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Supporting Information

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Iron-Catalyzed Sonogashira Reaction

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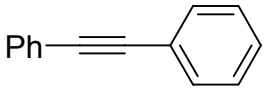
Supporting Information Available. Experimental details for compounds **3a-s** and **6a-b** and ^{1}H-NMR and ^{13}C-NMR spectra of all compounds are included.

General information: All reagents were purchased from commercial suppliers and used without further purification. $FeCl_3$ was purchased from Merck (98% purity). All experiments were carried out under argon. Flash chromatography was carried out with Merck silica gel 60 (63-200 mesh). Analytical TLC was performed with Merck silica gel 60 F_{254} plates, and the products were visualized by UV detection. ^{1}H-NMR and ^{13}C-NMR (300 or 400 MHz and 75 or 100 MHz, respectively) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard, and spin-spin coupling constants (J) are given in Hz. IR spectra were recorded on a Perkin-Elmer FT/IR 1760 as KBr pellets. Melting points were determined in open-end capillary tubes on a Büchi B-540 melting point apparatus and are uncorrected. Mass spectra were acquired on a Varian MAT 212 spectrometer (CI, 100 eV and EI, 70 eV). Microanalyses were obtained with a Vario EL element analyzer.

General procedure for Sonogashira reaction: A sealable tube equipped with a magnetic stir bar was charged with Cs_2CO_3 (2.0 equiv) and $FeCl_3$ (0.15 equiv). The aperture of the tube was then covered with a rubber septum, and an argon atmosphere was established. Alkyne (**1**, 1.0 equiv), aryliodide (**2**, 1.5 equiv), *N,N'*-dimethylethylendiamine (0.30 equiv) and toluene (1 mL/mmol of **1**) were added *via* syringe. The septum was then replaced by a teflon-coated screw cap, and the reaction vessel was placed in a 135 °C oil bath. After stirring at this temperature for 72 h, the heterogeneous mixture was cooled to room temperature and diluted with dichloromethane. The resulting solution was directly filtered through a pad of silica and concentrated to afford the product, which was purified by silica gel chromatography to yield arylated alkyne **3**. The identity and purity of the known products was confirmed by ^{1}H-and ^{13}C-NMR spectroscopic analysis, and the new products were fully characterized.

Diphenylacetylene[1] **(3a).** Following the general procedure using phenylacetylene (0.15 mL, 1.34 mmol) and iodobenzene (0.22 mL, 2.01 mmol) provided 161.3 mg (68% yield) of the coupling product as a white solid after purification by flash chromatography (pentane) of the crude oil.

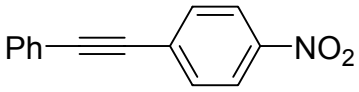


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.60-7.57 (m, 4H), 7.42-7.35 (m, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 131.5 (CH), 128.2 (CH), 128.1 (CH), 128.2 (C), 123.2 (C), 89.3 (C).

All spectral data correspond to those given in the literature.

1-Nitro-4-(phenylethynyl)benzene[2] **(3b).** Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and 4-iodonitrobenzene (339.9 mg, 1.34 mmol) provided 146.7 mg (74% yield) of the coupling product as a yellow solid after purification by flash chromatography (dichloromethane/pentane 3/7) of the crude oil.



^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.22 (d, J = 9.0 Hz, 2H) 7.66 (d, J = 9.0 Hz, 2H), 7.50-7.47 (m, 2H), 7.33-7.29 (m, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 146.9 (C), 132.2 (CH), 131.8 (CH), 130.2 (C), 129.3 (CH), 128.5 (CH), 123.6 (CH), 122.1 (C), 94.7 (C), 87.5 (C).

All spectral data correspond to those given in the literature.

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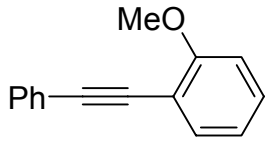
[1] Z. Novák, P. Nemes, A. Kotschy, *Org. Lett*. **2004**, *6*, 4917.

[2] N. Sakai, K. Annaka, T. Konakahara, *Org. Lett*. **2004**, *6*, 1527.

2-(Phenylethynyl)anisole[3] (3c). Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and 2-iodoanisole (0.18 mL, 1.34 mmol) provided 111.4 mg (60% yield) of the coupling product as a yellow oil after purification by flash chromatography (dichloromethane/pentane 3/7) of the crude oil.

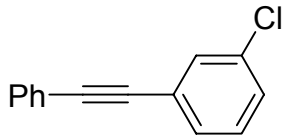


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.61-7.58 (m, 2H), 7.53 (d, J = 7.6 Hz, 1H), 7.39-7.29 (m, 4H), 6.95 (m, 2H), 3.92 (s, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 159.9 (C), 133.5 (CH), 131.6 (CH), 129.7 (CH), 128.2 (CH), 128.0 (CH), 123.5 (C), 120.4 (CH), 112.4 (C), 110.7 (CH), 93.4 (C), 85.7 (C), 55.8 (CH_3).

All spectral data correspond to those given in the literature.

1-Chloro-3-(phenylethynyl)benzene[4] (3d). Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and 1-iodo-3-chlorobenzene (0.20 mL, 1.34 mmol) provided 168.7 mg (89% yield) of the coupling product as a colorless liquid after purification by flash chromatography (pentane) of the crude oil.



^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.58-7.56 (m, 3H), 7.44 (dt, J = 7.4, 1.5, Hz, 1H), 7.40-7.27 (m, 5H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 134.0 (C), 131.5 (CH), 131.3 (CH), 129.6 (CH), 129.4 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 90.5 (C), 87.9 (C).

All spectral data correspond to those given in the literature.

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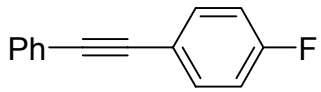
[3] D. Yue, T. Yao, R. C. Larock, *J. Org. Chem.* **2005**, *70*, 10292.

[4] M. R. Eberhard, Z. Wang, C. M. Jensen, *Chem. Commun.* **2002**, 818.

1-Fluoro-4-(phenylethynyl)benzene[4] (3e). Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and 1-iodo-4-fluorobenzene (0.16 mL, 1.34 mmol) provided 119.7 mg (69% yield) of the coupling product as a white solid after purification by flash chromatography (pentane) of the crude oil.

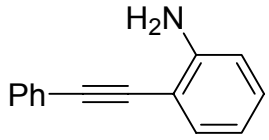


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.56-7.52 (m, 4H), 7.39-7.36 (m, 3H), 7.06 (t, J = 8.7, Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.3 (d, J = 249.1 Hz, C), 133.4 (d, J = 8.3 Hz, CH), 131.4 (CH), 128.2 (d, J = 4.2 Hz, CH), 122.9 (C), 115.6 (CH), 115.4 (CH), 119.3 (d, J = 3.5 Hz, C), 88.9 (C), 88.2 (C).

All spectral data correspond to those given in the literature.

2-(Phenylethynyl)aniline[2] (3f). Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and 2-iodoaniline (299.0 mg, 1.34 mmol) provided 153.9 mg (89% yield) of the coupling product as a yellow solid after purification by flash chromatography (ethyl acetate/pentane 2/8) of the crude oil.

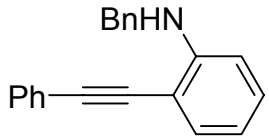


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.56-7.53 (m, 2H), 7.40-7.35 (m, 4H), 7.16 (ddd, J = 8.2, 7.4, 1.6 Hz, 1H), 6.76-6.71 (m, 2H), 4.27 (bs, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 147.7 (C), 132.1 (CH), 131.4 (CH), 129.7 (CH), 128.3 (CH), 128.2 (CH), 123.3 (C), 117.9 (CH), 114.3 (CH), 107.9 (C), 94.6 (C), 85.8 (C).

All spectral data correspond to those given in the literature.

***N*-Benzyl-2-(phenylethynyl)aniline[5] (3g).** Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol) and *N*-benzyl-2-iodoaniline[6] (414.0 mg, 1.34 mmol) provided 228.4 mg (86% yield) of the coupling product as an orange oil after purification by flash chromatography (diethyl ether/pentane 0.1/9.9) of the crude oil.

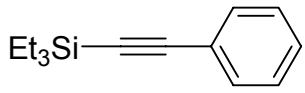


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.38-7.35 (m, 2H), 7.30-7.11 (m, 9H), 7.03 (m, 1H), 6.54 (dt, J = 7.5, 0.9 Hz, 1H), 6.45 (d, J = 8.3 Hz, 1H), 5.02 (bs, 1H), 4.30 (d, J = 5.4 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 148.5 (C), 138.9 (C), 131.9 (CH), 131.2 (CH), 129.9 (CH), 128.5 (CH), 128.2 (CH), 128.0 (CH), 127.0 (CH), 126.9 (CH), 123.1 (C), 116.5 (CH), 109.8 (CH), 107.4 (C), 95.2 (C), 85.9 (C), 47.6 (CH_2).

All spectral data correspond to those given in the literature.

(Triethylsilyl)phenylacetylene[7] (3h). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and iodobenzene (0.12 mL, 1.07 mmol) provided 122 mg (80% yield) of the coupling product as a colorless oil after purification by flash chromatography (pentane) of the crude oil.



^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.49-7.51 (m, 2H), 7.29-7.32 (m, 3H), 1.09 (t, J = 7.9 Hz, 9H), 0.71 (t, J = 7.9 Hz, 6H).

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[5] M. Nakamura, L. Ilies, S. Otsubo, E. Nakamura, *Angew. Chem*. **2006**, *118*, 958; *Angew. Chem. Int. Ed*. **2006**, *45*, 944.

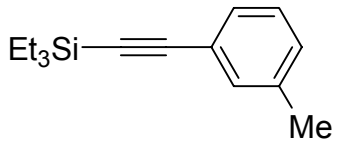
[6] *N*-Benzyl-2-iodoaniline was prepared following the literature procedure: E. L. Cropper, A. J. P. White, A. Ford, K. K. Hii, *J. Org. Chem*. **2006;** *71,* 1732.

[7] A. A. Selina, S. S. Karlov, E. V. Gauchenova, A. V. Churakov, L. G. Kuz'mina, J. A. K. Howard, J. Lorberth, G. S. Zaitseva, *Heteroatom Chem.* **2004**, *15*, 43.

^{13}C-NMR (100 MHz, $CDCl_3$) δ 132.0 (CH), 128.3 (CH), 128.1 (CH), 123.3 (C), 106.4 (C), 91.5 (C), 7.7 (CH_3), 4.6 (CH_2).

All spectral data correspond to those given in the literature.

(Triethylsilyl)-3-tolylacetylene[8] (3i). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and 3-iodotoluene (0.14 mL, 1.06 mmol) provided 147 mg (90% yield) of the coupling product as a colorless oil after purification by flash chromatography (petroleum ether) of the crude oil.



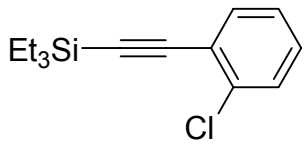
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.17-7.21 (m, 2H), 6.98-7.10 (m, 2H), 2.21 (s, 3H), 0.97 (t, J = 8.5 Hz, 9H), 0.58 (q, J = 8.5 Hz, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.8 (C), 132.6 (CH), 129.3 (CH), 129.2 (CH), 128.1 (CH), 123.2 (C), 106.7 (C), 91.1 (C), 21.2 (CH_3), 7.5 (CH_3), 4.5 (CH_2).

MS (EI) *m/z* (%) 230 (M^+, 19), 201 (100), 173 (75), 145 (83).

Calcd. for $C_{15}H_{22}Si$: C, 78.19; H, 9.62; found C, 78.12; H, 9.89.

2-Chlorophenyl(triethylsilyl)acetylene[8] (3j). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and 1-choro-2-iodobenzene (0.13 mL, 1.07 mmol) provided 130 mg (73% yield) of the coupling product as a colorless oil after purification by flash chromatography (petroleum ether) of the crude oil.



[8] C. Eaborn, D. R. M. Walton, *J. Organomet. Chem.* **1965**, *4*, 217.

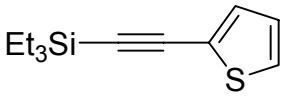
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, J = 7.3 Hz, 1.9 Hz, 1H), 7.38 (dd, J = 7.9 Hz, 1.4 Hz, 1H), 7.16-7.26 (m, 2H), 1.07 (t, J = 8.5 Hz, 9H), 0.70 (q, J = 8.5 Hz, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 136.2 (C), 133.6 (CH), 129.3 (CH), 129.1 (CH), 126.2 (CH), 123.2 (C), 102.4 (C), 97.8 (C), 7.6 (CH_3), 4.5 (CH_2).

MS (EI) *m/z* (%) 252 (M^++2, 4), 250 (M^+, 12), 221 (100), 193 (76), 165 (54), 129 (28), 63 (22).

Calcd. for $C_{14}H_{19}ClSi$: C, 67.03; H, 7.63; found C, 67.13; H, 7.84.

Triethylsilyl-2-thienylacetylene (3k). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and 2-iodothiophene (0.12 mL, 1.06 mmol) provided 62 mg (40% yield) of the coupling product as a colorless oil after purification by flash chromatography (petroleum ether) of the crude oil.



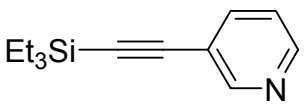
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.13-7.16 (m, 2H), 6.85-6.88 (m, 1H), 0.96 (t, J = 8.5 Hz, 9H), 0.59 (q, J = 8.5 Hz, 6H).

^{13}C-NMR (75 MHz, $CDCl_3$) δ 136.2 (C), 133.6 (CH), 129.3 (CH), 129.1 (CH), 126.2 (CH), 123.2 (C), 102.4 (C), 97.8 (C), 7.6 (CH_3), 4.5 (CH_2).

MS (EI) *m/z* (%) 222 (M^+, 25), 193 (100), 165 (93), 137 (82), 123 (42), 95 (50), 57 (57).

Calcd. for $C_{12}H_{18}SSi$: C, 64.80; H, 8.16; found C, 64.44; H, 8.39.

Triethylsilyl-3-pyridylacetylene (3l). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and 3-iodopyridine (217 mg, 1.06 mmol) provided 89 mg (58% yield) of the coupling product as a colorless oil after purification by flash chromatography (pentane/ethyl acetate 8/2) of the crude oil.



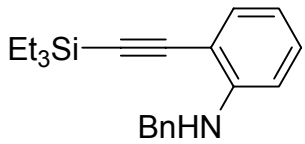
^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.49 (d, J = 4.7 Hz, 1H), 7.69-7.72 (m, 1H), 7.18-7.21 (m, 1H), 1.02 (t, J = 8.5 Hz, 9H), 0.66 (q, J = 8.5 Hz, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 152.6 (CH), 148.5 (CH), 138.8 (CH), 122.8 (CH), 120.4 (C), 102.7 (C), 95.8 (C), 7.6 (CH_3), 4.4 (CH_2).

MS (EI) *m/z* (%) 217 (M^+, 10), 188 (100), 160 (75), 132 (66).

Calcd. for $C_{13}H_{19}NSi$: C, 71.83; H, 8.81; N, 6.44; found C, 71.53; H, 9.06; N, 6.66.

Triethylsilyl-2-(aminobenzyl)phenylacetylene (3m). Following the general procedure using triethylsilylacetylene (0.13 mL, 0.71 mmol) and *N*-benzyl-2-iodoaniline (327 mg, 1.06 mmol) provided 191 mg (85% yield) of the coupling product as a colorless oil after purification by flash chromatography (pentane/ethyl acetate 98/2) of the crude oil.



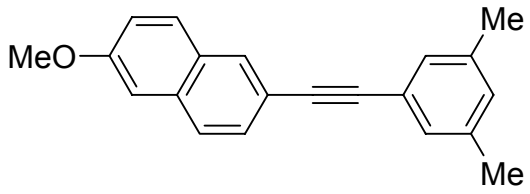
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.20-7.34 (m, 6H), 7.09-7.13 (m, 1H), 6.57 (dt, J = 7.4 Hz, 1.1 Hz, 1H), 6.53 (d, J = 8.2 Hz, 1H), 5.00 (br s, 1H), 4.33 (d, J = 5.2 Hz, 2H), 0.93 (t, J = 8.5 Hz, 9H), 0.58 (q, J = 8.5 Hz, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 149.2 (C), 138.8 (C), 132.1 (CH), 130.0 (CH), 128.6 (CH), 127.4 (CH), 127.3 (CH), 116.3 (CH), 109.6 (C), 107.7 (C), 103.1 (C), 97.8 (C), 47.9 (CH_2), 7.7 (CH_3), 4.6 (CH_2).

MS (EI) *m/z* (%) 321 (M^+, 10), 244 (38), 206 (66), 91 (36), 59 (22).

Calcd. for $C_{21}H_{27}NSi$: C, 78.44; H, 8.46; N, 4.36; found C, 78.67; H, 8.34; N, 4.23.

2-(3,5-Dimethylphenylethynyl)-6-methoxynaphthalene (3n). Following the general procedure using 2-ethynyl-6-methoxynaphthalene (100 mg, 0.53 mmol) and 5-iodo-*m*-xylene (0.11 mL, 0.79 mmol) provided 150 mg (99.9% yield) of the coupling product as a white solid after purification by flash chromatography (pentane/ethyl acetate 98/2) of the crude oil.



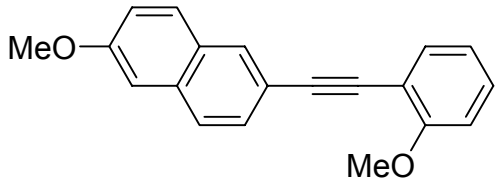
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.69-7.74 (m, 2H), 7.55-7.58 (m, 1H), 7.24 (s, 2H), 7.18 (dd, J = 9.1 Hz, 2.5 Hz, 1H), 7.12 (d, J = 2.2 Hz, 1H), 6.99 (s, 1H), 3.93 (s, 3H), 2.35 (s, 6H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 158.2 (C), 137.9 (C), 134.0 (C), 131.2 (CH), 130.1 (CH), 129.3 (CH), 129.2 (CH), 129.1 (CH), 128.5 (C), 126.8 (CH), 123.1 (C), 119.4 (CH), 118.4 (C), 89.5 (C), 89.4 (C), 55.4 (CH_3), 21.3 (CH_3).

MS (EI) *m/z* (%) 286 (M^+, 100), 271 (38), 243 (65), 226 (18), 143 (39), 114 (21).

Calcd. for $C_{21}H_{18}O$: C, 88.08; H, 6.34; found C, 87.71; H, 6.34.

2-(2-Methoxyphenylethynyl)-6-methoxynaphthalene (3o). Following the general procedure using 2-ethynyl-6-methoxynaphthalene (100 mg, 0.53 mmol) and 2-iodoanisole (0.10 mL, 0.79 mmol) provided 83 mg (54% yield) of the coupling product as an orange oil after purification by flash chromatography (pentane/dichloromethane 1/1) of the crude oil.



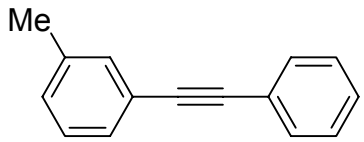
^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.71 (t, J = 8.8 Hz, 2H), 7.55-7.62 (m, 2H), 7.30-7.34 (m, 1H), 7.16 (dd, J = 8.8 Hz, 2.5 Hz, 1H), 7.12 (d, J = 2.7 Hz, 1H), 6.97 (dt, J = 7.7 Hz, 1.1 Hz, 1H), 6.92 (d, J = 8.2 Hz, 1H), 3.94 (s, 3H), 3.92 (s, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 159.9 (C), 158.2 (C), 134.0 (C), 133.5 (CH), 131.2 (CH), 129.6 (CH), 129.3 (CH), 129.2 (CH), 128.5 (C), 126.7 (CH), 120.5 (CH), 119.3 (CH), 118.5 (C), 112.7 (C), 110.7 (CH), 105.8 (CH), 94.1 (C), 85.5 (C), 55.9 (CH_3), 55.4 (CH_3).

MS (EI) *m/z* (%) 288 (M^+, 100), 245 (14), 202 (13).

Calcd. for $C_{20}H_{16}O_2$: C, 83.31; H, 5.59; found C, 83.56; H, 5.39.

3-(Phenylethynyl)toluene[4] (3p). Following the general procedure using 3-methylphenylacetylene (0.10 mL, 0.75 mmol) and phenyliodide (0.13 mL, 1.13 mmol) provided 73.6 mg (51% yield) of the coupling product as a colorless liquid after purification by flash chromatography (pentane) of the crude oil.

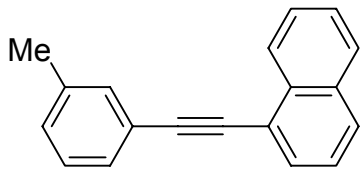


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.46-7.43 (m, 2H), 7.29-7.23 (m, 5H), 7.15 (t, J = 7.6 Hz, 1H), 7.06 (d, J = 7.6 Hz, 1H), 2.26 (s, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.9 (C), 132.1 (CH), 131.5 (CH), 129.1 (CH), 128.6 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 123.3 (C), 122.9 (C), 89.5 (C), 88.9 (C), 21.3 (CH_3).

All spectral data correspond to those given in the literature.

3-(1-Naphthylethynyl)toluene (3q). Following the general procedure using 3-methylphenylacetylene (0.10 mL, 0.75 mmol) and 1-iodonaphthalene (0.17 mL, 1.13 mmol) provided 109.8 mg (55% yield) of the coupling product as a yellow oil after purification by flash chromatography (pentane) of the crude oil.



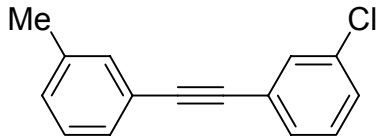
^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.54 (d, J = 8.3 Hz, 1H), 7.71-7.60 (m, 3H), 7.47-7.28 (m, 5H), 7.35 (t, J = 7.6, Hz, 1H), 7.24 (d, J = 7.5 Hz, 1H), 2.45 (s, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 138.1 (C), 133.2 (C), 133.1 (C), 132.2 (CH), 130.2 (CH), 129.3 (CH), 128.7 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 126.7 (CH); 126.4 (CH), 126.2 (CH), 125.2 (CH), 123.2 (C), 120.9 (C), 94.5 (C), 87.2 (C), 21.2 (CH_3).

MS (EI) *m/z* (%) 242 (100), 239 (20), 226 (11), 119 (13).

Calcd. for $C_{19}H_{14}$: C, 94.18; H, 5.82; found C, 94.03; H, 5.94.

3-(3-Chlorophenylethynyl)toluene[9] (3r). Following the general procedure using 3-methylphenylacetylene (0.10 mL, 0.75 mmol) and 1-iodo-3-chlorobenzene (0.17 mL, 1.13 mmol) provided 131.9 mg (77% yield) of the coupling product as a white solid after purification by flash chromatography (pentane) of the crude oil.



^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.55 (t, J = 1.7 Hz, 1H), 7.44-7.25 (m, 6H), 7.19 (d, J = 7.6 Hz, 1H), 2.38 (s, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.9 (C), 134.0 (C), 132.1 (CH), 131.3 (CH), 129.6 (CH), 129.4 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 125.0 (C), 122.4 (C), 90.7 (C), 87.6 (C), 21.3 (CH_3).

MS (EI) *m/z* (%) 228 (M+2, 34), 226 (M^+, 100), 189 (32), 94 (14).

Calcd. for $C_{15}H_{11}Cl$: C, 79.47; H, 4.89; found C, 79.11; H, 4.96.

(Cyclohexyl)phenylacetylene[10] (3s). Following the general procedure using cyclohexylacetylene (0.12 mL, 0.92 mmol) and iodobenzene (0.16 mL, 1.38 mmol) provided 24 mg (15% yield) of the coupling product as a colorless oil after purification by flash chromatography (pentane) of the crude oil.

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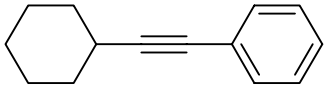
[9] D. Seyferth, M. O. Nestle, A. T. Wehman, *J. Am. Chem. Soc*. **1975**, *97*, 7417.

[10] J. Gong, P. L. Fuchs, *J. Am. Chem. Soc.* **1996**, *118*, 4486.

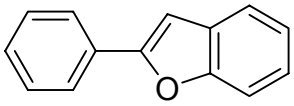


^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.30-7.37 (m, 2H), 7.16-7.22 (m, 3H), 2.48-2.54 (m, 1H), 1.23-1.82 (10H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 131.5 (CH), 128.1 (CH), 127.3 (CH), 124.1 (C), 94.5 (C), 80.5 (C), 32.8 (CH_2), 29.8 (CH), 26.1 (CH_2), 25.0 (CH_2).

All spectral data correspond to those given in the literature.

2-Phenylbenzo[*b*]furan[11] (6a). Following the general procedure using phenylacetylene (0.10 mL, 0.89 mmol), 1-iodophenol (300.4 mg, 1.34 mmol) and 3.0 equiv of base provided 88.8 mg (51% yield) of the domino coupling product as a white solid after purification by flash chromatography (pentane) of the crude oil.



^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, J = 8.4, 1.3 Hz, 2H), 7.63 (dd, J = 7.5, 1.5 Hz, 1H), 7.58 (d, J = 8.2 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 7.25-7.10 (m, 3H), 7.06 (d, J = 0.8 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 155.7 (C), 154.7 (C), 130.4 (C), 129.1 (C), 128.7 (CH), 128.4 (CH), 124.8 (CH), 124.1 (CH), 122.8 (CH), 120.8 (CH), 111.1 (CH), 101.2 (CH).

All spectral data correspond to those given in the literature.

2-(3-Tolyl)benzo[*b*]furan[12] (6b). Following the general procedure using 3-methylphenylacetylene (0.10 mL, 0.75 mmol), 1-iodophenol (253.1 mg, 1.13 mmol) and 3.0 equiv of base provided 78.8 mg (50% yield) of the domino coupling product as a white solid after purification by flash chromatography (pentane) of the crude oil.

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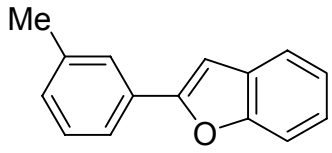
[11] J. –M. Becht, A. Gissot, A. Wagner, C. Miokowski, *Chem. Eur. J.* **2003**, *9*, 3209.

[12] J. N. Chatterjea, S. K. Roy, *J. Indian Chem. Soc*. **1957**, *34*, 98.



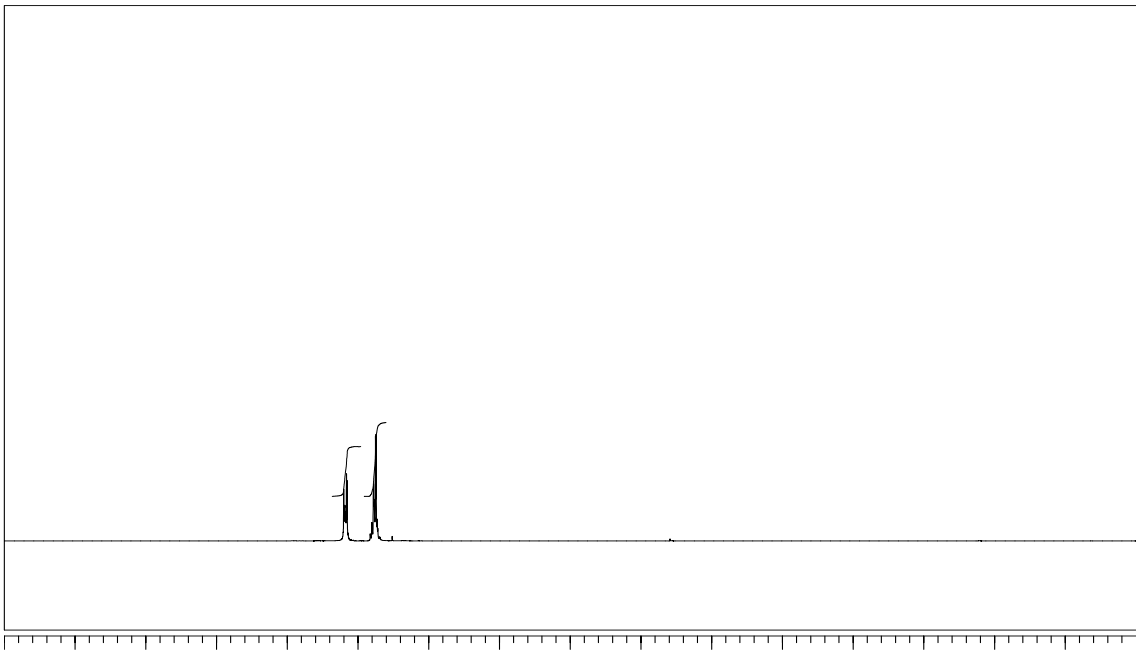
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.77-7.72 (m, 2H), 7.66-7.58 (m, 2H), 7.42-7.22 (m, 4H), 7.06 (d, J = 0.8 Hz, 1H), 2.49 (s, 3H).

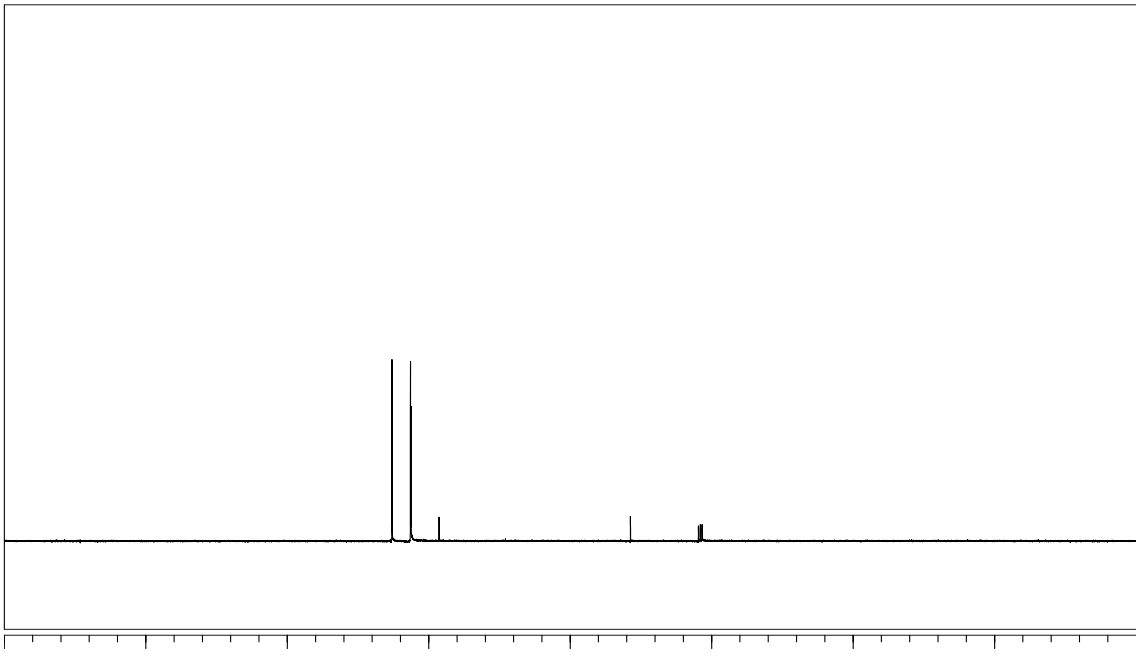
^{13}C-NMR (100 MHz, $CDCl_3$) δ 156.1 (C), 154.8 (C), 138.4 (C), 130.3 (C), 129.3 (CH), 129.2 (C), 128.7 (CH), 125.5 (CH), 124.1 (CH), 122.8 (CH), 122.1 (CH), 120.8 (CH), 111.1 (CH), 101.2 (CH), 21.5 (CH_3).

MS (EI) *m/z* (%) 208 (M^+, 100), 178 (11), 165 (12).

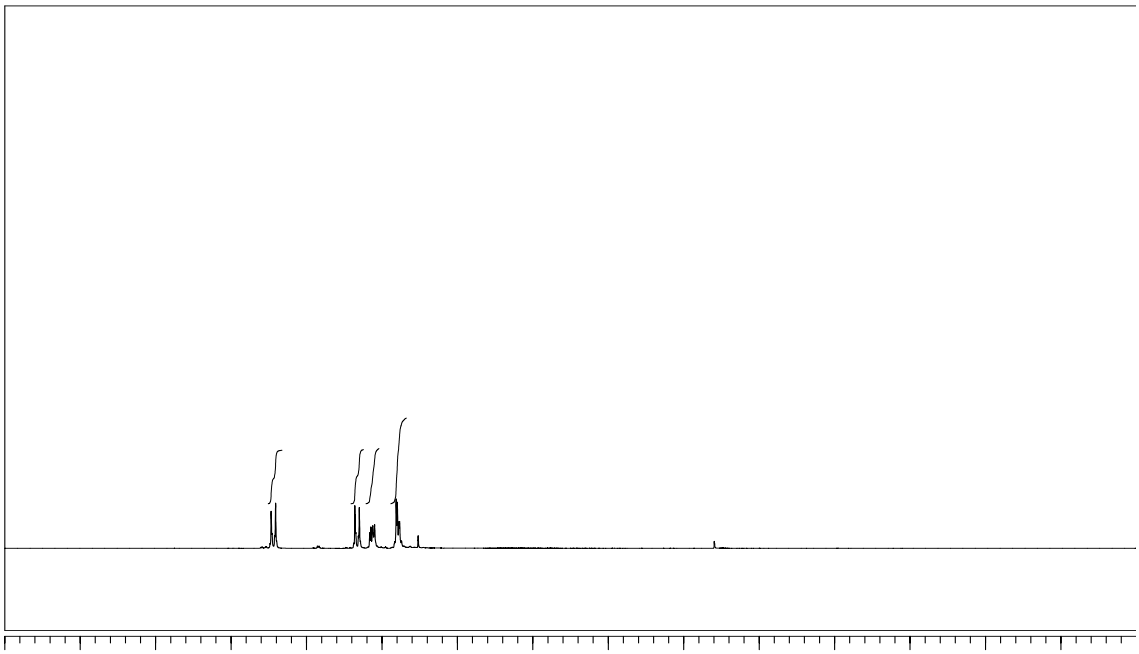
Calcd. for $C_{15}H_{12}O$: C, 86.51; H, 5.81; found C, 86.42; H, 5.78.

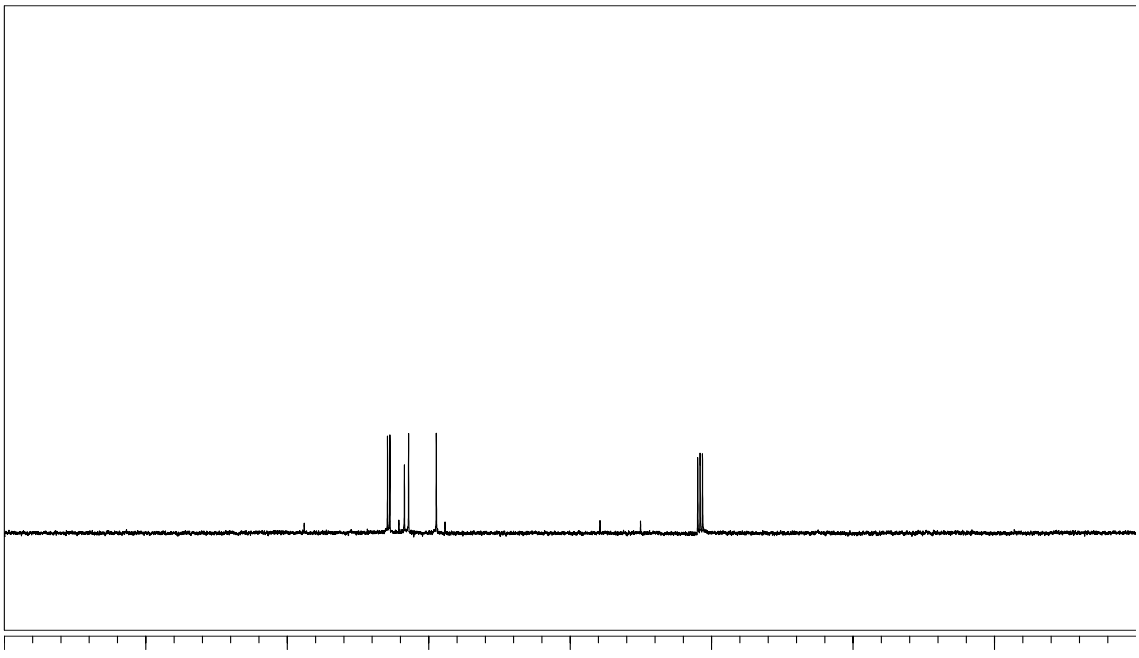
Diphenylacetylene (3a)



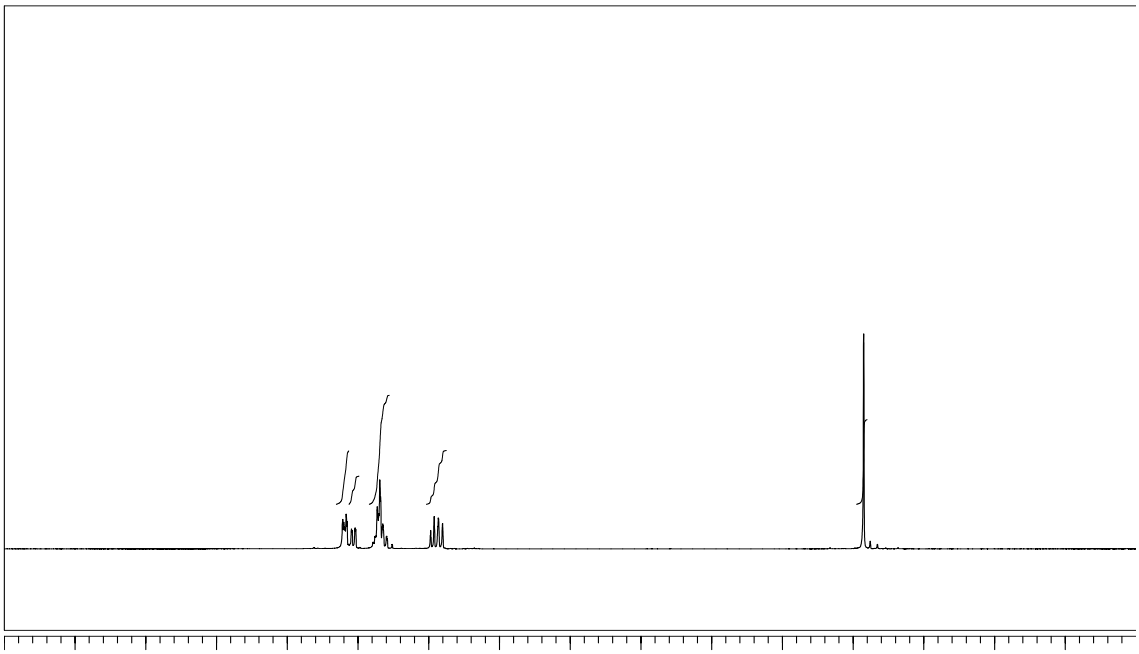


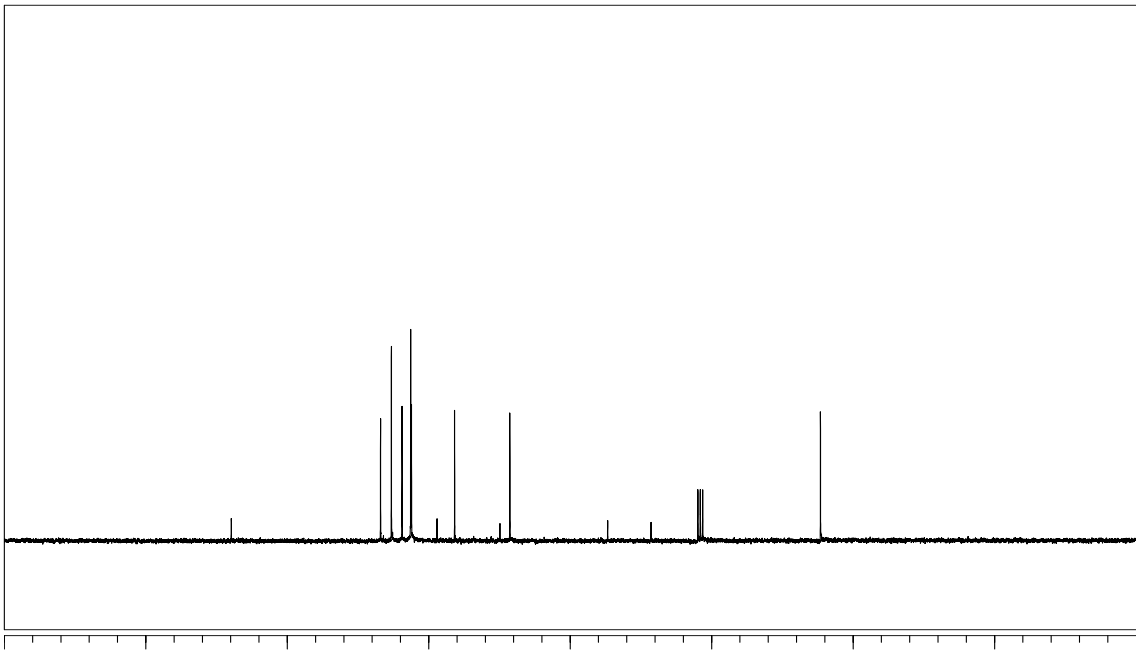
4-(Phenylethynyl)nitrobenzene (3b)



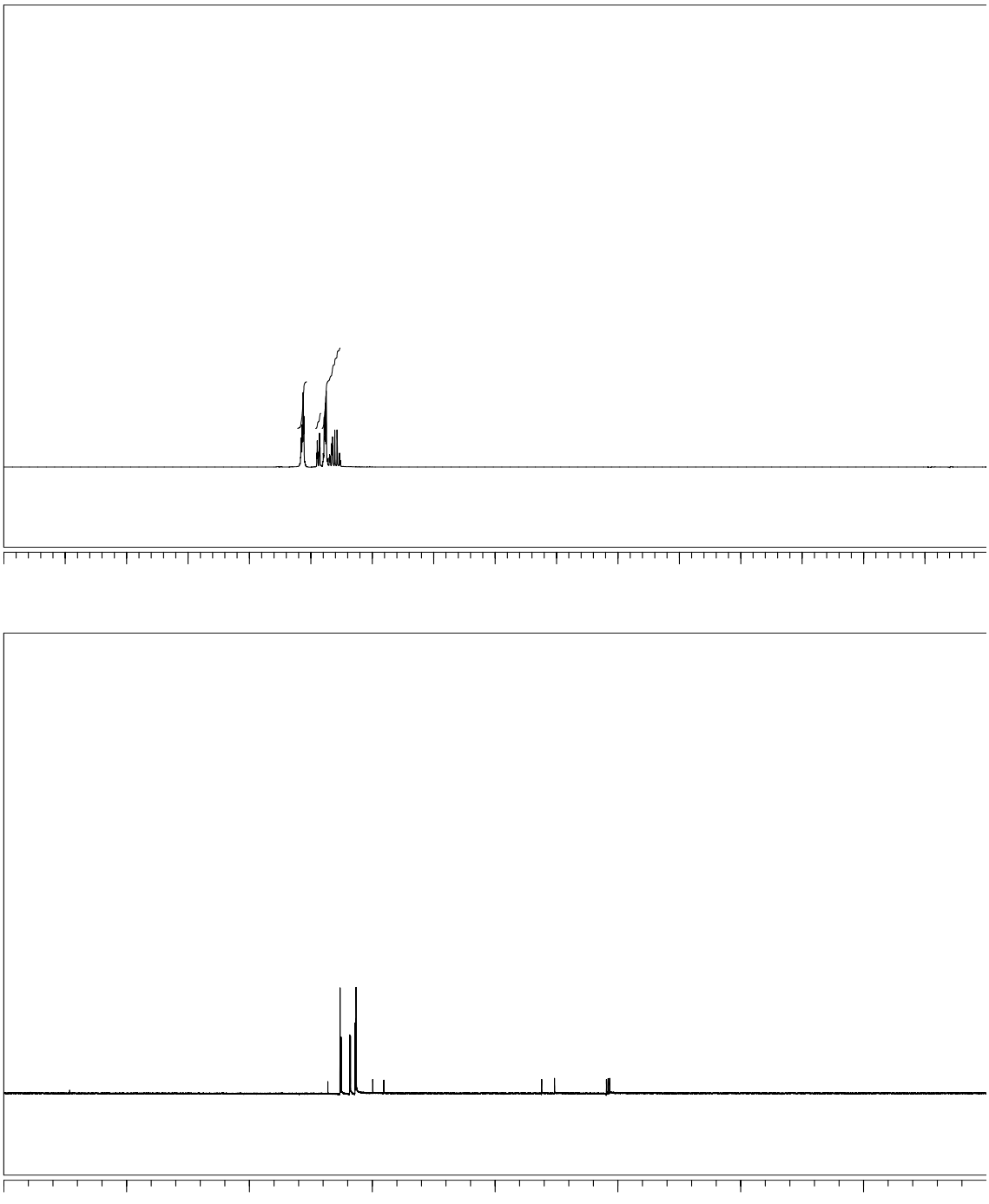


2-(Phenylethynyl)anisole (3c)

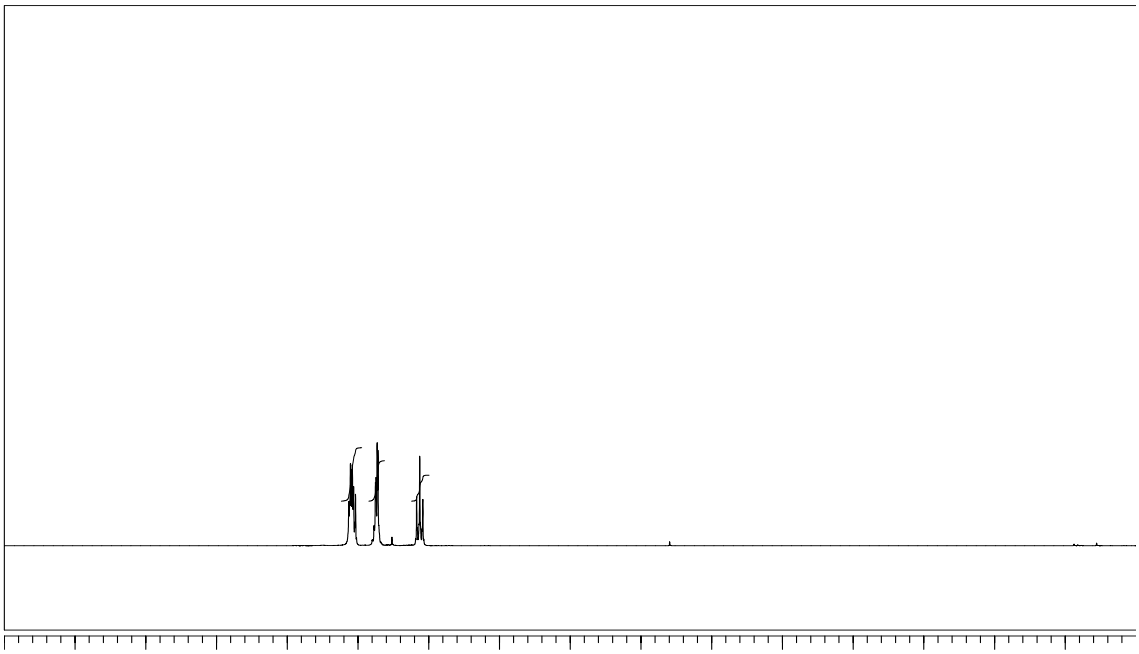


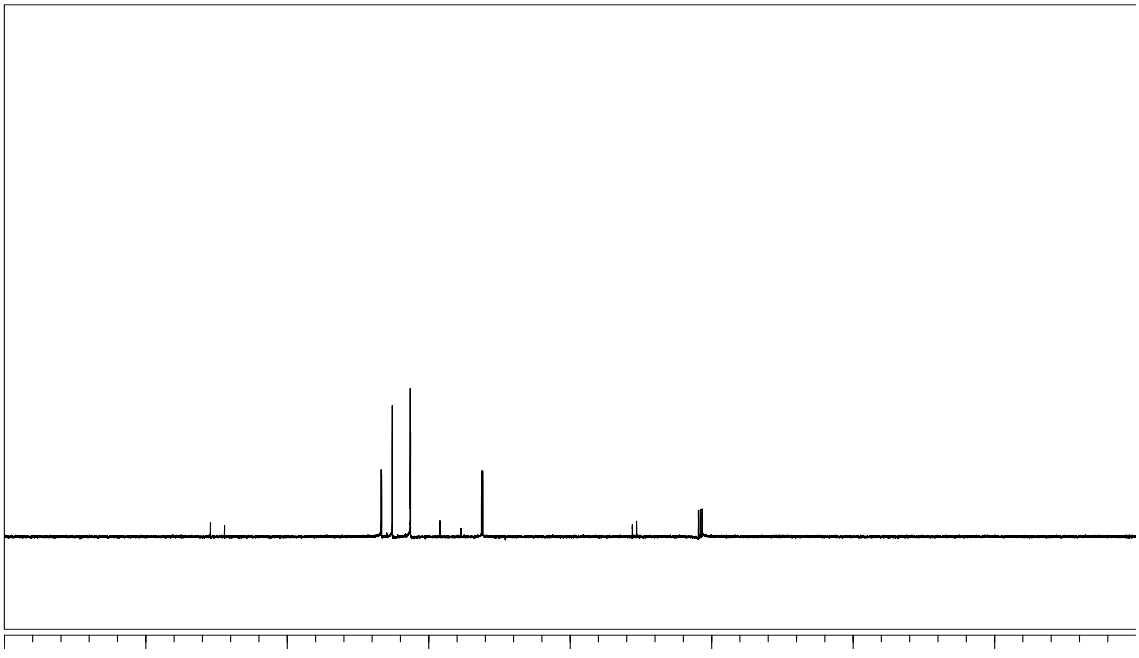


1-Chloro-3-(phenylethynyl)benzene (3d)

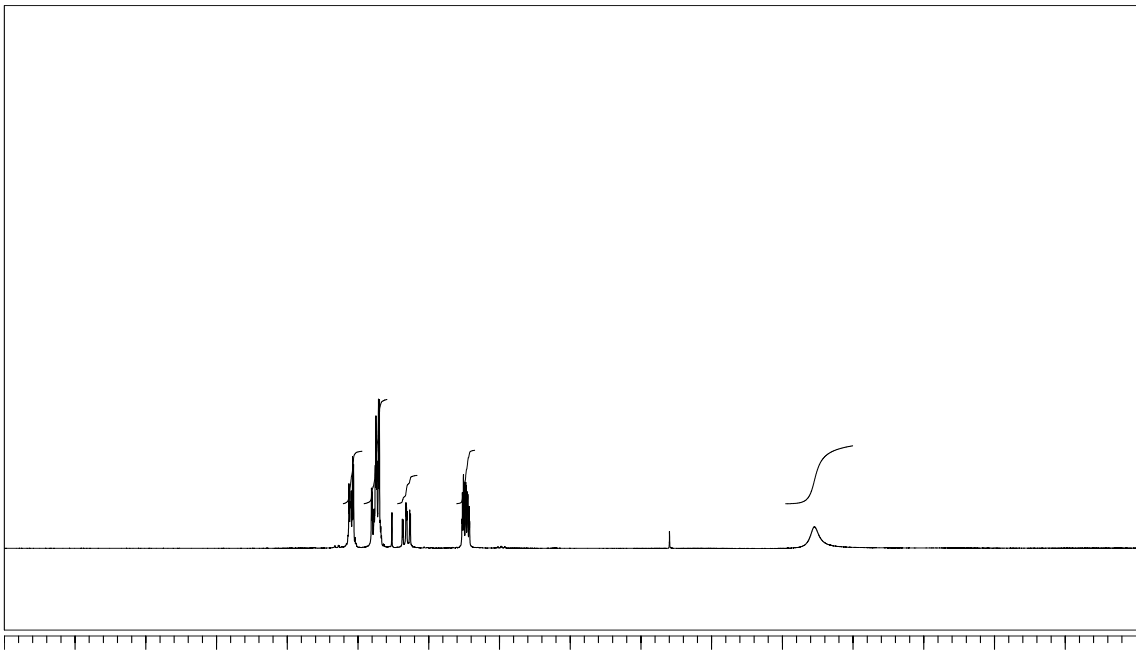


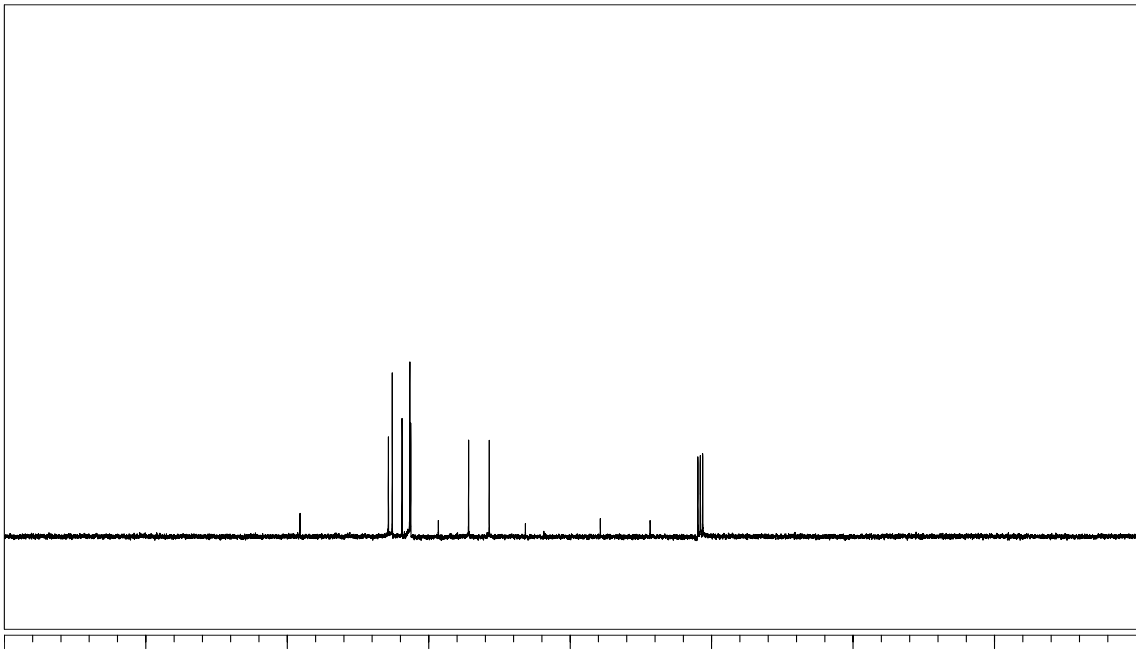
1-Fluoro-4-(phenylethynyl)benzene (3e)



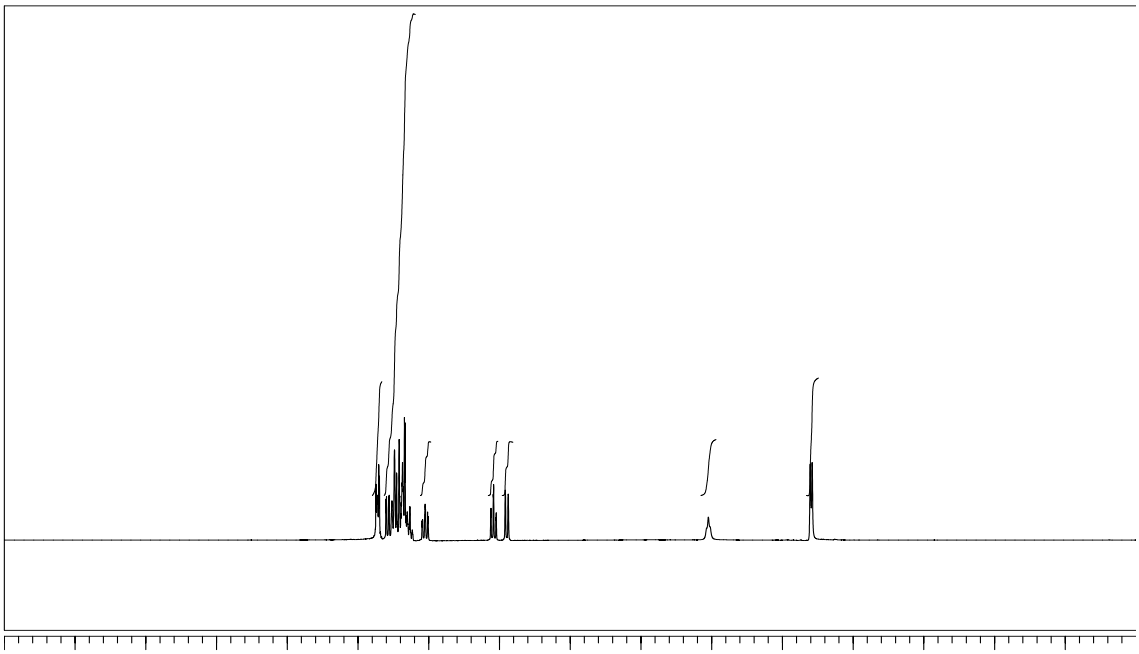


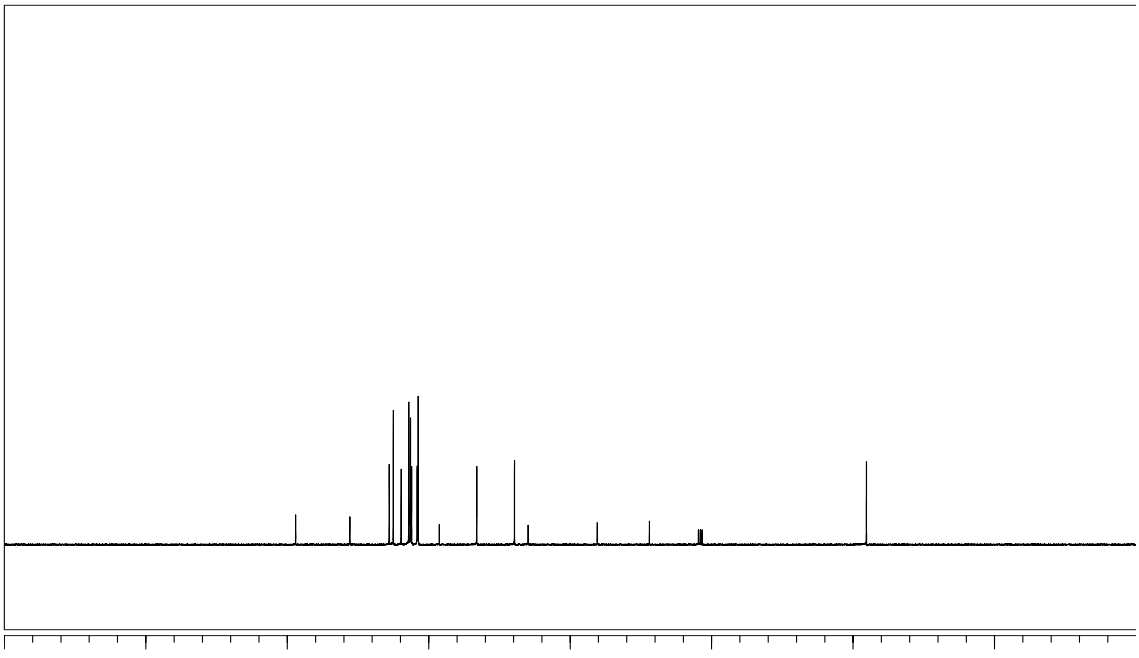
2-(Phenylethynyl)aniline (3f)



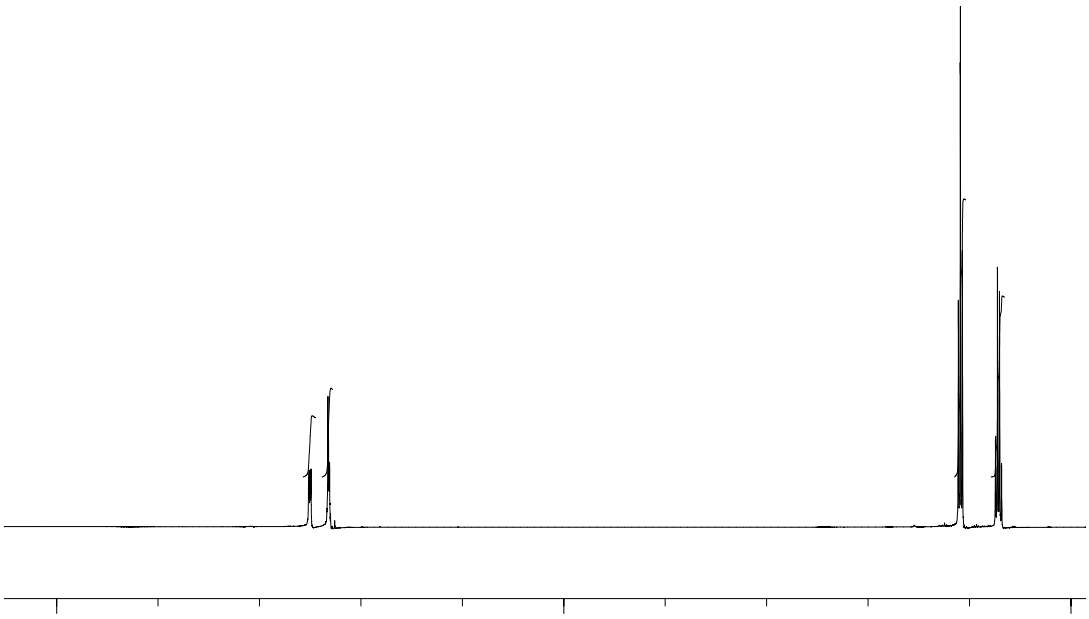


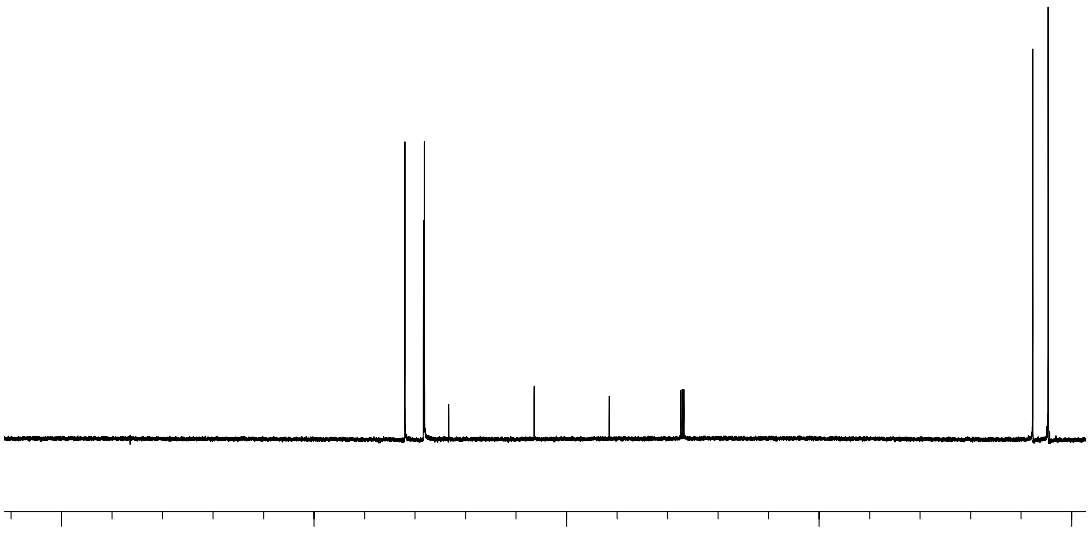
***N*-Benzyl-2-(phenylethynyl)aniline (3g)**



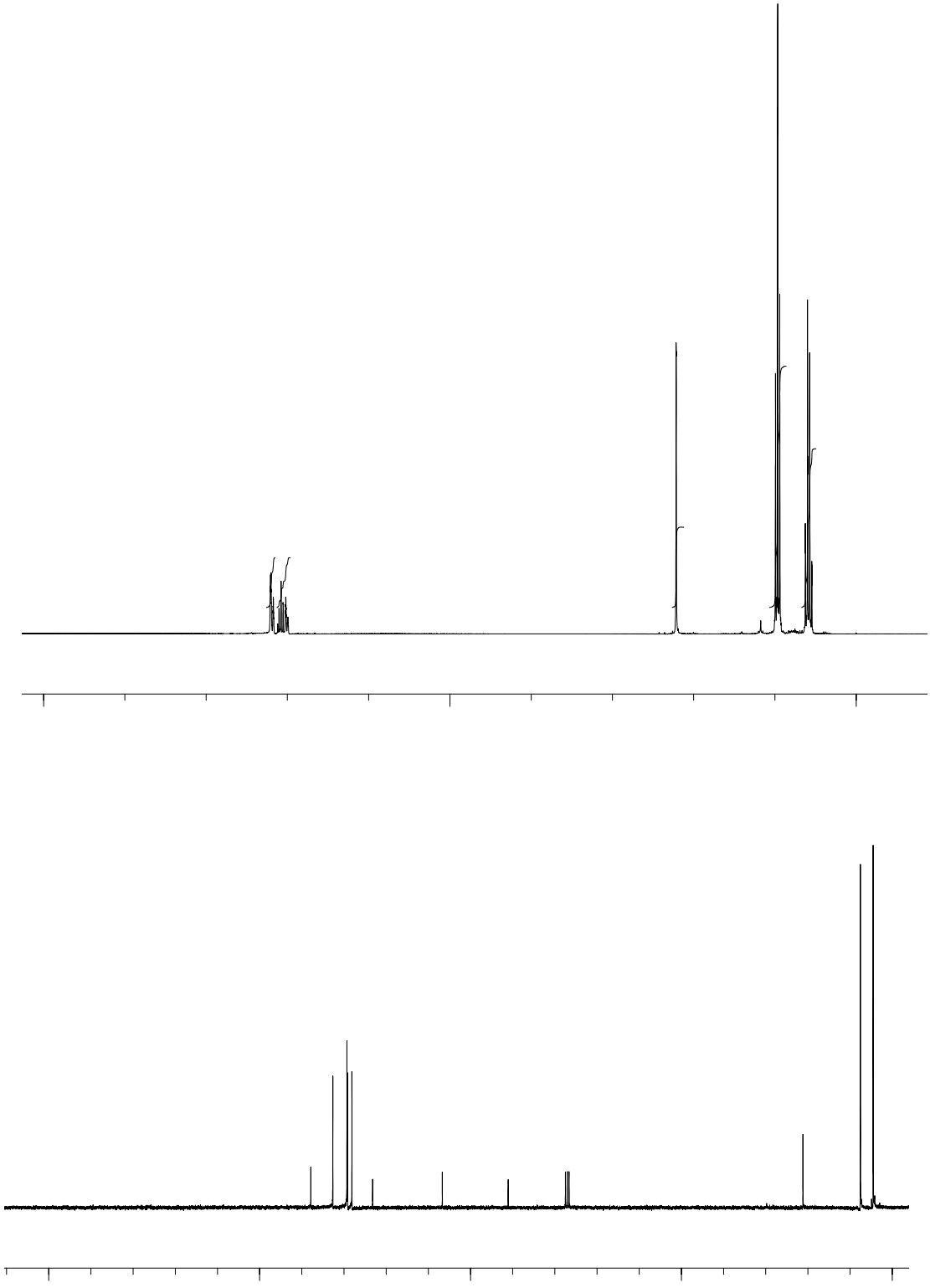


(Triethylsilyl)phenylacetylene (3h)

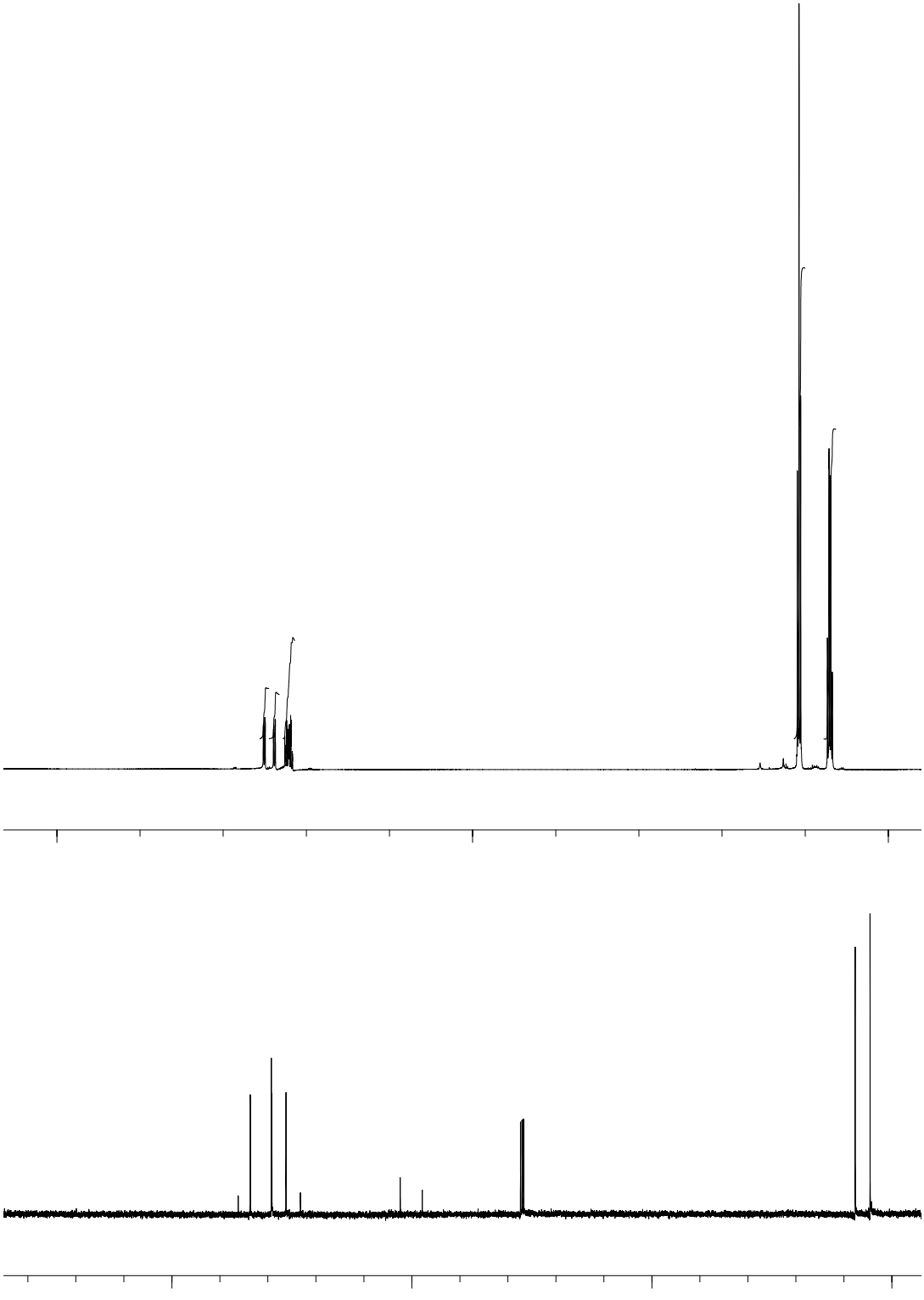




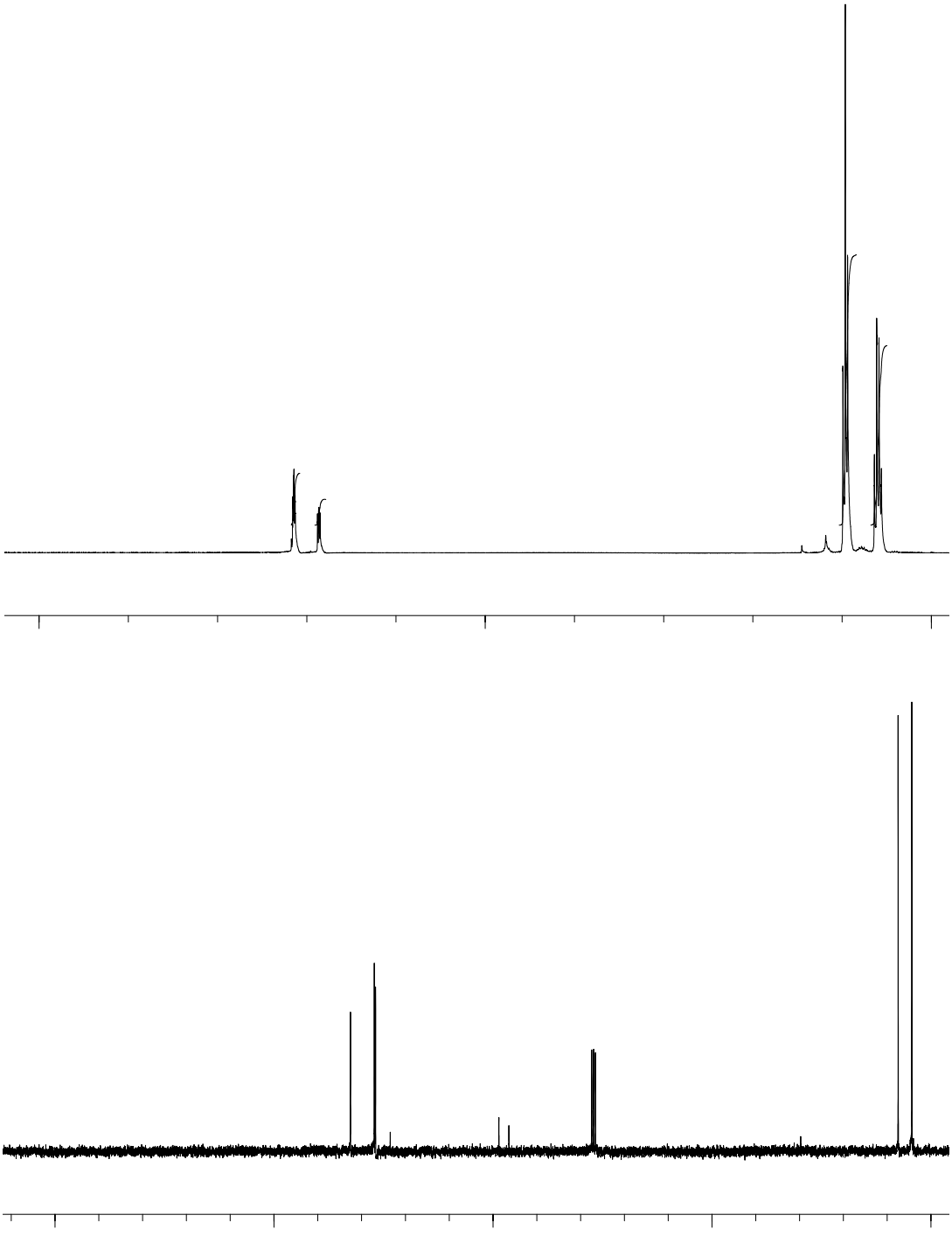
(Triethylsilyl)-3-tolylacetylene (3i)



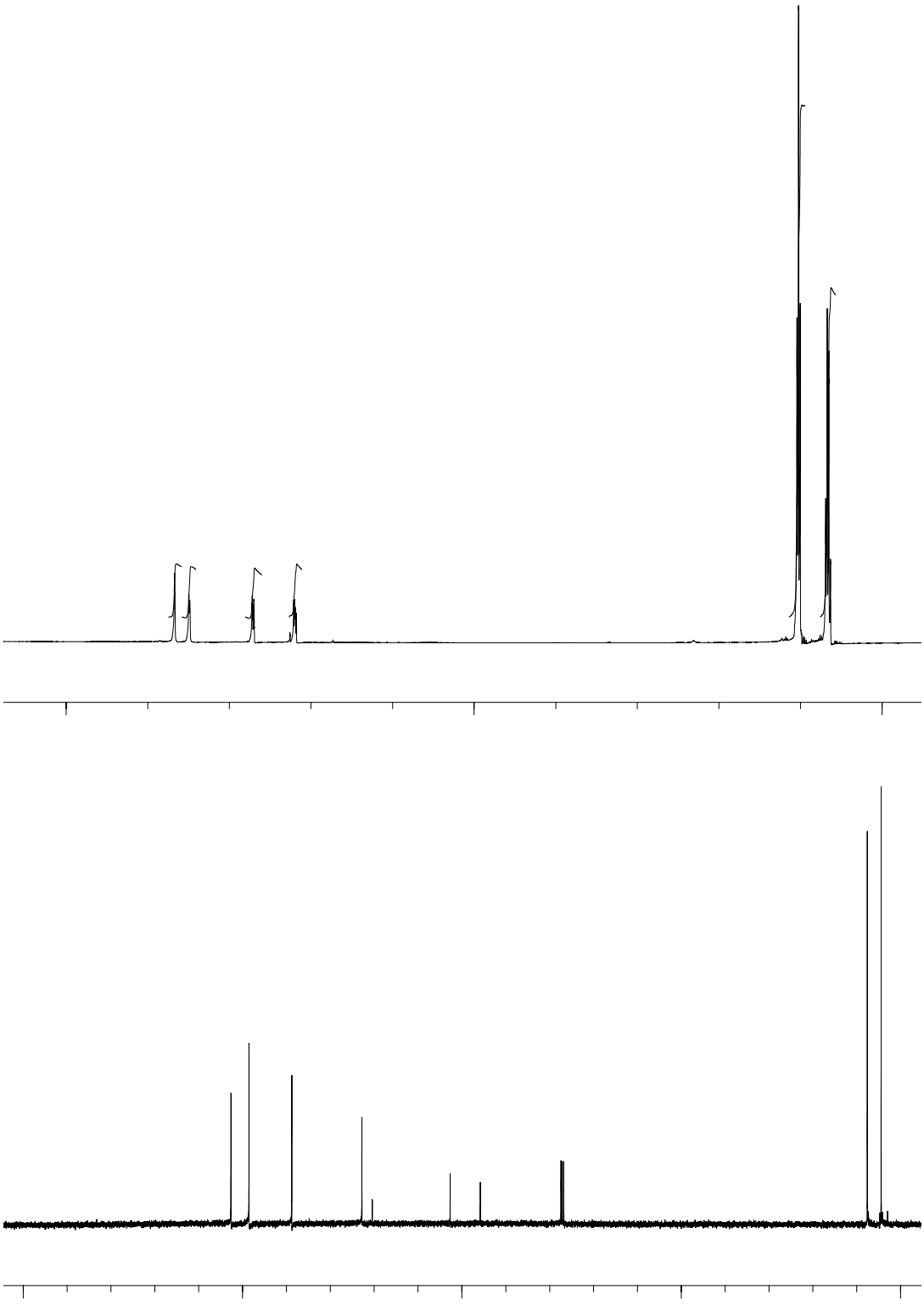
2-Chlorophenyl(triethylsilyl)acetylene (3j)



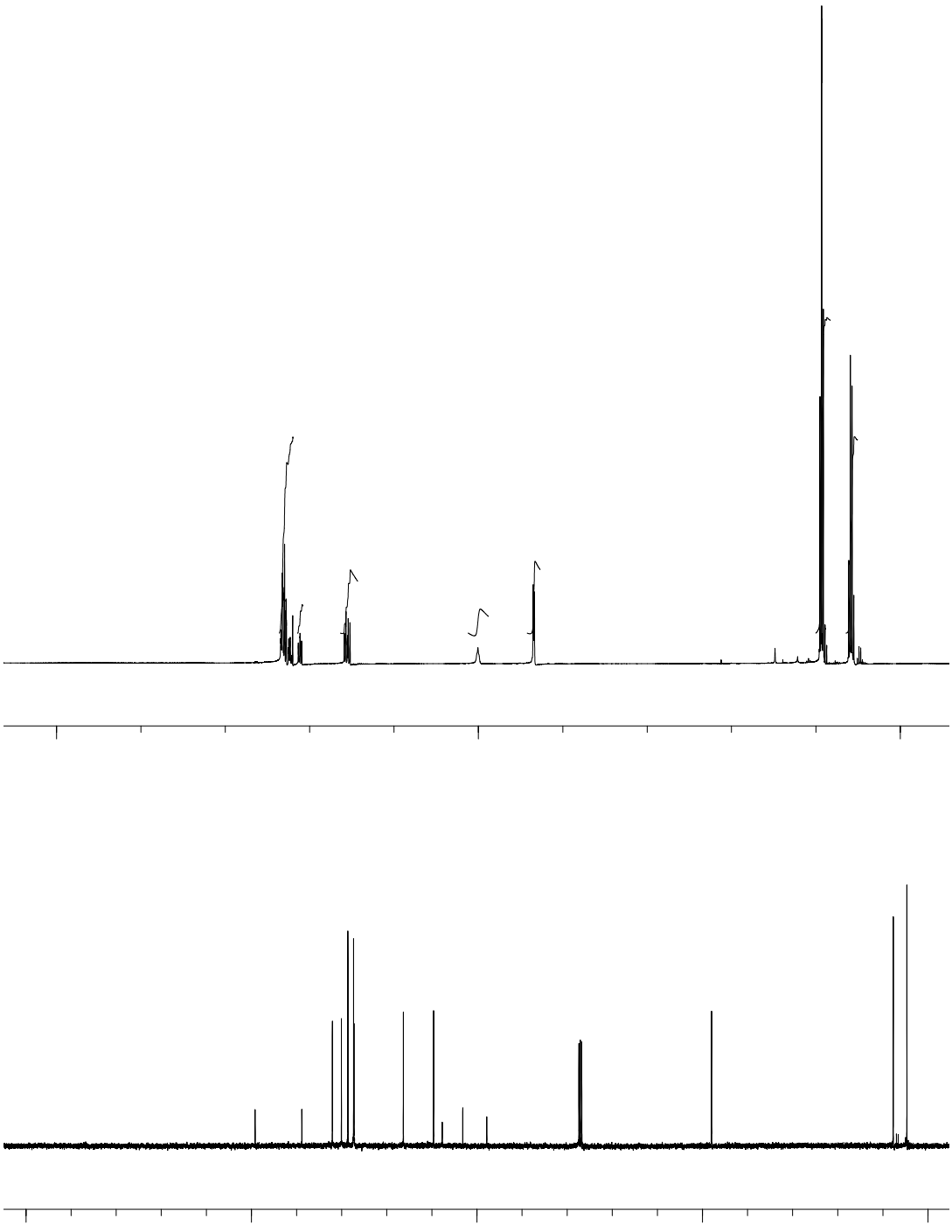
Triethylsilyl-2-thienylacetylene (3k)



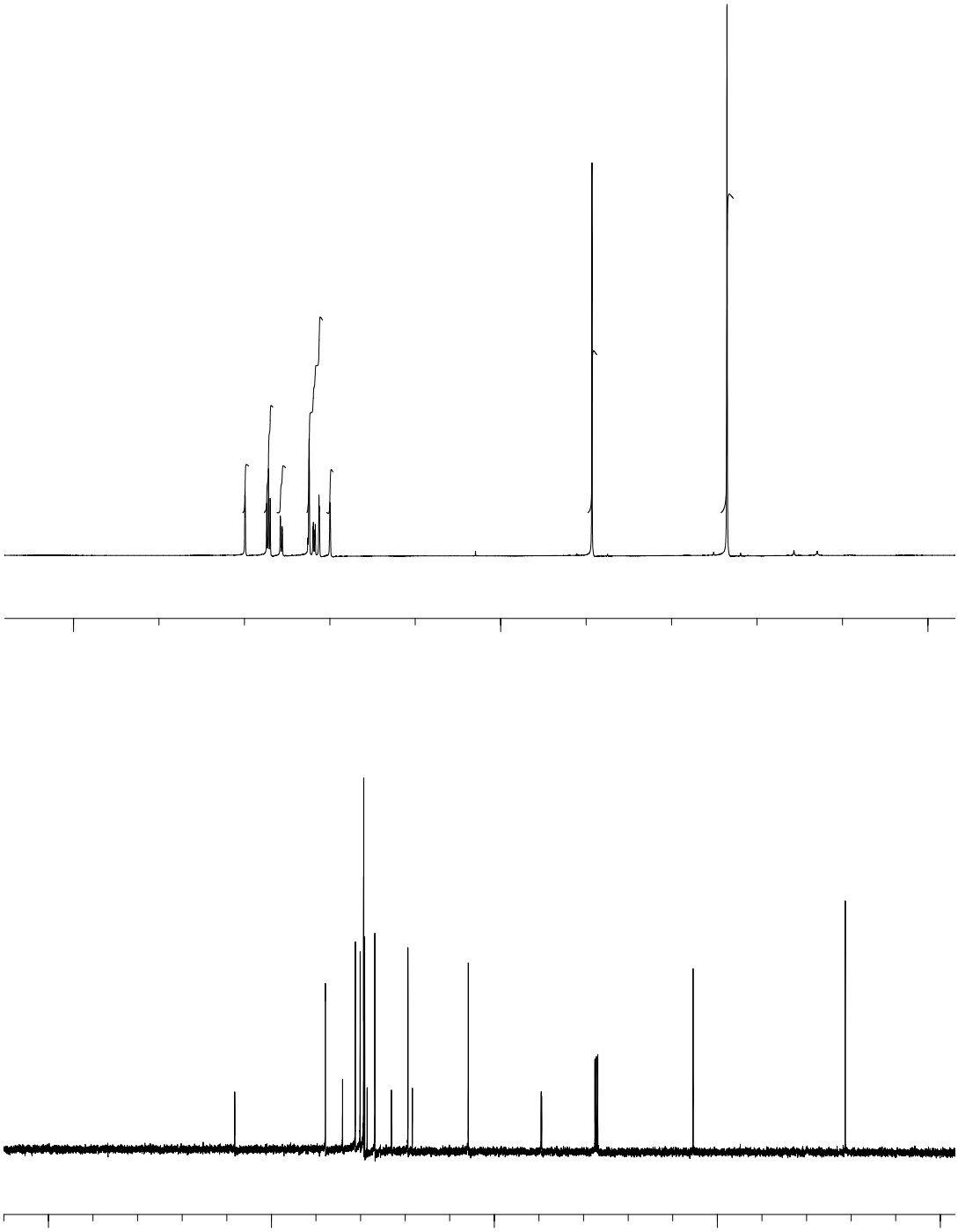
Triethylsilyl-3-pyridylacetylene (3l)



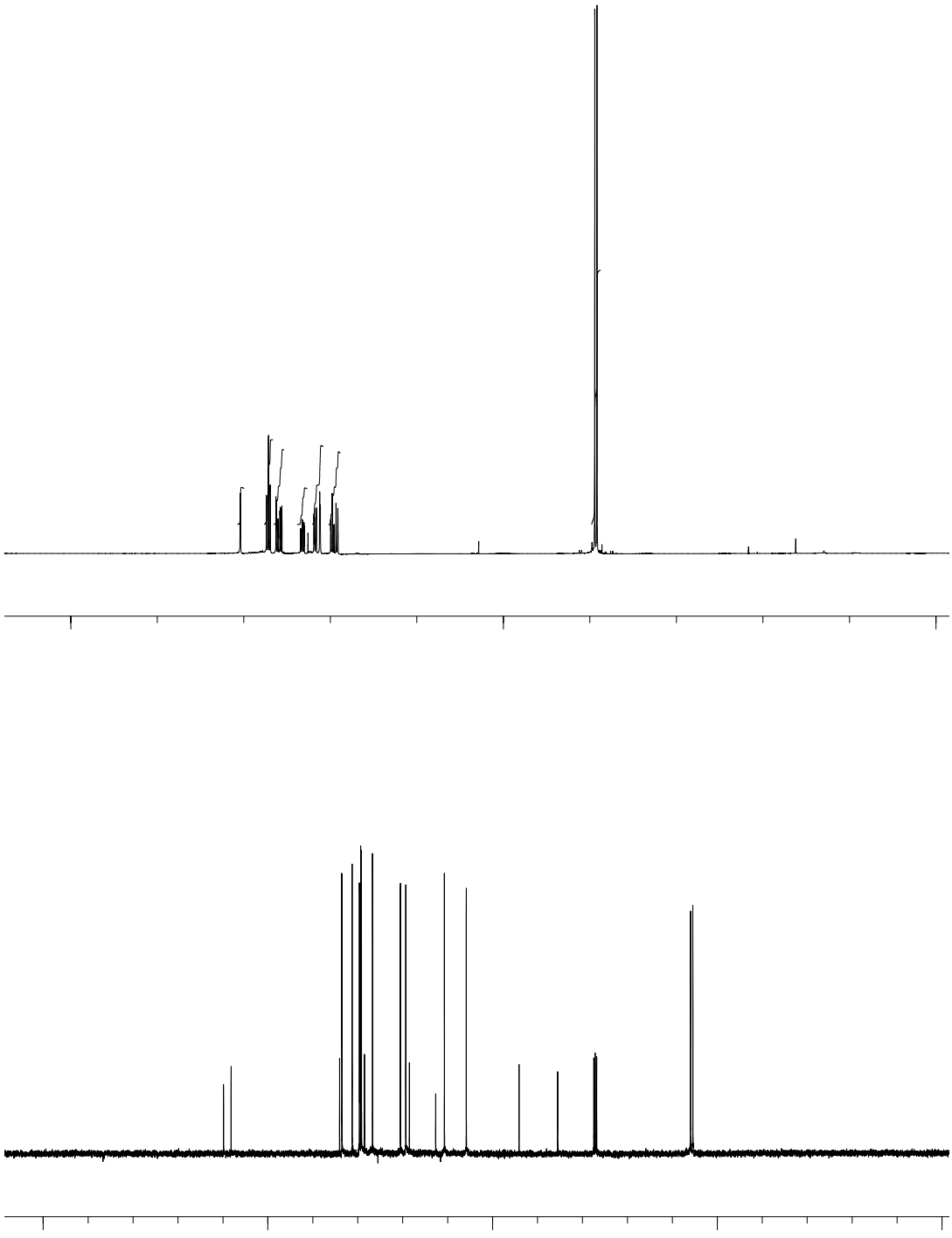
Triethylsilyl-2-(aminobenzyl)phenylacetylene (3m)



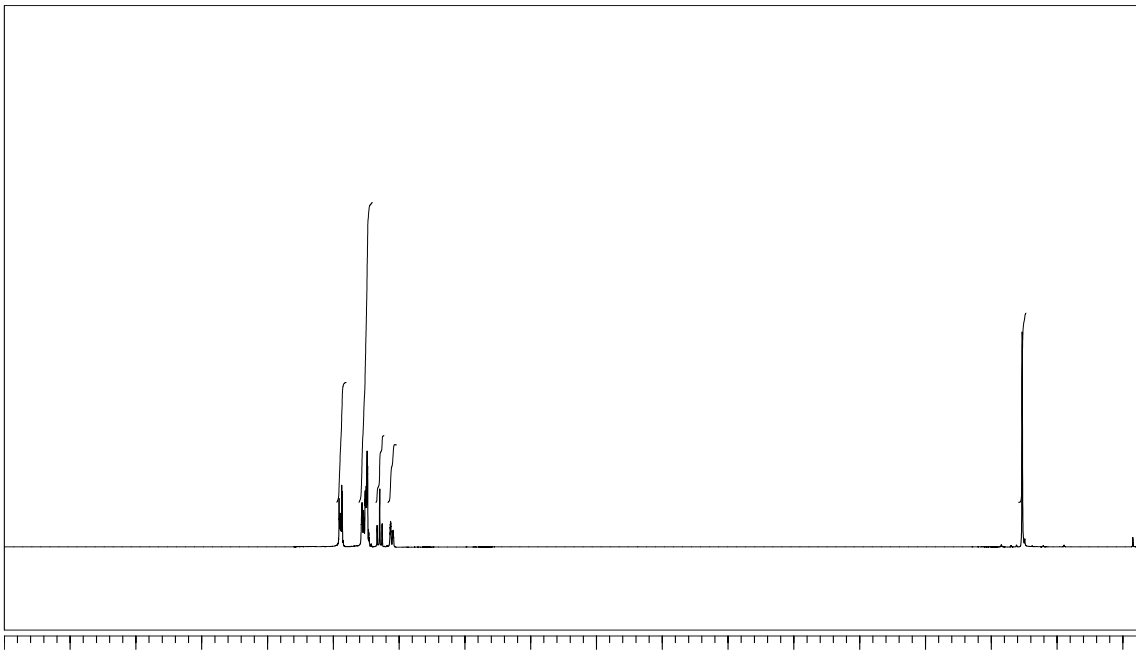
2-(3,5-Dimethylphenylethynyl)-6-methoxynaphthalene (3n)

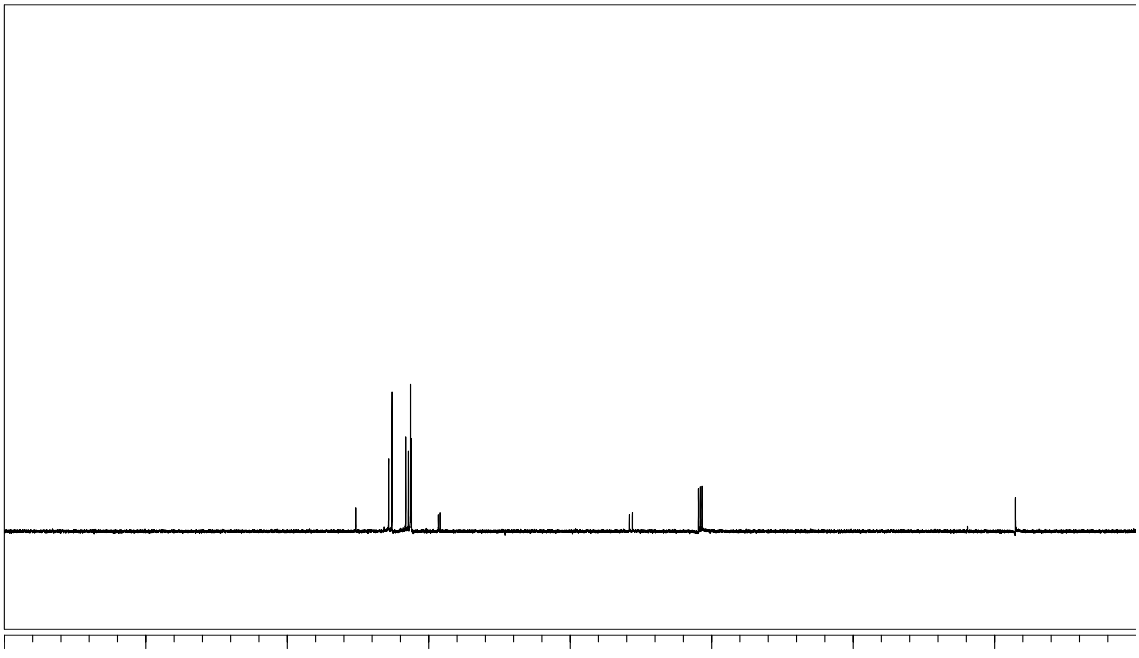


2-(2-Methoxyphenylethynyl)-6-methoxynaphthalene (3o)

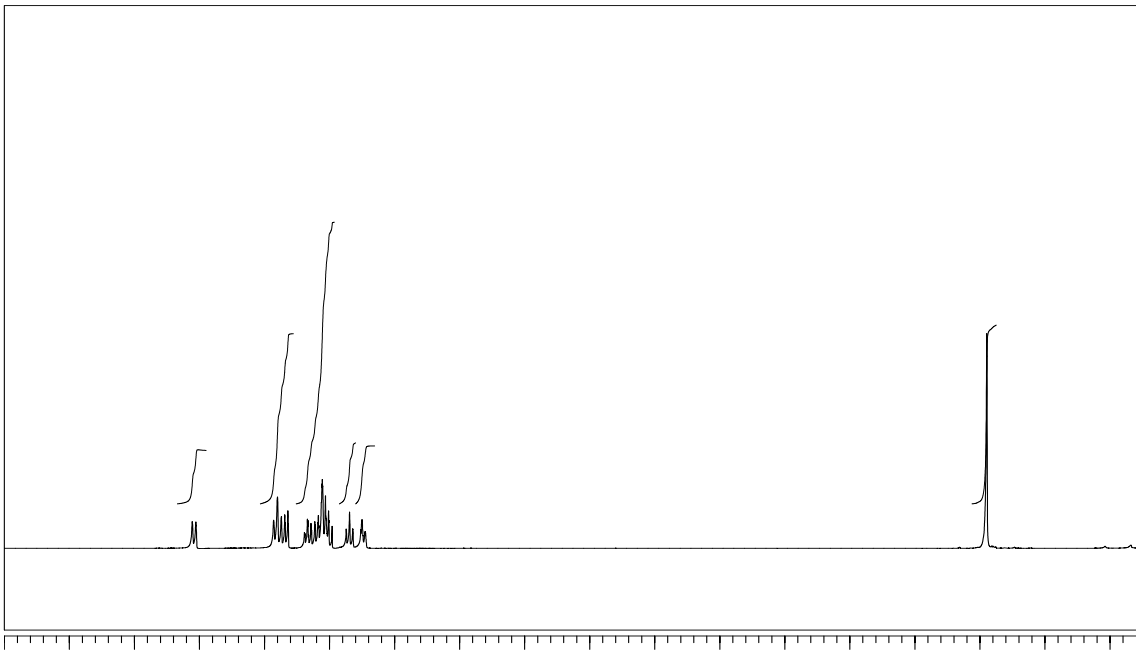


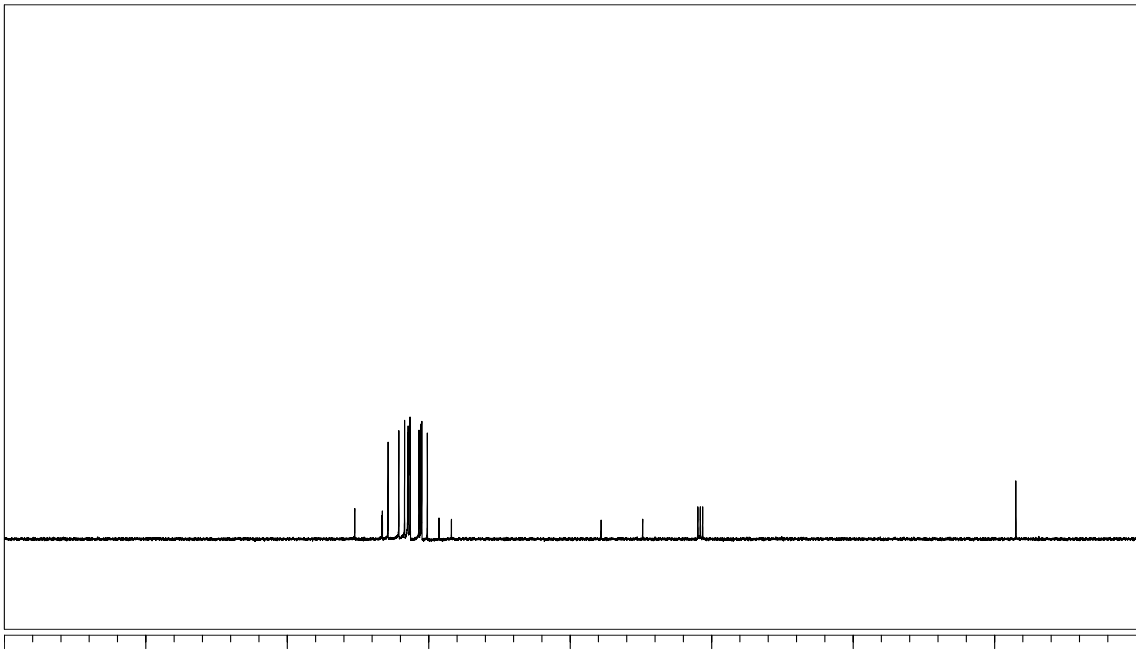
3-(Phenylethynyl)toluene (3p)



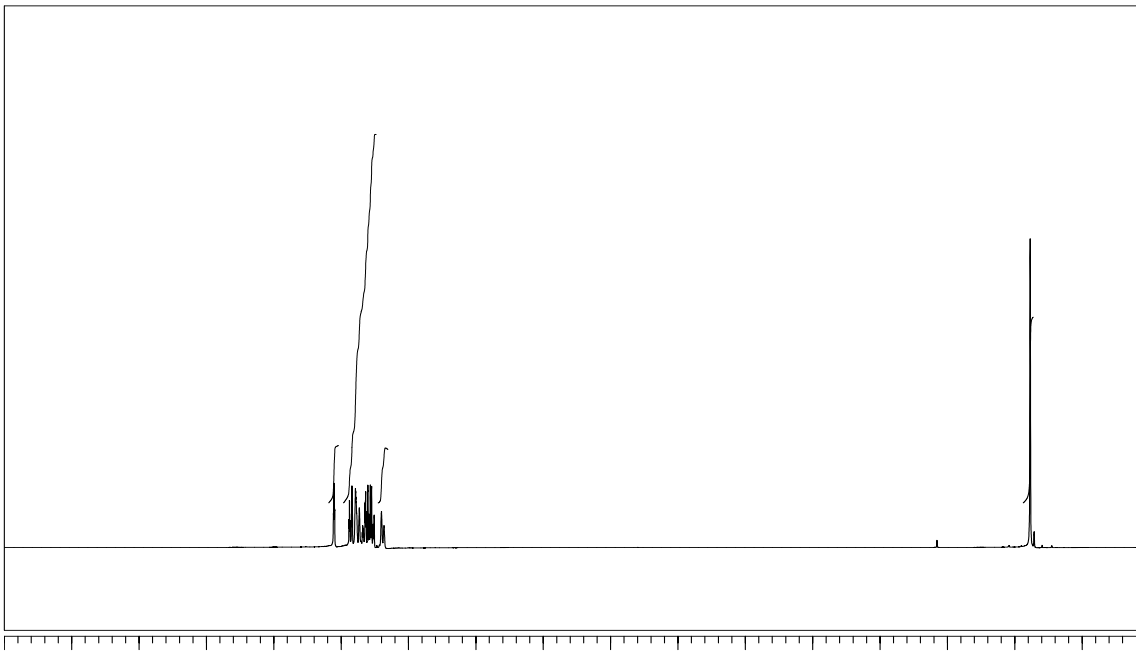


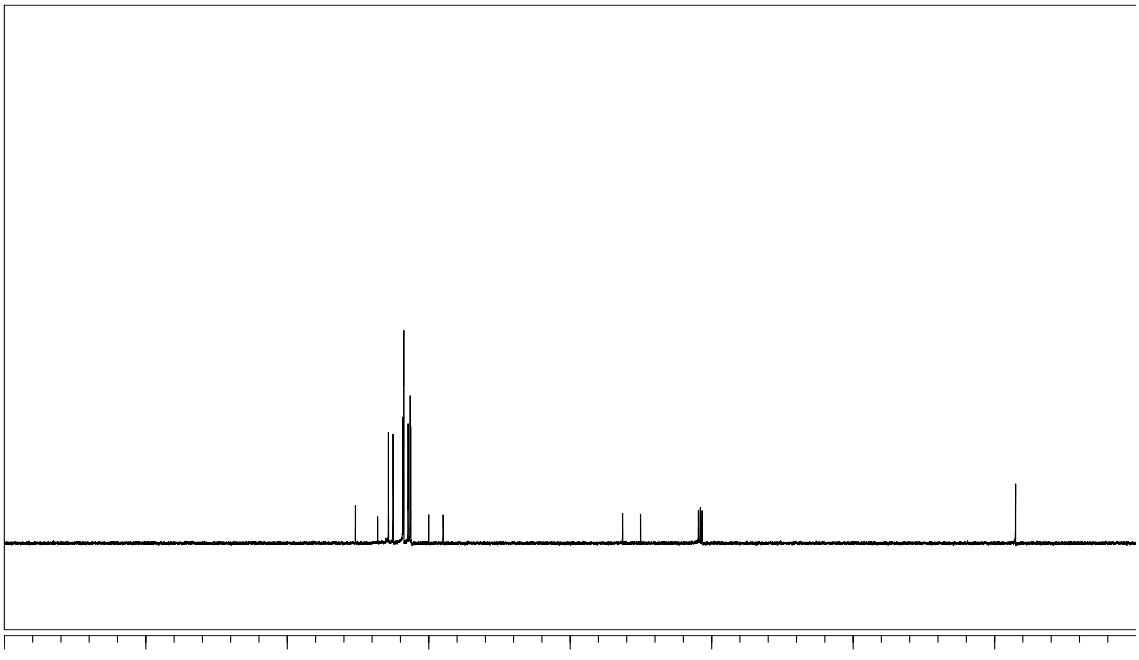
3-(1-Naphthylethynyl)toluene (3q)



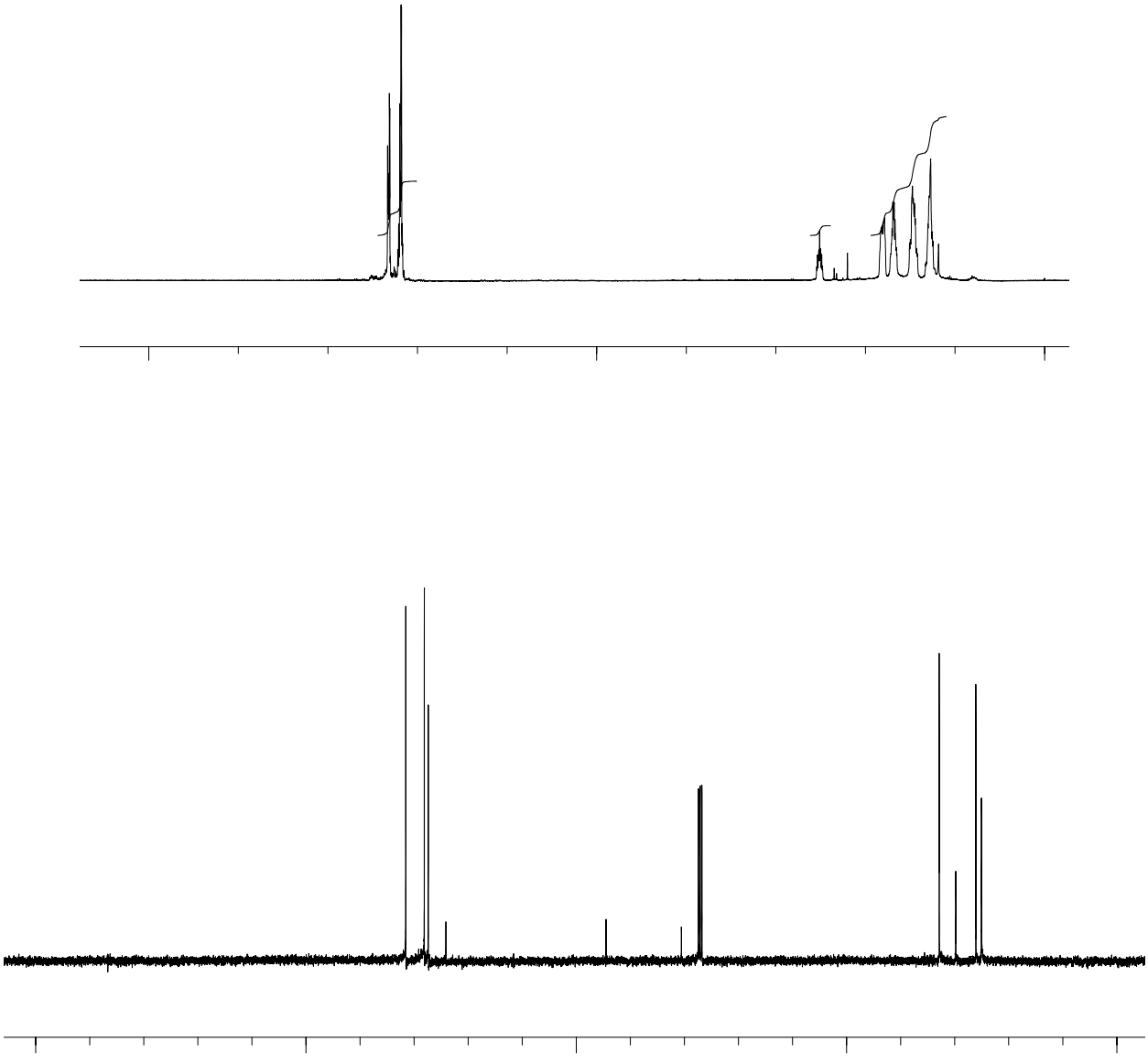


3-(3-Chlorophenylethynyl)toluene (3r)

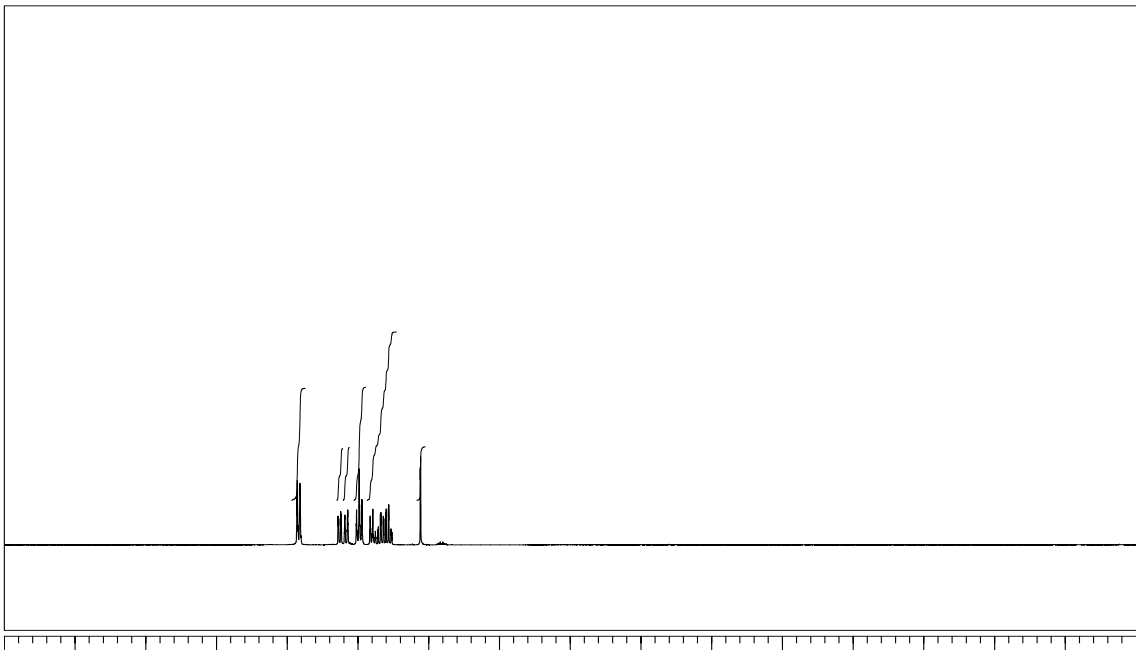


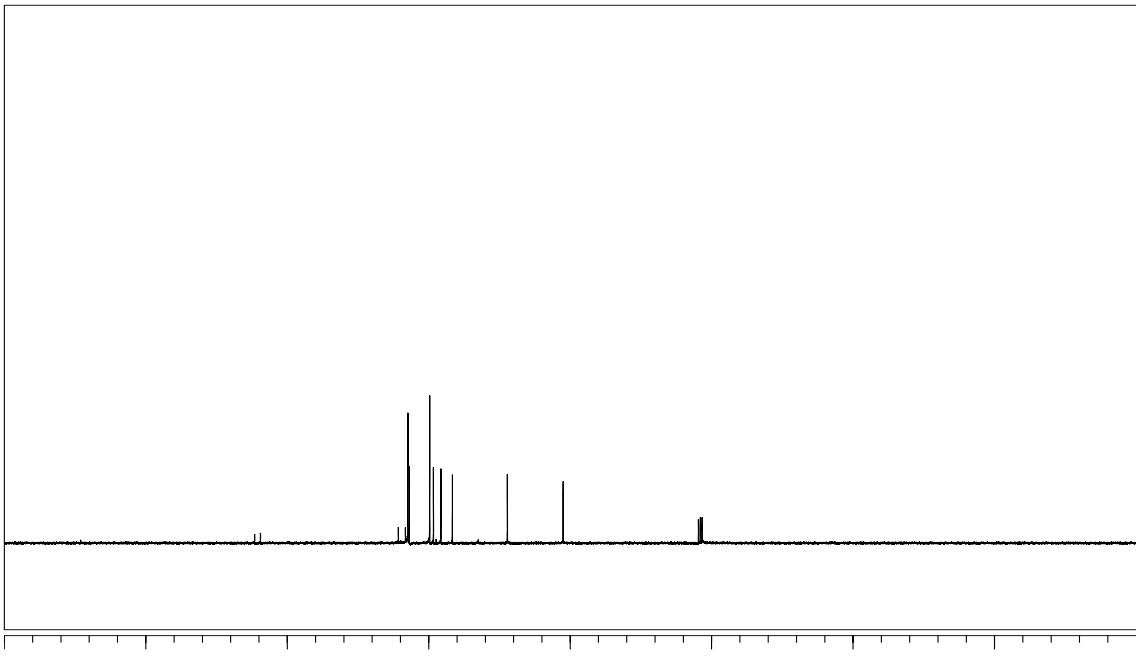


(Cyclohexyl)phenylacetylene (3s)



2-Phenylbenzo[*b*]furan (6a)





2-(3-Tolyl)benzo[*b*]furan (6b)