

Supporting Information for:

## Synthesis of Pyrrole-Based PSiP Pincer Ligands and Their Palladium, Rhodium, and Platinum Complexes

Julia F. Vidlak,<sup>a</sup> Mario N. Cosio,<sup>b</sup> Nihal K. Sriramaneni,<sup>a</sup> Nattamai Bhuvanesh,<sup>b</sup> Oleg V. Ozerov,<sup>\*b</sup> and Miles W. Johnson<sup>\*a</sup>

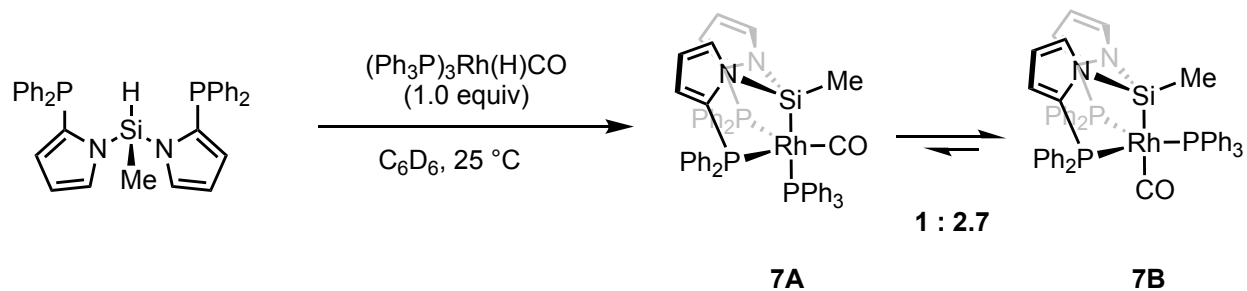
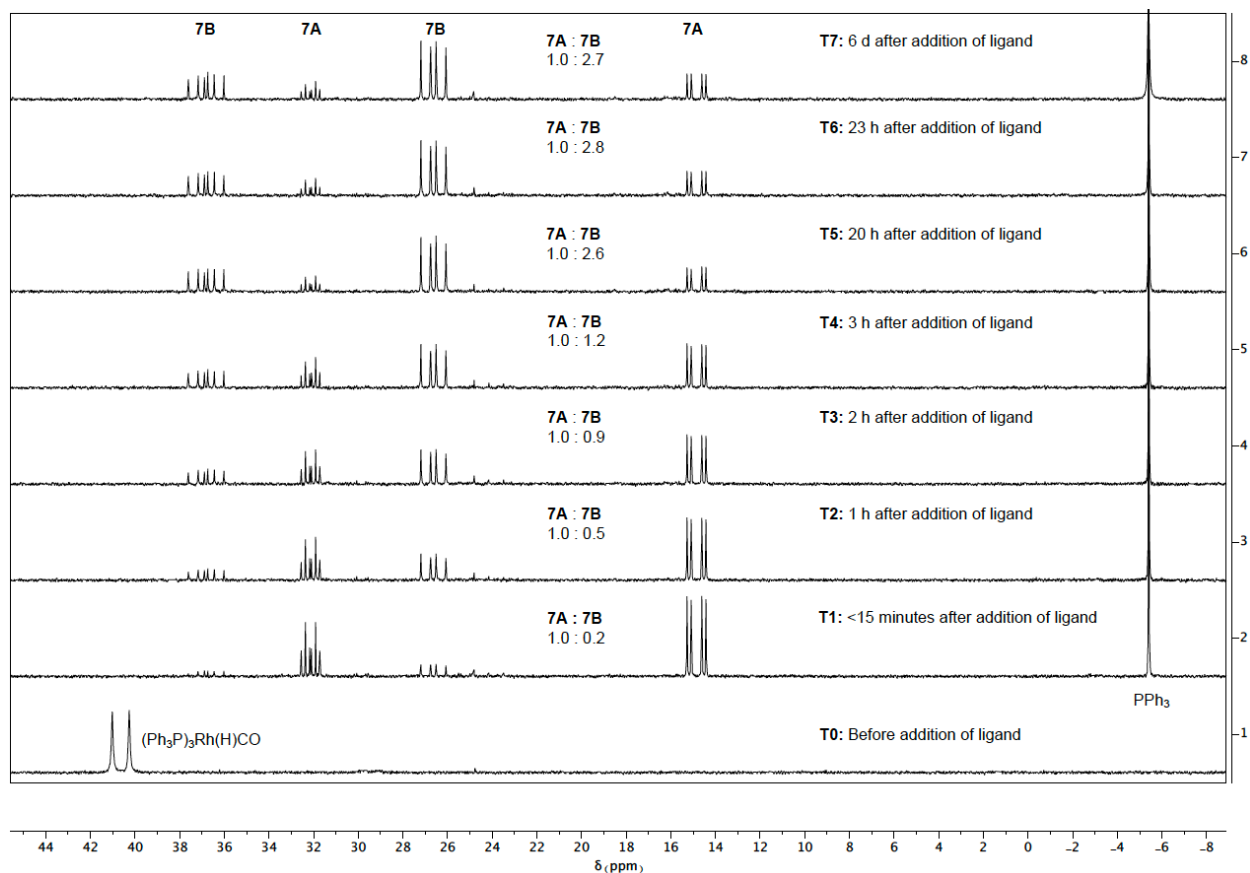
<sup>a</sup> Department of Chemistry, University of Richmond, Richmond, Virginia 23173, USA

<sup>b</sup> Department of Chemistry, Texas A&M University, College Station, Texas 77842, USA

### Table of Contents

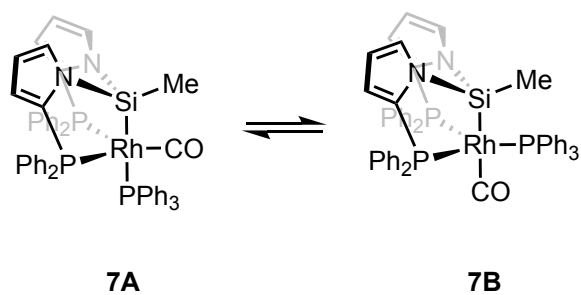
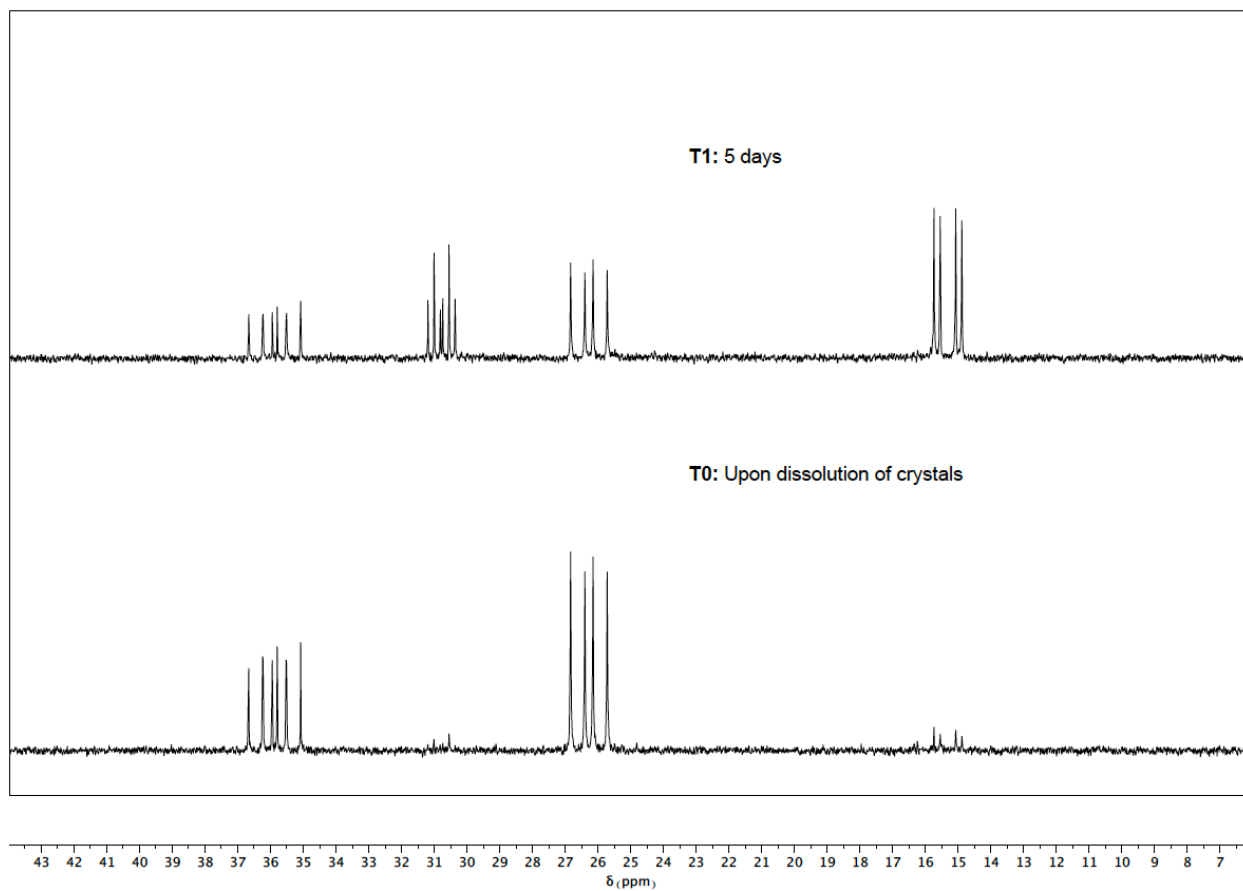
I.	Isomerization of [( <sup>Ph</sup> P <sub>Py</sub> ) <sub>2</sub> Si]Rh(CO)PPh <sub>3</sub> ( <b>7A/7B</b> ) .....	S-2
II.	Buried Volume Details for [( <sup>Ph</sup> P <sub>Py</sub> ) <sub>2</sub> Si]PdCl ( <b>4</b> ) and [( <sup>Ph</sup> P <sub>Ph</sub> ) <sub>2</sub> Si]PdCl.....	S-5
III.	Cyclic Voltammograms for [( <sup>Ph</sup> P <sub>Py</sub> ) <sub>2</sub> Si]PdCl ( <b>4</b> ) and [( <sup>Ph</sup> P <sub>Ph</sub> ) <sub>2</sub> Si]PdCl .....	S-7
IV.	Infrared Spectra of [( <sup>Ph</sup> P <sub>Py</sub> ) <sub>2</sub> Si]Rh(CO)PPh <sub>3</sub> ( <b>7A/7B</b> ) .....	S-9
V.	NMR Spectra of Proligands .....	S-11
VI.	NMR Spectra of Transition Metal Complexes .....	S-17
VII.	X-Ray Crystallographic Data.....	S-35
VIII.	References.....	S-38

## I. Isomerization of 7A/7B



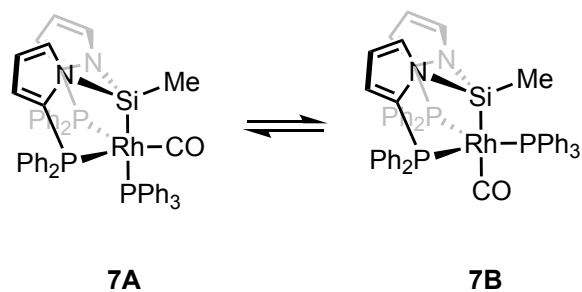
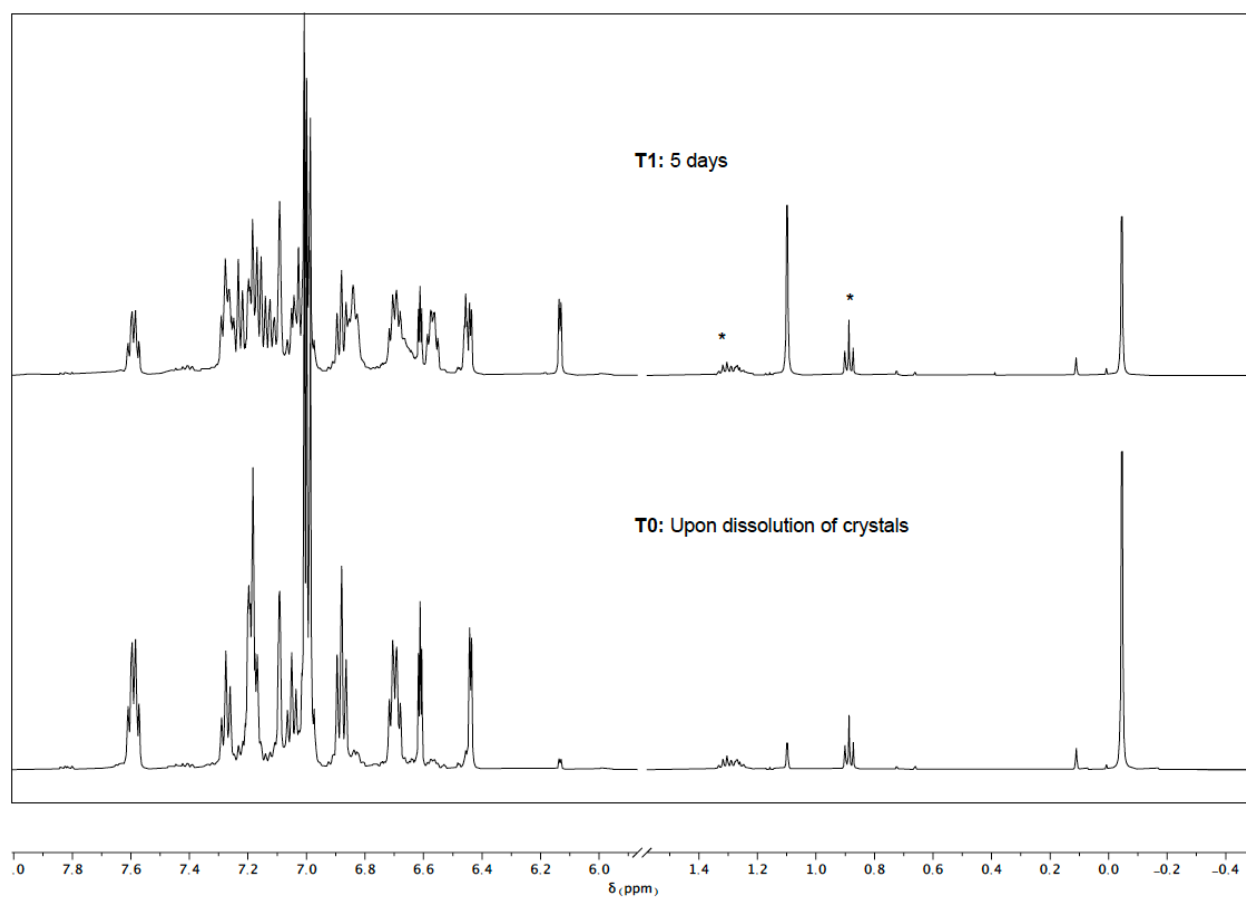
**Figure S1.** Time-course of reaction of  $(\text{Ph}_3\text{P})_3\text{Rh}(\text{H})\text{CO}$  with **2** in  $\text{C}_6\text{D}_6$  as monitored by  $^{31}\text{P}\{^1\text{H}\}$  NMR.

*Procedure:* A J. Young tube was charged with  $(\text{Ph}_3\text{P})_3\text{Rh}(\text{H})\text{CO}$  (18 mg, 0.020 mmol), proligand **2** (11 mg, 0.020 mmol), and benzene- $d_6$  (0.6 mL).  $^1\text{H}$  and  $^{31}\text{P}\{^1\text{H}\}$  spectra were acquired periodically. The ratio of **7A** to **7B** was found to be 1:2.6 and 1:2.7 in two experiments over the course of 3 to 6 days



Starting from  
crystalline **7B**

**Figure S2.** Conversion of crystals of **7B** to mixture of **7A/7B** in methylene chloride- $d_2$  as monitored by  $^{31}\text{P}\{^1\text{H}\}$  NMR (1.0:0.9 **7A:7B**). Equilibration was also observed in benzene- $d_6$  in which **7B** was the major species (1:3.2 of **7A:7B**).



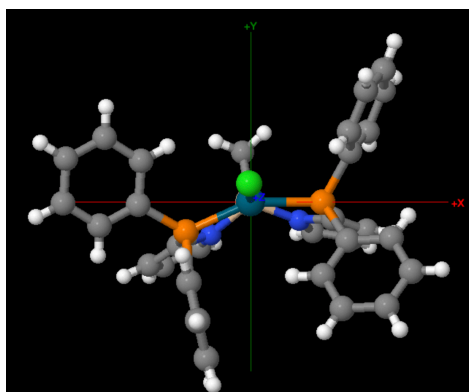
*Starting from  
crystalline 7B*

**Figure S3.** Conversion of crystals of **7B** to mixture of **7A/7B** in methylene chloride- $d_2$  as monitored by  $^1\text{H}$  NMR (1.0:0.9 **7A:7B**). Equilibration was also observed in benzene- $d_6$  in which **7B** was the major species (1:3.2 of **7A:7B**). Pentane is indicated with an asterisk (\*).

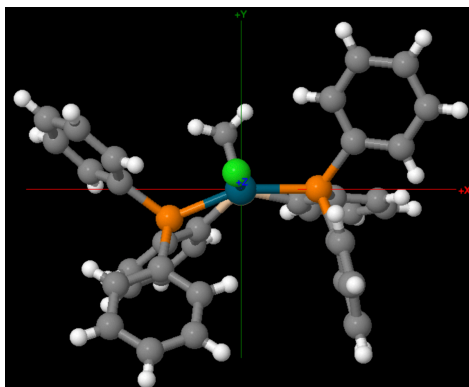
## II. Buried Volume Details for $[(^{\text{Ph}}\text{P}_{\text{Py}})_2\text{Si}]\text{PdCl}$ (4) and $[(^{\text{Ph}}\text{P}_{\text{Ph}})_2\text{Si}]\text{PdCl}$

Buried volume calculations were performed by inputting the following information into the SambVca 2.1 web application<sup>1</sup> based on the crystal structures of  $[(^{\text{Ph}}\text{P}_{\text{Py}})_2\text{Si}]\text{PdCl}$  (4) and  $[(^{\text{Ph}}\text{P}_{\text{Ph}})_2\text{Si}]\text{PdCl}$ .<sup>2</sup>

1. **Load file:** .xyz file of crystal structure used
2. **Select the atoms coordinated to the center of the sphere:** Palladium selected
3. **Select the atoms for z-axis definition:** Silicon selected with z-negative orientation
4. **Select the atoms for xz-plane definition:** Phosphorus selected that is to the right of the methyl group oriented upward.
5. **Select the atoms to be deleted:** Palladium and chloride selected.
6. **Check the chosen orientation:**

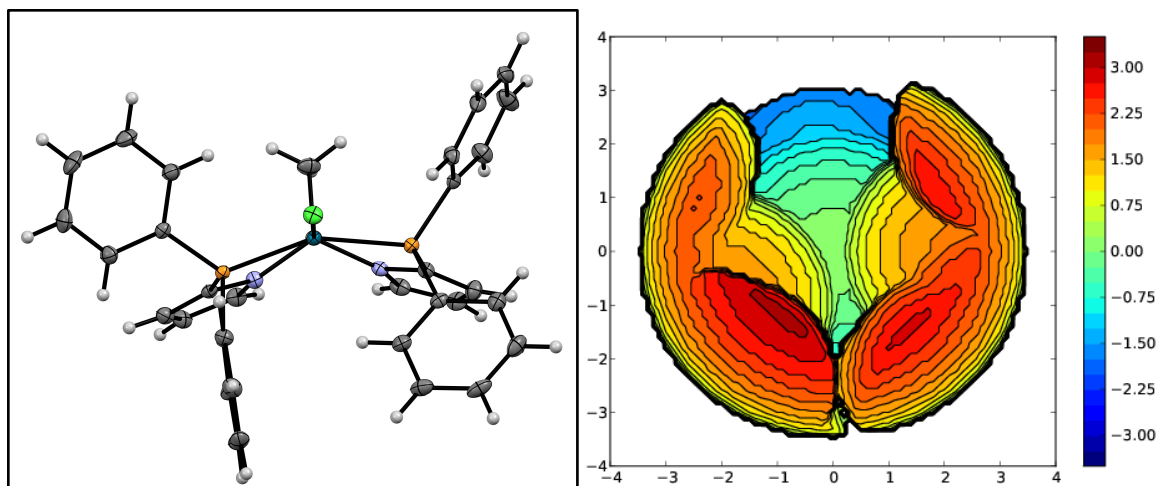


$[(^{\text{Ph}}\text{P}_{\text{Py}})_2\text{Si}]\text{PdCl}$  (4)

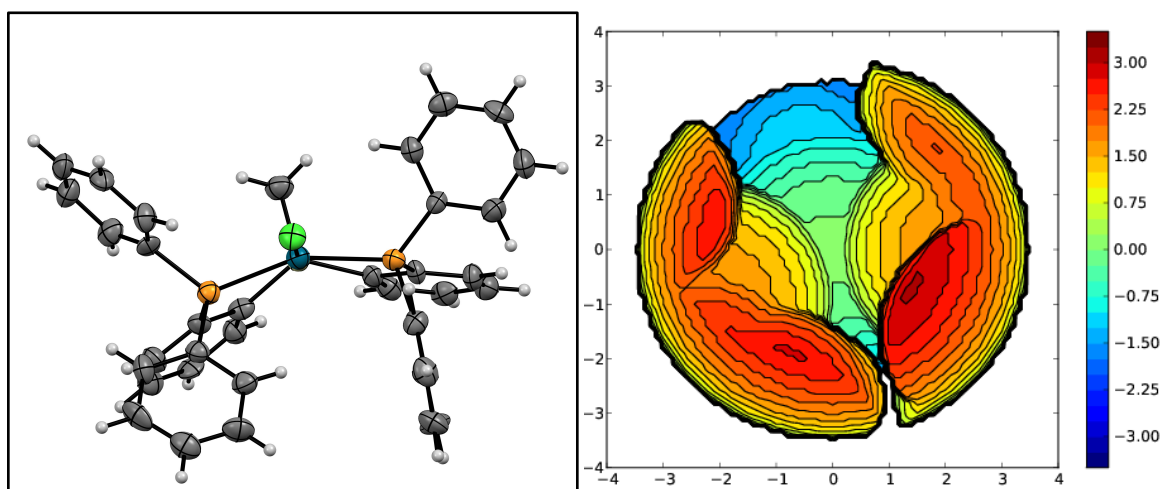


$[(^{\text{Ph}}\text{P}_{\text{Ph}})_2\text{Si}]\text{PdCl}$

7. **Select the atomic radii:** Bond scaled by 1.17
8. **Sphere radius:** 3.5
9. **Distance of the coordination point from the center of the sphere:** 0.0
10. **Mesh spacing for numerical integration:** 0.10
11. **Inclusion of H atoms:** H atoms included

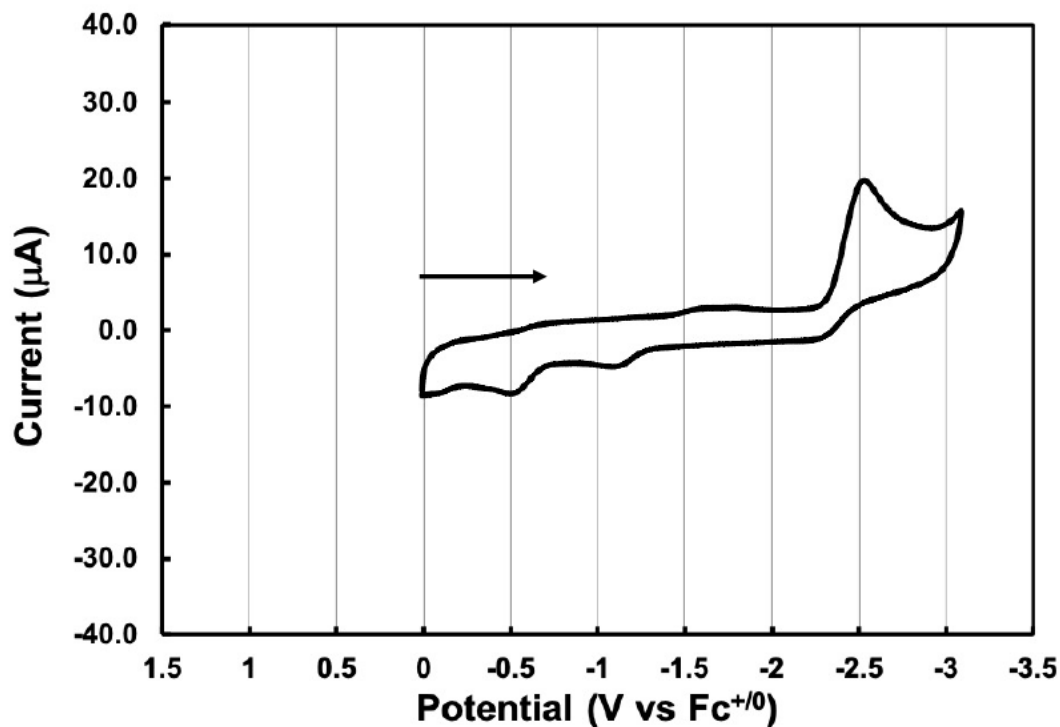


**Figure S4:** Steric map of  $[(^{\text{Ph}}\text{Ppy})_2\text{Si}]\text{PdCl}$ ;  $\%V_{\text{Bur}} = 71.4\%$

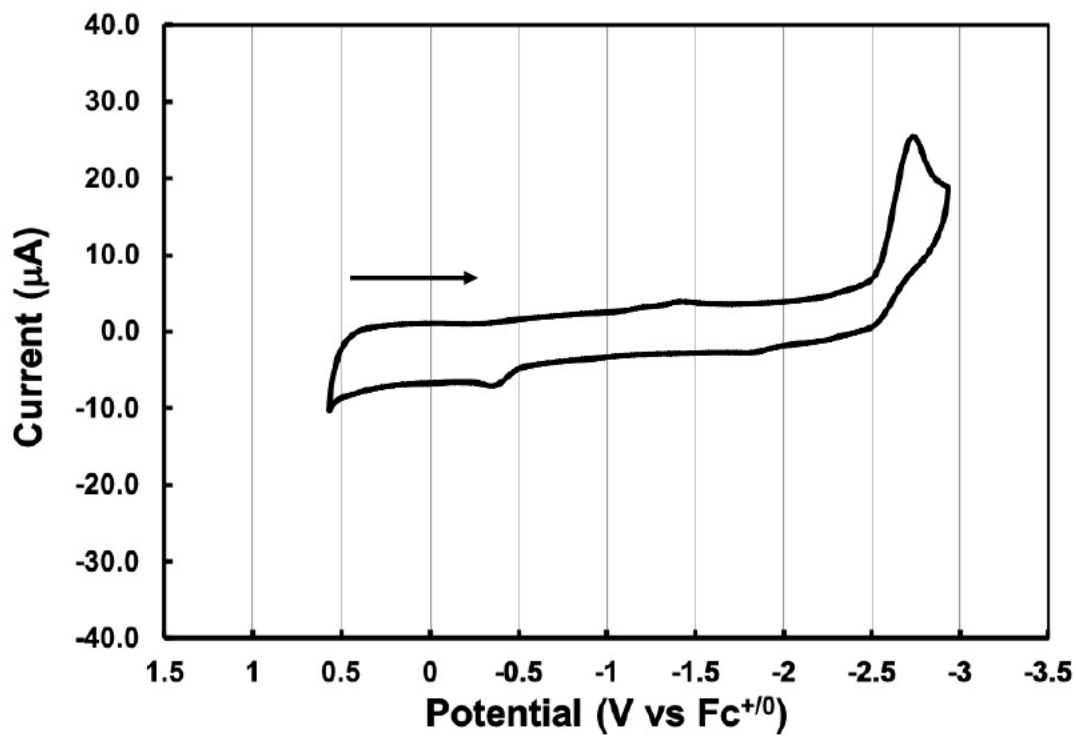


**Figure S5:** Steric map of  $[(^{\text{Ph}}\text{PPh})_2\text{Si}]\text{PdCl}$ ;  $\%V_{\text{Bur}} = 71.1\%$

### III. Cyclic Voltammograms of $[(^{\text{Ph}}\text{P}_{\text{Py}})_2\text{Si}]\text{PdCl}$ (4) and $[(^{\text{Ph}}\text{P}_{\text{Ph}})_2\text{Si}]\text{PdCl}$



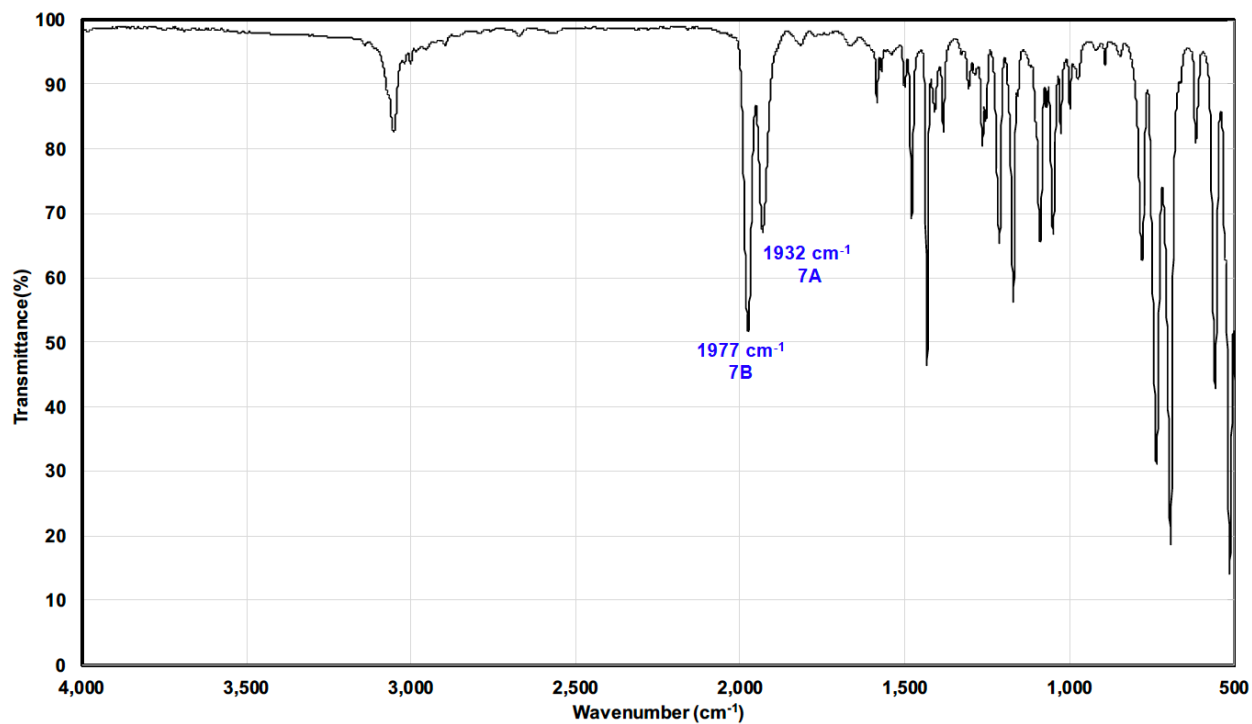
**Figure S6.** Cyclic voltammogram of  $[(^{\text{Ph}}\text{P}_{\text{Py}})_2\text{Si}]\text{PdCl}$  (4) in THF. Conditions: scan rate: 300 mV/s; supporting electrolyte: 0.1 M  $[n\text{-Bu}_4\text{N}][\text{PF}_6]$ ; working electrode: glassy carbon; auxiliary electrode: platinum wire; reference: silver wire referenced to the  $\text{Fc}^{+/0}$  couple.



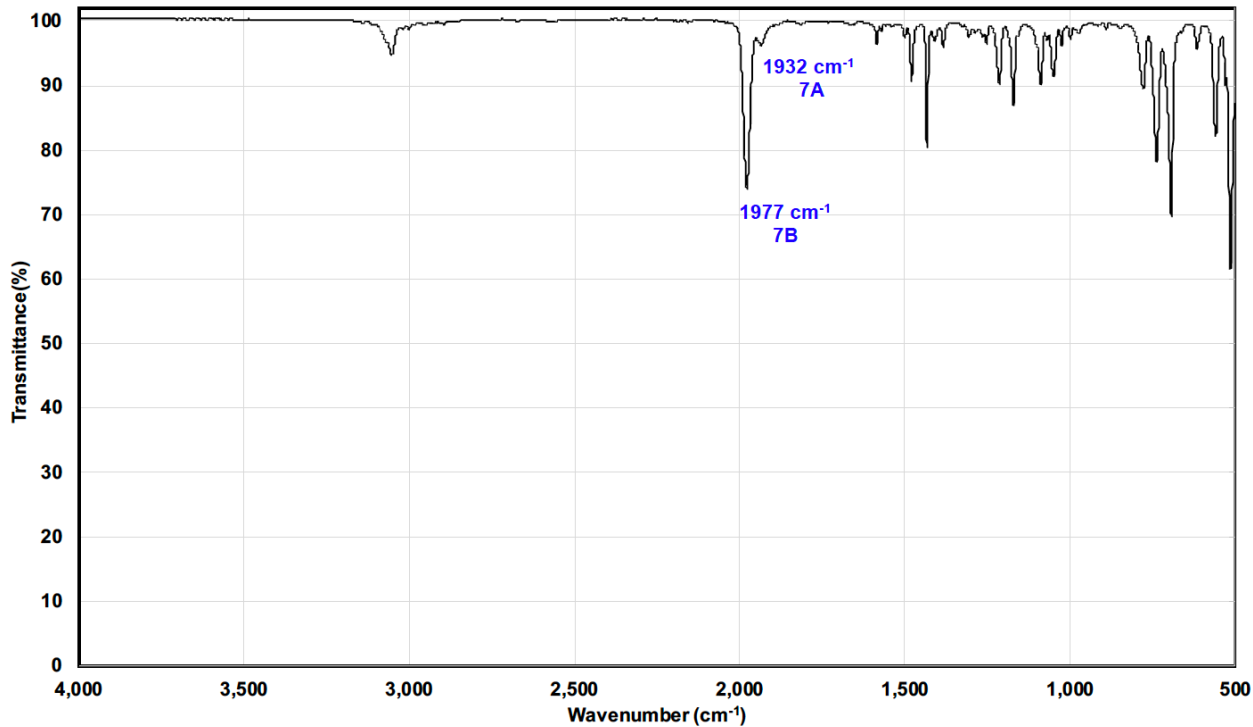
**Figure S7.** Cyclic voltammogram of  $[(\text{PhPPh})_2\text{Si}]\text{PdCl}$  in THF. Conditions: scan rate: 300 mV/s; supporting electrolyte: 0.1 M  $[n\text{-Bu}_4\text{N}][\text{PF}_6]$ ; working electrode: glassy carbon; auxiliary electrode: platinum wire; reference: silver wire referenced to the  $\text{Fc}^{+/0}$  couple.



#### IV. IR Spectra of [(<sup>Ph</sup>P<sub>Py</sub>)<sub>2</sub>Si]Rh(CO)PPh<sub>3</sub> (7A/7B)



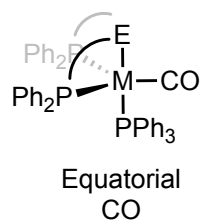
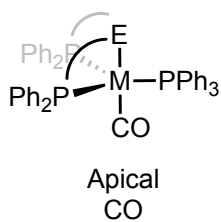
**Figure S8.** Infrared spectrum of an equilibrium mixture of 7A/7B.



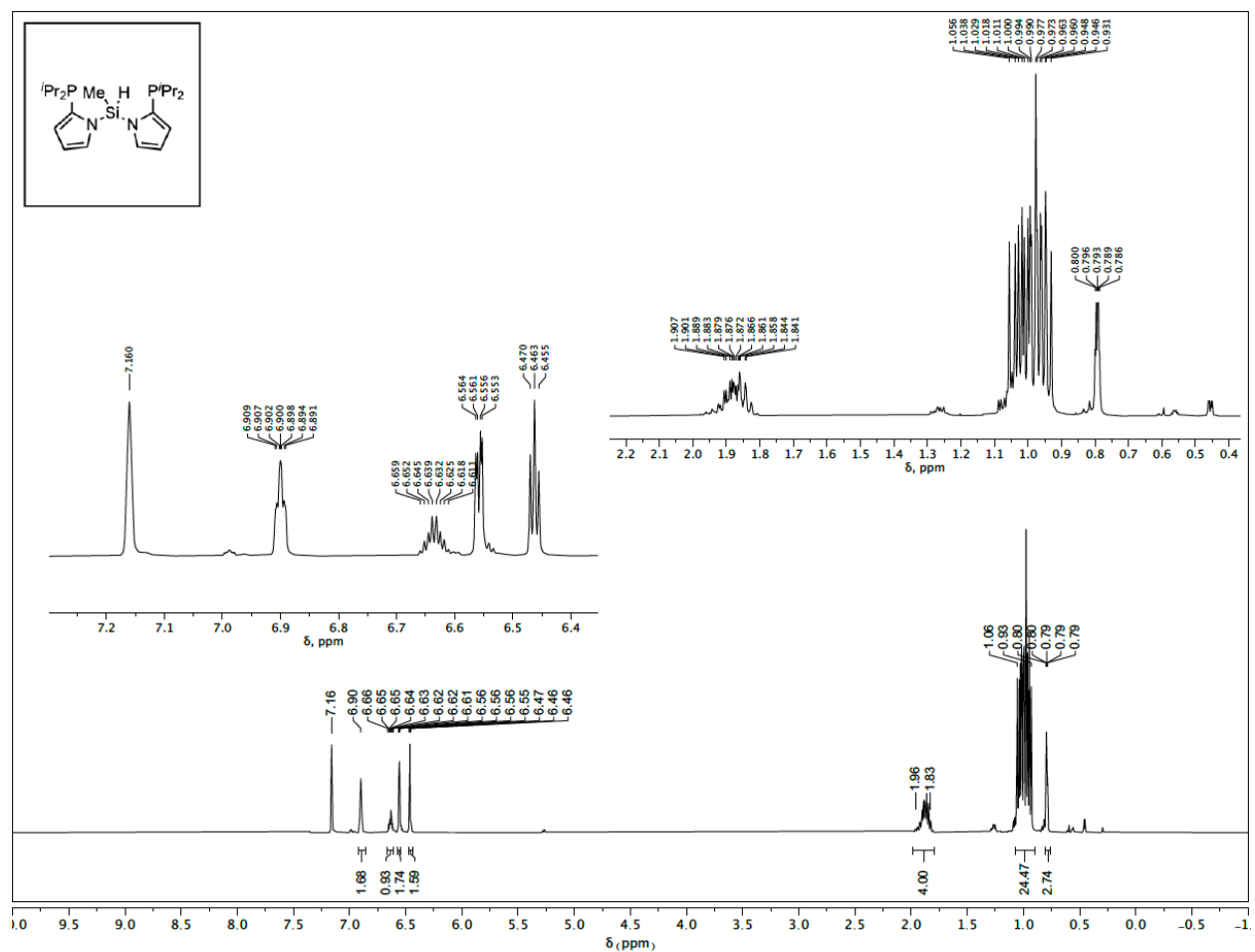
**Figure S9.** Infrared spectrum of crystalline 7B. Material was dissolved in DCM and immediately deposited as a thin film.

**Table S1:** Comparison of CO stretches and  $^2J_{P-P}$  (PPh<sub>2</sub>) for PEP complexes analogous to **7A/7B**.

Reference	Linker	Metal	E	Apical CO (cm <sup>-1</sup> )	$^2J_{P-P}$ (Hz)	Equatorial CO (cm <sup>-1</sup> )	$^2J_{P-P}$ (Hz)
3	Phenylene	Rh	SiMe	-	-	1897	34.9
4	Phenylene	Rh	GeMe	1968	99.1	1923	33.0
4	Phenylene	Rh	SnMe	1964	102.8	-	-
3	Phenylene	Ir	SiMe	1959	100.3	1918	25.7
5	Phenylene	Ir	SiF	1969	82.2	1937	28.0
<i>This work</i>	Pyrrole	Rh	SiMe	1977	88.0	1932	38.2

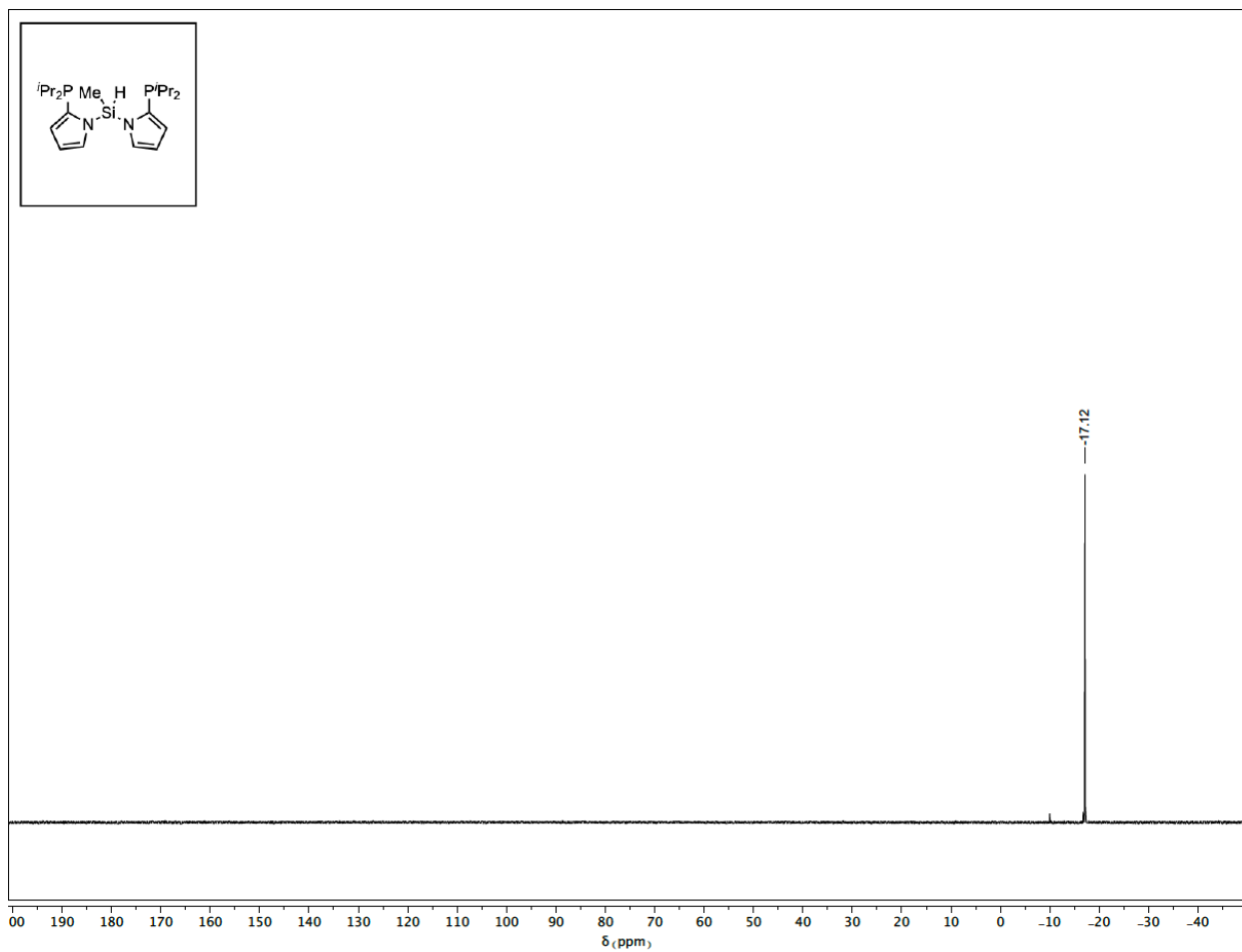


## V. NMR Spectra of Proligands

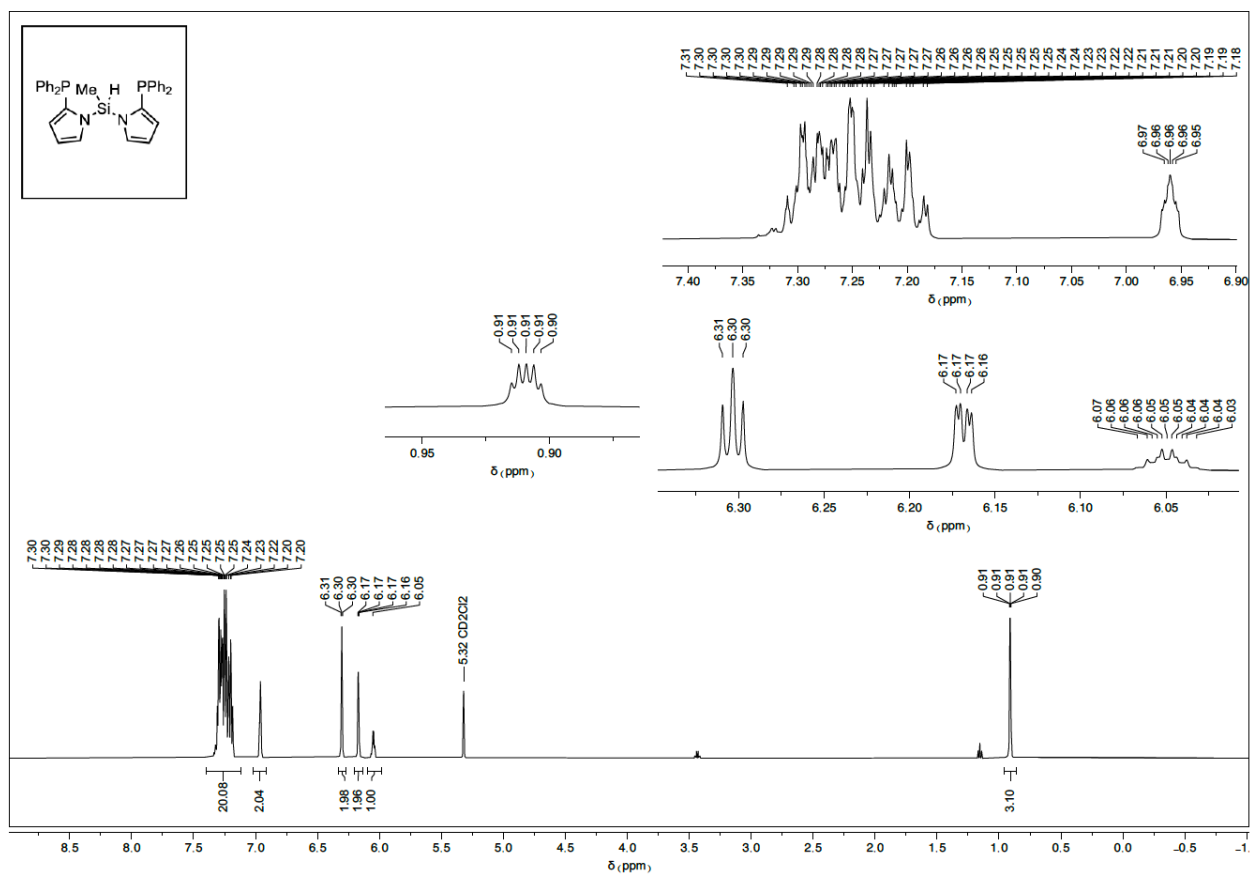


**Figure S10.**  $^1\text{H}$  NMR spectrum of bis(2-diisopropylphosphino-pyrrole)methylsilane (**1**) in  $\text{C}_6\text{D}_6$  (400 MHz).

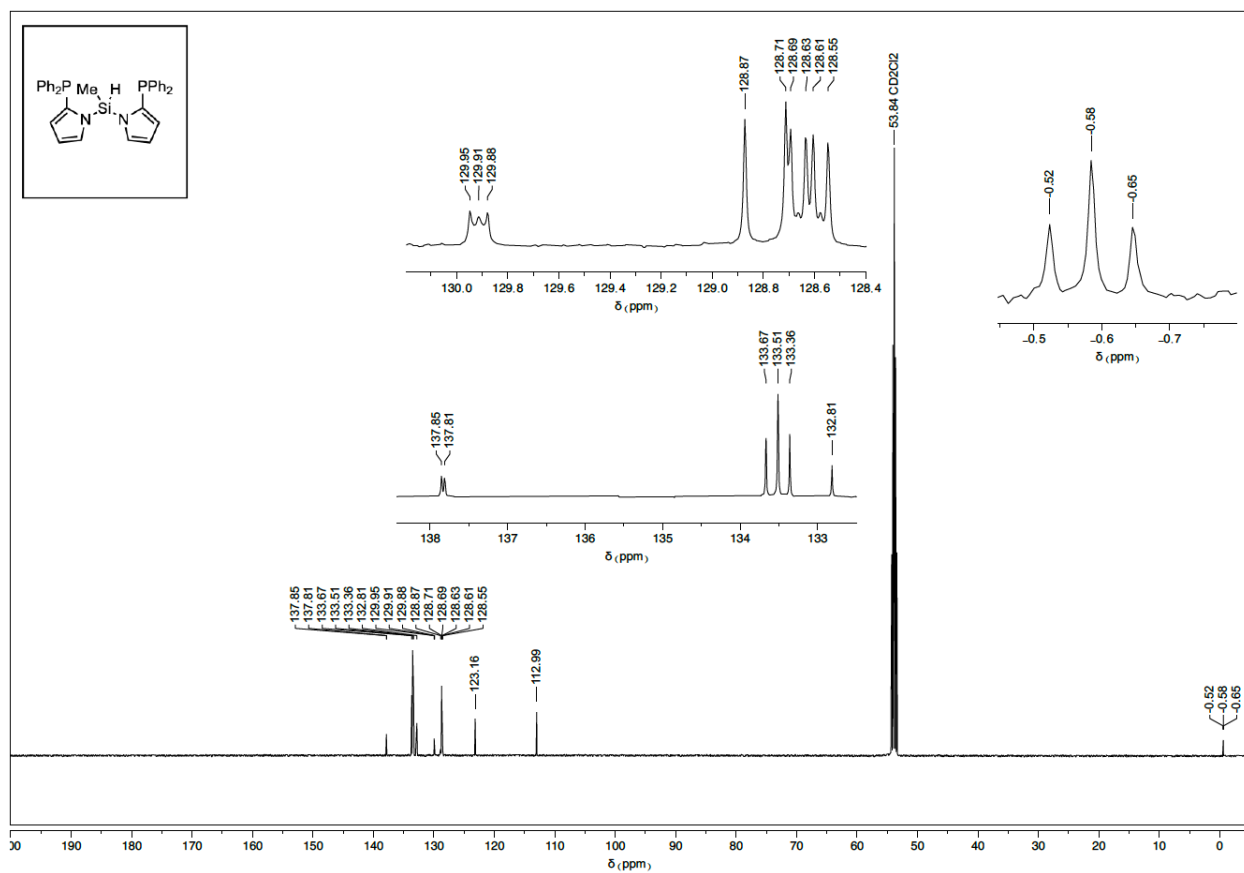




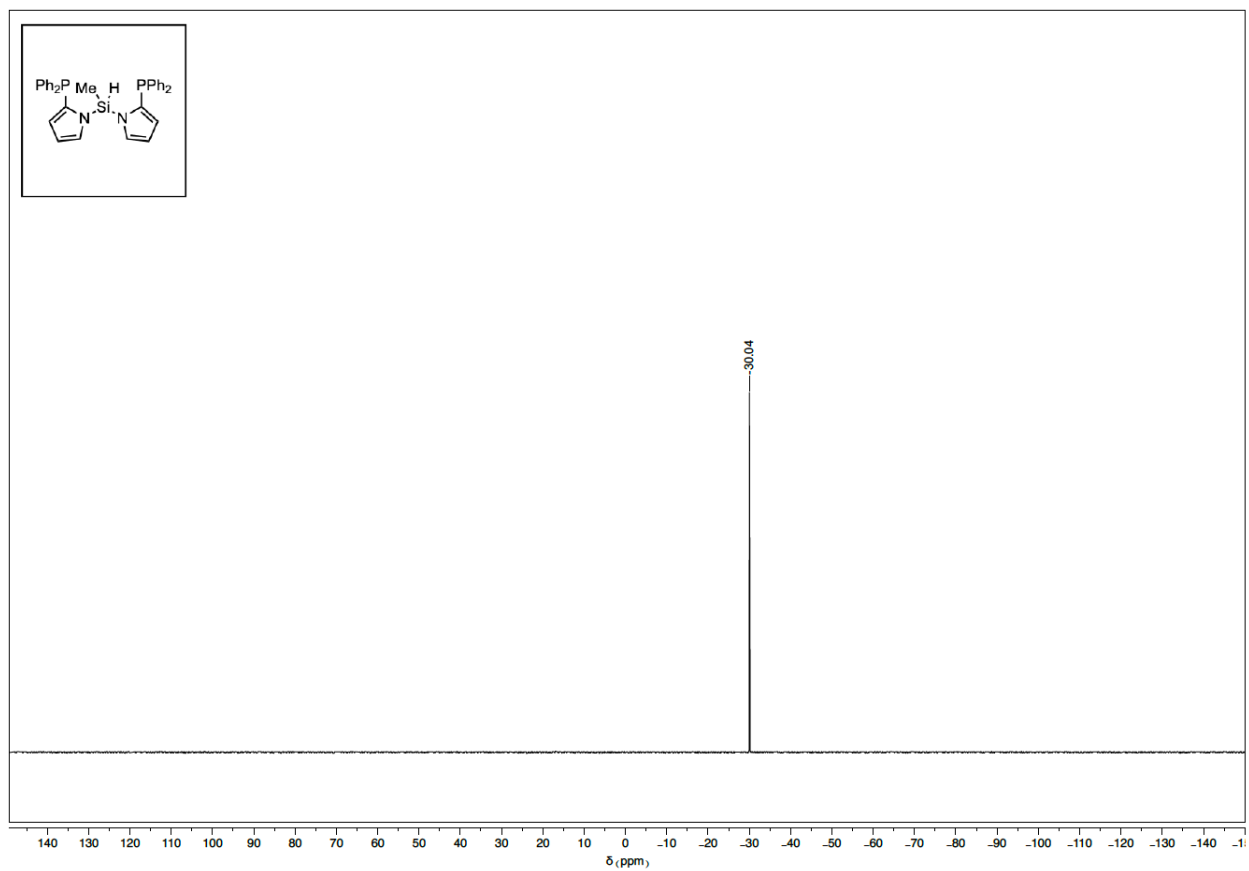
**Figure S12.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of bis(2-diisopropylphosphino-pyrrole)methylsilane (**1**) in  $\text{C}_6\text{D}_6$  (206 MHz).



**Figure S13.** <sup>1</sup>H NMR spectrum of bis(2-diphenylphosphino-pyrrole)methylsilane (**2**) in CD<sub>2</sub>Cl<sub>2</sub> (500 MHz).



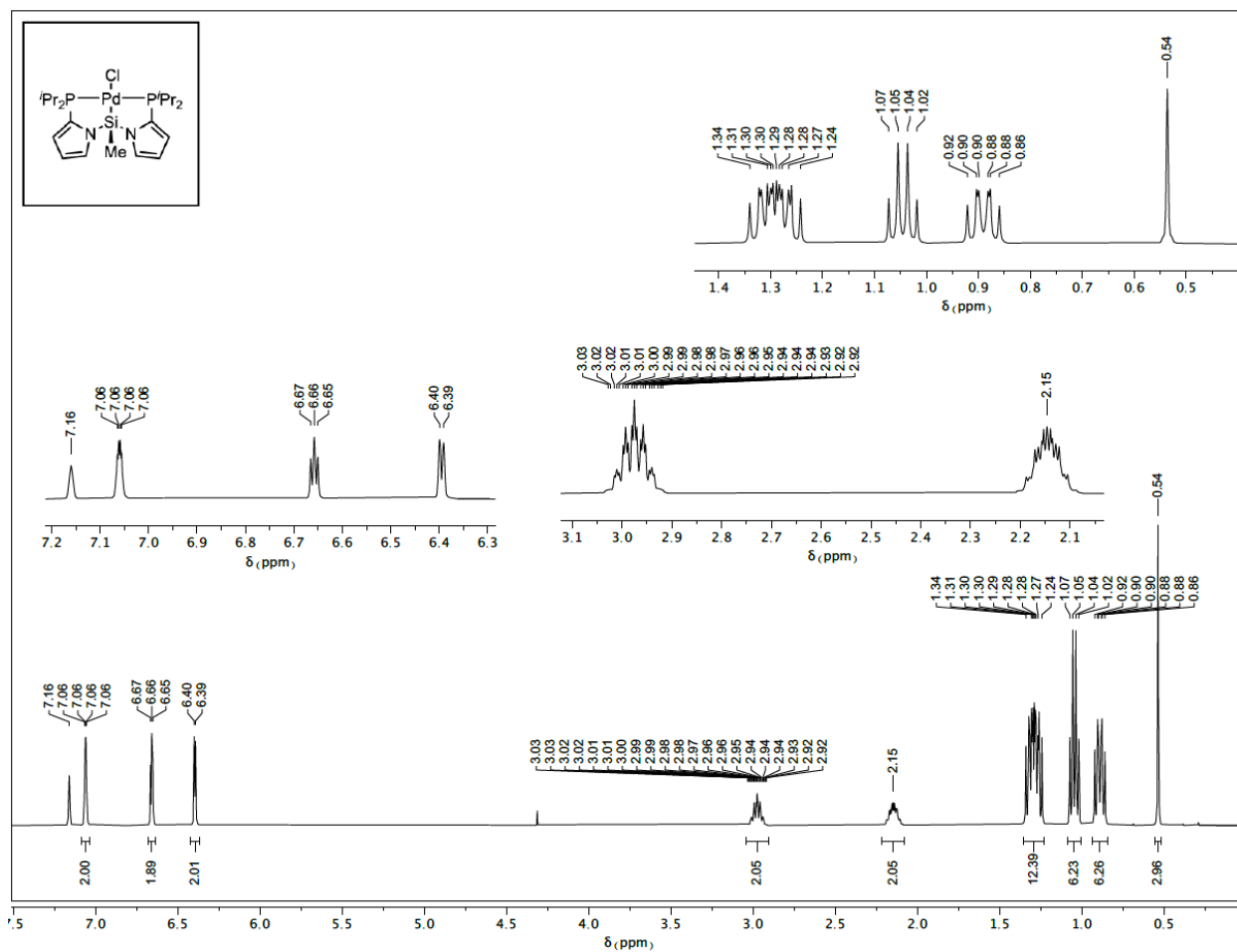
**Figure S14.**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of bis(2-diphenylphosphino-pyrrole)methylsilane (2) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).



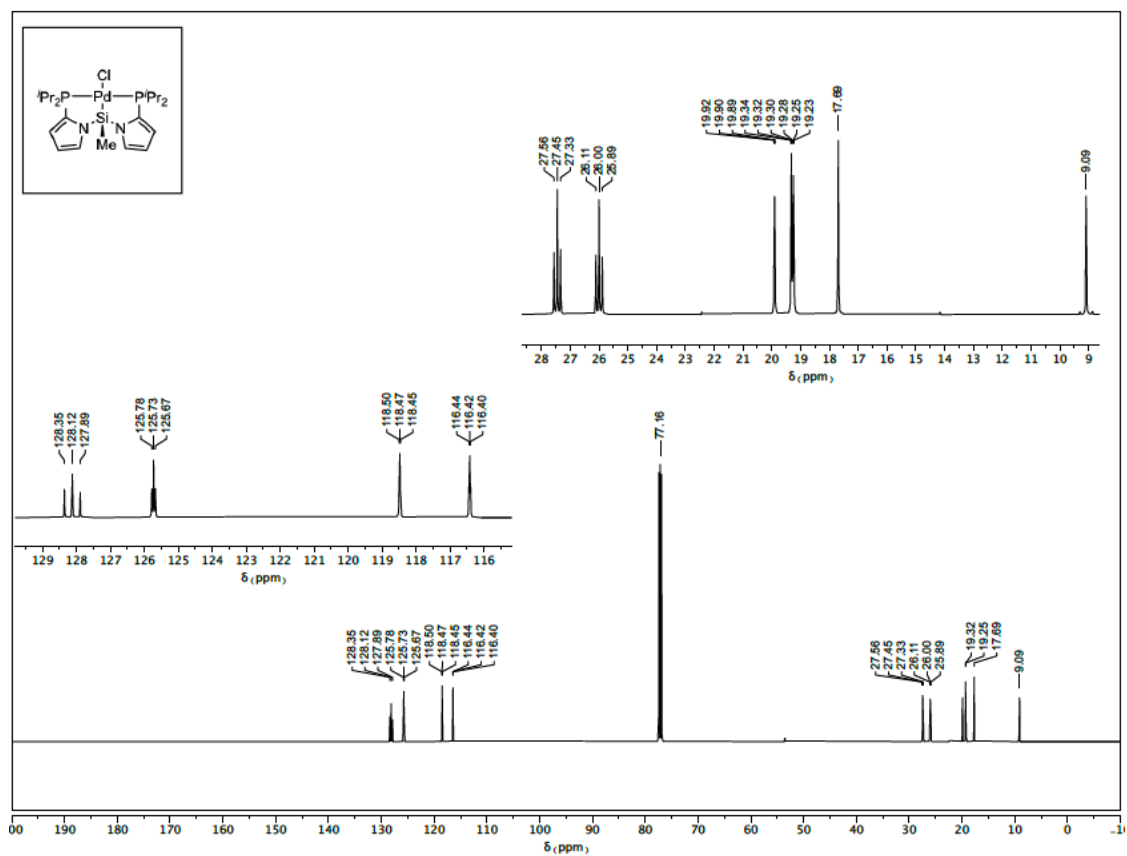
**Figure S15.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of bis(2-diphenylphosphino-pyrrole)methylsilane (**2**) in  $\text{CD}_2\text{Cl}_2$  (202 MHz).



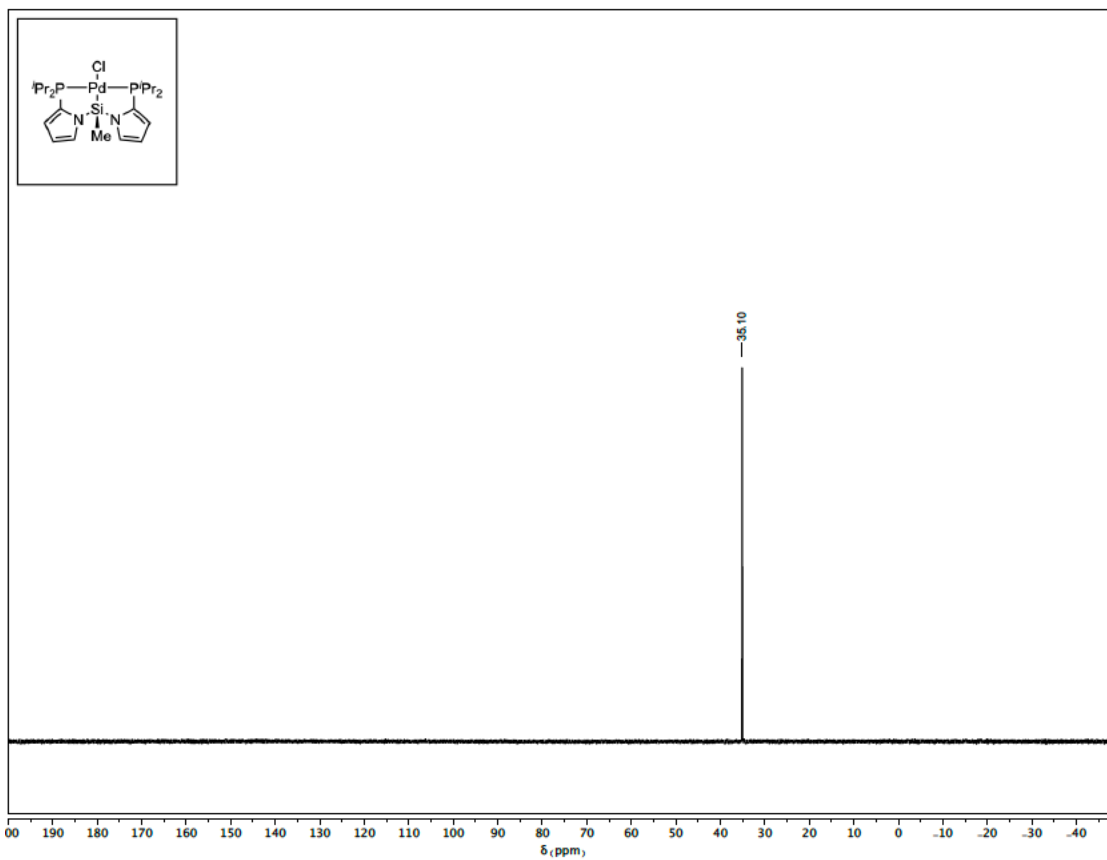
## VI. NMR Spectra of Transition Metal Complexes



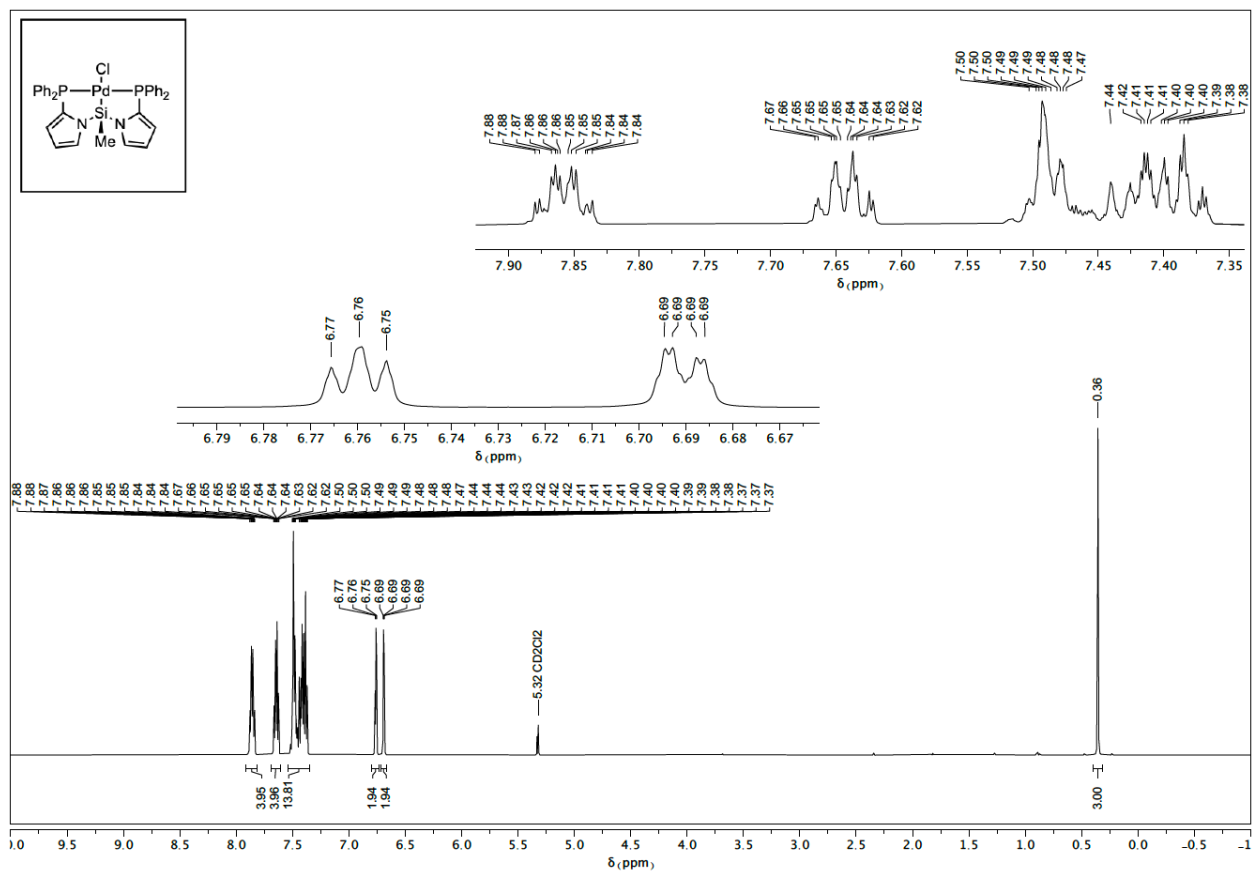
**Figure S16.**  $^1H$  NMR spectrum of  $[(iPrPpy)_2Si]PdCl$  (**3**) in  $C_6D_6$  (400 MHz).



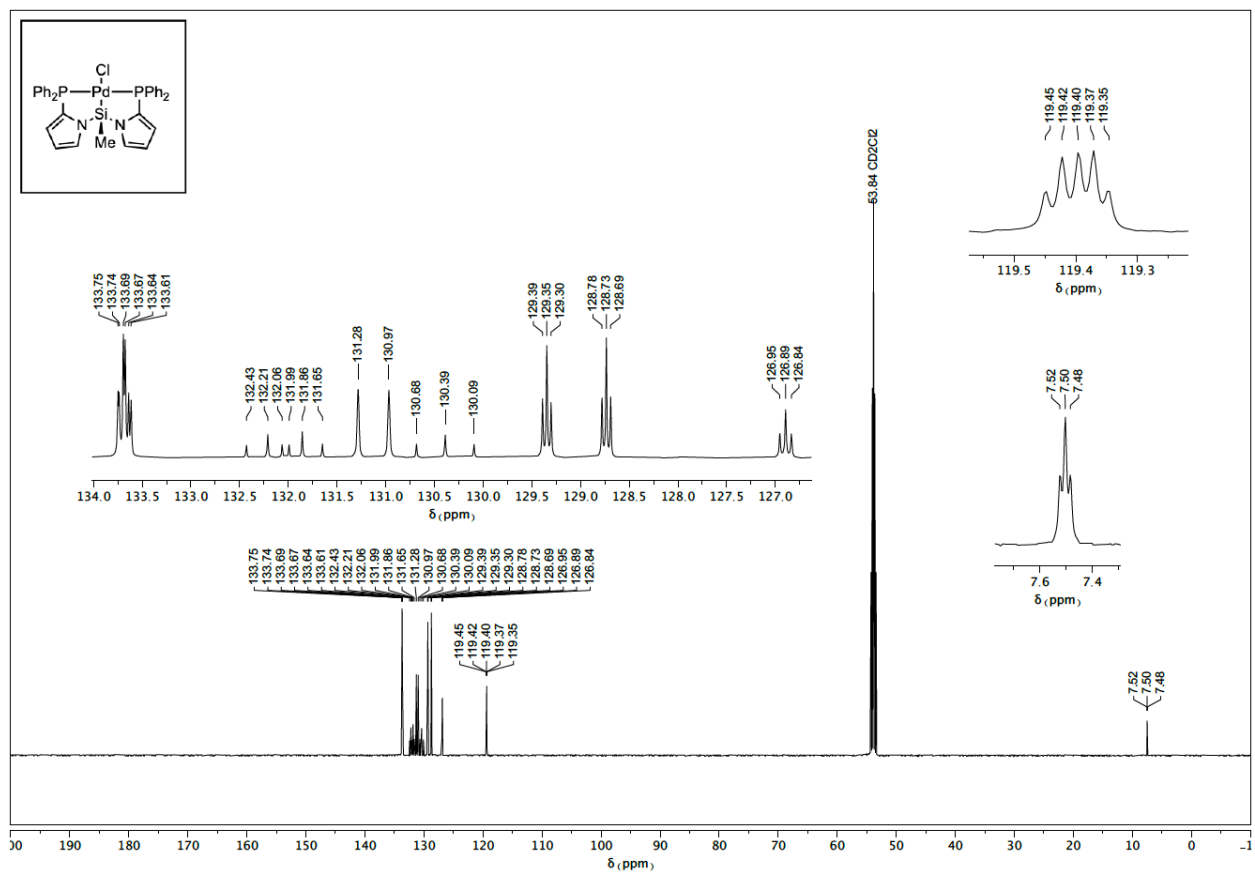
**Figure S17.**  $^{13}C\{^1H\}$  NMR spectrum of  $[(iPrPpy)_2Si]PdCl$  (**3**) in  $C_6D_6$  (126 MHz).



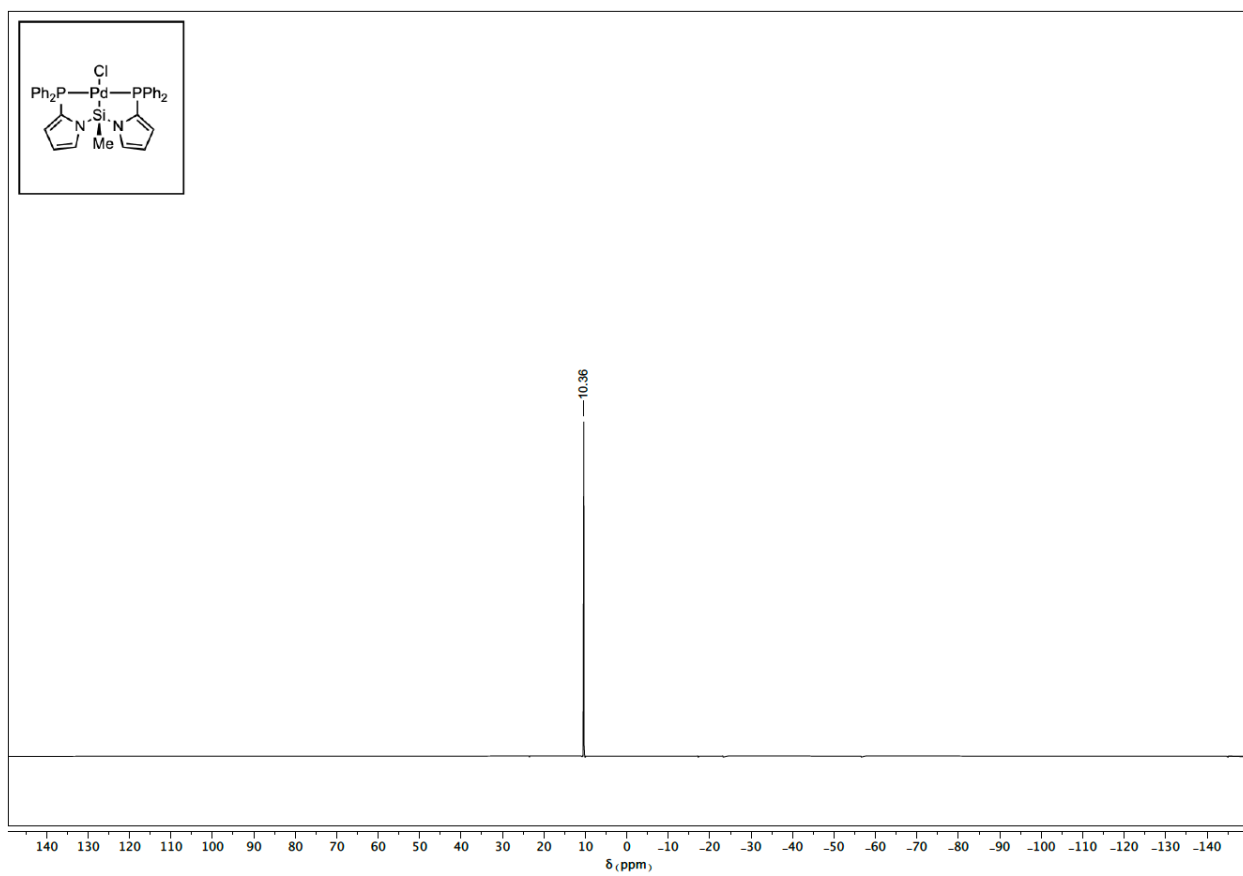
**Figure S18.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $[(i\text{PrPpy})_2\text{Si}]\text{PdCl}$  (**3**) in  $\text{C}_6\text{D}_6$  (202 MHz).



**Figure S19.** <sup>1</sup>H NMR spectrum of [(<sup>Ph</sup>Ppy)<sub>2</sub>Si]PdCl (**4**) in CD<sub>2</sub>Cl<sub>2</sub> (500 MHz).

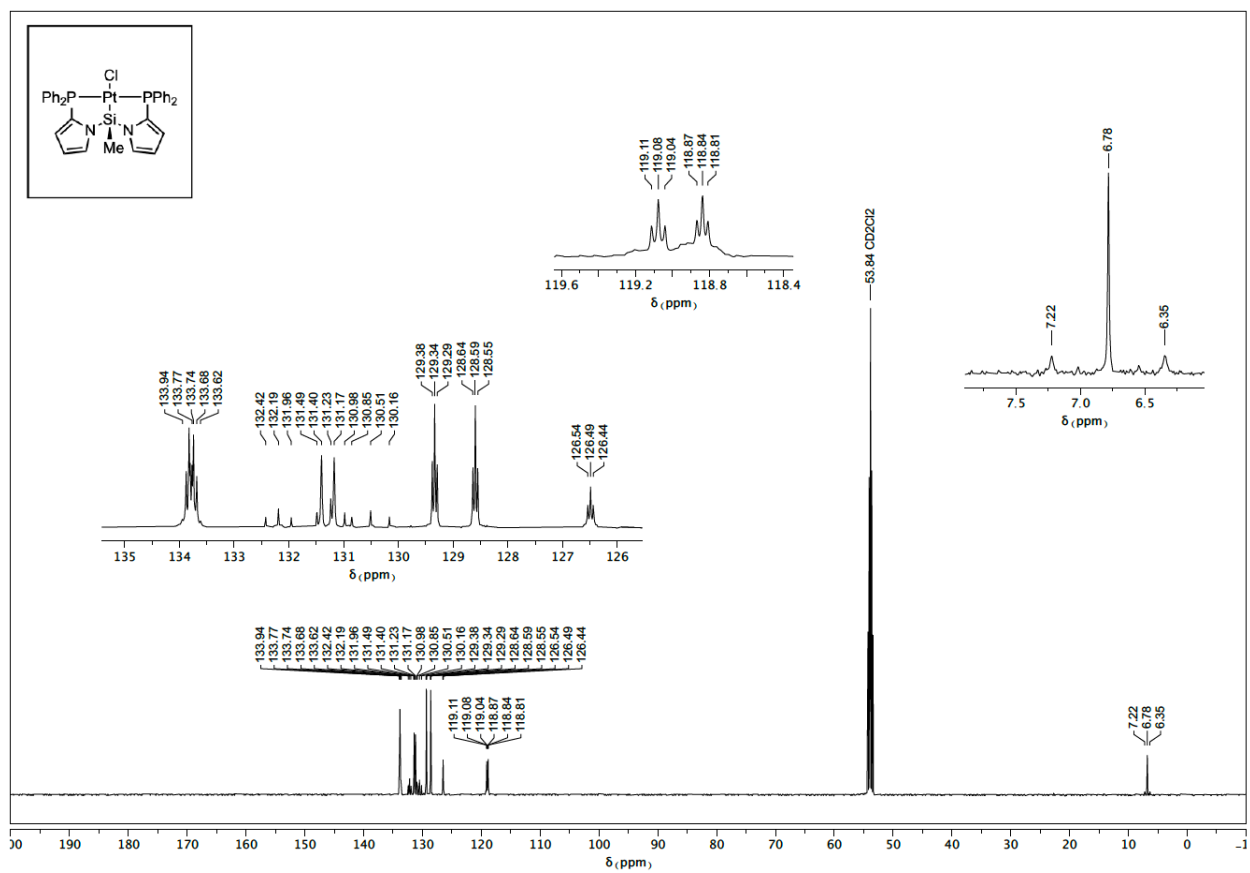


**Figure S20.**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPpy})_2\text{Si}]\text{PdCl}$  (**4**) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).



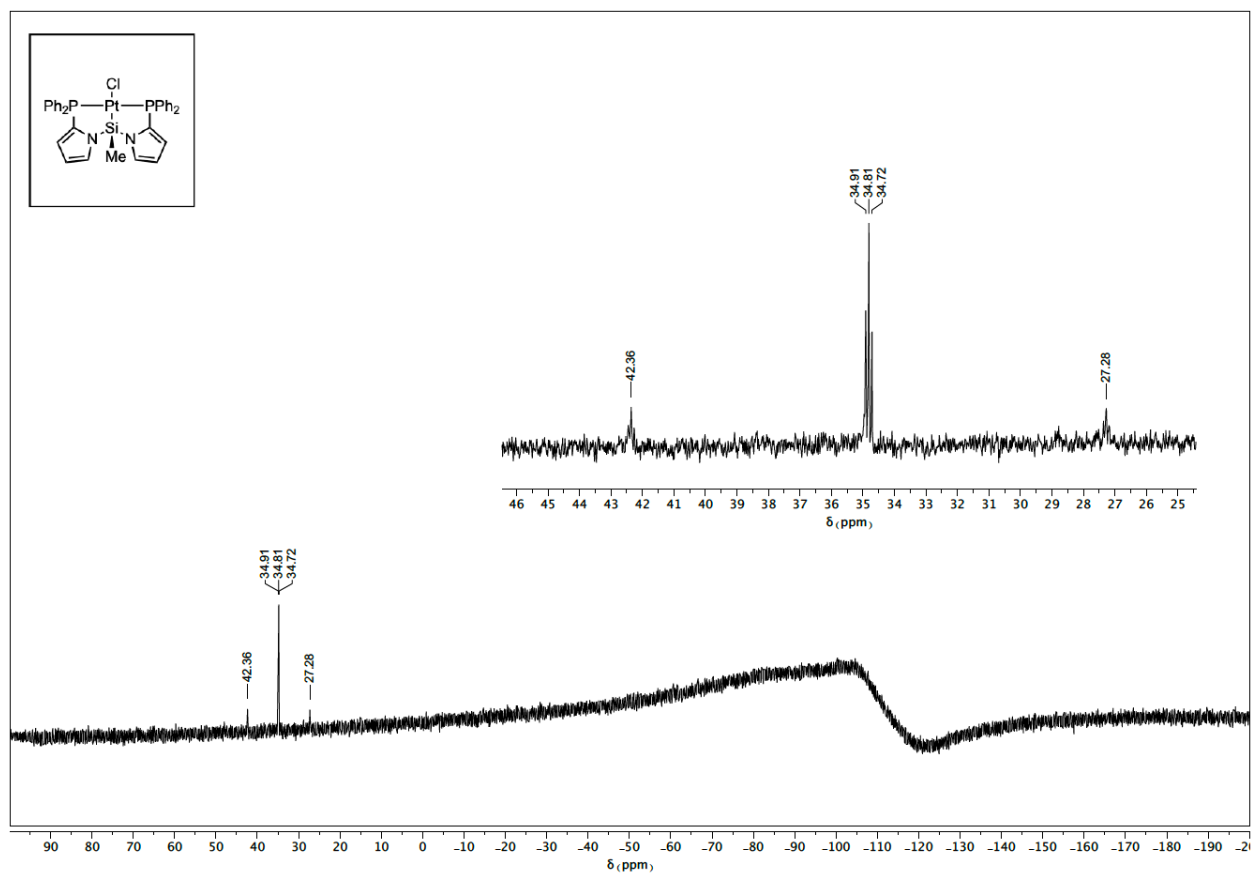
**Figure S21.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPpy})_2\text{Si}]\text{PdCl}$  (**4**) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).



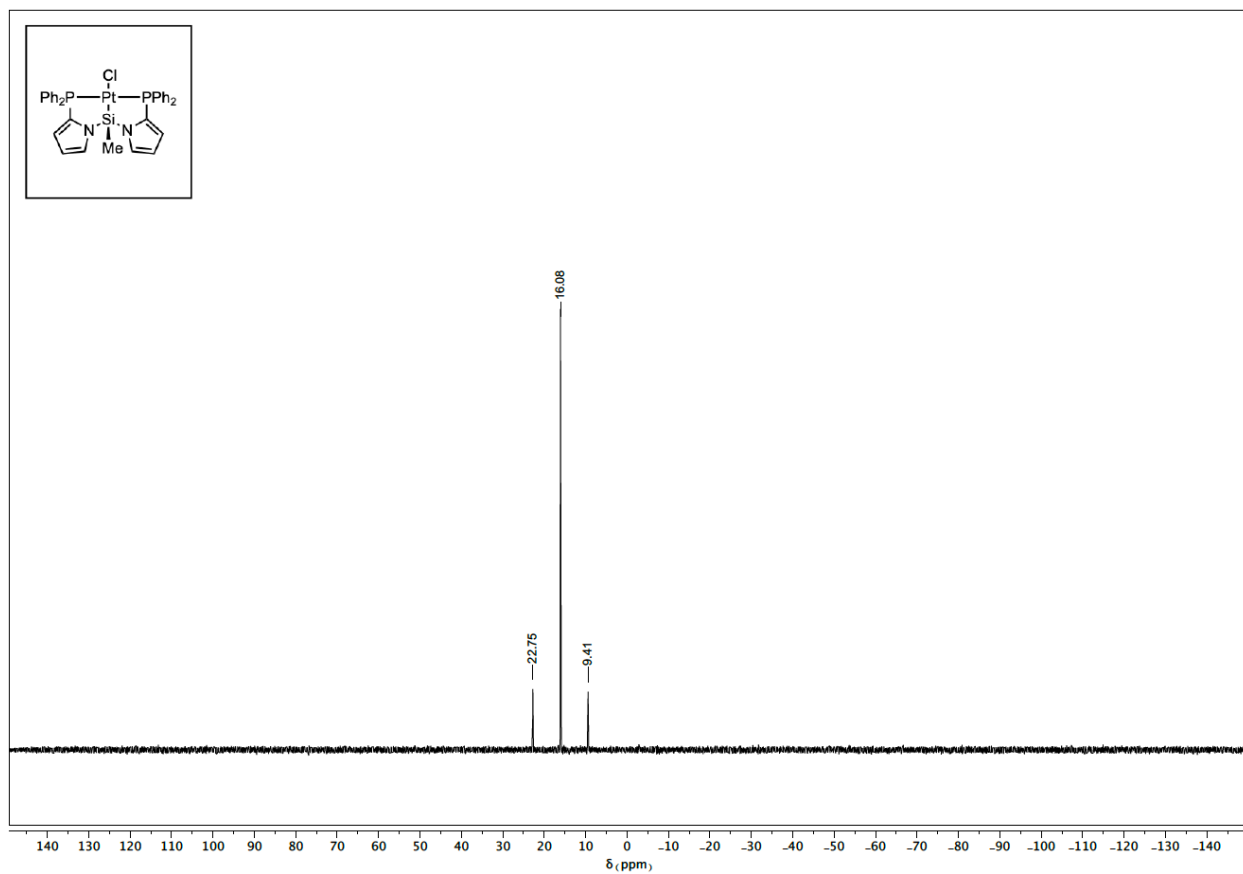


**Figure S23.**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPpy})_2\text{Si}]\text{PtCl}$  (5) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).

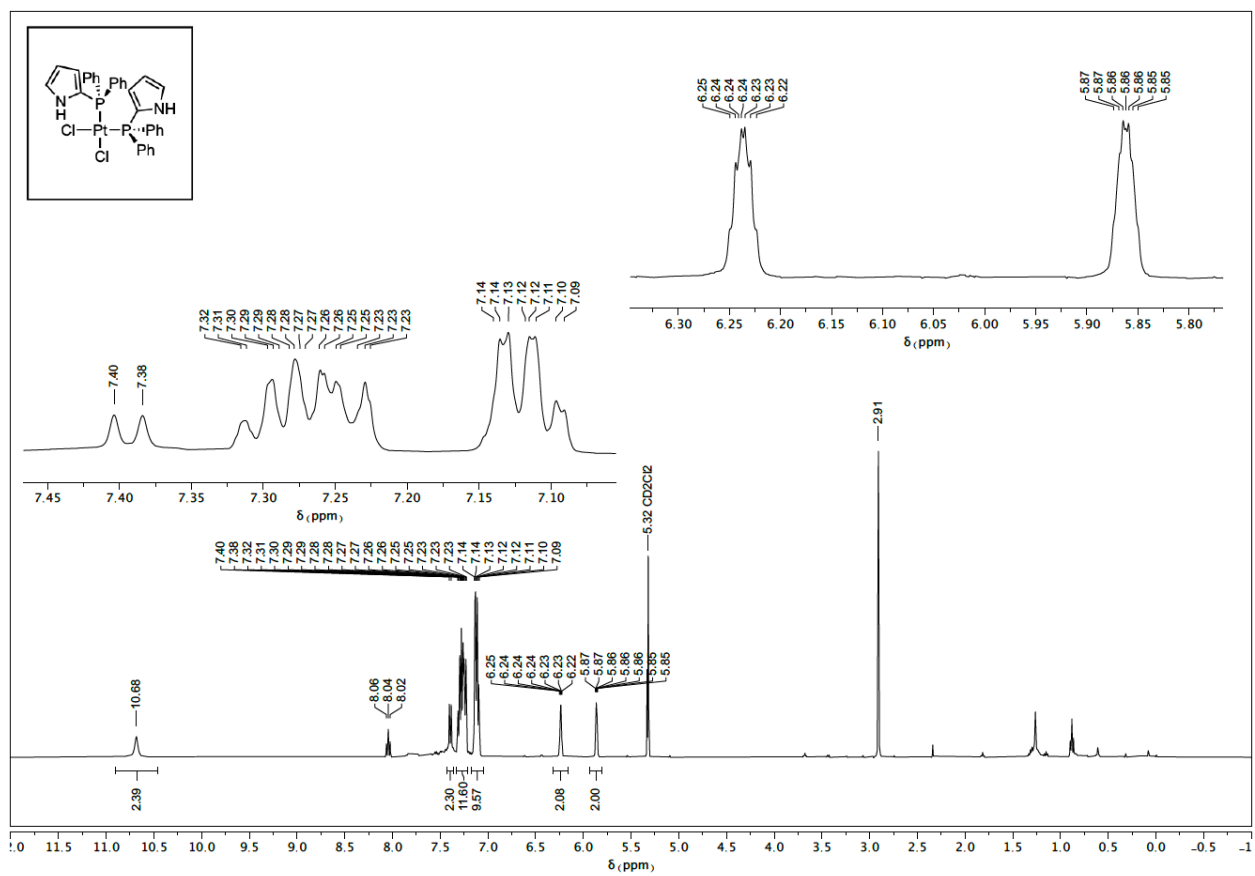




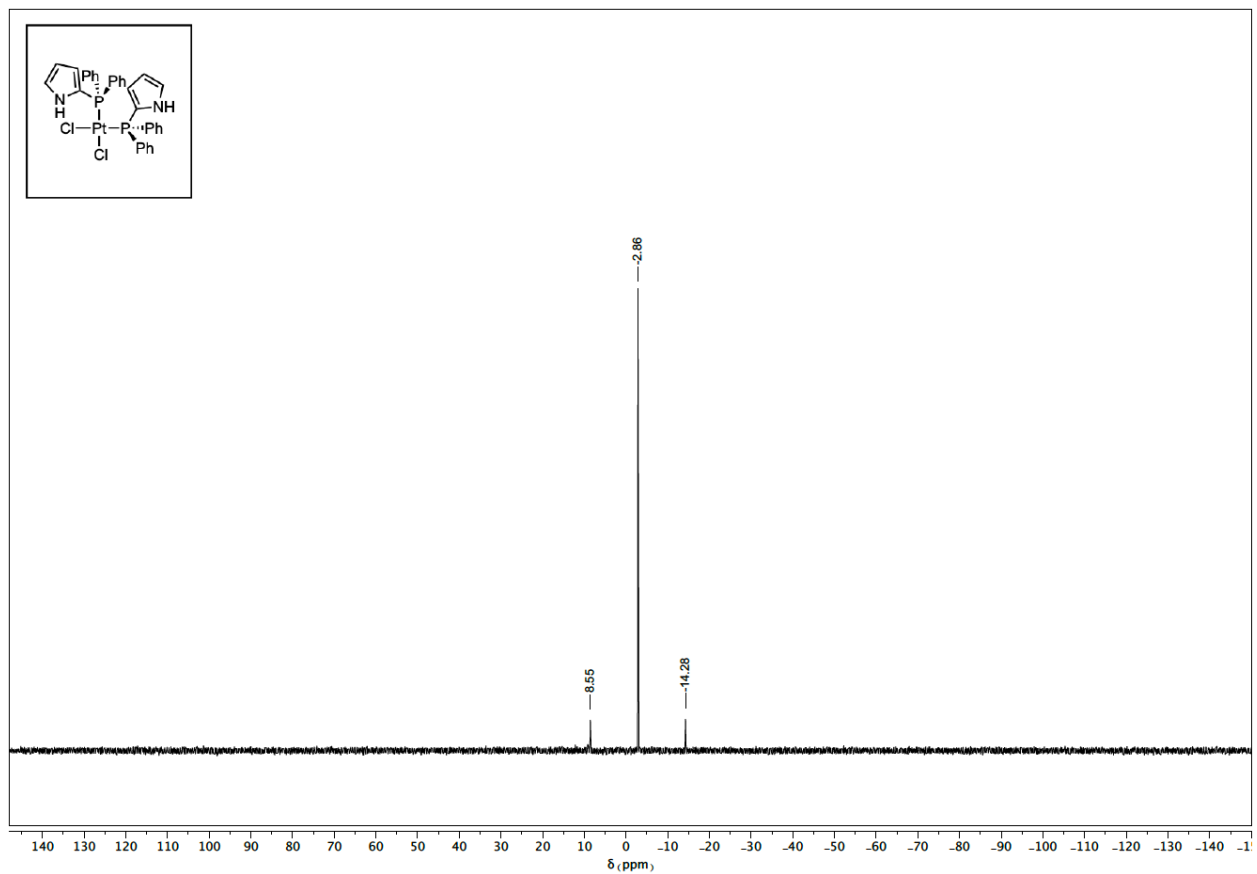
**Figure S24.**  $^{29}\text{Si}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPpy})_2\text{Si}]\text{PtCl}$  (5) in  $\text{CD}_2\text{Cl}_2$  (99 MHz).



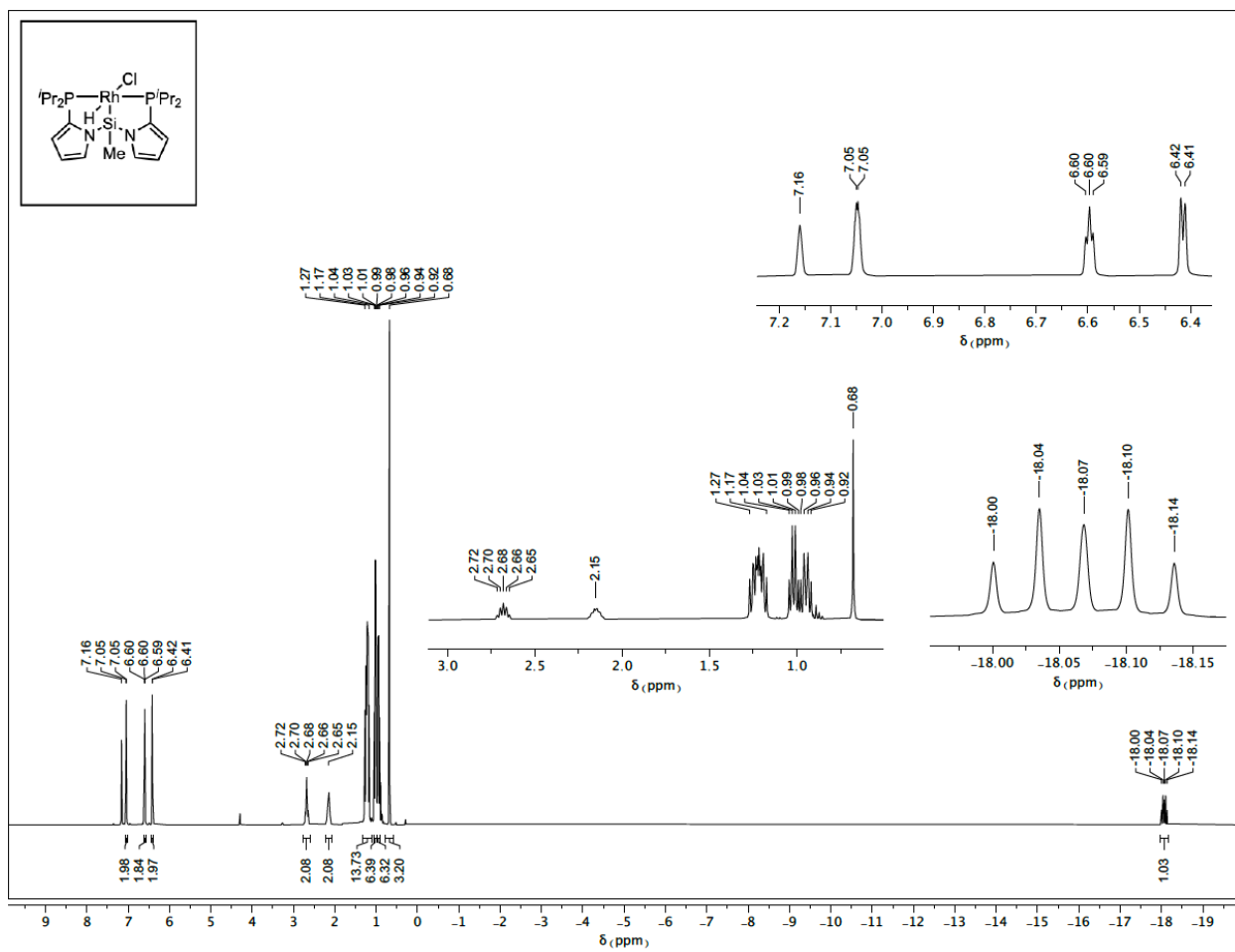
**Figure S25.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $[(^{\text{Ph}}\text{Ppy})_2\text{Si}]\text{PtCl}$  (**5**) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).



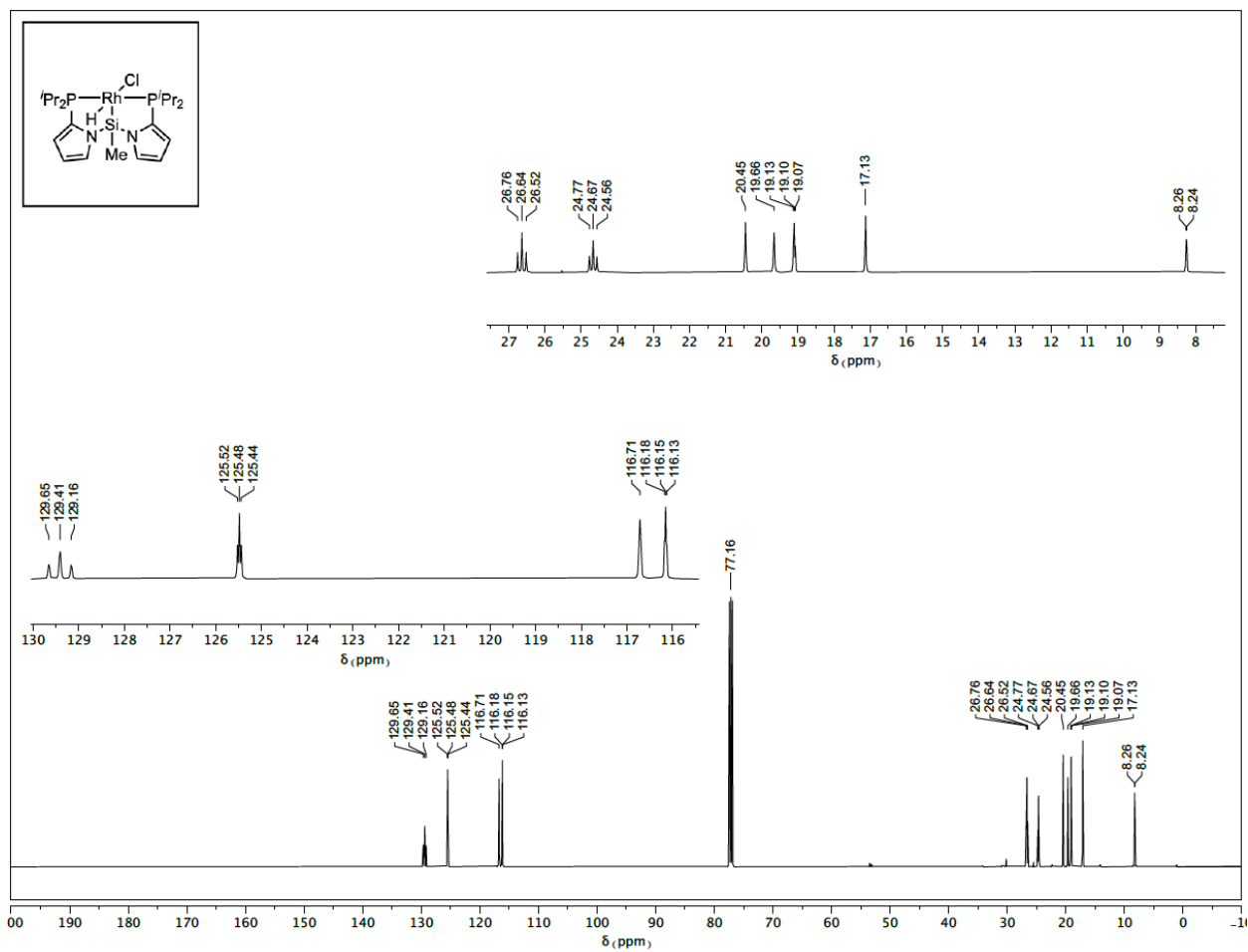
**Figure S26.** Crude  $^1\text{H}$  NMR spectrum of *cis*-bis[(2-diphenylphosphino)pyrrole] platinum dichloride in  $\text{CD}_2\text{Cl}_2$  (400 MHz).



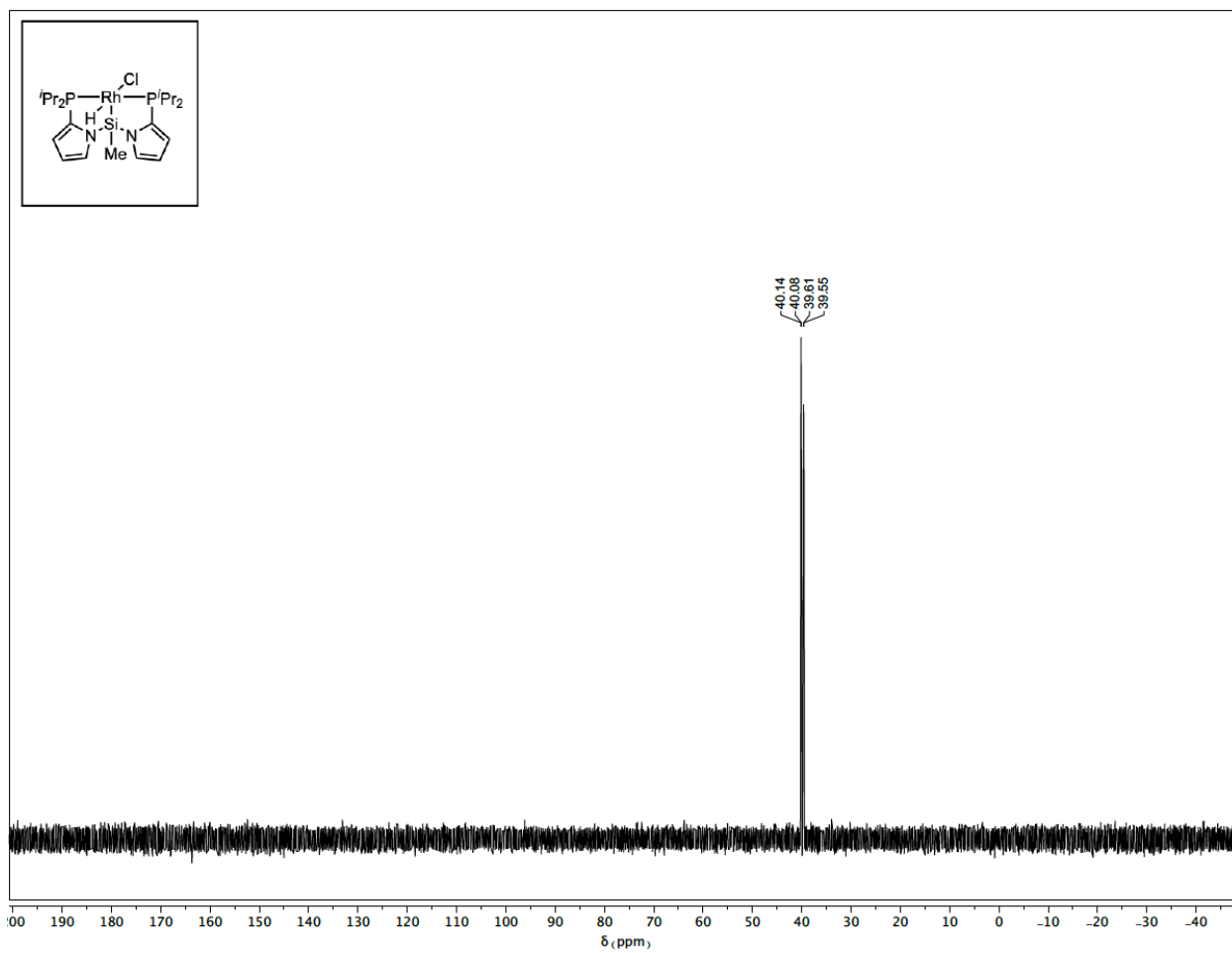
**Figure S27.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of *cis*-bis[(2-diphenylphosphino)pyrrole] platinum dichloride in  $\text{CD}_2\text{Cl}_2$  (162 MHz).



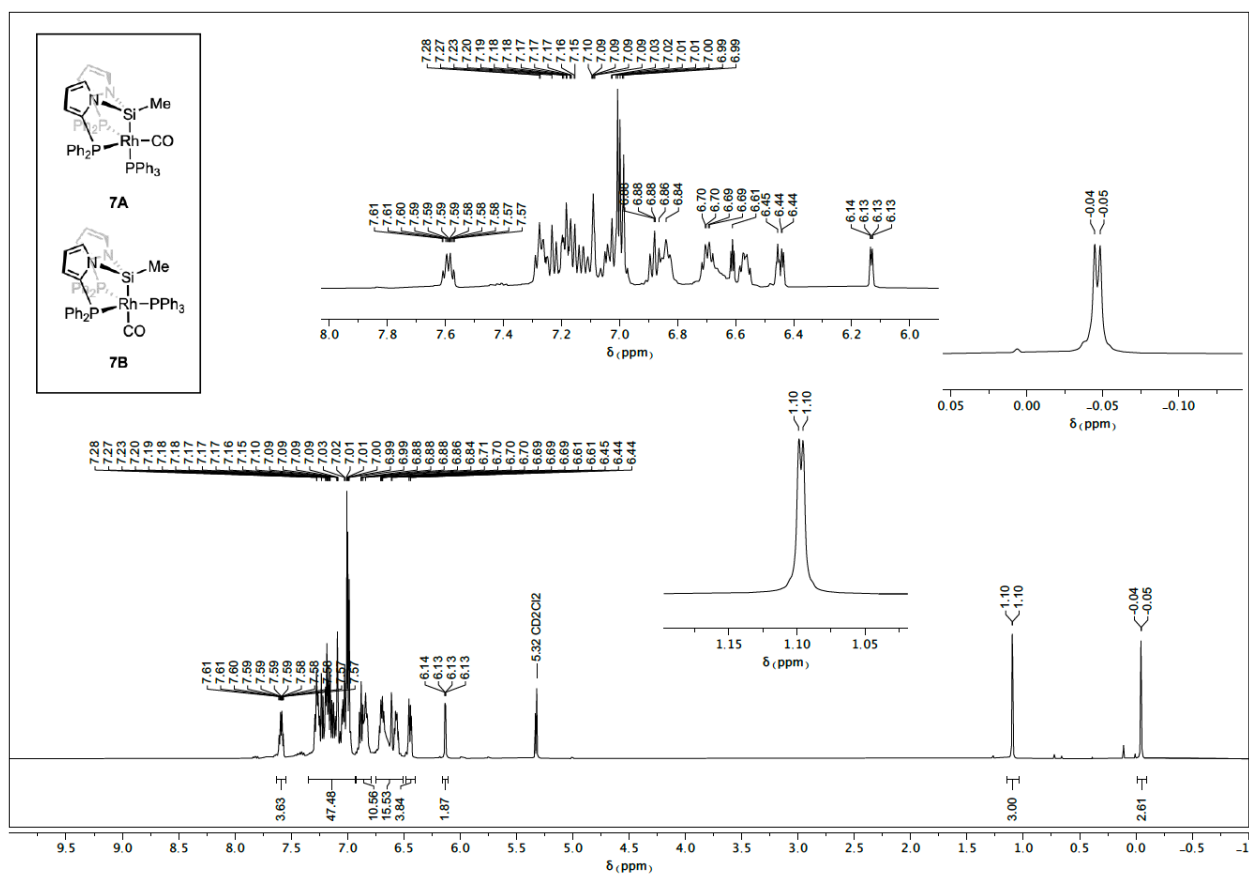
**Figure S28.** <sup>1</sup>H NMR spectrum of [(*i*PrPpy)<sub>2</sub>Si]Rh(H)Cl (**6**) in C<sub>6</sub>D<sub>6</sub> (400 MHz).



**Figure S29.**  $^{13}C\{^1H\}$  NMR spectrum of  $[(iPrPpy)_2Si]Rh(H)Cl$  (**6**) in  $CDCl_3$  (126 MHz).

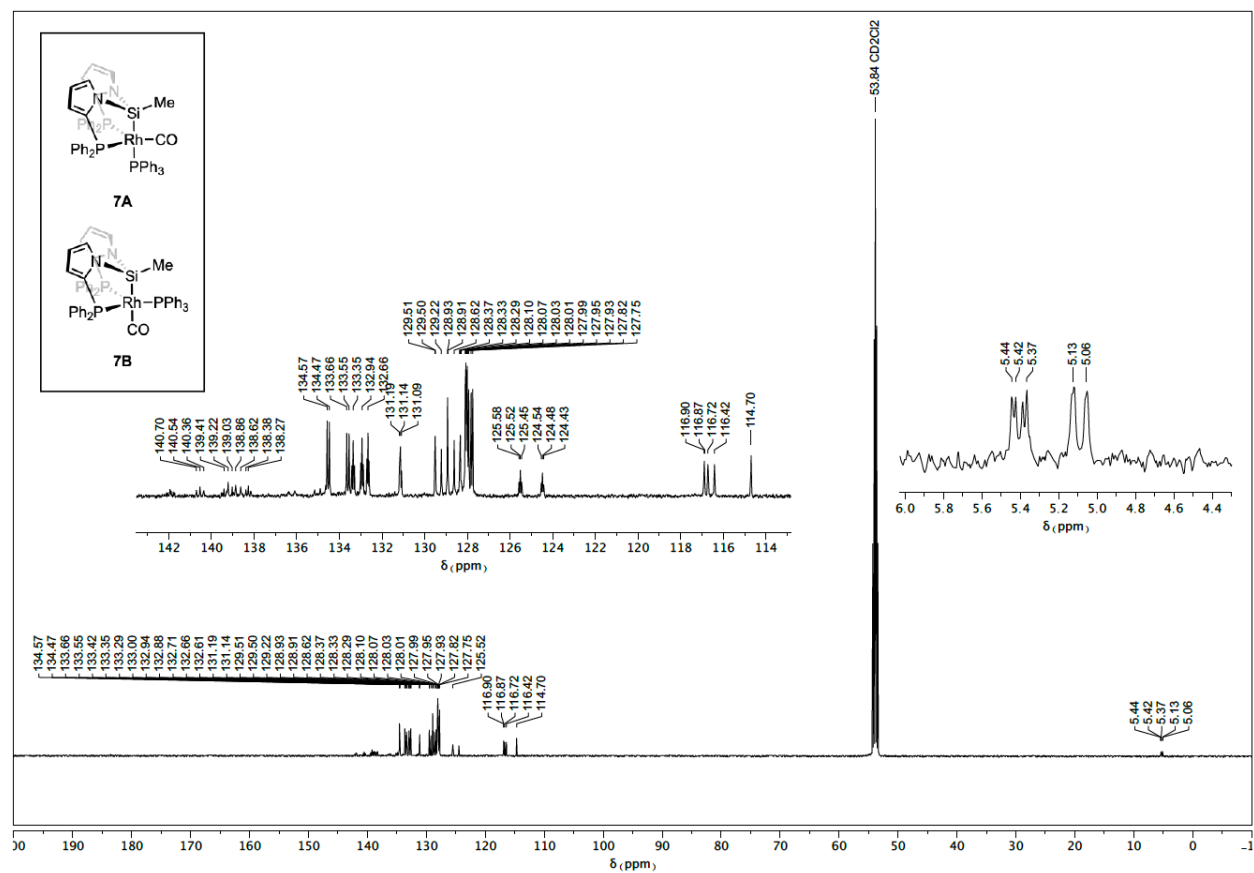


**Figure S30.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $[(i\text{PrPpy})_2\text{Si}]\text{Rh}(\text{H})\text{Cl}$  (**6**) in  $\text{C}_6\text{D}_6$  (206 MHz).

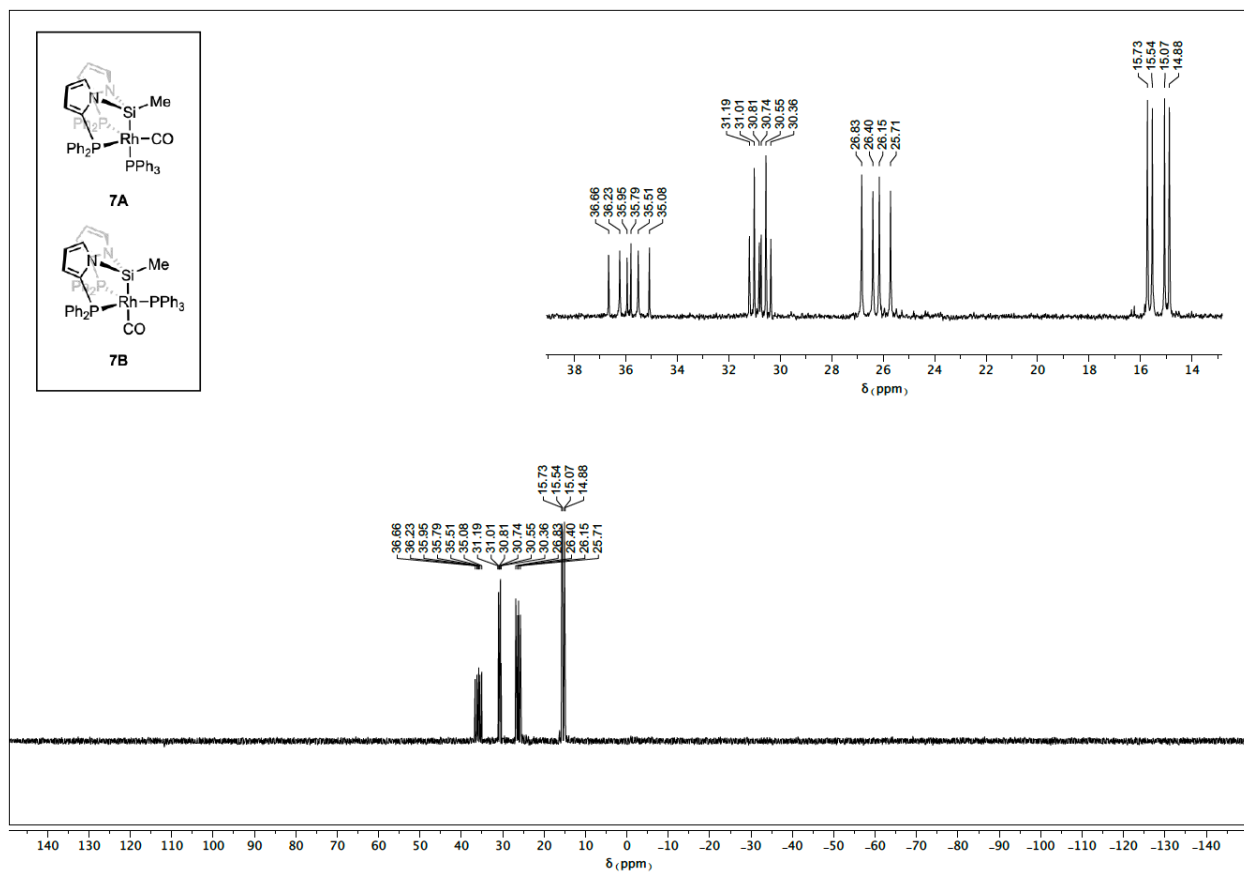


**Figure S31.**  $^1\text{H}$  NMR spectrum of  $[(^{\text{Ph}}\text{Ppy})_2\text{Si}]\text{Rh}(\text{CO})\text{PPh}_3$  (**7A/7B**) in  $\text{CD}_2\text{Cl}_2$  (500 MHz).





**Figure S32.**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPy})_2\text{Si}]\text{Rh}(\text{CO})\text{PPh}_3$  (**7A/7B**) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).



**Figure S33.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $[(\text{PhPpy})_2\text{Si}]\text{Rh}(\text{CO})\text{PPh}_3$  (**7A/7B**) in  $\text{CD}_2\text{Cl}_2$  (126 MHz).

## VII. X-Ray Crystallographic Data

### **X-Ray data collection, solution, and refinement for [(PhP<sub>Py</sub>)<sub>2</sub>Si]PdCl (4; CCDC 2143700).**

A Leica MZ 75 microscope was used to identify a suitable colorless block with very well defined faces with dimensions (max, intermediate, and min) 0.478 x 0.452 x 0.207 mm<sup>3</sup> from a representative sample of crystals of the same habit. The crystal mounted on a nylon loop was then placed in a cold nitrogen stream (Oxford) maintained at 110 K. A BRUKER Quest X-ray (fixed-Chi geometry) diffractometer with a PHOTON III detector was employed for crystal screening, unit cell determination, and data collection. The goniometer was controlled using the APEX3 software suite.<sup>6</sup> The sample was optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions. The X-ray radiation employed was generated from a Mo-I $\mu$ s X-ray tube ( $K_{\alpha} = 0.71073\text{\AA}$ ). 45 data frames were taken at widths of 1°. These reflections were used to determine the unit cell. The unit cell was verified by examination of the *h k l* overlays on several frames of data. No super-cell or erroneous reflections were observed. After careful examination of the unit cell, an extended data collection procedure (8 sets) was initiated using omega and phi scans. Integrated intensity information for each reflection was obtained by reduction of the data frames with the program APEX3. The integration method employed a three dimensional profiling algorithm and all data were corrected for Lorentz and polarization factors, as well as for crystal decay effects. Finally the data was merged and scaled to produce a suitable data set. The absorption correction program SADABS<sup>7</sup> was employed to correct the data for absorption effects. Systematic reflection conditions and statistical tests of the data suggested the space group *P2<sub>1</sub>/c*. A solution was obtained readily using XT/XS in APEX3.<sup>6,8</sup> Hydrogen atoms were placed in idealized positions and were set riding on the respective parent atoms. All non-hydrogen atoms were refined with anisotropic thermal parameters. Absence of additional symmetry and voids were confirmed using PLATON (ADDSYM).<sup>9</sup> The structure was refined (weighted least squares refinement on  $F^2$ ) to convergence.<sup>8,10</sup> Olex2 was employed for the final data presentation and structure plots.<sup>10</sup>

### **X-Ray data collection, solution, and refinement for [(PhP<sub>Py</sub>)<sub>2</sub>Si]PtCl (5; CCDC 2143692).**

A Leica MZ 75 microscope was used to identify a suitable colorless block with very well defined faces with dimensions (max, intermediate, and min) 0.402 x 0.381 x 0.217 mm<sup>3</sup> from a representative sample of crystals of the same habit. The crystal mounted on a nylon loop was then placed in a cold nitrogen stream (Oxford) maintained at 110 K. A BRUKER Quest X-ray (fixed-Chi geometry) diffractometer with a PHOTON III detector was employed for crystal screening, unit cell determination, and data collection. The goniometer was controlled using the APEX3 software suite.<sup>6</sup> The sample was optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions. The X-ray radiation employed was generated from a Mo-I $\mu$ s X-ray tube ( $K_{\alpha} = 0.71073\text{\AA}$ ). 45 data frames were taken at widths of 1°. These reflections were used to determine the unit cell. The unit cell was verified by examination of the *h k l* overlays on several frames of data. No super-cell or erroneous

reflections were observed. After careful examination of the unit cell, an extended data collection procedure (8 sets) was initiated using omega and phi scans. Integrated intensity information for each reflection was obtained by reduction of the data frames with the program APEX3. The integration method employed a three dimensional profiling algorithm and all data were corrected for Lorentz and polarization factors, as well as for crystal decay effects. Finally the data was merged and scaled to produce a suitable data set. The absorption correction program SADABS<sup>7</sup> was employed to correct the data for absorption effects. Systematic reflection conditions and statistical tests of the data suggested the space group  $P2_1/c$ . A solution was obtained readily using XT/XS in APEX3.<sup>6,8</sup> Hydrogen atoms were placed in idealized positions and were set riding on the respective parent atoms. All non-hydrogen atoms were refined with anisotropic thermal parameters. Absence of additional symmetry and voids were confirmed using PLATON (ADDSYM).<sup>9</sup> The structure was refined (weighted least squares refinement on  $F^2$ ) to convergence.<sup>8,10</sup> Olex2 was employed for the final data presentation and structure plots.<sup>10</sup>

### **X-Ray data collection, solution, and refinement for *cis*-bis[(2-diphenylphosphino)pyrrole] platinum dichloride (CCDC 2143693).**

A Leica MZ 75 microscope was used to identify a suitable colorless block with very well defined faces with dimensions (max, intermediate, and min) 0.207 x 0.124 x 0.081 mm<sup>3</sup> from a representative sample of crystals of the same habit. The crystal mounted on a nylon loop was then placed in a cold nitrogen stream (Oxford) maintained at 110 K. A BRUKER Quest X-ray (fixed-Chi geometry) diffractometer with a PHOTON III detector was employed for crystal screening, unit cell determination, and data collection. The goniometer was controlled using the APEX3 software suite.<sup>6</sup> The sample was optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions. The X-ray radiation employed was generated from a Mo-I $\mu$ s X-ray tube ( $K_{\alpha} = 0.71073\text{\AA}$ ). 45 data frames were taken at widths of 1°. These reflections were used to determine the unit cell. The unit cell was verified by examination of the  $h k l$  overlays on several frames of data. No super-cell or erroneous reflections were observed. After careful examination of the unit cell, an extended data collection procedure (5 sets) was initiated using omega and phi scans. Integrated intensity information for each reflection was obtained by reduction of the data frames with the program APEX3. The integration method employed a three dimensional profiling algorithm and all data were corrected for Lorentz and polarization factors, as well as for crystal decay effects. Finally the data was merged and scaled to produce a suitable data set. The absorption correction program SADABS<sup>7</sup> was employed to correct the data for absorption effects. Systematic reflection conditions and statistical tests of the data suggested the space group  $P-1$ . A solution was obtained readily using XT/XS in APEX3.<sup>6,8</sup> Hydrogen atoms were placed in idealized positions and were set riding on the respective parent atoms (except hydrogen atoms bonded to N1 and N2). All non-hydrogen atoms were refined with anisotropic thermal parameters. Absence of additional symmetry and voids were confirmed using PLATON (ADDSYM).<sup>9</sup> The structure was refined (weighted least squares refinement on  $F^2$ ) to convergence.<sup>8,10</sup> Olex2 was employed for the final data presentation and structure plots.<sup>10</sup>

**X-Ray data collection, solution, and refinement for  $[(\text{PhP}_{\text{Py}})_2\text{Si}]\text{Rh}(\text{CO})\text{PPh}_3$  (7; CCDC 2143691).**

A Leica MZ 75 microscope was used to identify a suitable yellow block with very well defined faces with dimensions (max, intermediate, and min)  $0.421 \times 0.387 \times 0.204 \text{ mm}^3$  from a representative sample of crystals of the same habit. The crystal mounted on a nylon loop was then placed in a cold nitrogen stream (Oxford) maintained at 110 K. A BRUKER Quest X-ray (fixed-Chi geometry) diffractometer with a PHOTON III detector was employed for crystal screening, unit cell determination, and data collection. The goniometer was controlled using the APEX3 software suite.<sup>6</sup> The sample was optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions. The X-ray radiation employed was generated from a Mo- $\text{I}\mu\text{s}$  X-ray tube ( $K_{\alpha} = 0.71073 \text{ \AA}$ ). 45 data frames were taken at widths of  $1^{\circ}$ . These reflections were used to determine the unit cell. The unit cell was verified by examination of the  $h k l$  overlays on several frames of data. No super-cell or erroneous reflections were observed. After careful examination of the unit cell, an extended data collection procedure (10 sets) was initiated using omega and phi scans. Integrated intensity information for each reflection was obtained by reduction of the data frames with the program APEX3. The integration method employed a three dimensional profiling algorithm and all data were corrected for Lorentz and polarization factors, as well as for crystal decay effects. Finally the data was merged and scaled to produce a suitable data set. The absorption correction program SADABS<sup>7</sup> was employed to correct the data for absorption effects. Systematic reflection conditions and statistical tests of the data suggested the space group  $P-1$ . A solution was obtained readily using XT/XS in APEX3.<sup>6,8</sup> Hydrogen atoms were placed in idealized positions and were set riding on the respective parent atoms. All non-hydrogen atoms were refined with anisotropic thermal parameters. Absence of additional symmetry and voids were confirmed using PLATON (ADDSYM).<sup>9</sup> The structure was refined (weighted least squares refinement on  $F^2$ ) to convergence.<sup>8,10</sup> Olex2 was employed for the final data presentation and structure plots.<sup>10</sup>

## VIII. References

- 1 L. Falivene, Z. Cao, A. Petta, L. Serra, A. Poater, R. Oliva, V. Scarano and L. Cavallo, *Nat. Chem.*, 2019, **11**, 872–879.
- 2 J. Takaya, S. Nakamura and N. Iwasawa, *Chem. Lett.*, 2012, **41**, 967–969.
- 3 H. Kameo, S. Ishii and H. Nakazawa, *Dalton Trans.*, 2013, **42**, 4663–4669.
- 4 H. Kameo, S. Ishii and H. Nakazawa, *Dalton Trans.*, 2012, **41**, 11386–11392.
- 5 H. Kameo, T. Kawamoto, S. Sakaki, D. Bourissou and H. Nakazawa, *Chem. – Eur. J.*, 2016, **22**, 2370–2375.
- 6 APEX3 “Program for Data Collection on Area Detectors” BRUKER AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711-5373 USA
- 7 SADABS, G. M. Sheldrick. “Program for Absorption Correction of Area Detector Frames”, BRUKER AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711-5373 USA.
- 8 (a) G. M. Sheldrick, *Acta Cryst.* 2008, **A64**, 112–122. (b) G. M. Sheldrick, *Acta Cryst.* 2015, **A71**, 3–8. (c) G. M. Sheldrick, *Acta Cryst.* 2015, **C71**, 3–8. (d) XT, XS, BRUKER AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711-5373 USA.
- 9 A. L. Spek, *J. Appl. Cryst.* 2003, **36**, 7–13.
- 10 O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Crystallogr.*, 2009, **42**, 339–341.