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Electronic Supplementary Information

Formation of (PNP)Rh complexes containing covalent rhodium-zinc bonds in studies of potential Rh-catalysed Negishi coupling

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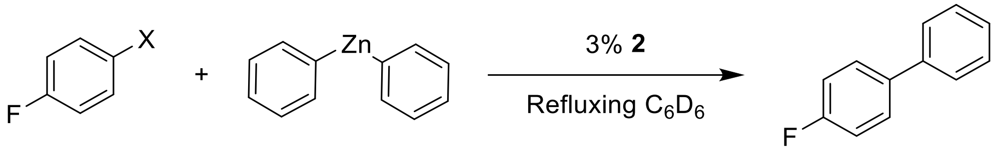
General Considerations. Unless otherwise specified, all manipulations were performed under an argon atmosphere using standard Schlenk or glovebox techniques. Toluene, ethyl ether, and pentane were dried and deoxygenated (by purging) using a solvent purification system by MBraun and stored over molecular sieves in an Ar-filled glove box. C_6D_6 was distilled from NaK/Ph_2CO/18-crown-6-ether and stored over molecular sieves in the glovebox. Liquid aryl halides (4-fluorochlorobenzene, 4-fluorobromobenzene, 4-fluoroiodobenzene, chlorobenzene, iodobenzene, and trifluorotoluene) were degassed and introduced into an argon filled glovebox and stored under molecular sieves. **1-Ph**[1]**, 1-Br**[1], and **1-I**[1] were previously characterized by our group. **1-Cl**[1] and **2**[2] were synthesized as described in the literature. Diphenylzinc was prepared as described in the literature.[3] Bis(4-fluorophenyl)zinc was prepared as described in the literature.[4] Phenylzinc chloride was prepared by mixing a 1:1 ratio of Ph_2Zn and $ZnCl_2$ in toluene and stirring overnight. $ZnCl_2$ was dried by refluxing in freshly distilled $SOCl_2$ followed by the removal of the volatiles via vacuum transfer.

Negishi Coupling Reactions

General procedure for Negishi Coupling Reactions. 2 (0.003 mmol, 100 µL of a 0.030 M solution in C_6D_6) was added to a J. Young tube and treated with *p*-FC_6H_4X (X= Cl, Br, I) (0.10 mmol, 100 µL of 1.0 M solution in *p*-FC_6H_4X / 0.33 M in $PhCF_3$ as an internal standard). Ph_2Zn (22 mg, 0.10 mmol) was added to the J. Young tube, and then the solution was diluted with 300 µL of C_6D_6). The J. Young tubes were placed in a 90 °C oil bath. Reaction progress was monitored by ^{19}F NMR spectroscopy.



Entry	X	Time	Starting Material Remaining	4-fluoro-biphenyl
1	Cl	24h	92%	0%
2	Br	24h	90%	4%
3	I	44 h	50%	7%

Coupling of 4-fluoroiodobenzene. In entry 3 there was a 36% conversion to a new product observed at -112.5 ppm by ^{19}F NMR spectroscopy. We hypothesize that this product is (phenyl)(4-fluorophenyl)zinc that is produced through an aryl-aryl transmetallation step.

Coupling of 4-fluorobromobenzene with slow addition of Ph_2Zn. 2 (0.007 mmol, 230 µL of a 0.030 M solution in C_6D_6) was added to a 100 mL Schlenk flask and treated with 4-fluorobromobenzene (25 µL, 0.23 mmol) and trifluorotoluene (10 µL, 0.08 mmol) was added as an internal standard. The solution was diluted with 1 mL of toluene. Ph_2Zn (50 mg, 0.23 mmol) was dissolved in 50 mL of toluene and transferred to an addition funnel. The addition funnel was mounted onto the Schlenk flask containing the reaction mixture, and the Ph_2Zn solution was added dropwise over a period of 6 hours while the reaction mixture was heated in a 90 °C oil bath. Within the first hour of Ph_2Zn addition, the reaction mixture had turned orange-brown color, which suggests that **4** is present as opposed to green (PNP)Rh species such as **1-Ph** or **(PNP)Rh(C_6H_4F)(Br)**.[5] The reaction progress was checked by ^{19}F NMR spectroscopy after 20 h, which showed no formation of 4-fluorobiphenyl, and >95% of the starting material remaining.

Observation of (*p*-FC_6H_4)Zn(C_6H_5). Bis(4-fluorophenyl)zinc (10 mg, 0.039 mmol) and Ph_2Zn (25 mg, 0.11 mmol) were dissolved in 500 μL of C_6D_6 in order to match the product ratio and concentration in the reaction mixture of the Negishi coupling of 4-fluoroiodobenzene with diphenyl zinc. After mixing the two diarylzinc reagents in solution for 1 hour, analysis by ^{19}F NMR showed a multiplet at -113.1 ppm.

Synthesis and Characterization

Treatment of 1-Cl with 1 eq. of Ph_2Zn. 1-Cl (38 mg, 0.059 mmol) was dissolved in C_6D_6 in a J. Young tube. Iodobenzene (13 μL, 0.12 mmol) was added to trap the 16-electron rhodium complex after the reductive elimination of biphenyl. The reaction mixture was treated with Ph_2Zn (13 mg, 0.059 mmol). Analysis by $^{31}P\{^1H\}$ NMR spectroscopy within 15 minutes of mixing showed **1-Ph** as the major product with trace amounts of **1-I** and **4**. The reaction mixture was left overnight at room temperature, and analysis by ^1H NMR spectroscopy in the morning showed the formation of biphenyl and **1-I** as the major rhodium complex.

Treatment of 2 with Ph_2Zn. 2 (10 mg, 0.018 mmol) was dissolved in C_6D_6 with iodobenzene (4 μL, 0.036 mmol) and treated with Ph_2Zn (4 mg, 0.018 mmol). Analysis with $^{31}P\{^1H\}$ NMR spectroscopy within 45 min. at room temperature showed **1-I** as the major product and **4** and **1-Cl** as minor side products. ^1H NMR spectroscopy showed the formation of C_6H_6.

Synthesis of (PNP)Rh(ZnPh)(Ph) (4). 3 (36 mg, 0.058 mmol) was added to a J. Young tube with Ph_2Zn (15 mg, 0.068 mmol) and dissolved in C_6D_6. The J. Young tube was rotated for 4 hours and was observed by $^{31}P\{^1H\}$ and ^1H NMR to convert >95% to **4**. The solution was diluted with pentane and filtered through Celite. The volatiles were removed and the red solid was redissolved in pentane and placed in a -35 °C freezer to recrystallize overnight. The product was isolated as a red solid (21 mg, 48% yield). $^{31}P\{^1H\}$ NMR (C_6D_6, 202 MHz): δ 46.2 (d, *J* = 119 Hz). ^1H NMR (C_6D_6, 500 MHz): δ 7.92 (d, *J* = 7.5 Hz, 2H, Ar-*H* of PNP), 7.76 (d, *J* = 7 Hz, 2H, Ar-*H*), 7.23 (d, *J* = 6.5 Hz, Ar-*H*), 7.18 (t, *J* = 7 Hz, 2H, *Ar*-H), 7.12 (m, 3H, Ar-*H*), 6.99 (t, *J* = 7 Hz, 1H, Ar-*H*), 6.95 (s, 2H, Ar-*H* of PNP), 6.86 (d, *J* = 8.5 Hz, 2H, Ar-*H* of PNP), 2.44 (m, 2H, C*H*Me_2), 2.19 (s, 6H, Ar-*Me*), 2.19 (m, 2H, C*H*Me_2), 1.12 (app. q (dvt), *J* = 8 Hz, 6H, PCHMe_2), 1.08 (app. q (dvt), *J* = 7 Hz, 6H, PCHMe_2), 0.95 (app. q (dvt), *J* = 6 Hz, 6H, PCHMe_2), 0.89 (app. q (dvt), *J* = 7.5 Hz, 6H, PCHMe_2). $^{13}C\{^1H\}$ NMR (C_6D_6, 100.6 MHz): δ 161.1 (dvt, *J* = 10 Hz, *J* = 1 Hz, *C*-N of PNP), 158.2 (dvt, *J* = 30.9 Hz, *J* = 9.9 Hz, *i*-Ph), 145.8 (d, *J* = 10.4 Hz, Zn-*C*), 140.8 (br. vt, *J* = 1.9 Hz, Ph), 137.3 (s, Ph), 132.4 (s, *Ar* PNP), 131.8 (s, *Ar* PNP), 128.6 (s, Ph), 126.7 (s, Ph), 125.9 (s, Ph), 125.1 (vt, *J* = 3.1 Hz, *Ar* PNP), 123.3 (vt, *J* = 18.2 Hz, *Ar* PNP), 121.2 (s, Ph), 116.5 (vt, *J* = 4.7 Hz, *Ar* of PNP), 20.61 (vt, *J* = 2.3 Hz, PCHMe_2), 20.57 (s, Ar-*Me*), 20.0 (vt, *J* = 2.4 Hz, PCHMe_2), 18.5 (vt, *J* = 2.7 Hz, PCHMe_2), 17.8 (s, PCHMe_2). Anal. Calcd for $C_{38}H_{50}NP_2RhZn$: C, 60.77; H, 6.71; N, 1.86 Found: C, 59.67; H, 6.34; N, 1.76.

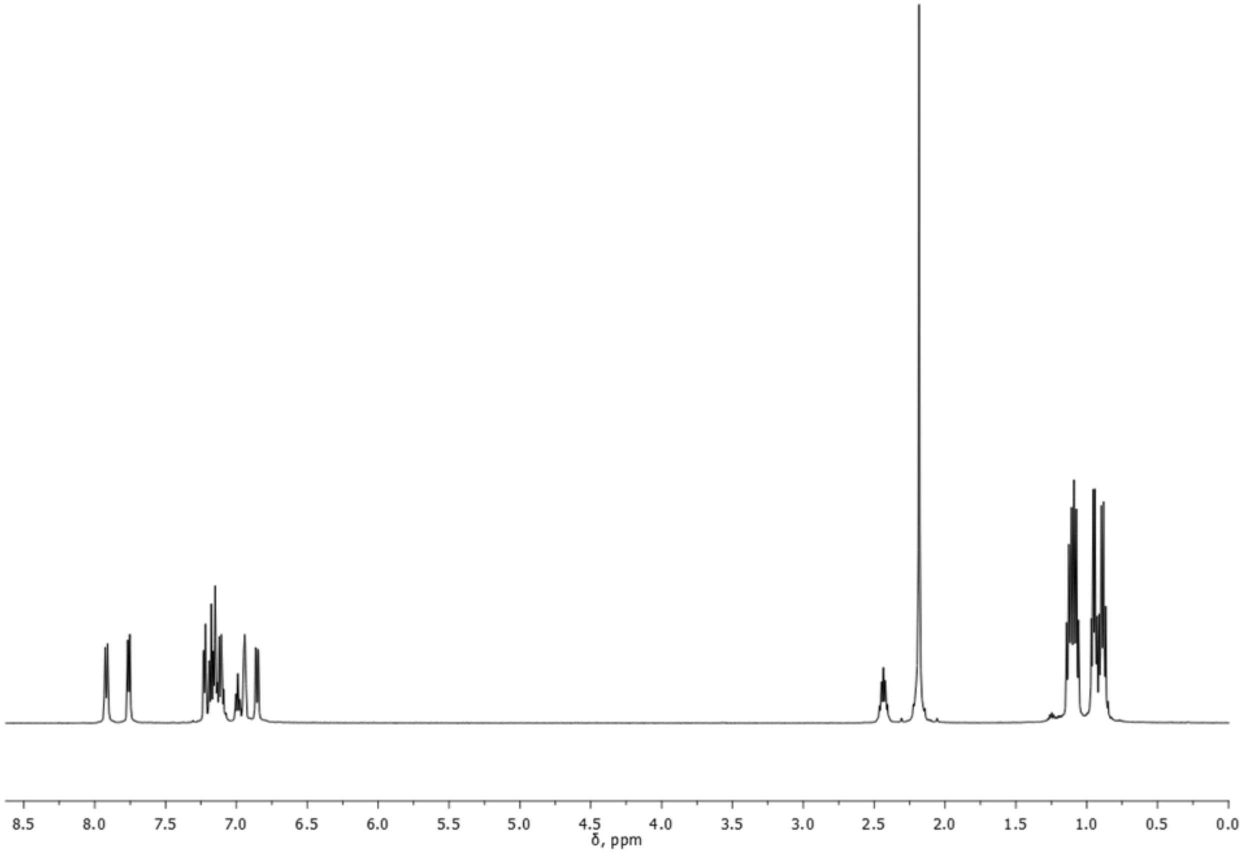


Figure S1: ^{1}H NMR spectrum of **4** in C_6D_6. Residual pentane can be observed at 0.86 and 1.23 ppm.

Treatment of 3 with 0.5 eq. Ph_2Zn. 3 (28 mg, 0.045 mmol) was dissolved in C_6D_6 in a J. Young tube and treated with Ph_2Zn (5 mg, 0.023 mmol). The reaction was mixed overnight at room temperature and examined by ^{1}H and ^{31}P{^{1}H} NMR spectroscopy. The mixture was observed to be a 1:1 mixture of **3** and **4** with 0.5 equivalents of free *tert*-butylethylene. The solution was transferred to a 10 mL Schlenk flask and the volatiles were removed. The solid residue was redissolved in C_6D_6 and transferred to a J. Young tube. The reaction was heated at 90 °C overnight. ^{1}H and ^{31}P{^{1}H} NMR showed no change in the distribution of products.

Treatment of 3 with 1 eq. of PhZnCl. 3 (31 mg, 0.05 mmol) was dissolved in C_6D_6 in a J. Young tube and treated with PhZnCl (9 mg, 0.05 mmol). Analysis by $^{31}P\{^1H\}$ NMR spectroscopy 20 minutes after mixing showed that about 34% of the reaction mixture was composed of **4**. The reaction was left for 24 h at room temperature and the color of the solution became brown. The mixture was analyzed by $^{31}P\{^1H\}$ NMR spectroscopy. Broad doublets were observed at 47.0 ppm ($J_{Rh\text{-}P}$ = 101 Hz) and 45.3 ppm (J = 116 Hz) and **4** still composed about 30% of the reaction mixture. The reaction was heated at 80 °C for 1 h, at which point the reaction turned green. Analysis by $^{31}P\{^1H\}$ NMR spectroscopy showed a mixture of approximately ten compounds. Compound **4** still composed about 30% of the mixture. **Treatment of 4 with PhCl. 4** (11 mg, 0.015 mmol) was dissolved in C_6D_6 in a J. Young tube and treated with chlorobenzene (8 μL, 0.079 mmol) and refluxed for 48 h. $^{31}P\{^1H\}$ NMR spectroscopy showed a 1:1 distribution of **6** and **1-Cl**. ^1H NMR spectroscopy showed the formation of approximately 1.5 equivalents of biphenyl.

***In Situ* observation of (PNP)Rh(ZnCl$_2$)$_{1.5}$ (6).** Method 1: **3** (18 mg, 0.029 mmol) was dissolved in C_6D_6 in a J. Young tube and treated with anhydrous $ZnCl_2$ (30 mg, 0.22 mmol) and mixed for 2 days at room temperature. The reaction had turned from an orange-red color to bright green. Analysis of the mixture by ^1H NMR spectroscopy shows complete consumption of **3** and free *tert*-butyl ethylene. Analysis by $^{31}P\{^1H\}$ NMR spectroscopy showed a broad doublet at 46.5 ppm (approximate $J_{Rh\text{-}P}$ = 100 Hz). Method 2: **3** (74 mg, 0.12 mmol) was dissolved in C_6D_6 and treated with anhydrous $ZnCl_2$ (24 mg, 0.18 mmol) in a J. Young tube and refluxed for 8 hours. The product could also be made in a shorter period of time by using >1.5 eq of anhydrous $ZnCl_2$ and refluxing the solution. Attempts to isolate the product via filtration through Celite and the removal of the volatiles under vacuum led to the partial formation of **7.** $^{31}P\{^1H\}$

NMR (202 MHz, C_6D_6): δ 46.5 (br. d, $J_{Rh\text{-}P}$ = 100 Hz); ^{1}H NMR (500 MHz, C_6D_6): δ 7.71 (br, 4H, Ar-*H*), 6.83 (br, 4H, Ar-*H*), 6.70 (d, *J* = 7 Hz, 4H, Ar-*H*), 2.52 (br, 4H, PC*H*Me_2), 2.27 (br, 4H, PC*H*Me_2), 2.14 (s, 12H, Ar-*Me*), 1.49 (br, 6H, PCH*Me*$_2$), 1.37 (br, 12H, PCH*Me*$_2$), 1.23 (br, 18H, PCH*Me*$_2$), 0.98 (br, 6H, PCH*Me*$_2$), 0.93 (br, 6H, PCH*Me*$_2$); ^{13}C{^{1}H} NMR (128 MHz, C_6D_6): δ 162.7 (br, *Ar*-N), 132.0 (s, *Ar*), 131.5 (br, *Ar*), 131.3 (br, *Ar*), 126.5 (s, *Ar*), 119.1 (br, *Ar*), 118.6 (br, *Ar*), 27.3 (br, P*C*HMe_2), 23.5 (br, P*C*HMe_2), 20.5 (s, Ar-*Me*), 20.3 (s, *Me*), 20.1 (s, *Me*), 18.7 (s, *Me*), 18.0 (s, *Me*).

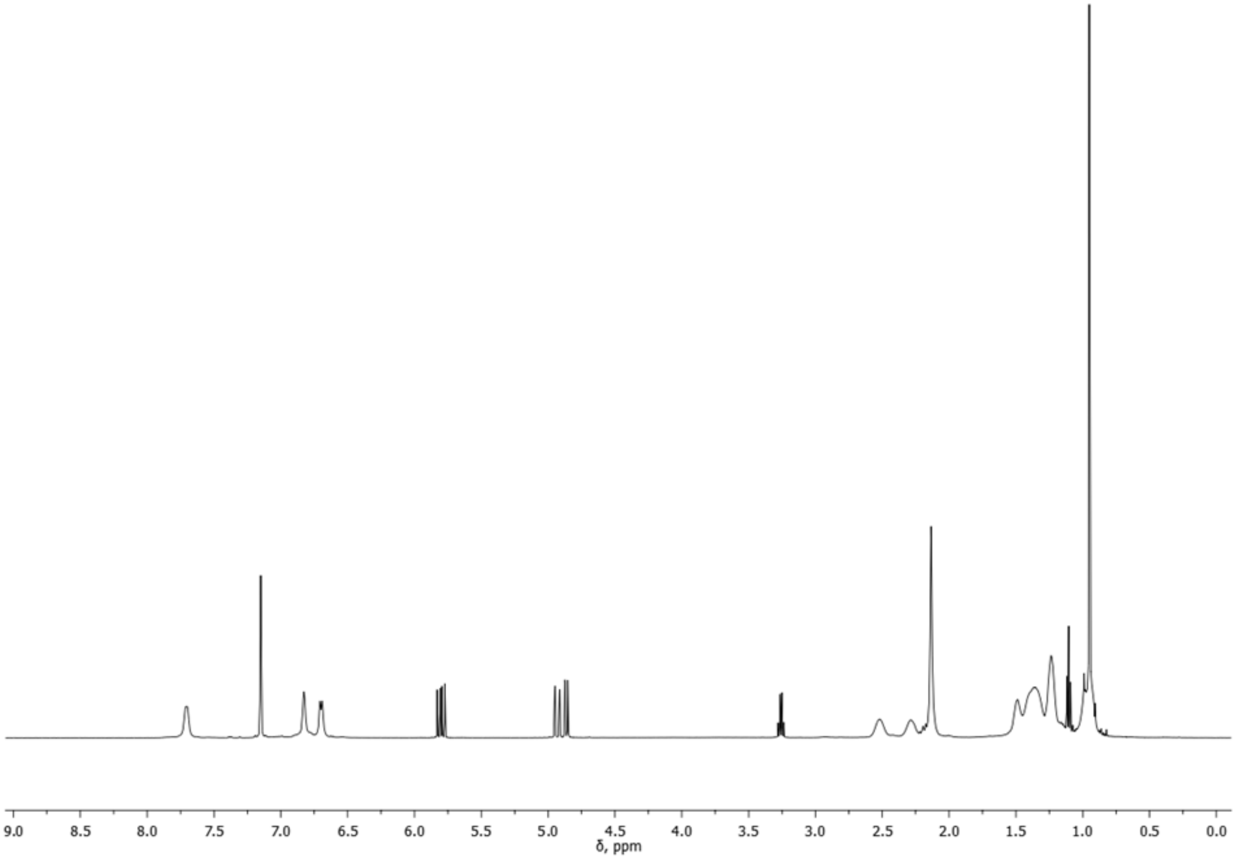


Figure S2: ^{1}H NMR spectrum of **6** characterized *in situ* in C_6D_6. Tert-butylethylene is observed at 5.80, 4.93, 4.86, and 0.95 ppm. Diethyl ether can be observed at 3.26 and 1.11 ppm.

Treatment of 3 with 0.6 eq. of $ZnCl_2$. **3** (75 mg, 0.12 mmol) was dissolved in C_6D_6 and treated with anhydrous $ZnCl_2$ (10 mg, 0.07 mmol). The mixture was heated overnight at 80 °C and produced a mixture of intractable products observed by ^1H NMR and $^{31}P\{^1H\}$ NMR containing **3** (~30%) and several other products. **6** accounted for ~5% of the reaction mixture.

Treatment of 3 with 1 eq. of $ZnCl_2$. **3** (75 mg, 0.11 mmol) was dissolved in C_6D_6 and treated with anhydrous $ZnCl_2$ (15 mg, 0.11 mmol) the reaction was heated at 80 °C for 48 hours. After this time, $^{31}P\{^1H\}$ NMR spectroscopy showed a mixture of products containing 23% **6** and 9% **3**.

Synthesis of $[(PNP)Rh(H)]_2[\mu\text{-}ZnCl_4]$ (7). Method 1: **3** (74 mg, 0.12 mmol) was dissolved in C_6D_6 and treated with anhydrous $ZnCl_2$ (24 mg, 0.18 mmol) in a J. Young tube and refluxed for 8 hours. Analysis by ^1H NMR spectroscopy indicated that the starting material had been converted to the product **6**. The reaction mixture was diluted with toluene and filtered through Celite and the volatiles were removed under vacuum. Analysis by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy showed a mixture of **6** and **7**. The volatiles were removed from the reaction to leave a fine flowing powder, which was exposed to the glovebox atmosphere and stirred as a solid for 5 days at which point the powder had turned purple. The product was extracted with pentane and filtered through Celite to yield **7** (48 mg, 63%). Method 2: **3** (26 mg, 0.042 mmol) was dissolved in C_6D_6 and treated with anhydrous $ZnCl_2$ (15 mg, 0.11 mmol) was refluxed for 8 h and showed complete conversion to **6**. The solution was then filtered through Celite to remove excess $ZnCl_2$ and transferred into a new J. Young tube. Water (1 μL, 0.056 mmol) was added to the solution and shaken vigorously for five seconds. Within less than a minute, the green solution had turned purple and contained a precipitate. Analysis by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy showed complete conversion to **7**. The solution was diluted with pentane and filtered through

Celite and the volatiles were removed under vacuum. The purple solid was recrystallized from pentane in a -35 °C freezer (16 mg, 63% yield). Method 3: **2** (14 mg, 0.025 mmol) was dissolved in C_6D_6 and treated with $ZnCl_2$ (10 mg, 0.073 mmol). Within 15 min. of mixing, the solution had turned from green to purple. Analysis by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy showed >95% conversion to **7**. Method 4: **(PNP)H** (300 mg, 0.70 mmol) and $[Rh(COD)Cl]_2$ (172 mg, 0.35 mmol) were dissolved in toluene and treated with $ZnCl_2$ (47 mg, 0.35 mmol) and stirred vigorously overnight at room temperature. The resulting solution was diluted with pentane and filtered through a pad of Celite and then the volatiles were removed under vacuum. The resulting solid was recrystallized by dissolving in diethyl ether and layering with pentane. **7** was isolated as a purple solid (352 mg, 80% yield). $^{31}P\{^1H\}$ NMR (121 MHz, C_6D_6): δ 49.6 (d, $J_{Rh\text{-}P}$ = 104 Hz), 49.5 (d, $J_{Rh\text{-}P}$ = 104 Hz); ^1H NMR (500 MHz, C_6D_6): δ 7.42 (br, 4H, Ar-*H*), 6.89 (br, 4H, Ar-*H*), 6.61 (dd, *J* = 8.5 Hz, *J* = 1.5 Hz, 4H, Ar-*H*), 3.05 (br, 2H, PC*H*Me₂), 2.83 (br, 2H, PC*H*Me₂), 2.22 (br, 2H, PC*H*Me₂), 2.14 (br, 2H, PC*H*Me₂), 2.10 (s, 12H, PC*H*Me₂), 1.53 (dt (apparent q.), *J* = 7.5 Hz, 12H, PCH*Me*₂), 1.45 (br, 6H, PCH*Me*₂), 1.36 (br, 6H, PCH*Me*₂), 1.30 (br, 12H, PCH*Me*₂), 1.12 (br, 6H, PCH*Me*₂), 1.07 (br, 6H, PCH*Me*₂), -19.98 (dt, $J_{Rh\text{-}H}$ = 26 Hz, $J_{P\text{-}H}$ = 10.5 Hz, 2H, Rh-H); $^{13}C\{^1H\}$ NMR (128 MHz, C_6D_6): δ 161.5 (br., C-N), 161.1 (br., C-N), 131.4 (s), 130.9 (s), 125.4 (br.), 124.9 (br.), 122.5 (s), 122.4 (s), 122.2 (s), 117.7 (br.), 116.2 (br.), 26.4 (br.), 25.5 (overlapping br.), 25.1 (br.), 20.6 (s, Ar-*Me*), 19.4 (s), 19.2 (s), 19.0 (s), 18.9 (s), 18.6 (br.). Anal. Calcd for $C_{52}H_{82}Cl_4N_2P_4Rh_2Zn$: C, 49.10; H, 6.50; N, 2.20 Found: C, 48.83; H, 5.95; N, 2.09.

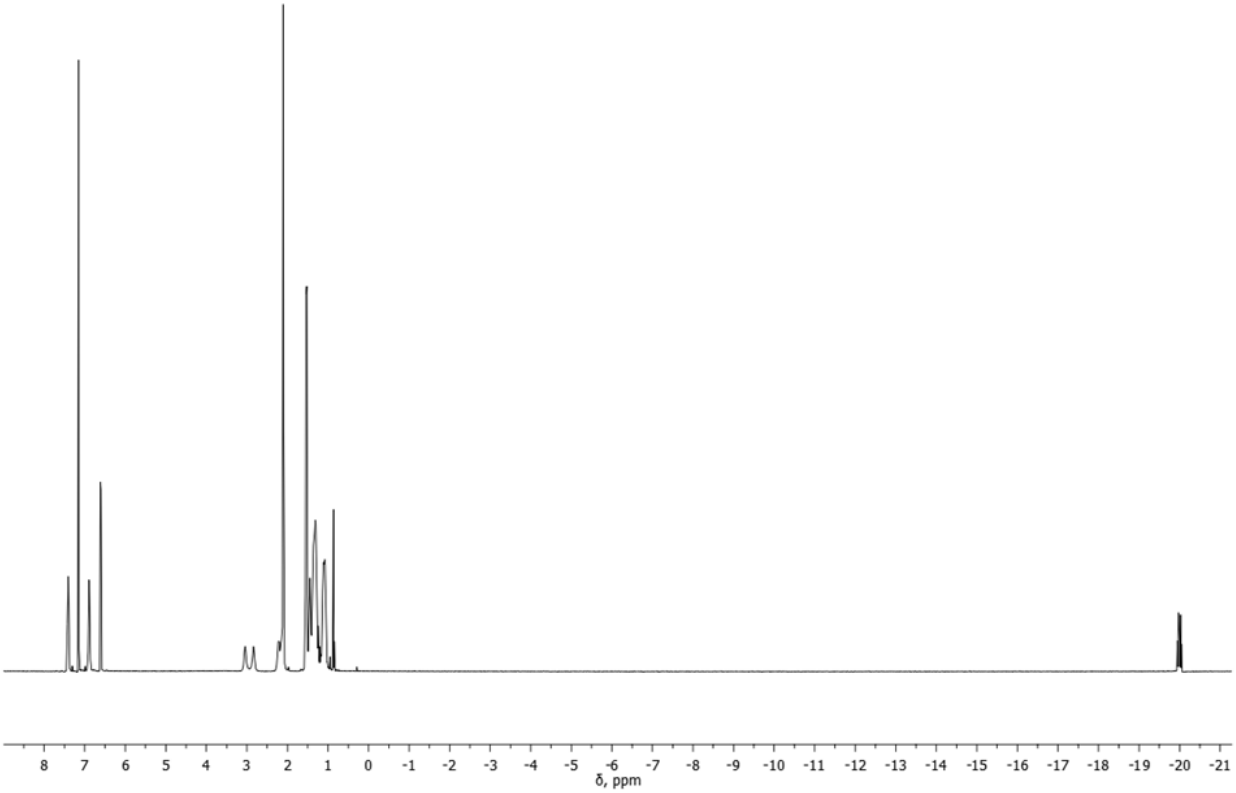


Figure S3: ^{1}H NMR Spectrum of **7** in C_6D_6. Residual silicone grease can be observed at 0.29 ppm. Pentane is visible at 0.86 ppm and 1.23 ppm.

Treatment of 6 with chlorobenzene. 3 (23 mg, 0.037 mmol) was treated with $ZnCl_2$ (15 mg, 0.11 mmol) in C_6D_6. The reaction was heated at 80 °C for 4 h, at which time complete conversion to **6** was observed. The reaction was then filtered through Celite in toluene, and the volatiles were removed. The residue was dissolved in 600 μL of C_6D_6 and transferred to a J. Young tube. Chlorobenzene (19 μL, 0.185 mmol) was added to the reaction and heated at 75 °C for 48 hours. Conversion to **1-Cl** was observed to be <10% by ^{1}H NMR spectroscopy.

Treatment of 6 with bromobenzene. 3 (23 mg, 0.037 mmol) was treated with $ZnCl_2$ (15 mg, 0.11 mmol) in C_6D_6. The reaction was heated at 80 °C for 4 h, at which time complete conversion to **6** was observed. The reaction was then filtered through Celite in toluene, and the volatiles were removed. The residue was dissolved in 600 µL C_6D_6 and transferred to a J. Young tube. Phenyl bromide (20 µL, 0.185 mmol) was added to the reaction which was then heated at 75 °C for 48 hours. At this time, conversion to **1-Br** was observed to be 73% by ^{1}H NMR spectroscopy.

Treatment of 6 with iodobenzene. 3 (23 mg, 0.037 mmol) was treated with $ZnCl_2$ (15 mg, 0.11 mmol) in C_6D_6. The reaction was heated at 80 °C for 4 h, at which time complete conversion to **6** was observed. The reaction was then filtered through Celite in toluene, and the volatiles were removed. The residue was dissolved in 600 µL C_6D_6 and transferred to a J. Young tube. Phenyl iodide (21 µL, 0.185 mmol) was added to the reaction, which was then heated for 1 h at 75 °C. Analysis by ^{1}H and ^{31}P{^{1}H} NMR spectroscopy showed complete conversion to **1-I**. A precipitate was also observed, presumably $ZnCl_2$.

Treatment of 3 with chlorobenzene. 3 (23 mg, 0.037 mmol) was dissolved in 600 µL of C_6D_6 in a J. Young tube and treated with chlorobenzene (19 µL, 0.185 mmol). The reaction was heated at 75 °C for 45 min. and was observed by ^{31}P{^{1}H} NMR spectroscopy to be >95% converted to **1-Cl**.

X-Ray Structural Determination Details

X-ray data collection, solution, and refinement for (PNP)Rh(Ph)(ZnPh) (4). A dark red, multi-faceted block of suitable size (0.24 x 0.14 x 0.11 mm) was selected from a representative sample of crystals of the same habit using an optical microscope and mounted onto a nylon loop. Low temperature (110 K) X-ray data were obtained on a Bruker APEXII CCD based diffractometer (Mo sealed X-ray tube, K_α = 0.71073 Å). All diffractometer manipulations, including data collection, integration and scaling were carried out using the Bruker APEXII software.[6] An absorption correction was applied using SADABS.[7] The space group was determined on the basis of systematic absences and intensity statistics and the structure was solved by direct methods and refined by full-matrix least squares on F^2. The structure was solved in the monoclinic C c space group using XS[8] (incorporated in SHELXLE). All non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogen atoms were placed in idealized positions and refined using riding model. The structure was refined (weighted least squares refinement on F^2) and the final least-squares refinement converged. Twin was found by using TwinRotMat incorporated in PLATON program with a twin matrix of -1 0 0 0 -1 0 1 0 1 and a BASF number of 0.07, which refined to 0.12 as final refinement.[9]

X-ray data collection, solution, and refinement for [(PNP)Rh(Ph)]$_2$(μ-Zn) (5). An orange, multi-faceted plate of suitable size (0.30 x 0.17 x 0.03 mm) was selected from a representative sample of crystals of the same habit using an optical microscope and mounted onto a nylon loop. Low temperature (110 K) X-ray data were obtained on a Bruker APEXII CCD based diffractometer (Mo sealed X-ray tube, K_α = 0.71073 Å). All diffractometer manipulations, including data collection, integration and scaling were carried out using the Bruker APEXII software.[6] An absorption correction was applied using SADABS.[7] The space group was determined on the basis of systematic absences and intensity statistics and the structure was solved by direct methods and refined by full-matrix least squares on F^2. The structure was solved in the monoclinic P 2_1/c space group using XS[8] (incorporated in SHELXLE). All non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogen atoms were placed in idealized positions and refined using riding model. The structure was refined (weighted least squares refinement on F^2) and the final least-squares refinement converged. No additional symmetry was found using ADDSYM incorporated in PLATON program.[9] The SQUEEZE protocol included in PLATON was used to account for disordered solvent molecules found in the crystal lattice that could not be satisfactorily modeled.[10]

X-ray data collection, solution, and refinement for [(PNP)Rh(H)]$_2$[μ-ZnCl$_4$] (7). A purple, multi-faceted block of suitable size (0.21 x 0.12 x 0.11 mm) was selected from a representative sample of crystals of the same habit using an optical microscope and mounted onto a nylon loop. Low temperature (110 K) X-ray data were obtained on a Bruker APEXII CCD based diffractometer (Cu sealed X-ray tube, K_α = 1.54178 Å). All diffractometer manipulations, including data collection, integration and scaling were carried out using the Bruker APEXII software.[6] An absorption correction was applied using SADABS.[7] The space group was determined on the basis of systematic absences and intensity statistics and the structure was solved by direct methods and refined by full-matrix least squares on F^2. The structure was solved in the monoclinic P 2_1/c space group using XS[8] (incorporated in SHELXLE). All non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogen atoms were placed in idealized positions and refined using riding model. Hydrogen atoms on Rh could not be located from difference Fourier maps and were removed. The structure was refined (weighted least squares refinement on F^2) and the final least-squares refinement converged. No additional symmetry was found using ADDSYM incorporated in PLATON program.[9] The SQUEEZE protocol included in PLATON was used to account for disordered solvent molecules found in the crystal lattice that could not be satisfactorily modeled.

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