Supporting Information

Decarboxylative/Sonogashira-type Cross-Coupling using PdCl₂(Cy*Phine)₂

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1. **General Considerations**

Unless otherwise noted, all reagents were purchased commercially from Strem Chemicals, Sigma-Aldrich, or Alfa Aesar and used as received without further purification. All operations were carried out in an argon atmosphere using glovebox and Schlenk techniques unless otherwise specified. Anhydrous tetrahydrofuran (THF) and toluene were obtained from an argon purged solvent purification system comprised of columns of activated alumina and molecular sieves. Anhydrous \( N,N' \)-dimethylformamide (DMF), acetonitrile (CH\(_3\)CN), dimethyl sulfoxide (DMSO) and 1,4-dioxane were purchased from Sigma-Aldrich as sure-sealed solvents and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) on 0.25 mm E. Merck silica gel plates (60F-254) using UV light as the visualizing agent. E. Column chromatography was carried out on silica gel (200-300 mesh) by elution with appropriate solvents. Yields refer to chromatographically and spectroscopically (\(^1\)H NMR) homogeneous materials, unless otherwise stated. Gas chromatography analysis was performed on an Agilent HP-7890 instrument with a flame ionization detector (FID) and an HP-5MS capillary column (30 m, 0.25 mm i.d., 0.25 μm film thicknesses) using helium as the carrier gas. Gas chromatography-mass spectrometry analysis was carried out on an Agilent HP-7890 instrument with an Agilent HP-5975 with triple-axis detector and HP-5MS capillary column using helium carrier gas. NMR spectra were from a Bruker DRX-600, instrument and calibrated using residual non-deuterated solvent (CDCl\(_3\): \( \delta_H = 7.26 \) ppm, \( \delta_C = 77.16 \) ppm; C\(_6\)D\(_6\): \( \delta_H = 7.16 \) ppm, \( \delta_C = 128.06 \) ppm) as an internal reference. Infrared (IR) spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer. High resolution mass spectra (HRMS) were recorded on an Agilent 6210 Series 1969A ESI-TOF (time of flight) mass spectrometer using ESI (electrospray ionization). PdCl\(_2\)(Cy*Phine)\(_2\) was prepared according to previously reported method.\(^1\)

2. **General procedure for decarboxylative cross-coupling (Method 1)**

To a sealable reaction tube equipped with a magnetic stir bar was charged with PdCl\(_2\)(Cy*Phine)\(_2\) (6.4 mg, 1 mol%), Cs\(_2\)CO\(_3\) (391 mg, 1.2 mmol), organic chloride (0.5 mmol), alkynyl carboxylic acid (0.6 mmol) and THF (2 mL). The tube was then crimp-sealed with a cap fitted with a Teflon-lined septum and heated to 80 °C for given hours with vigorous stirring. The mixture was cooled to room temperature, diluted with EtOAc and filtered through a pad of Celite. The filtrate was
concentrated in vacuo affording the crude product which was purified by flash chromatography on silica gel.

3. General procedure of tandem Sonogashira/decarboxylative cross-coupling for synthesis of symmetric di(heteroaryl)alkynes (Method 2)

To a sealable reaction tube equipped with a magnetic stir bar was charged with PdCl₂(Cy*Phine)₂ (25.7 mg, 5 mol%), Cs₂CO₃ (391 mg, 1.2 mmol), heteroaryl chloride (1.0 mmol), propiolic acid (0.4 mmol) and 1,4-dioxane (2 mL). The tube was then crimp-sealed with a cap fitted with a Teflon-lined septum and heated to 120 °C for 24 h with vigorous stirring. The mixture was cooled to room temperature, diluted with EtOAc and filtered through a pad of Celite. The filtrate was concentrated in vacuo affording the crude product which was purified by flash chromatography on silica gel.

4. Characterization data for products

1-methoxy-2-(phenylethynyl)benzene (3a). Following method 1, 71.5 mg (0.5 mmol) of 2-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3a as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. ¹H NMR (600 MHz, CDCl₃) δ = 7.60 (dd, J = 8.0, 1.6 Hz, 2 H), 7.54 (dd, J = 7.5, 1.7 Hz, 1 H), 7.40–7.34 (m, 4 H), 7.01–6.90 (m, 2 H), 3.95 (s, 3 H) ppm. ¹³C NMR (151 MHz, CDCl₃) δ = 159.9, 133.6, 131.7, 129.8, 128.8, 128.4, 128.3, 128.2, 126.3, 123.5, 120.5, 112.4, 110.7, 93.5, 85.7, 55.9 ppm. The physical data were in full accordance with the literature value.

1-methoxy-3-(phenylethynyl)benzene (3b). Following method 1, 71.5 mg (0.5 mmol) of 3-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3b as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. ¹H NMR (600 MHz, CDCl₃) δ = 7.62–7.55 (m, 2 H), 7.40–7.37 (m, 3 H), 7.30 (dd, J = 8.4, 7.5 Hz, 1 H), 7.19 (d, J = 7.5 Hz, 1 H), 7.12 (dd, J = 2.7, 1.4 Hz, 1 H), 6.94 (ddd, J = 8.3, 2.7, 1.0 Hz, 1 H), 3.86 (s, 3 H) ppm. ¹³C NMR (151 MHz,
S5

1-methoxy-4-(phenylethynyl)benzene (3c). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3c as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.52$ (dd, $J = 8.1$, 1.5 Hz, 2 H), 7.48 (d, $J = 8.9$ Hz, 2 H), 7.40–7.27 (m, 3 H), 6.89 (d, $J = 8.9$ Hz, 2 H), 3.83 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 160.2$, 133.6, 132.0, 129.8, 129.0, 128.9, 128.5, 124.2, 115.9, 89.9, 88.6, 55.9 ppm. The physical data were in full accordance with the literature value.\(^1\)

![1-methoxy-4-(phenylethynyl)benzene](image)

1-methyl-2-(phenylethynyl)benzene (3d). Following method 1, 63 mg (0.5 mmol) of 1-chloro-2-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3d as a colourless oil (95 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.64$–7.59 (m, 2 H), 7.57 (s, 1 H), 7.40 (dd, $J = 9.2$, 7.1 Hz, 3 H), 7.31–7.27 (m, 2 H), 7.24 (dd, $J = 7.9$, 4.2 Hz, 1 H), 2.59 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 140.2$, 131.9, 131.6, 129.5, 128.40, 128.36, 128.2, 125.6, 123.6, 123.1, 93.4, 88.4, 20.8 ppm. The physical data were in full accordance with the literature value.\(^1\)

![1-methyl-2-(phenylethynyl)benzene](image)

1-methyl-3-(phenylethynyl)benzene (3e). Following method 1, 63 mg (0.5 mmol) of 1-chloro-3-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3e as a yellow oil (95 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.63$–7.57 (m, 2 H), 7.45–7.35 (m, 5 H), 7.30 (td, $J = 7.6$, 1.6 Hz, 1 H), 7.20 (d, $J = 7.7$ Hz, 1 H), 2.41 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 138.1$, 132.2, 131.6, 129.2, 128.7, 128.5, 128.4, 128.3, 128.2, 123.4, 123.1, 89.7, 89.1, 21.3 ppm. The physical data were in full accordance with the literature value.\(^1\)

![1-methyl-3-(phenylethynyl)benzene](image)

1-methyl-4-(phenylethynyl)benzene (3f). Following method 1, 63 mg (0.5 mmol) of 1-chloro-4-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3f as a yellow solid (95
mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.58$–7.53 (m, 2 H), 7.47 (dd, $J = 8.1$, 1.6 Hz, 2 H), 7.39–7.32 (m, 3 H), 7.19 (d, $J = 7.8$ Hz, 2 H), 2.40 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 138.4$, 131.6, 131.5, 129.1, 128.3, 128.1, 123.5, 120.2, 89.6, 88.7, 21.5 ppm. The physical data were in full accordance with the literature value.$^2$

![Image](73x629 to 170x656)

4-(phenylethynyl)benzaldehyde (3g). Following method 1, 70 mg (0.5 mmol) of 4-chlorobenzaldehyde and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3g as a yellow solid (98 mg, 95%) using 6:94 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 10.04$ (s, 1 H), 7.89 (d, $J = 8.4$ Hz, 2 H), 7.70 (d, $J = 8.2$ Hz, 2 H), 7.61–7.50 (m, 2 H), 7.44–7.35 (m, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 192.18$, 192.17, 136.2, 132.9, 132.5, 130.4, 130.3, 129.7, 129.2, 123.2, 94.2, 89.3 ppm. The physical data were in full accordance with the literature value.$^1$

![Image](70x453 to 174x481)

1-(4-(phenylethynyl)phenyl)ethan-1-one (3h). Following method 1, 77 mg (0.5 mmol) of 1-(4-chlorophenyl)ethan-1-one and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3h as a yellow solid (100 mg, 91%) using 1:19 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.98$–7.93 (m, 2 H), 7.62 (d, $J = 8.3$ Hz, 2 H), 7.57 (dd, $J = 6.7$, 3.0 Hz, 2 H), 7.40–7.34 (m, 3 H), 2.62 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 197.3$, 136.2, 131.8, 131.7, 128.8, 128.5, 128.3, 128.2, 122.7, 92.7, 88.6, 26.6 ppm. The physical data were in full accordance with the literature value.$^2$

![Image](70x284 to 168x306)

4-(phenylethynyl)benzonitrile (3i). Following method 1, 68.5 mg (0.5 mmol) of 4-chlorobenzonitrile and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3i as a yellow solid (95 mg, 94%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.66$ (d, $J = 8.6$ Hz, 2 H), 7.63 (d, $J = 8.6$ Hz, 2 H), 7.58–7.54 (m, 2 H), 7.40 (dd, $J = 5.2$, 2.0 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 132.08$, 132.06, 131.8, 129.1, 128.5, 128.3, 122.2, 118.6, 111.5, 93.8, 87.7 ppm. The physical data were in full accordance with the literature value.$^2$

![Image](70x226 to 168x248)
1,3-dimethyl-2-(phenylethynyl)benzene (3j). Following method 1, 70 mg (0.5 mmol) of 2-chloro-1,3-dimethylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3j as a colourless oil (100 mg, 97%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.59 (dd, $J$ = 6.4, 1.9 Hz, 2 H), 7.43–7.31 (m, 3 H), 7.19–7.14 (m, 1 H), 7.11 (dd, $J$ = 7.8, 2.3 Hz, 2 H), 2.57 (s, 6 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 140.3, 131.4, 128.4, 128.1, 127.8, 126.7, 123.9, 123.0, 97.9, 87.1, 21.2 ppm. The physical data were in full accordance with the literature value.$^1$

4-(phenylethynyl)phenol (3k). Following method 1, 64.3 mg (0.5 mmol) of 4-chlorophenol and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3k as a yellow solid (80 mg, 82%) using 1:5 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.54 (dd, $J$ = 8.0, 1.6 Hz, 2 H), 7.46 (d, $J$ = 8.8 Hz, 2 H), 7.39–7.32 (m, 3 H), 6.84 (d, $J$ = 8.7 Hz, 2 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 155.8, 133.3, 131.5, 128.3, 128.0, 123.6, 115.6, 115.5, 89.3, 88.0 ppm. The physical data were in full accordance with the literature value.$^2$

4-(phenylethynyl)aniline (3l). Following method 1, 63.8 mg (0.5 mmol) of 4-chloroaniline and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3l as a yellow solid (81 mg, 84%) using 1:5 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.53 (d, $J$ = 6.8 Hz, 2 H), 7.42–7.30 (m, 5 H), 6.67 (d, $J$ = 8.5 Hz, 2 H), 3.84 (s, 2 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 146.6, 133.0, 131.4, 128.3, 127.7, 123.9, 114.8, 112.7, 90.1, 87.3 ppm. The physical data were in full accordance with the literature value.$^2$

2-(phenylethynyl)thiophene (3m). Following method 1, 59.3 mg (0.5 mmol) of 2-chlorothiophene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3m as a white oil (87 mg, 95%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.58–7.50 (m, 2 H), 7.37 (dd, $J$ = 4.7, 3.0 Hz, 3 H), 7.34–7.29 (m, 2 H), 7.04 (dd, $J$ = 5.0, 3.2, 1.9 Hz, 1 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 131.9, 131.4, 128.4, 128.4, 127.3, 127.1, 123.3, 122.9, 93.0, 82.6 ppm. The physical data were in full accordance with the literature value.$^1$
3-(phenylethynyl)pyridine (3n). Following method 1, 56.8 mg (0.5 mmol) of 3-chloropyridine and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3n as a white solid (88 mg, 98%) using 1:4 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 8.79$ (s, 1 H), 8.57 (d, $J = 4.0$ Hz, 1 H), 7.83 (d, $J = 7.9$ Hz, 1 H), 7.61–7.52 (m, 2 H), 7.42–7.37 (m, 3 H), 7.30 (ddd, $J = 7.9$, 4.9, 0.9 Hz, 1 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 152.2$, 148.5, 138.5, 128.8, 128.5, 123.1, 122.5, 92.7, 85.9 ppm. The physical data were in full accordance with the literature value.$^3$

4,6-dimethoxy-2-(phenylethynyl)pyrimidine (3o). Following method 1, 87 mg (0.5 mmol) of 2-chloro-4,6-dimethoxypyrimidine and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3o as a yellow solid (118 mg, 98%) using 1:19 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.72$–7.65 (m, 2 H), 7.44–7.35 (m, 3 H), 6.04 (s, 1 H), 4.01 (s, 6 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 171.1$, 151.2, 132.6, 129.5, 128.4, 121.5, 90.0, 88.1, 86.5, 54.4 ppm. The physical data were in full accordance with the literature value.$^1$

4-(methylthio)-2-(phenylethynyl)pyrimidine (3p). Following method 1, 80 mg (0.5 mmol) of 2-chloro-4-(methylthio)pyrimidine and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3p as a yellow oil (108 mg, 96%) using 1:25 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 8.50$ (s, 1 H), 7.64–7.59 (m, 2 H), 7.45–7.36 (m, 3 H), 7.11 (d, $J = 4.8$ Hz, 1 H), 2.60 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 173.2$, 157.0, 150.9, 132.4, 129.9, 128.5, 121.2, 118.5, 93.8, 86.8, 14.2 ppm. The physical data were in full accordance with the literature value.$^1$

3-methoxy-6-(phenylethynyl)pyridazine (3q). Following method 1, 72.3 mg (0.5 mmol) of 3-chloro-6-methoxypyridazine and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3q as a yellow solid (100 mg, 95%) using 1:4 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 7.63$–7.55 (m, 2 H), 7.50 (d, $J = 9.1$ Hz, 1 H), 7.36 (dd, $J = 5.3$, 2.0 Hz, 3 H),
6.93 (d, J = 9.1 Hz, 1 H), 4.15 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 163.4, 143.7, 132.3, 132.0, 129.2, 128.4, 121.9, 116.6, 92.1, 85.7, 55.1 ppm. The physical data were in full accordance with the literature value.$^4$

6-(phenylethynyl)imidazo[1,2-b]pyridazine (3r). Following method 1, 76.8 mg (0.5 mmol) of 6-chloroimidazo[1,2-b]pyridazine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3r as a yellow solid (108 mg, 98%) using 2:1 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.99 (d, J = 9.8 Hz, 2 H), 7.83 (s, 1 H), 7.63 (d, J = 8.1 Hz, 2 H), 7.42 (m, 3 H), 7.25 (d, J = 9.3 Hz, 1 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 138.7, 137.8, 134.6, 132.1, 129.7, 128.5, 125.2, 121.2, 120.6, 117.0, 92.4, 84.8 ppm. The physical data were in full accordance with the literature value.$^3$

1-methyl-3-(phenylethynyl)-1H-pyrrolo[2,3-b]pyridine (3s). Following method 1, 83 mg (0.5 mmol) of 3-chloro-1-methyl-1H-pyrrolo[2,3-b]pyridine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3s as a dark yellow solid (105 mg, 90%) using 1:4 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 8.38 (dd, J = 4.7, 1.6 Hz, 1 H), 8.08 (dd, J = 7.8, 1.6 Hz, 1 H), 7.58–7.52 (m, 2 H), 7.43 (s, 1 H), 7.38–7.28 (m, 3 H), 7.14 (dd, J = 7.8, 4.7 Hz, 1 H), 3.88 (s, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 147.3, 144.0, 132.3, 131.3, 128.4, 128.3, 127.8, 123.9, 121.4, 116.5, 95.7, 91.1, 82.3, 31.4 ppm. IR (film) $\nu$ max 2210, 1537, 1455, 1360, 1144, 792, 771, 754, 694 cm$^{-1}$. HRMS (ESI) calcd for C$_{16}$H$_{13}$N$_2$+ (M+H)$^+$, 233.1000, found: 233.1076.

(Z)-pent-1-en-4-yne-1,5-diyl dibenzene (3t). Following method 1, 76.3 mg (0.5 mmol) of (E)-(3-chloroprop-1-en-1-yl)benzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded 3t as a yellow solid (103 mg, 95%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.54 (dd, J = 7.7, 2.0 Hz, 2 H), 7.48–7.44 (m, 2 H), 7.42–7.34 (m, 6 H), 6.79 (d, J = 15.7 Hz, 1 H), 6.32 (dt, J = 15.7, 5.7 Hz, 1 H), 3.43 (dd, J = 5.6, 1.9 Hz, 2 H) ppm. $^{13}$C NMR (151
MHz, CDCl3) δ = 137.2, 131.7, 131.5, 128.6, 128.3, 127.9, 127.4, 126.3, 124.3, 123.7, 86.8, 83.0, 23.1 ppm. The physical data were in full accordance with the literature value.5

**prop-1-yne-1,3-diyl dibenzene (3u).** Following method 1, 63.3 mg (0.5 mmol) of benzyl chloride and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded the 3u as a colorless oil (93 mg, 97%) using petroleum ether as the column eluent. 1H NMR (600 MHz, CDCl3) δ = 7.55–7.51 (m, 1 H), 7.50–7.46 (m, 3 H), 7.41 (t, J = 7.5 Hz, 2 H), 7.38–7.29 (m, 5 H), 3.90 (s, 2 H) ppm. 13C NMR (151 MHz, CDCl3) δ = 136.8, 131.7, 128.6, 128.3, 128.0, 127.9, 126.7, 123.8, 87.6, 82.8, 25.8 ppm. The physical data were in full accordance with the literature value.6

![Propyne-1,3-diyl dibenzene](image)

**1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (3v).** Following method 1, 78.3 mg (0.5 mmol) of 4-methoxybenzyl chloride and 87.6 mg (0.6 mmol) of phenylpropionic acid afforded 3v as a yellow oil (110 mg, 99%) using petroleum ether as the column eluent. 1H NMR (600 MHz, CDCl3) δ = 7.53–7.42 (m, 2 H), 7.36 (d, J = 8.7 Hz, 2 H), 7.32 (dd, J = 5.1, 2.0 Hz, 3 H), 6.92 (d, J = 8.6 Hz, 2 H), 3.83 (s, 3 H), 3.81 (s, 2 H) ppm. 13C NMR (151 MHz, CDCl3) δ = 158.45, 131.67, 128.97, 128.83, 128.26, 127.80, 123.81, 114.02, 88.06, 82.47, 55.33, 24.93 ppm. The physical data were in full accordance with the literature value.6

![1-Methoxy-4-(3-phenylprop-2-yn-1-yl)benzene](image)

**1-((4-methoxyphenyl)ethynyl)naphthalene (4b).** Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 117.7 mg (0.6 mmol) of 3-(naphthalen-1-yl)propionic acid afforded 4b as a yellow solid (127 mg, 98%) using petroleum ether as the column eluent. 1H NMR (600 MHz, CDCl3) δ = 8.07 (s, 1 H), 7.83 (dd, J = 9.0, 5.4 Hz, 3 H), 7.65–7.42 (m, 5 H), 6.93 (d, J = 8.6 Hz, 2 H), 3.84 (s, 3 H) ppm. 13C NMR (151 MHz, CDCl3) δ = 159.7, 133.2, 133.1, 132.7, 131.2, 128.5, 128.0, 127.80, 127.77, 126.5, 121.0, 115.5, 114.1, 89.9, 88.6, 55.3 ppm. The physical data were in full accordance with the literature value.7

![1-((4-methoxyphenyl)ethynyl)naphthalene](image)
1-(hept-1-yn-1-yl)-4-methoxybenzene (4c). Following general method I, 71.5 mg (0.5 mmol) of 4-chloroanisole and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4c as a yellow oil (100 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.34 (d, $J$ = 8.8 Hz, 2 H), 6.81 (d, $J$ = 8.9 Hz, 2 H), 2.39 (t, $J$ = 7.1 Hz, 3 H), 1.65–1.52 (m, 2 H), 1.49–1.30 (m, 4 H), 0.93 (t, $J$ = 7.1 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 159.0, 132.8, 116.3, 113.8, 88.8, 80.2, 55.2, 31.2, 28.6, 22.3, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.$^8$

1-(but-1-yn-1-yl)-4-methoxybenzene (4d). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 58.9 mg (0.6 mmol) of pent-2-ynoic acid afforded 4d as a yellow oil (80 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.34 (d, $J$ = 8.7 Hz, 2 H), 6.81 (d, $J$ = 8.7 Hz, 2 H), 3.79 (s, 3 H), 2.41 (q, $J$ = 7.5 Hz, 2 H), 1.23 (t, $J$ = 7.5 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 159.0, 132.8, 116.2, 113.8, 90.0, 79.6, 55.2, 14.0, 13.1 ppm. The physical data were in full accordance with the literature value.$^9$

1-(cyclopropylethynyl)-4-methoxybenzene (4e). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 66.1 mg (0.6 mmol) of 3-cyclopropylpropionic acid afforded 4e as a yellow oil (85 mg, 99%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.32 (d, $J$ = 8.8 Hz, 2 H), 6.80 (d, $J$ = 8.9 Hz, 2 H), 3.79 (s, 3 H), 2.41 (q, $J$ = 7.5 Hz, 2 H), 1.43 (tt, $J$ = 8.2, 5.1 Hz, 1 H), 0.88–0.81 (m, 2 H), 0.81–0.73 (m, 2 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 158.9, 132.8, 115.9, 113.6, 91.6, 75.4, 55.1, 8.4 ppm. The physical data were in full accordance with the literature value.$^{10}$

4-(hept-1-yn-1-yl)phenol (4f). Following method 1, 64.3 mg (0.5 mmol) of 4-chlorophenol and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4f as a yellow oil (83 mg, 89%) using 1:9 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.20 (d, $J$ = 8.6 Hz, 2 H), 6.67 (dd, $J$ = 8.8, 7.5 Hz, 3 H), 2.29 (s, 2 H), 1.50 (q, $J$ = 7.3 Hz, 2 H), 1.39–1.17 (m, 4 H), 0.84 (t, $J$ = 7.1 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 155.0, 133.1, 129.5, 116.7, 115.4, 88.9, 80.2, 31.2, 28.6, 22.3, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.$^{11}$
4-(hept-1-yn-1-yl)phenol (4g). Following method 1, 63.8 mg (0.5 mmol) of 4-chloroaniline and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4g as a yellow oil (80 mg, 86%) using 1:9 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.20 (d, $J$ = 8.5 Hz, 2 H), 6.57 (d, $J$ = 8.5 Hz, 2 H), 3.71 (s, 2 H), 2.38 (t, $J$ = 7.1 Hz, 2 H), 1.65–1.52 (m, 2 H), 1.48–1.16 (m, 4 H), 0.93 (t, $J$ = 7.2 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 144.9, 131.7, 113.7, 112.7, 86.8, 79.7, 30.1, 27.7, 21.2, 18.4, 13.0 ppm. The physical data were in full accordance with the literature value.$^{12}$

2-(hept-1-yn-1-yl)thiophene (4h). Following method 1, 59.3 mg (0.5 mmol) of 2-chlorothiophene and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4h as a yellow oil (80 mg, 89%) using petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.18–7.15 (m, 1 H), 7.12 (dd, $J$ = 3.6, 1.1 Hz, 1 H), 6.94 (dd, $J$ = 5.2, 3.6 Hz, 1 H), 2.43 (t, $J$ = 7.2 Hz, 2 H), 1.61 (q, $J$ = 7.3 Hz, 2 H), 1.50–1.20 (m, 4 H), 0.94 (t, $J$ = 7.1 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 130.8, 126.7, 125.8, 124.3, 94.6, 73.7, 31.2, 28.3, 22.2, 19.7, 14.0 ppm. The physical data were in full accordance with the literature value.$^{8}$

3-(hept-1-yn-1-yl)pyridine (4i). Following method 1, 56.8 mg (0.5 mmol) of 3-chloropyridine and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4i as a yellow oil (78 mg, 90%) using 1:9 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 8.61 (s, 1 H), 8.50–8.40 (m, 1 H), 7.65 (d, $J$ = 7.9 Hz, 1 H), 7.19 (dd, $J$ = 7.9, 4.9 Hz, 2 H), 2.41 (t, $J$ = 7.1 Hz, 2 H), 1.60 (q, $J$ = 7.3 Hz, 2 H), 1.50–1.28 (m, 4 H), 0.91 (t, $J$ = 7.1 Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ = 152.3, 147.9, 138.4, 122.8, 121.2, 94.1, 31.1, 28.3, 22.2, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.$^{8}$

3-(hept-1-yn-1-yl)-6-methoxypyridazine (4j). Following method 1, 72.3 mg (0.5 mmol) of 3-chloro-6-methoxypyridazine and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded 4j as a brown oil (98 mg, 96%) using 1:9 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ = 7.32 (d, $J$ = 9.1 Hz, 1 H), 6.84 (d, $J$ = 9.1 Hz, 1 H), 4.08 (s, 3 H), 2.40 (t, $J$ = 7.1 Hz, 2
H), 1.59 (pentet, $J = 7.2$ Hz, 2 H), 1.45–1.33 (m, 2 H), 1.34–1.23 (m, 2 H), 0.87 (t, $J = 7.2$ Hz, 3 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta =$ 163.2, 144.0, 132.2, 130.7, 120.0, 116.5, 94.1, 54.9, 31.1, 27.9, 22.2, 19.3, 14.0 ppm. IR (film) $\nu_{\text{max}}$ 2955, 1593, 1542, 1463, 1411, 1324, 1289, 1267, 1012, 843, 797, 736 cm$^{-1}$. HRMS (ESI) calcd for C$_{12}$H$_{17}$N$_2$O$^+$ (M+H)$^+$, 205.1263, found: 205.1338.

**4-(cyclopropylethynyl)-2-(methylthio)pyridine (4k).** Following method 1, 80 mg (0.5 mmol) of 2-chloro-4-(methylthio)pyrimidine and 66.1 mg (0.6 mmol) of 3-cyclopropylpropionic acid afforded 4k as a yellow oil (90 mg, 95%) using 1:9 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta =$ 8.38 (d, $J = 5.1$ Hz, 1 H), 6.89 (d, $J = 5.1$ Hz, 1 H), 2.53 (s, 3 H), 1.53–1.39 (m, 1 H), 0.93 (ddq, $J =$ 10.1, 4.9, 2.3 Hz, 4 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta =$ 172.6, 156.4, 151.0, 118.0, 99.7, 74.0, 13.8, 8.9 ppm. IR (film) $\nu_{\text{max}}$ 2224, 1554, 1524, 1416, 1339, 1322, 1202, 864 cm$^{-1}$. HRMS (ESI) calcd for C$_{10}$H$_{11}$N$_2$S$^+$ (M+H)$^+$, 191.0565, found: 191.0638.

**3-(cyclopropylprop-2-yn-1-yl)benzene (4l).** Following method 1, 63.3 mg (0.5 mmol) of benzyl chloride and 66.1 mg (0.6 mmol) of 3-cyclopropylpropionic acid afforded 4l as a yellow oil (77 mg, 99%) using 3:97 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta =$ 7.42–7.30 (m, 4 H), 7.31–7.12 (m, 1 H), 3.61 (dd, $J =$ 2.0, 0.8 Hz, 2 H), 1.40–1.23 (m, 1 H), 0.87–0.76 (m, 2 H), 0.76–0.66 (m, 2 H) ppm. $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta =$ 137.5, 128.4, 127.9, 126.4, 85.6, 73.0, 25.2, 8.1 ppm. The physical data were in full accordance with the literature value.$^6$

**1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (4m).** Following method 1, 78.3 mg (0.5 mmol) of 4-methoxybenzyl chloride and 66.1 mg (0.6 mmol) of 3-cyclopropylpropionic acid afforded 4m as a yellow oil (91 mg, 98%) using 1:19 EtOAc:petroleum ether as the column eluent. $^1$H NMR (600 MHz, CDCl$_3$) $\delta =$ 7.26 (d, $J =$ 8.8 Hz, 2 H), 6.88 (d, $J =$ 8.7 Hz, 2 H), 3.82 (s, 3 H), 3.56–3.43 (m, 2 H), 1.35–1.21 (m, 1 H), 0.83–0.73 (m, 2 H), 0.71–0.59 (m, 2 H) ppm. $^{13}$C NMR
(151 MHz, CDCl3) δ = 158.2, 129.5, 128.8, 113.8, 85.3, 73.4, 55.3, 24.3, 8.0 ppm. IR (film) νmax 1510, 1246, 1174, 1033, 812 cm⁻¹. HRMS (ESI) calcd for C₁₃H₁₅O⁺ (M+H)⁺, 187.1045, found: 187.1075.

1,2-di(pyridin-3-yl)ethyne (6a). Following method 2, 113.6 mg (1.0 mmol) of 3-chloropyridine and 28.5 mg (0.4 mmol) of propiolic acid afforded 6a as a yellow oil (38 mg, 53%) using 2:1 EtOAc:petroleum ether as the column eluent. ¹H NMR (600 MHz, CDCl3) δ = 7.56 (d, J = 7.6 Hz, 2 H), 7.32 (ddd, J = 8.4, 7.4, 1.7 Hz, 2 H), 6.96 (td, J = 7.5, 1.1 Hz, 2 H), 6.92 (d, J = 8.3 Hz, 2 H), 3.95 (s, 6 H) ppm. ¹³C NMR (151 MHz, CDCl3) δ = 159.9, 133.6, 129.6, 120.4, 112.8, 110.7, 89.8, 55.9 ppm. The physical data were in full accordance with the literature value.

1,2-di(thiophen-2-yl)ethyne (6b). Following method 2, 118.0 mg (1.0 mmol) of 2-chlorothiophene and 28.5 mg (0.4 mmol) of propiolic acid afforded 6b as a yellow oil (45 mg, 58%) using 2:1 EtOAc:petroleum ether as the column eluent. ¹H NMR (600 MHz, CDCl3) δ = 7.56 (d, J = 7.6 Hz, 2 H), 7.32 (ddd, J = 8.4, 7.4, 1.7 Hz, 2 H), 6.96 (td, J = 7.5, 1.1 Hz, 2 H), 6.92 (d, J = 8.3 Hz, 2 H), 3.95 (s, 6 H) ppm. ¹³C NMR (151 MHz, CDCl3) δ = 159.9, 133.6, 129.6, 120.4, 112.8, 110.7, 89.8, 55.9 ppm. The physical data were in full accordance with the literature value.

1,2-bis(5-chloropyridin-3-yl)ethyne (6c). Following method 2, 148 mg (1.0 mmol) of 2,6-dichloropyridine and 28.5 mg (0.4 mmol) of propiolic acid afforded 6c as a white solid (24 mg, 24%) using 1:19 EtOAc:petroleum ether as the column eluent. ¹H NMR (600 MHz, CDCl3) δ = 8.65 (s, 1 H), 8.57 (s, 1 H), 7.84 – 7.83 (m, 1 H) ppm. ¹³C NMR (151 MHz, CDCl3) δ = 149.9, 148.5, 138.5, 132.0, 120.5, 89.0 ppm. IR (film) νmax 1737, 1374, 1241, 1046, 737 cm⁻¹. HRMS (ESI) calcd for C₁₂H₁Cl₂N₂⁺ (M+H)⁺, 248.9981, found: 248.9977.

References:


$^1$H NMR spectrum of 1-methoxy-2-(phenylethynyl)benzene (3a)

$^{13}$C NMR spectrum of 1-methoxy-2-(phenylethynyl)benzene (3a)
$^1$H NMR spectrum of 1-methoxy-3-(phenylethynyl)benzene (3b)

$^{13}$C NMR spectrum of 1-methoxy-3-(phenylethynyl)benzene (3b)
$^1$H NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene (3c)

$^{13}$C NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene (3c)
$^1$H NMR spectrum of 1-methyl-2-(phenylethynyl)benzene (3d)

$^{13}$C NMR spectrum of 1-methyl-2-(phenylethynyl)benzene (3d)
$^1$H NMR spectrum of 1-methyl-3-(phenylethynyl)benzene (3e)

$^{13}$C NMR spectrum of 1-methyl-3-(phenylethynyl)benzene (3e)
$^1$H NMR spectrum of 1-methyl-4-(phenylethynyl)benzene (3f)

$^{13}$C NMR spectrum of 1-methyl-4-(phenylethynyl)benzene (3f)
$^1$H NMR spectrum of 4-(phenylethynyl)benzaldehyde (3g)

$^{13}$C NMR spectrum of 4-(phenylethynyl)benzaldehyde (3g)
$^1$H NMR spectrum of 1-(4-(phenylethynyl)phenyl)ethan-1-one (3h)

$^{13}$C NMR spectrum of 1-(4-(phenylethynyl)phenyl)ethan-1-one (3h)
$^1$H NMR spectrum of 4-(phenylethynyl)benzonitrile (3i)

$^{13}$C NMR spectrum of 4-(phenylethynyl)benzonitrile (3i)
$^1$H NMR spectrum of 1,3-dimethyl-2-(phenylethynyl)benzene (3j)

$^{13}$C NMR spectrum of 1,3-dimethyl-2-(phenylethynyl)benzene (3j)
$^1$H NMR spectrum of 4-(phenylethynyl)phenol (3k)

$^{13}$C NMR spectrum of 4-(phenylethynyl)phenol (3k)
$^1$H NMR spectrum of 4-(phenylethynyl)aniline (3l)

$^{13}$C NMR spectrum of 4-(phenylethynyl)aniline (3l)
$^1$H NMR spectrum of 2-(phenylethynyl)thiophene (3m)

$^{13}$C NMR spectrum of 2-(phenylethynyl)thiophene (3m)
$^{1}H$ NMR spectrum of 3-(phenylethynyl)pyridine (3n)

$^{13}C$ NMR spectrum of 3-(phenylethynyl)pyridine (3n)
$^1$H NMR spectrum of 4,6-dimethoxy-2-(phenylethynyl)pyrimidine (3o)

$^{13}$C NMR spectrum of 4,6-dimethoxy-2-(phenylethynyl)pyrimidine (3o)
$^1$H NMR spectrum of 2-(methylthio)-4-(phenylethynyl)pyrimidine (3p)

$^{13}$C NMR spectrum of 2-(methylthio)-4-(phenylethynyl)pyrimidine (3p)
$^1$H NMR spectrum of 3-methoxy-6-(phenylethynyl)pyridazine (3q)

$^{13}$C NMR spectrum of 3-methoxy-6-(phenylethynyl)pyridazine (3q)
$^1$H NMR spectrum of 6-(phenylethynyl)imidazo[1,2-b]pyridazine (3r)

$^{13}$C NMR spectrum of 6-(phenylethynyl)imidazo[1,2-b]pyridazine (3r)
$^1$H NMR spectrum of 1-methyl-3-(phenylethynyl)-1H-pyrrolo[2,3-b]pyridine (3s)

$^{13}$C NMR spectrum of 1-methyl-3-(phenylethynyl)-1H-pyrrolo[2,3-b]pyridine (3s)
\(^1\)H NMR spectrum of (E)-pent-1-en-4-yne-1,5-diyldibenzene (3t)

\(^{13}\)C NMR spectrum of (E)-pent-1-en-4-yne-1,5-diyldibenzene (3t)
$^1$H NMR spectrum of prop-1-yn-1,3-diyl dibenzene (3u)

$^{13}$C NMR spectrum of prop-1-yn-1,3-diyl dibenzene (3u)
$^1$H NMR spectrum of 1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (3v)

$^{13}$C NMR spectrum of 1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (3v)
$^1$H NMR spectrum of 1-((4-methoxyphenyl)ethynyl)naphthalene (4b)

$^{13}$C NMR spectrum of 1-((4-methoxyphenyl)ethynyl)naphthalene (4b)
$^1$H NMR spectrum of 1-(hept-1-yn-1-yl)-4-methoxybenzene (4c)

$^{13}$C NMR spectrum of 1-(hept-1-yn-1-yl)-4-methoxybenzene (4c)
$^1$H NMR spectrum of 1-(but-1-yn-1-yl)-4-methoxybenzene (4d)

$^{13}$C NMR spectrum of 1-(but-1-yn-1-yl)-4-methoxybenzene (4d)
$^1$H NMR spectrum of 1-(cyclopropylethynyl)-4-methoxybenzene (4e)

$^{13}$C NMR spectrum of 1-(cyclopropylethynyl)-4-methoxybenzene (4e)
$^1$H NMR spectrum of 4-(hept-1-yn-1-yl)phenol (4f)

$^{13}$C NMR spectrum of 4-(hept-1-yn-1-yl)phenol (4f)
$^1$H NMR spectrum of 4-(hept-1-yn-1-yl)aniline (4g)

$^{13}$C NMR spectrum of 4-(hept-1-yn-1-yl)aniline (4g)
$^1$H NMR spectrum of 2-(hept-1-yn-1-yl)thiophene (4h)

$^{13}$C NMR spectrum of 2-(hept-1-yn-1-yl)thiophene (4h)
$^1$H NMR spectrum of 3-(hept-1-yn-1-yl)pyridine (4i)

$^{13}$C NMR spectrum of 3-(hept-1-yn-1-yl)pyridine (4i)
$^1$H NMR spectrum of 3-(hept-1-yn-1-yl)-6-methoxypyridazine (4j)

$^{13}$C NMR spectrum of 3-(hept-1-yn-1-yl)-6-methoxypyridazine (4j)
$^1$H NMR spectrum of 4-(cyclopropylethynyl)-2-(methylthio)pyrimidine (4k)

$^{13}$C NMR spectrum of 4-(cyclopropylethynyl)-2-(methylthio)pyrimidine (4k)
$^1$H NMR spectrum of (3-cyclopropylprop-2-yn-1-yl)benzene (4l)

$^{13}$C NMR spectrum of (3-cyclopropylprop-2-yn-1-yl)benzene (4l)
$^1$H NMR spectrum of 1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (4m)

$^{13}$C NMR spectrum of 1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (4m)
$^1$H NMR spectrum of 1,2-di(pyridin-3-yl)ethyne (6a)

$^{13}$C NMR spectrum of 1,2-di(pyridin-3-yl)ethyne (6a)
$^1$H NMR spectrum of 1,2-di(thiophen-2-yl)ethyne (6b)

$^{13}$C NMR spectrum of 1,2-di(thiophen-2-yl)ethyne (6b)
$^1$H NMR spectrum of 1,2-bis(5-chloropyridin-3-yl)ethyne (6c)

$^{13}$C NMR spectrum of 1,2-bis(5-chloropyridin-3-yl)ethyne (6c)