



## Supporting Information

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# Efficient Palladium-Catalyzed Coupling of Aryl Chlorides and Tosylates with Terminal Alkynes: Use of a Copper Co-Catalyst Inhibits the Reaction

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## General considerations:

**Reagents:** Pd(CH<sub>3</sub>CN)<sub>2</sub>Cl<sub>2</sub> was purchased from Strem (98% pure). **1** was prepared as previously described.<sup>1</sup> Cs<sub>2</sub>CO<sub>3</sub> was purchased from Strem; the bulk of the material was stored under nitrogen in a Vacuum Atmosphere glovebox. Small portions (2-3 g) were removed from the glovebox in glass vials, stored in the air in desiccator filled with anhydrous calcium sulfate and weighted in the air. Anhydrous acetonitrile was purchased from Aldrich in Sure/Seal<sup>®</sup> bottles and dispensed by syringe. All other reagents were commercially available and used without further purification. Flash column chromatography was performed with EM Science silica gel 60 (230-400 mesh).

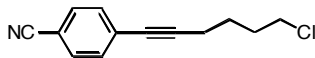
**Techniques:** The reported reactions are moderately sensitive to oxygen. Nevertheless, small amounts of oxygen are tolerated and neither glove-box techniques nor purification of the commercially available reagents are required. Standard argon/vacuum line techniques were used for all manipulations. All reactions were performed in Schlenk tubes dried in an oven at 140 °C overnight, equipped with a 10.3 mm Teflon-coated stirring bar and a Teflon valve. They were evacuated and backfilled with argon (this sequence was carried out two times). The solid reagents were weighed out in the air by adding them directly to the Schlenk tube with the Teflon valve removed after an evacuation/backfilling sequence and under the positive pressure of argon. All liquid reagents were added to the Schlenk tube via syringe. The Schlenk tube was sealed and heated in a preheated oil bath for the specified time and at the specified bath temperature.

**Analytical methods:** IR spectra were recorded on a Perkin-Elmer FT-IR 2000 instrument for all previously unknown compounds. Elemental analyses were performed by Atlantic Microlabs,

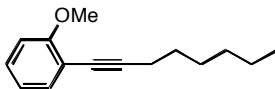
Inc., Norcross, GA.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker 400 MHz instrument with chemical shifts reported relative to residual deuterated solvent peaks or tetramethylsilane internal standard. Gas chromatographic analysis was performed on an Agilent 6890 instrument with a FID detector and an Agilent 10 mX0.10  $\mu\text{m}$  i.d. HP-1 capillary column. Mass spectra (GC/MS) were recorded on a Hewlett Packard model GCD. All yields reported in Tables 1-2 refer to isolated yields (average of two runs) of compounds estimated to be >95% pure as determined by  $^1\text{H}$  NMR. The procedures described in this section are representative and thus the yields for the individual reactions may differ slightly from the average yields reported in Table 1. Compounds described in the literature were characterized by comparing their melting points,  $^1\text{H}$ , and/or  $^{13}\text{C}$  NMR spectra to the previously reported data. Most new compounds were further characterized by elemental analysis (except for **14-17** and **23** — for them, a copy of their  $^1\text{H}$  NMR spectrum is included).

**General procedure for copper-free cross-coupling of aryl chlorides with alkynes.** An oven-dried Schlenk tube was evacuated and backfilled with argon two times and then charged under the positive pressure of argon with  $\text{PdCl}_2(\text{CH}_3\text{CN})_2$  (1.2 mg, 4.62  $\mu\text{mol}$ , 1 mol%), **1** (6.6 mg,  $1.38 \times 10^{-2}$  mmol, 3 mol%),  $\text{Cs}_2\text{CO}_3$  (391 mg, 1.20 mmol) followed by anhydrous acetonitrile (924  $\mu\text{L}$ ) and aryl chloride (0.462 mmol). The slightly yellow suspension was stirred for 5-10 min. Then the alkyne (0.6 mmol) was injected and the Schlenk tube was sealed with a Teflon valve. The reaction mixture was stirred at the desired temperature for the indicated period of time. The resulting suspension was allowed to reach room temperature, diluted with water (3 mL) and extracted with diethyl ether (4  $\times$  4 mL). The combined organic layers were dried over  $\text{MgSO}_4$ , concentrated, and the residue was purified by flash chromatography on silica gel to provide the desired product.

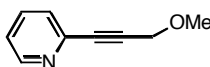
Compounds which were prepared using this procedure:



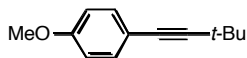
**4-(5-chloro-pent-1-ynyl)-benzonitrile (2)** (CAS No. 326474-52-6)<sup>2</sup> was obtained in 84% as a yellow oil yield after the flash chromatography on silica using hexane  $\square$  15% ether/hexane mixture as eluent.



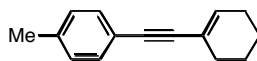
**1-methoxy-2-oct-1-ynyl-benzene (3)** (CAS No. 92991-35-0)<sup>3</sup> was obtained in 96% yield as a colorless liquid after the flash chromatography on silica using hexane  $\square$  5% ether/hexane mixture as eluent.



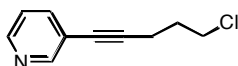
**2-(3-methoxy-prop-1-ynyl)-pyridine (4)** (CAS No. 113985-43-6)<sup>4</sup> was obtained in 78% yield as a colorless liquid after the flash chromatography on silica using 15%  $\square$  50% ether/hexane mixture as eluent. <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\square$  150.39, 143.21, 136.53, 127.51, 123.42, 86.08, 85.44, 60.57, 58.24.



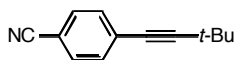
**1-(3,3-dimethyl-but-1-ynyl)-4-methoxybenzene (5)** (CAS No. 66582-09-0)<sup>5</sup> was obtained in 87% yield as a white solid (m.p. 36 °C, lit.<sup>6</sup> 39) after the flash chromatography on silica using hexane  $\square$  7% ether/hexane mixture as eluent.



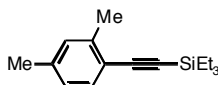
**1-cyclohex-1-enylethynyl-4-methylbenzene (6)** (CAS No. 66582-09-0)<sup>7</sup> was obtained in 81% yield as a colorless oil after the flash chromatography on silica using pentane as eluent. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\square$  7.34 (d, J=8 Hz, 2H), 7.12 (d, J=8 Hz, 2H), 6.20-6.23 (m, 1H), 2.36 (s, 3H), 2.14-2.26 (m, 4H), 1.62-1.72 (m, 4H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\square$  138.84, 135.88, 132.38, 130.01, 121.76, 121.58, 91.32, 87.62, 29.68, 26.13, 22.75, 21.89, 21.81.



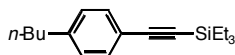
**3-(5-chloro-pent-1-ynyl)-pyridine (7)** was obtained as colorless liquid in 84% yield after the flash chromatography on silica using 15%  $\square$  50% ether/hexane mixture as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  8.64 (d,  $J=2$  Hz, 1H), 8.51 (dd,  $J_1=5$  Hz,  $J_2=2$  Hz, 1H), 7.69 (dt,  $J_1=8$  Hz,  $J_2=2$  Hz, 1H), 7.24 (dd,  $J_1=8$  Hz,  $J_2=4$  Hz, 1H), 3.75 (t,  $J=6$  Hz, 2H), 2.65 (t,  $J=6$  Hz, 2H), 2.09 (p,  $J=7$  Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  152.77, 148.60, 138.85, 123.29, 121.09, 96.52, 92.12, 43.96, 31.64, 17.29. IR (neat): 3030, 2960, 2232, 1561, 1476, 1407, 1291, 1186, 1023, 805, 706.  $\text{C}_{10}\text{H}_{10}\text{ClN}$  (179.65) calcd C 66.86, H 5.61, found C 66.68, H 5.63.



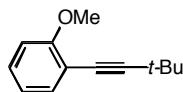
**4-(3,3-dimethyl-but-1-ynyl)-benzonitrile (8)** was obtained as white solid (m.p. 83-84  $^\circ\text{C}$ ) in 92% yield after the flash chromatography on silica using hexane  $\square$  7% ether/hexane mixture as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  8.57 (d,  $J=8$  Hz, 2H), 7.46 (d,  $J=8$  Hz, 2H), 1.34 (s, 9H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  132.50, 132.29, 129.55, 119.07, 111.11, 103.91, 78.39, 31.15, 28.52. IR (neat): 3072, 2974, 2867, 2229, 1921, 1604, 1361, 1292, 1104, 805, 756.  $\text{C}_{13}\text{H}_{13}\text{N}$  (183.25) calcd C 85.21, H 7.15, found C 85.00, H 7.18.



**(2,4-dimethyl-phenylethynyl)-triethylsilane (9)** was obtained as yellowish oil in 86% yield after the flash chromatography on silica using hexane as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  7.30 (s, 1H), 7.07 (dd,  $J_1=24$  Hz,  $J_2=7$  Hz, 2H), 2.43 (s, 3H), 2.30 (s, 3H), 1.08 (t,  $J=8$  Hz, 9H), 0.70 (q,  $J=8$  Hz, 6H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  137.87, 135.27, 133.90, 133.09, 129.65, 129.62, 123.31, 105.82, 95.49, 21.08, 20.62, 7.94, 4.95. IR (neat): 3004, 2954, 2875, 2358, 2147, 1497, 1456, 1120, 770.  $\text{C}_{16}\text{H}_{24}\text{Si}$  (244.16) calcd C 78.61, H 9.90, found C 78.52, H 9.89.

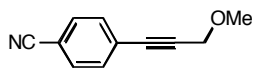


**(4-butyl-phenylethynyl)-triethylsilane (10)** was obtained as yellow oil in 82% yield after the flash chromatography on silica using pentane as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  7.41 (d,  $J=8$  Hz, 2H), 7.12 (d,  $J=8$  Hz, 2H), 2.61 (t,  $J=8$  Hz, 2H), 1.57-1.61 (m, 2H), 1.32-1.38 (m, 2H), 1.06 (t,  $J=8$  Hz, 9H), 0.93 (t,  $J=7$  Hz, 3H), 0.69 (q,  $J=8$  Hz, 6H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  143.91, 132.35, 128.69, 120.88, 107.06, 91.06, 35.97, 33.78, 22.65, 14.29, 7.90, 4.89. IR (neat): 3027, 2956, 2874, 2155, 1506, 1458, 1237, 1018, 736.  $\text{C}_{16}\text{H}_{24}\text{Si}$  (272.50) calcd C 79.34, H 10.34, found C 79.11, H 10.53.

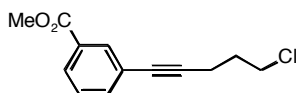


**1-(3,3-dimethyl-but-1-ynyl)-2-methoxybenzene (11)** was obtained in 93% yield as colorless oil after the flash chromatography on silica using hexane  $\square$  5% ether/hexane mixture as eluent.  $^1\text{H}$

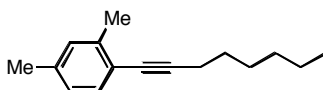
NMR (CDCl<sub>3</sub>):  $\delta$  7.35 (dd,  $J_1=6$  Hz,  $J_2=1$  Hz, 1H), 7.19-7.23 (m, 1H), 6.82-6.88 (m, 2H), 3.85 (s, 3H), 1.34 (s, 9H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  160.21, 134.03, 129.16, 120.73, 113.67, 111.20, 103.17, 75.41, 56.27, 31.51, 28.61. IR (neat): 3074, 2968, 2237, 1596, 1493, 1361, 1274, 1115, 1027, 751. C<sub>13</sub>H<sub>16</sub>O (188.27) calcd C 82.94, H 8.57, found C 82.73, H 8.69.



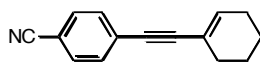
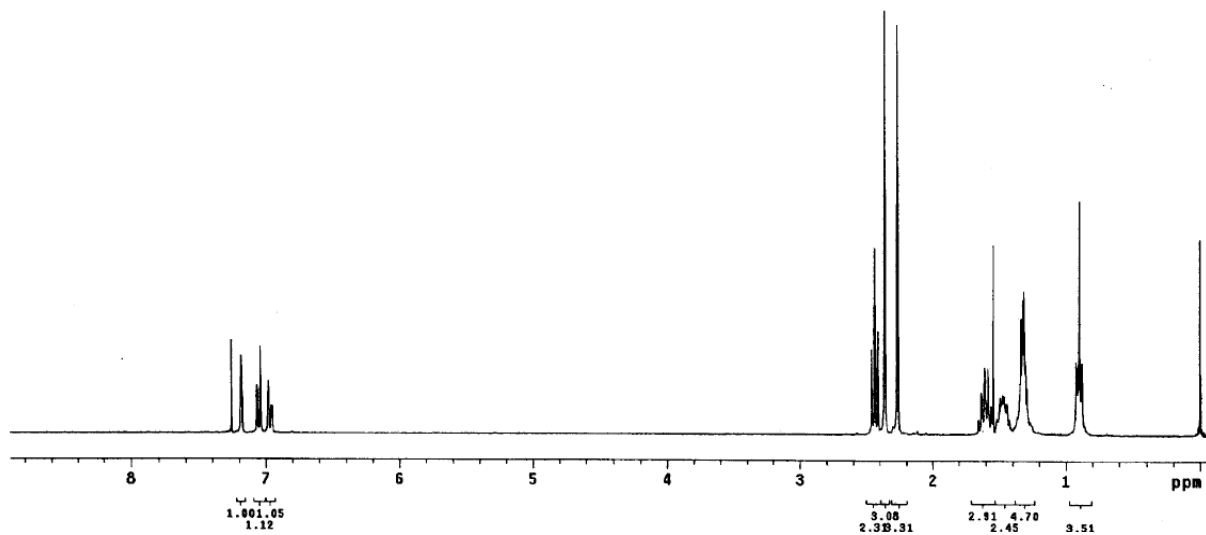
**4-(3-methoxy-prop-1-ynyl)-benzonitrile (12)** was obtained in 94% yield as white powder (m.p. 72-73 °C) after the flash chromatography on silica using hexane/15% ether/hexane mixture as eluent. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  7.63 (d,  $J=8$  Hz, 2H), 7.54 (d,  $J=8$  Hz, 2H), 4.35 (s, 2H), 3.48 (s, 3H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  132.62, 132.47, 127.73, 118.73, 112.25, 90.00, 85.08, 60.63, 58.30. IR (neat): 2948, 2228, 1658, 1450, 1021, 838. C<sub>11</sub>H<sub>9</sub>NO (171.20) calcd C 77.17, H 5.30, found C 76.96, H 5.24.



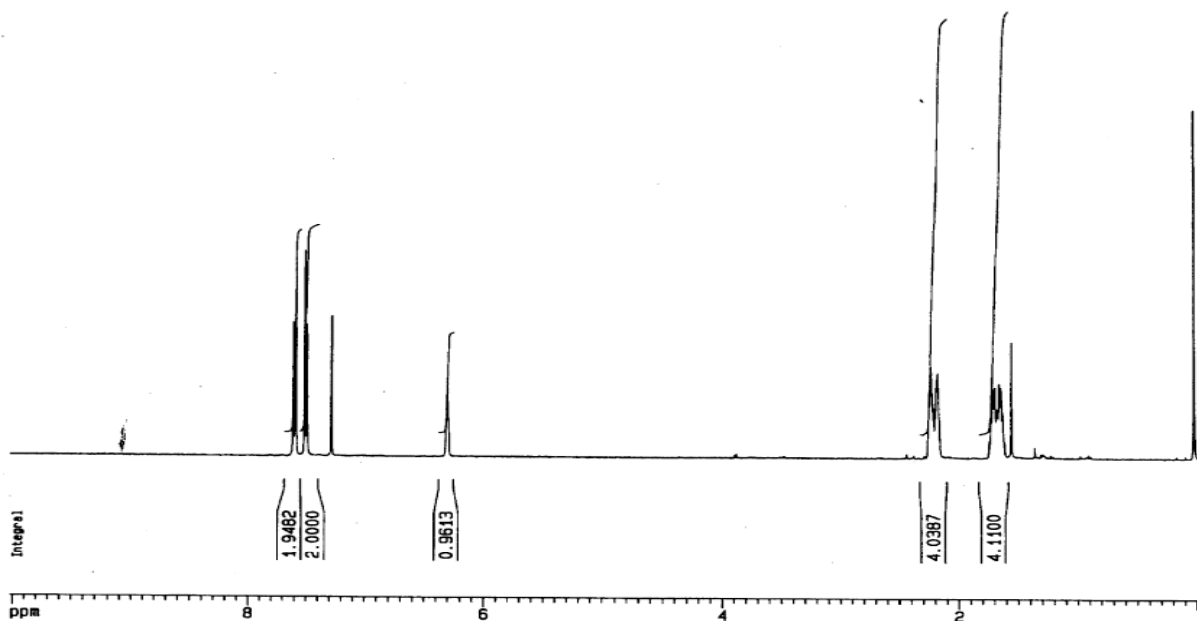
**3-(5-chloro-pent-1-ynyl)-benzoic acid methyl ester (13)** was obtained in 92% yield as colorless oil after the flash chromatography on silica using hexane/15% ether/hexane mixture as eluent. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  8.09 (t,  $J=2$  Hz, 1H), 7.97 (dt,  $J_1=6$  Hz,  $J_2=1$  Hz, 1H), 7.58 (dt,  $J_1=6$  Hz,  $J_2=1$  Hz, 1H), 7.39 (t,  $J=6$  Hz, 1H), 3.94 (s, 3H), 3.73 (t,  $J=4$  Hz, 2H), 2.64 (t,  $J=6$  Hz, 2H), 2.09 (p,  $J=3$  Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  166.87, 136.13, 133.14, 130.74, 129.19, 128.76, 124.43, 89.57, 81.00, 52.62, 44.06, 31.75, 17.23. IR (neat): 3068, 2952, 2229, 1725, 1600, 1438, 1298, 1107, 754. C<sub>13</sub>H<sub>13</sub>ClO<sub>2</sub> (236.69) calcd C 65.97, H 5.54, found C 66.02, H 5.47.

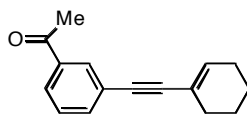


**2,4-dimethyl-1-oct-1-ynyl-benzene (14)** was obtained in 87% yield as colorless oil after the flash chromatography on silica using pentane as eluent. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  7.21 (s, 1H), 7.08 (d,  $J=8$  Hz, 1H), 6.99 (d,  $J=8$  Hz, 1H), 2.46 (t,  $J=7$  Hz, 2H), 2.39 (s, 3H), 2.29 (s, 3H), 1.59-1.65 (m, 2H), 1.48-1.53 (m, 2H), 1.32-1.37 (m, 4H), 0.93 (t,  $J=5$  Hz, 3H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  137.19, 135.22, 132.69, 129.65, 128.70, 123.9, 94.41, 79.96, 31.79, 29.31, 28.99, 23.01, 21.14, 20.64, 19.95, 14.49. IR (neat): 3019, 2929, 2226, 1610, 1497, 1039, 808. HIMS (EI) Calcd for [M<sup>+</sup>] 214.1716, found 214.1716.

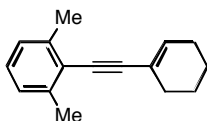
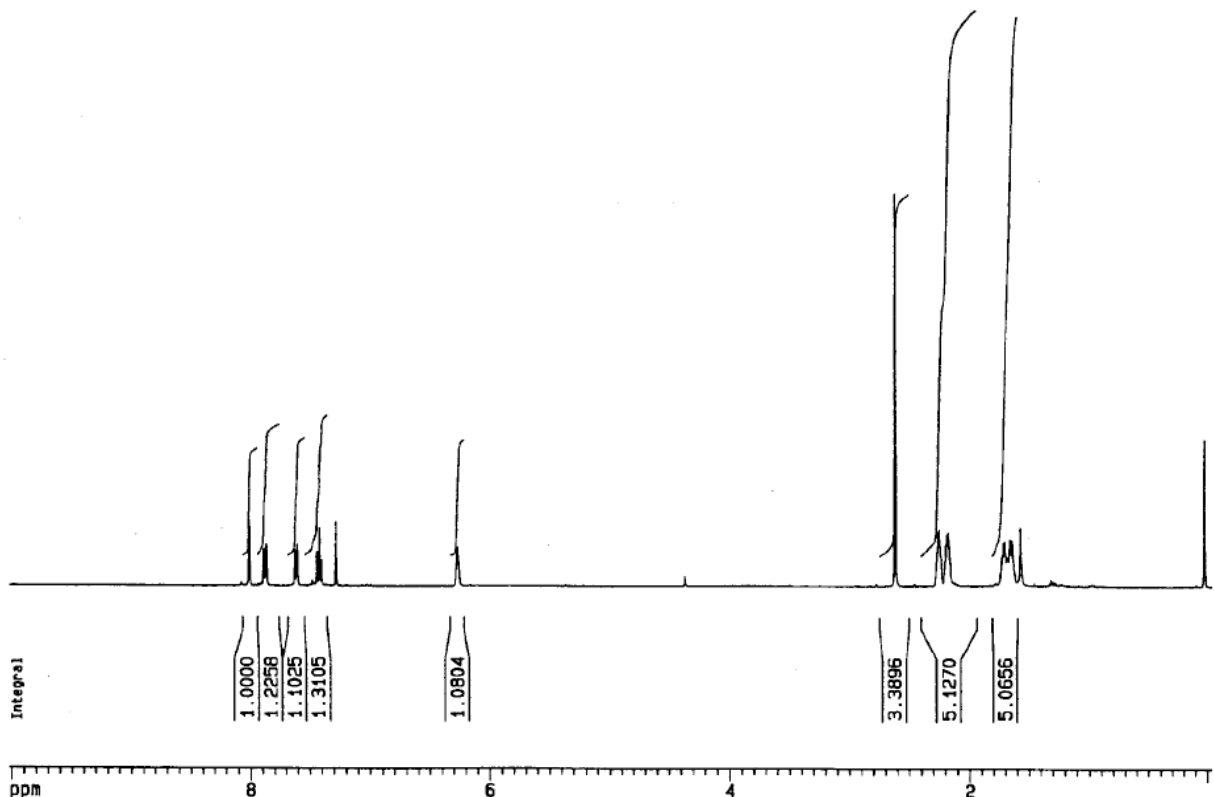


**4-cyclohex-1-enylethynyl-benzonitrile (15)** was obtained in 92% yield as yellow semisolid after the flash chromatography on silica using hexane  $\square$  7% ether/hexane mixture as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  7.57 (d,  $J=7$  Hz, 2H), 7.47(d,  $J=7$  Hz, 2H), 6.27-6.29 (m, 1H), 2.14-2.23 (m, 4H), 1.59-1.72 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  137.60, 132.31, 132.25, 129.22, 120.64, 119.01, 111.24, 96.33, 85.81, 29.33, 26.25, 22.60, 21.77. IR (neat): 3068, 2933, 2360, 2224, 2198, 1599, 1498, 838, 754. HIMS (ESI) Calcd for  $[\text{M}+\text{Na}]^+$  230.0940, found 230.0944.





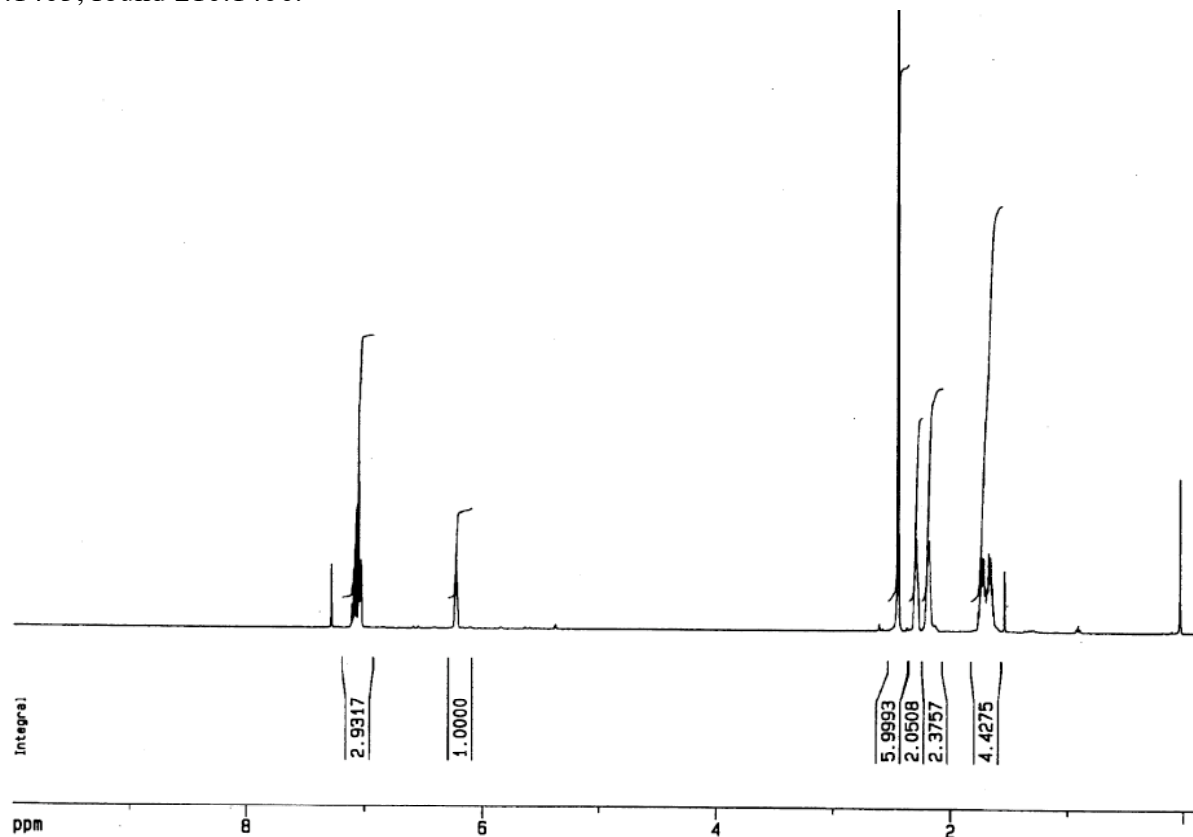
**1-(3-cyclohex-1-enylethynyl-phenyl)-ethanone (16)** was obtained in 94% yield as yellow oil after the flash chromatography on silica using hexane/25% ether/hexane mixture as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  8.01 (s, 1H), 7.88 (d,  $J=8$  Hz, 1H), 7.61 (d,  $J=7$  Hz, 1H), 7.42 (t,  $J=8$  Hz, 1H), 6.25-6.27 (m, 1H), 2.62 (s, 3H), 2.18-2.26 (m, 4H), 1.63-1.73 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  192.33, 137.53, 136.36, 136.03, 131.85, 128.96, 127.66, 124.85, 120.87, 92.72, 86.18, 29.52, 27.03, 26.20, 22.70, 21.87. IR (neat): 3064, 2934, 2205, 1687, 1594, 1423, 1233, 799, 686. HIMS (ESI) Calcd for  $[\text{M}+\text{Na}]^+$  247.1093, found 247.1101.



**2-cyclohex-1-enylethynyl-1,3-dimethylbenzene (17)** was obtained in 91% yield as colorless oil after the flash chromatography on silica using hexane as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  6.98-7.11 (m, 3H), 6.22-6.24 (m, 1H), 2.45 (s, 6H), 2.14-2.30 (m, 4H), 1.58-1.76 (m, 4H).  $^{13}\text{C}$  NMR



(CDCl<sub>3</sub>):  $\delta$  140.28, 134.61, 127.58, 126.94, 123.85, 121.64, 100.32, 84.88, 29.89, 26.16, 22.80, 21.99, 21.46. IR (neat): 3063, 2932, 2194, 1466, 1376, 918, 768. HIMS (EI) Calcd for [M<sup>+</sup>] 210.1403, found 210.1406.

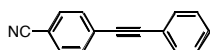


### General procedure for copper-free cross-coupling of aryl chlorides with phenyl acetylene.

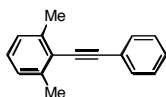
An oven-dried two-necked flask, equipped with reflux condenser, gas inlet/outlet and rubber stopper, was evacuated and backfilled with argon two times and then charged under the positive pressure of argon with PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (5.9 mg, 22.7  $\mu$ mol, 1 mol%), **1** (32.5 mg, 68.2  $\mu$ mol, 3 mol%), Cs<sub>2</sub>CO<sub>3</sub> (1.63 g, 4.99 mmol) followed by anhydrous acetonitrile (for **19**) or propionitrile (for **20-21**) (4.5 mL) and aryl chloride (2.27 mmol). The slightly yellow suspension was stirred for 25 min at room temperature. Then the reaction mixture was heated to the reflux temperature and the alkyne (0.6 mmol) was injected slowly over the course of reaction (2 hours) by means of ORION M2621 syringe pump. The reaction mixture was stirred for additional 30 min after the addition was complete. The resulting suspension was allowed to reach room temperature, diluted with water (3 mL) and extracted with diethyl ether (4  $\times$  4 mL). The combined organic layers

were dried over  $\text{MgSO}_4$ , concentrated, and the residue was purified by flash chromatography on silica gel to provide the desired product.

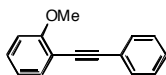
Compounds which were prepared using this procedure:



**4-cyanodiphenylacetylene (18)** (CAS No. 29822-79-5)<sup>8</sup> was obtained in 93% yield as a white solid (m.p. 49-51 °C; lit.<sup>9</sup> 108-109) after flash chromatography on silica using hexane  $\square$  10% ethyl acetate/hexane mixture as eluent.



**1,6-dimethyldiphenylacetylene (19)** (CAS No. 76339-11)<sup>10</sup> was obtained in 88% yield as a colorless oil after flash chromatography on silica using hexane as eluent.

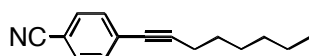


**2-methoxydiphenylacetylene (20)** (CAS No. 41398-67-8)<sup>11</sup> was obtained in 95% yield as a yellow oil after flash chromatography on silica using hexane  $\square$  5% ethyl acetate /hexane mixture as eluent.

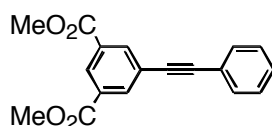
**General procedure for coupling of aryl tosylates with terminal acetylenes.** An oven-dried two-necked flask, equipped with reflux condenser, gas inlet/outlet and rubber stopper, was evacuated and backfilled with argon two times and then charged under the positive pressure of argon with  $\text{PdCl}_2(\text{CH}_3\text{CN})_2$  (7.7 mg, 29.6  $\square$ mol, 5 mol%), **1** (42.4 mg, 89  $\square$ mol, 15 mol%),  $\text{Cs}_2\text{CO}_3$  (0.87 g, 2.66 mmol) followed by propionitrile (1.8 mL) and aryl tosylate (0.59 mmol). The slightly yellow suspension was stirred for 25 min at room temperature. Then the reaction mixture was heated to the reflux temperature and the alkyne (0.88 mmol diluted with 1 mL of

propionitrile) was injected slowly over the course of reaction (8 hours). The reaction mixture was stirred for additional 2 hours after the addition was complete. The resulting suspension was allowed to reach room temperature, diluted with water (3 mL) and extracted with diethyl ether (4  $\square$  4 mL). The combined organic layers were dried over  $\text{MgSO}_4$ , concentrated, and the residue was purified by flash chromatography on silica gel to provide the desired product.

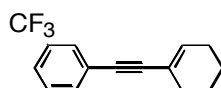
Compound which were prepared using this procedure:



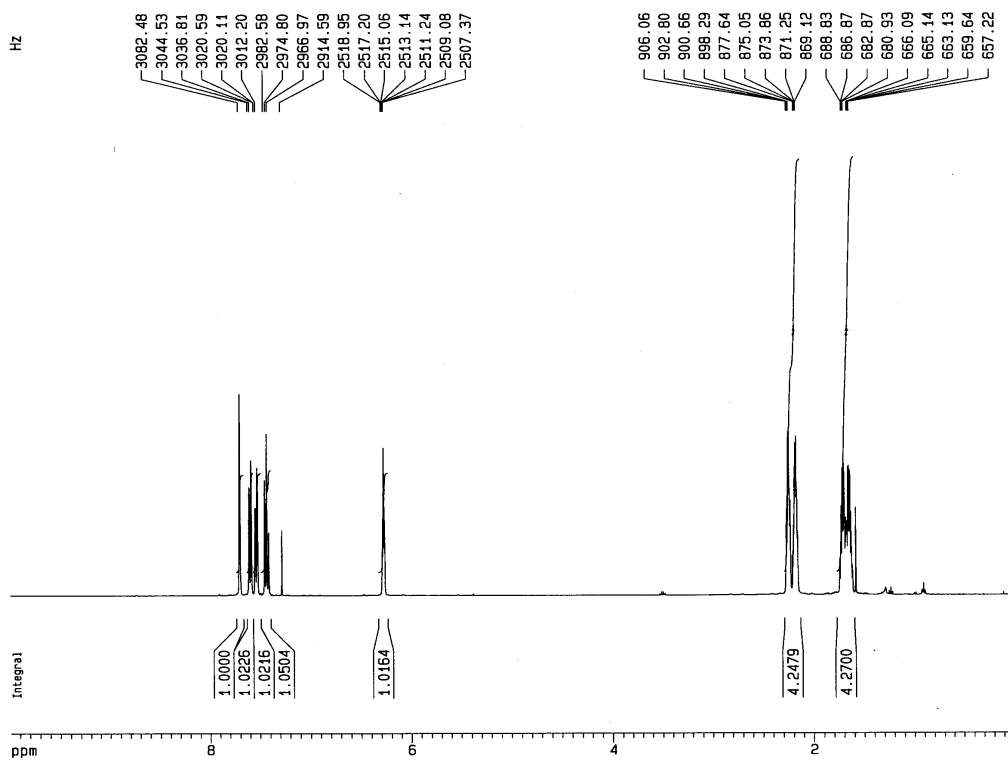
**4-oct-1-ynyl-benzonitrile (21)** (CAS No. 312708-98-8)<sup>12</sup> was obtained in 73% yield as a yellowish liquid after the flash chromatography on silica using hexane  $\square$  15% ether/hexane mixture as eluent.



**5-Phenylethynyl-isophthalic acid dimethyl ester (22)**<sup>13</sup> was obtained in 62% yield as a white solid (mp 111-113  $^{\circ}\text{C}$ , lit.<sup>13</sup> 115-116  $^{\circ}\text{C}$ ) after the flash chromatography on silica using 10% ethyl acetate/hexane mixture as eluent.



**1-Cyclohex-2-enylethynyl-3-trifluoromethyl-benzene (23)** was obtained in 78% yield as a yellowish liquid after the flash chromatography on silica using hexane as eluent.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\square$  7.70 (s, 1H), 7.6 (d,  $J=8\text{Hz}$ , 1H), 7.54 (d,  $J=8\text{ Hz}$ , 1H), 7.43 (t,  $J=8\text{Hz}$ , 1H), 6.26-6.29 (m, 1H), 2.15-2.27 (m, 4H), 1.58-1.74 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\square$  136.70, 134.85, 131.16 (d  $J=32\text{ Hz}$ ), 129.12, 125.57 (m), 125.14, 124.62 (m), 122.83, 120.74, 93.25, 85.71, 29.45, 26.20, 22.66, 21.84. IR (neat): 3063, 2932, 2194, 1466, 1376, 918, 768.



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