Electronic Supplementary Information

A triazine-based covalent organic framework-palladium hybrid for one-pot silicon-based cross-coupling of silanes and aryl iodides

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Figure S1. Thermogravimetric analysis (TGA) data of (A) COF-SDU1 and (B) Pd(II)/COF-SDU1.
Figure S2. $^{13}$C CP/MAS NMR spectrum of trif.

Figure S3. XPS spectrum of Pd(OAc)$_2$ (blue line) and Pd(II)/COF-SDU1 (pink line).
Figure S4. PXRD patterns of Pd(II)/COF-SDU1 (a) before reused, (b) after reused first time and (c) three times.
Figure S5. Fourier transforms infrared (FT-IR) spectra of Pd(II)/COF-SDU1 (a) before reused, (b) after reused first time and (c) three times in the region of 500-2000 cm\(^{-1}\) with 2 cm\(^{-1}\) resolution.
Table S1 Catalytic activity test of Pd(II)/COF-SDU1 towards oxidation of organosilanes to organo(alkoxy)silanes or organosilanols.

<table>
<thead>
<tr>
<th>Entry</th>
<th>R_{(4-n)}SiH_n</th>
<th>ROH</th>
<th>1</th>
<th>t (h)^a</th>
<th>Yield (%)^bc</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PhSiH_3</td>
<td>H_2O</td>
<td>la</td>
<td>2</td>
<td>97.8</td>
</tr>
<tr>
<td>2</td>
<td>Me_2PhSiH</td>
<td>H_2O</td>
<td>lb</td>
<td>2</td>
<td>97.3</td>
</tr>
<tr>
<td>3</td>
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<td>2</td>
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<td>4</td>
<td>PhSiH_3</td>
<td>CH_3OH</td>
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<td>3</td>
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<td>5</td>
<td>PhSiH_3</td>
<td>C_2H_5OH</td>
<td>le</td>
<td>3</td>
<td>97.0</td>
</tr>
</tbody>
</table>

^a Reaction conditions: R_{(4-n)}SiH_n (1 mmol), ROH (5 mmol), Pd(II)/COF-SDU1 (20 mg, 8.5 \times 10^{-3} mmol of Pd) and THF (2 mL), room temperature. ^b Isolated yield. ^c The values are the average of two independent experiments.

Table S2 Catalytic activity test of Pd(II)/COF-SDU1 towards silicon-based cross-coupling reaction of the organo(alkoxy)silanes or organosilanols with aryl iodides.

<table>
<thead>
<tr>
<th>Entry</th>
<th>R^1</th>
<th>R^2</th>
<th>R^3</th>
<th>t (h)^a</th>
<th>Yield (%)^bc</th>
</tr>
</thead>
<tbody>
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<td>OCH_3</td>
<td>OCH_3</td>
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<tr>
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<td>3</td>
<td>OH</td>
<td>CH_3</td>
<td>CH_3</td>
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<td>66.5</td>
</tr>
</tbody>
</table>

^a Reaction conditions: organo(alkoxy)silanes or organosilanols (1 mmol), aryl halides (1.1 mmol), Pd(II)/COF-SDU1 (20 mg, 8.5 \times 10^{-3} mmol of Pd), TBAF (3 mmol) and THF (2 mL), 80 °C. ^b Isolated yield. ^c The values are the average of two independent experiments.
NMR signals:1-6

Phenylsilanetriol (1a): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.74-7.55 (m, 2H), 7.42-7.24 (m, 3H).

Dimethylphenylsilanol (1b): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.64-7.60 (m, 2H), 7.46-7.37 (m, 3H), 3.15 (bs, 1H), 0.41 (s, 6H).

Diphenylsilanediol (1c): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.73-7.70 (m, 4H), 7.49-7.33 (m, 6H), 2.17 (bs, 2H).

Phenyltrimethoxylsilane (1d): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$=7.71-7.69 (m, 2H), 7.46-7.40 (m, 3H), 3.65 (s, 9H).

Phenyltriethoxysilane (1e): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$=7.70-7.66 (m, 2H), 7.40-7.30 (m, 3H), 3.90-3.83 (q, $J$ = 6.9 Hz, 6H), 1.25-1.20 (t, $J$ = 7.2 Hz, 9H).

4-methyl-biphenyl (2a): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.53-7.49 (m, 2H), 7.44-7.41 (m, 2H), 7.40-7.32 (m, 2H), 7.28-7.22 (m, 1H), 7.19-7.15 (m, 2H), 2.33-2.29 (d, $J$ = 3.0 Hz, 3H). $R_f$ = 0.5 (100% petroleum ether).

2-methyl-biphenyl (2b): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.37-7.32 (m, 2H), 7.29-7.24 (m, 3H), 7.21-7.16 (m, 4H), 2.20 (s, 3H). $R_f$ = 0.5 (100% petroleum ether).

4-methoxy-biphenyl (2c): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.57-7.51 (m, 4H), 7.44-7.39 (m, 2H), 7.33-7.28 (m, 1H), 7.01-6.56 (m, 2H), 3.86 (s, 3H). $R_f$ = 0.5 (100% petroleum ether).

4-fluoro-biphenyl (2d): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.51-7.41 (m, 4H), 7.40-7.25 (m, 3H), 7.09-7.02 (m, 2H). $R_f$ = 0.5 (100% petroleum ether).

4-hydroxy-biphenyl (2e): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=7.57-7.53 (m, 2H), 7.51-7.47 (m, 2H), 7.45-7.39 (m, 2H), 7.34-7.28 (m, 1H), 6.94-6.89 (m, 2H). $R_f$ = 0.5 (petroleum ether/n-hexane 1:1).

4-nitro-biphenyl (2f): $^1$H NMR (300 MHz, CDCl$_3$): $\delta$=8.33-8.28 (m, 2H), 7.77-7.72 (m, 2H), 7.65-7.61 (m, 2H), 7.53-7.42 (m, 3H). $R_f$ = 0.5 (petroleum ether/ethyl acetate 1:1).
References:


