Electric Supplementary Information

Preparation and Catalytic Performance of a Molecularly Imprinted Pd Complex Catalyst for Suzuki Cross-coupling Reactions

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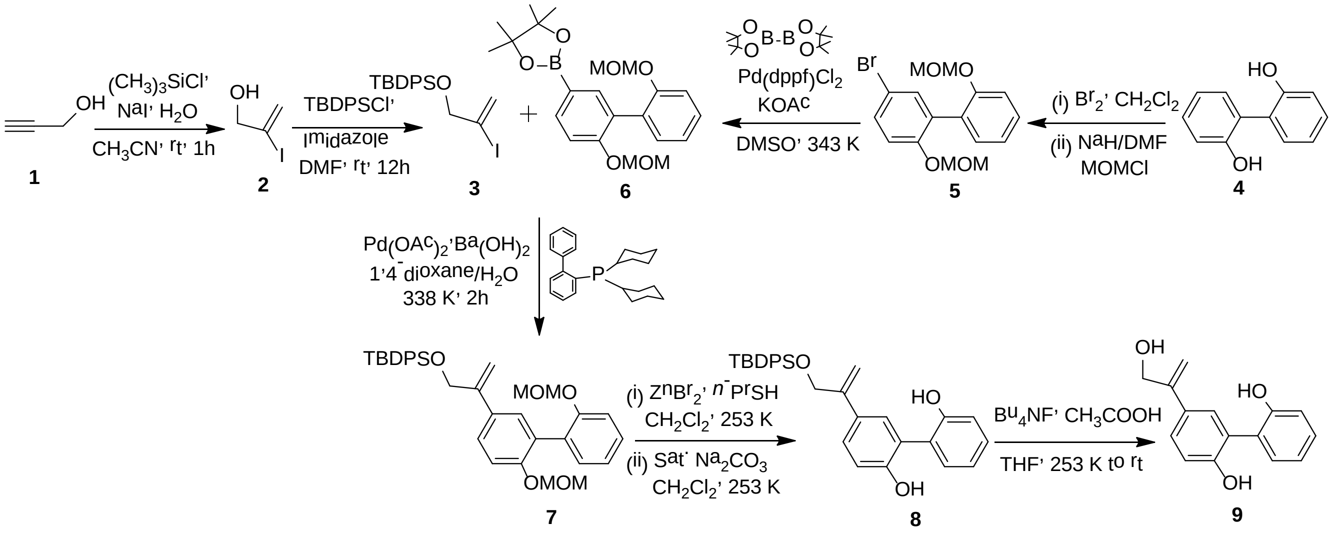
Contents

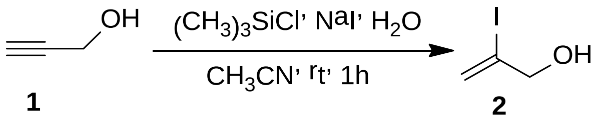
Preparation of template and (idppe)PdCl$_2$

Chemicals were purchased from commercial sources and used without further purification, unless otherwise indicated. All preparations and manipulations were performed using glove box and standard Schlenk techniques under Ar or N$_2$. The isopropenyl phosphene ligand (H$_2$C=C(CH$_3$)C$_6$H$_4$)$_2$-PCH$_2$CH$_2$P-(C$_6$H$_4$(CH$_3$)C=CH$_2$)$_2$ (idppe = 1,2-ethanediylbis[bis[4-(1-methylethenyl)phenyl]-phosphine]) and *trans*-(CH$_3$CN)$_2$PdCl$_2$, were synthesized following literature procedures.[1,2]



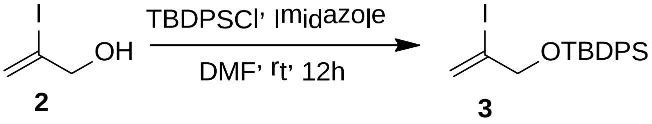
Scheme S1. Synthetic scheme of template.

1. Preparation of 2[3]



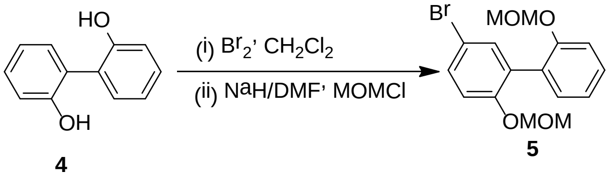
Chlorotrimethylsilane (14.68 mL, 160 mmol), water (1.44 mL), and propergyl alcohol (**1**, 4.72 mL, 80 mmol) were added slowly to a stirred solution of NaI (23.78 g, 160 mmol) in acetonitrile (160 mL). After 2 h at room temperature, water (50 mL) was added to the reaction mixture. The organic layer was extracted with diethyl ether (Et$_2$O; 120 mL), washed with 10% aqueous Na$_2$S$_2$O$_3$ (100 mL), and dried over anhydrous MgSO$_4$. The material was purified by column chromatography (silica gel, *n*-hexane, dichloromethane (DCM)). Yield: 4.4 g (30%). ^{1}H NMR (400 MHz, CDCl$_3$, Me$_4$Si, 298 K): δ = 2.25 (s, 1H; OH), 4.18 (s, 2H; CH$_2$), 5.86 (m, 1H; C=CH$_2$), 6.39 (m, 1H; C=CH$_2$) ppm. ^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$, Me$_4$Si, 298 K): δ = 71.04, 110.44, 124.48 ppm.

2. Preparation of 3[4]



Imidazole (1.38 g, 20.27 mmol) and *tert*-butylchlorodiphenylsilane (TBDPSCl) (2.67 mL) were added sequentially to a solution of 2-iodo-2-propen-1-ol (**2**, 1.5g, 8.15 mmol) in dimethylformamide (DMF; 25 mL) at room temperature under stirring. After 12 h, the reaction mixture was poured into saturated aqueous $NaHCO_3$ (35 mL). The organic layer was extracted three times with Et_2O (3 × 25 mL), and the combined organic layers were washed with H_2O (15 mL) and brine (15 mL). After drying over $MgSO_4$, the solvent was removed under vacuum. The crude compound was purified by flash column chromatography (silica gel, *n*-hexane). Yield: 2.7 g (78%). ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 1.07 (s, 9H; CH_3), 4.20-4.19 (t, 2H; CH_2), 5.85 (m, 1H; $C{=}CH_2$), 6.55 (m, 1H; $C{=}CH_2$), 7.36-7.86 (m, 10H; Ar) ppm. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 19.30, 26.72, 71.31, 108.78, 123.29, 127.78, 129.88, 132.95, 135.44 ppm.

3. Preparation of 5[5,6]

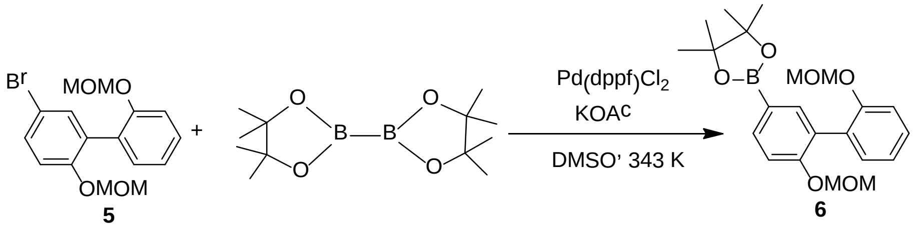


In a 100 mL Schlenk tube bromine (2.02 mL, 40 mmol) was added slowly to a solution of 2,2′-dihydroxybiphenyl (**4**, 7.44 g, 40 mmol) in DCM (50 mL) under vigorous stirring. After stirring for 24 h at room temperature, the reaction mixture was cooled to 273 K and filtered. The filtrate was concentrated and the crude product was purified by flash column chromatography (silica gel, DCM). The product was a mixture of 2,2′-dihydroxybiphenyl, 5-bromo-2,2′-dihydroxy-1,1′-biphenyl (**5**), and 5,5′-dibromo-2,2′-dihydroxy-1,1′-biphenyl. The mixture was used without further purification to the next step.

A 50 mL flask was charged with the crude mixture (1 g) dissolved in dry DMF (10 mL). A suspension of *n*-hexane-washed NaH (60% in paraffin oil, 880 mg, 22 mmol) in dry DMF (10 mL) was slowly added to the crude solution at 273 K. After stirring for 15 min chloromethyl methyl ether (MOMCl; 1 mL) was added to the reaction mixture at 273 K over 30 min. The reaction continued overnight at room temperature and was quenched by pouring the reaction mixture into ice water (40 mL). The organic layer was extracted with DCM and ethyl acetate and the combined organic layers were washed with water and brine. The organic extracts were dried over anhydrous $MgSO_4$, and the solvent was removed under vacuum. The crude compound was purified by column chromatography (silica gel, CH_2Cl_2/hexane; 2:1). Yield: 450 mg (35%) ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 3.31 (s, 3H; CH_3), 3.34 (s, 3H; CH_3), 5.02 (s, 2H; CH_2), 5.07 (s, 2H; CH_2), 7.02-7.39 (m, 7H, Ar) ppm.

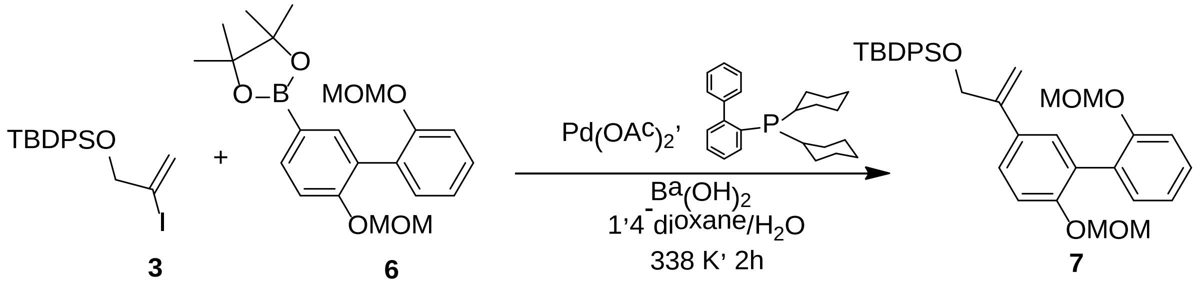
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 55.88, 55.91, 95.11, 95.23, 114.04, 115.34, 117.20, 121.76, 127.63, 129.15, 131.23, 131.26, 134.05, 154.09, 154.74 ppm.

4. Preparation of 6[7]



A mixture of Pd(dppf)Cl_2 (652 mg, 0.89 mmol), potassium acetate (7.72 g, 78.66 mmol), bis(pinacolato)diboron (7.03 g, 27.68 mmol), and **5** (9.32 g, 26.38 mmol) in dry dimethyl sulfoxide (DMSO; 100 mL) was degassed thoroughly. The reaction mixture was left at 343 K under nitrogen for 9 h. Toluene (150 mL) was added, and the reaction mixture was washed with water and brine, and then dried over anhydrous $MgSO_4$. The solvent was removed under vacuum and the crude compound was purified by column chromatography (silica gel, DCM). Yield: 7.72 g (73%). ^1H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 1.26 (s, 6H; CH_3), 1.31 (s, 6H; CH_3), 3.34 (s, 6H; CH_3), 5.05 (s, 2H; CH_2), 5.11 (s, 2H; CH_2), 7.02-7.78 (m, 7H; Ar) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 24.86, 25.03, 55.87, 83.49, 8358, 94.66, 94.69, 94.71, 95.13, 95.16, 95.17, 114.22, 115.27, 121.72, 128.41, 128.61, 129.11, 131.48, 135.68, 138.29, 154.98, 157.48 ppm.

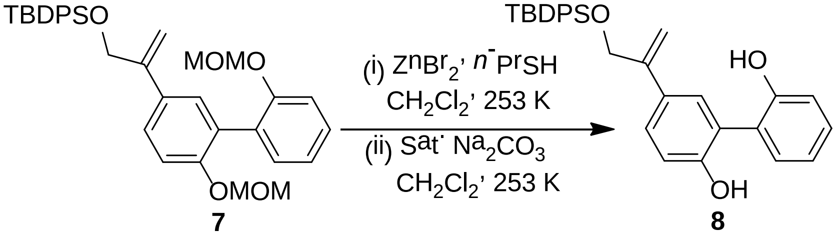
5. Preparation of 7[8]



A 50 mL Schlenk flask was charged sequentially with **3** (2.08 g, 4.92 mmol), **6** (1.5 g, 3.74 mmol), Pd$(OAc)_2$ (81.6 mg, 0.36 mmol), *o*-(dicyclohexylphosphino)biphenyl (637 mg, 1.81 mmol), $Ba(OH)_2$, (1.91 g, 11.14 mmol), 1,4-dioxane (23 mL), and H_2O (12.5 mL). The reaction mixture was degassed for 30 min and back filled with nitrogen. After stirring at 338 K for 1 h, the reaction mixture was cooled to room temperature and filtered through a short column (silica gel, Et_2O). The solvent was removed, and the crude was purified by column chromatography (silica gel, *n*-hexane/EtOAc; 5:1). Yield: 1.65 g (92%). ^1H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 1.06 (s, 9H; $3\times CH_3$), 3.27 (s, 3H; CH_3), 3.30 (s, 3H; CH_3), 4.54 (s, 2H; CH_2), 5.02-5.03 (2s, 4H; $2\times CH_2$), 5.44(d, 1H; $C{=}CH_2$), 5.50 (d, 1H; $C{=}CH_2$), 7.02-7.70 (m, 17H, Ar) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$,

Me_4Si, 298 K): δ = 19.33, 26.56, 55.96, 65.23, 95.09, 110.44, 115.19, 115.36, 121.72, 126.19, 127.69, 128.72, 128.83, 128.88, 129.10, 129.66, 131.47, 132.52, 133.49, 134.79, 135.20, 135.50, 145.80, 154.47, 154.87 ppm.

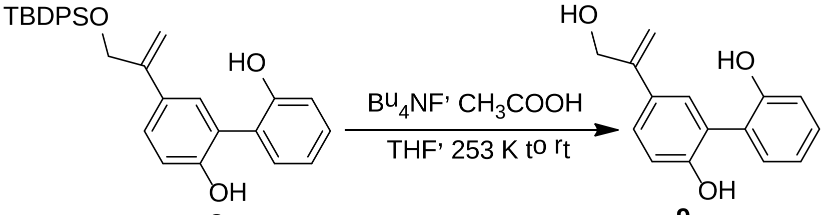
6. Preparation of 8[9]



7 **8**

n-PrSH (236 μL, 2.60 mmol) was added to a solution of **7** (590 mg, 1.04 mmol) in CH_2Cl_2 (7.7 mL) at 253 K. After stirring for 10 min, $ZnBr_2$ (234 mg, 1.04 mmol) was added in a single portion under nitrogen. The cooling bath was removed and after 15 min the mixture was then diluted with DCM (28 mL) at 253 K. Aqueous saturated $NaHCO_3$ was added slowly over 15 min at this temperature. The reaction mixture was filtered through celite with Et_2O. The organic layer was dried over Na_2SO_4. The solvent was removed under vacuum and the crude product was purified by column chromatography (silica gel, *n*-hexane/EtOAc; 2:1). Yield: 210 mg (42%). ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 1.06 (s, 9H; 3×CH_3), 4.53 (t, 2H; CH_2), 5.41 (d, 1H; C=CH_2), 5.45 (d, 1H; C=CH_2), 5.58 (br, OH), 6.92-7.69 (m, 17H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 19.30, 26.77, 65.42, 110.99, 116.49, 116.61, 121.62, 123.35, 123.48, 127.70, 128.95, 129.70, 130.02, 131.37, 132.58, 133.27, 133.44, 135.53, 135.64, 136.83, 145.63, 152.52, 152.82 ppm.

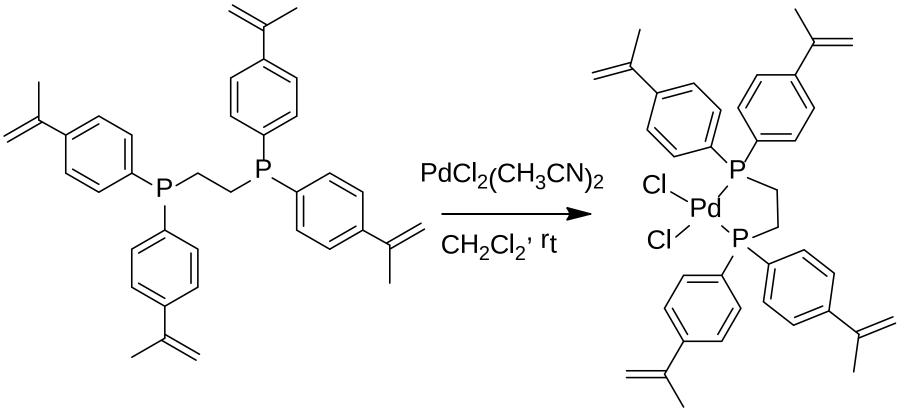
7. Preparation of 9 [10]



8 **9**

Bu_4NF (1 M, 5.36 mL) and CH_3COOH (0.59 mL) were added sequentially to a solution of **8** (1.00 g, 2.08 mmol) in anhydrous THF (18 mL) at 253 K, and the solution was gradually warmed to room temperature. After 12 h, aqueous saturated $NaHCO_3$ was added to the mixture at 273 K and extracted with ethyl acetate. The combined organic layer was washed with water and brine, and dried over $MgSO_4$. The solvent was removed and the crude compound was purified by column chromatography (silica gel, *n*-hexane/EtOac; 2:1). Yield: 163 mg (32%). ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 4.53 (s, 2H; CH_2), 5.28 (s, 1H, C=CH_2), 5.42 (s, 1H; C=CH_2), 5.88 (br, OH), 6.97-7.42 (m, 7H; Ar) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 65.24, 112.17, 116.75, 116.83, 121.66, 123.63, 123.91, 127.65, 129.05, 130.03, 131.37, 131.65, 146.03, 152.90, 152.95 ppm.

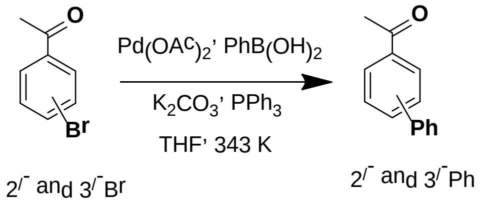
8. Preparation of (idppe)$PdCl_2$



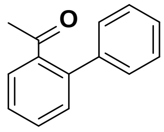
A solution of idppe (430 mg, 0.77 mmol) in DCM (20 mL) was added slowly to a solution of $(CH_3CN)_2PdCl_2$ (200 mg, 0.77 mmol) in DCM (20 mL) at room temperature. The reaction mixture was stirred overnight, and then filtered. The solvent was removed under vacuum to obtain the light yellow product. Quantitative yield. ^1H NMR (500 MHz, $CDCl_3$, Me_4Si, 298 K): $\delta = 2.15$ (s, 12H; CH_3), 2.42 (s, 2H; P-CH_2), 2.47 (s, 2H; P-CH_2), 5.20 (m, 4H; $C(CH_3){=}CH_2$), 5.45 (s, 4H; $C(CH_3){=}CH_2$), 7.54 (dd, 8H; Ar), 7.83 (q, 8H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): $\delta = 21.40$, 28.14, 28.53, 114.90, 125.98, 126.37, 126.82, 133.50, 142.00, 144.78 ppm. ^{31}P NMR (202.35 MHz, $CDCl_3$, 298 K): $\delta = 62.96$ ppm.

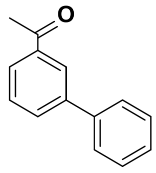
Preparation of standard compounds for catalytic reaction

9. Synthesis of 2- and 3-acetyl biphenyl

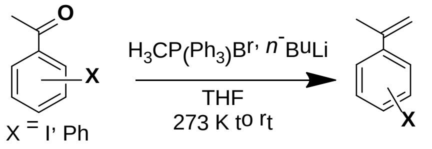


General procedure: A mixture of 3′-bromoacetophenone (10 g, 50.24 mmol), phenyl boronic acid (7.35 g, 60.29 mmol), triphenyl phosphine (2.62 g, 10 mmol), potassium carbonate (13.82 g, 100 mmol), and palladium acetate (1.13 g, 5.02 mmol) was degassed and the flask filled with nitrogen. After the addition of THF (100 mL), the reaction was stirred for 12 h at 343 K and passed down a column (silica gel, Et_2O). The solvent was removed and the crude product was purified by column chromatography (silica gel, *n*-hexane/DCM; 1/1).

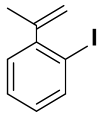
(a)  Eluent: *n*-hexane/DCM (1/1); yield: 64 %; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K): $\delta = 1.99$ (s, 3H; CH_3), 7.32-7.56 (m, 9H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): $\delta = 30.32$, 127.35, 127.77, 127.78, 128.58, 128.66, 128.76, 130.14, 130.61, 140.41, 140.63, 140.81, 204.72 ppm.

(b)  Eluent: *n*-hexane/DCM (1/1); yield: 47 %; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K): $\delta = 2.60$ (s, 3H; CH_3), 7.33-7.90 (m, 8H; Ar), 8.16 (s, 1H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): $\delta =$ 26.54, 126.72, 127.00, 127.03, 127.64, 128.76, 128.88, 131.52, 137.46, 139.97, 141.49, 197.82 ppm.

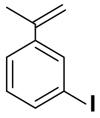
10. Synthesis of *o*-, *m*- and *p*-isopropenyl iodobenzene, and *o*-, *m*- and *p*-isopropenyl-biphenyl[11]



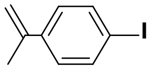
4-Acetylbiphenyl was purchased from Sigma-Aldrich and used without purification. *n*-BuLi (11.25 mL, 1.6 M in *n*-hexane) was added to a suspension of CH_3PPh_3Br (6.43 g, 18 mmol) in THF (30 mL) at 273 K. After 2 h, a solution of 4-acetylbiphenyl (2.94 g, 15 mmol) in THF (30 mL) was added to the triphenylphosphonium ylide solution. The reaction mixture was warmed to room temperature and stirred for 24 h. The reaction was quenched with aqueous NH_4Cl (30 mL) and extracted with EtOAc (100 mL). The organic layer was washed with brine (50 mL) and dried over anhydrous $MgSO_4$. After the solvent was removed, the crude product was purified by column chromatography (silica gel, *n*-hexane).



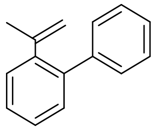
(a) Eluent: *n*-hexane; yield: 58 %; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K): δ = 2.06 (s, 3H; CH_3), 4.89 (s, 1H; CH_2), 5.22 (m, 1H; CH_2), 6.92-7.84 (m, 4H; C_6H_4) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 23.86, 96.89, 116.01, 128.02, 128.35, 128.48, 139.18, 148.38, 148.83 ppm.



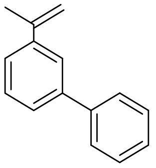
(b) Eluent: *n*-hexane; yield: 59 %; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K): δ = 2.11 (s, 3H; CH_3), 5.10 (s, 1H; CH_2), 5.34 (s, 1H; CH_2), 7.04-7.79 (m, 4H; C_6H4) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 21.64, 94.45, 113.61, 124.73, 129.87, 134.63, 136.25, 141.96, 143.53 ppm.



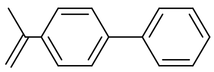
(c) Eluent: *n*-hexane; yield: 68 %; ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 2.11 (m, 3H; CH_3), 5.09 (m, 1H; CH_2), 5.36 (m, 1H; CH_2), 7.18-7.66 (m, 4H; C_6H_4) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ= 21.59, 92.85, 113.13, 127.41, 137.28, 140.74, 142.31 ppm.



(d) Eluent: *n*-hexane; yield: 13 %; ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 2.17 (m, 3H; CH_3), 5.10 (m, 1H; CH_2), 5.41 (m, 1H; CH_2), 7.29-7.67 (m, 9H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 21.88, 112.75, 124.45, 124.47, 126.28, 127.20, 127.25, 128.61, 128.71, 141.23, 141.32, 141.75, 143.25 ppm.



(e) Eluent: *n*-hexane; yield: 40 %; ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 1.65 (m, 3H; CH_3), 4.98 (m, 1H; CH_2), 5.06 (m, 1H; CH_2), 7.18-7.42 (m, 9H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 23.49, 116.21, 126.78, 127.14, 127.20, 128.00, 128.90, 129.08, 130.09, 139.57, 142.02, 142.79, 146.57 ppm.



(f) Eluent: *n*-hexane; yield: 88 %; ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 2.19 (m, 3H; CH_3), 5.11 (m, 1H; CH_2), 5.43 (m, 1H; CH_2), 7.32-7.61 (m, 9H; Ar) ppm. $^{13}C\{^1H\}$ NMR (125.65 MHz, $CDCl_3$, Me_4Si, 298 K): δ = 21.78, 112.44, 125.88, 126.90, 126.97, 127.25, 128.76, 140.11, 140.19, 140.76, 142.76 ppm.

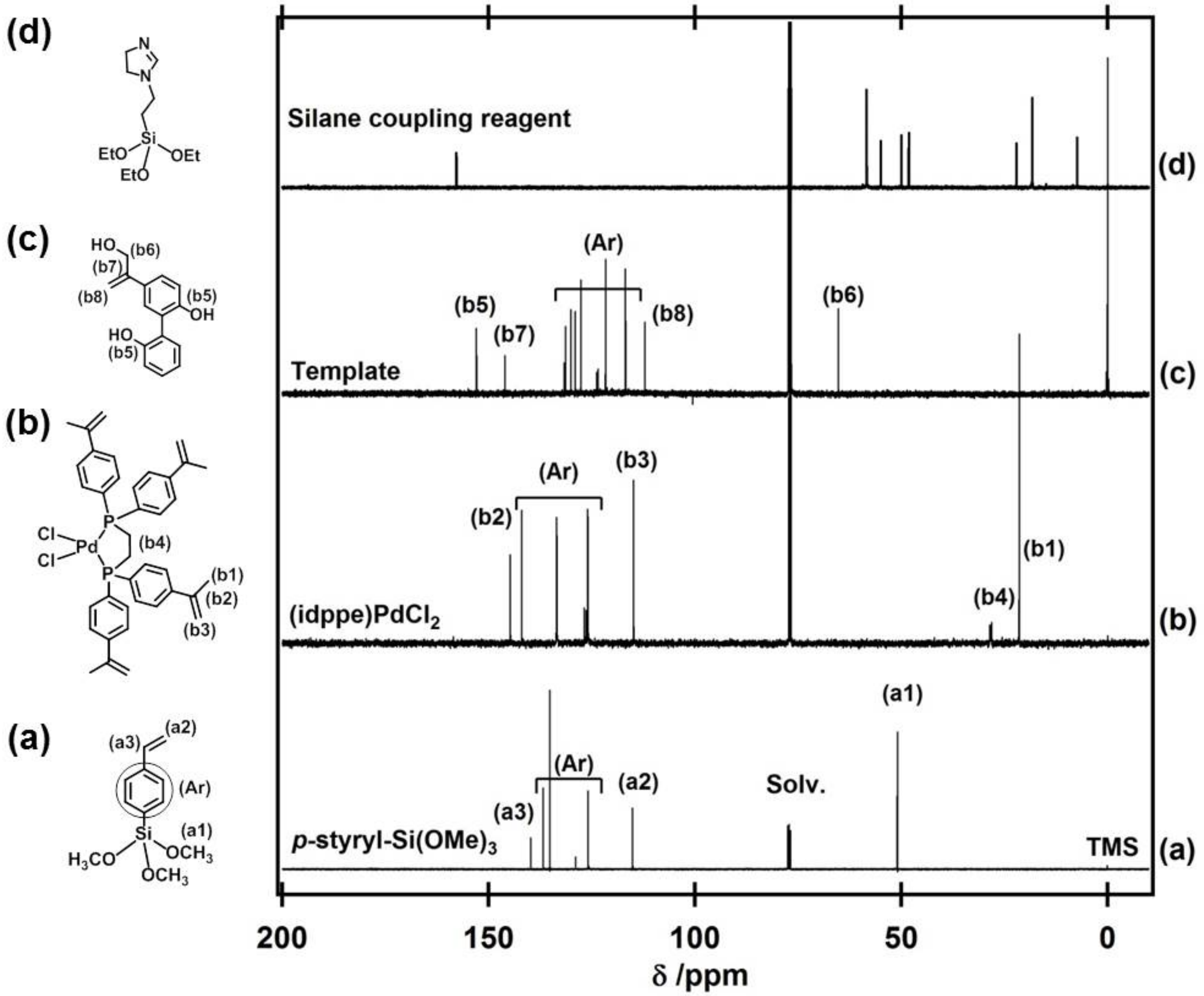


Fig. S1. ^{13}C LS NMR spectra (CDCl$_3$) of (a) *p*-styryltrimethoxysilane, (b) (idppe)PdCl$_2$, (c) template and (d) silane coupling reagent (triethoxy-3-(2-imidazolin-1-yl)propylsilane).

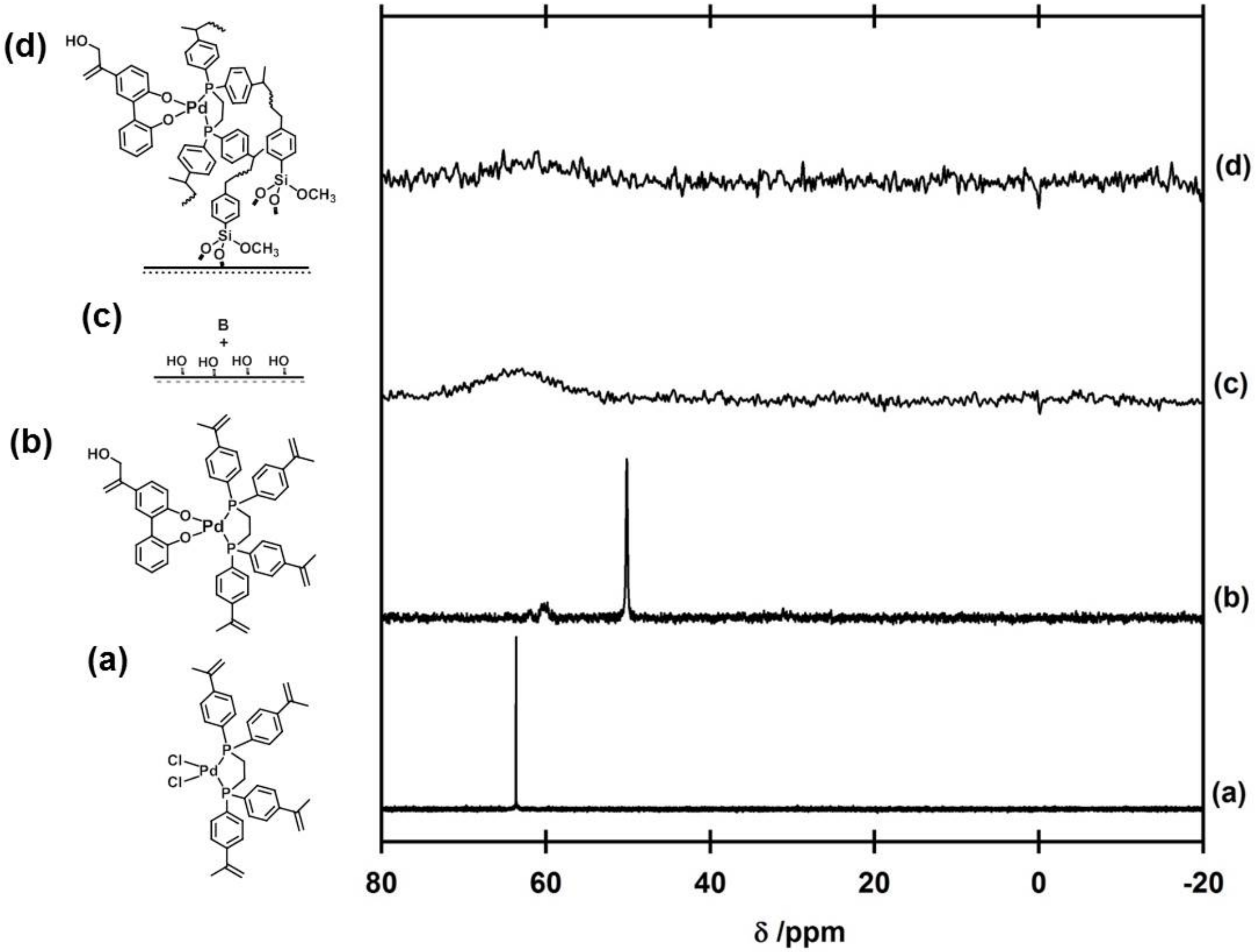


Fig. S2. ^{31}P LS NMR of (a) (idppe)PdCl$_2$, (b) **B** (THF-d^8) and ^{31}P SS MAS NMR of (c) **B** impregnated on SiO$_2$ and (d) **C**.

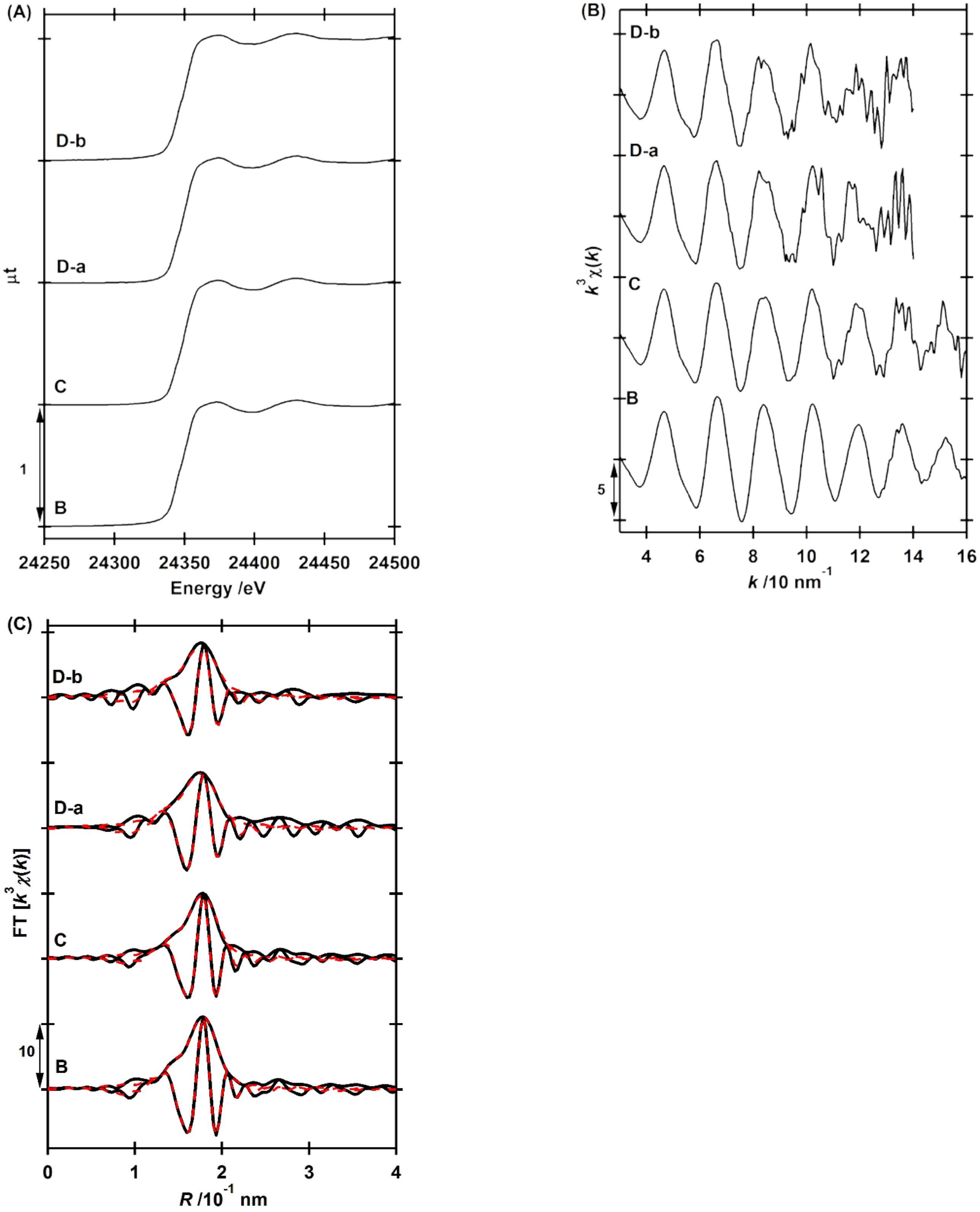


Fig. S3. Pd K-edge XANES spectra (A), EXAFS oscillations (B), and their EXAFS Fourier transforms (C) of **B**, **C**, **D-a**, and **D-b** measured at 20 K.

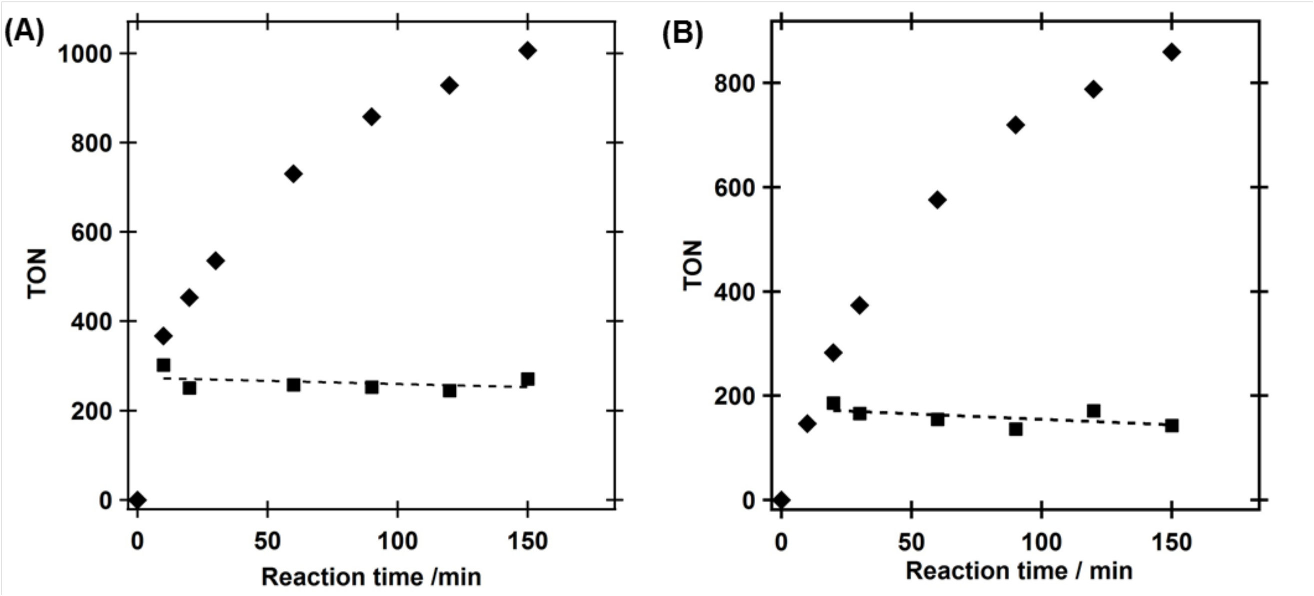


Fig. S4. Heterogeneity test of (A) **C** and (B) **D-b** under catalytic conditions. Reactions were conducted over the catalyst (◆) and the reaction filtrate (■).

Table S1. Curve fitting results for Pd K-edge EXAFS Fourier transforms of **B**, **C**, **D-a**, and **D-b**.

Shell	CN	Distance /nm	ΔE_0 /eV	σ^2 /×10^5 nm^2
B [a]				
Pd-P	2.0 (fix)	0.223 ± 0.001	5.4 ± 0.9	2.4 ± 0.2
Pd-O	2.0 (fix)	0.205 ± 0.001	6.2 ± 2.2	5.8 ± 0.8
C [b]				
Pd-P	1.7 ± 0.1	0.223 ± 0.001	5.4 (fix)	2.4 (fix)
Pd-O	2.0 ± 0.4	0.206 ± 0.002	6.2 (fix)	5.8 (fix)
D-a [c]				
Pd-P	1.6 ± 0.2	0.225 ± 0.001	5.4 (fix)	2.4 (fix)
Pd-O	1.9 ± 0.4	0.203 ± 0.002	6.2 (fix)	5.8 (fix)
D-b [d]				
Pd-P	1.5 ± 0.2	0.225 ± 0.001	5.4 (fix)	2.4 (fix)
Pd-O	1.6 ± 0.5	0.203 ± 0.002	6.2 (fix)	5.8 (fix)

[a] k = 30-160 nm^{-1}, R = 0.12-0.22 nm, R_f = 0.47%.

[b] k = 30-160 nm^{-1}, R = 0.12-0.22 nm, R_f = 1.0%.

[c] k = 30-140 nm^{-1}, R = 0.12-0.22 nm, R_f = 1.0%.

[d] k = 30-140 nm^{-1}, R = 0.12-0.22 nm, R_f = 1.3%.

Table S2. Results of Pd leaching from **C**, **D-a**, and **D-b**[a]

Catalyst	Time /h	TON	Pd leaching
C	2.5	1013	1.48%
D-a	2.5	987	0.22%
D-b	5	750	0.09%

[a] Pd (0.00244 mmol), Pd/*m*-isopropenyl iodobenzene/$PhB(OH)_2$/Cs_2CO_3/Dodecane = 1/1000/1500/2250/250, THF 6.1 mL, 333K. TON = [consumed *m*-isopropenyl iodobenzene (mol)/catalyst (mol)]×100.

Table S3. Results for catalyst reusability tests[a]

Catalyst	Conversion %
C	86%
C (reused)	89%
D-a	88%
D-a (reused)	90%

[a] Pd (0.0015 mmol), Pd/*m*-isopropenyl-iodobenzene/$PhB(OH)_2$/Cs_2CO_3/Dodecane = 1/200/300/450/50. THF 0.75 mL, 333 K, 3 h. [b] Conversion % = [consumed ArI/initial ArI] × 100.

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