Supporting Information

Nickel-Catalyzed Synthesis of (E)-Olefins from Benzylic Alcohol Derivatives and Arylacetonitriles via C-O Activation

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1. General information

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All solvents were dried by standard methods. Ni(COD)$_2$ and phosphine ligands are stored under nitrogen in the glove box. Unless otherwise noted, all coupling reactions were performed in 10-mL glass vessel tubes and carried out under N$_2$ atmosphere.

Flash column chromatography was performed using 200-300 mesh silica gel. Visualization on TLC was achieved by the use of UV light (254 nm). All reactions were monitored by GC, GC-MS. $^1$H NMR, $^{13}$C NMR spectra were measured on a Bruker Avance III-400 spectrometer. ($^1$H 400 MHz, $^{13}$C 101 MHz), using CDCl$_3$ as the solvent with tetramethyilsilane (TMS) as the internal standard. Chemical shifts are reported in ppm and referenced to residual solvent peaks (CHCl$_3$ in CDCl$_3$: 7.26 ppm for $^1$H and 77.0 ppm for $^{13}$C). The coupling constants $J$ are given in Hz. Mass spectra were recorded by GCMS-QP2010 ultra spectrometer. The GC yields were accorded to the authentic samples/tridecane calibration standard from Shimadzu GC-2010 plus equipped with FID system.

2. Synthesis of starting materials

Substrates 1 were prepared according to the literature procedure.

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\text{Typical procedure: To a solution of naphthalen-2-ylmethanol (3.16 g, 20 mmol) and 4,4-dimethylaminopyridine (DMAP, 10 mol%) in CH}_2\text{Cl}_2 \text{ (30 mL) was added triethylamine (3.35 mL, 24 mmol, 1.2 equiv) at room temperature. Then pivaloyl chloride (2.95 mL, 24 mmol, 1.2 equiv) was added dropwise over 3 min at 0 °C. After stirring for 15 min, the reaction mixture was quenched with saturated NaHCO}_3 \text{ (10 mL), then the layers were separated. The aqueous layer was extracted with CH}_2\text{Cl}_2 \text{ (25 mL × 3) and the combined organic layer was dried over Na}_2\text{SO}_4 \text{ and then filtrated. After evaporation of the solvent under reduced pressure, the crude residue was purified by flash column chromatography (Petroleum ether/EtOAc = 10:1) to afford the product 1a in 90% yield.}
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3. Typical procedure for the Nickel-catalyzed Synthesis of (E)-Olefins from Benzyl Alcohol Derivatives and Arylacetonitriles via C-O Activation

\[
\begin{align*}
\text{1a} & \quad \text{OPiv} \quad + \quad \text{H-CN} \quad \xrightarrow{5 \text{ mol\% Ni(COD)}_2, 5 \text{ mol\% DPPP} \quad 2.5 \text{ equiv } t-\text{BuOK}} \quad \text{3a} \\
1.5 \text{ mL dioxane, } 100^\circ \text{C, 12h}
\end{align*}
\]

In a nitrogen-filled glove box, a 10 mL sealed schlenk tube equipped with a magnetic stir bar was charged with 1a (0.1 mmol), Ni(COD)_2 (0.005 mmol), DPPP (0.005 mmol), t-BuOK (0.25 mmol) and 2a (0.15 mmol) and dioxane (1.5 mL). The reaction mixture was stirred at 100 °C for 12 hours. After cooling the reaction mixture to room temperature, the mixture was passed through a short silica gel column with EtOAc as eluent. The filtrate was concentrated and the residue was further purified by column chromatography on silica gel to give the product 3a in 81 % isolated yield (86% GC yield).

4. Characterization and analytical data of products (average yields based on two parallel reactions)

\( (E)-2\text{-styrylnaphthalene 3a} \), yield for pivalate 81%, 37.3 mg; yield for carbamate 65%, 29.9 mg; \(^1\)H NMR (400 MHz CDCl_3): \( \delta \) 7.86–7.80 (m, 4H), 7.76–7.74 (m, 1H), 7.57 (d, \( J = 7.6 \text{ Hz} \), 2H), 7.50–7.43 (m, 2H), 7.39 (t, \( J = 7.6 \text{ Hz} \), 2H), 7.31–7.25 (m, 3H). \(^{13}\)C NMR (100 MHz CDCl_3): \( \delta \) 137.38, 134.84, 133.73, 133.06, 129.05, 128.79, 128.75, 128.32, 128.01, 127.71, 126.64, 126.56, 126.35, 125.92, 123.53. This compound is known.\(^1\)
(\textit{E})-2-(2-

 methylstyryl)naphthalene \textbf{3b}, yield 71\%, 34.6 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.85–7.74 (m, 5H), 7.65 (d, $J$ = 7.2 Hz, 1H), 7.49–7.42 (m, 3H), 7.24–7.14 (m, 4H), 2.47 (s, 3H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 136.44, 135.88, 135.20, 133.75, 133.06, 130.48, 130.10, 128.32, 128.02, 127.72, 127.63, 126.86, 126.62, 126.36, 126.27, 125.91, 125.37, 123.64, 20.00. This compound is known.$^2$

![Image of 3c](image)

(\textit{E})-2-(3-

 methylstyryl)naphthalene \textbf{3c}, yield 70\%, 34.2 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.82–7.78 (m, 4H), 7.73–7.70 (m, 1H), 7.47–7.40 (m, 2H), 7.36–7.34 (m, 2H), 7.27–7.20 (m, 3H), 7.08 (d, $J$ = 19.0 Hz, 1H), 2.38 (s, 3H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 138.31, 137.35, 134.98, 133.78, 133.07, 129.19, 128.68, 128.62, 128.58, 128.34, 128.05, 127.76, 127.32, 126.62, 126.37, 125.91, 123.81, 123.58, 21.53. This compound is known.$^3$

![Image of 3d](image)

(\textit{E})-2-(4-

 methylstyryl)naphthalene \textbf{3d}, yield 79\%, 38.6 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.82–7.70 (m, 5H), 7.45–7.40 (m, 4H), 7.20–7.16 (m, 4H), 2.36 (s, 3H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 137.63, 135.07, 134.63, 133.79, 132.99, 129.48, 129.02, 128.29, 127.99, 127.82, 127.72, 126.51, 126.41, 126.32, 125.81, 123.56, 21.31. This compound is known.$^4$
(E)-2-(2,4,6-trimethylstyryl)naphthalene 3e, yield 91%, 49.5 mg; \(^1\)H NMR (400 MHz CDCl\(_3\)): \(\delta\) 7.80–7.72 (m, 5H), 7.46–7.42 (m, 2H), 7.25–7.17 (m, 1H), 6.92–6.91 (m, 2H), 6.77–6.72 (m, 1H), 2.39–2.37 (m, 6H), 2.31–2.69 (m, 3H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \(\delta\) 136.44, 136.28, 135.29, 134.11, 133.83, 133.07, 128.89, 128.35, 128.04, 127.79, 127.41, 126.41, 126.22, 125.88, 123.46, 21.18, 21.09. This compound is known.\(^5\)

(E)-2-(4-methoxystyryl)naphthalene 3f, yield 85%, 44.2 mg. \(^1\)H NMR (400 MHz CDCl\(_3\)): \(\delta\) 7.80–7.69 (m, 5H), 7.49–7.41 (m, 4H), 7.22–7.09 (m, 2H), 6.90 (d, \(J = 8.4\) Hz, 2H), 3.81 (s, 3H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \(\delta\) 159.41, 135.20, 133.81, 132.89, 130.21, 128.61, 128.26, 127.93, 127.79, 127.70, 126.72, 126.29, 126.13, 125.70, 123.51, 114.22, 55.35. This compound is known.\(^4\)

(E)-N,N-dibutyl-4-(2-(naphthalen-2-yl)vinyl)aniline 3g, yield 58%, 41.5 mg. \(^1\)H NMR (400 MHz CDCl\(_3\)): \(\delta\) 7.80–7.70 (m, 5H), 7.45–7.37 (m, 4H), 7.16 (d, \(J = 16.0\) Hz, 2H), 6.87 (d, \(J = 8.4\) Hz, 2H), 6.15 (d, \(J = 16.0\) Hz, 2H), 3.10–2.98 (m, 8H), 2.78–2.70 (m, 5H), 2.09 (t, \(J = 7.2\) Hz, 2H), 1.19 (s, 3H), 1.07 (s, 3H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \(\delta\) 159.66, 136.67, 134.12, 133.83, 132.89, 130.21, 128.61, 128.26, 127.93, 127.79, 127.70, 126.72, 126.29, 126.13, 125.70, 123.51, 114.22, 55.35. This compound is known.
Hz, 1H), 7.03 (d, $J = 16.4$ Hz, 1H), 6.64 (d, $J = 8.4$ Hz, 2H), 3.29 (t, $J = 7.6$ Hz, 4H), 1.59–1.55 (m, 4H), 1.39–1.32 (m, 4H), 0.96 (t, $J = 7.2$ Hz, 6H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 147.92, 135.93, 133.92, 132.59, 129.35, 128.11, 127.85, 127.80, 127.67, 126.17, 125.36, 125.31, 124.51, 123.69, 123.58, 111.69, 50.83, 29.53, 20.39, 14.05.

(E)-2-(2-[[1,1'-biphenyl]-4-yl]vinyl)naphthalene 3h, yield 61%, 37.4 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.81–7.69 (m, 6H), 7.58–7.56 (m, 6H), 7.33–7.27 (m, 2H), 7.23 (d, $J = 8.0$ Hz, 2H), 7.19 (s, 2H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 139.68, 139.40, 135.43, 133.85, 132.74, 132.07, 127.84, 127.80, 127.55, 127.33, 127.00, 126.70, 126.39, 126.34, 125.96, 125.92, 125.64, 125.35, 124.91, 122.52. This compound is known.$^6$

(E)-2-(4-fluorostyryl)naphthalene 3i, yield 70%, 34.7 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.83–7.81 (m, 4H), 7.71 (d, $J = 8.8$ Hz, 1H), 7.53–7.44 (m, 4H), 7.18 (s, 2H), 7.07 (t, $J = 8.8$ Hz, 2H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 162.40 (d, $J_{C-F} = 245.9$ Hz), 134.68, 133.72, 133.57 (d, $J_{C-F} = 3.4$ Hz), 133.07, 128.59 (d, $J_{C-F} = 2.3$ Hz), 128.37, 128.07, 127.99, 127.82, 127.73, 126.59, 126.40, 125.96, 123.43, 115.69 (d, $J_{C-F} = 21.5$ Hz). This compound is known.$^7$
(E)-1-(2-(naphthalen-2-yl)vinyl)naphthalene 3j, yield 76%, 42.6 mg. $^1$H NMR (400 MHz CDCl$_3$): δ 8.27 (d, $J$ = 8.0 Hz, 1H), 8.00 (d, $J$ = 16.0 Hz, 1H), 7.91–7.78 (m, 8H), 7.57–7.43 (m, 5H), 7.31 (d, $J$ = 16.0 Hz, 1H). $^{13}$C NMR (100 MHz CDCl$_3$): δ 135.15, 135.06, 133.82, 133.78, 133.16, 131.85, 131.46, 128.69, 128.42, 128.13, 128.09, 127.77, 126.82, 126.42, 126.16, 126.11, 126.02, 125.89, 125.76, 123.83, 123.73, 123.65. This compound is known. 

(E)-1-styrylnaphthalene 3k, yield 75%, 34.5 mg. $^1$H NMR (400 MHz CDCl$_3$): δ 8.21 (d, $J$ = 8.0 Hz, 1H), 7.90–7.85 (m, 2H), 7.79 (d, $J$ = 8.4 Hz, 1H), 7.74 (d, $J$ = 6.8 Hz, 1H), 7.60 (d, $J$ = 7.6 Hz, 2H), 7.53-7.38 (m, 3H), 7.40 (t, $J$ = 7.2 Hz, 2H), 7.29 (t, $J$ = 7.2 Hz, 1H), 7.16–7.07 (m, 1H). $^{13}$C NMR (100 MHz CDCl$_3$): δ 137.67, 135.07, 133.78, 131.82, 131.45, 128.80, 128.66, 128.08, 127.82, 126.73, 126.13, 125.87, 125.86, 125.74, 123.82, 123.67. This compound is known.
(E)-1-methyl-4-styrylnaphthalene 3l, yield 63%, 30.7 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 8.22–8.20 (m, 1H), 8.02–7.99 (m, 1H), 7.88–7.81 (m, 1H), 7.63–7.50 (m, 5H), 7.39–7.24 (m, 4H), 7.11–7.04 (m, 1H), 2.69 (s, 3H). $^{13}$C NMR (100 MHz CDCl$_3$): 137.84, 134.43, 133.48, 132.81, 131.54, 131.12, 128.80, 127.68, 126.68, 126.13, 125.80, 125.74, 124.77, 124.41, 123.47, 19.72. This compound is known.$^{10}$

![cis-trans mixture](image)

(E)-prop-1-ene-1,2-diyl dibenzene 3m (cis-trans mixture), yield 46%, 22.5 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.85–7.82 (m, 2H), 7.69–7.67 (m, 0.5H), 7.62–7.56 (m, 1.5H), 7.51–7.46 (m, 2.5H), 7.41–7.21 (m, 5H), 7.00–6.99 (m, 1H), 6.64 (s, 0.5H), 2.36–2.26 (m, 3H). This compound is known.$^{7}$

![cis-trans mixture](image)

(E)-2-methoxy-6-(1-phenylprop-1-en-2-yl)naphthalene 3n, yield 40%, 21.9 mg. $^1$H NMR (400 MHz CDCl$_3$): 7.86 (s, 1H), 7.76–7.69 (m, 3H), 7.40–7.36 (m, 4H), 7.26–7.25 (m, 1H), 7.16–7.13 (m, 2H), 6.97 (s, 1H), 3.92 (s, 3H), 2.37 (s, 3H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 157.72, 138.94, 138.53, 137.22, 133.85, 129.71, 129.24, 128.94, 128.21, 127.51, 126.70, 126.45, 124.90, 124.58, 118.98, 105.66, 55.34, 17.48. This compound is known.$^{11}$

![cis-trans mixture](image)

(E)-1,2-diphenylethene 3o, yield 73%, 26.3 mg; $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.50
(d, \( J = 7.6 \) Hz, 4H), 7.34 (dd, \( J = 7.6 \) Hz, 7.6 Hz, 4H), 7.24 (t, \( J = 7.6 \) Hz, 2H), 7.10 (s, 2H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \( \delta \) 137.40, 128.76, 127.69, 126.59. This compound is known.\(^1\)

\((E)-1\text{-methyl-4-styrylbenzene 3p}\), for benzyl pivalate: yield 77\%, 29.9 mg; for 4-methylbenzyl pivalate: yield 75\%, 29.1 mg. \(^1\)H NMR (400 MHz CDCl\(_3\)): \( \delta \) 7.49 (d, \( J = 8.0 \) Hz, 2H), 7.40 (d, \( J = 8.0 \) Hz, 2H), 7.34 (t, \( J = 7.6 \) Hz, 2H), 7.23 (t, \( J = 7.6 \) Hz, 1H), 7.15 (d, \( J = 8.0 \) Hz, 2H), 7.06 (d, \( J = 2.4 \) Hz, 2H), 2.35 (s, 3H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \( \delta \) 137.57, 137.55, 134.60, 129.44, 128.69, 128.67, 127.75, 127.45, 126.48, 126.44, 21.29. This compound is known.\(^1\)

\((E)-1\text{-methoxy-4-styrylbenzene 3q}\), yield 61\%, 25.6 mg. \(^1\)H NMR (400 MHz CDCl\(_3\)): \( \delta \) 7.49–7.44 (m, 4H), 7.34 (t, \( J = 7.6 \) Hz, 2H), 7.25–7.23 (m, 1H), 7.06 (d, \( J = 16.4 \) Hz, 1H), 6.97 (d, \( J = 16.4 \) Hz, 1H), 6.90 (d, \( J = 8.8 \) Hz, 2H), 3.82 (s, 3H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \( \delta \) 159.33, 137.67, 130.17, 128.67, 128.23, 127.74, 127.23, 126.64, 126.28, 114.16, 55.35. This compound is known.\(^4\)

\((E)-4\text{-styryl-1,1\text{-biphenyl 3r}\), yield 53\%, 27.1 mg; \(^1\)H NMR (400 MHz CDCl\(_3\)): \( \delta \) 7.63–7.59 (m, 6H), 7.53 (d, \( J = 7.6 \) Hz, 2H), 7.44 (t, \( J = 8.0 \) Hz, 2H), 7.38–7.35 (m, 3H), 7.28–7.24 (m, 1H), 7.15 (s, 2H). \(^{13}\)C NMR (100 MHz CDCl\(_3\)): \( \delta \) 140.70, 140.37, 137.36, 136.42, 128.83, 128.78, 128.73, 128.23, 127.68, 127.38, 127.36, 126.97, 126.89, 126.84, 126.81, 126.78, 126.75, 126.72, 126.69, 126.66.
126.95, 126.56. This compound is known.¹²

(E)-1-(tert-butyl)-4-styrylbenzene 3s, yield 62%, 29.3 mg. ¹H NMR (400 MHz CDCl₃): 7.52–7.45 (m, 4H), 7.39–7.33 (m, 4H), 7.26–7.22 (m, 1H), 7.13–7.09 (m, 2H), 1.33 (s, 9H). ¹³C NMR (100 MHz CDCl₃): δ 150.82, 137.58, 134.60, 128.67, 128.53, 127.96, 127.44, 126.44, 126.28, 125.64, 34.66, 31.33. This compound is known.⁷

(E)-1-phenoxy-4-styrylbenzene 3t, yield 74%, 40.3 mg. ¹H NMR (400 MHz CDCl₃): δ 7.48(d, J = 8.0 Hz, 2H), 7.36–7.23 (m, 7H), 7.17–7.03 (m, 6H), 6.90 (d, J = 8.0 Hz, 1H). ¹³C NMR (100 MHz CDCl₃): δ 157.62, 157.26, 139.32, 137.08, 129.98, 129.83, 129.50, 128.74, 128.07, 127.85, 126.63, 123.31, 121.69, 118.91, 118.17, 116.75. This compound is known.¹³

(E)-methyl(4-styrylphenyl)sulfane 3u, yield 45%, 20.3 mg. ¹H NMR (400 MHz CDCl₃): 7.50 (d, J = 7.6 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.35 (t, J = 7.6 Hz, 2H), 7.25–7.23 (m, 3H), 7.06 (s, 2H), 2.50 (s, 3H). ¹³C NMR (100 MHz CDCl₃): δ 137.85, 137.32, 134.31, 128.69, 128.09, 128.03, 127.56, 126.89, 126.73, 126.43, 15.84. This compound is known.⁴
(E)-2-styrylpyridine 3v, yield 61%, 22.1 mg; $^1\text{H}$ NMR (400 MHz CDCl$_3$): $\delta$ 8.60 (d, $J = 4.4$ Hz, 1H), 7.68–7.58 (m, 4H), 7.42–7.36 (m, 3H), 7.32–7.28 (m, 1H), 7.21–7.16 (m, 2H). $^{13}\text{C}$ NMR (100 MHz CDCl$_3$): $\delta$ 155.49, 149.38, 136.83, 136.57, 133.10, 128.75, 128.45, 127.60, 127.17, 122.14, 122.12. This compound is known.$^9$

(E)-prop-1-ene-1,2-diyl dibenzene 3w, yield 64%, 24.8 mg; $^1\text{H}$ NMR (400 MHz CDCl$_3$): $\delta$ 7.53–7.51 (m, 1.2H), 7.39–7.17 (m, 7H), 7.10–7.05 (m, 1H), 6.95–6.93 (m, 0.8H), 6.84–6.47 (m, 1H), 2.28–2.20 (m, 3H). This compound is known.$^1$

(E)-1,3-dimethoxy-5-(4-methoxystyryl)benzene 3x, yield 92%, 49.7 mg. $^1\text{H}$ NMR (400 MHz CDCl$_3$): $\delta$ 7.44 (d, $J = 8.8$ Hz, 2H), 7.04 (d, $J = 16.0$ Hz, 1H), 6.90 (d, $J = 16.0$ Hz, 1H), 6.89 (d, $J = 8.8$ Hz, 2H), 6.65 (d, $J = 2.0$ Hz, 2H), 6.37 (t, $J = 2.0$ Hz, 1H), 3.82 (s, 9H). $^{13}\text{C}$ NMR (100 MHz CDCl$_3$): $\delta$ 160.99, 159.42, 139.72, 129.95, 128.76, 127.82, 126.59, 114.16, 104.35, 99.64, 55.37, 55.34. This compound is known.$^{14}$
1,4-bis((E)-2-methylstyreryl)benzene $3y$, yield 62%, 38.4 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.61 (d, $J = 7.2$ Hz, 2H), 7.53 (s, 4H), 7.38 – 7.34 (m, 2H), 7.22 – 7.19 (m, 6H), 7.03 – 6.99 (m, 2H), 2.45 (s, 6H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 137.04, 136.36, 135.86, 130.48, 129.56, 127.61, 126.92, 126.37, 126.26, 125.29, 20.00. This compound is known.$^{15}$

(3r,5r,7r)-1-(2-methoxy-5-(6-((E)-styryl)naphthalen-2-yl)phenyl)adamantane $3z$, yield 40%, 37.6 mg. $^1$H NMR (400 MHz CDCl$_3$): $\delta$ 7.96 (s, 1H), 7.88 – 7.86 (m, 3H), 7.77 – 7.72 (m, 2H), 7.60 – 7.52 (m, 4H), 6.39 (t, $J = 7.6$ Hz, 2H), 7.31 – 7.26 (m, 3H), 6.99 (d, $J = 8.4$ Hz, 1H), 3.90 (s, 3H), 2.19 (s, 6H), 2.11 (s, 3H), 1.81 (s, 6H). $^{13}$C NMR (100 MHz CDCl$_3$): $\delta$ 158.62, 139.05, 138.90, 137.45, 134.51, 133.42, 133.09, 132.49, 128.87, 128.78, 128.74, 128.47, 128.35, 127.63, 126.54, 126.40, 126.14, 125.85, 125.53, 124.85, 123.85, 112.12, 61.75, 55.19, 40.65, 37.17, 29.16.
5. References

6. Copies of $^1$H NMR and $^{13}$C NMR spectra