Supplementary Information for

# Developing Highly Reducing Conjugated Porous Polymer: A Metal-Free and Recyclable Approach with Superior Performance for Pinacol C-C Coupling Under Visible Light

Feng Lan,<sup>a,c</sup> Cheng-Song Liu,<sup>a,c</sup> Cen Zhou,<sup>\*b</sup> Xiaozhou Huang,<sup>a</sup> Jun-Yan Wu,<sup>a</sup> Xiao Zhang<sup>\*a</sup>

<sup>*a*</sup>Fujian Key Laboratory of Polymer Science, Fujian Provincial Key Laboratory of Advanced Materials Oriented Chemical Engineering, College of Chemistry and Materials Science, Fujian Normal University, 8 Shangsan Lu, Fuzhou 350007, China. E-mail: zhangxiao@fjnu.edu.cn

<sup>b</sup>Fujian Engineering and Research Center of New Chinese Lacquer Materials, College of Materials and Chemical Engineering, Minjiang University, Fuzhou 350108, China. E-mail: zhoucen@mju.edu.cn

<sup>*c*</sup>These authors contributed equally.

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### **1.** General information

Unless stated otherwise, all reactions were carried out in flame-dried glassware under a dry nitrogen atmosphere. All reagents were commercially purchased and used without any further purification. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker instrument (400 MHz and 100 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protic solvent signals. Data for <sup>1</sup>H NMR are recorded as follows: chemical shift ( $\delta$ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for <sup>13</sup>C NMR are reported in terms of chemical shift ( $\delta$ , ppm).

Powder X-ray diffraction (PXRD) was recorded on a PANalytical X'pert PRO Xray Diffractometer using Cu-Kα radiation in the 2θ range of 10-90°. The nitrogen adsorption-desorption isotherms at 77 K were measured with Micromeritics ASAP2460 analyzers, and the BET surface area was estimated by the Brunauer-Emmett-Teller (BET) theory. Fourier Transform Infrared spectra were recorded with a Nicolet Is50 FT-IR spectrophotometer. UV-vis diffuse reflectance spectra (DRS) were performed on a Perkin Elmer Lambda 750 in the 300~800 nm range, with BaSO<sub>4</sub> as the reference substance. Electrochemical measurements were performed on an electrochemical workstation (CHI 660E, CH Instruments Inc., Shanghai).

### 2. Synthesis



#### Preparation of 2,7,12-triacetyl-5,5',10,10',15,15'-hexabutyltruxene (B):

Following a literature reported procedure<sup>[1]</sup>, AlCl<sub>3</sub> (5.89 g, 44.16 mmol) was slowly added to a solution of **A** (5.00 g, 7.36 mmol) and acetyl chloride (3.2 mL, 44.16 mmol) in dichloromethane (50 mL) at 0 °C. It was further stirred at room temperature for 12 hours. The mixture was gradually poured onto ice (~150 mL) with stirring. The resulting mixture was stirred at room temperature for 30 minutes. The aqueous solution was extracted with CH<sub>2</sub>Cl<sub>2</sub>. The resulting solution was dried over Na<sub>2</sub>SO<sub>4</sub>. After filtration, the solvent was evaporated under reduced pressure, the residue was purified by silica gel flash column chromatography using ethyl acetate/petroleum ether as the eluent to afford the desired product **B**<sup>[1]</sup> as a yellow solid in 83% yield (4.90 g). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.49 (d, *J* = 8.4 Hz, 3H), 8.11 (s, 3H), 8.06 (d, *J* = 8.4 Hz, 3H), 3.01-2.94 (m, 6H), 2.74 (s, 9H), 2.26-2.19 (m, 6H), 0.94-0.86 (m, 12H), 0.52-0.41 (m, 30H).

# **Preparation of 5,5',10,10',15,15'-hexabutyltruxene-2,7,12-tricarboxylic acid (C):** Following a literature reported procedure<sup>[1]</sup>, **B** (2.35 g, 2.90 mmol) was suspended

and stirred in 1,4-dioxane (60 mL). NaOH (4.55 g, 113.75 mmol) was dissolved in 30 mL of water. At 0 °C, bromine (2.00 mL) was added to the NaOH solution, which was stirred for 15 minutes. The NaOBr solution was added to the above suspension at room temperature. The resulting mixture was then stirred at 60 °C for 2 hours and cooled to room temperature. An aquous solution of NH<sub>2</sub>OH·HCl (2.62 g, 37.7 mmol) was added to the reaction mixture to quench NaOBr. Dioxane was removed under reduced pressure and the resulting aqueous suspension was acidified with 3 M HCl until pH < 1. The resulting solid was filtered, washed with water, and dried under vacuum. The desired product  $C^{[1]}$  was obtained as a yellow solid in 97% yield (2.30 g). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  13.08 (s, 3H), 8.51 (d, *J* = 8.8 Hz, 3H), 8.13-8.09 (m, 6H), 3.03-2.97 (m, 6H), 2.27-2.20 (m, 6H), 0.86-0.77 (m, 12H), 0.48-0.33 (m, 30H).

#### **Preparation of D:**

Thionyl chloride (4.70 g, 40.10 mmol) was slowly added to a solution of C (6.50 g, 8.01 mmol) in methanol (50 mL) at 0 °C. The mixture was stirred at 50 °C for 24 h and then cooled to room temperature. The resulting mixture was distilled to remove thionyl chloride. The residue was filtered, washed with methanol and dried under vacuum. The desired product **D** was obtained as a yellow solid in 88% yield (6.0 g). M.p. = 187.7-191.9 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.48 (d, *J* = 8.4 Hz, 3H), 8.18-8.15 (m, 6H), 4.01 (s, 9H), 3.03-2.95 (m, 6H), 2.26-2.19 (m, 6H), 0.94-0.85 (m, 12H), 0.55-0.41 (m, 30H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  167.3, 153.6, 147.9, 144.2, 137.9, 128.3, 128.0, 124.4, 123.3, 55.9, 52.1, 36.4, 26.4, 22.6, 13.7. IR (thin film): vmax (cm<sup>-1</sup>) = 2957, 2927, 2871, 2859, 1724, 1610, 1458, 1436, 1378, 1286, 1253, 1199, 1114, 1094, 768, 756. HRMS (ESI) calcd for C<sub>57</sub>H<sub>73</sub>O<sub>6</sub> [M+H]<sup>+</sup>: 853.5407. Found: 853.5375.

### **Preparation of TX-1:**

Hydrazine hydrate (50 mL) was added to a solution of **D** (6.0 g, 7.03 mmol) in methanol (50 mL). The mixture was stirred at 90 °C for 24 h and then cooled to room temperature. The resulting mixture was filtered, washed with methanol and dried under

vacuum. The desired product **TX-1** was obtained as a white solid in 47% yield (2.8 g). M.p. > 280 °C. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  9.93 (br, 3H), 8.42 (br, 3H), 8.05-7.92 (m, 6H), 4.67 (br, 6H), 2.96 (br, 6H), 2.21 (br, 6H), 0.80 (br, 12H), 0.35 (br, 30H). <sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  165.9, 153.0, 146.1, 141.8, 137.7, 131.6, 125.9, 124.1, 120.8, 55.4, 35.6, 26.3, 22.2, 13.7. IR (thin film): vmax (cm<sup>-1</sup>) = 3304, 3169, 2955, 2928, 2859, 1640, 1614, 1578, 1531, 1479, 1459, 1334, 1102. HRMS (ESI) calcd for C<sub>54</sub>H<sub>73</sub>N<sub>6</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 853.5744. Found: 853.5664.



### **Preparation of TX-OXD:**

Benzoic acid (0.214 g, 1.760 mmol), lithium chloride (0.165 g), pyridine (0.826 mL) and triphenyl phosphite (0.43 mL) were added to a solution of **TX-1** (0.500 g, 0.586 mmol) in N-methyl-2-pyrrolidone (3.3 mL). The mixture was stirred at 90 °C for 24 h and then cooled to room temperature. The mixture was added 50 mL of water and stirred for 30 minutes. Afterwards, the resulting mixture was filtered, washed with water, dichloromethane and dried in vacuo to give **TX-2** as a yellow solid (0.24 g). M.p. = 229.9 - 234.3 °C. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  10.74 (s, 3H), 10.63 (s, 3H), 8.57 (d, *J* = 8.4 Hz, 3H), 8.21 (s, 3H), 8.08 (d, *J* = 7.6 Hz, 3H), 8.00 (d, *J* = 7.2 Hz, 6H), 7.66-7.56 (m, 9H), 3.06 (s, 6H), 2.30 (s, 6H), 0.93-0.82 (m, 12H), 0.54-0.39 (m, 30H). <sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  166.1, 165.9, 153.2, 146.7, 142.5, 137.8, 132.7,

132.0, 130.9, 128.6, 127.5, 126.7, 124.4, 121.3, 55.6, 35.7, 26.4, 22.3, 13.8. IR (thin film): vmax (cm<sup>-1</sup>) = 3247, 3063, 2957, 2928, 2859, 1632, 1579, 1503, 1465, 1378, 1283, 1263, 708, 692. HRMS (ESI) calcd for  $C_{75}H_{85}N_6O_6 [M+H]^+$ : 1165.6531. Found: 1165.6561.

**TX-2** (0.20 g, 0.171 mmol) was added to phosphorus oxychloride (25 mL). The mixture was heated to 120 °C and stirred for another 12 h and then cooled to room temperature. The reaction mixture was poured gradually on ice and stirred for 30 minutes. Afterwards, the aqueous solution was extracted with CH<sub>2</sub>Cl<sub>2</sub>. The resulting solution was dried over Na<sub>2</sub>SO<sub>4</sub>. After filtration, the solvent was evaporated under reduced pressure, the residue was purified by silica gel flash column chromatography using ethyl acetate/petroleum ether as the eluent to afford the desired product **TX-OXD** as a yellow solid (0.087 g). M.p. > 280 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.64 (d, *J* = 6.8 Hz, 3H), 8.33-8.25 (m, 12H), 7.63-7.61 (m, 9H), 3.10-3.04 (m, 6H), 2.36-2.29 (m, 6H), 0.98-0.93 (m, 12H), 0.64-0.47 (m, 30H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  165.0, 164.6, 154.4, 147.5, 143.2, 138.0, 131.8, 129.1, 127.0, 125.5, 125.2, 124.0, 122.2, 120.7, 56.2, 36.6, 26.6, 22.7, 13.8. IR (thin film): vmax (cm<sup>-1</sup>) = 2955, 2926, 2858, 1613, 1546, 1489, 1473, 1419, 1377, 1258, 1067, 728, 688. HRMS (ESI) calcd for C<sub>75</sub>H<sub>79</sub>N<sub>6</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 1111.6214. Found: 1111.6171.

#### **Preparation of TX-OXD-P:**

Trimesic acid (0.123 g, 0.586 mmol), lithium chloride (0.165 g), pyridine (0.825 mL) and triphenyl phosphite (0.43 mL) were added to a solution of **TX-1** (0.500 g, 0.586 mmol) in N-methyl-2-pyrrolidone (3.3 mL). The mixture was stirred at 90 °C for 24 h and then cooled to room temperature. The mixture was added 30 mL of methanol and stirred for 30 minutes. Afterwards, the resulting mixture was filtered, washed with methanol and dried in vacuo to give **TX-2-P** as a yellow solid (0.48 g), which was used directly for the next step. **TX-2-P** (0.48 g, 0.476 mmol) was added to phosphorus oxychloride (25 mL). The mixture was heated to 120 °C and stirred for another 12 h and then cooled to room temperature. The reaction mixture was poured gradually on

ice and stirred for 30 minutes. Afterwards, the resulting mixture was filtered, washed with water, ethanol, methanol, acetone, dichloromethane and dried under vacuum. The desired product **TX-OXD-P** was obtained as a yellow solid (0.38 g).



#### **Preparation of TX-P:**

Following a literature reported procedure<sup>2</sup>, thionyl chloride (3.48 mL, 0.048 mmol) was added dropwise to the solution of **B** (0.386 g, 0.48 mmol) in the mixture of anhydrous ethanol (20 mL) and toluene (20 mL) at 0 °C under nitrogen atmosphere, and then stirred at room temperature for 72 h. The product was poured into water and filtered. The solid was thoroughly washed with methanol, chloroform, acetone, and tetrahydrofuran. Further purification was carried out by Soxhlet extraction with methanol, chloroform, and tetrahydrofuran for 24 h each, and then dried under vacuum. The desired product **TX-P** was obtained as a brown solid (0.314 g).

## 3. Characterization



Fig. S2 N<sub>2</sub>-physisorption isotherm and pore size distribution curve (inset) of TX-P.



Fig. S3 PXRD patterns TX-OXD-P and of TX-P.



Fig. S4 SEM image of TX-P.



Fig. S5 UV/vis absorption spectrum of TX-OXD in acetone.



Fig. S6 Emission spectrum of TX-OXD in acetone.



Fig. S7 C 1s XPS spectrum of TX-OXD-P.<sup>[3]</sup>



Fig. S8 N 1s XPS spectrum of TX-OXD-P.<sup>[4-5]</sup>



Fig. S9 O 1s XPS spectrum of TX-OXD-P.<sup>[6]</sup>



**Fig. S10** Fluorescence decay curves of TX-OXD-P and TX-P in solid state at 298 K. The emission lifetimes of TX-OXD-P and TX-P were measured at their emission maxima with excitation at 481 nm and 539 nm, respectively.

### 4. General procedure for visible-light-induced pinacol C-C coupling



To a sealed tube were added the substrate **1** (0.2 mmol, 1.0 equiv), DIPEA (0.4 mmol, 2.0 equiv), TX-OXD-P (5 mol%), and acetone (1 mL). The reaction mixture was degassed via freeze-pump-thaw for 3 cycles. After the mixture was thoroughly degassed, the vial was sealed and positioned approximately 2~3 cm from 30 W blue LEDs. The mixture was stirred at room temperature for the indicated time (monitored by TLC) under nitrogen atmosphere. Afterwards, the catalyst was separated by filtration and washed with dichloromethane. Then the filtrate was concentrated by rotary evaporation and the residue was purified by silica gel flash column chromatography using ethyl acetate/petroleum ether as the eluent to afford the desired products **2**. The analytical data of the products are summarized below. Two isomers were observed by NMR.



**2a**<sup>[7]</sup>, white solid, 24.8 mg, 88% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.31-7.09 (m, 8H), 5.52-5.41 (m, 2H), 4.63-4.56 (m, 2H).



**2b**<sup>[8]</sup>, white solid, 17.6 mg, 62% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.73-7.19 (m, 8H), 5.62-5.48 (m, 2H), 5.24-5.07 (m, 2H).



**2c**<sup>[9]</sup>, white solid, 20.7 mg, 59% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.63-7.09 (m, 6H), 5.65-5.38 (m, 2H), 2.75-2.71 (m, 2H).



**2d**<sup>[7]</sup>, white solid, 33.4 mg, 90% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.17-6.89 (m, 8H), 4.82-4.62 (m, 2H), 2.94-2.34 (m, 2H).



**2e**<sup>[7]</sup>, white solid, 17.5 mg, 63% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.17-6.89 (m, 8H), 4.82-4.62 (m, 2H), 2.97-2.37 (m, 2H).



**2f**<sup>[8]</sup>, white solid, 35.2 mg, 97% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.56-7.23 (m, 8H), 4.97-4.76 (m, 2H).



**2g**<sup>[7]</sup>, white solid, 30.2 mg, 82% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.58-7.25 (m, 18H), 4.92-4.82 (m, 2H), 2.91 (m, 2H).



**2h**<sup>[7]</sup>, white solid, 26.0 mg, 80% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.40-7.15 (m, 8H), 4.75-4.71 (m, 2H), 2.66-1.97 (m, 2H), 1.33 and 1.29 (s, 18H).



**2i**<sup>[10]</sup>, white solid, 19.1 mg, 77% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.20-7.04 (m, 8H), 4.75-4.68 (m, 2H), 2.34 and 2.30 (s, 6H).



**2j**<sup>[8]</sup>, white solid, 18.0 mg, 74% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.22-6.90 (m, 8H), 4.70-4.65 (m, 2H), 2.89 (s, 1H), 2.33 and 2.28 (s, 6H), 2.19 (s, 1H).



**2k**<sup>[7]</sup>, white solid, 23.3mg, 95% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62-6.90 (m, 8H), 5.17-4.95 (m, 2H), 2.27 and 2.16 (s, 6H).



**2l**<sup>[7]</sup>, white solid, 21.2 mg, 73% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.13-6.71 (m,



**2m**<sup>[10]</sup>, yellow solid, 19.4 mg, 71% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.22-6.73 (m, 8H), 5.24-5.02 (m, 2H), 3.68 and 3.64 (s, 6H), 3.46-3.12 (m, 2H).



**2n**<sup>[11]</sup>, white solid, 23.0 mg, 69% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.88-6.63 (m, 6H), 4.73-4.63 (m, 2H), 3.88-3.77 (m, 12H).



**20**<sup>[12]</sup>, white solid, 25.2 mg, 64% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 6.51-6.38 (m, 4H), 4.49-4.45 (m, 2H), 3.69-3.59 (m, 18H).



**2p**<sup>[7]</sup>, white solid, 16.8 mg, 71% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.26-7.19 (m, 10H), 2.58 and 2.28 (s, 2H), 1.58 and 1.50 (s, 6H).



**2q**<sup>[7]</sup>, yellow solid, 34.9 mg, 97% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.85-7.25 (m, 14H), 2.81 and 2.39 (s, 2H), 1.68 and 1.61 (s, 6H).



**2r**<sup>[9]</sup>, white solid, 19.0 mg, 61% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.23-7.08 (m, 8H), 2.52 and 2.22 (s, 2H), 1.55 and 1.48 (s, 6H).



**2s**<sup>[13]</sup>, white solid, 16.1 mg, 58% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.20-6.88 (m, 8H), 2.53 and 2.30 (s, 2H), 1.57 and 1.49 (s, 6H).



**2t**<sup>[7]</sup>, white solid, 32.0 mg, 85% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.51-7.28 (m, 8H), 2.65 and 2.26 (s, 2H), 1.57 and 1.53 (s, 6H).



**2u**<sup>[14]</sup>, white solid, 23.3 mg, 77% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.32-6.65 (m, 8H), 4.83 and 4.72 (s, 2H), 3.72 and 3.68 (s, 6H), 1.41 and 1.25 (s, 6H).



**2v**<sup>[7]</sup>, white solid, 33.0 mg, 90% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31-7.28 (m, 8H), 7.18-7.17 (m, 12H), 3.04 (s, 2H).



**2w**<sup>[7]</sup>, white solid, 39.9 mg, 91% yield. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.32-7.28 (m, 8H), 6.96-6.92 (m, 8H), 6.28 (s, 2H).



**2x**<sup>[15]</sup>, white solid, 39.1 mg, 90% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42-7.07 (m, 18H), 2.96 and 2.93 (s, 2H).



 $2y^{[7]}$ , white solid, 31.9 mg, 81% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34-7.29 (m,

4H), 7.18-7.11 (m, 10H), 7.00-6.96 (m, 4H), 2.99 (s, 2H), 2.29 and 2.27 (s, 6H).



**2z**<sup>[14]</sup>, white solid, 35.3 mg, 84% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.33-7.28 (m, 4H), 7.18-7.15 (m, 10H), 6.72-6.68 (m, 4H), 3.76 and 3.75 (s, 6H), 2.96 (s, 2H).



**2aa**<sup>[15]</sup>, white solid, 29.0 mg, 69% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.15 (d, J = 8.4 Hz, 8H), 6.96 (d, J = 8.0 Hz, 8H), 2.95 (s, 2H), 2.28 (s, 12H).

### 5. Mechanistic studies

#### General procedure for the adsorption of substrate 1a:

The substrate adsorption experiment was performed followed a modified incipient wetness impregnation method.<sup>[16]</sup> To a grinding bowl was added 97 mg of dried TX-OXD-P. Then, compound **1a** (140 mg) was dissolved in 10 mL of acetone, and the solution was added dropwisely to TX-OXD-P accompanied with milling. This operation was stopped util the solid was going to become wet. Afterwards, acetone was carefully removed by rotary evaporation.



Fig. S11 N<sub>2</sub>-physisorption isotherms of TX-OXD-P before and after adsorption of substrate 1a.



Fig. S12 Pore size distribution curves of TX-OXD-P before and after adsorption of substrate 1a.

**DFT calculations:** 

To evaluate the molecular sizes of the reactants, structures of representative substrates **1a**, **1o**, and DIPEA were selected and investigated by theoretical calculation. Geometry optimization was performed at the B3LYP/6-31G(d,p) level of theory, in conjunction with the SMD model to account for the solvation effect of acetone. The optimized structures and the maximum distances between two atoms farthest apart in each structure are included in **Fig. S13**. Theoretical results indicate that the sizes of DIPEA and reactants **1a** and **1o** are all smaller than the minimized pores, supporting that both reactants could enter into the pores.



**Fig. S13** Optimized structures of representative reaction components and the maximum distances between two atoms farthest apart.

## 6. Recyclability tests of TX-OXD-P

 Table S1. Recyclability tests of TX-OXD-P for visible-light-induced pinacol C-C coupling.

O II	TX-OXD-P (5 mol%) OH DIPEA (2.0 equiv) $R^{1}$
$R^1 R^2$	acetone, blue LEDs R <sup>2</sup> OH
1	2
cycle	results
1	<b>2w</b> , 86% yield, 36 h
2	<b>2w</b> , 88% yield, 36 h
3	<b>2w</b> , 89% yield, 36 h
4	<b>2w</b> , 87% yield, 36 h
5	<b>2w</b> , 88% yield, 48 h

cycle	results
1	<b>2aa</b> , 71% yield, 36 h
2	<b>2c</b> , 63% yield, 36 h
3	<b>2s</b> , 63% yield, 42 h
4	<b>2h</b> , 78% yield, 46 h
5	<b>2a</b> , 82% yield, 48 h



Fig. S14 FTIR spectra of TX-OXD-P before and after recycling experiments.

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# 8. Copies of NMR spectra











# <sup>1</sup>H NMR Spectrum of **TX-OXD**.





#### S30













# <sup>1</sup>H NMR Spectrum of **2m**.





# <sup>1</sup>H NMR Spectrum of **2n**.











### S38

# <sup>1</sup>H NMR Spectrum of **2s**.









# <sup>1</sup>H NMR Spectrum of **2aa**.

