Supporting Information

Palladium-Catalyzed Decarbonylative Alkynylation of Amides

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1. General Information

The reactions were carried out in schlenk tubes of 25 mL under N₂ atmosphere. Reagents were used as received unless otherwise noted, and solvents were purified according to standard operation procedure. Column chromatography was performed using Silica Gel 60 (300–400 mesh). The reactions were monitored by GC and GC-MS, GC-MS results were recorded on GC-MS QP2010, and GC analysis was performed on GC 2010 plus. The ¹H, and ¹³C NMR spectra were recorded on a Brucker ADVANCE III spectrometer at 400 MHz, and 100 MHz respectively, and chemical shifts were reported in parts per million (ppm). The electron ionization (EI) method was used as the ionization method for the HRMS measurement, and the mass analyzer type is TOF for EI. All solvents and reagents were purchased from Energy Chemical, Alfa Aesar, and Aladdin.

2. Experimental Procedure

2.1 General Experimental Procedure for the Synthesis of Internal Alkynes.



In an oven dried 25 mL Schlenk tube charged with amides 1 (0.2 mmol), $Pd(OAc)_2 (0.006 \text{ mmol}, 3 \text{ mol }\%)$, dppp (0.012 mmol, 6 mol %) and Na₂CO₃ (0.2 mmol, 1.0 equiv), after charging N₂ for three times, the terminal alkynes 2 (0.6 mmol, 3.0 equiv), and dioxane (2 mL) were added. The reaction mixture was reacted at 150 °C for 16 h. After completion of the reaction, the reaction mixture was concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (300-400 mesh) using petroleum ether-DCM as eluent.

2.2 Experimental Procedure for the Preparation of 2-(phenylethynyl)naphthalene for 1 mmol scale.



In an oven dried 100 mL Schlenk tube charged with 1-(2-naphthoyl)piperidine-2,6-dione **1a** (1.0 mmol), $Pd(OAc)_2$ (3 mol %), dppp (6 mol %) and Na_2CO_3 (1.0 equiv), after charging N_2 for three times, the ethynylbenzene **2a** (4.0 equiv), and dioxane (20 mL) were added. The reaction mixture was reacted at 150 °C for 32 h. After completion of the reaction, the reaction mixture was concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (300-400 mesh) using petroleum ether as eluent to afford a white solid in 71% yield (162.1 mg).

3. Characterization Data for the Products





The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 78% yield (35.6 mg). ¹H NMR (400 M, CDCl₃): δ 8.05 (s, 1H), 7.81–7.78 (m, 3H), 7.57 (d, *J* = 8.0 Hz, 3H), 7.49–7.47 (m, 2H), 7.37–7.34 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 133.1, 132.9, 131.7, 131.5, 128.5, 128.4, 128.3, 128.0, 127.83, 127.82, 126.7, 126.6, 123.4, 120.6, 89.9, 89.8. This compound is known.¹

2-(p-tolylethynyl)naphthalene (3b)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 71% yield (34.4 mg). ¹H NMR (400 M, CDCl₃): δ 8.04 (s, 1H), 7.81–7.79 (m, 3H), 7.57 (d, *J* = 7.6 Hz, 3H), 7.49–7.46 (m, 4H), 7.17 (d, *J* = 8.0 Hz, 2H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 138.5, 133.1, 132.8, 131.6, 131.3, 129.2, 128.5, 128.0, 127.8, 126.6, 126.5, 120.8, 120.2, 90.0, 89.2, 21.6. This compound is known.¹

2-((4-methoxyphenyl)ethynyl)naphthalene (3c)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ DCM (5/1) to afford a white solid in 75% yield (38.7 mg). ¹H NMR (400 M, CDCl₃): δ 8.02 (s, 1H), 7.80–7.78 (m, 3H), 7.57–7.46 (m, 5H), 6.89 (d, *J* = 8.0 Hz, 2H), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 159.7, 133.2, 133.1, 132.7, 131.1, 128.5, 128.0, 127.8, 127.7, 126.5, 121.0, 115.4,

114.1, 89.8, 88.5, 55.3. This compound is known.¹

2-((4-(tert-butyl)phenyl)ethynyl)naphthalene (3d)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white in 90% yield (51.2 mg). ¹H NMR (400 M, CDCl₃): δ 8.04 (s, 1H), 7.82–7.78 (m, 3H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.52–7.47 (m, 4H), 7.38 (d, *J* = 8.0 Hz, 2H), 1.33 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ 151.6, 133.1, 132.8, 131.4, 131.3, 128.5, 128.0, 127.79, 126.6, 126.5, 125.4, 120.9, 120.30, 90.0, 89.2, 34.8, 31.2. This compound is known.²

N,N-dimethyl-4-(naphthalen-2-ylethynyl)aniline (3e)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ DCM (5/1) to afford a white solid in 85% yield (46.1 mg). ¹H NMR (400 M, CDCl₃): δ 8.00 (s, 1H), 7.78–7.76 (m, 3H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.45 (d, *J* = 8.4 Hz, 4H), 6.67 (d, *J* = 8.4 Hz, 2H), 2.98 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 150.2, 133.2, 132.8, 132.5, 130.7, 128.6, 127.9, 127.8, 127.7, 126.4, 126.2, 121.5, 111.9, 110.0, 91.1, 87.8, 40.2. This compound is known.³

2-((2-methoxyphenyl)ethynyl)naphthalene (3f)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ DCM (5/1) to afford a white solid in 74% yield (38.2 mg). ¹H NMR (400 M, CDCl₃): δ 8.08 (s, 1H), 7.81–7.78 (m, 3H), 7.61 (d, J = 8.4 Hz, 1H), 7.54 (d, J = 7.2 Hz, 1H), 7.48–7.46 (m, 2H), 7.31 (t, J = 8.0 Hz, 1H), 7.97–7.89 (m, 2H), 3.92 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 160.0, 133.7,

133.07, 132.8, 131.4, 129.9, 128.6, 127.9, 127.8, 127.8, 126.6, 126.5, 120.9, 120.6, 112.5, 110.8, 93.9, 86.2, 55.9. This compound is known.⁴

2-(*m*-tolylethynyl)naphthalene (3g)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 72% yield (34.9 mg). ¹H NMR (400 M, CDCl₃): δ 8.04 (s, 1H), 7.81–7.79 (m, 3H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.49–7.47 (m, 2H), 7.41–7.37 (m, 2H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 1H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 138.1, 133.1, 132.8, 132.3, 131.4, 129.3, 128.8, 128.5, 128.3, 128.0, 127.8, 126.7, 126.6, 123.1, 120.7, 90.0, 89.5, 21.30. This compound is known.⁵

2-((4-fluorophenyl)ethynyl)naphthalene (3h)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 80% yield (39.4 mg), mp 121–123 °C; ¹H NMR (400 M, CDCl₃): δ 8.04 (s, 1H), 7.82–7.80 (m, 3H), 7.57–7.54 (m, 3H), 7.50–7.48 (m, 2H), 7.08–7.04 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 162.5 (d, $J_{C-F} = 248.0$ Hz), 133.6, 133.5, 132.9 (d, $J_{C-F} = 18.5$ Hz), 131.4, 128.3, 128.1, 127.8, 126.7, 126.6, 120.4, 119.4 (d, $J_{C-F} = 4.0$ Hz), 115.7 (d, $J_{C-F} = 21.9$ Hz), 115.6, 89.5, 88.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -110.84; HRMS (EI) m/z: [M]⁺ calcd. for C₁₈H₁₁F: 246.0845; found: 246.0841.

2-((4-(trifluoromethyl)phenyl)ethynyl)naphthalene (3i)



The title compound was prepared according to the general procedure and purified by column chromatography on

silica gel and eluted with petroleum ether to afford a white solid in 56% yield (33.2 mg), mp 110–112 °C;. ¹H NMR (400 M, CDCl₃): δ 8.08 (s, 1H), 7.83 (d, J = 7.2 Hz, 3H), 7.68–7.57 (m, 5H), 7.52–7.51 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 133.1, 133.0, 131.9, 131.86, 131.8, 128.3, 128.2, 127.9, 127.8, 127.2, 127.0, 126.7, 126.6, 125.3 (q, J_{C-F} = 3.7 Hz, J_{C-F} = 7.4 Hz), 119.8, 92.2, 88.3. ¹⁹F NMR (376 MHz, CDCl₃): δ -62.74; HRMS (EI) m/z: [M]⁺ calcd. for C₁₉H₁₁F₃: 296.0813; found: 296.0810.

2-((4-chlorophenyl)ethynyl)naphthalene (3j)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 53% yield (27.8 mg). ¹H NMR (400 M, CDCl₃): δ 8.05 (s, 1H), 7.82–7.80 (m, 3H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 4H), 7.34 (d, *J* = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 134.3, 133.0, 132.9, 132.9, 131.6, 128.8, 128.3, 128.1, 127.8, 127.8, 126.8, 126.6, 121.8, 120.2, 90.8, 88.6. This compound is known.⁶

2-((3-chlorophenyl)ethynyl)naphthalene (3k)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 58% yield (30.5 mg). ¹H NMR (400 M, CDCl₃): δ 8.04 (s, 1H), 7.81–7.79 (m, 3H), 7.56–7.48 (m, 4H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.32–7.27 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 134.3, 133.0, 131.7, 131.5, 129.8, 129.6, 128.6, 128.3, 128.1, 127.9, 127.8, 126.9, 126.7, 125.1, 120.1, 91.0, 88.3. This compound is known.⁷

2-(naphthalen-2-ylethynyl)thiophene (3l)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 75% yield (35.1 mg). ¹H NMR (400 M, CDCl₃): δ 8.03 (s, 1H), 7.81–7.79 (m, 3H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.50–7.48 (m, 2H), 7.32–7.29 (m, 2H), 7.03–7.01 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 133.0, 132.9, 132.0, 131.3, 128.1, 128.1, 127.85, 127.82, 127.4, 127.2, 126.8, 126.6, 123.4, 120.3, 93.5, 83.0. This compound is known.⁸

3-(naphthalen-2-ylethynyl)thiophene (3m)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 76% yield (35.6 mg). ¹H NMR (400 M, CDCl₃): δ 8.03 (s, 1H), 7.80–7.78 (m, 3H), 7.57–7.47 (m, 4H), 7.30 (s, 1H), 7.23 (d, *J* = 3.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 133.1, 132.8, 131.4, 129.9, 128.7, 128.4, 128.0, 127.8, 126.7, 126.6, 125.5, 122.4, 120.6, 89.3, 84.9. This compound is known.⁹

trimethyl(naphthalen-2-ylethynyl)silane (3n)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a colorless oil in 72% yield (32.3 mg). ¹H NMR (400 M, CDCl₃): δ 7.99 (s, 1 H), 7.78–7.73 (m, 3H), 7.51–7.45 (m, 3H), 0.28 (s, 9 H); ¹³C NMR (100 MHz, CDCl₃): δ 132.9, 132.8, 131.9, 128.5, 127.8, 127.7, 127.7, 126.7, 126.5, 120.4, 105.4, 94.5, 0.0. This compound is known.¹⁰

triisopropyl(naphthalen-2-ylethynyl)silane (30)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a colorless oil in 81% yield (50.0 mg). ¹H NMR (400 M, CDCl₃): δ 7.99 (s, 1 H), 7.79–7.74 (m, 3H), 7.52–7.46 (m, 3H), 1.16–1.10 (m, 21H); ¹³C NMR (100 MHz, CDCl₃): δ 132.9, 132.8, 131.9, 128.8, 127.8, 127.75, 127.73, 126.6, 126.5, 120.9, 107.5, 90.9, 18.7, 11.4. This compound is known.¹⁰

2-(oct-1-yn-1-yl)naphthalene (3p)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a pale yellow oil in 86% yield (40.6 mg). ¹H NMR (400 M, CDCl₃): δ 7.90 (s, 1 H), 7.78–7.72 (m, 3H), 7.46–7.41 (m, 3H), 2.46–2.42 (m, 2H), 1.63 (dt, J_I = 7.2 Hz, J_2 = 14.4 Hz, 2H), 1.51–1.46 (m, 2H), 1.33 (br, 4H), 0.92–0.89 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 133.1, 132.5, 131.0, 128.8, 127.8, 127.7, 127.6, 126.4, 126.2, 121.5, 90.9, 80.9, 31.5, 28.8, 28.7, 22.6, 19.6, 14.1. This compound is known.¹¹

1-(phenylethynyl)naphthalene (3q)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a pale yellow oil in 80% yield (36.5 mg). ¹H NMR (400 M, CDCl₃): 8.45 (d, J = 8.4 Hz, 1H), 7.81–7.73 (m, 3H), 7.63 (d, J = 6.8 Hz, 2H), 7.58–7.54 (m, 1H), 7.50–7.46 (m, 1H), 7.41–7.37 (m, 1H), 7.36–7.31 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 133.4, 133.3, 131.8, 130.5, 128.9, 128.6, 128.5, 128.5, 126.9, 126.6, 126.4, 125.4, 123.5, 121.0, 94.5, 87.8. This compound is known.¹²

4-(phenylethynyl)-1,1'-biphenyl (3r)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 60% yield (30.5 mg). ¹H NMR (400 M, CDCl₃): 7.61–7.54 (m, 8H), 7.46–7.42 (m, 2H), 7.37–7.34 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 141.0, 140.4, 132.1, 131.7, 128.9, 128.4, 128.3, 127.7, 127.1, 123.3, 122.2, 90.1, 89.3. This compound is known.¹³

1,2-diphenylethyne (3s)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 68% yield (24.2 mg). ¹H NMR (400 M, CDCl₃): 7.54–7.52 (m, 4H), 7.34–7.30 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 131.6, 128.4, 128.3, 123.3, 89.4. This compound is known.¹³

1-fluoro-4-(phenylethynyl)benzene (3t)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 63% yield (24.7 mg). ¹H NMR (400 M, CDCl₃): 7.51–7.48 (m, 4H), 7.33–7.32 (m, 3H), 7.02 (t, J = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 163.5 (d, $J_{C-F} = 248.0$ Hz), 133.5 (d, $J_{C-F} = 8.3$ Hz), 131.6, 128.4, 128.4, 123.1, 119.4 (d, $J_{C-F} = 3.5$ Hz), 115.7 (d, $J_{C-F} = 21.9$ Hz), 89.1, 88.4. ¹⁹F NMR (376 MHz, CDCl₃): δ -110.88; This compound is known.¹³

1-methyl-4-(phenylethynyl)benzene (3u)



The title compound was prepared according to the general procedure and purified by column chromatography on

silica gel and eluted with petroleum ether to afford a white solid in 32% yield (12.3 mg). ¹H NMR (400 M, CDCl₃): 7.52 (d, J = 6.8 Hz, 2H), 7.42 (d, J = 7.6 Hz, 2H), 7.33–7.29 (m, 3H), 7.15 (d, J = 7.6 Hz, 2H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 138.4, 131.6, 131.5, 129.1, 128.3, 128.1, 123.5, 120.2, 89.6, 88.7, 21.6. This compound is known.¹¹

2-(phenylethynyl)benzofuran (3w)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 76% yield (33.2 mg). ¹H NMR (400 M, CDCl₃): 7.49 (br, 3H), 7.39 (d, J = 8.0 Hz, 1H), 7.29–7.25 (m, 4H), 7.18–7.15 (m, 1H), 6.92 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 154.9, 138.8, 131.7, 129.2, 128.5, 127.8, 125.6, 123.3, 121.9, 121.2, 111.6, 111.3, 95.1, 79.7. This compound is known.⁶

2-(phenylethynyl)benzo[b]thiophene (3x)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether to afford a white solid in 83% yield (38.9 mg). ¹H NMR (400 M, CDCl₃): 7.75 (br, 2H), 7.54 (br, 2H), 7.49 (s, 1H), 7.35 (br, 5H); ¹³C NMR (100 MHz, CDCl₃): δ 140.3, 139.2, 131.6, 128.8, 128.7, 128.5, 125.4, 124.8, 123.8, 123.3, 122.6, 122.0, 94.9, 83.0. This compound is known.⁶

4. References

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5. Copies of ¹H, ¹³C NMR Spectra of the Products

¹H NMR Spectrum of 2-(phenylethynyl)naphthalene (3a)



¹³C NMR Spectrum of 2-(phenylethynyl)naphthalene (3a)









100 90 f1 (ppm) -10

 1 H NMR Spectrum of 2-((4-methoxyphenyl)ethynyl)naphthalene (3c)



 ^{13}C NMR Spectrum of 2-((4-methoxyphenyl)ethynyl)naphthalene (3c)





¹H NMR Spectrum of 2-((4-(tert-butyl)phenyl)ethynyl)naphthalene (3d)

100 90 f1 (ppm) 80 70 60 50 40 30 20 10 0

120 110

190

180 170 160

150 140 130

-10





¹³C NMR Spectrum of N,N-dimethyl-4-(naphthalen-2-ylethynyl)aniline (3e)



 $^1\mathrm{H}$ NMR Spectrum of 2-((2-methoxyphenyl)ethynyl)naphthalene (3f)



 $^{13}\mathrm{C}$ NMR Spectrum of 2-((2-methoxyphenyl)ethynyl)naphthalene (3f)



100 90 f1 (ppm) -10 ò









 $^{13}\mathrm{C}$ NMR Spectrum of 2-((4-fluorophenyl)ethynyl)naphthalene (3h)







¹³C NMR Spectrum of 2-((4-(trifluoromethyl)phenyl)ethynyl)naphthalene (3i)







 $^{13}\mathrm{C}$ NMR Spectrum of 2-((4-chlorophenyl)ethynyl)naphthalene (3j)



¹H NMR Spectrum of 2-((3-chlorophenyl)ethynyl)naphthalene (3k)



¹³C NMR Spectrum of 2-((3-chlorophenyl)ethynyl)naphthalene (3k)



¹H NMR Spectrum of 2-(naphthalen-2-ylethynyl)thiophene (31)









¹³C NMR Spectrum of 3-(naphthalen-2-ylethynyl)thiophene (3m)



¹H NMR Spectrum of trimethyl(naphthalen-2-ylethynyl)silane (**3n**)



¹³C NMR Spectrum of trimethyl(naphthalen-2-ylethynyl)silane (3n)





¹H NMR Spectrum of triisopropyl(naphthalen-2-ylethynyl)silane (30)

¹³C NMR Spectrum of triisopropyl(naphthalen-2-ylethynyl)silane (30)





¹H NMR Spectrum of 1-(phenylethynyl)naphthalene (**3q**)



¹³C NMR Spectrum of 1-(phenylethynyl)naphthalene (**3q**)

2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	535	762	530 212 893
	-94	-87	777







¹³C NMR Spectrum of 4-(phenylethynyl)-1,1'-biphenyl (**3r**)





¹³C NMR Spectrum of 1,2-diphenylethyne (3s)







S32







190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 20 20 10 0 -10 fl (ppm)



¹³C NMR Spectrum of 2-(phenylethynyl)benzo[b]thiophene (**3** \mathbf{x})

