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Synthesis of $[\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}}\text{Ir}(\text{PEt}_3)_3]^+\text{BF}_4^-$ (**2**).

Compound **3**, *mer*- $\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}}\text{Ir}(\text{PEt}_3)_3(\text{H})$ (300 mg, 0.46 mmol), was dissolved in 75 mL of tetrahydrofuran (THF) to form a yellow solution and cooled to -47°C . AgBF_4 (91 mg, 0.46 mmol) was dissolved in 25 mL of THF and added to the stirred solution over 2 hours. As the addition proceeded, the solution darkened to black-orange. When the addition was complete the solution was warmed to room temperature during which time it became opaque purple. After addition of 100 mL of pentane, the solution was filtered through a glass frit. The solvent was removed *in vacuo* to reveal a bright purple film containing a 1:1 product mixture of Compounds **2** and **4**. Yield: 320 mg (95%).

The bright purple film containing a 1:1 mixture of Compounds **2** and **4** (320 mg, 0.44 mmol) was dissolved in 30 mL of tetrahydrofuran and cooled to -30°C . Lithium diisopropylamide (31 mg, 0.30 mmol) in 10 mL of tetrahydrofuran was added dropwise to the stirred solution. The previously opaque solution became slightly more translucent while warming to room temperature. After addition of 70 mL of pentane, the solution was passed through a glass frit and the solvents were removed under vacuum.

The resultant film was dissolved in a minimal quantity of methylene chloride. Pentane was added until a purple precipitate of Compound **2** formed. The solution was passed through a glass frit to give a yellow filtrate of **3**, which could be recovered and reused. Methylene chloride was added to redissolve the purple precipitate of **2** and the solution was filtered. The methylene chloride was then removed under vacuum. The addition and vacuum removal of methylene chloride was repeated twice to facilitate residual tetrahydrofuran removal. Yield of **2**: 150 mg (46%).

High Resolution FAB-MS Calcd for $\text{C}_{24}\text{H}_{53}^{191}\text{IrOP}_3^+$: 641.2918. Found: 641.2926.

^1H NMR (methylene chloride- d_2 , 23°C): δ 9.35 (qd, $J_{\text{H-P}} = 6.0$ Hz, $J_{\text{H-H3}} = 1.6$ Hz, 1, H1), 6.45 (d, $J_{\text{H-H1}} = 1.6$ Hz, 1, H3), 2.38 (s, 3, ring CH_3), 2.33 (s, 3, ring CH_3), 1.95 (dq, $J_{\text{H-P}} = 7.5$ Hz, $J_{\text{H-H}} = 7.5$ Hz, 18, PEt_3 CH_2 's), 1.00 (dt, $J_{\text{H-P}} = 15.5$ Hz, $J_{\text{H-H}} = 7.5$ Hz, 27, PEt_3 CH_3 's).

$^{13}\text{C}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , 23°C): δ 170.7 (s, C4), 162.2 (q, $J_{\text{C-P}} = 22.7$ Hz, C1), 147.3 (s, C2), 112.2 (s, C3), 28.5 (d, $J_{\text{C-P}} = 2.8$ Hz, ring CH_3), 26.3 (br s, ring CH_3), 19.8 (d, $J_{\text{C-P}} = 32.8$ Hz, PEt_3 CH_2 's), 9.0 (br s, PEt_3 CH_3 's).

$^{31}\text{P}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , 23°C): δ 2.3 (s, PEt_3 's).

Independent Synthesis of $[\text{mer-}\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}_2-\text{C}(\text{Me})=\text{O}}-\text{Ir}(\text{PEt}_3)_3(\text{H})]^+\text{BF}_4^-$ (**4**).

Compound **3**, $\text{mer-}\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}}-\text{Ir}(\text{PEt}_3)_3(\text{H})$ (50 mg, 0.077 mmol), was dissolved in 10 mL of diethyl ether to form a yellow solution and cooled to -30°C . To this solution was added $\text{HBF}_4 \cdot \text{OEt}_2$ (12 mg, 0.077 mmol), dissolved in 5 mL of cold (-30°C) diethyl ether. As the addition proceeded, a white, microfine precipitate formed and remained suspended in the ether. Upon standing at -30°C , the precipitate dropped to the bottom of the flask, leaving a clear solution which was decanted. The residual ether was removed *in vacuo* and the product was washed with pentane and dried under vacuum. Yield of **4**: 54 mg (96%).

Anal. Calcd for $\text{C}_{24}\text{H}_{55}\text{BF}_4\text{IrOP}_3$: C, 39.39; H, 7.59. Found: C, 39.19; H, 7.49.

High Resolution FAB-MS Calcd for $\text{C}_{24}\text{H}_{55}^{193}\text{IrOP}_3^+$: 645.3096. Found: 645.3097.

^1H NMR (methylene chloride- d_2 , 22°C): δ 6.67 (d, $J_{\text{H-P}} = 3.0$ Hz, 1, H1), 3.86 (t, $J_{\text{H-P}} = 6.0$ Hz, 2, H3's) 2.22 (s, 3, ring CH_3), 1.86 (dq, $J_{\text{H-P}} = 7.5$ Hz, $J_{\text{H-H}} = 7.5$ Hz, 6, PEt_3 CH_2 's), 1.79-1.72 (m, 6, PEt_3 CH_2 's), 1.71 (s, 3, ring CH_3), 1.66-1.58 (m, 6, PEt_3 CH_2 's), 1.07 (dt,

$J_{\text{H-P}} = 14.5$ Hz, $J_{\text{H-H}} = 7.5$ Hz, 9, PEt_3 CH_3 's), 0.95 (m, 18, PEt_3 CH_3 's), -27.51 (td, $J_{\text{H-P}} = 16.5$ Hz, 12.0 Hz, 1, Ir-H).

$^{13}\text{C}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , 22°C): $\delta 215.7$ (s, C4), 125.7 (dt, $J_{\text{C-P}} = 75.0$ Hz, 14.0 Hz, C1), 120.8 (t, $J_{\text{C-P}} = 4.5$ Hz, C2), 50.3 (s, C3), 31.0 (s, ring CH_3), 28.7 (d, $J_{\text{C-P}} = 9.8$ Hz, ring CH_3), 20.1 (d, $J_{\text{C-P}} = 25.6$ Hz, PEt_3 CH_2 's), 16.8 (virtual t, $J_{\text{C-P}} = 34.3$ Hz, PEt_3 CH_2 's), 8.4 (d, $J_{\text{C-P}} = 2.2$ Hz, PEt_3 CH_3 's), 7.9 (s, PEt_3 CH_3 's).

$^{31}\text{P}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , 22°C): $\delta -6.0$ (d, $J_{\text{P-P}} = 17.0$ Hz, 2, PEt_3 's), -17.8 (t, $J_{\text{P-P}} = 17.0$ Hz, 1, PEt_3).

Formation of $[\text{CH}=\text{C}(\text{Me})-\text{CH}-\text{C}(\text{Me})=\text{O}-\text{Ir}-\text{O}-\text{C}(\text{Me})_2(\text{PEt}_3)_3]^+\text{BF}_4^-$ (5).

Compound 2, $[\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}=\text{Ir}(\text{PEt}_3)_3]^+\text{BF}_4^-$ (50 mg, 0.068 mmol), was dissolved in 0.5 mL of acetone- d_6 to form a purple solution, which turned yellow when cooled below -30°C . Upon warming, the solution reverted to its original purple color. Repeated cooling and warming cycles resulted in no significant product decomposition by NMR.

^1H NMR (acetone- d_6 , -40°C): $\delta 7.15$ (ddd, $J_{\text{H-P}} = 6.6$ Hz, 6.6 Hz, 5.4 Hz, 1, H1), 3.95 (m, 1, H3), 2.61 (s, 3, ring CH_3), 1.86 (s, 3, ring CH_3), 2.04, 1.88, 1.85, 1.80, 1.79, 1.73 (m's, 18, PEt_3 CH_2 's), 0.93, 0.88, 0.70 (br m's, 27, PEt_3 CH_3 's).

$^{13}\text{C}\{^1\text{H}\}$ NMR (acetone- d_6 , -40°C): $\delta 224.5$ (d, $J_{\text{C-P}} = 2.2$ Hz, C4), 137.6 (ddd, $J_{\text{C-P}} = 85.0$ Hz, 7.7 Hz, 6.4 Hz, C1), 127.6 (s, C2), 74.9 (s, C3), 64.9 (dd, $J_{\text{C-P}} = 3.5, 2.3$ Hz, central acetone C), 32.9 (d, $J_{\text{C-P}} = 4.6$ Hz, ring CH_3), 27.2 (d, $J_{\text{C-P}} = 9.3$ Hz, ring CH_3), 17.0 (d, $J_{\text{C-P}} = 38.1$ Hz, PEt_3 CH_2 's), 16.9 (d, $J_{\text{C-P}} = 33.6$ Hz, PEt_3 CH_2 's), 14.7 (d, $J_{\text{C-P}} = 22.3$ Hz,

PEt₃ CH₂'s), 8.9 (d, J_{C-P} = 7.0 Hz, PEt₃ CH₃'s), 8.6 (d, J_{C-P} = 5.5 Hz, PEt₃ CH₃'s), 7.9 (d, J_{C-P} = 5.5 Hz, PEt₃ CH₃'s).

³¹P{¹H} NMR (acetone-d₆, -40°C): δ-20.6 (dd, J_{P-P} = 10.5 Hz, 10.5 Hz, 1, PEt₃), -22.2 (dd, J_{P-P} = 19.0 Hz, 10.5 Hz, 1, PEt₃), -26.1 (dd, J_{P-P} = 19.0 Hz, 10.5 Hz, 1, PEt₃).

Synthesis of *mer*- $\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}-\text{Ir}(\text{PEt}_3)_3(\text{Cl})}$ (6).

Compound 2, $\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}=\text{Ir}(\text{PEt}_3)_3}^+\text{BF}_4^-$ (120 mg, 0.17 mmol), was dissolved in 20 mL of tetrahydrofuran to form a purple solution and cooled to -30°C. This solution was added dropwise to a -30°C solution of [PNP]⁺[Cl]⁻ (120 mg, 0.21 mmol) in 20 mL of tetrahydrofuran and 5 mL of methylene chloride. The purple solution instantly turned lemon yellow on contact with the [PNP]⁺[Cl]⁻ solution. The solvent was removed *in vacuo*. The resulting yellow brown solid was extracted with pentane and passed through a glass frit to give a translucent lemon yellow solution which produced yellow crystals after concentration and cooling at -30°C. Yield: 32 mg (28%). Anal. Calcd for C₂₄H₅₃ClIrOP₃: C, 42.49; H, 7.89. Found: C, 42.64; H, 8.22.

High Resolution FAB-MS Calcd for C₂₄H₅₄³⁵Cl¹⁹¹IrOP₃⁺: 677.2685. Found: 677.2697.

¹H NMR (benzene-d₆, 22°C): δ6.32 (d, J_{H-P} = 10.5 Hz, 1, H1), 4.76 (d, J_{H-H} = 2.0 Hz, 1, H3), 2.12 (s, 3, ring CH₃), 2.06 (s, 3, ring CH₃), 1.86-1.78 (m, 6, PEt₃ CH₂'s), 1.76 (dq, J_{H-P} = 7.5 Hz, J_{H-H} = 7.5 Hz, 6, PEt₃ CH₂'s), 2.08-2.01 (m, 6, PEt₃ CH₂'s), 1.14 (virtual t of t, J_{H-P} = 14.0 Hz, J_{H-H} = 7.5 Hz, 18, PEt₃ CH₃'s), 0.91 (dt, J_{H-P} = 13.5 Hz, J_{H-H} = 7.5 Hz, 9, PEt₃ CH₃'s).

$^{13}\text{C}\{^1\text{H}\}$ NMR (benzene- d_6 , 22°C): δ 156.2 (s, C4), 124.6 (s, C2), 99.1 (s, C3), 94.3 (q, $J_{\text{C-P}} = 7.7$ Hz, C1), 28.3 (s, ring CH_3), 25.6 (d, $J_{\text{C-P}} = 7.6$ Hz, ring CH_3), 19.0 (d, $J_{\text{C-P}} = 32.3$ Hz, PEt_3 CH_2 's), 14.8 (virtual t, $J_{\text{C-P}} = 30.0$ Hz, PEt_3 CH_2 's), 8.8 (br s, PEt_3 CH_3 's).

$^{31}\text{P}\{^1\text{H}\}$ NMR (benzene- d_6 , 22°C): δ -18.3 (d, $J_{\text{P-P}} = 17.6$ Hz, 2, PEt_3 's), -34.4 (t, $J_{\text{P-P}} = 17.6$ Hz, 1, PEt_3).

Synthesis of $[\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}}-\text{Ir}(\text{PMe}_3)_4]^+\text{BF}_4^-$ (7).

Compound 2, $[\overline{\text{CH}=\text{C}(\text{Me})-\text{CH}=\text{C}(\text{Me})-\text{O}}=\text{Ir}(\text{PEt}_3)_3]^+\text{BF}_4^-$ (300 mg, 0.41 mmol), was dissolved in 50 mL of tetrahydrofuran to form a purple solution and cooled to -77°C . Trimethylphosphine (1.04 g, 13.7 mmol) was dissolved in 10 mL of tetrahydrofuran and added to the stirred solution over 30 minutes. The solution lost the deep purple color and turned yellow during the addition. Upon warming to 25°C the solution became translucent orange. After three hours the tetrahydrofuran was removed *in vacuo*, and the resultant brown-yellow film was taken into a minimum of methylene chloride. The product was precipitated with diethyl ether and recovered in methylene chloride. The methylene chloride solution was passed through a glass frit and removed *in vacuo* to give a yellow, slightly crystalline film. Yield: 200 mg (70%).

To obtain crystals the above product was dissolved in tetrahydrofuran and vacuum deposited onto silanized silica. Thrice this volume of silica was placed into a column, followed by the product-containing fraction. The column was wetted with diethyl ether and eluted with a 20% mixture of tetrahydrofuran in ether. When this fraction ran clear, tetrahydrofuran was used to flush the column. A translucent yellow solution resulted. After *in vacuo* concentration, diffusion of ether into the tetrahydrofuran solution at -30°C gave

orange plates. Anal. Calcd for $C_{18}H_{44}BF_4IrOP_4$: C, 31.81; H, 6.54. Found: C, 31.72, H, 6.73.

High Resolution FAB-MS Calcd for $C_{18}H_{44}^{193}IrOP_4^+$: 593.1972. Found: 593.1964.

1H NMR (methylene chloride- d_2 , 23°C): δ 5.72 (br m, 1, H1), 4.38 (s, 1, H3), 1.74 (br s, 3, ring CH_3), 1.63 (d, J_{H-P} = 9.5 Hz, 9, PMe_3 CH_3 's), 1.59 (s, 3, ring CH_3), 1.55 (d, J_{H-P} = 8.0 Hz, 9, PMe_3 CH_3 's), 1.44 (virtual t, J_{H-P} = 7.0 Hz, 18, PMe_3 CH_3 's).

$^{13}C\{^1H\}$ NMR (methylene chloride- d_2 , 23°C): δ 154.2 (s, C4), 128.0 (m, C2), 107.6 (dtd, J_{C-P} = 74.0 Hz, 11.3 Hz, 5.0 Hz, C1), 100.4 (s, C3), 28.0 (d, J_{C-P} = 11.1 Hz, ring CH_3), 23.7 (d, J_{C-P} = 7.2 Hz, ring CH_3), 19.2 (d, J_{C-P} = 38.0 Hz, PMe_3 CH_3 's), 17.4 (d, J_{C-P} = 28.2 Hz, PMe_3 CH_3 's), 15.8 (virtual t, J_{C-P} = 38.0 Hz, PMe_3 CH_3 's).

$^{31}P\{^1H\}$ NMR (methylene chloride- d_2 , 23°C): δ -37.1 (dd, J_{P-P} = 20.0 Hz, 17.0 Hz, 2, PMe_3 's), -52.4 (td, J_{P-P} = 17.0 Hz, 8.3 Hz, 1, PMe_3), -55.7 (td, J_{P-P} = 20.0 Hz, 8.3 Hz, 1, PMe_3).

Synthesis of $[C(Me)-CH=C(Me)-O-Ir-O-N(Ph)=CH(PEt_3)_3]^+BF_4^-$ (8).

Compound 2, $[CH=C(Me)-CH=C(Me)-O-Ir(PEt_3)_3]^+BF_4^-$ (110 mg, 0.15 mmol), was dissolved in 20 mL of tetrahydrofuran to form a purple solution and cooled to $-30^\circ C$. Nitrosobenzene (18 mg, 0.17 mmol) was dissolved in 10 mL of tetrahydrofuran, cooled to $-30^\circ C$, and added to the stirred purple solution, causing it to turn orange-yellow. The solution was stirred at $25^\circ C$ for thirty minutes before the tetrahydrofuran was removed *in vacuo*. The resultant film was repeatedly extracted with toluene, and each successive extraction was passed through a glass frit. The toluene volume was reduced by ~75% and stored at $-30^\circ C$.

Multiple crops of microfine yellow crystals were recovered on a glass frit with subsequent *in vacuo* solvent reductions. The recovered microfine crystals were washed with cold toluene and pentane and dissolved in a minimum of acetone. Diethyl ether diffusion at -30°C gave a bright yellow crystalline product. Yield: 85 mg (66%).

Anal. Calcd for $\text{C}_{30}\text{H}_{58}\text{BF}_4\text{IrNO}_2\text{P}_3$: C, 43.06; H, 7.00. Found: C, 43.45; H, 6.95.

High Resolution FAB-MS Calcd for $\text{C}_{30}\text{H}_{58}^{193}\text{IrNO}_2\text{P}_3^+$: 750.3310. Found: 750.3313.

^1H NMR (methylene chloride- d_2 , -40°C): δ 7.53 (d, $J_{\text{H-H}} = 7.8$ Hz, 2, phenyl ortho), 7.45 (m, 3, phenyl meta & para), 7.25 (d, $J_{\text{H-P}} = 4.2$ Hz, 1, H1), 3.56 (d, $J_{\text{H-P}} = 5.4$ Hz, 1, H3), 2.21-2.16 (br m, 6, PEt_3 CH_2 's), 1.99-1.94 (br m, 6, PEt_3 CH_2 's), 1.91-1.86 (br m, 6, PEt_3 CH_2 's), 1.70 (s, 3, ring CH_3), 1.44 (d, $J_{\text{H-P}} = 6.6$ Hz, 3, ring CH_3), 1.16-1.10 (br m, 18, PEt_3 CH_3 's), 0.99 (m, 9, PEt_3 CH_3 's).

$^{13}\text{C}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , -40°C): δ 172.3 (d, $J_{\text{C-P}} = 10.5$ Hz, C4), 156.8 (s, C1), 139.8 (s, phenyl ipso), 129.7 (s, phenyl para), 129.5 (s, phenyl meta), 120.4 (s, phenyl ortho), 102.0 (t, $J_{\text{C-P}} = 4.1$ Hz, C3), 42.7 (ddd, $J_{\text{C-P}} = 78.7$ Hz, 6.3 Hz, 4.0 Hz, C2), 27.9 (s, ring CH_3), 19.0 (s, ring CH_3), 18.6 (d, $J_{\text{C-P}} = 23.7$ Hz, PEt_3 CH_2 's), 17.8 (d, $J_{\text{C-P}} = 24.0$ Hz, PEt_3 CH_2 's), 15.3 (d, $J_{\text{C-P}} = 24.8$ Hz, PEt_3 CH_2 's), 10.1 (d, $J_{\text{C-P}} = 5.9$ Hz, PEt_3 CH_3 's), 9.5 (d, $J_{\text{C-P}} = 5.4$ Hz, PEt_3 CH_3 's), 8.2 (d, $J_{\text{C-P}} = 6.4$ Hz, PEt_3 CH_3 's).

$^{31}\text{P}\{^1\text{H}\}$ NMR (methylene chloride- d_2 , -40°C): δ -22.9 (dd, $J_{\text{P-P}} = 10.5$ Hz, 10.5 Hz, 1, PEt_3), -34.3 (dd, $J_{\text{P-P}} = 15.0$ Hz, 10.5 Hz, 1, PEt_3), -34.4 (dd, $J_{\text{P-P}} = 15.0$ Hz, 10.5 Hz, 1, PEt_3).

STRUCTURE DETERMINATION SUMMARY

Compound 8

Crystal Data

Empirical Formula	$C_{33.5} H_{62} B F_4 Ir N O_2 P_3$
Color; Habit	Yellow prism
Crystal size (mm)	.25 x .45 x .50
Crystal System	Monoclinic
Space Group	$P2_1/c$
Unit Cell Dimensions	$a = 11.987(4) \text{ \AA}$ $b = 11.712(2) \text{ \AA}$ $c = 28.215(5) \text{ \AA}$ $\beta = 91.05(2)^\circ$
Volume	$3960.4(15) \text{ \AA}^3$
Z	4
Formula weight	882.8
Density(calc.)	1.480 g/cm^3
Absorption Coefficient	35.40 cm^{-1}
F(000)	1796

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoK α (λ = 0.71073 Å)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
2 θ Range	3.0 to 50.0°
Scan Type	ω
Scan Speed	Variable; 4.00 to 29.30°/min. in ω
Scan Range (ω)	0.80°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 97 reflections
Index Ranges	$0 \leq h \leq 14$, $0 \leq k \leq 13$ $-33 \leq l \leq 33$
Reflections Collected	7301
Independent Reflections	6944 (R_{int} = 2.31%)
Observed Reflections	3452 ($F > 6.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.7320 / 0.9912

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	N/A
Hydrogen Atoms	Riding model, common isotropic U refined
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0010F^2$
Number of Parameters Refined	372
Final R Indices (obs. data)	R = 4.97 %, wR = 5.75 %
R Indices (all data)	R = 11.26 %, wR = 7.48 %
Goodness-of-Fit	1.13
Largest and Mean Δ/σ	5.221, 0.095
Data-to-Parameter Ratio	9.3:1
Largest Difference Peak	1.46 eÅ ⁻³
Largest Difference Hole	-0.84 eÅ ⁻³

NOTE: The tetrafluoroborate anion was disordered and was, therefore, refined using DFIX constraints. Its F-B-F angles were fixed at 109.5° and a common B-F distance was refined. The toluene molecule of crystallization (1/2 equivalent) was refined isotropically. Its disordered methyl group was assigned an occupancy factor of 0.5.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U(eq)
Ir(1)	2706(1)	10130(1)	3593(1)	35(1)
P(1)	856(3)	10077(3)	3345(1)	41(1)
P(2)	2693(4)	11942(3)	3994(2)	42(1)
P(3)	3439(3)	10839(3)	2905(1)	43(1)
O(1)	4373(8)	9979(9)	3814(3)	56(4)
O(2)	2318(9)	9512(7)	4274(3)	48(4)
N(1)	2280(11)	8383(9)	4291(4)	46(5)
C(1)	2502(15)	7776(12)	3910(6)	60(7)
C(2)	2943(15)	8314(10)	3462(5)	51(6)
C(3)	4160(14)	8102(12)	3548(5)	53(6)
C(4)	4787(15)	8933(14)	3748(5)	58(6)
C(5)	2480(15)	7595(12)	3068(5)	65(7)
C(6)	6007(14)	8841(16)	3906(5)	73(7)
C(11)	-21(14)	9076(13)	3676(6)	59(6)
C(12)	-345(16)	9459(15)	4173(6)	83(8)
C(13)	661(13)	9647(13)	2706(5)	57(6)
C(14)	-292(16)	8861(16)	2576(6)	94(9)
C(15)	64(13)	11417(13)	3364(6)	55(6)
C(16)	-1124(13)	11422(15)	3158(6)	73(7)
C(21)	1696(14)	12003(14)	4476(6)	61(7)
C(22)	1740(19)	13013(16)	4815(7)	100(11)
C(23)	4019(13)	12222(13)	4303(6)	61(6)
C(24)	4256(15)	11418(16)	4720(6)	76(7)
C(25)	2423(16)	13261(12)	3643(6)	69(7)
C(26)	2886(20)	14354(14)	3778(7)	104(11)
C(31)	2523(13)	11691(13)	2514(5)	61(6)
C(32)	3029(15)	12664(16)	2220(7)	86(8)
C(33)	3994(18)	9755(14)	2499(6)	87(8)
C(34)	4522(25)	10133(18)	2051(8)	176(16)
C(35)	4622(13)	11798(15)	3008(7)	74(7)
C(36)	5762(17)	11325(25)	3095(10)	191(18)
C(41)	2034(15)	7904(14)	4750(6)	57(7)
C(42)	2486(15)	8484(14)	5145(6)	64(7)
C(43)	2315(16)	8027(15)	5591(5)	69(7)
C(44)	1720(17)	7036(17)	5638(6)	77(8)
C(45)	1249(17)	6498(16)	5240(7)	81(8)
C(46)	1459(17)	6924(15)	4790(6)	70(8)
B(1)	560(10)	621(10)	1371(4)	351(24)
F(1)	1076(13)	-227(13)	1557(6)	189(7)
F(2)	79(14)	1190(12)	1695(5)	180(7)
F(3)	-170(17)	258(18)	1072(7)	316(14)
F(4)	1256(18)	1262(16)	1161(8)	352(16)
C(54)	4268(32)	9467(31)	52(16)	180(14)
C(52)	4265(42)	10277(50)	500(19)	231(20)
C(51)	3832(54)	10433(55)	807(23)	152(23)
C(53)	5027(43)	10901(35)	375(18)	219(18)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

Ir(1)-P(1)	2.315 (3)	Ir(1)-P(2)	2.405 (4)
Ir(1)-P(3)	2.300 (4)	Ir(1)-O(1)	2.089 (9)
Ir(1)-O(2)	2.112 (9)	Ir(1)-C(2)	2.178 (13)
P(1)-C(11)	1.841 (16)	P(1)-C(13)	1.880 (15)
P(1)-C(15)	1.836 (15)	P(2)-C(21)	1.829 (17)
P(2)-C(23)	1.829 (16)	P(2)-C(25)	1.862 (15)
P(3)-C(31)	1.837 (16)	P(3)-C(33)	1.842 (18)
P(3)-C(35)	1.828 (17)	O(1)-C(4)	1.337 (20)
O(2)-N(1)	1.323 (14)	N(1)-C(1)	1.320 (20)
N(1)-C(41)	1.449 (21)	C(1)-C(2)	1.517 (22)
C(2)-C(3)	1.496 (25)	C(2)-C(5)	1.493 (21)
C(3)-C(4)	1.347 (23)	C(4)-C(6)	1.524 (24)
C(11)-C(12)	1.527 (24)	C(13)-C(14)	1.508 (25)
C(15)-C(16)	1.529 (22)	C(21)-C(22)	1.521 (25)
C(23)-C(24)	1.528 (23)	C(25)-C(26)	1.444 (23)
C(31)-C(32)	1.542 (24)	C(33)-C(34)	1.492 (31)
C(35)-C(36)	1.491 (27)	C(41)-C(42)	1.405 (23)
C(41)-C(46)	1.344 (25)	C(42)-C(43)	1.384 (23)
C(43)-C(44)	1.371 (27)	C(44)-C(45)	1.397 (27)
C(45)-C(46)	1.392 (27)	B(1)-F(1)	1.277 (19)
B(1)-F(2)	1.277 (19)	B(1)-F(3)	1.277 (23)
B(1)-F(4)	1.277 (24)	C(51)-C(52)	1.036 (84)
C(52)-C(53)	1.226 (72)	C(52)-C(54)	1.579 (70)
C(53)-C(54A)	1.548 (67)		

Table 3. Bond angles ($^{\circ}$)

P(1)-Ir(1)-P(2)	98.7(1)	P(1)-Ir(1)-P(3)	97.6(1)
P(2)-Ir(1)-P(3)	94.9(1)	P(1)-Ir(1)-O(1)	173.6(3)
P(2)-Ir(1)-O(1)	87.1(3)	P(3)-Ir(1)-O(1)	84.5(3)
P(1)-Ir(1)-O(2)	92.3(3)	P(2)-Ir(1)-O(2)	82.6(3)
P(3)-Ir(1)-O(2)	170.0(3)	O(1)-Ir(1)-O(2)	85.7(4)
P(1)-Ir(1)-C(2)	92.8(5)	P(2)-Ir(1)-C(2)	160.6(4)
P(3)-Ir(1)-C(2)	98.9(4)	O(1)-Ir(1)-C(2)	80.9(5)
O(2)-Ir(1)-C(2)	81.5(4)	Ir(1)-P(1)-C(11)	114.6(5)
Ir(1)-P(1)-C(13)	113.5(5)	C(11)-P(1)-C(13)	104.7(7)
Ir(1)-P(1)-C(15)	117.4(5)	C(11)-P(1)-C(15)	103.2(7)
C(13)-P(1)-C(15)	101.7(7)	Ir(1)-P(2)-C(21)	113.3(5)
Ir(1)-P(2)-C(23)	111.7(5)	C(21)-P(2)-C(23)	102.3(7)
Ir(1)-P(2)-C(25)	118.9(5)	C(21)-P(2)-C(25)	104.8(8)
C(23)-P(2)-C(25)	104.2(8)	Ir(1)-P(3)-C(31)	118.0(5)
Ir(1)-P(3)-C(33)	115.1(6)	C(31)-P(3)-C(33)	102.7(8)
Ir(1)-P(3)-C(35)	113.3(6)	C(31)-P(3)-C(35)	102.4(8)
C(33)-P(3)-C(35)	103.5(9)	Ir(1)-O(1)-C(4)	113.1(9)
Ir(1)-O(2)-N(1)	112.6(8)	O(2)-N(1)-C(1)	120.1(12)
O(2)-N(1)-C(41)	115.3(11)	C(1)-N(1)-C(41)	124.5(12)
N(1)-C(1)-C(2)	122.2(12)	Ir(1)-C(2)-C(1)	102.4(9)
Ir(1)-C(2)-C(3)	105.3(9)	C(1)-C(2)-C(3)	98.5(12)
Ir(1)-C(2)-C(5)	129.1(11)	C(1)-C(2)-C(5)	104.8(12)
C(3)-C(2)-C(5)	112.1(13)	C(2)-C(3)-C(4)	118.9(14)
O(1)-C(4)-C(3)	120.9(15)	O(1)-C(4)-C(6)	112.4(14)
C(3)-C(4)-C(6)	126.6(16)	P(1)-C(11)-C(12)	115.9(11)
P(1)-C(13)-C(14)	118.6(11)	P(1)-C(15)-C(16)	118.0(11)
P(2)-C(21)-C(22)	118.8(13)	P(2)-C(23)-C(24)	113.8(11)
P(2)-C(25)-C(26)	122.2(13)	P(3)-C(31)-C(32)	119.2(12)
P(3)-C(33)-C(34)	119.2(13)	P(3)-C(35)-C(36)	120.3(15)
N(1)-C(41)-C(42)	116.1(14)	N(1)-C(41)-C(46)	121.3(15)
C(42)-C(41)-C(46)	122.5(16)	C(41)-C(42)-C(43)	118.1(16)
C(42)-C(43)-C(44)	120.1(15)	C(43)-C(44)-C(45)	120.5(16)
C(44)-C(45)-C(46)	119.5(17)	C(41)-C(46)-C(45)	119.0(17)
F(1)-B(1)-F(2)	109.5(13)	F(1)-B(1)-F(3)	109.5(14)
F(2)-B(1)-F(3)	109.5(14)	F(1)-B(1)-F(4)	109.5(14)
F(2)-B(1)-F(4)	109.5(14)	F(3)-B(1)-F(4)	109.5(16)
C(51)-C(52)-C(53)	121.5(66)	C(51)-C(52)-C(54)	141.6(60)
C(53)-C(52)-C(54)	96.8(43)	C(52)-C(53)-C(54A)	118.5(43)
C(52)-C(54)-C(53A)	143.1(37)		

Table 4. Anisotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	40(1)	29(1)	35(1)	1(1)	1(1)	3(1)
P(1)	40(2)	40(2)	44(2)	-8(2)	2(1)	2(2)
P(2)	46(3)	37(2)	44(3)	-1(2)	-5(2)	-5(2)
P(3)	41(2)	47(2)	43(2)	6(2)	6(2)	9(2)
O(1)	44(5)	68(7)	58(6)	1(7)	0(4)	5(6)
O(2)	70(8)	36(5)	39(6)	2(5)	17(5)	-1(4)
N(1)	60(9)	39(7)	38(8)	-1(6)	-3(7)	9(5)
C(1)	85(16)	32(10)	64(12)	2(9)	7(11)	4(9)
C(2)	88(13)	21(7)	44(9)	12(8)	16(9)	3(6)
C(3)	83(13)	43(8)	34(8)	23(9)	14(8)	1(7)
C(4)	63(12)	64(11)	47(9)	42(10)	14(9)	1(8)
C(5)	98(15)	49(10)	49(10)	-12(10)	18(10)	-14(8)
C(6)	66(13)	113(14)	38(9)	35(11)	2(9)	11(9)
C(11)	49(11)	65(10)	64(11)	-19(8)	14(9)	18(9)
C(12)	79(14)	106(14)	65(12)	-7(11)	31(11)	33(10)
C(13)	55(10)	67(10)	47(9)	-11(8)	-7(8)	3(7)
C(14)	111(19)	94(14)	77(14)	-65(13)	13(13)	-42(11)
C(15)	46(10)	60(9)	58(10)	7(8)	7(9)	8(8)
C(16)	51(11)	81(12)	86(14)	14(10)	5(10)	6(10)
C(21)	60(12)	73(11)	50(11)	3(9)	-21(10)	-26(9)
C(22)	121(22)	89(15)	89(19)	-10(14)	6(15)	-32(13)
C(23)	59(11)	58(10)	66(11)	4(9)	-2(9)	-15(9)
C(24)	71(13)	100(14)	58(11)	-1(11)	-5(10)	-22(10)
C(25)	104(17)	39(8)	63(12)	-4(9)	1(11)	-5(8)
C(26)	163(25)	56(11)	94(16)	5(14)	-9(16)	15(10)
C(31)	58(11)	77(11)	46(9)	-12(9)	-5(8)	33(8)
C(32)	63(13)	105(15)	90(14)	-4(11)	-7(11)	56(12)
C(33)	112(17)	64(11)	88(14)	8(11)	46(12)	8(10)
C(34)	289(39)	105(18)	138(21)	86(23)	139(24)	33(17)
C(35)	37(10)	99(14)	86(13)	-21(10)	-12(9)	18(11)
C(36)	45(14)	278(38)	250(37)	-27(20)	12(19)	150(31)
C(41)	66(12)	59(11)	47(11)	14(9)	6(9)	11(8)
C(42)	75(13)	68(11)	50(10)	6(10)	14(10)	6(9)
C(43)	91(14)	75(12)	40(9)	-1(11)	7(9)	7(9)
C(44)	94(16)	97(15)	41(10)	34(13)	3(10)	23(10)
C(45)	88(16)	69(12)	88(15)	-15(11)	10(13)	21(11)
C(46)	82(15)	72(12)	56(13)	-7(11)	11(11)	12(9)

The anisotropic displacement exponent takes the form:

$$-2\pi^2 (h^2 a^{*2} U_{11} + \dots + 2hka^*b^*U_{12})$$

Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
H(1A)	2363	6969	3915	87
H(3A)	4484	7385	3460	87
H(5A)	2729	6822	3111	87
H(5B)	1680	7617	3068	87
H(5C)	2741	7880	2771	87
H(6A)	6272	8079	3853	87
H(6B)	6445	9371	3729	87
H(6C)	6069	9020	4237	87
H(11A)	372	8365	3708	87
H(11B)	-688	8933	3492	87
H(12A)	-799	8886	4318	87
H(12B)	314	9583	4364	87
H(12C)	-758	10158	4146	87
H(13A)	1332	9273	2609	87
H(13B)	587	10334	2522	87
H(14A)	-303	8705	2242	87
H(14B)	-219	8158	2748	87
H(14C)	-974	9233	2660	87
H(15A)	487	12003	3212	87
H(15B)	5	11625	3692	87
H(16A)	-1465	12158	3193	87
H(16B)	-1077	11239	2827	87
H(16C)	-1566	10856	3314	87
H(21A)	1815	11337	4669	87
H(21B)	957	11947	4341	87
H(22A)	1184	12937	5053	87
H(22B)	2464	13070	4964	87
H(22C)	1594	13689	4631	87
H(23A)	4021	12990	4422	87
H(23B)	4612	12156	4081	87
H(24A)	4961	11595	4869	87
H(24B)	3673	11492	4947	87
H(24C)	4270	10650	4602	87
H(25A)	1628	13358	3633	87
H(25B)	2648	13119	3323	87
H(26A)	2643	14935	3560	87
H(26B)	2648	14546	4092	87
H(26C)	3686	14303	3777	87
H(31A)	1953	12014	2708	87
H(31B)	2159	11182	2294	87
H(32A)	2455	13037	2035	87
H(32B)	3373	13205	2432	87
H(32C)	3582	12361	2012	87
H(33A)	3387	9251	2420	87
H(33B)	4552	9316	2665	87
H(34A)	4769	9500	1863	87
H(34B)	3965	10555	1876	87
H(34C)	5145	10621	2124	87
H(35A)	4458	12272	3275	87
H(35B)	4660	12290	2737	87
H(36A)	6293	11931	3139	87
H(36B)	5759	10854	3374	87
H(36C)	5964	10872	2827	87
H(42A)	2895	9180	5102	87

H(43A)	2611	8411	5866	87
H(44A)	1652	6690	5944	87
H(45A)	769	5851	5282	87
H(46A)	1206	6543	4507	87