Electronic Supplementary Information for Dalton Transactions

New Bimetallic Complexes of Late Transition Metals Involving Pyrazole-Bridged Bis *N*-Heterocyclic Carbene Ligands

Sang-Jin Jeon and Robert M. Waymouth*

Department of Chemistry, Stanford University,

Stanford, California 94305.

Supporting Information

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General Considerations. All organometallic reactions were performed under an inert atmosphere of N_2 using standard Schlenk line or glove box techniques. Degassing of solvents was achieved via multiple freeze pump thaw cycles. 2,6-Diisopropyl-phenyl imidazole[1] and 3,5-Bis(chloromethyl)-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole[2] were prepared by literature procedure. Sodium hexamethyldisilazane (NaHMDS), $NiCl_2$(dme), $[Rh(COD)Cl]_2$, and $[Ir(COD)Cl]_2$ were purchased from Strem and stored in an inert atmosphere. Celite was baked out at 140 °C prior to use. THF-d_8 and CD_2Cl_2 were purchased from Cambridge Isotope, Inc., degassed, dried over sodium benzophenone and CaH_2 respectively, and vacuum transferred. DMSO-d_6 was purchased from Cambridge Isotope, Inc., fractionally distilled, and stored in activated 4Å molecular sieves prior to use. NMR spectra were taken on a Varian Unity Inova 500 MHz and Mercury 400 MHz NMR spectrometer. Chemical shifts were referenced to residual solvent peak. High resolution mass spectroscopy was performed at Stanford University Mass Spectrometry Laboratory. Elemental analyses were performed by Robertson Microlit Laboratories, Inc., Madison, N.J. X-ray diffraction study was performed by the X-ray crystallographic laboratory at the University of Minnesota. A single crystal was mounted on a Bruker-Siemens SMART Platform CCD diffractometer at 173(2) K and the data collection was carried out using MoKα radiation with a frame time of 30-45 seconds and a detector distance of 4.89-4.97 cm. The X-ray structures were solved using SHELXS-97 and refined using SHELXL-97.

Synthesis of Ligand Precursors.

3,5-Bis-[3-(2,6-diisopropyl-phenyl)-3*H*-imidazolium-1-ylmethyl]-1-(tetrahydro-pyran-2-yl)-1*H*-pyrazole diiodide (1). 3,5-Bis(chloromethyl)-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole (1.02 g, 4.09 mmol), 2,6-diisopropyl-phenyl imidazole (2.06 g, 9.01 mmol), and NaI (1.23 g, 8.18 mmol) were dissolved in extra dried acetone (70 mL). The reaction was stirred for 20 h at 60 ^oC and solvent was removed in vacuo. The resulting powder was redissolved in CH_2Cl_2 (25 mL) and filtered through Celite. The filtrate was concentrated and poured into ether (60 mL) to precipitate the product. The pale yellow solids were collected on a Frit and dried in a vacuum (3.50 g, 96% yield). ^1H NMR (500

MHz, DMSO-d_6): δ 1.06-1.17 (m, 24H, isopropyl CH_3), 1.41-1.52 (m, 1H, thp CH), 1.52-1.59 (m, 1H, thp CH), 1.61-1,74 (m, 1H, thp CH), 1.83-1.99 (m, 2H, thp CH_2), 2.11-2.21 (m, 2H, isopropyl CH), 2.22-2.34 (m, 3H, 2H from isopropyl CH and 1H from thp CH), 3.66-3.70 (m, 2H, thp CH_2), 5.64 (s, 2H, bridged CH_2), 5.70 (dd, 1H, J = 9.5 Hz, 2.5 Hz, thp CH), 5.84 (d, 1H, J = 15.5 Hz, bridged CH_2), 5.89 (d, 1H, J = 15.5 Hz, bridged CH_2), 6.76 (s, 1H, pyrazolyl CH), 7.43-7.48 (m, 4H, *m*-Ph), 7.61-7.66 (m, 2H, *p*-Ph), 8.11 (s, 2H, NCH), 8.14-8.17 (m, 2H, NCH), 9.59 (s, 1H, imidazolium CH), 9.70 (s, 1H, imidazolium CH). ^{13}C NMR (125 MHz, DMSO-d_6): δ 22.34 (thp CH_2), 24.36-24.77 (8 isopropyl CH_3), 25.25 (thp CH_2), 28.67-28.78 (4 isopropyl CH), 29.24 (thp CH_2), 43.73 (bridged CH_2), 47.44 (bridged CH_2), 67.16 (thp CH_2), 84.45 (thp CH), 108.80 (pyrazolyl CH), 124.06 (NCH), 124.53 (NCH), 125.17 (*m*-Ph), 125.84 (NCH), 125.88 (NCH), 131.20 (*ipso*-Ph), 131.25 (*ipso*-Ph), 132.24 (*p*-Ph), 138.25 (imidazolium C), 138.75 (imidazolium C), 145.67, 145.73 (*o*-Ph), 145.74, 145.78 (pyrazolyl C). HRMS calcd for $C_{40}H_{54}N_6ONaI_2$ $(M + Na)^+$: 911.2346, found 911.2347.

3,5-Bis-[3-(2,6-diisopropyl-phenyl)-3*H*-imidazolium-1-ylmethyl]-1*H*-pyrazole diiodide (2). The N-thp protected salt **1** (3.50 g, 3.94 mmol) and pyridinium *p*-toluenesulfonate (1.55 g, 6.17 mmol) were dissolved in ethanol (35 mL) and stirred for 12 h at 55 ^{o}C. The resulting cloudy yellow reaction mixture was filtered to give an off-white solid, which was washed with ether (30 mL), and dried in vacuo (1.70 g, 54% yield). The filtrate was evaporated and purified by column chromatography on silica gel (CH_2Cl_2 / acetone : 1 / 1) to obtain an additional 0.54 g of material (combined yield: 71%). ^{1}H NMR (500 MHz, DMSO-d_6): δ 1.13 (d, 24H, J = 7 Hz, isopropyl CH_3), 2.27 (m, 4H, isopropyl CH), 5.70 (br, 4H, brided CH_2), 6.59 (s, 1H, pyrazolyl CH), 7.46 (d, 4H, J = 8 Hz, *m*-Ph), 7.63 (t, 2H, J = 8 Hz, *p*-Ph), 8.11 (s, 2H, NCH), 8.16 (s, 2H, NCH), 9.70 (s, 2H, imidazolium CH). ^{13}C NMR (125 MHz, DMSO-d_6): δ 24.58 (8 isopropyl CH_3), 28.76 (4 isopropyl CH), 44.42 (bridged CH_2), 47.52 (bridged CH_2), 105.67 (pyrazolyl CH), 124.16 (NCH), 125.18 (*m*-Ph), 125.96 (NCH), 131.20 (*ipso*-Ph), 132.25 (*p*-Ph), 138.72 (imidazolium C), 145.78 (*o*-Ph, pyrazolyl C). HRMS calcd for $C_{35}H_{46}N_6I$ $(M - I)^+$: 677.2829, found 677.2825.

3,5-Bis-[3-(2,6-diisopropyl-phenyl)-3*H*-imidazolium-1-ylmethyl]-1*H*-pyrazole dihexafluorophosphate (3). To a solution of deprotected salt **2** (986 mg, 1.23 mmol) in MeOH / H_2O (15 mL / 12 mL) was added NH_4PF_6 (601 mg, 3.69 mmol). The clear solution immediately turned cloudy and was stirred for 2 h. The reaction mixture was filtered, washed with water (10 mL) and ether (10 mL), and dried under vacuum to give an off-white product (972 mg, 94% yield). ^{1}H NMR (500 MHz, DMSO-d_6): δ 1.12 (d, 24H, J = 7 Hz, isopropyl CH_3), 2.22-2.26 (m, 4H, isopropyl CH), 5.61 (br, 4H, bridged CH_2), 6.50 (s, 1H, pyrazolyl CH), 7.45 (d, 4H, J = 8 Hz, *m*-Ph), 7.62 (t, 2H, J = 8 Hz, *p*-Ph), 8.03 (s, 2H, NCH), 8.13 (s, 2H, NCH), 9.60 (s, 2H, imidazolium CH). ^{13}C NMR (125 MHz, DMSO-d_6): δ 24.49 (isopropyl CH_3), 28.75 (isopropyl CH), 44.46 (bridged CH_2), 47.50 (bridged CH_2), 105.53 (pyrazolyl CH), 123.97, 124.29 (NCH), 125.16 (*m*-Ph), 125.79, 126.16 (NCH), 131.28 (*ipso*-Ph), 132.25 (*p*-Ph), 138.10, 138.75 (imidazolium C), 145.77 (*o*-Ph), 146.82 (pyrazolyl C). ^{19}F NMR (376 MHz, DMSO-d_6): δ -70.54 (d, J = 712 Hz). ^{31}P NMR (162 MHz, DMSO-d_6): δ -143.12 (sept, J = 712 Hz). HRMS calcd for $C_{35}H_{46}N_6F_6P$ $(M - PF_6)^+$: 695.3426, found 695.3433.

Synthesis of Metal Carbene Complexes.

$(dIPrpy)_2Ni_2^{2+}(PF_6^-)_2$ complex 4. In a nitrogen glove box, the ligand precursor **3** (84.1 mg, 0.1 mmol) and NaHMDS (57.9 mg, 0.3 mmol) were dissolved in THF (3 mL) and stirred at room temperature. After 15 min, this reaction mixture was added to the flask containing $NiCl_2$(dme) (44 mg, 0.2 mmol) rinsing with THF (3 mL). The resulting red-brown cloudy solution was stirred for 10 h and then removed from the box. Solvent was removed in vacuo and the residue was column chromatographed on silica gel (CH_2Cl_2 / acetone : 9 / 1) to give the product as a orange-red solid (56 mg, 74.5 % yield). Slow evaporation of CH_2Cl_2 / THF solution yielded X-ray-quality crystals. ^{1}H NMR (500 MHz, CD_2Cl_2): δ 0.27 (d, 6H, J = 6.5 Hz, isopropyl CH_3), 0.75 (d, 6H, J = 6.5 Hz, isopropyl CH_3), 0.78 (d, 6H, J = 6.5 Hz, isopropyl CH_3), 1.06 (d, 6H, J = 6.5 Hz, isopropyl CH_3), 2.19-2.25 (m, 2H, isopropyl CH), 3.07 (d, 2H, J = 16.5 Hz, bridged CH_2), 3.12-3.17 (m, 2H, isopropyl CH), 4.78 (d, 2H, J = 16.5 Hz, bridged CH_2), 6.23 (s, 1H, pyrazolyl CH), 7.01 (d, 2H, J = 2 Hz, NCH), 7.10 (d, 2H, J = 2 Hz, NCH),7.27 (d,

2H, *J* = 8 Hz, *m*-Ph), 7.36 (d, 2H, *J* = 8 Hz, *m*-Ph), 7.63 (t, 2H, *J* = 7.5 Hz, *p*-Ph). ^{13}C NMR (125 MHz, CD_2Cl_2): δ 19.59, 22.92, 27.07, 27.25, 28.02, 30.52 (isopropyl CH and CH_3), 47.23 (bridged CH_2), 102.88 (pyrazolyl CH), 122.37 (NCH), 124.62 (*m*-Ph), 125.46 (*m*-Ph), 127.22 (NCH), 131.89 (*p*-Ph), 134.38 (*ipso*-Ph), 145.45 (*o*-Ph), 145.88 (*o*-Ph), 148.85 (pyrazolyl C), 153.22 (carbene). ^{19}F NMR (376 MHz, CD_2Cl_2): δ -72.65 (d, *J* = 712 Hz). ^{31}P NMR (162 MHz, CD_2Cl_2): δ -143.01 (sept, *J* = 712 Hz). Anal. Calcd for $C_{70}H_{86}F_{12}N_{12}Ni_2P_2$: C 55.94, H 5.77, N 11.18; Found: C 56.00, H 5.73, N 11.10.

(dIPrpy)Rh$_2^+$(COD)$_2$(PF$_6^-$) complex 5. In a nitrogen glove box, the ligand precursor **3** (84.1 mg, 0.1 mmol) and NaHMDS (57.9 mg, 0.3 mmol) were dissolved in THF (3 mL) and stirred at room temperature. After 15 min, this reaction mixture was added to a solution of $[Rh(COD)Cl]_2$ (49.3 mg, 0.1 mmol) in THF (2 mL). The resulting dark yellow solution was stirred for 10 h and then removed from the box and filtered through Celite in air. The dark yellow filtrate was concentrated and purified by column chromatography on silica gel (DCM / MeOH : 97 / 3) to give the product as a dark yellow solid (95 mg, 85% yield). Slow diffusion of pentane into a CH_2Cl_2 solution yielded X-ray-quality crystals. ^{1}H NMR (500 MHz, CD_2Cl_2): δ 0.73 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.13 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.17 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.23 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.58-1.64 (m, 2H, CH_{2cod}), 1.65-1.74 (m, 2H, CH_{2cod}), 1.95-2.07 (m, 7H, CH_{2cod}), 2.14-2.24 (m, 7H, CH_{2cod}), 2.31-2.40 (m, 2H, isopropyl CH), 2.93-3.05 (m, 2H, isopropyl CH), 3.28-3.31 (m, 2H, CH_{cod}), 3.78-3.83 (m, 2H, CH_{cod}), 4.03-4.06 (m, 2H, CH_{cod}), 5.24 (d, 2H, *J* = 15.5 Hz, bridged CH_2). 5.72 (d, 2H, *J* = 15.5 Hz, bridged CH_2), 6.17-6.22 (m, 2H, CH_{cod}), 6.44 (s, 1H, pyrazolyl CH), 6.79-6.80 (m, 2H, NCH), 7.25 (d, 2H, *J* = 8 Hz, *m*-Ph), 7.31 (d, 2H, *J* = 8 Hz, *m*-Ph), 7.31-7.32 (m, 2H, NCH), 7.47 (t, 2H, *J* = 8 Hz, *p*-Ph). ^{13}C NMR (125 MHz, CD_2Cl_2): δ 22.75, 22.89, 25.14, 26.57 (isopropyl CH_3), 27.83 (CH_{2cod}), 28.27, 28.99 (isopropyl CH), 30.01 (CH_{2cod}), 31.52 (CH_{2cod}), 34.81 (CH_{2cod}), 49.08 (bridged CH_2), 74.08 (d, $^1J_{RhC}$ = 12 Hz, CH_{cod}), 74.99 (d, $^1J_{RhC}$ = 11 Hz, CH_{cod}), 93.83 (d, $^1J_{RhC}$ = 8 Hz, CH_{cod}), 97.26 (d, $^1J_{RhC}$ = 7 Hz, CH_{cod}), 101.42 (pyrazolyl CH), 121.34 (NCH), 123.80 (*m*-Ph), 124.47 (*m*-Ph), 125.07 (NCH), 130.46 (*p*-Ph), 135.94 (*ipso*-Ph), 144.63 (*o*-Ph), 145.37 (*o*-Ph), 146.47 (pyrazolyl C), 176.75 (d, $^1J_{Rh\text{-}Carbene}$ = 52 Hz). ^{19}F NMR (376 MHz, CD_2Cl_2): δ -73.26 (d, *J* = 712

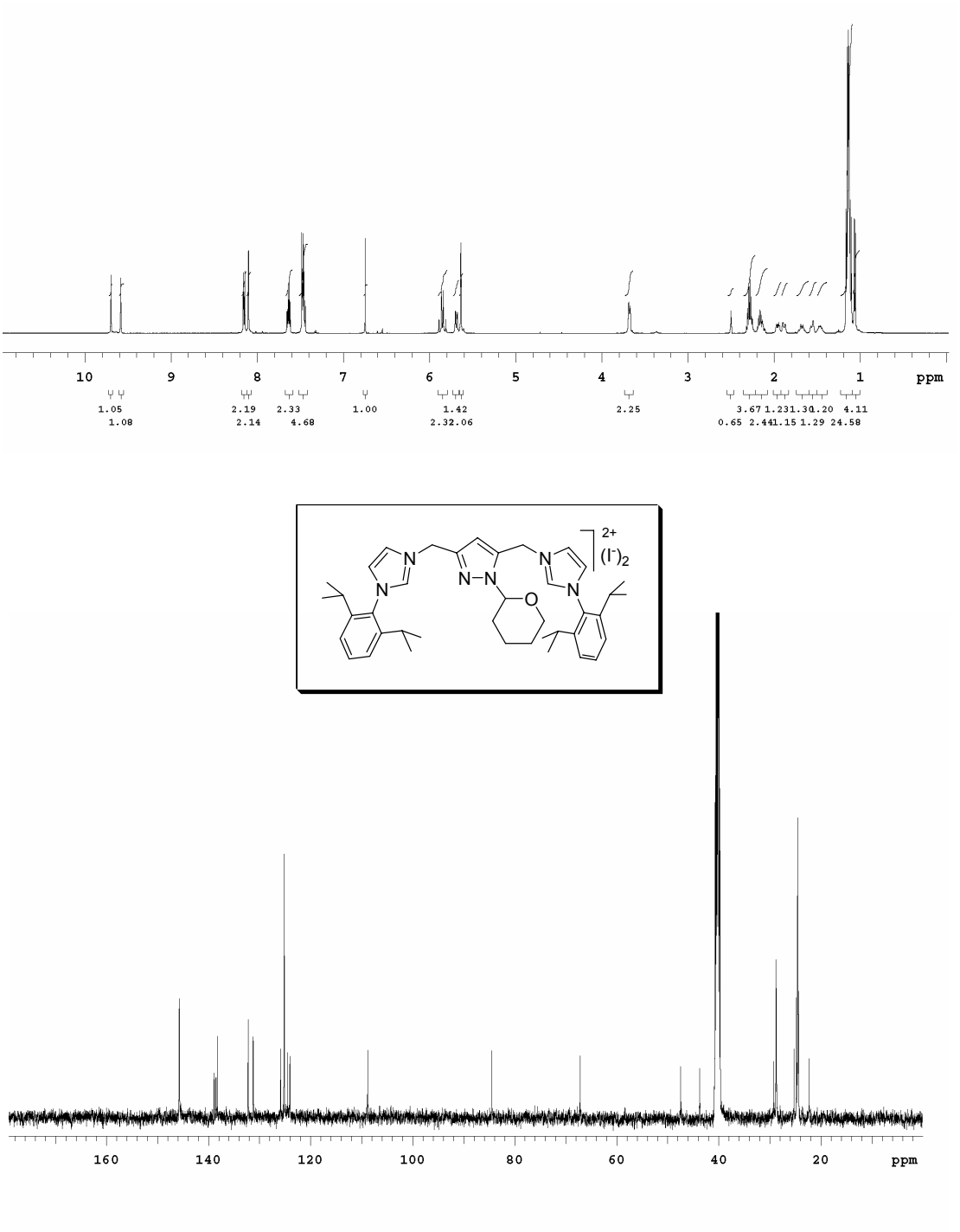
Hz). ^{31}P NMR (162 MHz, CD_2Cl_2): δ -143.22 (sept, *J* = 712 Hz). Anal. Calcd for $C_{51}H_{67}F_6N_6PRh_2$: C 54.94, H 6.06, N 7.54; Found: C 54.72, H 6.11, N 7.42.

(dIPrpy)Ir$_2^+$(COD)$_2$(PF$_6^-$) complex 6. In a nitrogen glove box, the ligand precursor **3** (84.1 mg, 0.1 mmol) and NaHMDS (57.9 mg, 0.3 mmol) were dissolved in THF (3 mL) and stirred at room temperature. After 15 min, this reaction mixture was added to a solution of $[Ir(COD)Cl]_2$ (67.2 mg, 0.1 mmol) in THF (2 mL). The resulting orange-red solution was stirred for 10 h and then removed from the box and filtered through Celite in air. The orange-red filtrate was concentrated and purified by column chromatography on silica gel (DCM / MeOH : 97 / 3) to give the product as an orange crystalline solid (115 mg, 89% yield). Slow diffusion of pentane into a CH_2Cl_2 solution yielded X-ray-quality crystals. ^{1}H NMR (500 MHz, CD_2Cl_2): δ 0.73 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 0.93-1.09 (m, 2H, CH_{2cod}), 1.14 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.17 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.19 (d, 6H, *J* = 7 Hz, isopropyl CH_3), 1.23-1.35 (m, 2H, CH_{2cod}), 1.73-1.84 (m, 2H, CH_{2cod}), 1.86-1.99 (m, 8H, CH_{2cod}), 2.02-2.13 (m, 2H, CH_{2cod}), 2.26-2.32 (m, 2H, isopropyl CH), 2.87-2.92 (m, 2H, isopropyl CH), 3.07-3.11 (m, 2H, CH_{cod}), 3.37-3.42 (m, 2H, CH_{cod}), 3.68-3.73 (m, 2H, CH_{cod}), 5.23 (d, 2H, *J* = 15.5 Hz, bridged CH_2). 5.50 (d, 2H, *J* = 15.5 Hz, bridged CH_2), 5.49-5.56 (m, 2H, CH_{cod}), 6.53 (s, 1H, pyrazolyl CH), 6.86 (s, 2H, NCH), 7.25 (d, 2H, *J* = 8 Hz, *m*-Ph), 7.27 (d, 2H, *J* = 8 Hz, *m*-Ph), 7.33 (s, 2H, NCH), 7.48 (t, 2H, *J* = 8 Hz, *p*-Ph). ^{13}C NMR (125 MHz, CD_2Cl_2): δ 22.73, 22.79, 25.12, 26.52 (isopropyl CH_3), 27.60 (CH_{2cod}), 28.33, 29.10 (isopropyl CH), 29.76 (CH_{2cod}), 33.16 (CH_{2cod}), 36.30 (CH_{2cod}), 48.85 (bridged CH_2), 58.53 (CH_{cod}), 60.04 (CH_{cod}), 82.01 (CH_{cod}), 84.80 (CH_{cod}), 101.84 (pyrazolyl CH), 120.88 (NCH), 123.83 (*m*-Ph), 124.48 (*m*-Ph), 125.47 (NCH), 130.52 (*p*-Ph), 135.88 (*ipso*-Ph), 144.59 (*o*-Ph), 145.39 (*o*-Ph), 146.52 (pyrazolyl C), 173.03 (carbene). ^{19}F NMR (376 MHz, CD_2Cl_2): δ -73.26 (d, *J* = 712 Hz). ^{31}P NMR (162 MHz, CD_2Cl_2): δ -143.25 (sept, *J* = 712 Hz). Anal. Calcd for $C_{51}H_{67}F_6Ir_2N_6P$: C 47.36, H 5.22, N 6.50; Found: C 47.16, H 5.20, N 6.35.

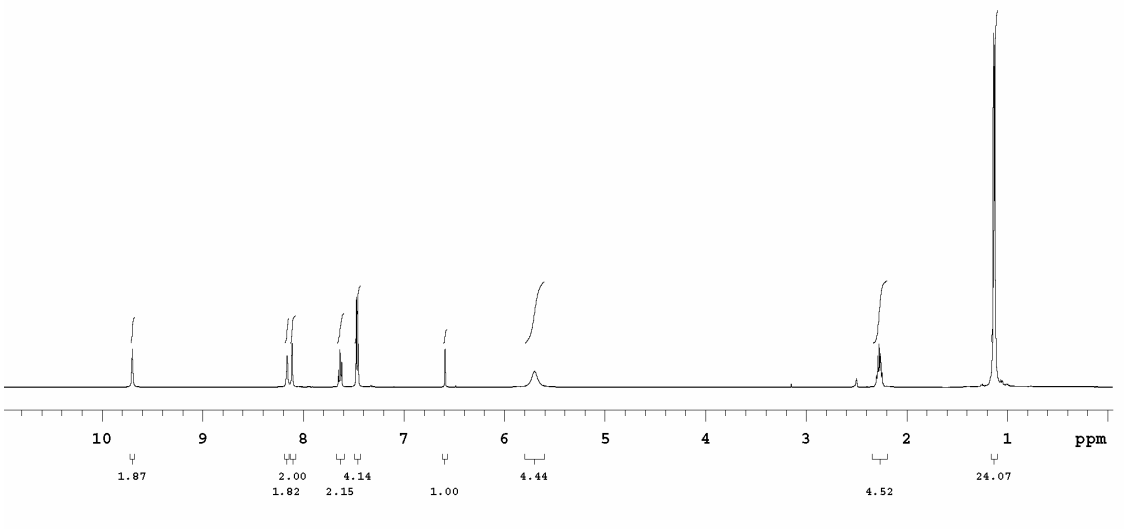
References.

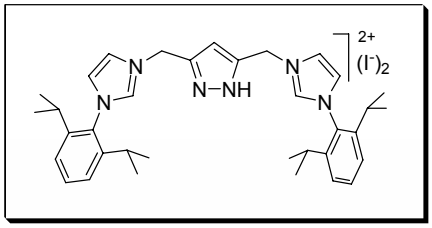
(1) Liu, J. P.; Chen, J. B.; Zhao, J. F.; Zhao, Y. H.; Li, L.; Zhang, H. B. *Synthesis* **2003**, 2661-2666.

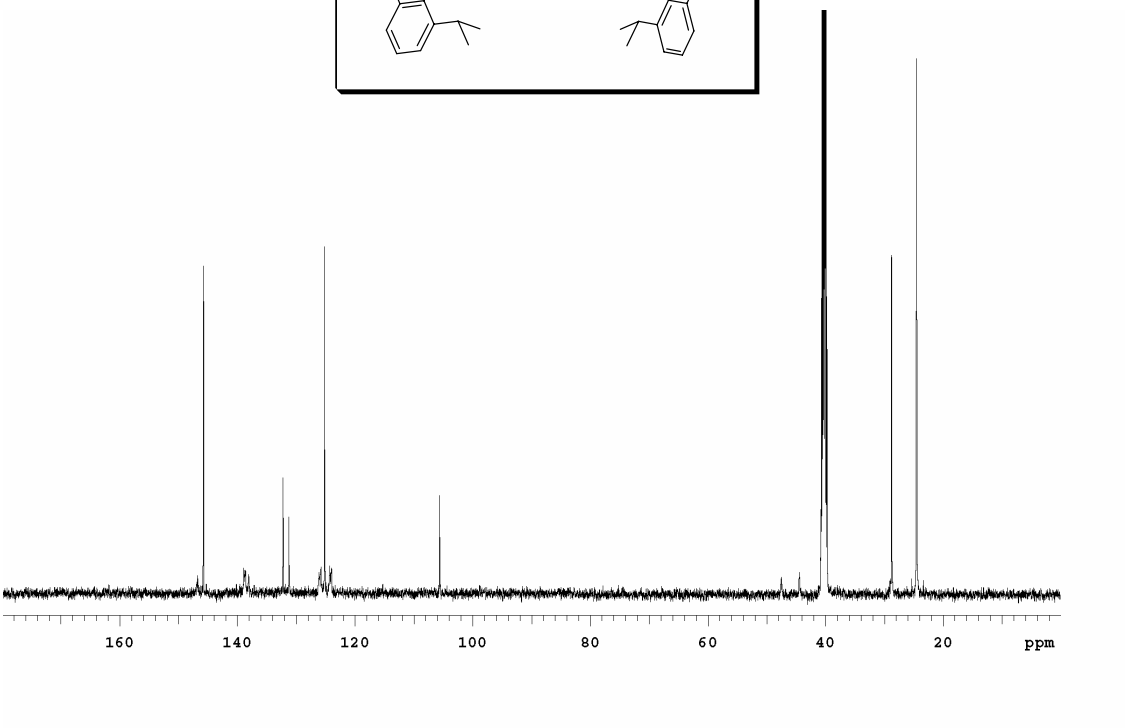
(2) Röder, J. C.; Meyer, F.; Pritzkow, H. *organometallics* **2001**, *20*, 811-817.



^{1}H and ^{13}C NMR of Salt 1 in DMSO-d_6







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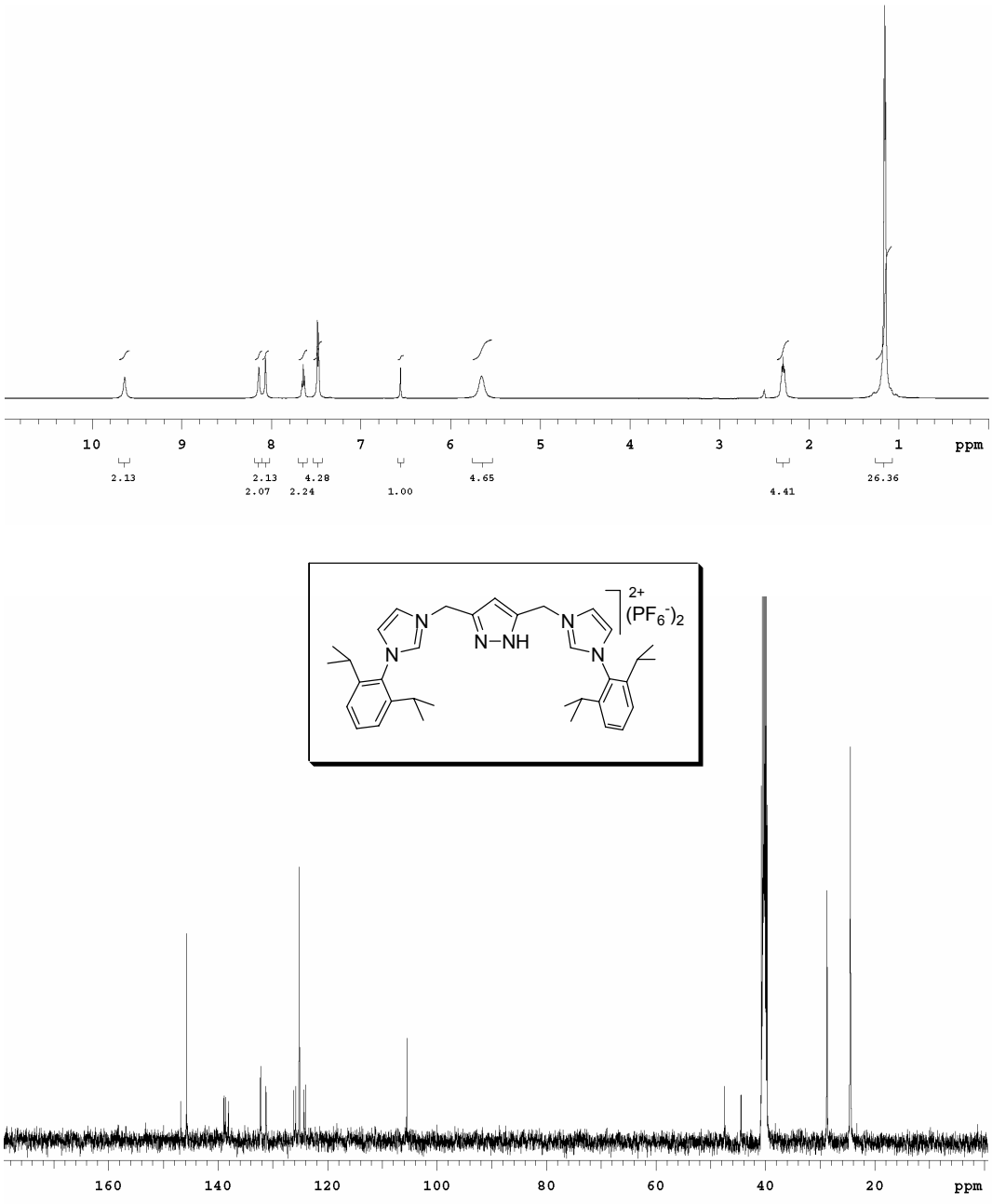

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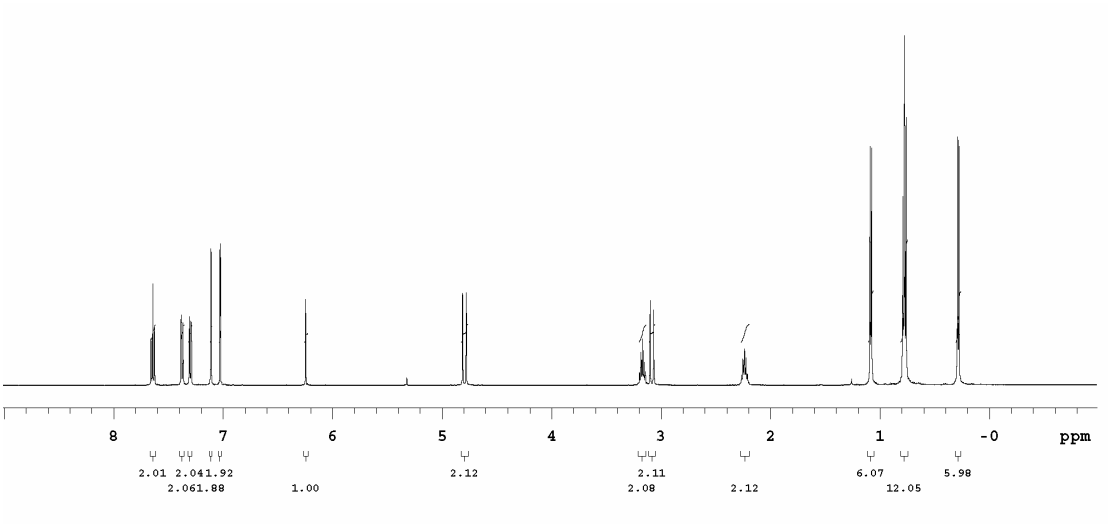

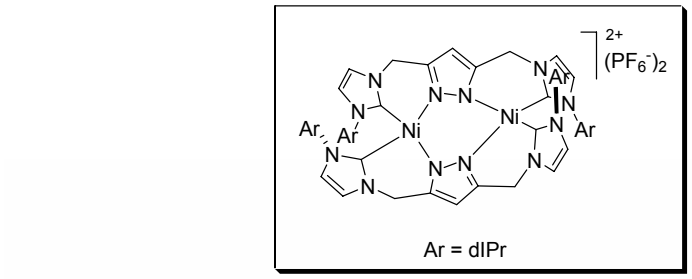

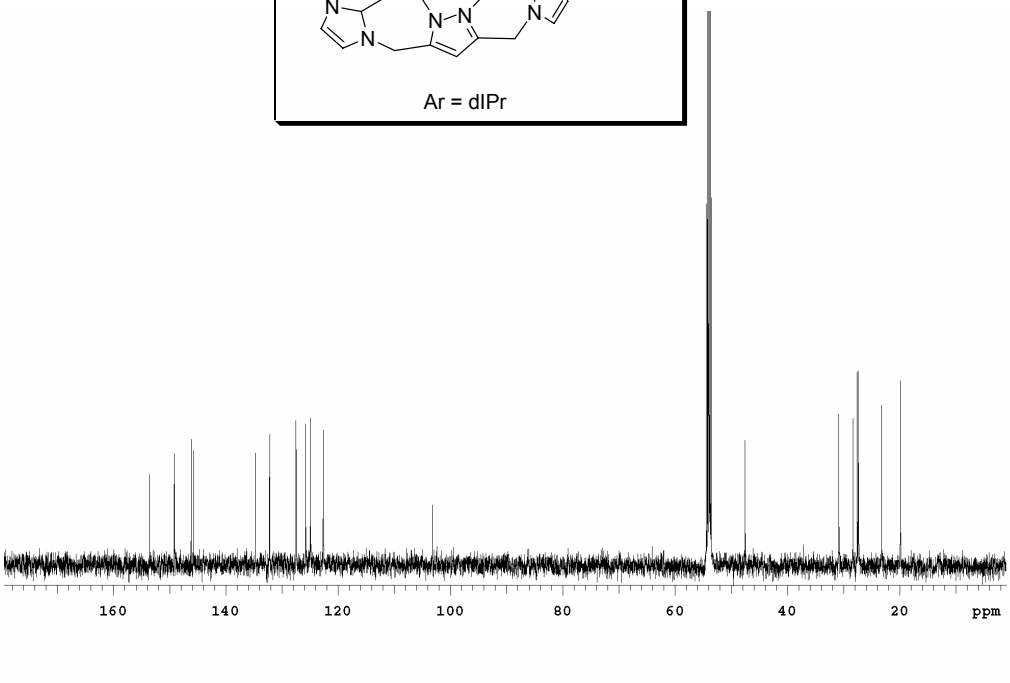

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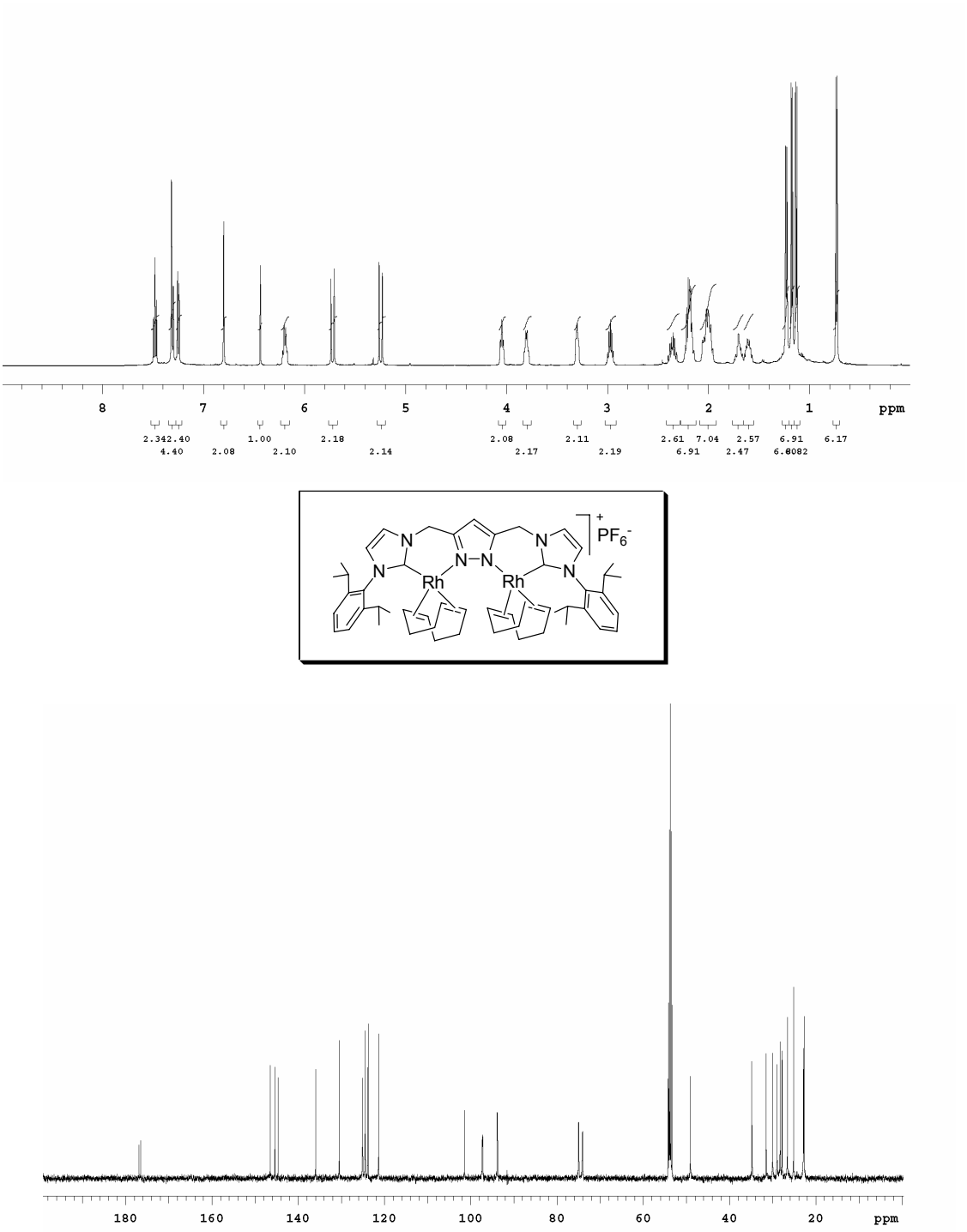
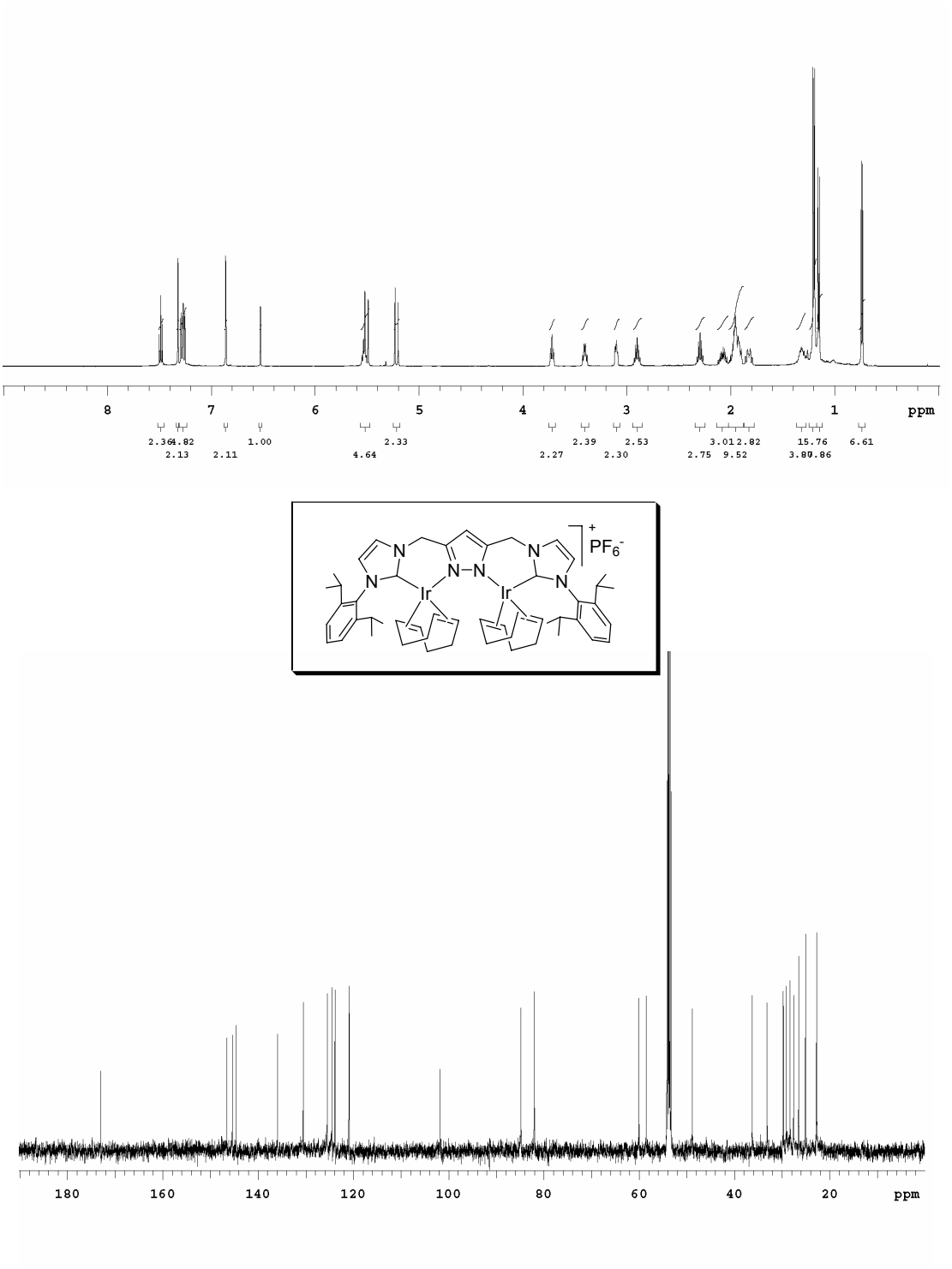
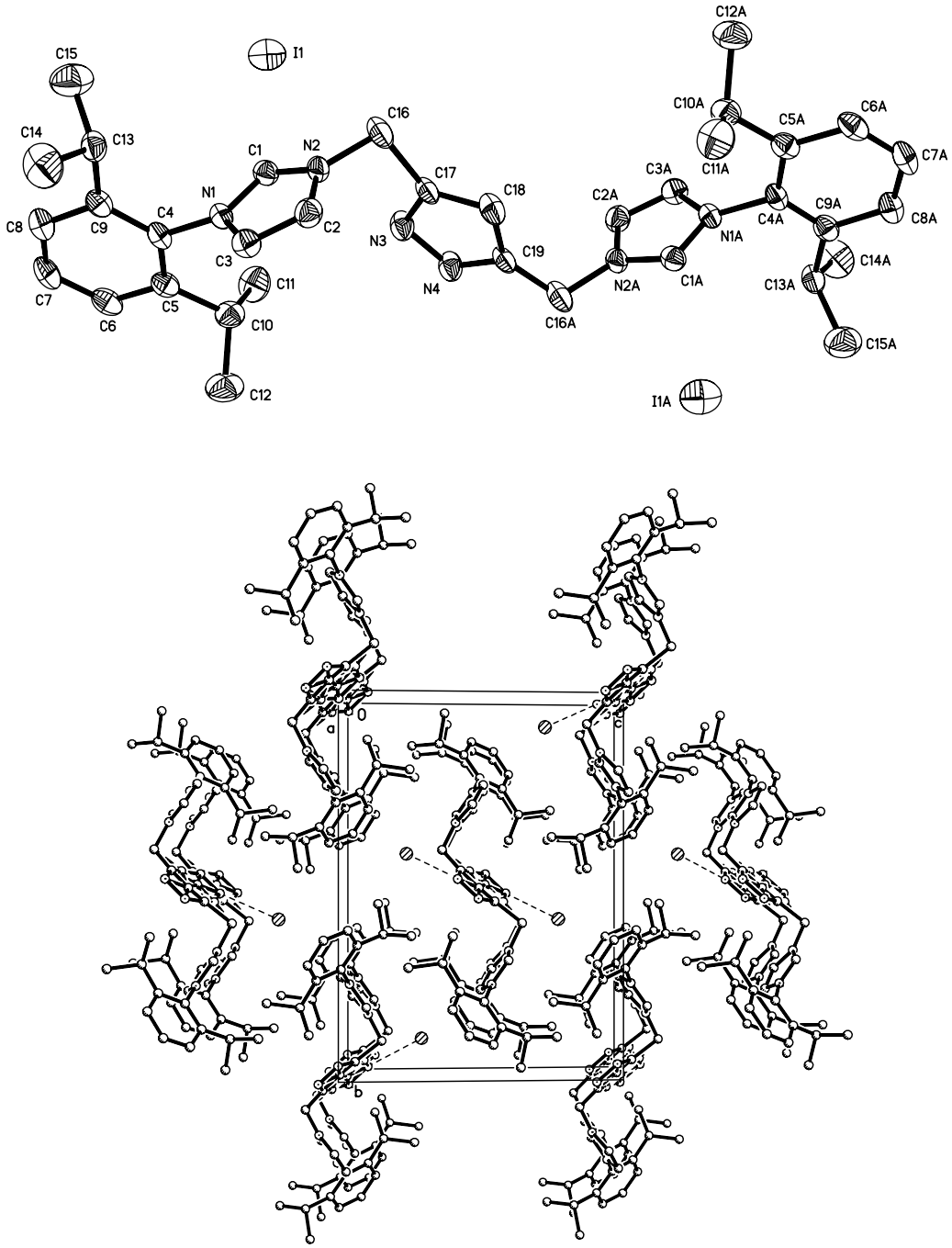


^{1}H and ^{13}C NMR of the Complex 5 (BisRh) in CD_2Cl_2



^{1}H and ^{13}C NMR of the Complex 6 (BisIr) in CD_2Cl_2

X-ray Structure of 2 (Iodide salt)



Electronic Supplementary Information for Dalton Transactions

Data collection

A crystal (approximate dimensions 0.40 x 0.20 x 0.15 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker SMART Platform CCD diffractometer for a data collection at 173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 114 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 30 seconds and a detector distance of 4.900 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.84 Å. Four major sections of frames were collected with 0.30º steps in ω at four different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from the xyz centroids of 4052 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-97 [4] and refined using SHELXL-97.[4] The space group $P2_1/n$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0457$ and $wR2 = 0.1231$ (F^2, all data).

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1 SMART V5.054, Bruker Analytical X-ray Systems, Madison, WI (2001).

2 An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

3 SAINT+ V6.45, Bruker Analytical X-Ray Systems, Madison, WI (2003).

4 SHELXTL V6.14, Bruker Analytical X-Ray Systems, Madison, WI (2000).

Tables for 2 (Iodide salt)

Table 1. Crystal data and structure refinement.

Identification code	07065	
Empirical formula	C_{35} H_{46} I_2 N_6	
Formula weight	804.58	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 7.7975(7) Å	α = 90°
	b = 17.8932(17) Å	β = 96.196(2)°
	c = 13.1964(13) Å	γ = 90°
Volume	1830.4(3) Å^3	
Z	2	
Density (calculated)	1.460 Mg/m^3	
Absorption coefficient	1.749 mm^{-1}	
F(000)	808	
Crystal color, morphology	colorless, rod	
Crystal size	0.40 x 0.20 x 0.15 mm^3	
Theta range for data collection	1.92 to 25.05°	
Index ranges	$-9 \le h \le 9$, $-21 \le k \le 21$, $-15 \le l \le 15$	
Reflections collected	14689	
Independent reflections	3247 [R(int) = 0.0259]	
Observed reflections	2974	
Completeness to theta = 25.05°	99.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7793 and 0.5413	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3247 / 15 / 209	
Goodness-of-fit on F^2	1.011	
Final R indices [I>2sigma(I)]	R1 = 0.0457, wR2 = 0.1201	
R indices (all data)	R1 = 0.0497, wR2 = 0.1231	
Largest diff. peak and hole	2.020 and -0.842 e.Å^{-3}	

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

	x	y	z	U_{eq}
I1	6393(1)	5868(1)	7757(1)	50(1)
N1	3165(4)	7467(2)	5329(3)	25(1)
N2	1552(5)	6642(2)	5934(3)	27(1)
C1	3171(6)	6861(2)	5915(3)	28(1)
C2	481(6)	7109(3)	5338(3)	33(1)
C3	1483(6)	7626(2)	4955(3)	31(1)
C4	4689(5)	7869(2)	5079(3)	26(1)
C5	5499(5)	7601(2)	4261(3)	30(1)
C6	6938(6)	8010(3)	4026(4)	35(1)
C7	7488(6)	8638(3)	4582(4)	37(1)
C8	6653(6)	8876(3)	5385(4)	36(1)
C9	5202(6)	8493(2)	5659(3)	30(1)
C10	4889(6)	6904(3)	3673(3)	35(1)
C11	6005(8)	6230(3)	4024(5)	52(1)
C12	4831(8)	7016(4)	2521(4)	53(1)
C13	4268(6)	8762(3)	6540(4)	34(1)
C14	3528(9)	9542(3)	6339(5)	60(2)
C15	5418(8)	8729(4)	7539(4)	60(2)
C16	1058(7)	5938(2)	6404(4)	39(1)
C17	731(11)	5387(7)	5445(7)	29(2)
C18	-713(10)	4957(5)	5102(7)	35(2)
C19	-230(11)	4596(7)	4254(7)	29(2)
N3	2035(9)	5312(4)	4882(5)	34(1)
N4	1404(8)	4815(4)	4161(5)	34(1)

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

N(1)-C(1)	1.332(5)	C(13)-C(15)	1.514(7)
N(1)-C(3)	1.380(6)	C(13)-C(14)	1.524(7)
N(1)-C(4)	1.457(5)	C(13)-H(13)	1.0000
N(2)-C(1)	1.324(6)	C(14)-H(14A)	0.9800
N(2)-C(2)	1.368(6)	C(14)-H(14B)	0.9800
N(2)-C(16)	1.473(5)	C(14)-H(14C)	0.9800
C(1)-H(1)	0.9500	C(15)-H(15A)	0.9800
C(2)-C(3)	1.344(6)	C(15)-H(15B)	0.9800
C(2)-H(2)	0.9500	C(15)-H(15C)	0.9800
C(3)-H(3)	0.9500	C(16)-C(19)#1	1.401(10)
C(4)-C(9)	1.387(6)	C(16)-C(17)	1.603(10)
C(4)-C(5)	1.393(6)	C(16)-H(16A)	0.9600
C(5)-C(6)	1.402(6)	C(16)-H(16B)	0.9600
C(5)-C(10)	1.518(6)	C(17)-N(3)	1.330(5)
C(6)-C(7)	1.383(7)	C(17)-C(18)	1.397(5)
C(6)-H(6)	0.9500	C(18)-C(19)	1.380(5)
C(7)-C(8)	1.370(7)	C(18)-H(18A)	0.9600
C(7)-H(7)	0.9500	C(19)-N(4)	1.351(5)
C(8)-C(9)	1.404(6)	C(19)-C(16)#1	1.401(10)
C(8)-H(8)	0.9500	N(3)-N(4)	1.355(5)
C(9)-C(13)	1.515(6)	N(4)-H(4A)	0.9001
C(10)-C(12)	1.529(7)		
C(10)-C(11)	1.530(7)	C(1)-N(1)-C(3)	108.5(4)
C(10)-H(10)	1.0000	C(1)-N(1)-C(4)	125.6(4)
C(11)-H(11A)	0.9800	C(3)-N(1)-C(4)	125.8(3)
C(11)-H(11B)	0.9800	C(1)-N(2)-C(2)	109.4(4)
C(11)-H(11C)	0.9800	C(1)-N(2)-C(16)	123.6(4)
C(12)-H(12A)	0.9800	C(2)-N(2)-C(16)	126.3(4)
C(12)-H(12B)	0.9800	N(2)-C(1)-N(1)	108.0(4)
C(12)-H(12C)	0.9800	N(2)-C(1)-H(1)	126.0

N(1)-C(1)-H(1)	126.0	H(11A)-C(11)-H(11B)	109.5
C(3)-C(2)-N(2)	107.0(4)	C(10)-C(11)-H(11C)	109.5
C(3)-C(2)-H(2)	126.5	H(11A)-C(11)-H(11C)	109.5
N(2)-C(2)-H(2)	126.5	H(11B)-C(11)-H(11C)	109.5
C(2)-C(3)-N(1)	107.1(4)	C(10)-C(12)-H(12A)	109.5
C(2)-C(3)-H(3)	126.5	C(10)-C(12)-H(12B)	109.5
N(1)-C(3)-H(3)	126.5	H(12A)-C(12)-H(12B)	109.5
C(9)-C(4)-C(5)	125.2(4)	C(10)-C(12)-H(12C)	109.5
C(9)-C(4)-N(1)	117.7(4)	H(12A)-C(12)-H(12C)	109.5
C(5)-C(4)-N(1)	117.0(4)	H(12B)-C(12)-H(12C)	109.5
C(4)-C(5)-C(6)	115.6(4)	C(15)-C(13)-C(9)	111.6(4)
C(4)-C(5)-C(10)	122.4(4)	C(15)-C(13)-C(14)	111.4(5)
C(6)-C(5)-C(10)	122.0(4)	C(9)-C(13)-C(14)	111.3(4)
C(7)-C(6)-C(5)	121.1(4)	C(15)-C(13)-H(13)	107.5
C(7)-C(6)-H(6)	119.4	C(9)-C(13)-H(13)	107.5
C(5)-C(6)-H(6)	119.4	C(14)-C(13)-H(13)	107.5
C(8)-C(7)-C(6)	121.1(4)	C(13)-C(14)-H(14A)	109.5
C(8)-C(7)-H(7)	119.4	C(13)-C(14)-H(14B)	109.5
C(6)-C(7)-H(7)	119.4	H(14A)-C(14)-H(14B)	109.5
C(7)-C(8)-C(9)	120.7(5)	C(13)-C(14)-H(14C)	109.5
C(7)-C(8)-H(8)	119.7	H(14A)-C(14)-H(14C)	109.5
C(9)-C(8)-H(8)	119.7	H(14B)-C(14)-H(14C)	109.5
C(4)-C(9)-C(8)	116.3(4)	C(13)-C(15)-H(15A)	109.5
C(4)-C(9)-C(13)	123.0(4)	C(13)-C(15)-H(15B)	109.5
C(8)-C(9)-C(13)	120.6(4)	H(15A)-C(15)-H(15B)	109.5
C(5)-C(10)-C(12)	112.0(4)	C(13)-C(15)-H(15C)	109.5
C(5)-C(10)-C(11)	110.8(4)	H(15A)-C(15)-H(15C)	109.5
C(12)-C(10)-C(11)	111.1(4)	H(15B)-C(15)-H(15C)	109.5
C(5)-C(10)-H(10)	107.6	C(19)#1-C(16)-N(2)	116.5(6)
C(12)-C(10)-H(10)	107.6	C(19)#1-C(16)-C(17)	21.2(3)
C(11)-C(10)-H(10)	107.6	N(2)-C(16)-C(17)	102.8(5)
C(10)-C(11)-H(11A)	109.5	C(19)#1-C(16)-H(16A)	98.1
C(10)-C(11)-H(11B)	109.5	N(2)-C(16)-H(16A)	109.7

C(17)-C(16)-H(16A)	118.9	C(17)-C(18)-H(18A)	128.9
C(19)#1-C(16)-H(16B)	113.0	N(4)-C(19)-C(18)	106.3(7)
N(2)-C(16)-H(16B)	110.3	N(4)-C(19)-C(16)#1	121.2(6)
C(17)-C(16)-H(16B)	106.6	C(18)-C(19)-C(16)#1	132.4(6)
H(16A)-C(16)-H(16B)	108.3	C(17)-N(3)-N(4)	102.5(7)
N(3)-C(17)-C(18)	113.7(7)	C(19)-N(4)-N(3)	113.7(7)
N(3)-C(17)-C(16)	115.9(6)	C(19)-N(4)-H(4A)	119.5
C(18)-C(17)-C(16)	130.4(6)	N(3)-N(4)-H(4A)	126.8
C(19)-C(18)-C(17)	103.9(7)		
C(19)-C(18)-H(18A)	127.1		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	45(1)	57(1)	46(1)	0(1)	5(1)	15(1)
N1	27(2)	22(2)	28(2)	2(1)	7(1)	-2(1)
N2	36(2)	19(2)	27(2)	-1(1)	6(2)	-6(1)
C1	32(2)	24(2)	27(2)	0(2)	6(2)	1(2)
C2	28(2)	36(2)	34(2)	-2(2)	2(2)	-3(2)
C3	29(2)	30(2)	33(2)	3(2)	2(2)	4(2)
C4	22(2)	25(2)	33(2)	9(2)	5(2)	-1(2)
C5	27(2)	33(2)	30(2)	6(2)	5(2)	5(2)
C6	31(2)	42(3)	35(2)	10(2)	13(2)	5(2)
C7	28(2)	35(2)	50(3)	15(2)	10(2)	-2(2)
C8	33(2)	30(2)	44(3)	5(2)	7(2)	-4(2)
C9	28(2)	28(2)	33(2)	7(2)	5(2)	2(2)
C10	34(2)	42(3)	31(2)	-4(2)	10(2)	0(2)
C11	58(3)	37(3)	61(3)	-9(3)	7(3)	6(2)
C12	53(3)	76(4)	34(3)	-6(3)	15(2)	-2(3)
C13	35(2)	29(2)	38(2)	-4(2)	13(2)	-7(2)
C14	69(4)	40(3)	74(4)	-5(3)	23(3)	15(3)
C15	53(3)	85(5)	42(3)	-4(3)	9(3)	0(3)
C16	47(3)	29(2)	43(3)	5(2)	18(2)	-7(2)
C17	37(5)	21(3)	31(6)	4(4)	14(3)	-4(4)
C18	40(4)	25(5)	44(5)	2(4)	15(4)	-3(4)
C19	37(5)	21(3)	31(6)	4(4)	14(3)	-4(4)
N3	38(3)	27(3)	39(3)	-8(2)	16(2)	-6(2)
N4	38(3)	27(3)	39(3)	-8(2)	16(2)	-6(2)

Table 5. Torsion angles [°].

C2-N2-C1-N1	0.9(5)	C4-C5-C10-C12	135.4(5)
C16-N2-C1-N1	172.4(4)	C6-C5-C10-C12	-45.7(6)
C3-N1-C1-N2	-1.0(5)	C4-C5-C10-C11	-100.0(5)
C4-N1-C1-N2	-177.8(4)	C6-C5-C10-C11	78.9(5)
C1-N2-C2-C3	-0.5(5)	C4-C9-C13-C15	117.3(5)
C16-N2-C2-C3	-171.7(4)	C8-C9-C13-C15	-63.1(6)
N2-C2-C3-N1	-0.2(5)	C4-C9-C13-C14	-117.6(5)
C1-N1-C3-C2	0.7(5)	C8-C9-C13-C14	61.9(6)
C4-N1-C3-C2	177.5(4)	C1-N2-C16-C19#1	-115.3(6)
C1-N1-C4-C9	-96.5(5)	C2-N2-C16-C19#1	54.7(7)
C3-N1-C4-C9	87.2(5)	C1-N2-C16-C17	-98.1(6)
C1-N1-C4-C5	84.6(5)	C2-N2-C16-C17	71.9(6)
C3-N1-C4-C5	-91.7(5)	C19#1-C16-C17-N3	-171(3)
C9-C4-C5-C6	-0.3(6)	N2-C16-C17-N3	55.6(11)
N1-C4-C5-C6	178.6(4)	C19#1-C16-C17-C18	7.8(17)
C9-C4-C5-C10	178.7(4)	N2-C16-C17-C18	-125.3(12)
N1-C4-C5-C10	-2.5(6)	N3-C17-C18-C19	-0.3(17)
C4-C5-C6-C7	-0.1(6)	C16-C17-C18-C19	-179.5(8)
C10-C5-C6-C7	-179.0(4)	C17-C18-C19-N4	0.9(16)
C5-C6-C7-C8	0.5(7)	C17-C18-C19-C16#1	176.5(9)
C6-C7-C8-C9	-0.6(7)	C18-C17-N3-N4	-0.4(13)
C5-C4-C9-C8	0.1(6)	C16-C17-N3-N4	179.0(8)
N1-C4-C9-C8	-178.7(4)	C18-C19-N4-N3	-1.2(13)
C5-C4-C9-C13	179.7(4)	C16#1-C19-N4-N3	-177.4(9)
N1-C4-C9-C13	0.9(6)	C17-N3-N4-C19	1.0(10)
C7-C8-C9-C4	0.3(7)		
C7-C8-C9-C13	-179.3(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

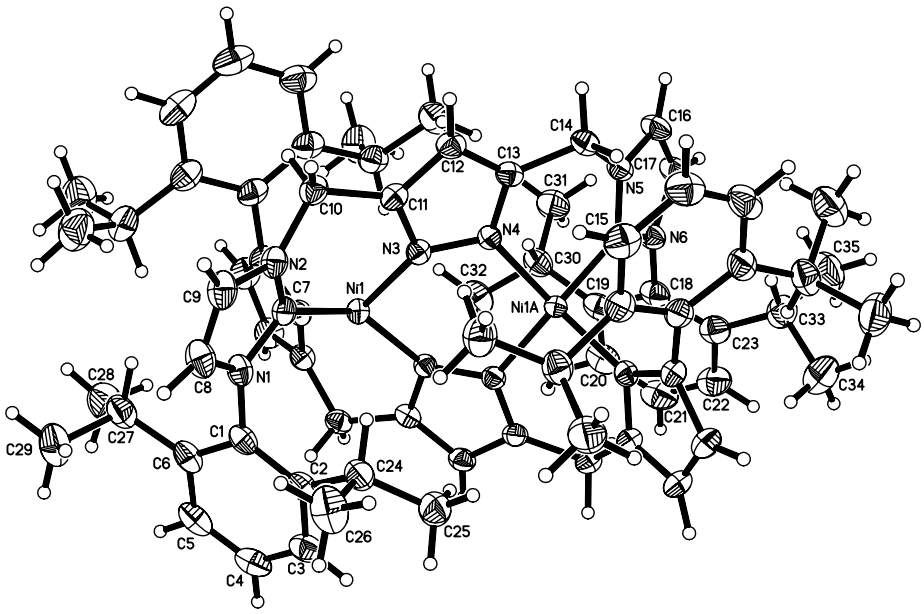
Table 6. Hydrogen bonds [Å and °].

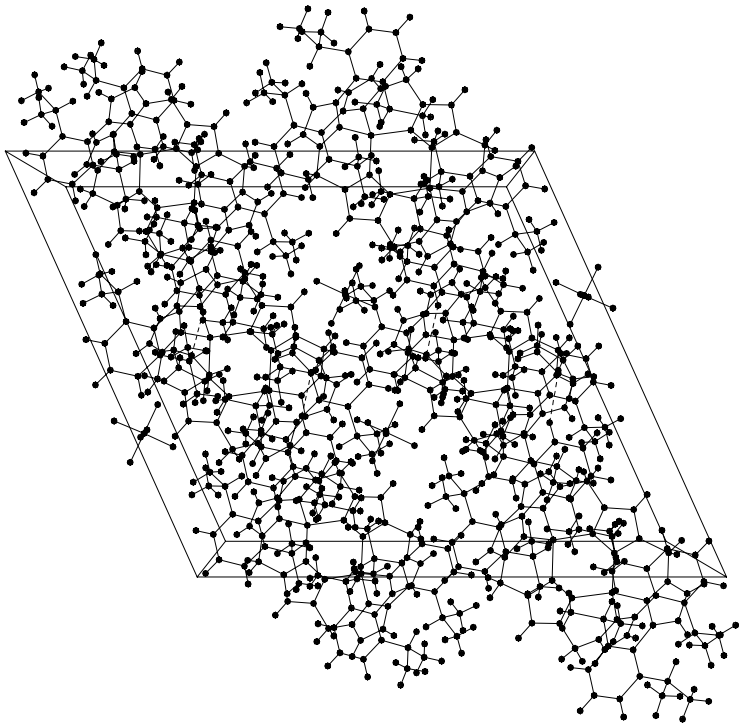
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C1-H1...I1	0.95	2.84	3.750(4)	161.9
N4-H4A...I1#2	0.90	2.53	3.434(6)	178.0

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 -x+1,-y+1,-z+1

X-ray Structure of 4 (Dimeric Ni complex)





Electronic Supplementary Information for Dalton Transactions

Data collection

A crystal (approximate dimensions 0.45 x 0.30 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a CCD area detector diffractometer for a data collection at 173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 182 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 30 seconds and a detector distance of 4.9 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.77 Å. Four major sections of frames were collected with 0.30º steps in ω at four different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from 2961 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using Bruker SHELXTL[4] and refined using Bruker SHELXTL.[4] The space group C2/c was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0481$ and $wR2 = 0.1379$ (F^2, all data).

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1 SMART V5.054, Bruker Analytical X-ray Systems, Madison, WI (2001).

2 An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

3 SAINT+ V6.45, Bruker Analytical X-Ray Systems, Madison, WI (2003).

4 SHELXTL V6.14, Bruker Analytical X-Ray Systems, Madison, WI (2000).

5 A. Altomare, M. C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna. Sir97: a new tool for crystal structure determination and refinement. *J. Appl. Cryst.* **32**, 115-119 (1998).

6 M. C. Burla, M. Camalli, B. Carrozzini, G. L. Cascarano, C. Giacovazzo, G. Polidori, R. Spagna. Sir2002: a new Direct Methods program for automatic solution and refinement of crystal structures. *J. Appl. Cryst.* (2003), in preparation.

7 A. L. Spek, *Acta. Cryst.* **A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht

University, Utrecht, The Netherlands, A. L. Spek (2000).

Tables for 4 (dimeric Ni complex)

Table 1. Crystal data and structure refinement.

Identification code	07006a	
Empirical formula	$C_{78}\ H_{102}\ F_{12}\ N_{12}\ Ni_2\ O_2\ P_2$	
Formula weight	1647.08	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 20.8499(18) Å	α = 90°
	b = 17.6914(15) Å	β = 114.251(1)°
	c = 23.633(2) Å	γ = 90°
Volume	7948.0(12) Å^3	
Z	4	
Density (calculated)	1.376 Mg/m^3	
Absorption coefficient	0.596 mm^{-1}	
F(000)	3456	
Crystal color, morphology	Red, Block	
Crystal size	0.45 x 0.30 x 0.08 mm^3	
Theta range for data collection	1.57 to 27.50°	
Index ranges	$-27 \le h \le 24$, $0 \le k \le 22$, $0 \le l \le 30$	
Reflections collected	37711	
Independent reflections	9020 [R(int) = 0.0334]	
Observed reflections	7198	
Completeness to theta = 27.50°	98.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9539 and 0.7753	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9020 / 4 / 495	
Goodness-of-fit on F^2	1.089	
Final R indices [I>2sigma(I)]	R1 = 0.0481, wR2 = 0.1299	
R indices (all data)	R1 = 0.0617, wR2 = 0.1379	
Largest diff. peak and hole	1.259 and -0.878 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	5652(1)	7631(1)	2152(1)	20(1)
N1	6318(1)	6502(1)	1704(1)	28(1)
N2	5297(1)	6817(1)	1014(1)	26(1)
N3	4663(1)	7497(1)	1760(1)	21(1)
N4	4205(1)	7744(1)	2005(1)	21(1)
N5	3081(1)	8464(1)	2196(1)	25(1)
N6	3603(1)	8817(1)	3149(1)	27(1)
C1	6900(1)	6401(1)	2309(1)	29(1)
C2	6764(1)	6041(1)	2776(1)	30(1)
C3	7316(1)	5988(2)	3365(1)	36(1)
C4	7977(1)	6254(2)	3470(1)	39(1)
C5	8111(1)	6562(2)	2995(1)	39(1)
C6	7575(1)	6646(1)	2401(1)	33(1)
C7	5784(1)	7013(1)	1576(1)	24(1)
C8	6149(1)	5996(2)	1211(1)	36(1)
C9	5516(1)	6192(2)	785(1)	34(1)
C10	4630(1)	7220(1)	706(1)	28(1)
C11	4297(1)	7422(1)	1142(1)	24(1)
C12	3598(1)	7612(1)	979(1)	26(1)
C13	3563(1)	7809(1)	1533(1)	23(1)
C14	2918(1)	8027(1)	1625(1)	26(1)
C15	3608(1)	8277(1)	2738(1)	24(1)
C16	2739(1)	9108(2)	2254(1)	33(1)
C17	3065(1)	9331(2)	2847(1)	36(1)
C18	4137(1)	8905(1)	3782(1)	30(1)
C19	4785(1)	9230(1)	3868(1)	32(1)
C20	5291(2)	9290(2)	4478(1)	41(1)
C21	5148(2)	9059(2)	4973(1)	47(1)
C22	4503(2)	8774(2)	4877(1)	44(1)
C23	3976(2)	8689(2)	4279(1)	35(1)

C24	6067(1)	5658(2)	2652(1)	33(1)
C25	5901(2)	5613(2)	3221(1)	42(1)
C26	6074(2)	4859(2)	2404(2)	51(1)
C27	7742(1)	6958(2)	1882(1)	39(1)
C28	8209(2)	7663(2)	2078(2)	52(1)
C29	8087(2)	6343(2)	1638(2)	54(1)
C30	4929(1)	9567(2)	3340(1)	34(1)
C31	4614(2)	10368(2)	3202(2)	47(1)
C32	5709(2)	9602(2)	3474(2)	43(1)
C33	3252(2)	8411(2)	4194(1)	41(1)
C34	3284(2)	7724(2)	4597(2)	57(1)
C35	2845(2)	9059(2)	4333(2)	63(1)
P1	6781(1)	4538(1)	221(1)	40(1)
F1	6458(2)	4465(2)	714(2)	139(2)
F2	7522(2)	4589(2)	778(1)	138(2)
F3	6846(1)	3637(1)	240(1)	61(1)
F4	6715(1)	5432(1)	212(1)	75(1)
F5	6039(1)	4462(1)	-335(1)	90(1)
F6	7125(2)	4593(2)	-249(1)	91(1)
O1	4769(2)	7653(2)	8720(2)	92(1)
C37	5297(2)	7439(3)	8510(2)	85(1)
C38	5460(3)	6651(3)	8620(3)	115(2)
C39	5101(4)	6398(3)	9032(3)	112(2)
C40	4627(3)	6998(3)	9011(3)	106(2)

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

Ni(1)-C(15)#1	1.851(2)	C(6)-C(27)	1.510(4)
Ni(1)-C(7)	1.851(2)	C(8)-C(9)	1.335(4)
Ni(1)-N(3)	1.8966(19)	C(8)-H(8A)	0.9500
Ni(1)-N(4)#1	1.9000(19)	C(9)-H(9A)	0.9500
N(1)-C(7)	1.368(3)	C(10)-C(11)	1.502(3)
N(1)-C(8)	1.395(3)	C(10)-H(10A)	0.9900
N(1)-C(1)	1.458(3)	C(10)-H(10B)	0.9900
N(2)-C(7)	1.345(3)	C(11)-C(12)	1.388(3)
N(2)-C(9)	1.387(3)	C(12)-C(13)	1.383(3)
N(2)-C(10)	1.463(3)	C(12)-H(12A)	0.9500
N(3)-C(11)	1.349(3)	C(13)-C(14)	1.500(3)
N(3)-N(4)	1.375(3)	C(14)-H(14A)	0.9900
N(4)-C(13)	1.350(3)	C(14)-H(14B)	0.9900
N(4)-Ni(1)#1	1.9000(19)	C(15)-Ni(1)#1	1.851(2)
N(5)-C(15)	1.342(3)	C(16)-C(17)	1.340(4)
N(5)-C(16)	1.381(3)	C(16)-H(16A)	0.9500
N(5)-C(14)	1.468(3)	C(17)-H(17A)	0.9500
N(6)-C(15)	1.365(3)	C(18)-C(23)	1.400(4)
N(6)-C(17)	1.393(3)	C(18)-C(19)	1.405(4)
N(6)-C(18)	1.462(3)	C(19)-C(20)	1.398(4)
C(1)-C(2)	1.401(4)	C(19)-C(30)	1.520(4)
C(1)-C(6)	1.403(3)	C(20)-C(21)	1.383(4)
C(2)-C(3)	1.398(4)	C(20)-H(20A)	0.9500
C(2)-C(24)	1.516(3)	C(21)-C(22)	1.364(5)
C(3)-C(4)	1.378(4)	C(21)-H(21A)	0.9500
C(3)-H(3A)	0.9500	C(22)-C(23)	1.396(4)
C(4)-C(5)	1.376(4)	C(22)-H(22A)	0.9500
C(4)-H(4B)	0.9500	C(23)-C(33)	1.520(4)
C(5)-C(6)	1.397(4)	C(24)-C(25)	1.523(4)
C(5)-H(5A)	0.9500	C(24)-C(26)	1.533(4)

C(24)-H(24A)	1.0000	C(33)-H(33A)	1.0000
C(25)-H(25A)	0.9800	C(34)-H(34A)	0.9800
C(25)-H(25B)	0.9800	C(34)-H(34B)	0.9800
C(25)-H(25C)	0.9800	C(34)-H(34C)	0.9800
C(26)-H(26A)	0.9800	C(35)-H(35A)	0.9800
C(26)-H(26B)	0.9800	C(35)-H(35B)	0.9800
C(26)-H(26C)	0.9800	C(35)-H(35C)	0.9800
C(27)-C(28)	1.533(4)	P(1)-F(6)	1.552(2)
C(27)-C(29)	1.541(4)	P(1)-F(2)	1.568(3)
C(27)-H(27A)	1.0000	P(1)-F(5)	1.572(2)
C(28)-H(28A)	0.9800	P(1)-F(1)	1.574(3)
C(28)-H(28B)	0.9800	P(1)-F(4)	1.587(2)
C(28)-H(28C)	0.9800	P(1)-F(3)	1.599(2)
C(29)-H(29A)	0.9800	O(1)-C(37)	1.432(4)
C(29)-H(29B)	0.9800	O(1)-C(40)	1.438(4)
C(29)-H(29C)	0.9800	C(37)-C(38)	1.435(5)
C(30)-C(32)	1.524(4)	C(37)-H(37A)	0.9900
C(30)-C(31)	1.539(4)	C(37)-H(37B)	0.9900
C(30)-H(30A)	1.0000	C(38)-C(39)	1.520(8)
C(31)-H(31A)	0.9800	C(38)-H(38A)	0.9900
C(31)-H(31B)	0.9800	C(38)-H(38B)	0.9900
C(31)-H(31C)	0.9800	C(39)-C(40)	1.438(5)
C(32)-H(32A)	0.9800	C(39)-H(39A)	0.9900
C(32)-H(32B)	0.9800	C(39)-H(39B)	0.9900
C(32)-H(32C)	0.9800	C(40)-H(40A)	0.9900
C(33)-C(34)	1.527(4)	C(40)-H(40B)	0.9900
C(33)-C(35)	1.539(4)		

C(15)#1-Ni(1)-C(7)	95.75(10)	N(3)-Ni(1)-N(4)#1	101.30(8)
C(15)#1-Ni(1)-N(3)	145.30(9)	C(7)-N(1)-C(8)	109.6(2)
C(7)-Ni(1)-N(3)	91.65(9)	C(7)-N(1)-C(1)	124.94(19)
C(15)#1-Ni(1)-N(4)#1	91.38(9)	C(8)-N(1)-C(1)	124.3(2)
C(7)-Ni(1)-N(4)#1	145.70(9)	C(7)-N(2)-C(9)	110.8(2)

C(7)-N(2)-C(10)	122.8(2)	N(2)-C(7)-Ni(1)	126.78(17)
C(9)-N(2)-C(10)	126.4(2)	N(1)-C(7)-Ni(1)	125.49(17)
C(11)-N(3)-N(4)	107.84(18)	C(9)-C(8)-N(1)	107.2(2)
C(11)-N(3)-Ni(1)	123.97(16)	C(9)-C(8)-H(8A)	126.4
N(4)-N(3)-Ni(1)	123.95(14)	N(1)-C(8)-H(8A)	126.4
C(13)-N(4)-N(3)	107.54(18)	C(8)-C(9)-N(2)	107.1(2)
C(13)-N(4)-Ni(1)#1	123.52(15)	C(8)-C(9)-H(9A)	126.5
N(3)-N(4)-Ni(1)#1	124.81(14)	N(2)-C(9)-H(9A)	126.5
C(15)-N(5)-C(16)	111.2(2)	N(2)-C(10)-C(11)	112.90(19)
C(15)-N(5)-C(14)	122.53(19)	N(2)-C(10)-H(10A)	109.0
C(16)-N(5)-C(14)	126.2(2)	C(11)-C(10)-H(10A)	109.0
C(15)-N(6)-C(17)	109.5(2)	N(2)-C(10)-H(10B)	109.0
C(15)-N(6)-C(18)	125.33(19)	C(11)-C(10)-H(10B)	109.0
C(17)-N(6)-C(18)	124.7(2)	H(10A)-C(10)-H(10B)	107.8
C(2)-C(1)-C(6)	122.3(2)	N(3)-C(11)-C(12)	109.7(2)
C(2)-C(1)-N(1)	118.2(2)	N(3)-C(11)-C(10)	123.4(2)
C(6)-C(1)-N(1)	119.5(2)	C(12)-C(11)-C(10)	126.7(2)
C(3)-C(2)-C(1)	117.6(2)	C(13)-C(12)-C(11)	104.8(2)
C(3)-C(2)-C(24)	119.7(2)	C(13)-C(12)-H(12A)	127.6
C(1)-C(2)-C(24)	122.5(2)	C(11)-C(12)-H(12A)	127.6
C(4)-C(3)-C(2)	120.7(3)	N(4)-C(13)-C(12)	110.0(2)
C(4)-C(3)-H(3A)	119.7	N(4)-C(13)-C(14)	123.0(2)
C(2)-C(3)-H(3A)	119.7	C(12)-C(13)-C(14)	126.9(2)
C(5)-C(4)-C(3)	120.8(3)	N(5)-C(14)-C(13)	112.68(18)
C(5)-C(4)-H(4B)	119.6	N(5)-C(14)-H(14A)	109.1
C(3)-C(4)-H(4B)	119.6	C(13)-C(14)-H(14A)	109.1
C(4)-C(5)-C(6)	121.0(2)	N(5)-C(14)-H(14B)	109.1
C(4)-C(5)-H(5A)	119.5	C(13)-C(14)-H(14B)	109.1
C(6)-C(5)-H(5A)	119.5	H(14A)-C(14)-H(14B)	107.8
C(5)-C(6)-C(1)	117.4(3)	N(5)-C(15)-N(6)	105.28(19)
C(5)-C(6)-C(27)	120.0(2)	N(5)-C(15)-Ni(1)#1	126.52(17)
C(1)-C(6)-C(27)	122.6(2)	N(6)-C(15)-Ni(1)#1	125.57(17)
N(2)-C(7)-N(1)	105.2(2)	C(17)-C(16)-N(5)	106.6(2)

C(17)-C(16)-H(16A)	126.7	H(25A)-C(25)-H(25C)	109.5
N(5)-C(16)-H(16A)	126.7	H(25B)-C(25)-H(25C)	109.5
C(16)-C(17)-N(6)	107.4(2)	C(24)-C(26)-H(26A)	109.5
C(16)-C(17)-H(17A)	126.3	C(24)-C(26)-H(26B)	109.5
N(6)-C(17)-H(17A)	126.3	H(26A)-C(26)-H(26B)	109.5
C(23)-C(18)-C(19)	122.6(2)	C(24)-C(26)-H(26C)	109.5
C(23)-C(18)-N(6)	119.1(2)	H(26A)-C(26)-H(26C)	109.5
C(19)-C(18)-N(6)	118.3(2)	H(26B)-C(26)-H(26C)	109.5
C(20)-C(19)-C(18)	117.0(3)	C(6)-C(27)-C(28)	112.4(3)
C(20)-C(19)-C(30)	119.8(3)	C(6)-C(27)-C(29)	110.2(2)
C(18)-C(19)-C(30)	123.0(2)	C(28)-C(27)-C(29)	110.6(2)
C(21)-C(20)-C(19)	121.0(3)	C(6)-C(27)-H(27A)	107.8
C(21)-C(20)-H(20A)	119.5	C(28)-C(27)-H(27A)	107.8
C(19)-C(20)-H(20A)	119.5	C(29)-C(27)-H(27A)	107.8
C(22)-C(21)-C(20)	120.6(3)	C(27)-C(28)-H(28A)	109.5
C(22)-C(21)-H(21A)	119.7	C(27)-C(28)-H(28B)	109.5
C(20)-C(21)-H(21A)	119.7	H(28A)-C(28)-H(28B)	109.5
C(21)-C(22)-C(23)	121.4(3)	C(27)-C(28)-H(28C)	109.5
C(21)-C(22)-H(22A)	119.3	H(28A)-C(28)-H(28C)	109.5
C(23)-C(22)-H(22A)	119.3	H(28B)-C(28)-H(28C)	109.5
C(22)-C(23)-C(18)	117.3(3)	C(27)-C(29)-H(29A)	109.5
C(22)-C(23)-C(33)	119.7(3)	C(27)-C(29)-H(29B)	109.5
C(18)-C(23)-C(33)	123.0(2)	H(29A)-C(29)-H(29B)	109.5
C(2)-C(24)-C(25)	113.5(2)	C(27)-C(29)-H(29C)	109.5
C(2)-C(24)-C(26)	109.1(2)	H(29A)-C(29)-H(29C)	109.5
C(25)-C(24)-C(26)	109.2(2)	H(29B)-C(29)-H(29C)	109.5
C(2)-C(24)-H(24A)	108.3	C(19)-C(30)-C(32)	113.5(2)
C(25)-C(24)-H(24A)	108.3	C(19)-C(30)-C(31)	108.9(2)
C(26)-C(24)-H(24A)	108.3	C(32)-C(30)-C(31)	109.4(2)
C(24)-C(25)-H(25A)	109.5	C(19)-C(30)-H(30A)	108.3
C(24)-C(25)-H(25B)	109.5	C(32)-C(30)-H(30A)	108.3
H(25A)-C(25)-H(25B)	109.5	C(31)-C(30)-H(30A)	108.3
C(24)-C(25)-H(25C)	109.5	C(30)-C(31)-H(31A)	109.5

C(30)-C(31)-H(31B)	109.5	F(2)-P(1)-F(1)	87.5(2)
H(31A)-C(31)-H(31B)	109.5	F(5)-P(1)-F(1)	92.1(2)
C(30)-C(31)-H(31C)	109.5	F(6)-P(1)-F(4)	89.45(14)
H(31A)-C(31)-H(31C)	109.5	F(2)-P(1)-F(4)	90.18(15)
H(31B)-C(31)-H(31C)	109.5	F(5)-P(1)-F(4)	91.32(14)
C(30)-C(32)-H(32A)	109.5	F(1)-P(1)-F(4)	91.73(14)
C(30)-C(32)-H(32B)	109.5	F(6)-P(1)-F(3)	91.39(14)
H(32A)-C(32)-H(32B)	109.5	F(2)-P(1)-F(3)	89.57(13)
C(30)-C(32)-H(32C)	109.5	F(5)-P(1)-F(3)	88.94(12)
H(32A)-C(32)-H(32C)	109.5	F(1)-P(1)-F(3)	87.41(14)
H(32B)-C(32)-H(32C)	109.5	F(4)-P(1)-F(3)	179.12(13)
C(23)-C(33)-C(34)	112.8(3)	C(37)-O(1)-C(40)	107.0(4)
C(23)-C(33)-C(35)	109.6(3)	O(1)-C(37)-C(38)	110.6(3)
C(34)-C(33)-C(35)	110.3(3)	O(1)-C(37)-H(37A)	109.5
C(23)-C(33)-H(33A)	108.0	C(38)-C(37)-H(37A)	109.5
C(34)-C(33)-H(33A)	108.0	O(1)-C(37)-H(37B)	109.5
C(35)-C(33)-H(33A)	108.0	C(38)-C(37)-H(37B)	109.5
C(33)-C(34)-H(34A)	109.5	H(37A)-C(37)-H(37B)	108.1
C(33)-C(34)-H(34B)	109.5	C(37)-C(38)-C(39)	105.1(3)
H(34A)-C(34)-H(34B)	109.5	C(37)-C(38)-H(38A)	110.7
C(33)-C(34)-H(34C)	109.5	C(39)-C(38)-H(38A)	110.7
H(34A)-C(34)-H(34C)	109.5	C(37)-C(38)-H(38B)	110.7
H(34B)-C(34)-H(34C)	109.5	C(39)-C(38)-H(38B)	110.7
C(33)-C(35)-H(35A)	109.5	H(38A)-C(38)-H(38B)	108.8
C(33)-C(35)-H(35B)	109.5	C(40)-C(39)-C(38)	105.8(3)
H(35A)-C(35)-H(35B)	109.5	C(40)-C(39)-H(39A)	110.6
C(33)-C(35)-H(35C)	109.5	C(38)-C(39)-H(39A)	110.6
H(35A)-C(35)-H(35C)	109.5	C(40)-C(39)-H(39B)	110.6
H(35B)-C(35)-H(35C)	109.5	C(38)-C(39)-H(39B)	110.6
F(6)-P(1)-F(2)	90.7(2)	H(39A)-C(39)-H(39B)	108.7
F(6)-P(1)-F(5)	89.62(17)	C(39)-C(40)-O(1)	109.8(3)
F(2)-P(1)-F(5)	178.47(14)	C(39)-C(40)-H(40A)	109.7
F(6)-P(1)-F(1)	177.9(2)	O(1)-C(40)-H(40A)	109.7

C(39)-C(40)-H(40B)	109.7	H(40A)-C(40)-H(40B)	108.2
O(1)-C(40)-H(40B)	109.7		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni1	19(1)	19(1)	21(1)	-3(1)	9(1)	-3(1)
N1	27(1)	28(1)	30(1)	-6(1)	15(1)	-1(1)
N2	28(1)	28(1)	25(1)	-6(1)	13(1)	-4(1)
N3	21(1)	21(1)	22(1)	0(1)	9(1)	-2(1)
N4	19(1)	22(1)	21(1)	1(1)	8(1)	0(1)
N5	21(1)	25(1)	28(1)	2(1)	9(1)	3(1)
N6	27(1)	26(1)	28(1)	-1(1)	11(1)	5(1)
C1	26(1)	24(1)	35(1)	-6(1)	13(1)	4(1)
C2	29(1)	25(1)	37(1)	-3(1)	14(1)	5(1)
C3	37(1)	29(1)	38(1)	0(1)	12(1)	9(1)
C4	31(1)	36(1)	41(2)	-2(1)	4(1)	10(1)
C5	25(1)	34(1)	56(2)	-6(1)	13(1)	4(1)
C6	28(1)	26(1)	45(2)	-5(1)	17(1)	3(1)
C7	25(1)	24(1)	26(1)	-1(1)	14(1)	-3(1)
C8	39(1)	34(1)	40(2)	-13(1)	20(1)	0(1)
C9	40(1)	36(1)	32(1)	-14(1)	19(1)	-5(1)
C10	29(1)	32(1)	22(1)	-3(1)	10(1)	-3(1)
C11	25(1)	24(1)	21(1)	-1(1)	8(1)	-5(1)
C12	22(1)	30(1)	23(1)	0(1)	4(1)	-4(1)
C13	21(1)	21(1)	25(1)	2(1)	7(1)	-2(1)
C14	20(1)	28(1)	27(1)	0(1)	6(1)	0(1)
C15	23(1)	22(1)	27(1)	1(1)	12(1)	1(1)
C16	28(1)	31(1)	38(1)	4(1)	11(1)	11(1)
C17	33(1)	32(1)	43(2)	0(1)	15(1)	13(1)
C18	33(1)	26(1)	30(1)	-5(1)	11(1)	8(1)
C19	33(1)	26(1)	35(1)	-6(1)	11(1)	7(1)
C20	37(1)	39(2)	40(2)	-11(1)	8(1)	4(1)
C21	48(2)	50(2)	31(1)	-10(1)	5(1)	11(1)
C22	59(2)	45(2)	30(1)	-2(1)	18(1)	14(1)

C23	42(1)	30(1)	33(1)	-2(1)	16(1)	9(1)
C24	30(1)	33(1)	39(1)	2(1)	16(1)	3(1)
C25	45(2)	37(2)	49(2)	4(1)	26(1)	6(1)
C26	52(2)	39(2)	69(2)	-16(2)	32(2)	-10(1)
C27	29(1)	40(2)	51(2)	2(1)	20(1)	3(1)
C28	41(2)	48(2)	72(2)	2(2)	26(2)	-7(1)
C29	55(2)	57(2)	62(2)	-7(2)	36(2)	6(2)
C30	32(1)	31(1)	37(1)	-5(1)	13(1)	3(1)
C31	50(2)	38(2)	57(2)	8(1)	25(2)	13(1)
C32	37(1)	34(1)	58(2)	-7(1)	20(1)	-1(1)
C33	49(2)	42(2)	39(2)	-1(1)	25(1)	4(1)
C34	71(2)	57(2)	49(2)	6(2)	33(2)	-2(2)
C35	60(2)	62(2)	79(3)	-3(2)	41(2)	13(2)
P1	58(1)	34(1)	29(1)	-3(1)	17(1)	-3(1)
F1	293(5)	64(2)	158(3)	-7(2)	191(4)	-3(2)
F2	156(3)	70(2)	85(2)	13(1)	-55(2)	-50(2)
F3	68(1)	37(1)	54(1)	-6(1)	0(1)	1(1)
F4	118(2)	33(1)	80(2)	1(1)	49(2)	0(1)
F5	52(1)	65(2)	115(2)	28(1)	-5(1)	-3(1)
F6	112(2)	87(2)	112(2)	-11(2)	85(2)	-14(2)
O1	84(2)	102(3)	104(3)	31(2)	52(2)	19(2)
C37	70(3)	123(4)	73(3)	9(3)	40(2)	-5(3)
C38	131(5)	70(3)	177(6)	-19(4)	97(5)	-16(3)
C39	165(6)	77(3)	84(4)	-1(3)	41(4)	-21(4)
C40	101(4)	127(5)	125(5)	47(4)	83(4)	31(4)

Table 5. Torsion angles [°].

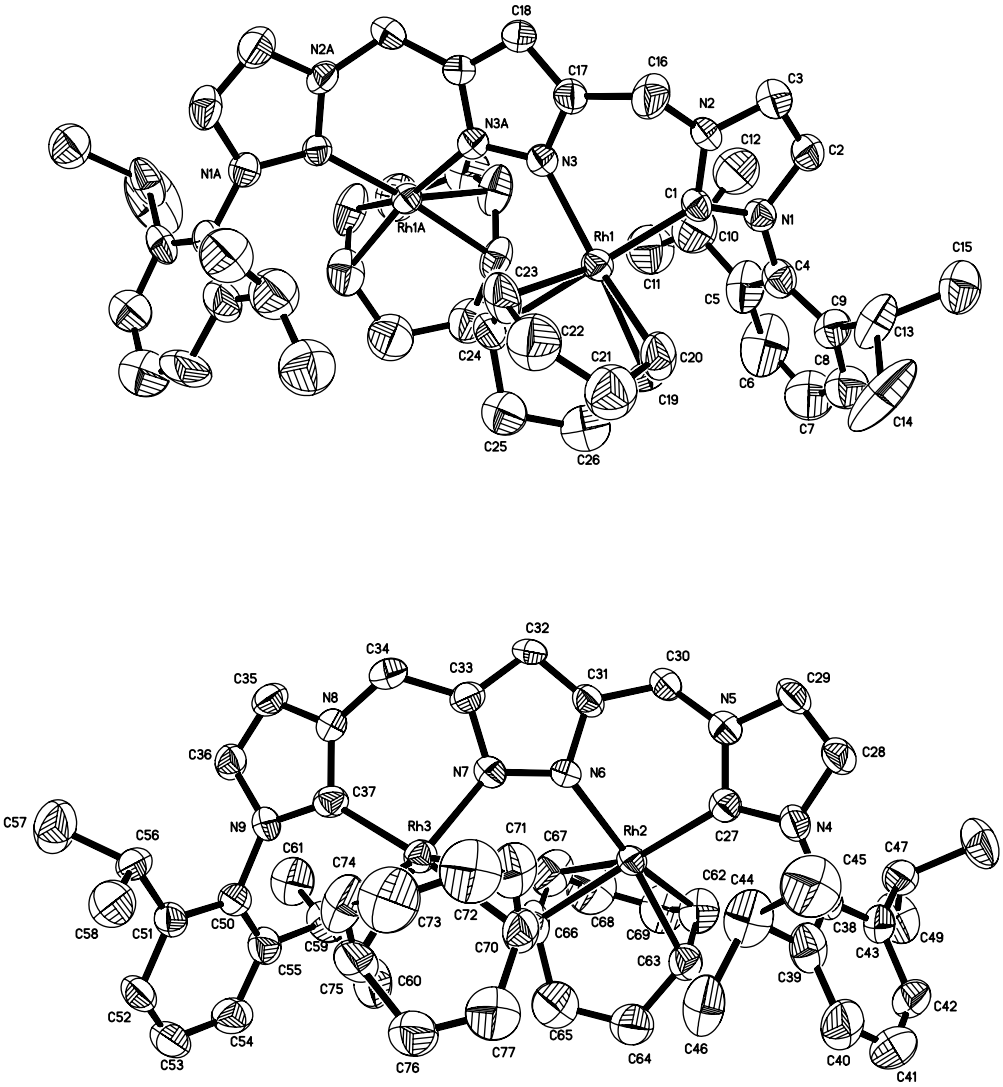
C15#1-Ni1-N3-C11	67.9(2)	C10-N2-C7-Ni1	16.4(3)
C7-Ni1-N3-C11	-34.68(19)	C8-N1-C7-N2	0.3(3)
N4#1-Ni1-N3-C11	177.27(17)	C1-N1-C7-N2	-167.9(2)
C15#1-Ni1-N3-N4	-86.1(2)	C8-N1-C7-Ni1	163.25(18)
C7-Ni1-N3-N4	171.29(17)	C1-N1-C7-Ni1	-4.9(3)
N4#1-Ni1-N3-N4	23.24(15)	C15#1-Ni1-C7-N2	-127.6(2)
C11-N3-N4-C13	0.5(2)	N3-Ni1-C7-N2	18.5(2)
Ni1-N3-N4-C13	158.11(15)	N4#1-Ni1-C7-N2	131.4(2)
C11-N3-N4-Ni1#1	158.26(15)	C15#1-Ni1-C7-N1	73.1(2)
Ni1-N3-N4-Ni1#1	-44.2(2)	N3-Ni1-C7-N1	-140.9(2)
C7-N1-C1-C2	73.5(3)	N4#1-Ni1-C7-N1	-27.9(3)
C8-N1-C1-C2	-92.9(3)	C7-N1-C8-C9	-0.2(3)
C7-N1-C1-C6	-108.0(3)	C1-N1-C8-C9	168.0(2)
C8-N1-C1-C6	85.6(3)	N1-C8-C9-N2	0.1(3)
C6-C1-C2-C3	5.4(4)	C7-N2-C9-C8	0.0(3)
N1-C1-C2-C3	-176.1(2)	C10-N2-C9-C8	-179.2(2)
C6-C1-C2-C24	-169.6(2)	C7-N2-C10-C11	-42.0(3)
N1-C1-C2-C24	8.9(3)	C9-N2-C10-C11	137.2(2)
C1-C2-C3-C4	-2.8(4)	N4-N3-C11-C12	-0.5(2)
C24-C2-C3-C4	172.4(2)	Ni1-N3-C11-C12	-158.08(16)
C2-C3-C4-C5	-1.4(4)	N4-N3-C11-C10	175.1(2)
C3-C4-C5-C6	3.0(4)	Ni1-N3-C11-C10	17.5(3)
C4-C5-C6-C1	-0.4(4)	N2-C10-C11-N3	24.0(3)
C4-C5-C6-C27	-177.6(2)	N2-C10-C11-C12	-161.2(2)
C2-C1-C6-C5	-3.9(4)	N3-C11-C12-C13	0.3(3)
N1-C1-C6-C5	177.7(2)	C10-C11-C12-C13	-175.1(2)
C2-C1-C6-C27	173.2(2)	N3-N4-C13-C12	-0.4(2)
N1-C1-C6-C27	-5.2(4)	Ni1#1-N4-C13-C12	-158.45(16)
C9-N2-C7-N1	-0.2(3)	N3-N4-C13-C14	176.7(2)
C10-N2-C7-N1	179.1(2)	Ni1#1-N4-C13-C14	18.7(3)
C9-N2-C7-Ni1	-162.88(18)	C11-C12-C13-N4	0.1(3)

C11-C12-C13-C14	-176.9(2)	C20-C21-C22-C23	1.5(5)
C15-N5-C14-C13	-43.6(3)	C21-C22-C23-C18	0.3(4)
C16-N5-C14-C13	135.4(2)	C21-C22-C23-C33	-176.5(3)
N4-C13-C14-N5	24.7(3)	C19-C18-C23-C22	-3.0(4)
C12-C13-C14-N5	-158.7(2)	N6-C18-C23-C22	179.2(2)
C16-N5-C15-N6	-0.3(3)	C19-C18-C23-C33	173.7(2)
C14-N5-C15-N6	178.8(2)	N6-C18-C23-C33	-4.1(4)
C16-N5-C15-Ni1#1	-162.60(18)	C3-C2-C24-C25	30.4(3)
C14-N5-C15-Ni1#1	16.5(3)	C1-C2-C24-C25	-154.7(2)
C17-N6-C15-N5	0.0(3)	C3-C2-C24-C26	-91.7(3)
C18-N6-C15-N5	-172.0(2)	C1-C2-C24-C26	83.2(3)
C17-N6-C15-Ni1#1	162.56(18)	C5-C6-C27-C28	-46.3(3)
C18-N6-C15-Ni1#1	-9.5(3)	C1-C6-C27-C28	136.7(3)
C15-N5-C16-C17	0.4(3)	C5-C6-C27-C29	77.5(3)
C14-N5-C16-C17	-178.6(2)	C1-C6-C27-C29	-99.5(3)
N5-C16-C17-N6	-0.4(3)	C20-C19-C30-C32	27.8(3)
C15-N6-C17-C16	0.2(3)	C18-C19-C30-C32	-157.4(2)
C18-N6-C17-C16	172.3(2)	C20-C19-C30-C31	-94.4(3)
C15-N6-C18-C23	-106.2(3)	C18-C19-C30-C31	80.4(3)
C17-N6-C18-C23	82.9(3)	C22-C23-C33-C34	-46.6(4)
C15-N6-C18-C19	76.0(3)	C18-C23-C33-C34	136.8(3)
C17-N6-C18-C19	-94.9(3)	C22-C23-C33-C35	76.7(3)
C23-C18-C19-C20	3.8(4)	C18-C23-C33-C35	-99.9(3)
N6-C18-C19-C20	-178.5(2)	C40-O1-C37-C38	4.2(7)
C23-C18-C19-C30	-171.1(2)	O1-C37-C38-C39	-10.8(7)
N6-C18-C19-C30	6.6(3)	C37-C38-C39-C40	13.2(7)
C18-C19-C20-C21	-1.9(4)	C38-C39-C40-O1	-11.2(7)
C30-C19-C20-C21	173.2(3)	C37-O1-C40-C39	4.7(7)
C19-C20-C21-C22	-0.7(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2

X-ray Structure of 5 (Bisrhodium Complex)



Data collection

A crystal (approximate dimensions 0.50 x 0.50 x 0.40 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Siemens SMART Platform CCD diffractometer for a data collection at 173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 233 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 30 seconds and a detector distance of 4.957 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.84 Å. Four major sections of frames were collected with 0.30º steps in ω at four different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from the xyz centroids of 3958 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-97 [4] and refined using SHELXL-97.[4] The space group *C*2 was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The function SQUEEZE in the program PLATON[5] was used to model solvent accessible void space. A void space of 2332.0 $Å^3$, containing 773 electrons per cell, was found. The final full matrix least squares refinement converged to $R1 = 0.0397$ and $wR2 = 0.1047$ (F^2, all data).

Structure description

The sample is structurally analogous to the Ir sample. The sample consists of one Rh dimer, with a PF_6^- counterion, on general positions, and a second dimer, with a PF_6^- counterion, on different 2-fold axis. The PF_6^- counterion on the 2-fold axis (P2-F12) was modeled as disordered over two positions (91:9), as well as over the 2-fold axis. Two isopropyl groups were modeled as disordered over two positions: C10-C12 (78:22) and C44-C46 (72:28). An ISOR restraint (restrains the U_{ij}'s of the atoms to be more isotropic in character) was used to help model the displacement parameters on the isopropyl group C10-C12. A DELU restraint (forces the atoms to have displacement parameters in the same direction) was also used to help model the displacement parameters. An ISOR restraint was used to help model the displacement parameters for all of the coordinated COD ligands. A DELU restraint was also used to help model the displacement parameters of the COD ligands. While the COD ligands appear to be disordered, no disorder model could be found that would make sense structurally and chemically (thus the use of the restraints). As stated earlier, the SQUEEZE function removed 2332.0 $Å^3$ of solvent accessible void space, containing 773 electrons per cell. This corresponds to roughly 4.5 CH_2Cl_2 molecules per asymmetric unit (4 CH_2Cl_2 molecules per asymmetric unit would have 672electrons and 5 per asymmetric unit would have 840 electrons).

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1 SMART V5.054, Bruker Analytical X-ray Systems, Madison, WI (2001).

2 An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

3 SAINT+ V6.45, Bruker Analytical X-Ray Systems, Madison, WI (2003).

4 SHELXTL V6.14, Bruker Analytical X-Ray Systems, Madison, WI (2000).

5 A. L. Spek, *Acta. Cryst.* **A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht

University, Utrecht, The Netherlands, A. L. Spek (2000).

Tables for 5 (Bisrhodium complex)

Table 1. Crystal data and structure refinement.

Identification code	07066s	
Empirical formula	C_{51} H_{67} F_6 N_6 P Rh_2	
Formula weight	1114.90	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*C*2	
Unit cell dimensions	*a* = 30.849(4) Å	α = 90°
	b = 18.153(2) Å	β = 92.222(2)°
	c = 16.475(2) Å	γ = 90°
Volume	9219.1(19) Å^3	
Z	6	
Density (calculated)	1.205 Mg/m^3	
Absorption coefficient	0.615 mm^{-1}	
F(000)	3444	
Crystal color, morphology	yellow, block	
Crystal size	0.50 x 0.50 x 0.40 mm^3	
Theta range for data collection	1.24 to 25.04°	
Index ranges	$-36 \le h \le 36$, $-21 \le k \le 21$, $-19 \le l \le 19$	
Reflections collected	45510	
Independent reflections	16350 [*R*(int) = 0.0311]	
Observed reflections	15285	
Completeness to theta = 25.04°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7911 and 0.7486	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	16350 / 347 / 959	
Goodness-of-fit on F^2	1.026	
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0397, *wR*2 = 0.1027	
R indices (all data)	*R*1 = 0.0425, *wR*2 = 0.1047	
Absolute structure parameter	0.06(2)	

Largest diff. peak and hole	0.736 and -1.122 e.Å^{-3}

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

	x	y	z	U_{eq}
Rh1	4575(1)	2040(1)	5977(1)	32(1)
Rh2	1922(1)	1696(1)	7620(1)	31(1)
Rh3	1549(1)	2898(1)	9667(1)	33(1)
P1	4615(1)	5973(1)	6484(1)	39(1)
F1	4852(2)	6412(3)	7204(3)	105(2)
F2	4827(2)	6459(2)	5825(3)	106(2)
F3	4360(1)	5494(2)	5807(2)	71(1)
F4	4384(2)	5471(2)	7136(3)	83(1)
F5	5009(1)	5415(2)	6431(3)	73(1)
F6	4228(1)	6531(2)	6527(2)	72(1)
P2	5025(2)	4705(1)	9933(4)	45(1)
F7	5257(3)	4708(4)	9098(5)	70(2)
F8	5428(3)	5109(6)	10349(6)	81(5)
F9	4814(4)	5476(3)	9718(7)	86(4)
F10	4620(3)	4307(6)	9515(5)	100(6)
F11	5232(4)	3933(4)	10143(5)	84(4)
F12	4792(3)	4705(5)	10766(5)	70(2)
P2'	5025(2)	4705(1)	9933(4)	45(1)
F7'	5310(20)	4670(50)	9160(30)	70(2)
F8'	5080(30)	5568(7)	9960(60)	81(5)
F9'	4604(17)	4800(60)	9370(40)	86(4)
F10'	4970(30)	3843(8)	9910(60)	100(6)
F11'	5445(17)	4610(60)	10500(50)	84(4)
F12'	4740(20)	4740(50)	10700(30)	70(2)
N1	4936(1)	2446(2)	7786(2)	36(1)
N2	4748(2)	3348(2)	7012(2)	42(1)
N3	4896(1)	2872(2)	5356(2)	35(1)
N4	1557(1)	2120(3)	5834(2)	38(1)
N5	1145(1)	1462(2)	6545(2)	37(1)
N6	1347(1)	1775(2)	8246(2)	34(1)

N7	1275(1)	2011(2)	9014(2)	36(1)
N8	1003(1)	1944(2)	10668(2)	38(1)
N9	1551(1)	2204(2)	11452(2)	36(1)
C1	4771(2)	2604(3)	7019(3)	31(1)
C2	5003(2)	3092(3)	8233(3)	49(1)
C3	4886(2)	3675(4)	7736(3)	54(1)
C4	5010(2)	1738(3)	8132(3)	45(1)
C5	5385(2)	1364(4)	7937(4)	65(2)
C6	5450(3)	674(5)	8288(5)	98(3)
C7	5159(3)	359(5)	8751(5)	87(2)
C8	4801(2)	755(4)	8966(4)	64(2)
C9	4714(2)	1445(3)	8659(3)	46(1)
C10	5736(2)	1729(4)	7408(4)	78(2)
C11	6063(4)	1254(7)	7035(7)	94(2)
C12	5995(4)	2263(7)	7905(6)	82(2)
C10'	5736(2)	1729(4)	7408(4)	78(2)
C11'	5868(13)	1026(12)	7030(20)	94(2)
C12'	6060(8)	1840(20)	8080(14)	82(2)
C13	4307(2)	1846(4)	8882(3)	60(2)
C14	3896(3)	1397(8)	8847(7)	139(5)
C15	4372(2)	2214(4)	9751(4)	71(2)
C16	4582(3)	3769(3)	6325(3)	62(2)
C17	4832(2)	3575(3)	5567(3)	42(1)
C18	5000	4047(4)	5000	56(2)
C19	4385(2)	1052(4)	6580(4)	63(2)
C20	4042(2)	1533(4)	6551(4)	63(2)
C21	3634(3)	1451(5)	5972(5)	82(2)
C22	3695(3)	1846(5)	5173(5)	85(2)
C23	4145(2)	1934(4)	4915(3)	60(2)
C24	4440(2)	1365(3)	4887(3)	51(1)
C25	4357(3)	579(4)	5107(4)	71(2)
C26	4451(3)	396(5)	6017(5)	81(2)
C27	1539(2)	1795(3)	6570(3)	34(1)
C28	1183(2)	1983(3)	5358(3)	44(1)
C29	926(2)	1560(3)	5809(3)	45(1)

C30	990(2)	988(3)	7189(3)	42(1)
C31	999(2)	1366(3)	7994(3)	35(1)
C32	704(2)	1336(3)	8580(3)	39(1)
C33	885(1)	1746(3)	9222(3)	39(1)
C34	690(2)	1947(4)	9991(3)	49(1)
C35	937(2)	1573(3)	11402(3)	43(1)
C36	1284(2)	1744(3)	11884(3)	41(1)
C37	1373(2)	2333(3)	10682(3)	36(1)
C38	1923(2)	2520(3)	5519(3)	40(1)
C39	1981(2)	3271(3)	5724(3)	53(2)
C40	2337(3)	3622(4)	5406(4)	72(2)
C41	2607(3)	3256(4)	4891(5)	70(2)
C42	2537(2)	2543(4)	4674(4)	55(2)
C43	2182(2)	2153(3)	4986(3)	42(1)
C44	1683(2)	3657(3)	6289(4)	65(2)
C45	1361(13)	4085(17)	5664(19)	95(3)
C46	2045(10)	4162(18)	6740(20)	70(3)
C44'	1683(2)	3657(3)	6289(4)	65(2)
C45'	1224(4)	3841(11)	5838(9)	95(3)
C46'	1895(6)	4337(6)	6785(8)	70(3)
C47	2123(2)	1360(3)	4731(3)	47(1)
C48	1909(2)	1326(4)	3873(4)	68(2)
C49	2529(3)	918(4)	4760(5)	72(2)
C50	1941(2)	2532(3)	11820(3)	35(1)
C51	1886(2)	3132(3)	12343(3)	39(1)
C52	2263(2)	3433(3)	12714(3)	49(1)
C53	2658(2)	3161(4)	12565(4)	59(2)
C54	2706(2)	2550(4)	12053(4)	54(1)
C55	2342(2)	2219(3)	11672(3)	45(1)
C56	1445(2)	3447(3)	12509(3)	47(1)
C57	1285(3)	3216(5)	13359(4)	84(2)
C58	1429(2)	4292(4)	12475(5)	69(2)
C59	2393(2)	1538(3)	11146(4)	52(1)
C60	2810(2)	1541(5)	10697(4)	77(2)
C61	2328(3)	851(4)	11660(5)	69(2)

C62	2412(2)	1173(4)	6965(4)	55(1)
C63	2531(2)	1883(3)	7091(3)	47(1)
C64	2870(2)	2146(4)	7704(4)	65(2)
C65	2780(3)	2034(5)	8549(5)	83(2)
C66	2329(2)	1718(5)	8740(3)	63(2)
C67	2203(2)	1037(5)	8601(4)	68(2)
C68	2485(3)	450(5)	8250(6)	94(2)
C69	2601(3)	526(5)	7429(5)	79(2)
C70	1847(2)	3412(4)	8613(4)	60(2)
C71	1429(3)	3583(4)	8602(4)	66(2)
C72	1228(3)	4284(6)	8887(6)	100(2)
C73	1332(4)	4493(6)	9664(6)	103(2)
C74	1534(3)	3912(3)	10271(4)	69(2)
C75	1951(3)	3704(3)	10276(4)	63(2)
C76	2305(2)	3983(4)	9712(4)	72(2)
C77	2193(3)	3925(6)	8888(5)	93(2)

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

Rh(1)-C(1)	2.069(4)	N(2)-C(1)	1.351(6)
Rh(1)-N(3)	2.095(4)	N(2)-C(3)	1.386(7)
Rh(1)-C(20)	2.137(6)	N(2)-C(16)	1.443(7)
Rh(1)-C(19)	2.142(6)	N(3)-C(17)	1.340(6)
Rh(1)-C(23)	2.163(5)	N(3)-N(3)#1	1.359(7)
Rh(1)-C(24)	2.200(5)	N(4)-C(27)	1.351(6)
Rh(2)-C(27)	2.067(5)	N(4)-C(28)	1.392(6)
Rh(2)-N(6)	2.093(4)	N(4)-C(38)	1.454(7)
Rh(2)-C(62)	2.116(5)	N(5)-C(27)	1.355(6)
Rh(2)-C(63)	2.128(5)	N(5)-C(29)	1.376(6)
Rh(2)-C(67)	2.165(6)	N(5)-C(30)	1.460(6)
Rh(2)-C(66)	2.191(5)	N(6)-C(31)	1.355(6)
Rh(3)-C(37)	2.054(5)	N(6)-N(7)	1.361(5)
Rh(3)-C(74)	2.094(6)	N(7)-C(33)	1.354(6)
Rh(3)-N(7)	2.097(4)	N(8)-C(37)	1.344(6)
Rh(3)-C(75)	2.141(6)	N(8)-C(35)	1.405(6)
Rh(3)-C(71)	2.171(6)	N(8)-C(34)	1.447(6)
Rh(3)-C(70)	2.203(6)	N(9)-C(37)	1.380(6)
P(1)-F(2)	1.561(4)	N(9)-C(36)	1.388(6)
P(1)-F(6)	1.570(4)	N(9)-C(50)	1.455(6)
P(1)-F(1)	1.584(4)	C(2)-C(3)	1.376(9)
P(1)-F(5)	1.587(3)	C(2)-H(2A)	0.9500
P(1)-F(3)	1.598(4)	C(3)-H(3A)	0.9500
P(1)-F(4)	1.599(4)	C(4)-C(9)	1.389(8)
P(2)-F(11)	1.574(4)	C(4)-C(5)	1.390(8)
P(2)-F(12)	1.574(4)	C(5)-C(6)	1.390(11)
P(2)-F(7)	1.575(4)	C(5)-C(10)	1.565(11)
P(2)-F(10)	1.576(4)	C(6)-C(7)	1.329(12)
P(2)-F(9)	1.577(4)	C(6)-H(6A)	0.9500
P(2)-F(8)	1.578(4)	C(7)-C(8)	1.375(11)
N(1)-C(1)	1.374(6)	C(7)-H(7A)	0.9500
N(1)-C(2)	1.396(7)	C(8)-C(9)	1.373(9)
N(1)-C(4)	1.420(7)	C(8)-H(8A)	0.9500

C(9)-C(13)	1.510(8)	C(20)-H(20A)	1.0000
C(10)-C(11)	1.477(8)	C(21)-C(22)	1.517(11)
C(10)-C(12)	1.482(8)	C(21)-H(21A)	0.9900
C(10)-H(10)	1.0000	C(21)-H(21B)	0.9900
C(11)-H(11A)	0.9800	C(22)-C(23)	1.476(10)
C(11)-H(11B)	0.9800	C(22)-H(22A)	0.9900
C(11)-H(11C)	0.9800	C(22)-H(22B)	0.9900
C(12)-H(12A)	0.9800	C(23)-C(24)	1.379(9)
C(12)-H(12B)	0.9800	C(23)-H(23A)	1.0000
C(12)-H(12C)	0.9800	C(24)-C(25)	1.498(10)
C(11')-H(11D)	0.9800	C(24)-H(24A)	1.0000
C(11')-H(11E)	0.9800	C(25)-C(26)	1.553(11)
C(11')-H(11F)	0.9800	C(25)-H(25A)	0.9900
C(12')-H(12D)	0.9800	C(25)-H(25B)	0.9900
C(12')-H(12E)	0.9800	C(26)-H(26A)	0.9900
C(12')-H(12F)	0.9800	C(26)-H(26B)	0.9900
C(13)-C(14)	1.504(12)	C(28)-C(29)	1.347(8)
C(13)-C(15)	1.586(10)	C(28)-H(28A)	0.9500
C(13)-H(13A)	1.0000	C(29)-H(29A)	0.9500
C(14)-H(14A)	0.9800	C(30)-C(31)	1.493(7)
C(14)-H(14B)	0.9800	C(30)-H(30A)	0.9900
C(14)-H(14C)	0.9800	C(30)-H(30B)	0.9900
C(15)-H(15A)	0.9800	C(31)-C(32)	1.354(7)
C(15)-H(15B)	0.9800	C(32)-C(33)	1.393(7)
C(15)-H(15C)	0.9800	C(32)-H(32A)	0.9500
C(16)-C(17)	1.533(8)	C(33)-C(34)	1.469(7)
C(16)-H(16A)	0.9900	C(34)-H(34A)	0.9900
C(16)-H(16B)	0.9900	C(34)-H(34B)	0.9900
C(17)-C(18)	1.383(7)	C(35)-C(36)	1.345(7)
C(18)-C(17)#1	1.383(7)	C(35)-H(35A)	0.9500
C(18)-H(18A)	0.9500	C(36)-H(36A)	0.9500
C(19)-C(20)	1.372(10)	C(38)-C(43)	1.382(7)
C(19)-C(26)	1.527(11)	C(38)-C(39)	1.414(8)
C(19)-H(19A)	1.0000	C(39)-C(40)	1.390(10)
C(20)-C(21)	1.554(10)	C(39)-C(44)	1.505(9)

C(40)-C(41)	1.383(11)	C(52)-C(53)	1.346(9)
C(40)-H(40A)	0.9500	C(52)-H(52A)	0.9500
C(41)-C(42)	1.358(10)	C(53)-C(54)	1.404(9)
C(41)-H(41A)	0.9500	C(53)-H(53A)	0.9500
C(42)-C(43)	1.416(8)	C(54)-C(55)	1.401(8)
C(42)-H(42A)	0.9500	C(54)-H(54A)	0.9500
C(43)-C(47)	1.509(8)	C(55)-C(59)	1.522(8)
C(44)-C(45)	1.604(8)	C(56)-C(58)	1.536(9)
C(44)-C(46)	1.607(8)	C(56)-C(57)	1.560(9)
C(44)-H(44)	1.0000	C(56)-H(56A)	1.0000
C(45)-H(45A)	0.9800	C(57)-H(57A)	0.9800
C(45)-H(45B)	0.9800	C(57)-H(57B)	0.9800
C(45)-H(45C)	0.9800	C(57)-H(57C)	0.9800
C(46)-H(46A)	0.9800	C(58)-H(58A)	0.9800
C(46)-H(46B)	0.9800	C(58)-H(58B)	0.9800
C(46)-H(46C)	0.9800	C(58)-H(58C)	0.9800
C(45')-H(45D)	0.9800	C(59)-C(60)	1.507(9)
C(45')-H(45E)	0.9800	C(59)-C(61)	1.525(9)
C(45')-H(45F)	0.9800	C(59)-H(59A)	1.0000
C(46')-H(46D)	0.9800	C(60)-H(60A)	0.9800
C(46')-H(46E)	0.9800	C(60)-H(60B)	0.9800
C(46')-H(46F)	0.9800	C(60)-H(60C)	0.9800
C(47)-C(49)	1.486(9)	C(61)-H(61A)	0.9800
C(47)-C(48)	1.539(8)	C(61)-H(61B)	0.9800
C(47)-H(47A)	1.0000	C(61)-H(61C)	0.9800
C(48)-H(48A)	0.9800	C(62)-C(63)	1.354(9)
C(48)-H(48B)	0.9800	C(62)-C(69)	1.507(10)
C(48)-H(48C)	0.9800	C(62)-H(62A)	1.0000
C(49)-H(49A)	0.9800	C(63)-C(64)	1.502(8)
C(49)-H(49B)	0.9800	C(63)-H(63A)	1.0000
C(49)-H(49C)	0.9800	C(64)-C(65)	1.445(10)
C(50)-C(55)	1.391(7)	C(64)-H(64A)	0.9900
C(50)-C(51)	1.402(7)	C(64)-H(64B)	0.9900
C(51)-C(52)	1.406(7)	C(65)-C(66)	1.549(10)
C(51)-C(56)	1.509(8)	C(65)-H(65A)	0.9900

C(65)-H(65B)	0.9900	N(3)-Rh(1)-C(19)	166.6(2)
C(66)-C(67)	1.312(11)	C(20)-Rh(1)-C(19)	37.4(3)
C(66)-H(66A)	1.0000	C(1)-Rh(1)-C(23)	150.0(2)
C(67)-C(68)	1.506(12)	N(3)-Rh(1)-C(23)	87.5(2)
C(67)-H(67A)	1.0000	C(20)-Rh(1)-C(23)	82.0(2)
C(68)-C(69)	1.419(11)	C(19)-Rh(1)-C(23)	97.5(3)
C(68)-H(68A)	0.9900	C(1)-Rh(1)-C(24)	173.0(2)
C(68)-H(68B)	0.9900	N(3)-Rh(1)-C(24)	94.62(18)
C(69)-H(69A)	0.9900	C(20)-Rh(1)-C(24)	89.8(2)
C(69)-H(69B)	0.9900	C(19)-Rh(1)-C(24)	82.3(2)
C(70)-C(71)	1.327(10)	C(23)-Rh(1)-C(24)	36.8(2)
C(70)-C(77)	1.475(11)	C(27)-Rh(2)-N(6)	86.38(16)
C(70)-H(70A)	1.0000	C(27)-Rh(2)-C(62)	90.5(2)
C(71)-C(72)	1.499(12)	N(6)-Rh(2)-C(62)	156.7(2)
C(71)-H(71A)	1.0000	C(27)-Rh(2)-C(63)	97.39(19)
C(72)-C(73)	1.361(13)	N(6)-Rh(2)-C(63)	166.00(19)
C(72)-H(72A)	0.9900	C(62)-Rh(2)-C(63)	37.2(2)
C(72)-H(72B)	0.9900	C(27)-Rh(2)-C(67)	150.8(3)
C(73)-C(74)	1.566(12)	N(6)-Rh(2)-C(67)	89.6(2)
C(73)-H(73A)	0.9900	C(62)-Rh(2)-C(67)	81.9(3)
C(73)-H(73B)	0.9900	C(63)-Rh(2)-C(67)	93.4(2)
C(74)-C(75)	1.338(10)	C(27)-Rh(2)-C(66)	174.0(3)
C(74)-H(74A)	1.0000	N(6)-Rh(2)-C(66)	93.06(17)
C(75)-C(76)	1.547(10)	C(62)-Rh(2)-C(66)	92.3(2)
C(75)-H(75A)	1.0000	C(63)-Rh(2)-C(66)	81.8(2)
C(76)-C(77)	1.392(11)	C(67)-Rh(2)-C(66)	35.1(3)
C(76)-H(76A)	0.9900	C(37)-Rh(3)-C(74)	92.3(2)
C(76)-H(76B)	0.9900	C(37)-Rh(3)-N(7)	85.44(17)
C(77)-H(77A)	0.9900	C(74)-Rh(3)-N(7)	154.6(3)
C(77)-H(77B)	0.9900	C(37)-Rh(3)-C(75)	97.2(2)
		C(74)-Rh(3)-C(75)	36.8(3)
C(1)-Rh(1)-N(3)	85.36(17)	N(7)-Rh(3)-C(75)	168.5(3)
C(1)-Rh(1)-C(20)	93.0(2)	C(37)-Rh(3)-C(71)	154.5(2)
N(3)-Rh(1)-C(20)	156.0(2)	C(74)-Rh(3)-C(71)	82.8(3)
C(1)-Rh(1)-C(19)	96.1(2)	N(7)-Rh(3)-C(71)	88.4(2)

C(75)-Rh(3)-C(71)	93.8(3)	F(9)-P(2)-F(8)	89.7(2)
C(37)-Rh(3)-C(70)	170.1(3)	C(1)-N(1)-C(2)	110.5(4)
C(74)-Rh(3)-C(70)	91.2(2)	C(1)-N(1)-C(4)	127.3(4)
N(7)-Rh(3)-C(70)	95.32(19)	C(2)-N(1)-C(4)	122.1(4)
C(75)-Rh(3)-C(70)	80.2(2)	C(1)-N(2)-C(3)	113.9(5)
C(71)-Rh(3)-C(70)	35.3(3)	C(1)-N(2)-C(16)	123.6(5)
F(2)-P(1)-F(6)	90.4(3)	C(3)-N(2)-C(16)	122.4(5)
F(2)-P(1)-F(1)	92.5(3)	C(17)-N(3)-N(3)#1	107.6(3)
F(6)-P(1)-F(1)	88.3(2)	C(17)-N(3)-Rh(1)	118.9(3)
F(2)-P(1)-F(5)	88.9(3)	N(3)#1-N(3)-Rh(1)	132.05(14)
F(6)-P(1)-F(5)	179.3(3)	C(27)-N(4)-C(28)	111.3(4)
F(1)-P(1)-F(5)	91.8(2)	C(27)-N(4)-C(38)	126.9(4)
F(2)-P(1)-F(3)	91.8(3)	C(28)-N(4)-C(38)	121.6(4)
F(6)-P(1)-F(3)	91.6(2)	C(27)-N(5)-C(29)	112.1(4)
F(1)-P(1)-F(3)	175.7(3)	C(27)-N(5)-C(30)	124.0(4)
F(5)-P(1)-F(3)	88.4(2)	C(29)-N(5)-C(30)	123.6(4)
F(2)-P(1)-F(4)	178.0(3)	C(31)-N(6)-N(7)	107.6(4)
F(6)-P(1)-F(4)	88.6(2)	C(31)-N(6)-Rh(2)	119.2(3)
F(1)-P(1)-F(4)	89.3(3)	N(7)-N(6)-Rh(2)	130.6(3)
F(5)-P(1)-F(4)	92.1(3)	C(33)-N(7)-N(6)	107.6(4)
F(3)-P(1)-F(4)	86.4(3)	C(33)-N(7)-Rh(3)	119.2(3)
F(11)-P(2)-F(12)	90.1(2)	N(6)-N(7)-Rh(3)	129.9(3)
F(11)-P(2)-F(7)	90.0(2)	C(37)-N(8)-C(35)	112.9(4)
F(12)-P(2)-F(7)	179.9(3)	C(37)-N(8)-C(34)	123.5(4)
F(11)-P(2)-F(10)	89.8(2)	C(35)-N(8)-C(34)	123.4(4)
F(12)-P(2)-F(10)	90.0(2)	C(37)-N(9)-C(36)	110.6(4)
F(7)-P(2)-F(10)	90.0(2)	C(37)-N(9)-C(50)	127.2(4)
F(11)-P(2)-F(9)	179.6(4)	C(36)-N(9)-C(50)	122.0(4)
F(12)-P(2)-F(9)	89.9(2)	N(2)-C(1)-N(1)	103.6(4)
F(7)-P(2)-F(9)	89.9(2)	N(2)-C(1)-Rh(1)	118.2(3)
F(10)-P(2)-F(9)	89.8(2)	N(1)-C(1)-Rh(1)	138.2(4)
F(11)-P(2)-F(8)	90.6(4)	C(3)-C(2)-N(1)	107.6(5)
F(12)-P(2)-F(8)	90.0(2)	C(3)-C(2)-H(2A)	126.2
F(7)-P(2)-F(8)	90.0(2)	N(1)-C(2)-H(2A)	126.2
F(10)-P(2)-F(8)	179.6(4)	C(2)-C(3)-N(2)	104.3(5)

C(2)-C(3)-H(3A)	127.8	C(10)-C(12)-H(12C)	109.5
N(2)-C(3)-H(3A)	127.8	H(12A)-C(12)-H(12C)	109.5
C(9)-C(4)-C(5)	122.0(6)	H(12B)-C(12)-H(12C)	109.5
C(9)-C(4)-N(1)	119.9(5)	H(11D)-C(11')-H(11E)	109.5
C(5)-C(4)-N(1)	118.1(5)	H(11D)-C(11')-H(11F)	109.5
C(4)-C(5)-C(6)	116.9(7)	H(11E)-C(11')-H(11F)	109.5
C(4)-C(5)-C(10)	121.5(6)	H(12D)-C(12')-H(12E)	109.5
C(6)-C(5)-C(10)	121.4(6)	H(12D)-C(12')-H(12F)	109.5
C(7)-C(6)-C(5)	122.4(7)	H(12E)-C(12')-H(12F)	109.5
C(7)-C(6)-H(6A)	118.8	C(14)-C(13)-C(9)	115.9(8)
C(5)-C(6)-H(6A)	118.8	C(14)-C(13)-C(15)	109.9(7)
C(6)-C(7)-C(8)	119.4(8)	C(9)-C(13)-C(15)	110.2(5)
C(6)-C(7)-H(7A)	120.3	C(14)-C(13)-H(13A)	106.8
C(8)-C(7)-H(7A)	120.3	C(9)-C(13)-H(13A)	106.8
C(9)-C(8)-C(7)	121.9(7)	C(15)-C(13)-H(13A)	106.8
C(9)-C(8)-H(8A)	119.0	C(13)-C(14)-H(14A)	109.5
C(7)-C(8)-H(8A)	119.0	C(13)-C(14)-H(14B)	109.5
C(8)-C(9)-C(4)	117.1(6)	H(14A)-C(14)-H(14B)	109.5
C(8)-C(9)-C(13)	120.2(6)	C(13)-C(14)-H(14C)	109.5
C(4)-C(9)-C(13)	122.7(5)	H(14A)-C(14)-H(14C)	109.5
C(11)-C(10)-C(12)	104.5(8)	H(14B)-C(14)-H(14C)	109.5
C(11)-C(10)-C(5)	118.9(8)	C(13)-C(15)-H(15A)	109.5
C(12)-C(10)-C(5)	109.8(7)	C(13)-C(15)-H(15B)	109.5
C(11)-C(10)-H(10)	107.8	H(15A)-C(15)-H(15B)	109.5
C(12)-C(10)-H(10)	107.8	C(13)-C(15)-H(15C)	109.5
C(5)-C(10)-H(10)	107.8	H(15A)-C(15)-H(15C)	109.5
C(10)-C(11)-H(11A)	109.5	H(15B)-C(15)-H(15C)	109.5
C(10)-C(11)-H(11B)	109.5	N(2)-C(16)-C(17)	110.1(5)
H(11A)-C(11)-H(11B)	109.5	N(2)-C(16)-H(16A)	109.6
C(10)-C(11)-H(11C)	109.5	C(17)-C(16)-H(16A)	109.6
H(11A)-C(11)-H(11C)	109.5	N(2)-C(16)-H(16B)	109.6
H(11B)-C(11)-H(11C)	109.5	C(17)-C(16)-H(16B)	109.6
C(10)-C(12)-H(12A)	109.5	H(16A)-C(16)-H(16B)	108.2
C(10)-C(12)-H(12B)	109.5	N(3)-C(17)-C(18)	110.6(5)
H(12A)-C(12)-H(12B)	109.5	N(3)-C(17)-C(16)	120.9(5)

C(18)-C(17)-C(16)	128.4(5)	C(23)-C(24)-Rh(1)	70.1(3)
C(17)#1-C(18)-C(17)	103.5(6)	C(25)-C(24)-Rh(1)	111.3(4)
C(17)#1-C(18)-H(18A)	128.2	C(23)-C(24)-H(24A)	113.8
C(17)-C(18)-H(18A)	128.2	C(25)-C(24)-H(24A)	113.8
C(20)-C(19)-C(26)	126.5(7)	Rh(1)-C(24)-H(24A)	113.8
C(20)-C(19)-Rh(1)	71.1(4)	C(24)-C(25)-C(26)	114.1(6)
C(26)-C(19)-Rh(1)	109.0(5)	C(24)-C(25)-H(25A)	108.7
C(20)-C(19)-H(19A)	113.9	C(26)-C(25)-H(25A)	108.7
C(26)-C(19)-H(19A)	113.9	C(24)-C(25)-H(25B)	108.7
Rh(1)-C(19)-H(19A)	113.9	C(26)-C(25)-H(25B)	108.7
C(19)-C(20)-C(21)	124.5(6)	H(25A)-C(25)-H(25B)	107.6
C(19)-C(20)-Rh(1)	71.5(3)	C(19)-C(26)-C(25)	113.3(7)
C(21)-C(20)-Rh(1)	112.8(4)	C(19)-C(26)-H(26A)	108.9
C(19)-C(20)-H(20A)	113.6	C(25)-C(26)-H(26A)	108.9
C(21)-C(20)-H(20A)	113.6	C(19)-C(26)-H(26B)	108.9
Rh(1)-C(20)-H(20A)	113.6	C(25)-C(26)-H(26B)	108.9
C(22)-C(21)-C(20)	111.4(6)	H(26A)-C(26)-H(26B)	107.7
C(22)-C(21)-H(21A)	109.4	N(4)-C(27)-N(5)	103.7(4)
C(20)-C(21)-H(21A)	109.4	N(4)-C(27)-Rh(2)	138.2(4)
C(22)-C(21)-H(21B)	109.4	N(5)-C(27)-Rh(2)	118.0(3)
C(20)-C(21)-H(21B)	109.4	C(29)-C(28)-N(4)	106.5(4)
H(21A)-C(21)-H(21B)	108.0	C(29)-C(28)-H(28A)	126.8
C(23)-C(22)-C(21)	116.7(7)	N(4)-C(28)-H(28A)	126.8
C(23)-C(22)-H(22A)	108.1	C(28)-C(29)-N(5)	106.3(4)
C(21)-C(22)-H(22A)	108.1	C(28)-C(29)-H(29A)	126.8
C(23)-C(22)-H(22B)	108.1	N(5)-C(29)-H(29A)	126.8
C(21)-C(22)-H(22B)	108.1	N(5)-C(30)-C(31)	112.3(4)
H(22A)-C(22)-H(22B)	107.3	N(5)-C(30)-H(30A)	109.1
C(24)-C(23)-C(22)	124.0(7)	C(31)-C(30)-H(30A)	109.1
C(24)-C(23)-Rh(1)	73.0(3)	N(5)-C(30)-H(30B)	109.1
C(22)-C(23)-Rh(1)	109.3(4)	C(31)-C(30)-H(30B)	109.1
C(24)-C(23)-H(23A)	114.3	H(30A)-C(30)-H(30B)	107.9
C(22)-C(23)-H(23A)	114.3	C(32)-C(31)-N(6)	110.5(4)
Rh(1)-C(23)-H(23A)	114.3	C(32)-C(31)-C(30)	128.7(4)
C(23)-C(24)-C(25)	125.9(6)	N(6)-C(31)-C(30)	120.7(4)

C(31)-C(32)-C(33)	105.0(4)	C(43)-C(42)-H(42A)	120.0
C(31)-C(32)-H(32A)	127.5	C(38)-C(43)-C(42)	117.7(5)
C(33)-C(32)-H(32A)	127.5	C(38)-C(43)-C(47)	124.8(5)
N(7)-C(33)-C(32)	109.3(4)	C(42)-C(43)-C(47)	117.5(5)
N(7)-C(33)-C(34)	121.9(5)	C(39)-C(44)-C(45)	101.9(15)
C(32)-C(33)-C(34)	128.6(4)	C(39)-C(44)-C(46)	97.2(15)
N(8)-C(34)-C(33)	112.4(4)	C(45)-C(44)-C(46)	115.1(10)
N(8)-C(34)-H(34A)	109.1	C(39)-C(44)-H(44)	113.6
C(33)-C(34)-H(34A)	109.1	C(45)-C(44)-H(44)	113.6
N(8)-C(34)-H(34B)	109.1	C(46)-C(44)-H(44)	113.6
C(33)-C(34)-H(34B)	109.1	C(44)-C(45)-H(45A)	109.5
H(34A)-C(34)-H(34B)	107.9	C(44)-C(45)-H(45B)	109.5
C(36)-C(35)-N(8)	105.0(4)	H(45A)-C(45)-H(45B)	109.5
C(36)-C(35)-H(35A)	127.5	C(44)-C(45)-H(45C)	109.5
N(8)-C(35)-H(35A)	127.5	H(45A)-C(45)-H(45C)	109.5
C(35)-C(36)-N(9)	108.1(4)	H(45B)-C(45)-H(45C)	109.5
C(35)-C(36)-H(36A)	126.0	C(44)-C(46)-H(46A)	109.5
N(9)-C(36)-H(36A)	126.0	C(44)-C(46)-H(46B)	109.5
N(8)-C(37)-N(9)	103.4(4)	H(46A)-C(46)-H(46B)	109.5
N(8)-C(37)-Rh(3)	120.0(3)	C(44)-C(46)-H(46C)	109.5
N(9)-C(37)-Rh(3)	136.5(3)	H(46A)-C(46)-H(46C)	109.5
C(43)-C(38)-C(39)	123.1(5)	H(46B)-C(46)-H(46C)	109.5
C(43)-C(38)-N(4)	117.5(5)	H(45D)-C(45')-H(45E)	109.5
C(39)-C(38)-N(4)	119.3(5)	H(45D)-C(45')-H(45F)	109.5
C(40)-C(39)-C(38)	116.5(6)	H(45E)-C(45')-H(45F)	109.5
C(40)-C(39)-C(44)	122.0(6)	H(46D)-C(46')-H(46E)	109.5
C(38)-C(39)-C(44)	121.4(6)	H(46D)-C(46')-H(46F)	109.5
C(41)-C(40)-C(39)	121.1(6)	H(46E)-C(46')-H(46F)	109.5
C(41)-C(40)-H(40A)	119.5	C(49)-C(47)-C(43)	114.5(5)
C(39)-C(40)-H(40A)	119.5	C(49)-C(47)-C(48)	109.8(5)
C(42)-C(41)-C(40)	121.7(6)	C(43)-C(47)-C(48)	109.8(5)
C(42)-C(41)-H(41A)	119.2	C(49)-C(47)-H(47A)	107.5
C(40)-C(41)-H(41A)	119.2	C(43)-C(47)-H(47A)	107.5
C(41)-C(42)-C(43)	119.9(6)	C(48)-C(47)-H(47A)	107.5
C(41)-C(42)-H(42A)	120.0	C(47)-C(48)-H(48A)	109.5

C(47)-C(48)-H(48B)	109.5	C(56)-C(57)-H(57A)	109.5
H(48A)-C(48)-H(48B)	109.5	C(56)-C(57)-H(57B)	109.5
C(47)-C(48)-H(48C)	109.5	H(57A)-C(57)-H(57B)	109.5
H(48A)-C(48)-H(48C)	109.5	C(56)-C(57)-H(57C)	109.5
H(48B)-C(48)-H(48C)	109.5	H(57A)-C(57)-H(57C)	109.5
C(47)-C(49)-H(49A)	109.5	H(57B)-C(57)-H(57C)	109.5
C(47)-C(49)-H(49B)	109.5	C(56)-C(58)-H(58A)	109.5
H(49A)-C(49)-H(49B)	109.5	C(56)-C(58)-H(58B)	109.5
C(47)-C(49)-H(49C)	109.5	H(58A)-C(58)-H(58B)	109.5
H(49A)-C(49)-H(49C)	109.5	C(56)-C(58)-H(58C)	109.5
H(49B)-C(49)-H(49C)	109.5	H(58A)-C(58)-H(58C)	109.5
C(55)-C(50)-C(51)	123.7(5)	H(58B)-C(58)-H(58C)	109.5
C(55)-C(50)-N(9)	119.1(4)	C(60)-C(59)-C(55)	112.8(6)
C(51)-C(50)-N(9)	117.2(4)	C(60)-C(59)-C(61)	114.1(6)
C(50)-C(51)-C(52)	116.8(5)	C(55)-C(59)-C(61)	109.2(5)
C(50)-C(51)-C(56)	122.5(4)	C(60)-C(59)-H(59A)	106.7
C(52)-C(51)-C(56)	120.7(5)	C(55)-C(59)-H(59A)	106.7
C(53)-C(52)-C(51)	121.2(5)	C(61)-C(59)-H(59A)	106.7
C(53)-C(52)-H(52A)	119.4	C(59)-C(60)-H(60A)	109.5
C(51)-C(52)-H(52A)	119.4	C(59)-C(60)-H(60B)	109.5
C(52)-C(53)-C(54)	121.1(5)	H(60A)-C(60)-H(60B)	109.5
C(52)-C(53)-H(53A)	119.4	C(59)-C(60)-H(60C)	109.5
C(54)-C(53)-H(53A)	119.4	H(60A)-C(60)-H(60C)	109.5
C(55)-C(54)-C(53)	120.4(6)	H(60B)-C(60)-H(60C)	109.5
C(55)-C(54)-H(54A)	119.8	C(59)-C(61)-H(61A)	109.5
C(53)-C(54)-H(54A)	119.8	C(59)-C(61)-H(61B)	109.5
C(50)-C(55)-C(54)	116.7(5)	H(61A)-C(61)-H(61B)	109.5
C(50)-C(55)-C(59)	123.1(5)	C(59)-C(61)-H(61C)	109.5
C(54)-C(55)-C(59)	120.2(5)	H(61A)-C(61)-H(61C)	109.5
C(51)-C(56)-C(58)	113.6(5)	H(61B)-C(61)-H(61C)	109.5
C(51)-C(56)-C(57)	112.3(6)	C(63)-C(62)-C(69)	124.7(6)
C(58)-C(56)-C(57)	106.8(5)	C(63)-C(62)-Rh(2)	71.9(3)
C(51)-C(56)-H(56A)	108.0	C(69)-C(62)-Rh(2)	111.2(5)
C(58)-C(56)-H(56A)	108.0	C(63)-C(62)-H(62A)	113.9
C(57)-C(56)-H(56A)	108.0	C(69)-C(62)-H(62A)	113.9

Rh(2)-C(62)-H(62A)	113.9	C(67)-C(68)-H(68B)	107.8
C(62)-C(63)-C(64)	125.5(5)	H(68A)-C(68)-H(68B)	107.2
C(62)-C(63)-Rh(2)	70.9(3)	C(68)-C(69)-C(62)	116.9(7)
C(64)-C(63)-Rh(2)	112.1(4)	C(68)-C(69)-H(69A)	108.1
C(62)-C(63)-H(63A)	113.5	C(62)-C(69)-H(69A)	108.1
C(64)-C(63)-H(63A)	113.5	C(68)-C(69)-H(69B)	108.1
Rh(2)-C(63)-H(63A)	113.5	C(62)-C(69)-H(69B)	108.1
C(65)-C(64)-C(63)	116.7(6)	H(69A)-C(69)-H(69B)	107.3
C(65)-C(64)-H(64A)	108.1	C(71)-C(70)-C(77)	123.2(7)
C(63)-C(64)-H(64A)	108.1	C(71)-C(70)-Rh(3)	71.0(4)
C(65)-C(64)-H(64B)	108.1	C(77)-C(70)-Rh(3)	110.1(5)
C(63)-C(64)-H(64B)	108.1	C(71)-C(70)-H(70A)	114.8
H(64A)-C(64)-H(64B)	107.3	C(77)-C(70)-H(70A)	114.8
C(64)-C(65)-C(66)	117.0(6)	Rh(3)-C(70)-H(70A)	114.8
C(64)-C(65)-H(65A)	108.0	C(70)-C(71)-C(72)	127.5(8)
C(66)-C(65)-H(65A)	108.0	C(70)-C(71)-Rh(3)	73.7(4)
C(64)-C(65)-H(65B)	108.0	C(72)-C(71)-Rh(3)	107.0(5)
C(66)-C(65)-H(65B)	108.0	C(70)-C(71)-H(71A)	113.5
H(65A)-C(65)-H(65B)	107.3	C(72)-C(71)-H(71A)	113.5
C(67)-C(66)-C(65)	125.0(7)	Rh(3)-C(71)-H(71A)	113.5
C(67)-C(66)-Rh(2)	71.4(4)	C(73)-C(72)-C(71)	116.5(9)
C(65)-C(66)-Rh(2)	109.0(4)	C(73)-C(72)-H(72A)	108.2
C(67)-C(66)-H(66A)	114.3	C(71)-C(72)-H(72A)	108.2
C(65)-C(66)-H(66A)	114.3	C(73)-C(72)-H(72B)	108.2
Rh(2)-C(66)-H(66A)	114.3	C(71)-C(72)-H(72B)	108.2
C(66)-C(67)-C(68)	124.3(7)	H(72A)-C(72)-H(72B)	107.3
C(66)-C(67)-Rh(2)	73.5(4)	C(72)-C(73)-C(74)	119.1(9)
C(68)-C(67)-Rh(2)	109.0(5)	C(72)-C(73)-H(73A)	107.5
C(66)-C(67)-H(67A)	114.2	C(74)-C(73)-H(73A)	107.5
C(68)-C(67)-H(67A)	114.2	C(72)-C(73)-H(73B)	107.5
Rh(2)-C(67)-H(67A)	114.2	C(74)-C(73)-H(73B)	107.5
C(69)-C(68)-C(67)	117.9(7)	H(73A)-C(73)-H(73B)	107.0
C(69)-C(68)-H(68A)	107.8	C(75)-C(74)-C(73)	123.6(7)
C(67)-C(68)-H(68A)	107.8	C(75)-C(74)-Rh(3)	73.5(4)
C(69)-C(68)-H(68B)	107.8	C(73)-C(74)-Rh(3)	107.7(5)

C(75)-C(74)-H(74A)	114.7	C(75)-C(76)-H(76A)	108.7
C(73)-C(74)-H(74A)	114.7	C(77)-C(76)-H(76B)	108.7
Rh(3)-C(74)-H(74A)	114.7	C(75)-C(76)-H(76B)	108.7
C(74)-C(75)-C(76)	127.1(6)	H(76A)-C(76)-H(76B)	107.6
C(74)-C(75)-Rh(3)	69.7(4)	C(76)-C(77)-C(70)	119.8(7)
C(76)-C(75)-Rh(3)	110.5(4)	C(76)-C(77)-H(77A)	107.4
C(74)-C(75)-H(75A)	113.6	C(70)-C(77)-H(77A)	107.4
C(76)-C(75)-H(75A)	113.6	C(76)-C(77)-H(77B)	107.4
Rh(3)-C(75)-H(75A)	113.6	C(70)-C(77)-H(77B)	107.4
C(77)-C(76)-C(75)	114.0(6)	H(77A)-C(77)-H(77B)	106.9
C(77)-C(76)-H(76A)	108.7		

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	31(1)	33(1)	33(1)	-2(1)	1(1)	-1(1)
Rh2	28(1)	35(1)	29(1)	2(1)	-3(1)	0(1)
Rh3	34(1)	33(1)	33(1)	3(1)	2(1)	-1(1)
P1	38(1)	38(1)	39(1)	-3(1)	2(1)	4(1)
F1	99(3)	88(3)	123(4)	-60(3)	-54(3)	7(3)
F2	129(4)	67(3)	127(4)	25(3)	78(3)	1(3)
F3	71(2)	71(2)	69(2)	-16(2)	-30(2)	14(2)
F4	117(3)	64(2)	70(3)	18(2)	30(2)	0(2)
F5	43(2)	66(2)	110(3)	-29(2)	-13(2)	19(2)
F6	67(2)	67(3)	82(3)	-9(2)	10(2)	29(2)
P2	63(2)	38(1)	33(2)	3(2)	9(2)	-3(3)
F7	77(3)	89(3)	45(3)	-2(5)	17(3)	5(7)
F8	84(7)	87(10)	73(7)	-51(7)	6(5)	-18(6)
F9	124(11)	44(5)	93(9)	7(5)	36(8)	19(6)
F10	81(8)	162(17)	58(7)	-42(8)	8(5)	-38(10)
F11	105(11)	73(7)	72(7)	16(5)	4(6)	51(6)
F12	77(3)	89(3)	45(3)	-2(5)	17(3)	5(7)
P2'	63(2)	38(1)	33(2)	3(2)	9(2)	-3(3)
F7'	77(3)	89(3)	45(3)	-2(5)	17(3)	5(7)
F8'	84(7)	87(10)	73(7)	-51(7)	6(5)	-18(6)
F9'	124(11)	44(5)	93(9)	7(5)	36(8)	19(6)
F10'	81(8)	162(17)	58(7)	-42(8)	8(5)	-38(10)
F11'	105(11)	73(7)	72(7)	16(5)	4(6)	51(6)
F12'	77(3)	89(3)	45(3)	-2(5)	17(3)	5(7)
N1	35(2)	38(2)	35(2)	-2(2)	0(2)	-3(2)
N2	61(3)	33(2)	32(2)	-8(2)	3(2)	-4(2)
N3	41(2)	32(2)	32(2)	-1(2)	2(2)	0(2)
N4	37(2)	48(2)	29(2)	-3(2)	-3(2)	3(2)
N5	37(2)	38(2)	36(2)	-3(2)	-3(2)	-1(2)
N6	28(2)	43(2)	31(2)	-3(2)	0(1)	-8(2)

N7	33(2)	42(2)	32(2)	0(2)	-3(2)	-4(2)
N8	36(2)	47(3)	31(2)	-2(2)	6(2)	-5(2)
N9	39(2)	44(2)	25(2)	-1(2)	-3(2)	-3(2)
C1	32(2)	33(2)	29(2)	-3(2)	-4(2)	2(2)
C2	58(3)	54(4)	34(3)	-4(2)	-2(2)	-17(3)
C3	67(4)	52(3)	45(3)	-8(3)	11(3)	-7(3)
C4	48(3)	50(3)	37(3)	0(2)	-8(2)	3(2)
C5	61(4)	84(4)	49(3)	12(3)	-4(3)	34(3)
C6	107(6)	98(6)	90(6)	26(5)	16(4)	76(5)
C7	89(3)	81(3)	90(3)	5(2)	2(2)	11(2)
C8	65(2)	62(2)	66(2)	5(2)	-2(2)	1(2)
C9	48(3)	53(3)	36(3)	12(2)	-13(2)	1(2)
C10	76(2)	82(2)	77(2)	5(1)	4(1)	4(1)
C11	93(3)	95(3)	94(3)	-1(1)	5(1)	3(1)
C12	80(3)	84(3)	82(3)	5(1)	3(1)	1(1)
C10'	76(2)	82(2)	77(2)	5(1)	4(1)	4(1)
C11'	93(3)	95(3)	94(3)	-1(1)	5(1)	3(1)
C12'	80(3)	84(3)	82(3)	5(1)	3(1)	1(1)
C13	31(3)	99(6)	49(3)	31(3)	2(2)	12(3)
C14	90(7)	197(13)	133(9)	89(9)	47(6)	-13(7)
C15	70(4)	83(5)	61(4)	2(3)	14(3)	15(4)
C16	98(5)	44(3)	44(3)	12(3)	11(3)	27(3)
C17	60(3)	31(3)	35(3)	0(2)	1(2)	9(2)
C18	103(7)	26(4)	38(4)	0	9(4)	0
C19	80(4)	56(4)	53(3)	7(3)	11(3)	-36(3)
C20	50(3)	98(5)	44(3)	-18(3)	16(2)	-27(3)
C21	77(2)	93(3)	78(2)	-2(2)	8(2)	-4(2)
C22	77(2)	96(3)	82(2)	-3(2)	1(2)	-3(2)
C23	40(3)	97(4)	41(3)	-15(3)	-10(2)	-16(3)
C24	57(3)	57(3)	39(3)	-16(2)	9(2)	-20(3)
C25	76(2)	69(2)	68(2)	-6(2)	8(2)	-11(2)
C26	90(3)	74(2)	81(2)	2(2)	7(2)	-8(2)
C27	34(2)	33(2)	34(2)	-4(2)	3(2)	4(2)
C28	32(2)	63(3)	36(2)	-1(2)	-8(2)	6(2)
C29	34(2)	63(4)	36(3)	-9(2)	-11(2)	1(2)

C30	43(3)	51(3)	33(3)	-1(2)	-5(2)	-13(2)
C31	41(3)	33(2)	32(2)	-1(2)	-5(2)	-7(2)
C32	26(2)	51(3)	38(3)	1(2)	-3(2)	-14(2)
C33	31(2)	48(3)	37(2)	8(2)	2(2)	-3(2)
C34	30(2)	71(4)	46(3)	-2(3)	6(2)	-8(2)
C35	39(3)	45(3)	44(3)	2(2)	12(2)	-6(2)
C36	54(3)	37(2)	33(2)	1(2)	6(2)	-10(2)
C37	36(3)	31(2)	40(3)	1(2)	4(2)	-5(2)
C38	41(3)	50(3)	28(2)	2(2)	-3(2)	2(2)
C39	80(4)	37(3)	41(3)	10(2)	-7(3)	4(3)
C40	114(6)	48(4)	54(4)	11(3)	5(4)	-26(4)
C41	78(5)	63(4)	69(4)	15(4)	9(4)	-21(4)
C42	43(3)	74(4)	48(3)	3(3)	12(2)	-4(3)
C43	47(3)	48(3)	32(2)	5(2)	1(2)	0(2)
C44	98(5)	28(3)	69(4)	2(3)	14(3)	13(3)
C45	96(4)	93(4)	96(4)	3(2)	3(2)	6(2)
C46	138(9)	22(5)	50(4)	-7(4)	21(6)	-5(5)
C44'	98(5)	28(3)	69(4)	2(3)	14(3)	13(3)
C45'	96(4)	93(4)	96(4)	3(2)	3(2)	6(2)
C46'	138(9)	22(5)	50(4)	-7(4)	21(6)	-5(5)
C47	42(3)	54(3)	45(3)	-6(3)	6(2)	-3(2)
C48	74(4)	84(5)	45(3)	-24(3)	-8(3)	12(4)
C49	79(5)	66(4)	71(4)	-8(4)	-10(4)	21(4)
C50	39(3)	34(2)	32(2)	6(2)	-1(2)	-4(2)
C51	52(3)	32(3)	34(2)	-1(2)	3(2)	-12(2)
C52	63(4)	46(3)	37(3)	-5(2)	-6(2)	-21(3)
C53	61(4)	63(4)	51(3)	-4(3)	-9(3)	-22(3)
C54	42(3)	67(4)	53(3)	10(3)	-4(2)	-5(3)
C55	51(3)	49(3)	35(3)	2(2)	-2(2)	-3(2)
C56	43(3)	49(3)	51(3)	-15(2)	19(2)	-15(2)
C57	121(7)	67(4)	65(4)	-8(4)	33(4)	-29(5)
C58	81(5)	49(4)	80(5)	-24(3)	25(4)	-15(3)
C59	46(3)	55(4)	54(3)	-10(3)	-7(2)	17(3)
C60	61(4)	113(6)	57(4)	-15(4)	-3(3)	28(4)
C61	82(5)	49(4)	76(4)	-4(3)	8(4)	16(3)

C62	45(3)	62(3)	58(3)	-1(3)	5(3)	29(3)
C63	40(3)	62(3)	37(2)	3(2)	-1(2)	2(2)
C64	61(2)	71(2)	64(2)	7(2)	-2(2)	-7(2)
C65	77(2)	96(3)	77(2)	-7(2)	4(2)	-5(2)
C66	33(2)	128(5)	28(2)	-4(3)	-9(2)	18(3)
C67	60(4)	85(4)	58(4)	34(3)	-1(3)	18(3)
C68	95(3)	92(3)	94(3)	9(2)	4(2)	12(2)
C69	82(3)	74(2)	81(2)	4(2)	5(2)	13(2)
C70	89(4)	54(4)	37(3)	4(3)	7(3)	-27(3)
C71	91(4)	51(3)	57(4)	26(3)	-2(3)	5(3)
C72	102(3)	96(3)	101(3)	4(2)	3(2)	7(2)
C73	107(3)	98(3)	104(3)	1(2)	10(2)	11(2)
C74	116(5)	30(3)	65(4)	-8(3)	39(4)	-1(3)
C75	93(4)	46(3)	47(3)	15(3)	-12(3)	-35(3)
C76	73(2)	72(2)	69(2)	5(2)	-2(2)	-14(2)
C77	94(3)	95(3)	89(3)	-4(2)	7(2)	-12(2)

Table 5. Torsion angles [°].

C1-Rh1-N3-C17	-46.1(4)	C70-Rh3-N7-N6	12.3(4)
C20-Rh1-N3-C17	40.9(7)	C3-N2-C1-N1	-0.4(6)
C19-Rh1-N3-C17	-142.9(9)	C16-N2-C1-N1	-178.6(5)
C23-Rh1-N3-C17	104.7(4)	C3-N2-C1-Rh1	180.0(4)
C24-Rh1-N3-C17	140.9(4)	C16-N2-C1-Rh1	1.7(7)
C1-Rh1-N3-N3#1	149.6(5)	C2-N1-C1-N2	0.9(6)
C20-Rh1-N3-N3#1	-123.4(6)	C4-N1-C1-N2	177.1(5)
C19-Rh1-N3-N3#1	52.7(12)	C2-N1-C1-Rh1	-179.6(4)
C23-Rh1-N3-N3#1	-59.6(5)	C4-N1-C1-Rh1	-3.4(8)
C24-Rh1-N3-N3#1	-23.5(6)	N3-Rh1-C1-N2	41.4(4)
C27-Rh2-N6-C31	46.6(4)	C20-Rh1-C1-N2	-114.7(4)
C62-Rh2-N6-C31	-36.3(7)	C19-Rh1-C1-N2	-152.1(4)
C63-Rh2-N6-C31	152.8(7)	C23-Rh1-C1-N2	-35.4(7)
C67-Rh2-N6-C31	-104.5(4)	C24-Rh1-C1-N2	131.5(16)
C66-Rh2-N6-C31	-139.4(4)	N3-Rh1-C1-N1	-138.1(5)
C27-Rh2-N6-N7	-154.3(4)	C20-Rh1-C1-N1	65.8(6)
C62-Rh2-N6-N7	122.8(6)	C19-Rh1-C1-N1	28.4(6)
C63-Rh2-N6-N7	-48.2(9)	C23-Rh1-C1-N1	145.1(5)
C67-Rh2-N6-N7	54.6(5)	C24-Rh1-C1-N1	-48(2)
C66-Rh2-N6-N7	19.7(5)	C1-N1-C2-C3	-1.1(6)
C31-N6-N7-C33	-0.1(5)	C4-N1-C2-C3	-177.5(5)
Rh2-N6-N7-C33	-161.0(4)	N1-C2-C3-N2	0.8(6)
C31-N6-N7-Rh3	-159.0(3)	C1-N2-C3-C2	-0.3(7)
Rh2-N6-N7-Rh3	40.1(6)	C16-N2-C3-C2	178.0(5)
C37-Rh3-N7-C33	45.3(4)	C1-N1-C4-C9	-100.4(6)
C74-Rh3-N7-C33	-40.5(7)	C2-N1-C4-C9	75.4(7)
C75-Rh3-N7-C33	149.1(9)	C1-N1-C4-C5	80.6(7)
C71-Rh3-N7-C33	-109.9(4)	C2-N1-C4-C5	-103.6(6)
C70-Rh3-N7-C33	-144.5(4)	C9-C4-C5-C6	0.7(10)
C37-Rh3-N7-N6	-157.9(4)	N1-C4-C5-C6	179.7(7)
C74-Rh3-N7-N6	116.3(6)	C9-C4-C5-C10	-174.3(5)
C75-Rh3-N7-N6	-54.1(11)	N1-C4-C5-C10	4.7(9)
C71-Rh3-N7-N6	46.9(4)	C4-C5-C6-C7	3.9(13)

C10-C5-C6-C7	178.8(8)	C24-Rh1-C19-C26	23.3(5)
C5-C6-C7-C8	-7.1(15)	C26-C19-C20-C21	5.4(10)
C6-C7-C8-C9	5.8(13)	Rh1-C19-C20-C21	105.4(6)
C7-C8-C9-C4	-1.4(10)	C26-C19-C20-Rh1	-100.0(6)
C7-C8-C9-C13	177.3(7)	C1-Rh1-C20-C19	-96.1(4)
C5-C4-C9-C8	-1.8(8)	N3-Rh1-C20-C19	178.5(4)
N1-C4-C9-C8	179.2(5)	C23-Rh1-C20-C19	113.7(4)
C5-C4-C9-C13	179.6(6)	C24-Rh1-C20-C19	77.6(4)
N1-C4-C9-C13	0.6(8)	C1-Rh1-C20-C21	143.4(5)
C4-C5-C10-C11	-163.5(8)	N3-Rh1-C20-C21	58.0(8)
C6-C5-C10-C11	21.8(12)	C19-Rh1-C20-C21	-120.5(7)
C4-C5-C10-C12	76.4(9)	C23-Rh1-C20-C21	-6.8(6)
C6-C5-C10-C12	-98.4(10)	C24-Rh1-C20-C21	-43.0(6)
C8-C9-C13-C14	-45.3(8)	C19-C20-C21-C22	-90.3(9)
C4-C9-C13-C14	133.3(7)	Rh1-C20-C21-C22	-7.7(9)
C8-C9-C13-C15	80.3(8)	C20-C21-C22-C23	26.6(10)
C4-C9-C13-C15	-101.1(6)	C21-C22-C23-C24	50.4(10)
C1-N2-C16-C17	-55.7(8)	C21-C22-C23-Rh1	-31.8(9)
C3-N2-C16-C17	126.2(6)	C1-Rh1-C23-C24	177.4(4)
N3#1-N3-C17-C18	-0.6(6)	N3-Rh1-C23-C24	101.1(4)
Rh1-N3-C17-C18	-168.5(3)	C20-Rh1-C23-C24	-100.5(4)
N3#1-N3-C17-C16	176.7(5)	C19-Rh1-C23-C24	-66.3(4)
Rh1-N3-C17-C16	8.8(7)	C1-Rh1-C23-C22	-61.8(8)
N2-C16-C17-N3	48.9(8)	N3-Rh1-C23-C22	-138.1(6)
N2-C16-C17-C18	-134.3(5)	C20-Rh1-C23-C22	20.3(6)
N3-C17-C18-C17#1	0.2(2)	C19-Rh1-C23-C22	54.5(6)
C16-C17-C18-C17#1	-176.8(7)	C24-Rh1-C23-C22	120.8(7)
C1-Rh1-C19-C20	87.0(4)	C22-C23-C24-C25	0.2(10)
N3-Rh1-C19-C20	-177.5(7)	Rh1-C23-C24-C25	102.4(6)
C23-Rh1-C19-C20	-66.2(4)	C22-C23-C24-Rh1	-102.2(6)
C24-Rh1-C19-C20	-99.8(4)	C1-Rh1-C24-C23	-169.1(16)
C1-Rh1-C19-C26	-149.8(5)	N3-Rh1-C24-C23	-79.5(4)
N3-Rh1-C19-C26	-54.3(12)	C20-Rh1-C24-C23	76.9(4)
C20-Rh1-C19-C26	123.1(7)	C19-Rh1-C24-C23	113.6(4)
C23-Rh1-C19-C26	57.0(6)	C1-Rh1-C24-C25	69.1(18)

N3-Rh1-C24-C25	158.6(5)	Rh2-N6-C31-C32	163.8(3)
C20-Rh1-C24-C25	-45.0(5)	N7-N6-C31-C30	-176.4(4)
C19-Rh1-C24-C25	-8.2(5)	Rh2-N6-C31-C30	-12.9(6)
C23-Rh1-C24-C25	-121.9(6)	N5-C30-C31-C32	139.8(5)
C23-C24-C25-C26	-89.1(8)	N5-C30-C31-N6	-44.1(6)
Rh1-C24-C25-C26	-8.8(8)	N6-C31-C32-C33	-0.4(6)
C20-C19-C26-C25	45.0(10)	C30-C31-C32-C33	176.0(5)
Rh1-C19-C26-C25	-35.1(8)	N6-N7-C33-C32	-0.1(6)
C24-C25-C26-C19	29.4(10)	Rh3-N7-C33-C32	161.4(3)
C28-N4-C27-N5	-0.7(5)	N6-N7-C33-C34	-174.9(5)
C38-N4-C27-N5	-176.2(4)	Rh3-N7-C33-C34	-13.4(7)
C28-N4-C27-Rh2	179.8(4)	C31-C32-C33-N7	0.3(6)
C38-N4-C27-Rh2	4.2(8)	C31-C32-C33-C34	174.6(6)
C29-N5-C27-N4	1.4(5)	C37-N8-C34-C33	55.1(7)
C30-N5-C27-N4	175.1(4)	C35-N8-C34-C33	-129.4(5)
C29-N5-C27-Rh2	-179.0(3)	N7-C33-C34-N8	-42.7(7)
C30-N5-C27-Rh2	-5.3(6)	C32-C33-C34-N8	143.6(5)
N6-Rh2-C27-N4	142.3(5)	C37-N8-C35-C36	-0.1(6)
C62-Rh2-C27-N4	-60.8(5)	C34-N8-C35-C36	-176.0(5)
C63-Rh2-C27-N4	-24.2(6)	N8-C35-C36-N9	-0.1(6)
C67-Rh2-C27-N4	-135.0(6)	C37-N9-C36-C35	0.2(6)
C66-Rh2-C27-N4	58(2)	C50-N9-C36-C35	175.6(5)
N6-Rh2-C27-N5	-37.2(4)	C35-N8-C37-N9	0.2(5)
C62-Rh2-C27-N5	119.7(4)	C34-N8-C37-N9	176.1(4)
C63-Rh2-C27-N5	156.4(4)	C35-N8-C37-Rh3	178.2(3)
C67-Rh2-C27-N5	45.5(6)	C34-N8-C37-Rh3	-5.9(7)
C66-Rh2-C27-N5	-121.9(18)	C36-N9-C37-N8	-0.2(5)
C27-N4-C28-C29	-0.2(6)	C50-N9-C37-N8	-175.3(4)
C38-N4-C28-C29	175.6(5)	C36-N9-C37-Rh3	-177.8(4)
N4-C28-C29-N5	1.1(6)	C50-N9-C37-Rh3	7.2(8)
C27-N5-C29-C28	-1.6(6)	C74-Rh3-C37-N8	118.7(5)
C30-N5-C29-C28	-175.3(5)	N7-Rh3-C37-N8	-35.9(4)
C27-N5-C30-C31	56.0(6)	C75-Rh3-C37-N8	155.4(4)
C29-N5-C30-C31	-131.1(5)	C71-Rh3-C37-N8	40.6(8)
N7-N6-C31-C32	0.4(6)	C70-Rh3-C37-N8	-130.7(12)

C74-Rh3-C37-N9	-64.1(6)	C55-C50-C51-C52	1.6(7)
N7-Rh3-C37-N9	141.3(5)	N9-C50-C51-C52	178.3(4)
C75-Rh3-C37-N9	-27.4(6)	C55-C50-C51-C56	-178.3(5)
C71-Rh3-C37-N9	-142.1(6)	N9-C50-C51-C56	-1.6(7)
C70-Rh3-C37-N9	46.5(16)	C50-C51-C52-C53	0.6(8)
C27-N4-C38-C43	100.4(6)	C56-C51-C52-C53	-179.5(5)
C28-N4-C38-C43	-74.8(6)	C51-C52-C53-C54	-2.0(9)
C27-N4-C38-C39	-83.6(7)	C52-C53-C54-C55	1.2(9)
C28-N4-C38-C39	101.3(6)	C51-C50-C55-C54	-2.3(8)
C43-C38-C39-C40	-4.4(8)	N9-C50-C55-C54	-179.0(5)
N4-C38-C39-C40	179.8(5)	C51-C50-C55-C59	176.0(5)
C43-C38-C39-C44	178.3(5)	N9-C50-C55-C59	-0.7(7)
N4-C38-C39-C44	2.5(8)	C53-C54-C55-C50	0.9(8)
C38-C39-C40-C41	2.4(10)	C53-C54-C55-C59	-177.4(5)
C44-C39-C40-C41	179.7(6)	C50-C51-C56-C58	-134.6(5)
C39-C40-C41-C42	0.2(11)	C52-C51-C56-C58	45.5(7)
C40-C41-C42-C43	-1.0(11)	C50-C51-C56-C57	104.0(6)
C39-C38-C43-C42	3.7(8)	C52-C51-C56-C57	-75.8(6)
N4-C38-C43-C42	179.6(5)	C50-C55-C59-C60	147.8(5)
C39-C38-C43-C47	-178.1(5)	C54-C55-C59-C60	-34.0(7)
N4-C38-C43-C47	-2.2(7)	C50-C55-C59-C61	-84.2(6)
C41-C42-C43-C38	-0.9(9)	C54-C55-C59-C61	94.0(6)
C41-C42-C43-C47	-179.3(6)	C27-Rh2-C62-C63	101.6(4)
C40-C39-C44-C45	83.4(17)	N6-Rh2-C62-C63	-176.4(4)
C38-C39-C44-C45	-99.4(16)	C67-Rh2-C62-C63	-106.7(4)
C40-C39-C44-C46	-34.2(15)	C66-Rh2-C62-C63	-73.1(4)
C38-C39-C44-C46	143.0(15)	C27-Rh2-C62-C69	-137.4(5)
C38-C43-C47-C49	-133.2(6)	N6-Rh2-C62-C69	-55.4(8)
C42-C43-C47-C49	45.0(7)	C63-Rh2-C62-C69	121.0(7)
C38-C43-C47-C48	102.8(6)	C67-Rh2-C62-C69	14.3(5)
C42-C43-C47-C48	-79.0(6)	C66-Rh2-C62-C69	47.9(6)
C37-N9-C50-C55	-84.8(6)	C69-C62-C63-C64	0.4(10)
C36-N9-C50-C55	100.6(6)	Rh2-C62-C63-C64	104.1(6)
C37-N9-C50-C51	98.3(6)	C69-C62-C63-Rh2	-103.6(6)
C36-N9-C50-C51	-76.3(6)	C27-Rh2-C63-C62	-81.0(4)

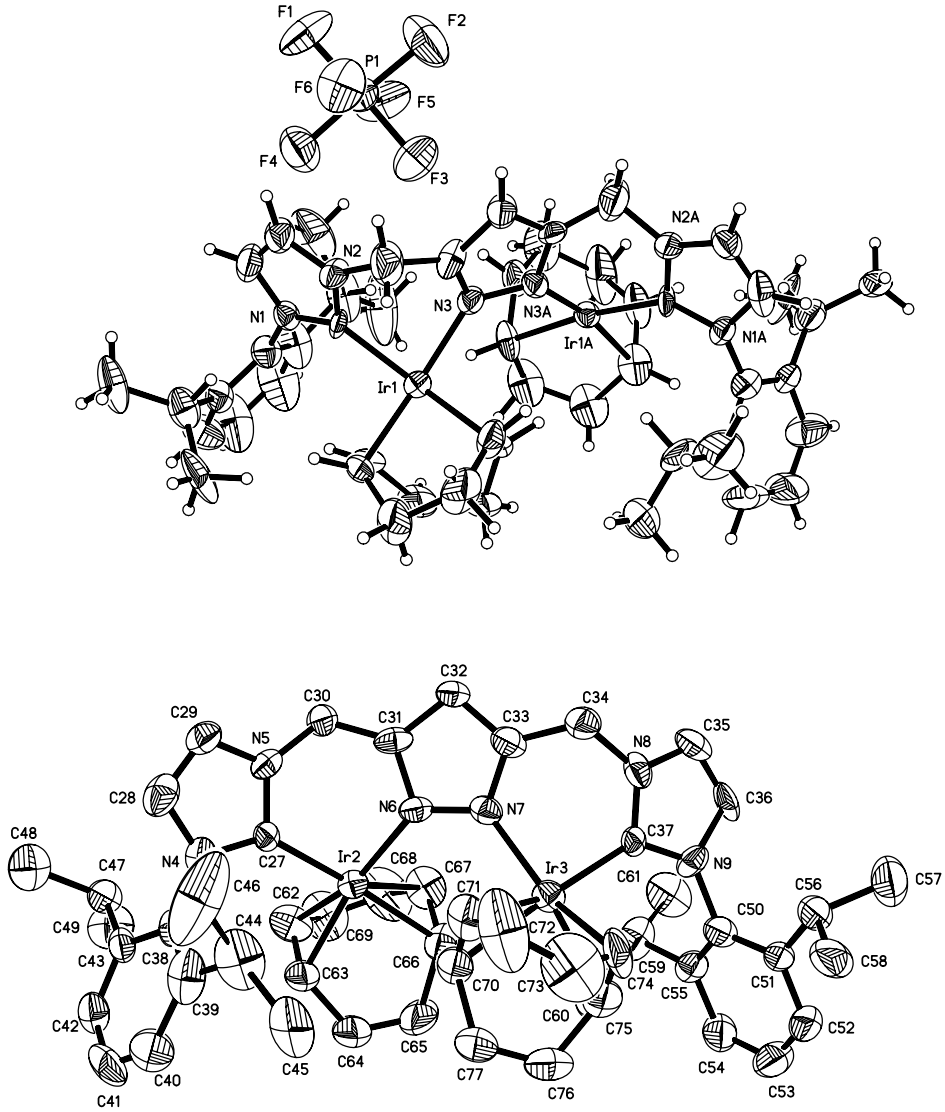
N6-Rh2-C63-C62	174.1(6)	Rh2-C67-C68-C69	14.6(11)
C67-Rh2-C63-C62	71.8(4)	C67-C68-C69-C62	-3.0(13)
C66-Rh2-C63-C62	105.0(4)	C63-C62-C69-C68	71.4(10)
C27-Rh2-C63-C64	157.4(4)	Rh2-C62-C69-C68	-10.6(10)
N6-Rh2-C63-C64	52.5(10)	C37-Rh3-C70-C71	173.6(12)
C62-Rh2-C63-C64	-121.6(6)	C74-Rh3-C70-C71	-75.8(5)
C67-Rh2-C63-C64	-49.8(5)	N7-Rh3-C70-C71	79.6(4)
C66-Rh2-C63-C64	-16.6(5)	C75-Rh3-C70-C71	-111.1(5)
C62-C63-C64-C65	-64.9(9)	C37-Rh3-C70-C77	-67.0(15)
Rh2-C63-C64-C65	16.9(8)	C74-Rh3-C70-C77	43.7(6)
C63-C64-C65-C66	-5.2(11)	N7-Rh3-C70-C77	-161.0(6)
C64-C65-C66-C67	71.6(10)	C75-Rh3-C70-C77	8.4(6)
C64-C65-C66-Rh2	-8.6(10)	C71-Rh3-C70-C77	119.4(8)
C27-Rh2-C66-C67	169.3(17)	C77-C70-C71-C72	-2.9(12)
N6-Rh2-C66-C67	84.9(4)	Rh3-C70-C71-C72	99.2(8)
C62-Rh2-C66-C67	-72.4(4)	C77-C70-C71-Rh3	-102.1(7)
C63-Rh2-C66-C67	-108.1(4)	C37-Rh3-C71-C70	-177.4(5)
C27-Rh2-C66-C65	-69(2)	C74-Rh3-C71-C70	102.4(5)
N6-Rh2-C66-C65	-153.4(6)	N7-Rh3-C71-C70	-101.6(4)
C62-Rh2-C66-C65	49.3(6)	C75-Rh3-C71-C70	67.1(5)
C63-Rh2-C66-C65	13.5(6)	C37-Rh3-C71-C72	57.6(9)
C67-Rh2-C66-C65	121.7(7)	C74-Rh3-C71-C72	-22.6(6)
C65-C66-C67-C68	1.5(11)	N7-Rh3-C71-C72	133.4(6)
Rh2-C66-C67-C68	102.0(7)	C75-Rh3-C71-C72	-57.9(6)
C65-C66-C67-Rh2	-100.6(6)	C70-Rh3-C71-C72	-125.0(8)
C27-Rh2-C67-C66	-177.7(4)	C70-C71-C72-C73	-54.5(13)
N6-Rh2-C67-C66	-95.9(4)	Rh3-C71-C72-C73	27.7(11)
C62-Rh2-C67-C66	105.9(4)	C71-C72-C73-C74	-17.0(15)
C63-Rh2-C67-C66	70.4(4)	C72-C73-C74-C75	78.0(13)
C27-Rh2-C67-C68	61.0(8)	C72-C73-C74-Rh3	-3.5(12)
N6-Rh2-C67-C68	142.8(6)	C37-Rh3-C74-C75	98.9(4)
C62-Rh2-C67-C68	-15.4(6)	N7-Rh3-C74-C75	-176.8(4)
C63-Rh2-C67-C68	-50.9(6)	C71-Rh3-C74-C75	-106.2(4)
C66-Rh2-C67-C68	-121.3(8)	C70-Rh3-C74-C75	-71.8(4)
C66-C67-C68-C69	-68.1(11)	C37-Rh3-C74-C73	-140.3(6)

N7-Rh3-C74-C73	-56.1(8)	C37-Rh3-C75-C76	152.5(5)
C75-Rh3-C74-C73	120.8(7)	C74-Rh3-C75-C76	-123.2(7)
C71-Rh3-C74-C73	14.5(6)	N7-Rh3-C75-C76	49.9(12)
C70-Rh3-C74-C73	48.9(6)	C71-Rh3-C75-C76	-50.6(5)
C73-C74-C75-C76	0.4(11)	C70-Rh3-C75-C76	-17.8(5)
Rh3-C74-C75-C76	100.9(7)	C74-C75-C76-C77	-53.7(11)
C73-C74-C75-Rh3	-100.6(7)	Rh3-C75-C76-C77	25.9(9)
C37-Rh3-C75-C74	-84.2(4)	C75-C76-C77-C70	-20.1(12)
N7-Rh3-C75-C74	173.1(8)	C71-C70-C77-C76	84.2(11)
C71-Rh3-C75-C74	72.7(4)	Rh3-C70-C77-C76	4.2(11)
C70-Rh3-C75-C74	105.4(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1

X-ray Structure of 6 (Bisiridium complex)



Data collection

A crystal (approximate dimensions 0.50 x 0.30 x 0.15 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker SMART Platform CCD diffractometer for a data collection at 173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 264 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 4.894 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.84 Å. Four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from the xyz centroids of 3447 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97 (Sheldrick, 1997).[4] The space group C2 was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The function SQUEEZE in the program PLATON[6] was used to model solvent accessible void space in the sample. 2309.3 $Å^3$ of solvent accessible void space, containing 769 electrons per unit cell was found. The sample was also modeled as a racemic twin (twin law: -1 0 0 0 -1 0 0 0 -1), in a 94:6 ratio. The final full matrix least squares refinement converged to $R1 = 0.0466$ and $wR2 = 0.1209$ (F^2, all data).

Structure description

The structure is the one suggested. The structure quality is questionable at best, but does show connectivity. The sample consists of one Ir dimer on a general position and a second Ir dimer on a 2-fold axis. The sample also contains one PF_6^- on a general position and a second PF_6^- disordered over an inversion center (50:50) as well as rotationally disordered (91:9). The sample also appears to have at least 4 CH_2Cl_2 molecules in the asymmetric unit, and may have as many as 5. As stated earlier, the SQUEEZE function from the program PLATON removed 2309.3 $Å^3$ of solvent accessible void space, containing 769 electrons per unit cell. Since *C*2 has a Z = 4, 16 CH_2Cl_2 molecules would have an electron count of 672 e^- and 20 CH_2Cl_2 molecules would have an electron count of 840 e^-. Several of the propyl groups were modeled as disordered over two positions: C10-C12 (86:14), C13-C15 (81:19), and C44-C46 (67:33). Some of the thermal ellipsoids still look large, but attempts to model further disorder were unsuccessful.

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1 SMART V5.054, Bruker Analytical X-ray Systems, Madison, WI (2001).

2 An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

3 SAINT+ V6.45, Bruker Analytical X-Ray Systems, Madison, WI (2003).

4 A. Altomare, M. C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna. Sir97: a new tool for crystal structure determination and refinement. *J. Appl. Cryst.* **32**, 115-119 (1998).

5 SHELXTL V6.14, Bruker Analytical X-Ray Systems, Madison, WI (2000).

6 A. L. Spek, *Acta. Cryst.* **A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, A. L. Spek (2000).

Tables for 6 (Bisiridium complex)

Table 1. Crystal data and structure refinement.

Identification code	zzzs	
Empirical formula	C_{51} H_{67} F_6 Ir_2 N_6 P	
Formula weight	1293.48	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*C*2	
Unit cell dimensions	*a* = 30.809(8) Å	α = 90°
	b = 18.203(5) Å	β = 91.924(4)°
	c = 16.420(4) Å	γ = 90°
Volume	9204(4) Å^3	
Z	6	
Density (calculated)	1.400 Mg/m^3	
Absorption coefficient	4.411 mm^{-1}	
F(000)	3828	
Crystal color, morphology	orange, block	
Crystal size	0.50 x 0.30 x 0.15 mm^3	
Theta range for data collection	1.24 to 25.08°	
Index ranges	$-36 \le h \le 36$, $-21 \le k \le 21$, $-19 \le l \le 19$	
Reflections collected	45218	
Independent reflections	16290 [*R*(int) = 0.0549]	
Observed reflections	14518	
Completeness to theta = 25.08°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.5575 and 0.2164	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	16290 / 230 / 935	
Goodness-of-fit on F^2	1.036	
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0466, *wR*2 = 0.1176	
R indices (all data)	*R*1 = 0.0525, *wR*2 = 0.1209	

Absolute structure parameter	0.059(9)
Largest diff. peak and hole	3.350 and -1.661 e.Å^{-3}

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

	x	y	z	U_{eq}
Ir1	5424(1)	7957(1)	4021(1)	33(1)
Ir2	8085(1)	8300(1)	2408(1)	32(1)
Ir3	8466(1)	7103(1)	363(1)	36(1)
P1	5389(1)	4043(2)	3505(2)	42(1)
F1	5148(3)	3589(4)	2798(5)	83(3)
F2	5205(3)	3547(5)	4212(6)	93(3)
F3	5639(2)	4510(4)	4203(5)	67(2)
F4	5596(3)	4531(5)	2838(5)	80(2)
F5	4984(2)	4587(4)	3583(6)	76(2)
F6	5784(3)	3485(4)	3460(5)	75(2)
P2	5035(4)	5294(2)	-58(6)	45(2)
F7	4624(5)	5666(8)	-491(9)	96(5)
F8	4835(5)	4507(6)	-244(11)	82(5)
F9	4799(5)	5311(9)	785(8)	67(4)
F10	5235(5)	6079(5)	130(11)	82(5)
F11	5446(4)	4920(8)	375(9)	96(5)
F12	5271(4)	5277(9)	-900(7)	67(4)
P2'	5035(4)	5294(2)	-58(6)	45(2)
F7'	4700(30)	5940(50)	-170(80)	96(5)
F8'	4750(30)	4800(60)	-660(50)	82(5)
F9'	4770(30)	4990(70)	680(40)	67(4)
F10'	5320(30)	5790(60)	550(60)	82(5)
F11'	5370(30)	4650(50)	50(80)	96(5)
F12'	5300(30)	5600(70)	-800(50)	67(4)
N1	5070(3)	7566(5)	2226(5)	36(2)
N2	5261(3)	6634(4)	2977(5)	39(2)
N3	5109(3)	7103(5)	4623(4)	35(2)
N4	8444(3)	7899(5)	4203(5)	44(2)

N5	8866(3)	8544(4)	3485(5)	37(2)
N6	8661(2)	8225(5)	1773(5)	35(2)
N7	8735(2)	8012(5)	1005(5)	34(2)
N8	9005(2)	8053(5)	-683(5)	39(2)
N9	8452(3)	7784(4)	-1424(5)	37(2)
C1	5236(3)	7402(6)	2970(5)	34(2)
C2	5011(4)	6900(8)	1777(7)	61(4)
C3	5111(4)	6336(7)	2251(7)	52(3)
C4	4999(2)	8248(4)	1872(4)	46(2)
C5	4631(2)	8641(5)	2075(5)	72(4)
C6	4561(3)	9341(6)	1758(7)	112(7)
C7	4859(4)	9647(4)	1238(7)	111(7)
C8	5227(3)	9254(4)	1036(5)	71(3)
C9	5297(2)	8555(4)	1353(4)	42(2)
C10	4277(4)	8299(9)	2614(7)	86(5)
C11	4002(9)	8873(17)	3010(12)	143(11)
C12	3989(6)	7797(16)	2118(16)	110(7)
C10'	4277(4)	8299(9)	2614(7)	86(5)
C11'	4050(40)	8280(90)	3410(40)	143(11)
C12'	4120(50)	7660(70)	2100(100)	110(7)
C13	5703(4)	8139(7)	1126(7)	59(3)
C14	6133(17)	8490(60)	1490(50)	98(7)
C15	5800(30)	7710(50)	320(40)	73(5)
C13'	5703(4)	8139(7)	1126(7)	59(3)
C14'	6104(5)	8657(14)	1149(15)	98(7)
C15'	5628(8)	7802(12)	247(8)	73(5)
C16	5415(6)	6228(9)	3710(8)	73(4)
C17	5174(4)	6438(6)	4418(6)	46(3)
C18	5000	5947(9)	5000	53(4)
C19	5597(4)	8939(7)	3399(8)	59(3)
C20	5948(4)	8500(8)	3448(7)	58(3)
C21	6355(4)	8605(14)	4021(8)	108(7)
C22	6305(4)	8141(13)	4838(8)	92(5)

C23	5849(3)	8054(8)	5077(6)	54(3)
C24	5549(4)	8627(6)	5104(6)	46(2)
C25	5642(6)	9426(7)	4887(9)	77(4)
C26	5554(6)	9605(7)	3999(9)	79(4)
C27	8466(3)	8228(5)	3458(5)	29(1)
C28	8823(3)	8042(8)	4666(7)	55(3)
C29	9075(3)	8461(7)	4211(7)	49(3)
C30	9019(4)	9011(6)	2844(6)	45(3)
C31	9008(3)	8630(5)	2049(6)	36(2)
C32	9310(3)	8701(6)	1412(6)	39(2)
C33	9126(3)	8258(7)	796(6)	46(2)
C34	9325(3)	8064(6)	25(6)	45(2)
C35	9066(3)	8406(6)	-1385(7)	47(3)
C36	8716(4)	8249(7)	-1849(6)	46(3)
C37	8638(3)	7674(5)	-663(5)	29(1)
C38	8089(3)	7493(6)	4518(6)	41(2)
C39	8058(5)	6724(7)	4341(8)	65(4)
C40	7665(7)	6404(9)	4698(10)	89(6)
C41	7405(6)	6735(9)	5180(9)	75(4)
C42	7473(4)	7464(7)	5365(7)	52(3)
C43	7830(3)	7820(6)	5064(6)	39(2)
C44	8335(5)	6351(7)	3795(9)	81(4)
C45	8162(12)	5653(11)	3251(14)	75(6)
C46	8809(13)	6210(20)	4260(30)	149(14)
C44'	8335(5)	6351(7)	3795(9)	81(4)
C45'	7980(17)	5850(30)	3280(30)	75(6)
C46'	8740(30)	5870(40)	4190(70)	149(14)
C47	7866(4)	8636(6)	5290(7)	45(3)
C48	8077(5)	8658(8)	6161(8)	66(4)
C49	7458(5)	9071(9)	5221(10)	74(4)
C50	8060(3)	7454(6)	-1800(6)	43(2)
C51	8122(4)	6853(6)	-2322(6)	43(2)
C52	7752(4)	6540(7)	-2681(7)	53(3)

C53	7344(5)	6832(8)	-2515(8)	68(4)
C54	7292(4)	7419(9)	-2026(7)	60(3)
C55	7665(4)	7762(5)	-1646(6)	41(2)
C56	8559(4)	6555(6)	-2503(7)	54(3)
C57	8708(6)	6744(9)	-3346(8)	81(5)
C58	8599(5)	5686(8)	-2430(10)	85(5)
C59	7607(4)	8430(7)	-1120(8)	60(3)
C60	7182(4)	8436(12)	-660(8)	86(5)
C61	7667(5)	9147(7)	-1657(10)	79(5)
C62	7585(4)	8806(7)	3053(9)	58(3)
C63	7484(3)	8068(7)	2947(7)	48(2)
C64	7152(4)	7814(10)	2328(8)	77(4)
C65	7230(4)	7887(13)	1531(9)	101(6)
C66	7678(3)	8225(8)	1311(6)	50(2)
C67	7815(4)	8921(7)	1444(8)	55(3)
C68	7521(6)	9543(9)	1824(11)	88(5)
C69	7389(5)	9433(9)	2590(10)	80(4)
C70	8178(4)	6601(6)	1416(7)	49(2)
C71	8598(5)	6404(6)	1382(7)	56(3)
C72	8793(9)	5703(11)	1128(11)	115(7)
C73	8639(7)	5468(8)	403(13)	103(5)
C74	8481(5)	6073(7)	-226(7)	65(3)
C75	8055(5)	6310(6)	-211(8)	59(3)
C76	7693(5)	6045(10)	347(9)	86(5)
C77	7842(6)	6050(9)	1182(9)	79(4)

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

Ir(1)-C(1)	2.067(9)	N(1)-C(4)	1.385(10)
Ir(1)-N(3)	2.097(9)	N(1)-C(2)	1.428(16)
Ir(1)-C(19)	2.136(12)	N(2)-C(3)	1.374(14)
Ir(1)-C(20)	2.138(10)	N(2)-C(1)	1.399(13)
Ir(1)-C(23)	2.144(10)	N(2)-C(16)	1.477(15)
Ir(1)-C(24)	2.180(10)	N(3)-C(17)	1.274(13)
Ir(2)-C(27)	2.056(9)	N(3)-N(3)#1	1.430(15)
Ir(2)-N(6)	2.092(7)	N(4)-C(27)	1.367(12)
Ir(2)-C(67)	2.093(11)	N(4)-C(28)	1.396(14)
Ir(2)-C(62)	2.110(11)	N(4)-C(38)	1.433(14)
Ir(2)-C(63)	2.121(10)	N(5)-C(29)	1.346(13)
Ir(2)-C(66)	2.164(10)	N(5)-C(27)	1.360(12)
Ir(3)-C(37)	2.063(9)	N(5)-C(30)	1.444(13)
Ir(3)-C(74)	2.111(12)	N(6)-N(7)	1.347(11)
Ir(3)-N(7)	2.115(8)	N(6)-C(31)	1.364(12)
Ir(3)-C(75)	2.121(12)	N(7)-C(33)	1.342(12)
Ir(3)-C(71)	2.131(10)	N(8)-C(37)	1.325(12)
Ir(3)-C(70)	2.173(11)	N(8)-C(35)	1.338(13)
P(1)-F(4)	1.563(8)	N(8)-C(34)	1.499(13)
P(1)-F(6)	1.587(7)	N(9)-C(37)	1.372(12)
P(1)-F(1)	1.589(7)	N(9)-C(36)	1.378(13)
P(1)-F(2)	1.590(8)	N(9)-C(50)	1.467(13)
P(1)-F(3)	1.603(7)	C(2)-C(3)	1.319(19)
P(1)-F(5)	1.603(7)	C(2)-H(2A)	0.9500
P(2)-F(10)	1.583(6)	C(3)-H(3A)	0.9500
P(2)-F(8)	1.584(6)	C(4)-C(5)	1.3900
P(2)-F(7)	1.585(6)	C(4)-C(9)	1.3900
P(2)-F(11)	1.585(6)	C(5)-C(6)	1.3900
P(2)-F(12)	1.585(6)	C(5)-C(10)	1.559(16)
P(2)-F(9)	1.585(6)	C(6)-C(7)	1.3900
N(1)-C(1)	1.342(12)	C(6)-H(6)	0.9500

C(7)-C(8)	1.3900	C(15')-H(15E)	0.9800
C(7)-H(7)	0.9500	C(15')-H(15F)	0.9800
C(8)-C(9)	1.3900	C(16)-C(17)	1.452(18)
C(8)-H(8)	0.9500	C(16)-H(16A)	0.9900
C(9)-C(13)	1.520(13)	C(16)-H(16B)	0.9900
C(10)-C(12)	1.495(19)	C(17)-C(18)	1.426(15)
C(10)-C(11)	1.506(18)	C(18)-C(17)#1	1.426(15)
C(10)-H(10)	1.0000	C(18)-H(18A)	0.9500
C(11)-H(11A)	0.9800	C(19)-C(20)	1.345(19)
C(11)-H(11B)	0.9800	C(19)-C(26)	1.570(18)
C(11)-H(11C)	0.9800	C(19)-H(19A)	1.0000
C(12)-H(12A)	0.9800	C(20)-C(21)	1.552(18)
C(12)-H(12B)	0.9800	C(20)-H(20A)	1.0000
C(12)-H(12C)	0.9800	C(21)-C(22)	1.60(2)
C(11')-H(11D)	0.9800	C(21)-H(21A)	0.9900
C(11')-H(11E)	0.9800	C(21)-H(21B)	0.9900
C(11')-H(11F)	0.9800	C(22)-C(23)	1.479(17)
C(12')-H(12D)	0.9800	C(22)-H(22A)	0.9900
C(12')-H(12E)	0.9800	C(22)-H(22B)	0.9900
C(12')-H(12F)	0.9800	C(23)-C(24)	1.396(17)
C(13)-C(14)	1.57(2)	C(23)-H(23A)	1.0000
C(13)-C(15)	1.57(2)	C(24)-C(25)	1.527(17)
C(13)-H(13)	1.0000	C(24)-H(24A)	1.0000
C(14)-H(14A)	0.9800	C(25)-C(26)	1.51(2)
C(14)-H(14B)	0.9800	C(25)-H(25A)	0.9900
C(14)-H(14C)	0.9800	C(25)-H(25B)	0.9900
C(15)-H(15A)	0.9800	C(26)-H(26A)	0.9900
C(15)-H(15B)	0.9800	C(26)-H(26B)	0.9900
C(15)-H(15C)	0.9800	C(28)-C(29)	1.334(17)
C(14')-H(14D)	0.9800	C(28)-H(28A)	0.9500
C(14')-H(14E)	0.9800	C(29)-H(29A)	0.9500
C(14')-H(14F)	0.9800	C(30)-C(31)	1.477(15)
C(15')-H(15D)	0.9800	C(30)-H(30A)	0.9900

C(30)-H(30B)	0.9900	C(46')-H(46D)	0.9800
C(31)-C(32)	1.431(14)	C(46')-H(46E)	0.9800
C(32)-C(33)	1.398(15)	C(46')-H(46F)	0.9800
C(32)-H(32A)	0.9500	C(47)-C(49)	1.488(17)
C(33)-C(34)	1.467(14)	C(47)-C(48)	1.552(18)
C(34)-H(34A)	0.9900	C(47)-H(47A)	1.0000
C(34)-H(34B)	0.9900	C(48)-H(48A)	0.9800
C(35)-C(36)	1.331(16)	C(48)-H(48B)	0.9800
C(35)-H(35A)	0.9500	C(48)-H(48C)	0.9800
C(36)-H(36A)	0.9500	C(49)-H(49A)	0.9800
C(38)-C(43)	1.357(15)	C(49)-H(49B)	0.9800
C(38)-C(39)	1.431(17)	C(49)-H(49C)	0.9800
C(39)-C(44)	1.43(2)	C(50)-C(55)	1.370(15)
C(39)-C(40)	1.48(2)	C(50)-C(51)	1.407(15)
C(40)-C(41)	1.29(2)	C(51)-C(52)	1.387(15)
C(40)-H(40A)	0.9500	C(51)-C(56)	1.491(17)
C(41)-C(42)	1.38(2)	C(52)-C(53)	1.40(2)
C(41)-H(41A)	0.9500	C(52)-H(52A)	0.9500
C(42)-C(43)	1.382(16)	C(53)-C(54)	1.348(19)
C(42)-H(42A)	0.9500	C(53)-H(53A)	0.9500
C(43)-C(47)	1.535(15)	C(54)-C(55)	1.434(16)
C(44)-C(45)	1.632(18)	C(54)-H(54A)	0.9500
C(44)-C(46)	1.65(2)	C(55)-C(59)	1.506(15)
C(44)-H(44)	1.0000	C(56)-C(57)	1.512(17)
C(45)-H(45A)	0.9800	C(56)-C(58)	1.59(2)
C(45)-H(45B)	0.9800	C(56)-H(56A)	1.0000
C(45)-H(45C)	0.9800	C(57)-H(57A)	0.9800
C(46)-H(46A)	0.9800	C(57)-H(57B)	0.9800
C(46)-H(46B)	0.9800	C(57)-H(57C)	0.9800
C(46)-H(46C)	0.9800	C(58)-H(58A)	0.9800
C(45')-H(45D)	0.9800	C(58)-H(58B)	0.9800
C(45')-H(45E)	0.9800	C(58)-H(58C)	0.9800
C(45')-H(45F)	0.9800	C(59)-C(60)	1.533(18)

C(59)-C(61)	1.59(2)	C(72)-C(73)	1.34(3)
C(59)-H(59A)	1.0000	C(72)-H(72A)	0.9900
C(60)-H(60A)	0.9800	C(72)-H(72B)	0.9900
C(60)-H(60B)	0.9800	C(73)-C(74)	1.58(2)
C(60)-H(60C)	0.9800	C(73)-H(73A)	0.9900
C(61)-H(61A)	0.9800	C(73)-H(73B)	0.9900
C(61)-H(61B)	0.9800	C(74)-C(75)	1.38(2)
C(61)-H(61C)	0.9800	C(74)-H(74A)	1.0000
C(62)-C(63)	1.388(18)	C(75)-C(76)	1.543(19)
C(62)-C(69)	1.489(19)	C(75)-H(75A)	1.0000
C(62)-H(62A)	1.0000	C(76)-C(77)	1.43(2)
C(63)-C(64)	1.493(16)	C(76)-H(76A)	0.9900
C(63)-H(63A)	1.0000	C(76)-H(76B)	0.9900
C(64)-C(65)	1.345(19)	C(77)-H(77A)	0.9900
C(64)-H(64A)	0.9900	C(77)-H(77B)	0.9900
C(64)-H(64B)	0.9900		
C(65)-C(66)	1.565(17)	C(1)-Ir(1)-N(3)	84.8(3)
C(65)-H(65A)	0.9900	C(1)-Ir(1)-C(19)	94.4(4)
C(65)-H(65B)	0.9900	N(3)-Ir(1)-C(19)	166.6(4)
C(66)-C(67)	1.350(18)	C(1)-Ir(1)-C(20)	93.1(4)
C(66)-H(66A)	1.0000	N(3)-Ir(1)-C(20)	156.7(5)
C(67)-C(68)	1.592(18)	C(19)-Ir(1)-C(20)	36.7(5)
C(67)-H(67A)	1.0000	C(1)-Ir(1)-C(23)	149.6(5)
C(68)-C(69)	1.35(2)	N(3)-Ir(1)-C(23)	87.7(4)
C(68)-H(68A)	0.9900	C(19)-Ir(1)-C(23)	99.3(5)
C(68)-H(68B)	0.9900	C(20)-Ir(1)-C(23)	82.4(5)
C(69)-H(69A)	0.9900	C(1)-Ir(1)-C(24)	172.7(5)
C(69)-H(69B)	0.9900	N(3)-Ir(1)-C(24)	95.8(3)
C(70)-C(71)	1.346(18)	C(19)-Ir(1)-C(24)	83.2(4)
C(70)-C(77)	1.482(17)	C(20)-Ir(1)-C(24)	89.2(4)
C(70)-H(70A)	1.0000	C(23)-Ir(1)-C(24)	37.7(5)
C(71)-C(72)	1.48(2)	C(27)-Ir(2)-N(6)	86.8(3)
C(71)-H(71A)	1.0000	C(27)-Ir(2)-C(67)	150.3(5)

N(6)-Ir(2)-C(67)	88.8(4)	F(1)-P(1)-F(2)	94.0(6)
C(27)-Ir(2)-C(62)	90.7(4)	F(4)-P(1)-F(3)	90.0(5)
N(6)-Ir(2)-C(62)	157.6(4)	F(6)-P(1)-F(3)	91.2(4)
C(67)-Ir(2)-C(62)	82.3(5)	F(1)-P(1)-F(3)	178.6(5)
C(27)-Ir(2)-C(63)	96.8(4)	F(2)-P(1)-F(3)	87.4(5)
N(6)-Ir(2)-C(63)	164.1(4)	F(4)-P(1)-F(5)	92.4(5)
C(67)-Ir(2)-C(63)	95.4(5)	F(6)-P(1)-F(5)	177.6(5)
C(62)-Ir(2)-C(63)	38.3(5)	F(1)-P(1)-F(5)	91.9(4)
C(27)-Ir(2)-C(66)	172.7(5)	F(2)-P(1)-F(5)	89.7(5)
N(6)-Ir(2)-C(66)	93.3(3)	F(3)-P(1)-F(5)	88.4(4)
C(67)-Ir(2)-C(66)	36.9(5)	F(10)-P(2)-F(8)	179.9(3)
C(62)-Ir(2)-C(66)	91.9(5)	F(10)-P(2)-F(7)	90.0(2)
C(63)-Ir(2)-C(66)	81.1(4)	F(8)-P(2)-F(7)	90.0(2)
C(37)-Ir(3)-C(74)	93.6(4)	F(10)-P(2)-F(11)	90.0(2)
C(37)-Ir(3)-N(7)	84.6(3)	F(8)-P(2)-F(11)	90.0(2)
C(74)-Ir(3)-N(7)	155.4(5)	F(7)-P(2)-F(11)	179.9(3)
C(37)-Ir(3)-C(75)	98.4(4)	F(10)-P(2)-F(12)	90.0(2)
C(74)-Ir(3)-C(75)	38.1(6)	F(8)-P(2)-F(12)	90.1(2)
N(7)-Ir(3)-C(75)	166.3(4)	F(7)-P(2)-F(12)	90.0(2)
C(37)-Ir(3)-C(71)	153.4(4)	F(11)-P(2)-F(12)	90.0(2)
C(74)-Ir(3)-C(71)	79.8(5)	F(10)-P(2)-F(9)	90.0(2)
N(7)-Ir(3)-C(71)	90.9(4)	F(8)-P(2)-F(9)	90.0(2)
C(75)-Ir(3)-C(71)	92.2(5)	F(7)-P(2)-F(9)	90.0(2)
C(37)-Ir(3)-C(70)	170.1(4)	F(11)-P(2)-F(9)	90.0(2)
C(74)-Ir(3)-C(70)	90.4(4)	F(12)-P(2)-F(9)	179.9(3)
N(7)-Ir(3)-C(70)	95.5(4)	C(1)-N(1)-C(4)	129.1(8)
C(75)-Ir(3)-C(70)	79.2(4)	C(1)-N(1)-C(2)	108.6(9)
C(71)-Ir(3)-C(70)	36.4(5)	C(4)-N(1)-C(2)	122.0(8)
F(4)-P(1)-F(6)	90.0(5)	C(3)-N(2)-C(1)	111.8(9)
F(4)-P(1)-F(1)	88.6(5)	C(3)-N(2)-C(16)	126.7(10)
F(6)-P(1)-F(1)	88.5(4)	C(1)-N(2)-C(16)	121.5(9)
F(4)-P(1)-F(2)	176.6(6)	C(17)-N(3)-N(3)#1	108.1(6)
F(6)-P(1)-F(2)	87.9(5)	C(17)-N(3)-Ir(1)	120.1(7)

N(3)#1-N(3)-Ir(1)	130.3(3)	C(6)-C(5)-C(10)	118.4(7)
C(27)-N(4)-C(28)	110.0(9)	C(5)-C(6)-C(7)	120.0
C(27)-N(4)-C(38)	127.5(8)	C(5)-C(6)-H(6)	120.0
C(28)-N(4)-C(38)	122.4(9)	C(7)-C(6)-H(6)	120.0
C(29)-N(5)-C(27)	112.9(8)	C(8)-C(7)-C(6)	120.0
C(29)-N(5)-C(30)	123.6(9)	C(8)-C(7)-H(7)	120.0
C(27)-N(5)-C(30)	122.9(8)	C(6)-C(7)-H(7)	120.0
N(7)-N(6)-C(31)	108.2(7)	C(7)-C(8)-C(9)	120.0
N(7)-N(6)-Ir(2)	131.2(5)	C(7)-C(8)-H(8)	120.0
C(31)-N(6)-Ir(2)	117.9(6)	C(9)-C(8)-H(8)	120.0
C(33)-N(7)-N(6)	108.9(8)	C(8)-C(9)-C(4)	120.0
C(33)-N(7)-Ir(3)	118.3(7)	C(8)-C(9)-C(13)	118.9(6)
N(6)-N(7)-Ir(3)	128.1(6)	C(4)-C(9)-C(13)	121.1(6)
C(37)-N(8)-C(35)	114.6(8)	C(12)-C(10)-C(11)	109.2(17)
C(37)-N(8)-C(34)	121.7(8)	C(12)-C(10)-C(5)	110.4(13)
C(35)-N(8)-C(34)	123.7(9)	C(11)-C(10)-C(5)	112.5(16)
C(37)-N(9)-C(36)	108.4(8)	C(12)-C(10)-H(10)	108.2
C(37)-N(9)-C(50)	129.6(8)	C(11)-C(10)-H(10)	108.2
C(36)-N(9)-C(50)	121.9(9)	C(5)-C(10)-H(10)	108.2
N(1)-C(1)-N(2)	104.4(8)	C(10)-C(11)-H(11A)	109.5
N(1)-C(1)-Ir(1)	137.7(8)	C(10)-C(11)-H(11B)	109.5
N(2)-C(1)-Ir(1)	117.9(7)	H(11A)-C(11)-H(11B)	109.5
C(3)-C(2)-N(1)	109.4(10)	C(10)-C(11)-H(11C)	109.5
C(3)-C(2)-H(2A)	125.3	H(11A)-C(11)-H(11C)	109.5
N(1)-C(2)-H(2A)	125.3	H(11B)-C(11)-H(11C)	109.5
C(2)-C(3)-N(2)	105.7(10)	C(10)-C(12)-H(12A)	109.5
C(2)-C(3)-H(3A)	127.2	C(10)-C(12)-H(12B)	109.5
N(2)-C(3)-H(3A)	127.2	H(12A)-C(12)-H(12B)	109.5
N(1)-C(4)-C(5)	118.7(6)	C(10)-C(12)-H(12C)	109.5
N(1)-C(4)-C(9)	121.2(6)	H(12A)-C(12)-H(12C)	109.5
C(5)-C(4)-C(9)	120.0	H(12B)-C(12)-H(12C)	109.5
C(4)-C(5)-C(6)	120.0	H(11D)-C(11')-H(11E)	109.5
C(4)-C(5)-C(10)	121.5(7)	H(11D)-C(11')-H(11F)	109.5

H(11E)-C(11')-H(11F)	109.5	H(16A)-C(16)-H(16B)	108.0
H(12D)-C(12')-H(12E)	109.5	N(3)-C(17)-C(18)	110.7(10)
H(12D)-C(12')-H(12F)	109.5	N(3)-C(17)-C(16)	123.4(11)
H(12E)-C(12')-H(12F)	109.5	C(18)-C(17)-C(16)	125.9(11)
C(9)-C(13)-C(14)	113(4)	C(17)-C(18)-C(17)#1	102.4(13)
C(9)-C(13)-C(15)	129(3)	C(17)-C(18)-H(18A)	128.8
C(14)-C(13)-C(15)	111(3)	C(17)#1-C(18)-H(18A)	128.8
C(9)-C(13)-H(13)	99.1	C(20)-C(19)-C(26)	120.4(13)
C(14)-C(13)-H(13)	99.1	C(20)-C(19)-Ir(1)	71.7(7)
C(15)-C(13)-H(13)	99.1	C(26)-C(19)-Ir(1)	108.5(8)
C(13)-C(14)-H(14A)	109.5	C(20)-C(19)-H(19A)	115.9
C(13)-C(14)-H(14B)	109.5	C(26)-C(19)-H(19A)	115.9
H(14A)-C(14)-H(14B)	109.5	Ir(1)-C(19)-H(19A)	115.9
C(13)-C(14)-H(14C)	109.5	C(19)-C(20)-C(21)	126.4(13)
H(14A)-C(14)-H(14C)	109.5	C(19)-C(20)-Ir(1)	71.6(6)
H(14B)-C(14)-H(14C)	109.5	C(21)-C(20)-Ir(1)	113.3(9)
C(13)-C(15)-H(15A)	109.5	C(19)-C(20)-H(20A)	112.9
C(13)-C(15)-H(15B)	109.5	C(21)-C(20)-H(20A)	112.9
H(15A)-C(15)-H(15B)	109.5	Ir(1)-C(20)-H(20A)	112.9
C(13)-C(15)-H(15C)	109.5	C(20)-C(21)-C(22)	110.3(11)
H(15A)-C(15)-H(15C)	109.5	C(20)-C(21)-H(21A)	109.6
H(15B)-C(15)-H(15C)	109.5	C(22)-C(21)-H(21A)	109.6
H(14D)-C(14')-H(14E)	109.5	C(20)-C(21)-H(21B)	109.6
H(14D)-C(14')-H(14F)	109.5	C(22)-C(21)-H(21B)	109.6
H(14E)-C(14')-H(14F)	109.5	H(21A)-C(21)-H(21B)	108.1
H(15D)-C(15')-H(15E)	109.5	C(23)-C(22)-C(21)	113.4(12)
H(15D)-C(15')-H(15F)	109.5	C(23)-C(22)-H(22A)	108.9
H(15E)-C(15')-H(15F)	109.5	C(21)-C(22)-H(22A)	108.9
C(17)-C(16)-N(2)	111.2(12)	C(23)-C(22)-H(22B)	108.9
C(17)-C(16)-H(16A)	109.4	C(21)-C(22)-H(22B)	108.9
N(2)-C(16)-H(16A)	109.4	H(22A)-C(22)-H(22B)	107.7
C(17)-C(16)-H(16B)	109.4	C(24)-C(23)-C(22)	124.4(14)
N(2)-C(16)-H(16B)	109.4	C(24)-C(23)-Ir(1)	72.6(6)

C(22)-C(23)-Ir(1)	110.7(8)	C(31)-C(30)-H(30A)	109.3
C(24)-C(23)-H(23A)	113.9	N(5)-C(30)-H(30B)	109.3
C(22)-C(23)-H(23A)	113.9	C(31)-C(30)-H(30B)	109.3
Ir(1)-C(23)-H(23A)	113.9	H(30A)-C(30)-H(30B)	107.9
C(23)-C(24)-C(25)	125.0(12)	N(6)-C(31)-C(32)	109.2(8)
C(23)-C(24)-Ir(1)	69.8(6)	N(6)-C(31)-C(30)	122.8(9)
C(25)-C(24)-Ir(1)	111.8(8)	C(32)-C(31)-C(30)	127.4(9)
C(23)-C(24)-H(24A)	114.0	C(33)-C(32)-C(31)	102.6(8)
C(25)-C(24)-H(24A)	114.0	C(33)-C(32)-H(32A)	128.7
Ir(1)-C(24)-H(24A)	114.0	C(31)-C(32)-H(32A)	128.7
C(26)-C(25)-C(24)	113.7(10)	N(7)-C(33)-C(32)	110.8(9)
C(26)-C(25)-H(25A)	108.8	N(7)-C(33)-C(34)	123.0(10)
C(24)-C(25)-H(25A)	108.8	C(32)-C(33)-C(34)	126.3(9)
C(26)-C(25)-H(25B)	108.8	C(33)-C(34)-N(8)	113.0(8)
C(24)-C(25)-H(25B)	108.8	C(33)-C(34)-H(34A)	109.0
H(25A)-C(25)-H(25B)	107.7	N(8)-C(34)-H(34A)	109.0
C(25)-C(26)-C(19)	115.1(12)	C(33)-C(34)-H(34B)	109.0
C(25)-C(26)-H(26A)	108.5	N(8)-C(34)-H(34B)	109.0
C(19)-C(26)-H(26A)	108.5	H(34A)-C(34)-H(34B)	107.8
C(25)-C(26)-H(26B)	108.5	C(36)-C(35)-N(8)	104.8(9)
C(19)-C(26)-H(26B)	108.5	C(36)-C(35)-H(35A)	127.6
H(26A)-C(26)-H(26B)	107.5	N(8)-C(35)-H(35A)	127.6
N(5)-C(27)-N(4)	103.1(8)	C(35)-C(36)-N(9)	108.8(9)
N(5)-C(27)-Ir(2)	119.4(6)	C(35)-C(36)-H(36A)	125.6
N(4)-C(27)-Ir(2)	137.4(7)	N(9)-C(36)-H(36A)	125.6
C(29)-C(28)-N(4)	107.1(10)	N(8)-C(37)-N(9)	103.4(8)
C(29)-C(28)-H(28A)	126.5	N(8)-C(37)-Ir(3)	121.7(7)
N(4)-C(28)-H(28A)	126.5	N(9)-C(37)-Ir(3)	134.9(7)
C(28)-C(29)-N(5)	106.8(9)	C(43)-C(38)-C(39)	121.8(11)
C(28)-C(29)-H(29A)	126.6	C(43)-C(38)-N(4)	119.0(10)
N(5)-C(29)-H(29A)	126.6	C(39)-C(38)-N(4)	118.5(11)
N(5)-C(30)-C(31)	111.7(9)	C(44)-C(39)-C(38)	123.7(14)
N(5)-C(30)-H(30A)	109.3	C(44)-C(39)-C(40)	125.0(14)

C(38)-C(39)-C(40)	110.7(12)	H(45E)-C(45')-H(45F)	109.5
C(41)-C(40)-C(39)	126.1(14)	H(46D)-C(46')-H(46E)	109.5
C(41)-C(40)-H(40A)	117.0	H(46D)-C(46')-H(46F)	109.5
C(39)-C(40)-H(40A)	117.0	H(46E)-C(46')-H(46F)	109.5
C(40)-C(41)-C(42)	119.5(13)	C(49)-C(47)-C(43)	116.3(11)
C(40)-C(41)-H(41A)	120.3	C(49)-C(47)-C(48)	112.5(10)
C(42)-C(41)-H(41A)	120.3	C(43)-C(47)-C(48)	105.9(9)
C(41)-C(42)-C(43)	119.3(13)	C(49)-C(47)-H(47A)	107.3
C(41)-C(42)-H(42A)	120.3	C(43)-C(47)-H(47A)	107.3
C(43)-C(42)-H(42A)	120.3	C(48)-C(47)-H(47A)	107.3
C(38)-C(43)-C(42)	121.6(11)	C(47)-C(48)-H(48A)	109.5
C(38)-C(43)-C(47)	123.0(10)	C(47)-C(48)-H(48B)	109.5
C(42)-C(43)-C(47)	114.7(10)	H(48A)-C(48)-H(48B)	109.5
C(39)-C(44)-C(45)	121.5(18)	C(47)-C(48)-H(48C)	109.5
C(39)-C(44)-C(46)	109(2)	H(48A)-C(48)-H(48C)	109.5
C(45)-C(44)-C(46)	113.7(15)	H(48B)-C(48)-H(48C)	109.5
C(39)-C(44)-H(44)	103.5	C(47)-C(49)-H(49A)	109.5
C(45)-C(44)-H(44)	103.5	C(47)-C(49)-H(49B)	109.5
C(46)-C(44)-H(44)	103.5	H(49A)-C(49)-H(49B)	109.5
C(44)-C(45)-H(45A)	109.5	C(47)-C(49)-H(49C)	109.5
C(44)-C(45)-H(45B)	109.5	H(49A)-C(49)-H(49C)	109.5
H(45A)-C(45)-H(45B)	109.5	H(49B)-C(49)-H(49C)	109.5
C(44)-C(45)-H(45C)	109.5	C(55)-C(50)-C(51)	124.8(10)
H(45A)-C(45)-H(45C)	109.5	C(55)-C(50)-N(9)	118.6(10)
H(45B)-C(45)-H(45C)	109.5	C(51)-C(50)-N(9)	116.6(9)
C(44)-C(46)-H(46A)	109.5	C(52)-C(51)-C(50)	116.8(11)
C(44)-C(46)-H(46B)	109.5	C(52)-C(51)-C(56)	120.0(10)
H(46A)-C(46)-H(46B)	109.5	C(50)-C(51)-C(56)	123.2(9)
C(44)-C(46)-H(46C)	109.5	C(51)-C(52)-C(53)	119.5(12)
H(46A)-C(46)-H(46C)	109.5	C(51)-C(52)-H(52A)	120.3
H(46B)-C(46)-H(46C)	109.5	C(53)-C(52)-H(52A)	120.3
H(45D)-C(45')-H(45E)	109.5	C(54)-C(53)-C(52)	122.8(12)
H(45D)-C(45')-H(45F)	109.5	C(54)-C(53)-H(53A)	118.6

C(52)-C(53)-H(53A)	118.6	H(60A)-C(60)-H(60B)	109.5
C(53)-C(54)-C(55)	119.6(12)	C(59)-C(60)-H(60C)	109.5
C(53)-C(54)-H(54A)	120.2	H(60A)-C(60)-H(60C)	109.5
C(55)-C(54)-H(54A)	120.2	H(60B)-C(60)-H(60C)	109.5
C(50)-C(55)-C(54)	116.5(10)	C(59)-C(61)-H(61A)	109.5
C(50)-C(55)-C(59)	124.1(10)	C(59)-C(61)-H(61B)	109.5
C(54)-C(55)-C(59)	119.5(11)	H(61A)-C(61)-H(61B)	109.5
C(51)-C(56)-C(57)	113.7(12)	C(59)-C(61)-H(61C)	109.5
C(51)-C(56)-C(58)	114.5(10)	H(61A)-C(61)-H(61C)	109.5
C(57)-C(56)-C(58)	105.7(10)	H(61B)-C(61)-H(61C)	109.5
C(51)-C(56)-H(56A)	107.5	C(63)-C(62)-C(69)	126.4(13)
C(57)-C(56)-H(56A)	107.5	C(63)-C(62)-Ir(2)	71.3(7)
C(58)-C(56)-H(56A)	107.5	C(69)-C(62)-Ir(2)	111.6(9)
C(56)-C(57)-H(57A)	109.5	C(63)-C(62)-H(62A)	113.3
C(56)-C(57)-H(57B)	109.5	C(69)-C(62)-H(62A)	113.3
H(57A)-C(57)-H(57B)	109.5	Ir(2)-C(62)-H(62A)	113.3
C(56)-C(57)-H(57C)	109.5	C(62)-C(63)-C(64)	122.1(12)
H(57A)-C(57)-H(57C)	109.5	C(62)-C(63)-Ir(2)	70.4(7)
H(57B)-C(57)-H(57C)	109.5	C(64)-C(63)-Ir(2)	111.5(8)
C(56)-C(58)-H(58A)	109.5	C(62)-C(63)-H(63A)	115.0
C(56)-C(58)-H(58B)	109.5	C(64)-C(63)-H(63A)	115.0
H(58A)-C(58)-H(58B)	109.5	Ir(2)-C(63)-H(63A)	115.0
C(56)-C(58)-H(58C)	109.5	C(65)-C(64)-C(63)	119.4(11)
H(58A)-C(58)-H(58C)	109.5	C(65)-C(64)-H(64A)	107.5
H(58B)-C(58)-H(58C)	109.5	C(63)-C(64)-H(64A)	107.5
C(55)-C(59)-C(60)	114.1(12)	C(65)-C(64)-H(64B)	107.5
C(55)-C(59)-C(61)	109.0(10)	C(63)-C(64)-H(64B)	107.5
C(60)-C(59)-C(61)	112.6(12)	H(64A)-C(64)-H(64B)	107.0
C(55)-C(59)-H(59A)	106.9	C(64)-C(65)-C(66)	116.8(11)
C(60)-C(59)-H(59A)	106.9	C(64)-C(65)-H(65A)	108.1
C(61)-C(59)-H(59A)	106.9	C(66)-C(65)-H(65A)	108.1
C(59)-C(60)-H(60A)	109.5	C(64)-C(65)-H(65B)	108.1
C(59)-C(60)-H(60B)	109.5	C(66)-C(65)-H(65B)	108.1

H(65A)-C(65)-H(65B)	107.3	C(72)-C(71)-Ir(3)	111.3(10)
C(67)-C(66)-C(65)	127.3(12)	C(70)-C(71)-H(71A)	111.7
C(67)-C(66)-Ir(2)	68.7(6)	C(72)-C(71)-H(71A)	111.7
C(65)-C(66)-Ir(2)	108.9(8)	Ir(3)-C(71)-H(71A)	111.7
C(67)-C(66)-H(66A)	114.0	C(73)-C(72)-C(71)	113.1(16)
C(65)-C(66)-H(66A)	114.0	C(73)-C(72)-H(72A)	109.0
Ir(2)-C(66)-H(66A)	114.0	C(71)-C(72)-H(72A)	109.0
C(66)-C(67)-C(68)	123.4(12)	C(73)-C(72)-H(72B)	109.0
C(66)-C(67)-Ir(2)	74.4(7)	C(71)-C(72)-H(72B)	109.0
C(68)-C(67)-Ir(2)	107.7(9)	H(72A)-C(72)-H(72B)	107.8
C(66)-C(67)-H(67A)	114.6	C(72)-C(73)-C(74)	116.9(13)
C(68)-C(67)-H(67A)	114.6	C(72)-C(73)-H(73A)	108.1
Ir(2)-C(67)-H(67A)	114.6	C(74)-C(73)-H(73A)	108.1
C(69)-C(68)-C(67)	117.0(12)	C(72)-C(73)-H(73B)	108.1
C(69)-C(68)-H(68A)	108.1	C(74)-C(73)-H(73B)	108.1
C(67)-C(68)-H(68A)	108.1	H(73A)-C(73)-H(73B)	107.3
C(69)-C(68)-H(68B)	108.1	C(75)-C(74)-C(73)	118.6(12)
C(67)-C(68)-H(68B)	108.1	C(75)-C(74)-Ir(3)	71.3(7)
H(68A)-C(68)-H(68B)	107.3	C(73)-C(74)-Ir(3)	109.4(9)
C(68)-C(69)-C(62)	117.4(11)	C(75)-C(74)-H(74A)	116.4
C(68)-C(69)-H(69A)	107.9	C(73)-C(74)-H(74A)	116.4
C(62)-C(69)-H(69A)	107.9	Ir(3)-C(74)-H(74A)	116.4
C(68)-C(69)-H(69B)	107.9	C(74)-C(75)-C(76)	128.2(13)
C(62)-C(69)-H(69B)	107.9	C(74)-C(75)-Ir(3)	70.5(7)
H(69A)-C(69)-H(69B)	107.2	C(76)-C(75)-Ir(3)	112.4(9)
C(71)-C(70)-C(77)	118.3(12)	C(74)-C(75)-H(75A)	112.6
C(71)-C(70)-Ir(3)	70.1(7)	C(76)-C(75)-H(75A)	112.6
C(77)-C(70)-Ir(3)	112.2(8)	Ir(3)-C(75)-H(75A)	112.6
C(71)-C(70)-H(70A)	116.0	C(77)-C(76)-C(75)	110.6(12)
C(77)-C(70)-H(70A)	116.0	C(77)-C(76)-H(76A)	109.5
Ir(3)-C(70)-H(70A)	116.0	C(75)-C(76)-H(76A)	109.5
C(70)-C(71)-C(72)	130.0(16)	C(77)-C(76)-H(76B)	109.5
C(70)-C(71)-Ir(3)	73.5(7)	C(75)-C(76)-H(76B)	109.5

H(76A)-C(76)-H(76B)	108.1	C(76)-C(77)-H(77B)	108.1
C(76)-C(77)-C(70)	116.8(11)	C(70)-C(77)-H(77B)	108.1
C(76)-C(77)-H(77A)	108.1	H(77A)-C(77)-H(77B)	107.3
C(70)-C(77)-H(77A)	108.1		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ir1	33(1)	33(1)	33(1)	-2(1)	0(1)	0(1)
Ir2	31(1)	30(1)	36(1)	1(1)	-2(1)	1(1)
Ir3	39(1)	29(1)	39(1)	1(1)	4(1)	0(1)
P1	41(1)	39(2)	46(2)	-4(1)	2(1)	4(1)
F1	91(6)	65(5)	90(6)	-35(4)	-41(5)	6(4)
F2	104(7)	89(6)	91(6)	23(5)	50(5)	1(5)
F3	62(4)	71(5)	68(5)	-15(4)	-16(3)	11(4)
F4	104(6)	72(5)	66(5)	12(4)	24(4)	-6(5)
F5	45(4)	61(5)	120(7)	-23(4)	-20(4)	13(3)
F6	70(5)	70(5)	84(5)	-10(4)	3(4)	34(4)
P2	60(4)	34(2)	41(3)	12(5)	12(3)	3(5)
F7	94(9)	128(13)	66(10)	52(8)	1(7)	21(9)
F8	121(11)	55(7)	73(10)	-14(7)	19(8)	-42(7)
F9	76(7)	73(9)	54(5)	5(11)	17(5)	-15(12)
F10	121(11)	55(7)	73(10)	-14(7)	19(8)	-42(7)
F11	94(9)	128(13)	66(10)	52(8)	1(7)	21(9)
F12	76(7)	73(9)	54(5)	5(11)	17(5)	-15(12)
P2'	60(4)	34(2)	41(3)	12(5)	12(3)	3(5)
F7'	94(9)	128(13)	66(10)	52(8)	1(7)	21(9)
F8'	121(11)	55(7)	73(10)	-14(7)	19(8)	-42(7)
F9'	76(7)	73(9)	54(5)	5(11)	17(5)	-15(12)
F10'	121(11)	55(7)	73(10)	-14(7)	19(8)	-42(7)
F11'	94(9)	128(13)	66(10)	52(8)	1(7)	21(9)
F12'	76(7)	73(9)	54(5)	5(11)	17(5)	-15(12)
N1	40(4)	38(5)	30(4)	8(4)	0(3)	0(4)
N2	57(5)	24(4)	36(5)	-2(3)	-3(4)	5(4)
N3	44(4)	31(4)	32(4)	-5(4)	2(3)	0(4)
N4	53(5)	46(5)	35(4)	-3(4)	1(4)	4(4)

N5	36(4)	37(5)	36(4)	-8(3)	-6(3)	-4(3)
N6	28(4)	32(4)	44(5)	-6(4)	0(3)	-6(3)
N7	28(4)	36(4)	38(4)	8(4)	1(3)	0(3)
N8	33(4)	55(6)	29(4)	3(4)	7(3)	11(4)
N9	50(5)	23(4)	38(5)	-6(3)	-7(4)	-5(3)
C1	55(6)	35(5)	13(4)	-4(4)	-1(4)	-5(5)
C2	52(7)	98(12)	34(6)	-18(6)	4(5)	-13(7)
C3	77(8)	39(6)	42(6)	-5(5)	13(6)	2(6)
C4	50(5)	45(5)	43(6)	6(5)	0(4)	3(4)
C5	61(7)	99(9)	56(8)	25(7)	6(5)	38(7)
C6	111(12)	124(13)	103(13)	48(11)	32(9)	86(12)
C7	169(17)	73(10)	93(12)	36(9)	41(11)	69(10)
C8	74(8)	48(6)	90(10)	24(6)	7(6)	0(5)
C9	47(5)	45(5)	32(5)	8(4)	-5(4)	4(4)
C10	47(6)	155(14)	56(8)	38(8)	0(5)	53(8)
C11	136(19)	230(30)	66(13)	24(13)	39(12)	115(18)
C12	48(12)	170(20)	108(14)	48(12)	14(11)	11(12)
C10'	47(6)	155(14)	56(8)	38(8)	0(5)	53(8)
C11'	136(19)	230(30)	66(13)	24(13)	39(12)	115(18)
C12'	48(12)	170(20)	108(14)	48(12)	14(11)	11(12)
C13	56(6)	73(10)	47(6)	18(5)	5(5)	12(6)
C14	53(8)	200(20)	46(12)	71(11)	5(8)	-6(10)
C15	64(13)	106(14)	51(7)	31(7)	27(7)	54(10)
C13'	56(6)	73(10)	47(6)	18(5)	5(5)	12(6)
C14'	53(8)	200(20)	46(12)	71(11)	5(8)	-6(10)
C15'	64(13)	106(14)	51(7)	31(7)	27(7)	54(10)
C16	111(12)	67(9)	41(7)	13(6)	-1(7)	0(8)
C17	75(7)	29(5)	31(5)	-3(4)	-12(5)	23(5)
C18	93(13)	29(8)	37(8)	0	4(8)	0
C19	63(6)	55(7)	59(7)	-3(5)	8(6)	-32(5)
C20	42(5)	91(9)	42(5)	-13(6)	19(4)	-35(5)
C21	39(6)	230(20)	58(8)	-23(9)	16(5)	-40(9)
C22	36(5)	173(18)	65(7)	-21(8)	-17(5)	-13(8)

C23	41(5)	87(8)	34(5)	-11(6)	-4(4)	1(5)
C24	63(6)	45(5)	32(5)	-13(4)	17(5)	-29(4)
C25	110(11)	48(6)	75(7)	-17(6)	25(8)	-45(7)
C26	126(13)	29(6)	85(8)	-3(5)	28(9)	-29(7)
C27	37(3)	23(3)	27(3)	-8(3)	8(3)	-1(3)
C28	41(6)	77(9)	46(6)	-8(6)	-9(5)	15(6)
C29	36(5)	65(8)	44(6)	-5(5)	-7(5)	-5(5)
C30	50(6)	51(7)	33(5)	13(5)	-7(4)	-15(5)
C31	27(5)	25(5)	57(6)	-6(4)	-2(4)	-1(4)
C32	32(5)	42(6)	42(6)	2(4)	2(4)	-13(4)
C33	37(5)	56(6)	45(6)	7(5)	6(4)	-8(5)
C34	34(5)	52(7)	49(6)	-5(5)	4(4)	-1(4)
C35	42(6)	47(7)	55(7)	4(5)	21(5)	-7(5)
C36	67(7)	44(6)	27(5)	10(5)	23(5)	-4(6)
C37	37(3)	23(3)	27(3)	-8(3)	8(3)	-1(3)
C38	52(6)	50(6)	20(5)	4(4)	-3(4)	-3(5)
C39	102(10)	44(7)	49(7)	4(6)	-6(7)	26(7)
C40	147(16)	54(9)	67(10)	13(7)	-3(10)	-51(10)
C41	107(12)	64(9)	55(8)	26(8)	18(8)	-28(9)
C42	64(7)	58(8)	36(6)	-1(5)	7(5)	2(6)
C43	42(5)	48(6)	27(5)	1(4)	5(4)	1(5)
C44	130(11)	30(6)	84(10)	5(6)	21(8)	26(6)
C45	144(17)	20(9)	62(9)	23(8)	16(12)	19(9)
C46	240(20)	90(20)	108(18)	-40(20)	-79(18)	80(20)
C44'	130(11)	30(6)	84(10)	5(6)	21(8)	26(6)
C45'	144(17)	20(9)	62(9)	23(8)	16(12)	19(9)
C46'	240(20)	90(20)	108(18)	-40(20)	-79(18)	80(20)
C47	52(6)	37(6)	47(6)	-12(5)	12(5)	-3(5)
C48	78(9)	53(8)	68(9)	-13(7)	9(7)	14(7)
C49	63(8)	68(9)	90(11)	17(8)	-15(7)	19(7)
C50	39(5)	46(6)	44(6)	3(5)	5(4)	-6(5)
C51	57(6)	39(6)	32(5)	2(4)	6(4)	-21(5)
C52	61(7)	61(8)	37(6)	-2(5)	-4(5)	-15(6)

C53	73(9)	73(9)	58(8)	-2(7)	-11(7)	-25(7)
C54	35(6)	98(10)	47(7)	-8(7)	2(5)	1(6)
C55	56(6)	28(5)	40(6)	4(4)	-4(5)	-6(4)
C56	65(7)	41(6)	57(7)	-18(5)	26(6)	-16(5)
C57	125(13)	65(9)	55(8)	-8(7)	30(8)	-20(9)
C58	89(10)	72(10)	98(11)	-42(8)	57(9)	-36(8)
C59	51(6)	66(9)	63(7)	-21(6)	-5(5)	26(6)
C60	62(8)	150(17)	47(7)	-28(9)	-3(6)	27(9)
C61	96(10)	29(6)	114(12)	-23(7)	45(9)	13(7)
C62	37(6)	59(6)	78(8)	2(5)	12(5)	14(5)
C63	33(5)	64(6)	48(5)	3(5)	0(4)	-6(5)
C64	52(7)	118(13)	61(7)	7(8)	-2(5)	-36(8)
C65	46(7)	188(18)	69(6)	-44(11)	8(6)	-46(9)
C66	22(4)	85(7)	43(6)	-15(6)	-5(4)	11(5)
C67	51(6)	50(6)	64(7)	18(5)	-4(5)	28(5)
C68	89(10)	62(8)	117(11)	27(7)	31(9)	52(8)
C69	74(9)	66(8)	101(10)	-3(7)	18(8)	40(7)
C70	74(6)	29(5)	43(6)	7(4)	3(5)	-12(4)
C71	86(7)	32(5)	48(6)	20(5)	-1(6)	3(5)
C72	190(20)	76(11)	81(9)	31(8)	38(10)	68(12)
C73	144(15)	31(7)	138(12)	-6(6)	39(10)	41(9)
C74	110(8)	46(7)	42(6)	-12(5)	41(6)	-6(6)
C75	95(7)	30(6)	51(6)	5(5)	1(6)	-24(5)
C76	82(9)	95(12)	82(8)	1(8)	1(7)	-53(9)
C77	99(10)	76(10)	64(7)	-1(7)	15(7)	-51(8)

Table 5. Torsion angles [°].

C1-Ir1-N3-C17	46.1(8)	C75-Ir3-N7-N6	56.0(19)
C19-Ir1-N3-C17	133.5(17)	C71-Ir3-N7-N6	-46.8(8)
C20-Ir1-N3-C17	-39.6(13)	C70-Ir3-N7-N6	-10.6(8)
C23-Ir1-N3-C17	-104.4(8)	C4-N1-C1-N2	-176.0(9)
C24-Ir1-N3-C17	-141.3(8)	C2-N1-C1-N2	-2.0(11)
C1-Ir1-N3-N3#1	-149.6(10)	C4-N1-C1-Ir1	6.5(17)
C19-Ir1-N3-N3#1	-62(2)	C2-N1-C1-Ir1	-179.5(9)
C20-Ir1-N3-N3#1	124.7(11)	C3-N2-C1-N1	-0.3(12)
C23-Ir1-N3-N3#1	59.9(10)	C16-N2-C1-N1	-177.9(10)
C24-Ir1-N3-N3#1	23.1(10)	C3-N2-C1-Ir1	177.8(7)
C27-Ir2-N6-N7	157.4(9)	C16-N2-C1-Ir1	0.2(15)
C67-Ir2-N6-N7	-51.9(9)	N3-Ir1-C1-N1	137.4(11)
C62-Ir2-N6-N7	-118.5(12)	C19-Ir1-C1-N1	-29.2(12)
C63-Ir2-N6-N7	53.8(18)	C20-Ir1-C1-N1	-65.9(12)
C66-Ir2-N6-N7	-15.2(9)	C23-Ir1-C1-N1	-146.2(11)
C27-Ir2-N6-C31	-43.8(7)	C24-Ir1-C1-N1	42(4)
C67-Ir2-N6-C31	106.8(8)	N3-Ir1-C1-N2	-39.9(8)
C62-Ir2-N6-C31	40.3(15)	C19-Ir1-C1-N2	153.5(9)
C63-Ir2-N6-C31	-147.4(12)	C20-Ir1-C1-N2	116.8(9)
C66-Ir2-N6-C31	143.5(8)	C23-Ir1-C1-N2	36.5(13)
C31-N6-N7-C33	1.5(11)	C24-Ir1-C1-N2	-135(3)
Ir2-N6-N7-C33	161.8(8)	C1-N1-C2-C3	3.8(13)
C31-N6-N7-Ir3	156.4(6)	C4-N1-C2-C3	178.3(10)
Ir2-N6-N7-Ir3	-43.3(11)	N1-C2-C3-N2	-3.8(13)
C37-Ir3-N7-C33	-47.8(8)	C1-N2-C3-C2	2.6(14)
C74-Ir3-N7-C33	39.1(13)	C16-N2-C3-C2	-180.0(12)
C75-Ir3-N7-C33	-151.2(16)	C1-N1-C4-C5	-80.0(11)
C71-Ir3-N7-C33	106.0(8)	C2-N1-C4-C5	106.7(9)
C70-Ir3-N7-C33	142.2(8)	C1-N1-C4-C9	97.2(11)
C37-Ir3-N7-N6	159.4(7)	C2-N1-C4-C9	-76.1(11)
C74-Ir3-N7-N6	-113.7(10)	N1-C4-C5-C6	177.2(8)

C9-C4-C5-C6	0.0	C23-Ir1-C19-C20	63.6(8)
N1-C4-C5-C10	-6.5(11)	C24-Ir1-C19-C20	97.8(8)
C9-C4-C5-C10	176.4(10)	C1-Ir1-C19-C26	154.0(10)
C4-C5-C6-C7	0.0	N3-Ir1-C19-C26	68(2)
C10-C5-C6-C7	-176.5(10)	C20-Ir1-C19-C26	-116.8(13)
C5-C6-C7-C8	0.0	C23-Ir1-C19-C26	-53.2(11)
C6-C7-C8-C9	0.0	C24-Ir1-C19-C26	-19.0(10)
C7-C8-C9-C4	0.0	C26-C19-C20-C21	-4.6(19)
C7-C8-C9-C13	-179.6(9)	Ir1-C19-C20-C21	-105.8(13)
N1-C4-C9-C8	-177.1(9)	C26-C19-C20-Ir1	101.2(11)
C5-C4-C9-C8	0.0	C1-Ir1-C20-C19	93.3(8)
N1-C4-C9-C13	2.4(10)	N3-Ir1-C20-C19	177.3(8)
C5-C4-C9-C13	179.5(9)	C23-Ir1-C20-C19	-116.9(8)
C4-C5-C10-C12	-78.9(15)	C24-Ir1-C20-C19	-79.7(8)
C6-C5-C10-C12	97.6(14)	C1-Ir1-C20-C21	-144.2(13)
C4-C5-C10-C11	158.9(12)	N3-Ir1-C20-C21	-60.2(16)
C6-C5-C10-C11	-24.7(15)	C19-Ir1-C20-C21	122.5(15)
C8-C9-C13-C14	68(4)	C23-Ir1-C20-C21	5.6(12)
C4-C9-C13-C14	-112(4)	C24-Ir1-C20-C21	42.8(13)
C8-C9-C13-C15	-79(5)	C19-C20-C21-C22	95(2)
C4-C9-C13-C15	102(5)	Ir1-C20-C21-C22	10.9(19)
C3-N2-C16-C17	-123.8(13)	C20-C21-C22-C23	-30(2)
C1-N2-C16-C17	53.4(17)	C21-C22-C23-C24	-47.6(19)
N3#1-N3-C17-C18	1.0(12)	C21-C22-C23-Ir1	35.0(19)
Ir1-N3-C17-C18	168.5(5)	C1-Ir1-C23-C24	-178.3(7)
N3#1-N3-C17-C16	-175.7(11)	N3-Ir1-C23-C24	-102.6(6)
Ir1-N3-C17-C16	-8.2(15)	C19-Ir1-C23-C24	65.9(7)
N2-C16-C17-N3	-49.3(18)	C20-Ir1-C23-C24	98.6(7)
N2-C16-C17-C18	134.5(11)	C1-Ir1-C23-C22	60.7(17)
N3-C17-C18-C17#1	-0.4(5)	N3-Ir1-C23-C22	136.4(13)
C16-C17-C18-C17#1	176.2(15)	C19-Ir1-C23-C22	-55.1(13)
C1-Ir1-C19-C20	-89.2(7)	C20-Ir1-C23-C22	-22.4(13)
N3-Ir1-C19-C20	-175.4(14)	C24-Ir1-C23-C22	-121.0(15)

C22-C23-C24-C25	0.5(18)	C63-Ir2-C27-N4	23.3(11)
Ir1-C23-C24-C25	-103.0(11)	C66-Ir2-C27-N4	-50(3)
C22-C23-C24-Ir1	103.5(11)	C27-N4-C28-C29	0.4(13)
C1-Ir1-C24-C23	173(3)	C38-N4-C28-C29	-176.8(10)
N3-Ir1-C24-C23	78.6(7)	N4-C28-C29-N5	-2.4(13)
C19-Ir1-C24-C23	-114.9(8)	C27-N5-C29-C28	3.7(13)
C20-Ir1-C24-C23	-78.6(8)	C30-N5-C29-C28	175.0(10)
C1-Ir1-C24-C25	-66(3)	C29-N5-C30-C31	132.4(10)
N3-Ir1-C24-C25	-160.7(10)	C27-N5-C30-C31	-57.2(13)
C19-Ir1-C24-C25	5.9(10)	N7-N6-C31-C32	2.3(11)
C20-Ir1-C24-C25	42.2(11)	Ir2-N6-C31-C32	-161.0(7)
C23-Ir1-C24-C25	120.8(13)	N7-N6-C31-C30	174.2(9)
C23-C24-C25-C26	89.7(18)	Ir2-N6-C31-C30	10.9(13)
Ir1-C24-C25-C26	9.7(17)	N5-C30-C31-N6	45.5(13)
C24-C25-C26-C19	-27(2)	N5-C30-C31-C32	-144.2(10)
C20-C19-C26-C25	-48.5(18)	N6-C31-C32-C33	-5.0(11)
Ir1-C19-C26-C25	30.8(16)	C30-C31-C32-C33	-176.4(10)
C29-N5-C27-N4	-3.4(11)	N6-N7-C33-C32	-5.0(12)
C30-N5-C27-N4	-174.7(9)	Ir3-N7-C33-C32	-162.6(7)
C29-N5-C27-Ir2	179.3(7)	N6-N7-C33-C34	174.2(10)
C30-N5-C27-Ir2	7.9(12)	Ir3-N7-C33-C34	16.5(14)
C28-N4-C27-N5	1.8(11)	C31-C32-C33-N7	6.0(12)
C38-N4-C27-N5	178.7(10)	C31-C32-C33-C34	-173.1(11)
C28-N4-C27-Ir2	178.4(8)	N7-C33-C34-N8	39.9(15)
C38-N4-C27-Ir2	-4.7(17)	C32-C33-C34-N8	-141.1(11)
N6-Ir2-C27-N5	35.1(7)	C37-N8-C34-C33	-51.8(13)
C67-Ir2-C27-N5	-46.9(12)	C35-N8-C34-C33	129.3(11)
C62-Ir2-C27-N5	-122.6(8)	C37-N8-C35-C36	0.2(13)
C63-Ir2-C27-N5	-160.5(7)	C34-N8-C35-C36	179.2(9)
C66-Ir2-C27-N5	126(3)	N8-C35-C36-N9	-1.6(12)
N6-Ir2-C27-N4	-141.1(10)	C37-N9-C36-C35	2.4(12)
C67-Ir2-C27-N4	136.9(11)	C50-N9-C36-C35	-174.1(10)
C62-Ir2-C27-N4	61.3(11)	C35-N8-C37-N9	1.2(11)

C34-N8-C37-N9	-177.8(8)	C41-C42-C43-C38	7.3(18)
C35-N8-C37-Ir3	-178.6(7)	C41-C42-C43-C47	178.4(12)
C34-N8-C37-Ir3	2.5(12)	C38-C39-C44-C45	-147.8(15)
C36-N9-C37-N8	-2.1(10)	C40-C39-C44-C45	23(2)
C50-N9-C37-N8	174.0(10)	C38-C39-C44-C46	77(3)
C36-N9-C37-Ir3	177.6(8)	C40-C39-C44-C46	-112(2)
C50-N9-C37-Ir3	-6.3(15)	C38-C43-C47-C49	127.4(12)
C74-Ir3-C37-N8	-117.0(9)	C42-C43-C47-C49	-43.5(15)
N7-Ir3-C37-N8	38.3(7)	C38-C43-C47-C48	-106.9(11)
C75-Ir3-C37-N8	-155.1(8)	C42-C43-C47-C48	82.2(12)
C71-Ir3-C37-N8	-42.7(13)	C37-N9-C50-C55	84.3(13)
C70-Ir3-C37-N8	130(2)	C36-N9-C50-C55	-100.1(12)
C74-Ir3-C37-N9	63.3(10)	C37-N9-C50-C51	-98.0(12)
N7-Ir3-C37-N9	-141.4(9)	C36-N9-C50-C51	77.7(13)
C75-Ir3-C37-N9	25.2(10)	C55-C50-C51-C52	-2.3(16)
C71-Ir3-C37-N9	137.6(11)	N9-C50-C51-C52	-179.9(9)
C70-Ir3-C37-N9	-50(3)	C55-C50-C51-C56	177.2(10)
C27-N4-C38-C43	-102.5(12)	N9-C50-C51-C56	-0.4(15)
C28-N4-C38-C43	74.1(14)	C50-C51-C52-C53	0.4(16)
C27-N4-C38-C39	86.7(14)	C56-C51-C52-C53	-179.1(11)
C28-N4-C38-C39	-96.7(13)	C51-C52-C53-C54	1(2)
C43-C38-C39-C44	-177.5(12)	C52-C53-C54-C55	-1(2)
N4-C38-C39-C44	-7.0(18)	C51-C50-C55-C54	2.6(16)
C43-C38-C39-C40	11.0(16)	N9-C50-C55-C54	-179.8(10)
N4-C38-C39-C40	-178.5(10)	C51-C50-C55-C59	-176.7(11)
C44-C39-C40-C41	-178.5(16)	N9-C50-C55-C59	0.9(16)
C38-C39-C40-C41	-7(2)	C53-C54-C55-C50	-1.0(17)
C39-C40-C41-C42	3(3)	C53-C54-C55-C59	178.3(12)
C40-C41-C42-C43	-3(2)	C52-C51-C56-C57	73.4(14)
C39-C38-C43-C42	-12.0(17)	C50-C51-C56-C57	-106.1(13)
N4-C38-C43-C42	177.6(10)	C52-C51-C56-C58	-48.3(16)
C39-C38-C43-C47	177.7(11)	C50-C51-C56-C58	132.2(12)
N4-C38-C43-C47	7.3(15)	C50-C55-C59-C60	-148.2(12)

C54-C55-C59-C60	32.6(16)	C27-Ir2-C66-C65	62(3)
C50-C55-C59-C61	84.9(13)	N6-Ir2-C66-C65	152.8(11)
C54-C55-C59-C61	-94.3(13)	C67-Ir2-C66-C65	-123.6(14)
C27-Ir2-C62-C63	-100.1(7)	C62-Ir2-C66-C65	-49.1(12)
N6-Ir2-C62-C63	176.6(8)	C63-Ir2-C66-C65	-12.2(11)
C67-Ir2-C62-C63	108.8(8)	C65-C66-C67-C68	-3(2)
C66-Ir2-C62-C63	73.1(8)	Ir2-C66-C67-C68	-101.1(12)
C27-Ir2-C62-C69	137.2(11)	C65-C66-C67-Ir2	98.1(13)
N6-Ir2-C62-C69	53.9(17)	C27-Ir2-C67-C66	178.6(7)
C67-Ir2-C62-C69	-13.8(11)	N6-Ir2-C67-C66	97.1(7)
C63-Ir2-C62-C69	-122.7(14)	C62-Ir2-C67-C66	-103.6(8)
C66-Ir2-C62-C69	-49.6(11)	C63-Ir2-C67-C66	-67.5(7)
C69-C62-C63-C64	0(2)	C27-Ir2-C67-C68	-60.7(14)
Ir2-C62-C63-C64	-103.6(11)	N6-Ir2-C67-C68	-142.2(10)
C69-C62-C63-Ir2	103.4(13)	C62-Ir2-C67-C68	17.2(10)
C27-Ir2-C63-C62	82.5(8)	C63-Ir2-C67-C68	53.2(11)
N6-Ir2-C63-C62	-175.2(12)	C66-Ir2-C67-C68	120.7(13)
C67-Ir2-C63-C62	-70.4(8)	C66-C67-C68-C69	63(2)
C66-Ir2-C63-C62	-104.6(8)	Ir2-C67-C68-C69	-20(2)
C27-Ir2-C63-C64	-159.8(10)	C67-C68-C69-C62	9(3)
N6-Ir2-C63-C64	-57.5(19)	C63-C62-C69-C68	-76(2)
C67-Ir2-C63-C64	47.3(11)	Ir2-C62-C69-C68	6(2)
C62-Ir2-C63-C64	117.7(14)	C37-Ir3-C70-C71	-174(2)
C66-Ir2-C63-C64	13.1(10)	C74-Ir3-C70-C71	72.1(8)
C62-C63-C64-C65	68(2)	N7-Ir3-C70-C71	-83.9(7)
Ir2-C63-C64-C65	-12(2)	C75-Ir3-C70-C71	108.8(8)
C63-C64-C65-C66	1(3)	C37-Ir3-C70-C77	72(3)
C64-C65-C66-C67	-67(2)	C74-Ir3-C70-C77	-41.2(11)
C64-C65-C66-Ir2	10(2)	N7-Ir3-C70-C77	162.7(11)
C27-Ir2-C66-C67	-175(3)	C75-Ir3-C70-C77	-4.5(11)
N6-Ir2-C66-C67	-83.6(7)	C71-Ir3-C70-C77	-113.3(14)
C62-Ir2-C66-C67	74.6(8)	C77-C70-C71-C72	1(2)
C63-Ir2-C66-C67	111.4(8)	Ir3-C70-C71-C72	-104.2(14)

C77-C70-C71-Ir3	105.2(10)	C75-Ir3-C74-C73	-114.6(13)
C37-Ir3-C71-C70	177.8(8)	C71-Ir3-C74-C73	-7.7(12)
C74-Ir3-C71-C70	-104.7(8)	C70-Ir3-C74-C73	-42.7(12)
N7-Ir3-C71-C70	98.2(7)	C73-C74-C75-C76	-1(2)
C75-Ir3-C71-C70	-68.5(8)	Ir3-C74-C75-C76	-103.7(13)
C37-Ir3-C71-C72	-55.1(18)	C73-C74-C75-Ir3	102.4(12)
C74-Ir3-C71-C72	22.4(14)	C37-Ir3-C75-C74	85.1(7)
N7-Ir3-C71-C72	-134.7(14)	N7-Ir3-C75-C74	-173.0(14)
C75-Ir3-C71-C72	58.6(15)	C71-Ir3-C75-C74	-70.4(8)
C70-Ir3-C71-C72	127.1(17)	C70-Ir3-C75-C74	-104.7(8)
C70-C71-C72-C73	51(3)	C37-Ir3-C75-C76	-150.6(11)
Ir3-C71-C72-C73	-35(2)	C74-Ir3-C75-C76	124.3(14)
C71-C72-C73-C74	29(3)	N7-Ir3-C75-C76	-49(2)
C72-C73-C74-C75	-88(2)	C71-Ir3-C75-C76	53.9(12)
C72-C73-C74-Ir3	-10(2)	C70-Ir3-C75-C76	19.6(11)
C37-Ir3-C74-C75	-99.0(7)	C74-C75-C76-C77	50(2)
N7-Ir3-C74-C75	176.1(8)	Ir3-C75-C76-C77	-32.3(18)
C71-Ir3-C74-C75	106.9(8)	C75-C76-C77-C70	29(2)
C70-Ir3-C74-C75	71.9(8)	C71-C70-C77-C76	-91.6(18)
C37-Ir3-C74-C73	146.4(11)	Ir3-C70-C77-C76	-13(2)
N7-Ir3-C74-C73	61.4(15)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1