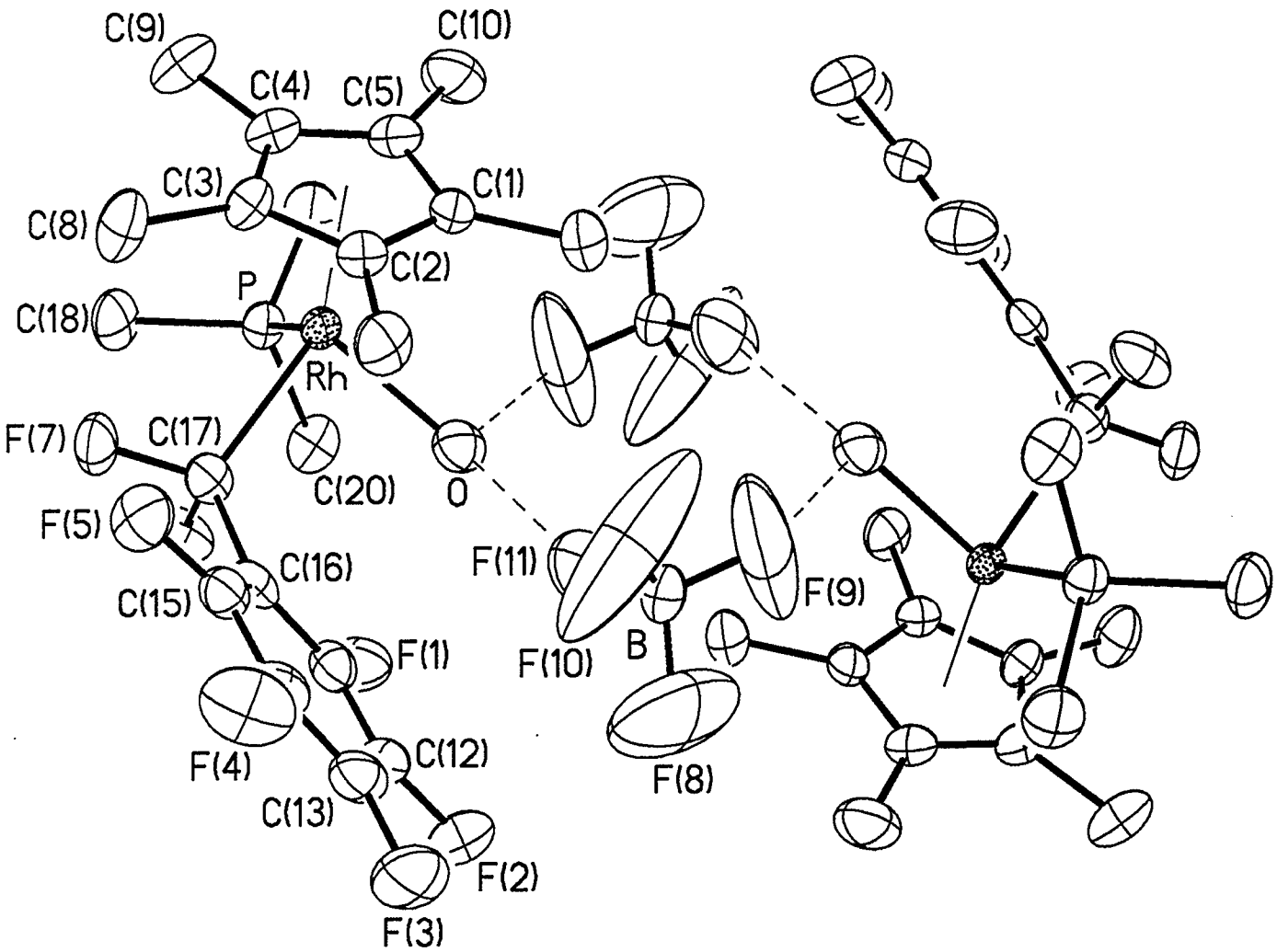

Table 1S. Crystal data and structure refinement for 3.

Identification code	rph24
Empirical formula	$C_{20}H_{26}BF_{11}OPRh$
Formula weight	636.10
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Crystal color	Orange-red block
Unit cell dimensions	a = 8.682(3) Å alpha = 74.50(1)° b = 11.033(2) Å beta = 76.11(2)° c = 14.376(3) Å gamma = 73.02(3)°
Volume, Z	1249.4(7) $Å^3$, 2
Density (calculated)	1.691 g/cm^3
Absorption coefficient	0.841 mm^{-1}
F(000)	636
Crystal size	0.40 x 0.30 x 0.20 mm
θ range for data collection	2.21 to 22.49°
Limiting indices	$-1 \le h \le 9$, $-11 \le k \le 11$, $-15 \le l \le 15$
Reflections collected	4015
Independent reflections	3246 (R_{int} = 0.0254)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3246 / 10 / 281
Goodness-of-fit on F^2	1.612
Final R indices [I>2σ(I)]	R1 = 0.0674, wR2 = 0.1949
R indices (all data)	R1 = 0.0746, wR2 = 0.2004
Largest diff. peak and hole	1.685 and -1.122 $eÅ^{-3}$

Table 25. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 3. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Rh	8907.8(7)	2941.6(5)	7586.6(4)	39(1)
P	9084(3)	4826(2)	7964(2)	47(1)
F(1)	13518(7)	1710(7)	7786(5)	86(2)
F(2)	15374(8)	-688(8)	7791(6)	104(2)
F(3)	15339(9)	-1960(6)	6429(7)	116(3)
F(4)	13285(10)	-860(7)	5135(6)	108(2)
F(5)	11362(8)	1491(6)	5116(4)	74(2)
F(6)	12031(7)	3757(5)	6717(4)	73(2)
F(7)	10746(7)	3643(5)	5630(4)	69(2)
C(1)	7103(5)	1643(3)	8150(2)	49(2)
C(2)	7792(5)	1575(3)	7159(2)	48(2)
C(3)	7334(5)	2833(3)	6568(2)	50(2)
C(4)	6362(5)	3679(3)	7194(3)	56(2)
C(5)	6219(4)	2943(4)	8172(2)	54(2)
C(6)	7279(7)	529(4)	9027(3)	69(3)
C(7)	8829(7)	376(4)	6797(4)	66(3)
C(8)	7799(8)	3207(5)	5467(2)	75(3)
C(9)	5613(8)	5108(3)	6876(4)	83(3)
C(10)	5291(7)	3453(5)	9076(3)	91(4)
C(11)	13367(7)	1122(6)	7150(4)	56(2)
C(12)	14387(7)	-108(6)	7135(4)	65(3)
C(13)	14348(7)	-775(4)	6450(5)	73(3)

C(14)	13290(8)	-211(5)	5779(4)	69(3)
C(15)	12270(7)	1019(5)	5794(4)	55(2)
C(16)	12308(6)	1686(4)	6480(4)	48(2)
C(17)	11092(11)	3005(8)	6557(6)	50(2)
C(18)	9275(14)	6172(8)	6928(7)	70(3)
C(19)	7395(14)	5571(10)	8792(8)	74(3)
C(20)	10790(13)	4565(10)	8597(8)	70(3)
B	11640(8)	-2116(5)	8766(5)	62(3)
F(8)	13116(11)	-2730(8)	8559(11)	273(9)
F(9)	11070(19)	-2550(9)	9652(7)	280(10)
F(10)	10805(20)	-2264(9)	8210(12)	426(19)
F(11)	11568(10)	-920(6)	8642(6)	138(4)
O	10338(8)	1763(6)	8697(5)	67(2)

Table 3S. Bond lengths [Å] and angles [°] for 3.

Rh-C(17)	2.113(9)	Rh-O	2.164(7)
Rh-C(4)	2.273(4)	Rh-C(3)	2.275(4)
Rh-C(5)	2.283(4)	Rh-C(2)	2.286(4)
Rh-C(1)	2.291(4)	Rh-P	2.338(2)
P-C(19)	1.796(10)	P-C(18)	1.815(9)
P-C(20)	1.834(10)	F(1)-C(11)	1.302(7)
F(2)-C(12)	1.333(8)	F(3)-C(13)	1.342(7)
F(4)-C(14)	1.314(7)	F(5)-C(15)	1.304(7)
F(6)-C(17)	1.412(10)	F(7)-C(17)	1.388(10)
C(1)-C(2)	1.42	C(1)-C(5)	1.42
C(1)-C(6)	1.51	C(2)-C(3)	1.42
C(2)-C(7)	1.51	C(3)-C(4)	1.42
C(3)-C(8)	1.51	C(4)-C(5)	1.42
C(4)-C(9)	1.51	C(5)-C(10)	1.51
C(11)-C(12)	1.39	C(11)-C(16)	1.39
C(12)-C(13)	1.39	C(13)-C(14)	1.39
C(14)-C(15)	1.39	C(15)-C(16)	1.39
C(16)-C(17)	1.542(9)	B-F(11)	1.268(4)
B-F(8)	1.268(4)	B-F(9)	1.268(4)
B-F(10)	1.268(4)		

C(17)-Rh-O	89.5(3)	C(17)-Rh-C(4)	124.1(3)
O-Rh-C(4)	145.3(2)	C(17)-Rh-C(3)	98.2(3)
O-Rh-C(3)	142.5(2)	C(4)-Rh-C(3)	36.38(6)
C(17)-Rh-C(5)	158.6(3)	O-Rh-C(5)	109.0(2)
C(4)-Rh-C(5)	36.32(6)	C(3)-Rh-C(5)	60.54(8)
C(17)-Rh-C(2)	104.8(2)	O-Rh-C(2)	106.3(2)
C(4)-Rh-C(2)	60.52(8)	C(3)-Rh-C(2)	36.28(6)
C(5)-Rh-C(2)	60.38(8)	C(17)-Rh-C(1)	137.8(2)
O-Rh-C(1)	89.6(2)	C(4)-Rh-C(1)	60.45(8)
C(3)-Rh-C(1)	60.43(8)	C(5)-Rh-C(1)	36.18(6)
C(2)-Rh-C(1)	36.15(6)	C(17)-Rh-P	88.1(2)
O-Rh-P	90.4(2)	C(4)-Rh-P	98.25(11)
C(3)-Rh-P	126.30(11)	C(5)-Rh-P	102.10(11)
C(2)-Rh-P	158.72(11)	C(1)-Rh-P	134.07(11)
C(19)-P-C(18)	101.9(5)	C(19)-P-C(20)	101.1(5)
C(18)-P-C(20)	106.1(5)	C(19)-P-Rh	117.1(4)
C(18)-P-Rh	115.5(4)	C(20)-P-Rh	113.3(3)
C(2)-C(1)-C(5)	108.0	C(2)-C(1)-C(6)	126.0
C(5)-C(1)-C(6)	126.0	C(2)-C(1)-Rh	71.74(13)
C(5)-C(1)-Rh	71.62(13)	C(6)-C(1)-Rh	122.33(12)
C(1)-C(2)-C(3)	108.0	C(1)-C(2)-C(7)	126.0
C(3)-C(2)-C(7)	126.0	C(1)-C(2)-Rh	72.11(13)
C(3)-C(2)-Rh	71.45(12)	C(7)-C(2)-Rh	122.13(12)
C(4)-C(3)-C(2)	108.0	C(4)-C(3)-C(8)	126.0
C(2)-C(3)-C(8)	126.0	C(4)-C(3)-Rh	71.74(13)
C(2)-C(3)-Rh	72.27(13)	C(8)-C(3)-Rh	121.71(12)
C(3)-C(4)-C(5)	108.0	C(3)-C(4)-C(9)	126.0
C(5)-C(4)-C(9)	126.0	C(3)-C(4)-Rh	71.87(13)
C(5)-C(4)-Rh	72.21(13)	C(9)-C(4)-Rh	121.64(12)
C(4)-C(5)-C(1)	108.0	C(4)-C(5)-C(10)	126.0
C(1)-C(5)-C(10)	126.0	C(4)-C(5)-Rh	71.48(13)
C(1)-C(5)-Rh	72.20(13)	C(10)-C(5)-Rh	122.02(12)
F(1)-C(11)-C(12)	116.6(5)	F(1)-C(11)-C(16)	123.3(5)

C(12)-C(11)-C(16)	120.0	F(2)-C(12)-C(13)	118.8(6)
F(2)-C(12)-C(11)	121.2(6)	C(13)-C(12)-C(11)	120.0
F(3)-C(13)-C(12)	120.2(6)	F(3)-C(13)-C(14)	119.8(6)
C(12)-C(13)-C(14)	120.0	F(4)-C(14)-C(15)	121.3(6)
F(4)-C(14)-C(13)	118.7(6)	C(15)-C(14)-C(13)	120.0
F(5)-C(15)-C(16)	124.1(5)	F(5)-C(15)-C(14)	115.8(5)
C(16)-C(15)-C(14)	120.0	C(15)-C(16)-C(11)	120.0
C(15)-C(16)-C(17)	120.5(5)	C(11)-C(16)-C(17)	119.3(5)
F(7)-C(17)-F(6)	101.8(6)	F(7)-C(17)-C(16)	108.6(6)
F(6)-C(17)-C(16)	104.4(6)	F(7)-C(17)-Rh	110.6(6)
F(6)-C(17)-Rh	114.3(5)	C(16)-C(17)-Rh	116.0(5)
F(11)-B-F(8)	109.52(10)	F(11)-B-F(9)	109.47(10)
F(8)-B-F(9)	109.45(11)	F(11)-B-F(10)	109.47(10)
F(8)-B-F(10)	109.46(10)	F(9)-B-F(10)	109.45(10)

Symmetry transformations used to generate equivalent atoms:

Table 4S. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 3.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + \ldots + 2hka^*b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Rh	39(1)	33(1)	41(1)	-8(1)	-6(1)	-4(1)
P	55(1)	39(1)	44(1)	-12(1)	-7(1)	-7(1)
F(1)	62(4)	120(5)	88(4)	-43(4)	-22(3)	-15(4)
F(2)	61(4)	113(6)	115(5)	3(4)	-35(4)	4(4)
F(3)	90(5)	52(4)	171(8)	-18(4)	0(5)	11(3)
F(4)	110(6)	99(5)	134(6)	-88(5)	6(5)	-21(4)
F(5)	85(4)	82(4)	60(3)	-26(3)	-20(3)	-10(3)
F(6)	65(4)	63(3)	100(4)	-37(3)	10(3)	-33(3)
F(7)	84(4)	47(3)	54(3)	3(2)	1(3)	-5(3)
C(1)	39(5)	56(5)	50(5)	0(4)	-10(4)	-17(4)
C(2)	42(5)	50(5)	53(5)	-13(4)	-11(4)	-9(4)
C(3)	56(6)	47(5)	48(5)	-8(4)	-20(4)	-7(4)
C(4)	45(5)	57(5)	65(6)	-15(4)	-19(4)	-2(4)
C(5)	38(5)	63(6)	61(6)	-17(5)	-12(4)	-6(4)
C(6)	74(7)	65(6)	64(6)	4(5)	-9(5)	-27(5)
C(7)	78(7)	45(5)	75(6)	-15(5)	-10(5)	-15(5)
C(8)	103(9)	65(6)	50(5)	-5(5)	-27(6)	-8(6)
C(9)	79(8)	70(7)	96(8)	-18(6)	-46(7)	16(6)
C(10)	63(7)	115(10)	82(8)	-31(7)	7(6)	-6(7)
C(11)	50(6)	70(6)	50(5)	-10(5)	-7(4)	-25(5)
C(12)	40(6)	64(6)	77(7)	0(5)	0(5)	-11(5)
C(13)	56(6)	41(5)	100(8)	-14(5)	18(6)	-7(5)
C(14)	61(7)	66(6)	82(7)	-31(6)	1(6)	-15(5)
C(15)	53(6)	47(5)	56(5)	-12(4)	4(4)	-12(4)
C(16)	45(5)	45(5)	50(5)	-16(4)	13(4)	-20(4)
C(17)	52(5)	42(5)	55(5)	-14(4)	-2(4)	-12(4)
C(18)	89(8)	39(5)	73(6)	-4(4)	-8(6)	-13(5)
C(19)	79(8)	66(6)	71(6)	-29(5)	-3(6)	-6(6)
C(20)	86(8)	57(6)	74(6)	-21(5)	-26(6)	-14(5)
B	69(8)	44(6)	63(7)	2(5)	-9(6)	-15(5)
F(8)	175(14)	154(12)	425(27)	-102(14)	10(15)	42(10)
F(9)	421(25)	134(9)	177(12)	18(8)	111(14)	-87(12)
F(10)	751(42)	84(7)	626(35)	-23(12)	-618(36)	-20(14)
F(11)	131(7)	70(5)	193(9)	-25(5)	23(6)	-40(5)
O	59(4)	59(4)	81(4)	-12(3)	-7(3)	-20(3)

Table 5S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 3.

	x	y	z	U(eq)
H(6A)	6697(8)	831(5)	9612(3)	104
H(6B)	8415(8)	182(5)	9073(3)	104
H(6C)	6836(9)	-136(4)	8954(4)	104
H(7A)	9140(8)	591(5)	6097(4)	99
H(7B)	8213(9)	-272(3)	6972(4)	99
H(7C)	9792(7)	47(4)	7091(4)	99
H(8A)	8461(9)	2457(6)	5220(2)	112
H(8B)	8405(8)	3859(5)	5300(3)	112
H(8C)	6826(9)	3541(6)	5181(2)	112
H(9A)	5015(9)	5455(4)	7440(5)	125
H(9B)	4884(8)	5231(4)	6433(4)	125
H(9C)	6463(9)	5549(3)	6552(4)	125
H(10A)	5386(8)	2759(6)	9644(3)	137
H(10B)	4159(7)	3803(6)	9020(4)	137
H(10C)	5738(9)	4121(5)	9139(4)	137
H(18A)	10159(14)	5883(8)	6428(7)	105
H(18B)	9493(14)	6852(8)	7138(7)	105
H(18C)	8275(14)	6492(8)	6670(7)	105
H(19A)	7177(14)	4939(10)	9380(8)	111
H(19B)	6443(14)	5907(10)	8487(8)	111
H(19C)	7662(14)	6267(10)	8954(8)	111
H(20A)	11790(13)	4162(10)	8220(8)	104
H(20B)	10603(13)	4013(10)	9233(8)	104
H(20C)	10871(13)	5384(10)	8669(8)	104

CRYSTALLOGRAPHIC TABLES FOR

COMPLEX 4

ORTEP of **4**

Table 6S. Crystal data and structure refinement for **4**.

Table 7S. Atomic coordinates for **4**.

Table 8S. Bond lengths and angles for **4**.

Table 9S. Anisotropic displacement coefficients for **4**.

Table 10S. Hydrogen atom coordinates for **4**.

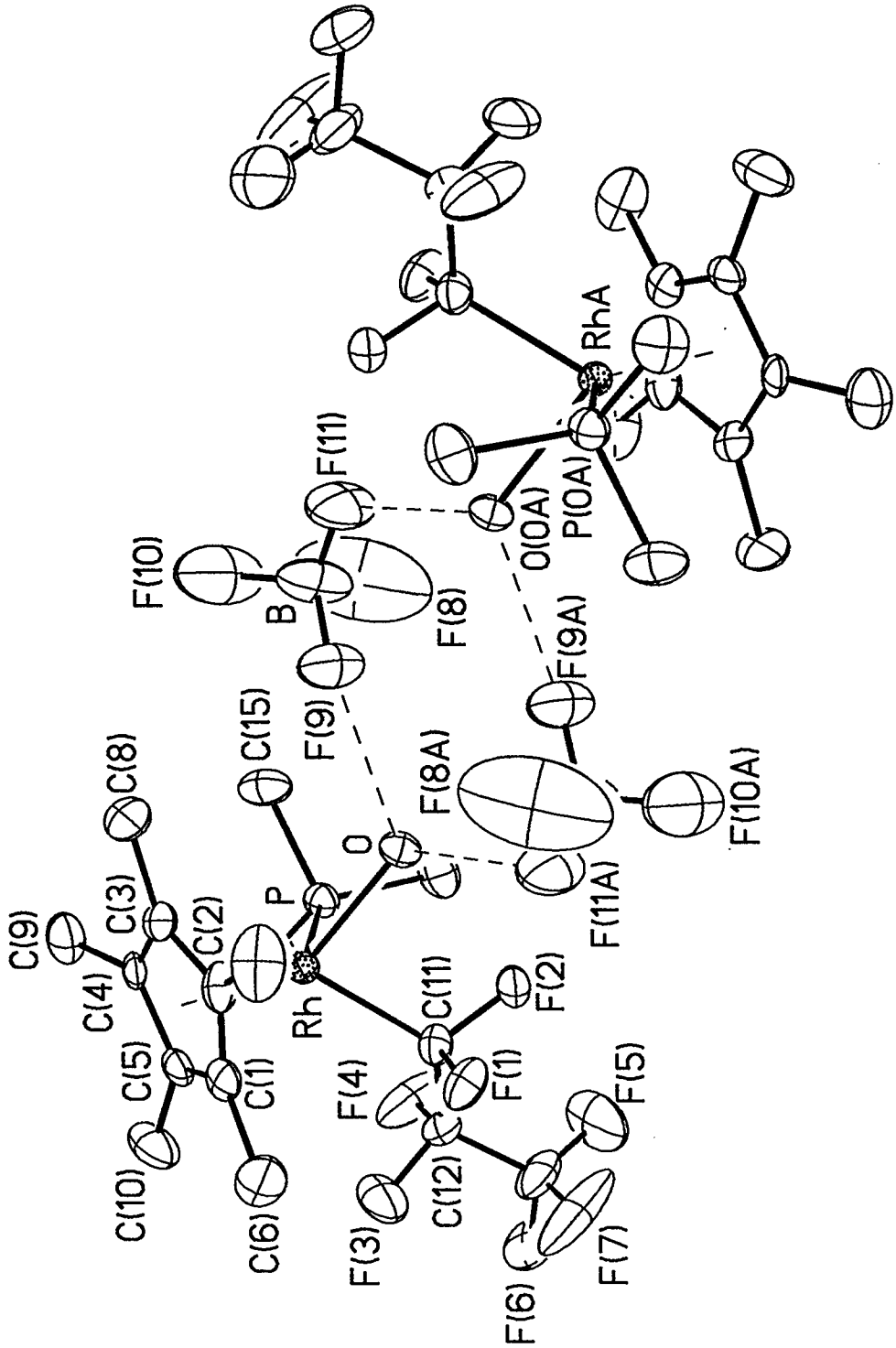
RhA
P(0A)
O(0A)
F(11)
F(10)
B
F(8)
F(9)
F(9A)
C(15)
C(8)
F(8A)
F(10A)
O
P
C(3)
C(2)
F(11A)
C(9)
C(4)
Rh
C(11)
F(2)
C(5)
C(1)
F(4)
F(1)
F(5)
C(10)
C(12)
F(3)
F(7)
C(6)
F(6)

Table 6S. Crystal data and structure refinement for 4.

Identification code	rph47
Empirical formula	$C_{16}H_{26}BF_{11}OPRh$
Formula weight	588.06
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 10.115(1) Å alpha = 90° b = 16.270(2) Å beta = 92.74(1)° c = 14.070(2) Å gamma = 90°
Volume, Z	2312.7(4) $Å^3$, 4
Density (calculated)	1.689 g/cm^3
Absorption coefficient	0.900 mm^{-1}
F(000)	1176
Crystal size	0.40 x 0.30 x 0.20 mm
Crystal color	Orange block
θ range for data collection	2.37 to 22.50°
Limiting indices	$-1 \le h \le 10$, $-1 \le k \le 17$, $-15 \le l \le 15$
Reflections collected	3928
Independent reflections	3019 (R_{int} = 0.0416)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3017 / 0 / 280
Goodness-of-fit on F^2	1.035
Final R indices [I>2σ(I)]	R1 = 0.0481, wR2 = 0.0841
R indices (all data)	R1 = 0.0825, wR2 = 0.0919
Largest diff. peak and hole	0.452 and −0.422 $eÅ^{-3}$

Table 7S. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 4. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Rh	2251.9(6)	8373.9(4)	2376.7(4)	34(1)
P	4260(2)	9008(1)	2129(1)	43(1)
O	1355(5)	9379(3)	1496(3)	53(2)
F(1)	546(4)	9049(3)	3733(3)	66(2)
F(2)	1969(5)	9958(3)	3313(3)	65(2)
F(3)	2379(7)	8295(4)	4848(3)	118(2)
F(4)	3922(5)	9057(5)	4360(3)	120(3)
F(5)	2694(13)	10356(4)	5075(5)	214(6)
F(6)	3077(6)	9450(4)	6056(3)	102(2)
F(7)	1190(8)	9641(7)	5489(5)	191(5)
F(8)	2562(12)	10105(10)	-979(9)	326(10)
C(1)	1146(9)	7288(5)	2902(6)	47(2)
C(2)	676(8)	7485(5)	1987(6)	52(2)
C(3)	1727(9)	7354(5)	1348(5)	45(2)
C(4)	2857(8)	7125(4)	1880(6)	40(2)
C(5)	2553(9)	7127(4)	2876(5)	48(2)
C(6)	332(9)	7178(5)	3775(6)	101(4)
C(7)	-711(8)	7676(6)	1682(6)	97(4)
C(8)	1583(9)	7406(5)	285(5)	79(3)
C(9)	4141(8)	6818(5)	1511(6)	73(3)
C(10)	3438(9)	6784(5)	3671(5)	85(3)
C(11)	1878(8)	9129(5)	3532(5)	47(2)
C(12)	2664(10)	9033(6)	4488(5)	56(3)
C(13)	2381(14)	9637(8)	5265(7)	83(4)
C(14)	4420(8)	10104(5)	2356(6)	70(3)
C(15)	4560(8)	8951(5)	860(5)	70(3)
C(16)	5741(7)	8605(5)	2696(5)	67(3)
B	1716(14)	9583(13)	-1205(12)	102(6)
F(9)	1094(6)	9355(4)	-439(4)	108(2)
F(10)	2351(12)	8950(7)	-1535(6)	261(8)
F(11)	879(7)	9881(4)	-1871(4)	134(3)

Table 8S. Bond lengths [Å] and angles [°] for 4.

Rh-C(11)	2.086(7)	Rh-C(5)	2.164(7)
Rh-C(2)	2.202(8)	Rh-O	2.219(5)
Rh-C(1)	2.236(8)	Rh-C(3)	2.249(7)
Rh-C(4)	2.243(7)	Rh-P	2.319(2)
P-C(16)	1.788(7)	P-C(14)	1.819(8)
P-C(15)	1.827(7)	F(1)-C(11)	1.396(8)
F(2)-C(11)	1.389(8)	F(3)-C(12)	1.339(9)
F(4)-C(12)	1.295(10)	F(5)-C(13)	1.244(14)
F(6)-C(13)	1.324(11)	F(7)-C(13)	1.261(12)
F(8)-B	1.24(2)	C(1)-C(2)	1.388(10)
C(1)-C(5)	1.449(10)	C(1)-C(6)	1.522(9)
C(2)-C(3)	1.441(9)	C(2)-C(7)	1.480(10)
C(3)-C(4)	1.387(10)	C(3)-C(8)	1.498(9)
C(4)-C(5)	1.449(10)	C(4)-C(9)	1.506(9)
C(5)-C(10)	1.506(10)	C(11)-C(12)	1.538(10)
C(12)-C(13)	1.507(12)	B-F(10)	1.31(2)
B-F(11)	1.32(2)	B-F(9)	1.327(12)
C(11)-Rh-C(5)	109.1(3)	C(11)-Rh-C(2)	115.1(3)
C(5)-Rh-C(2)	63.5(3)	C(11)-Rh-O	85.2(3)
C(5)-Rh-O	156.9(3)	C(2)-Rh-O	94.2(3)
C(11)-Rh-C(1)	95.5(3)	C(5)-Rh-C(1)	38.4(3)
C(2)-Rh-C(1)	36.4(3)	O-Rh-C(1)	124.7(3)
C(11)-Rh-C(3)	152.8(3)	C(5)-Rh-C(3)	62.8(3)
C(2)-Rh-C(3)	37.8(2)	O-Rh-C(3)	96.1(2)
C(1)-Rh-C(3)	61.5(3)	C(11)-Rh-C(4)	147.0(3)
C(5)-Rh-C(4)	38.3(3)	C(2)-Rh-C(4)	62.1(3)
O-Rh-C(4)	127.2(2)	C(1)-Rh-C(4)	62.3(3)
C(3)-Rh-C(4)	36.0(3)	C(11)-Rh-P	92.8(2)
C(5)-Rh-P	110.8(3)	C(2)-Rh-P	152.0(2)
O-Rh-P	85.8(2)	C(1)-Rh-P	148.9(2)
C(3)-Rh-P	114.4(2)	C(4)-Rh-P	95.8(2)
C(16)-P-C(14)	102.4(4)	C(16)-P-C(15)	104.0(4)
C(14)-P-C(15)	101.7(4)	C(16)-P-Rh	119.4(3)
C(14)-P-Rh	118.7(3)	C(15)-P-Rh	108.2(3)
C(2)-C(1)-C(5)	108.2(8)	C(2)-C(1)-C(6)	126.9(9)
C(5)-C(1)-C(6)	124.7(8)	C(2)-C(1)-Rh	70.5(5)
C(5)-C(1)-Rh	68.1(4)	C(6)-C(1)-Rh	131.2(5)
C(1)-C(2)-C(3)	108.3(8)	C(1)-C(2)-C(7)	126.5(9)
C(3)-C(2)-C(7)	124.6(8)	C(1)-C(2)-Rh	73.1(5)
C(3)-C(2)-Rh	72.9(4)	C(7)-C(2)-Rh	126.8(6)
C(4)-C(3)-C(2)	108.5(7)	C(4)-C(3)-C(8)	126.3(8)
C(2)-C(3)-C(8)	125.1(9)	C(4)-C(3)-Rh	71.8(4)
C(2)-C(3)-Rh	69.4(4)	C(8)-C(3)-Rh	127.6(5)
C(3)-C(4)-C(5)	108.3(7)	C(3)-C(4)-C(9)	127.2(7)
C(5)-C(4)-C(9)	124.1(8)	C(3)-C(4)-Rh	72.2(4)
C(5)-C(4)-Rh	67.8(4)	C(9)-C(4)-Rh	131.3(5)
C(4)-C(5)-C(1)	106.2(8)	C(4)-C(5)-C(10)	124.8(8)
C(1)-C(5)-C(10)	126.7(8)	C(4)-C(5)-Rh	73.8(4)
C(1)-C(5)-Rh	73.5(5)	C(10)-C(5)-Rh	131.2(6)
F(2)-C(11)-F(1)	102.1(6)	F(2)-C(11)-C(12)	104.8(7)
F(1)-C(11)-C(12)	106.1(7)	F(2)-C(11)-Rh	112.5(5)
F(1)-C(11)-Rh	108.4(5)	C(12)-C(11)-Rh	121.1(6)
F(4)-C(12)-F(3)	108.1(9)	F(4)-C(12)-C(13)	107.6(9)

F(3)-C(12)-C(13)	105.0(8)	F(4)-C(12)-C(11)	110.2(7)
F(3)-C(12)-C(11)	108.1(8)	C(13)-C(12)-C(11)	117.4(8)
F(5)-C(13)-F(7)	107.7(12)	F(5)-C(13)-F(6)	105.4(12)
F(7)-C(13)-F(6)	105.5(10)	F(5)-C(13)-C(12)	113.5(11)
F(7)-C(13)-C(12)	113.7(11)	F(6)-C(13)-C(12)	110.4(9)
F(8)-B-F(10)	106.6(13)	F(8)-B-F(11)	110(2)
F(10)-B-F(11)	110(2)	F(8)-B-F(9)	109(2)
F(10)-B-F(9)	109(2)	F(11)-B-F(9)	111.5(11)

Symmetry transformations used to generate equivalent atoms:

Table 9S. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 4.

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Rh	38(1)	33(1)	31(1)	-2(1)	-3(1)	1(1)
P	46(2)	38(1)	46(1)	0(1)	0(1)	-5(1)
O	60(4)	55(4)	42(3)	8(3)	-13(3)	14(3)
F(1)	47(3)	87(4)	65(3)	-26(3)	2(3)	10(3)
F(2)	98(4)	42(3)	54(3)	-7(2)	-2(3)	17(3)
F(3)	231(8)	63(4)	55(3)	2(3)	-50(4)	11(5)
F(4)	52(4)	253(8)	55(3)	-31(4)	-11(3)	42(5)
F(5)	485(20)	55(5)	97(5)	-24(4)	-47(8)	-37(7)
F(6)	134(6)	117(5)	51(3)	-27(3)	-25(4)	11(5)
F(7)	115(6)	353(15)	106(5)	-133(7)	1(5)	53(8)
F(8)	181(10)	442(22)	347(16)	162(15)	-80(11)	-212(14)
C(1)	53(6)	28(5)	60(6)	-7(4)	7(5)	-16(5)
C(2)	43(6)	40(6)	71(6)	-19(5)	-9(5)	-3(5)
C(3)	59(6)	32(5)	43(5)	-12(4)	-3(5)	-16(5)
C(4)	53(6)	17(4)	51(5)	-7(4)	16(5)	0(4)
C(5)	78(7)	22(5)	44(5)	-2(4)	-10(6)	-2(5)
C(6)	134(10)	65(7)	112(8)	-10(6)	75(8)	-43(7)
C(7)	46(6)	94(8)	146(10)	-45(8)	-33(7)	-1(6)
C(8)	128(9)	65(7)	41(5)	-16(5)	-28(6)	-11(7)
C(9)	73(7)	48(6)	98(7)	-11(5)	14(6)	-4(5)
C(10)	119(9)	59(7)	74(6)	15(6)	-36(6)	-4(7)
C(11)	45(6)	43(5)	52(5)	-7(5)	1(5)	9(5)
C(12)	76(7)	58(6)	34(5)	-3(5)	2(5)	10(6)
C(13)	96(10)	105(10)	46(7)	-30(7)	-4(7)	23(9)
C(14)	59(6)	52(6)	99(7)	-1(6)	1(6)	-8(5)
C(15)	74(7)	85(7)	52(5)	11(5)	5(5)	-25(6)
C(16)	46(5)	76(7)	80(6)	-2(5)	10(5)	-3(5)
B	48(9)	163(17)	96(12)	59(12)	27(9)	5(11)
F(9)	124(5)	129(5)	72(3)	20(4)	17(4)	-3(4)
F(10)	290(13)	340(15)	162(8)	100(9)	107(8)	257(13)
F(11)	133(6)	186(7)	82(4)	35(5)	0(4)	88(6)

Table 10S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 4.

	x	y	z	U(eq)
H(6A)	908(9)	7045(5)	4315(6)	152
H(6B)	-295(9)	6742(5)	3663(6)	152
H(6C)	-133(9)	7679(5)	3897(6)	152
H(7A)	-765(8)	7787(6)	1011(6)	145
H(7B)	-1003(8)	8149(6)	2022(6)	145
H(7C)	-1265(8)	7215(6)	1816(6)	145
H(8A)	701(9)	7580(5)	99(5)	118
H(8B)	1746(9)	6875(5)	16(5)	118
H(8C)	2208(9)	7795(5)	59(5)	118
H(9A)	4754(8)	6705(5)	2036(6)	109
H(9B)	4506(8)	7230(5)	1110(6)	109
H(9C)	3980(8)	6324(5)	1150(6)	109
H(10A)	3026(9)	6857(5)	4265(5)	128
H(10B)	4272(9)	7067(5)	3690(5)	128
H(10C)	3581(9)	6208(5)	3563(5)	128
H(14A)	3662(8)	10385(5)	2078(6)	105
H(14B)	5207(8)	10306(5)	2080(6)	105
H(14C)	4476(8)	10200(5)	3030(6)	105
H(15A)	3801(8)	9157(5)	499(5)	105
H(15B)	4717(8)	8390(5)	686(5)	105
H(15C)	5321(8)	9278(5)	728(5)	105
H(16A)	5760(7)	8019(5)	2618(5)	101
H(16B)	5763(7)	8737(5)	3361(5)	101
H(16C)	6496(7)	8843(5)	2412(5)	101