

# Synthesis of an Azaphosphatriptycene and Its Rhodium Carbonyl Complex

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*Supporting Information*

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## 1. General Considerations

Unless otherwise indicated, manipulations were carried out in resealable glassware on a Schlenk line under an atmosphere of argon, or in an MBraun glovebox under an atmosphere of nitrogen. Glassware and magnetic stir bars were dried in a ventilated oven at 160 °C and allowed to cool under vacuum. Tetrahydrofuran (THF, EMD Millipore OmniSolv), toluene (EMD Millipore OmniSolv) and dichloromethane (EMD Millipore OmniSolv) were sparged with ultra-high purity argon (NexAir) for 45 minutes prior to first use and dried using an MBraun solvent purification system. THF and toluene were further dried over sodium benzophenone ketyl, degassed by successive freeze-pump-thaw cycles and transferred under vacuum to an oven-dried resealable flasks. Dichloromethane was further dried by stirring overnight with calcium hydride (Alfa Aesar, coarse powder), degassed by successive freeze-pump-thaw cycles, and transferred under vacuum into oven-dried resealable Schlenk flask. Benzene-*d*<sub>6</sub> (Cambridge Isotope Laboratories) was dried over sodium benzophenone ketyl, degassed by successive freeze-pump-thaw cycles and transferred under vacuum into oven-dried resealable flask. 1-Octene (Acros Organics), cyclohexene (Alfa Aesar), 2,3-dihydrofuran (Alfa Aesar) and 3,4-dihydro-2*H*-pyran (Aldrich) were dried by stirring overnight with calcium hydride (Alfa Aesar, coarse powder), degassed by successive freeze-pump-thaw cycles, and transferred under vacuum into oven-dried resealable Schlenk flasks. Anhydrous pentane (EMD Millipore Drisolv, sealed under a nitrogen atmosphere) and (acetylacetonato)dicarbonyl-rhodium(I) (Aldrich) were used as received and stored in a glovebox. Ethyl Acetate (BDH), methanol (BDH), ethanol (200 proof, Koptec), chloroform-*d* (Cambridge Isotope Laboratories), *tert*-butyllithium (1.7 M in pentane, Aldrich), nitric acid (BDH, 68-70%, ACS grade), tris(2,4-di-*tert*-butylphenyl)phosphite (Strem Chemicals), anhydrous magnesium sulfate (Alfa Aesar, powder), sodium hydroxide (EMD Millipore), nonanal (Aldrich, analytical standard), cyclohexanecarboxaldehyde (Aldrich), di-(4-*tert*-butylphenyl)amine (TCI America), tris(dibenzylideneacetone)dipalladium(0) (Aldrich), tris(*o*-tolyl)phosphine (Aldrich), sodium *tert*-butoxide (TCI America), 1-bromo-4-*tert*-butylbenzene (Oakwood Chemicals), *N*-bromosuccinimide (Aldrich), sodium metal (Alfa Aesar), benzophenone (Alfa Aesar), calcium hydride (Alfa Aesar), nitrogen (NexAir), syngas (CO/H<sub>2</sub>=1:1, NexAir) and argon (both industrial and ultra-high purity grades, NexAir) were used as received. Tap water was purified in a Barnstead International automated still prior to use.

Tris(4-*tert*-butylphenyl)amine<sup>1</sup> and tris(2-bromo-4-*tert*-butylphenyl)amine<sup>2</sup> were prepared according to reported procedures, and characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy.

<sup>1</sup>H, <sup>13</sup>C and <sup>31</sup>P NMR spectra were obtained using a Varian Mercury Vx 400 spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts were referenced with respect to solvent signals and reported relative to tetramethylsilane. <sup>31</sup>P NMR chemical shifts were reported with 85% aqueous H<sub>3</sub>PO<sub>4</sub> as external standard. Chemical shifts  $\delta$  are reported in ppm. Fourier Transform Infrared spectra were collected using microcrystalline samples on a Shimadzu IRAffinity-1S Fourier transform infrared spectrophotometer. Samples were exposed to air as briefly as possible prior to data collection. Only characteristic peaks were reported in cm<sup>-1</sup>. EI-MS and GC-MS were recorded on MicroMass AutoSpec M by Georgia Institute of Technology mass spec facility. Elemental analyses were performed by Atlantic Microlab, Inc. in Norcross, GA.

Hydroformylation reactions were performed in a 300-mL stainless steel stirred Parr reactor (Parr Instrument Company, model 452HC, maximum allowable working pressure: 2000 psi (137.9 bar) at 350° C). The 300-mL stainless steel Parr reactor was thoroughly washed with nitric acid (1 M, 3 x 300 mL), neutralized with 1 M NaOH solution, followed by several rinse cycles with distilled water and acetone. The reactor was dried in pieces in a ventilated oven at 160 °C for 12 h, then reconstructed and purged with 7 bar nitrogen 10 times with heating to 140 °C. The reactor was cooled to room temperature under N<sub>2</sub> (7 bar) before each experiment. Syngas was added to the vessel via high pressure stainless steel tubing, using a Matheson Tri-Gas control system (PAN-5500 Series Analytical Grade Panel) and 500D Syringe Pump (Teledyne Isco). Gas chromatography-flame ionization detection (GC-FID) analyses were performed using a Shimadzu GC-2010 Plus gas chromatograph fitted with a Supelco PTA-5 (30 m x 0.25 mm x 0.50  $\mu$ m, length x inside diameter x film thickness) capillary GC column. Calibration curves for 1-octene, nonanal, cyclohexene, cyclohexanecarboxaldehyde, 2,3-dihydrofuran and 3,4-dihydro-2*H*-pyran were recorded on the GC-FID.

## 2. Synthesis of Azaphosphatriptycene 1

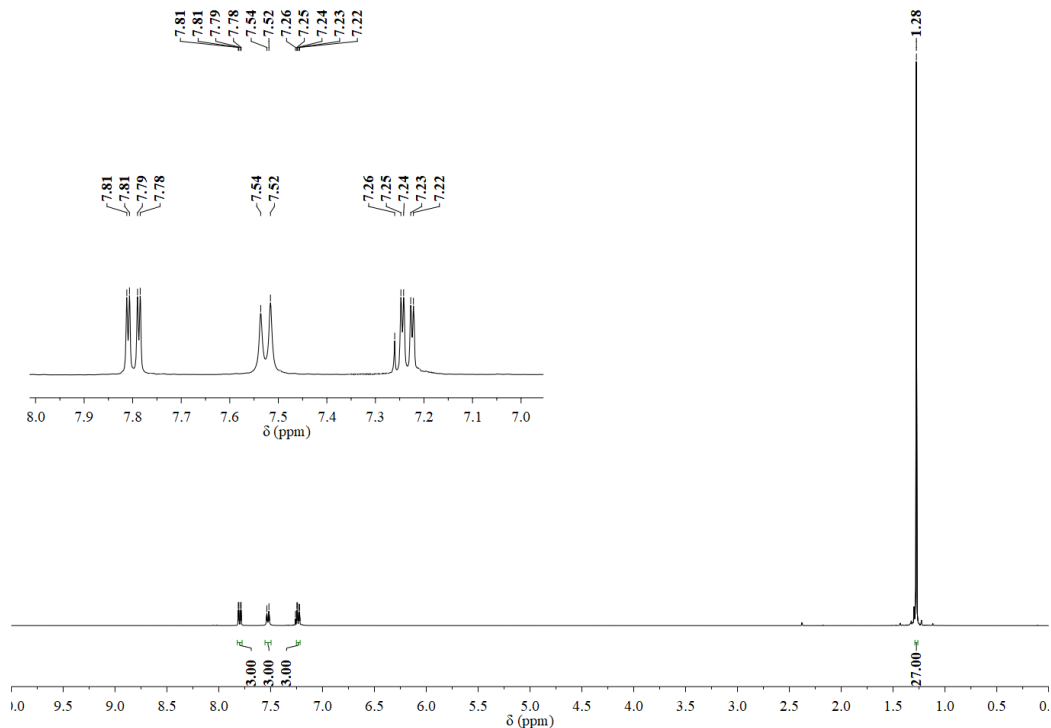
Tris(2-bromo-4-*tert*-butylphenyl)amine<sup>2</sup> (4.056 g, 6.237 mmol) was dissolved in THF (150 mL) in a 500-mL Schlenk flask and the solution was cooled to -78 °C. *tert*-Butyllithium (1.7 M in pentane, 22.0 mL, 37.4 mmol) was added dropwise at -78 °C and the resulting solution was stirred at -78 °C for 2 h. A solution of tris(2,4-di-*tert*-butylphenyl) phosphite (4.035 g, 6.237 mmol) in THF (20 mL) was added dropwise to the reaction mixture, which was allowed to warm slowly to ambient temperature, then refluxed for 96 h. The solution was cooled to room temperature, quenched with distilled water, then extracted with ethyl acetate (3 portions, 50 mL each). The combined organic portions were washed with distilled water and dried over MgSO<sub>4</sub>. The resulting mixture was filtered, then

concentrated in vacuo. Addition of methanol (50 mL) to the residue caused a microcrystalline solid to form. The off-white precipitate was collected by filtration and recrystallized from CH<sub>2</sub>Cl<sub>2</sub>/EtOH to afford **1** as a white solid, 1.945 g (71%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (ppm) 7.80 (dd, <sup>3</sup>J<sub>H-P</sub> = 8.8 Hz, *J*<sub>H-H, meta</sub> = 2.2 Hz, 3H), 7.53 (d, *J*<sub>H-H, ortho</sub> = 8.0 Hz, 3H), 7.23 (dd, *J*<sub>H-H, ortho</sub> = 8.0 Hz, *J*<sub>H-H, meta</sub> = 2.0 Hz, 3H), 1.28 (s, 27H, C(CH<sub>3</sub>)<sub>3</sub>); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ (ppm) 155.47 (d, <sup>2</sup>J<sub>C-P</sub> = 1.2 Hz), 148.01 (d, <sup>1</sup>J<sub>C-P</sub> = 10.8 Hz), 144.69 (d, <sup>3</sup>J<sub>C-P</sub> = 7.5 Hz), 129.88 (d, <sup>2</sup>J<sub>C-P</sub> = 34.4 Hz), 126.30 (d, <sup>3</sup>J<sub>C-P</sub> = 1.4 Hz), 125.62 (s), 34.73 (s, C(CH<sub>3</sub>)<sub>3</sub>), 31.57 (s, CH<sub>3</sub>); <sup>31</sup>P(<sup>1</sup>H) NMR (162 MHz, CDCl<sub>3</sub>): δ (ppm) -77.0 (s). MS(EI): calcd m/z 441.26, found m/z 441.3 (M<sup>+</sup>). Anal. calcd (%) for C<sub>30</sub>H<sub>36</sub>NP (441.60): C 81.60, H 8.22, N 3.17; found C 81.72, H 8.27, N 3.19.

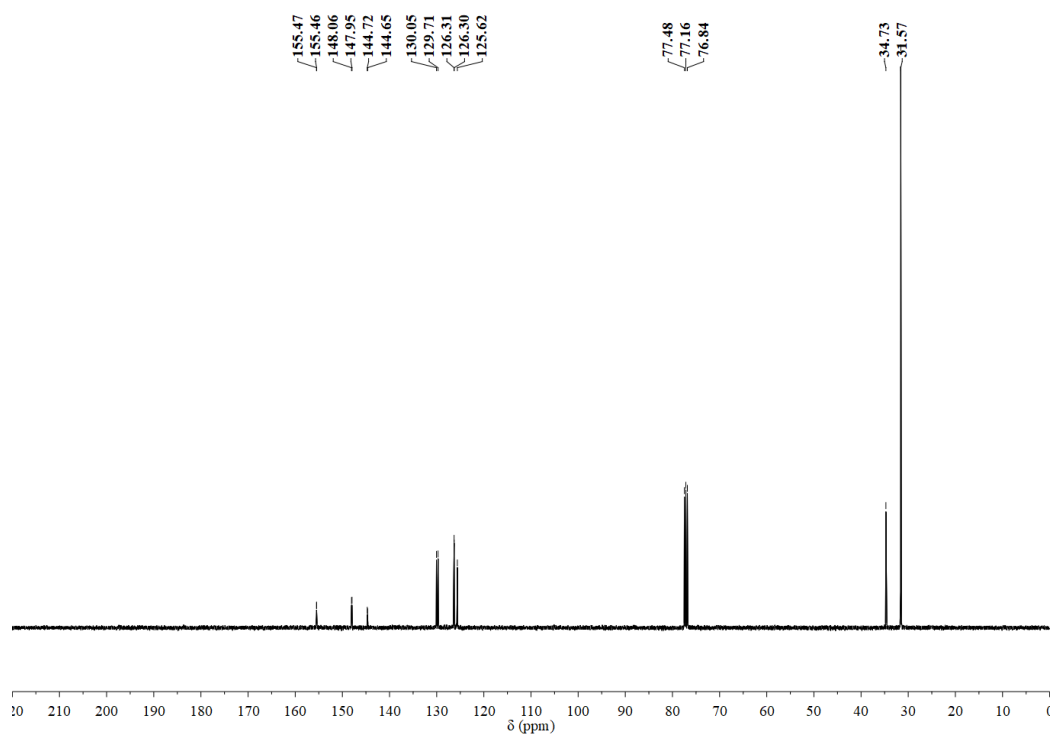
### 3. Preparation of Complex 2, [(1)Rh(acac)(CO)]

In a glovebox under nitrogen atmosphere, ligand **1** (0.017 g, 0.039 mmol) and (acetylacetonato)dicarbonyl-rhodium(I) [Rh(acac)(CO)<sub>2</sub>] (0.010 g, 0.039 mmol) were dissolved in toluene (0.5 mL); bubbles were observed immediately. The solution was layered with pentane (5 mL) and stored at -35 °C for 3 days. The solution was filtered and a yellow crystalline solid was collected. The solid was dried under vacuum for 12 h to afford the title complex (0.015 g, 58%). <sup>1</sup>H NMR (400 MHz, C<sub>6</sub>D<sub>6</sub>): δ (ppm) 8.70 (dd, <sup>3</sup>J<sub>H-P</sub> = 12.4 Hz, *J*<sub>H-H, meta</sub> = 2.0 Hz, 3H), 7.69 (dd, *J*<sub>H-H, ortho</sub> = 8.0 Hz, <sup>4</sup>J<sub>H-P</sub> = 2.4 Hz, 3H), 7.03 (dd, *J*<sub>H-H, ortho</sub> = 8.0 Hz, *J*<sub>H-H, meta</sub> = 2.4 Hz, 3H), 5.43 (s, 1H, CH, acac), 2.00 (s, 3H, CH<sub>3</sub>, acac), 1.86 (s, 3H, CH<sub>3</sub>, acac), 1.08 (s, 27H, C(CH<sub>3</sub>)<sub>3</sub>); <sup>13</sup>C NMR (101 MHz, C<sub>6</sub>D<sub>6</sub>): δ (ppm) 189.17 (dd, <sup>1</sup>J<sub>C-Rh</sub> = 72.3 Hz, <sup>2</sup>J<sub>C-P</sub> = 29.5 Hz, CO), 188.34 (s, CO, acac), 185.00 (s, CO, acac), 154.78 (d, <sup>2</sup>J<sub>C-P</sub> = 1.9 Hz), 148.91 (d, <sup>1</sup>J<sub>C-P</sub> = 11.2 Hz), 141.49 (d, <sup>2</sup>J<sub>C-P</sub> = 45.3 Hz), 131.37 (d, <sup>3</sup>J<sub>C-P</sub> = 16.6 Hz), 127.44 (d, <sup>4</sup>J<sub>C-P</sub> = 1.4 Hz), 125.90 (d, <sup>3</sup>J<sub>C-P</sub> = 3.7 Hz), 101.53 (d, <sup>3</sup>J<sub>C-Rh</sub> = 1.7 Hz, CH, acac), 34.78 (s, C(CH<sub>3</sub>)<sub>3</sub>), 31.40 (s, CH<sub>3</sub>), 27.57 (s, CH<sub>3</sub>, acac). <sup>31</sup>P(<sup>1</sup>H) NMR (162 MHz, C<sub>6</sub>D<sub>6</sub>): δ (ppm) -1.28 (d, <sup>1</sup>J<sub>P-Rh</sub> = 189.0 Hz). IR: ν (cm<sup>-1</sup>): 2961 (w), 1985 (s, CO), 1584(w), 1524(w), 1474(s), 1391(w), 1051 (s), 729(s). Anal. calcd (%) for C<sub>36</sub>H<sub>43</sub>NO<sub>3</sub>PRh (671.62): C 64.38, H 6.45, N 2.09; found C 64.37, H 7.04, N 1.90.

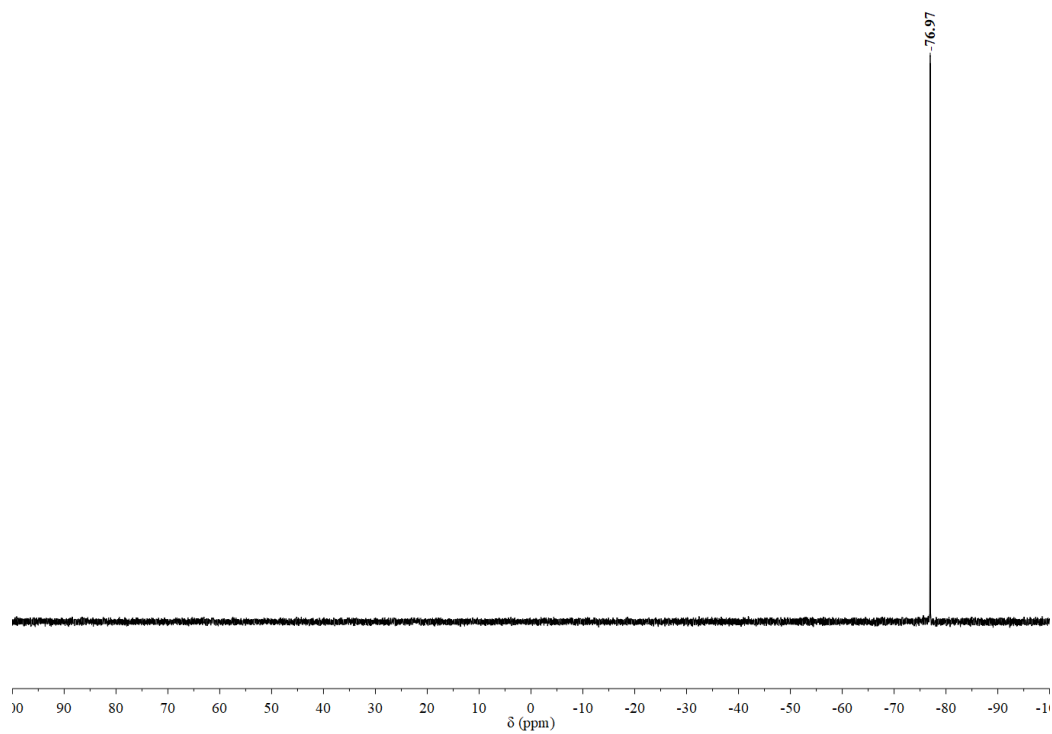
### 4. Selected Spectra



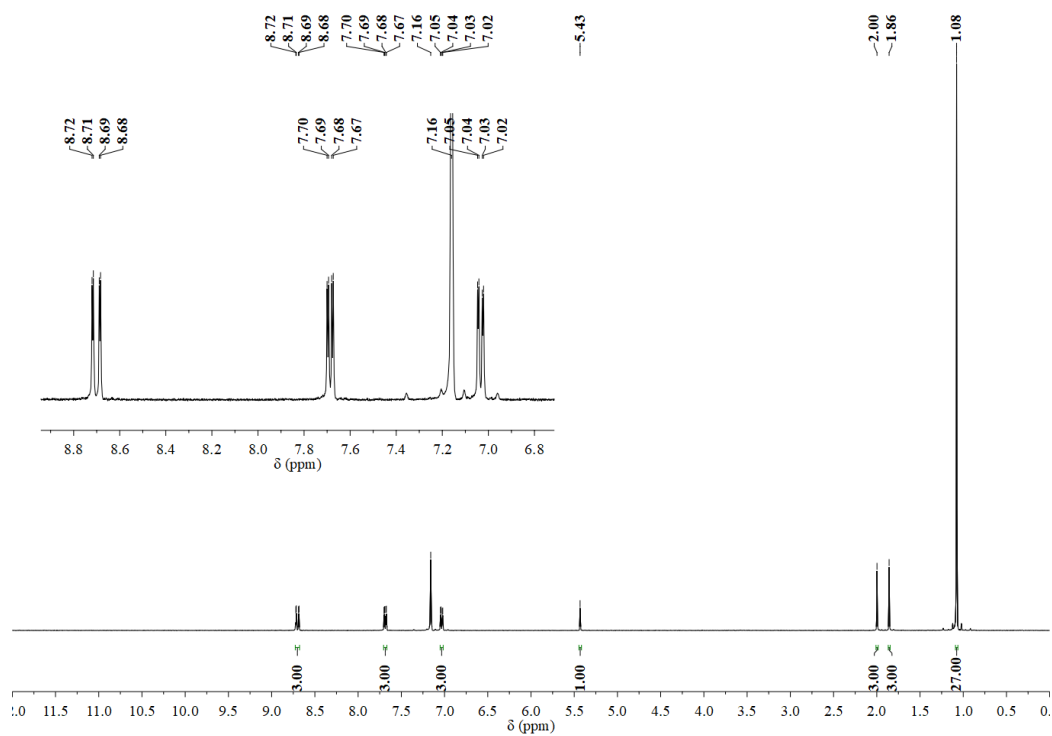
**Figure S1.** <sup>1</sup>H NMR spectrum of **1** in CDCl<sub>3</sub> solution.



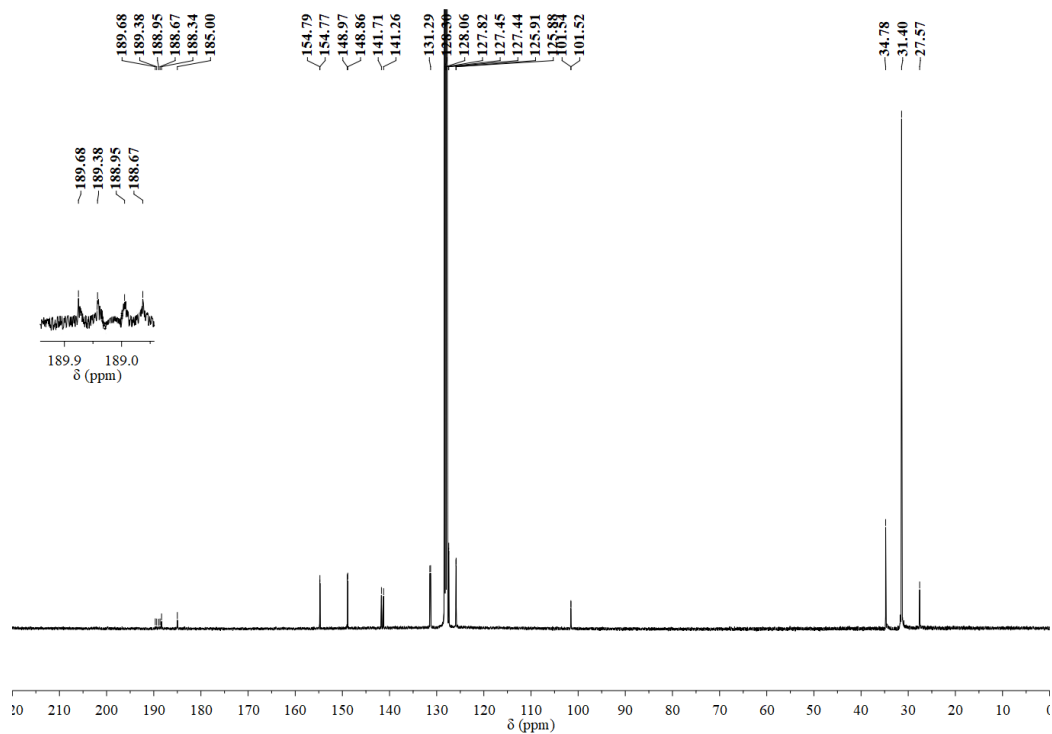
**Figure S2.**  $^{13}\text{C}$  NMR spectrum of **1** in  $\text{CDCl}_3$  solution.



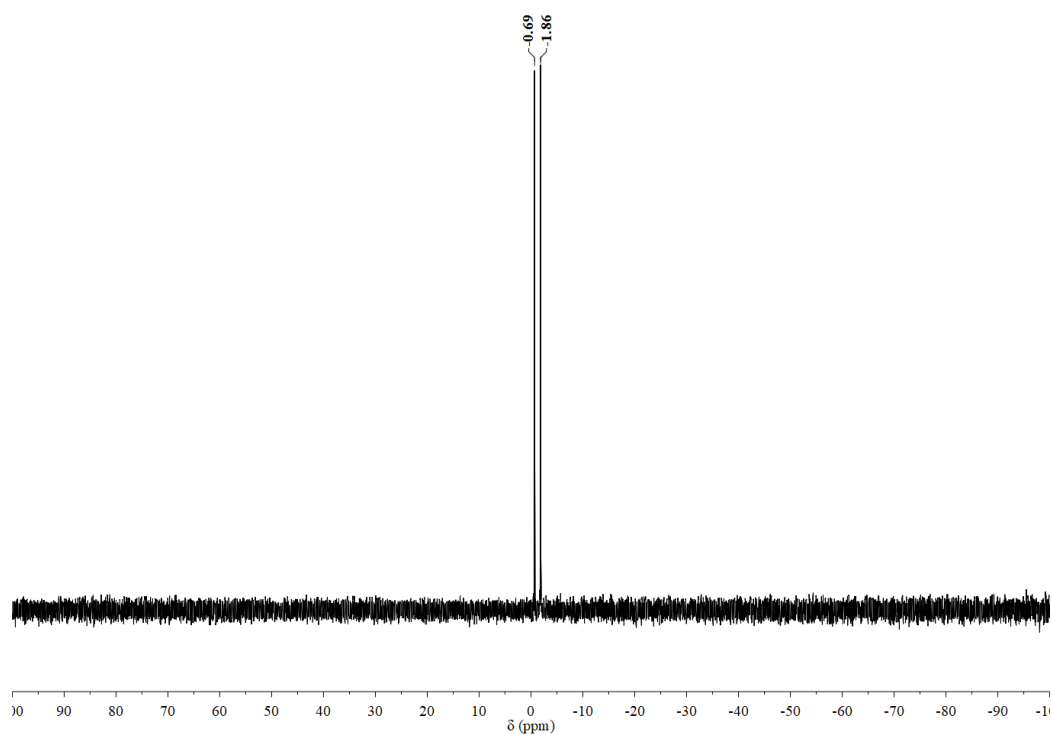
**Figure S3.**  $^{31}\text{P}$  NMR spectrum of **1** in  $\text{CDCl}_3$  solution.



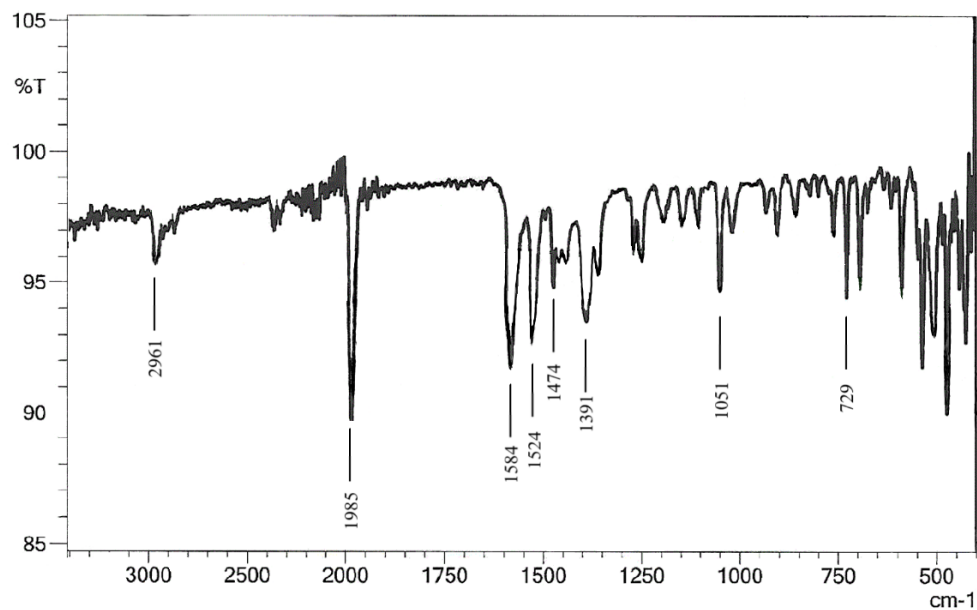
**Figure S4.** <sup>1</sup>H NMR spectrum of **2** in C<sub>6</sub>D<sub>6</sub> solution.



**Figure S5.** <sup>13</sup>C NMR spectrum of **2** in C<sub>6</sub>D<sub>6</sub> solution.



**Figure S6.**  $^{31}\text{P}$  NMR spectrum of **2** in  $\text{C}_6\text{D}_6$  solution.



**Figure S7.** IR spectrum of **2**.

## 5. General Procedure for Hydroformylation

Ligand **1**,  $[\text{Rh}(\text{acac})(\text{CO})_2]$  and toluene were mixed in a glovebox and transferred via gastight syringe into a previously dried 300-mL stainless steel Parr reactor. The reactor was purged twice with  $\text{N}_2$  (7 bar), then three times with syngas (7 bar). Substrate was added into the Parr reactor via another gastight syringe, then the reactor was purged twice more with syngas (7 bar), and released to 1 bar. The reactor was then heated to the desired working

temperature, a continuous working pressure of syngas was established, and stirring was initiated. The reaction mixture was stirred for the desired reaction time, then agitation was stopped and the reactor was cooled to room temperature in an ice bath. The gas inside the Parr reactor was slowly vented in the hood to a 4.0 mL cold bath of toluene (0 °C). The reactor was opened and the organic phase was diluted to a defined volume. Aliquots taken from the organic phase and from cold toluene were analyzed by GC-FID. Amounts of starting substrates and products were determined by analysis of both the organic phase and the toluene solution for gas venting, based on predetermined calibration curves. Aldehyde TOF (turnover frequency) was calculated from the molar quantity of aldehyde formed per mole of Rh(acac)(CO)<sub>2</sub> precatalyst per hour of reaction time.

Hydroformylation experiments of 2,3-dihydrofuran and 3,4-dihydro-2*H*-pyran were reproduced from literature reports under their reported conditions.<sup>3-4</sup> The hydroformylation products were determined by GC-MS and GC-FID and isomer aldehydes were identified by different retention times on GC based on boiling points. None of the experiments produced hydrogenation products.

## 6. Hydroformylation of 1-Octene

Hydroformylation experiments of 1-octene<sup>5</sup> were conducted by dissolving catalyst precursors in toluene in the autoclave, then pressurizing with syngas and heating up to the desired pressure and temperature over 40 min. 1-Octene was added under pressure, and the reactions run for 1 h. To compare results in our laboratory to those of experiments reported using the same reaction conditions, catalyst precursor and 1-octene were dissolved in toluene in the autoclave, then the desired pressure of syngas was added and the mixture was heated over 20 min to the desired temperature, followed by a reaction time of 1 h. The results are summarized in Table S1.

**Table S1. Hydroformylation of 1-octene.<sup>a</sup>**

$n\text{-Hex} \xrightarrow[\text{H}_2/\text{CO}]{[\text{Rh}] / \text{L cat.}} n\text{-Hex} \text{---} \text{CH}_2 \text{---} \text{CH}_2 \text{---} \text{CHO} \text{ (I)} + n\text{-Hex} \text{---} \text{CH} \text{---} \text{CH}_2 \text{---} \text{CHO} \text{ (b)}$						
Entry	L	L/M ratio	Alkene conv (%)	Aldehyde (%)	I : b ratio	Aldehyde TOF (h <sup>-1</sup> )
1	PPh <sub>3</sub>	20	42	42	2.8	2100 <sup>5</sup>
2	PPh <sub>3</sub>	20	76	28	2.8	1390
3	<b>1</b>	20	1	0.3	- <sup>b</sup>	15
4	<b>1</b>	4	80	56	3.1	2780
5 <sup>c</sup>	<b>1</b>	2	99	53	1.5	5300
6 <sup>c,d</sup>	<b>1</b>	2	20	13	3.2	1270

<sup>a</sup> [Rh(acac)(CO)<sub>2</sub>], 0.020 mol%; [1-octene]<sub>initial</sub> = 0.84 M in toluene; H<sub>2</sub>/CO (1:1) 20 bar. Unless noted otherwise, 80 °C, 1 h. <sup>b</sup> Only *n*-aldehyde detected. <sup>c</sup> Reaction time 0.5 h. <sup>d</sup> Reaction temp 60 °C.

## 7. X-ray Diffraction Studies for Complex 2, [(1)Rh(acac)(CO)]

### Experimental.

Single crystals of **2** were recrystallized from a mixture of THF and pentane by vapor diffusion. A suitable crystal was selected and mounted on a loop with Paratone oil a Bruker D8 Venture diffractometer. The crystal was kept at 100(2) K during data collection. Using Olex2,<sup>6</sup> the structure was solved with the SHELXT<sup>7</sup> structure solution program using Intrinsic Phasing, and refined with the SHELXL<sup>8</sup> refinement package using Least Squares minimization.

### Crystal Data.

C<sub>72</sub>H<sub>86</sub>N<sub>2</sub>O<sub>6</sub>P<sub>2</sub>Rh<sub>2</sub> (*M* = 1343.19 g/mol): triclinic, space group P-1 (no. 2), *a* = 10.6289(5) Å, *b* = 10.8230(5) Å, *c* = 17.8290(8) Å, α = 84.254(2)°, β = 78.440(2)°, γ = 63.6990(10)°, *V* = 1801.22(15) Å<sup>3</sup>, *Z* = 1, *T* = 100(2) K, μ(MoKα) = 0.551 mm<sup>-1</sup>, *D*<sub>calc</sub> = 1.238 g/cm<sup>3</sup>, 16475 reflections measured (4.342° ≤ 2θ ≤ 56.564°), 8804 unique (*R*<sub>int</sub> = 0.0260, *R*<sub>sigma</sub> = 0.0454) which were used in all calculations. The final *R*<sub>1</sub> was 0.0461 (*I* > 2σ(*I*)) and *wR*<sub>2</sub> was 0.1130 (all data).

**Table S2.** Crystal data and structure refinement for **2**.

<b>Empirical formula</b>	C <sub>72</sub> H <sub>86</sub> N <sub>2</sub> O <sub>6</sub> P <sub>2</sub> Rh <sub>2</sub>
<b>Formula weight</b>	1343.19
<b>Temperature/K</b>	100(2)
<b>Crystal system</b>	triclinic
<b>Space group</b>	P-1
<b>a/Å</b>	10.6289(5)
<b>b/Å</b>	10.8230(5)
<b>c/Å</b>	17.8290(8)
<b>α/°</b>	84.254(2)
<b>β/°</b>	78.440(2)
<b>γ/°</b>	63.6990(10)
<b>Volume/Å<sup>3</sup></b>	1801.22(15)
<b>Z</b>	1
<b>ρ<sub>calc</sub>/g/cm<sup>3</sup></b>	1.238
<b>μ/mm<sup>-1</sup></b>	0.551
<b>F(000)</b>	700.0
<b>Crystal size/mm<sup>3</sup></b>	0.254 × 0.119 × 0.113
<b>Radiation</b>	MoKα (λ = 0.71073)
<b>2θ range for data collection/°</b>	4.342 to 56.564
<b>Index ranges</b>	-14 ≤ h ≤ 10, -14 ≤ k ≤ 13, -23 ≤ l ≤ 23
<b>Reflections collected</b>	16475
<b>Independent reflections</b>	8804 [R <sub>int</sub> = 0.0260, R <sub>sigma</sub> = 0.0454]
<b>Data/restraints/parameters</b>	8804/378/390
<b>Goodness-of-fit on F<sup>2</sup></b>	1.206
<b>Final R indexes [I ≥ 2σ (I)]</b>	R <sub>1</sub> = 0.0461, wR <sub>2</sub> = 0.0909
<b>Final R indexes [all data]</b>	R <sub>1</sub> = 0.0618, wR <sub>2</sub> = 0.1130
<b>Largest diff. peak/hole / e Å<sup>-3</sup></b>	1.01/-0.76



**Table S3.** Bond lengths in Å for **2**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Rh1	P1	2.2080(8)	C24	C23	1.375(4)
Rh1	O3	2.044(2)	C12	C17	1.374(4)
Rh1	O2	2.067(2)	C23	C22	1.405(4)
Rh1	C1	1.821(3)	C14	C15	1.403(4)
P1	C2	1.832(3)	C8	C6	1.530(4)
P1	C13	1.834(3)	C8	C11	1.548(5)
P1	C22	1.823(3)	C8	C9	1.523(5)
O3	C32	1.288(4)	C8	C10	1.529(5)
O2	C34	1.249(4)	C5	C6	1.390(4)
O1	C1	1.142(4)	C33	C32	1.396(4)
N1	C12	1.466(4)	C33	C34	1.399(5)
N1	C3	1.460(4)	C26	C28	1.527(4)
N1	C23	1.467(3)	C32	C35	1.512(5)
C2	C7	1.398(4)	C15	C16	1.399(4)
C2	C3	1.398(4)	C15	C18	1.531(5)
C7	C6	1.412(4)	C31	C28	1.536(5)
C13	C12	1.393(4)	C28	C29	1.529(4)
C13	C14	1.386(4)	C28	C30	1.532(5)
C27	C22	1.397(4)	C34	C36	1.503(4)
C27	C26	1.401(4)	C16	C17	1.390(4)
C25	C24	1.399(4)	C18	C19	1.535(5)
C25	C26	1.396(4)	C18	C20	1.529(6)
C4	C3	1.382(4)	C18	C21	1.531(5)
C4	C5	1.387(4)			

**Table S4.** Bond angles for **2**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O3	Rh1	P1	94.54(6)	C9	C8	C11	107.7(3)
O3	Rh1	O2	89.26(9)	C9	C8	C10	109.9(3)
O2	Rh1	P1	175.79(7)	C10	C8	C6	108.5(3)

C1	Rh1	P1	86.43(10)	C10	C8	C11	108.7(3)
C1	Rh1	O3	178.88(12)	C4	C5	C6	121.9(3)
C1	Rh1	O2	89.78(11)	C7	C6	C8	119.4(3)
C2	P1	Rh1	126.62(10)	C5	C6	C7	118.3(3)
C2	P1	C13	95.00(13)	C5	C6	C8	122.3(3)
C13	P1	Rh1	119.44(10)	C32	C33	C34	126.3(3)
C22	P1	Rh1	118.48(10)	C27	C22	P1	126.3(2)
C22	P1	C2	95.14(13)	C27	C22	C23	119.5(3)
C22	P1	C13	95.13(13)	C23	C22	P1	114.2(2)
C32	O3	Rh1	126.2(2)	C27	C26	C28	119.6(3)
C34	O2	Rh1	126.6(2)	C25	C26	C27	117.5(3)
C12	N1	C23	108.9(2)	C25	C26	C28	122.9(3)
C3	N1	C12	107.5(2)	O3	C32	C33	125.6(3)
C3	N1	C23	108.0(2)	O3	C32	C35	115.4(3)
C7	C2	P1	126.6(2)	C33	C32	C35	119.0(3)
C7	C2	C3	120.0(3)	C14	C15	C18	120.1(3)
C3	C2	P1	113.3(2)	C16	C15	C14	116.9(3)
C2	C7	C6	120.0(3)	C16	C15	C18	123.0(3)
C12	C13	P1	113.9(2)	O1	C1	Rh1	178.5(3)
C14	C13	P1	126.1(2)	C26	C28	C31	109.1(3)
C14	C13	C12	120.0(3)	C26	C28	C29	112.0(3)
C22	C27	C26	121.2(3)	C26	C28	C30	109.7(3)
C26	C25	C24	122.2(3)	C29	C28	C31	108.9(3)
C3	C4	C5	119.6(3)	C29	C28	C30	108.0(3)
C23	C24	C25	119.3(3)	C30	C28	C31	109.2(3)
C13	C12	N1	118.6(3)	O2	C34	C33	126.0(3)
C17	C12	N1	121.2(3)	O2	C34	C36	116.1(3)
C17	C12	C13	120.2(3)	C33	C34	C36	117.9(3)
C2	C3	N1	119.2(2)	C17	C16	C15	122.4(3)
C4	C3	N1	120.6(3)	C12	C17	C16	119.2(3)
C4	C3	C2	120.2(3)	C15	C18	C19	108.3(3)
C24	C23	N1	121.7(2)	C20	C18	C15	110.2(3)
C24	C23	C22	120.3(3)	C20	C18	C19	109.3(3)

C22	C23	N1	118.0(2)	C20	C18	C21	108.5(3)
C13	C14	C15	121.2(3)	C21	C18	C15	112.3(3)
C6	C8	C11	109.6(3)	C21	C18	C19	108.1(3)
C9	C8	C6	112.4(3)				

## 8. References

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