Supporting Information

One-pot synthesis of unsymmetrical diarylacetylenes *via* Sonogashira/Deacetonation/Sonogashira cross-coupling of two different aryl chlorides with 2-methyl-3-butyn-2-ol

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Experimental Section

General

GC analysis was performed on Agilent 4890D gas chromatograph. Melting points were measured using a WC-1 microscopic apparatus and were uncorrected. ^{1}H and ^{13}C spectra were recorded on a Bruker DPX-400 spectrometer with $CDCl_3$ as the solvent and TMS as an internal standard. Mass spectra were measured on an LC-MSD-Trap-XCT instrument. High-resolution mass spectra were measured on a MALDI-FTMS. Ethyl acetate and hexane (analytical grade) were used for column chromatography without purification. All chemicals were bought from commercial sources and used as-received unless otherwise noted.

General procedure for the one-pot synthesis of unsymmetrical diarylacetylenes *via* Sonogashira/deacetonation/Sonogashira (SDS) cross-coupling of two different aryl chlorides with 2-methyl-3-butyn-2-ol

A mixture of $Ar^{1}Cl$ (0.5 mmol), 2-methyl-3-butyn-2-ol (0.6 mmol), $Ar^{2}Cl$ (0.6 mmol), $PdCl_2$ (4 mol%), X-Phos (4 mol%) and K_2CO_3 (2 mmol) was dissolved in CH_3CN (2 mL) in a 10 mL vial under a nitrogen atmosphere. After the reaction was heated at 110 ^{o}C for 16 h, the mixture was filtered through a pad of Celite and washed with ethyl acetate. The mixture was added into H_2O (25 mL) and extracted with ethyl acetate (10 mL) three times. The combined organic layer was dried over anhydrous Na_2SO_4 and filtered. After removal of the solvent in vacuum, the residue was purified by flash chromatography on silica gel (hexane/ethyl acetate) to give the pure product.

General procedure for the Sonogashira cross-coupling of Aryl Chlrorides with phenylacetylene

A mixture of ArCl (0.5 mmol), terminal alkyne (0.6 mmol), $PdCl_2$ (2 mol%), X-Phos (4 mol%) and K_2CO_3 (0.75 mmol) was dissolved in CH_3CN (2 mL) in a 10 mL vial under a nitrogen atmosphere. After the reaction was heated at 110 ^{o}C for 16 h, the mixture was filtered through a pad of Celite and washed with ethyl acetate. The mixture was added into H_2O (25 mL) and extracted with ethyl acetate (10 mL) three times. The combined organic layer was dried over anhydrous Na_2SO_4 and filtered. After removal of the solvent in vacuum, the residue was purified by flash chromatography on silica gel (hexane/ethyl acetate) to give the pure product.

General procedure for the one-pot synthesis of symmetrical diarylacetylenes *via* Sonogashira/deacetonation/Sonogashira (SDS) cross-coupling of one aryl chloride with 2-methyl-3-butyn-2-ol

A mixture of 2-methyl-3-butyn-2-ol (0.5 mmol), ArCl (1.2 mmol), $PdCl_2$ (4 mol%), X-Phos (4 mol%) and K_2CO_3 (2 mmol) was dissolved in CH_3CN (2 mL) in a 10 mL vial under a nitrogen atmosphere. After the reaction was heated at 110 ^{o}C for 16 h, the mixture was filtered through a pad of Celite and washed with ethyl acetate. The mixture was added into H_2O (25 mL) and extracted with ethyl acetate (10 mL) three times. The combined organic layer was dried over anhydrous Na_2SO_4 and filtered. After removal of the solvent in vacuum, the residue was purified by flash chromatography on silica gel (hexane/ethyl acetate) to give the pure product.

2-Methyl-4-(4-nitrophenyl)but-3-yn-2-ol. (2ax) [1]

Yellow solid, mp 100–102 ^{o}C, yield: 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17 (d, J=8.8 Hz, 2H), 7.55 (d, J=8.8 Hz, 2H), 2.33 (s, 1H), 1.64 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.5, 132.8, 130.1, 123.9, 99.5, 80.8, 66.0, 31.6.

1-Methyl-4-(2-(4-nitrophenyl)ethynyl)benzene. (2ab) [2]

Yellow solid, mp 149–150 °C, yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.21 (d, J=8.8 Hz, 2H), 7.65 (d, J=8.8 Hz, 2H), 7.45 (d, J=8.0 Hz, 2H), 7.19 (d, J=8.0 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.2, 140.1, 132.6, 132.2, 130.9, 129.7, 124.0, 119.4, 95.5, 87.5, 22.0.

1-Methyl-3-(2-(4-nitrophenyl)ethynyl)benzene. (2ac) [3]

Yellow solid, mp 106–107 °C, yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.8 Hz, 2H), 7.66 (d, J=8.8 Hz, 2H), 7.40–7.35 (m, 2H), 7.30–7.26 (m, 1H), 7.21 (d, J=8.0 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.9, 137.3, 131.4, 131.2, 129.4, 129.2, 127.9, 127.4, 122.6, 120.9, 94.0, 86.2, 20.2.

1-Methyl-2-(2-(4-nitrophenyl)ethynyl)benzene. (2ad)

Yellow solid, mp 78–79 °C, yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.24 (d, J=8.8 Hz, 2H), 7.67 (d, J=8.8 Hz, 2H), 7.52 (d, J=7.6 Hz, 1H), 7.33–7.27 (m, 2H), 7.21 (t, J=7.0 Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.9, 139.6, 131.1, 131.1, 129.5, 128.7, 128.3, 124.8, 122.6, 120.8, 92.8, 90.4, 19.7; HRMS: $[M+H]^+$ calcd. for $C_{15}H_{12}NO_2^+$, m/z 238.0863, Found: 238.0858.

1,2-Dimethyl-4-(2-(4-nitrophenyl)ethynyl)benzene. (2ae) [4]

Yellow solid, mp 102–103 °C, yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.20 (d, J=8.8 Hz, 2H), 7.64 (d, J=8.8 Hz, 2H), 7.35–7.28 (m, 2H), 7.14 (d, J=7.6 Hz, 1H), 2.29 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8, 137.5, 135.9, 131.8, 131.1, 129.6, 128.8, 128.3, 122.6, 118.3, 94.3, 85.9, 18.9, 18.6.

1,4-Dimethyl-2-(2-(4-nitrophenyl)ethynyl)benzene. (2af) [5]

Yellow solid, mp 94–95 °C, yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.8 Hz, 2H), 7.65 (d, J=8.4 Hz, 2H), 7.35 (s, 1H), 7.16–7.09 (m, 2H), 2.48 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8, 136.5, 134.3, 131.5, 131.0, 129.5, 129.3, 128.5, 122.6, 120.6, 93.1, 90.1, 19.7, 19.2.

1,3-Dimethyl-2-(2-(4-nitrophenyl)ethynyl)benzene. (2ag)

Yellow solid, mp 90–91 °C, yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27 (d, J=8.8 Hz, 2H), 7.66 (d, J=8.8 Hz, 2H), 7.21–7.16 (m, 1H), 7.11–7.08 (m, 2H), 2.52 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8, 139.7, 131.0, 129.7, 127.8, 125.9, 122.7, 120.8, 94.8, 91.7, 20.1; HRMS: $[M+H]^+$ calcd. for $C_{16}H_{14}NO_2^+$, m/z 252.1019, Found: 252.1020.

1,3,5-Trimethyl-2-(2-(4-nitrophenyl)ethynyl)benzene.(2ah) [5]

Yellow solid, mp 89–90 °C, yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.21 (d, J=8.8 Hz, 2H), 7.64 (d, J=8.8 Hz, 2H), 6.91 (s, 2H), 2.48 (s, 6H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.7, 139.6, 138.1, 130.9, 130.0, 126.8, 122.7, 117.9, 94.3, 92.1, 20.4, 19.9.

1-Methoxy-4-((4-nitrophenyl)ethynyl)benzene. (2ai) [6]

Yellow solid, mp 111–112 °C, yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.21 (d, J=8.4 Hz, 2H), 7.63 (d, J=8.8 Hz, 2H), 7.50 (d, J=8.8 Hz, 2H), 6.91 (d, J=8.4 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.1, 145.4, 132.1, 130.7, 129.4, 122.3, 112.9, 112.8, 93.8, 85.3, 54.1.

Methyl 4-(2-(2,6-dimethylphenyl)ethynyl)benzoate. (2jg)

Pale yellow solid, mp 70–71 °C, yield: 93%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, J=8.4 Hz, 2H), 7.59 (d, J=8.0 Hz, 2H), 7.18–7.13 (m, 1H), 7.09–7.06 (m, 2H), 3.93 (s, 3H), 2.52 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 139.5, 130.2, 128.5, 128.3, 127.5, 127.3, 125.8, 121.4, 96.0, 89.2, 51.2, 20.1; HRMS: $[M+H]^+$ calcd. for $C_{18}H_{17}O_2^+$, m/z 265.1223, Found: 265.1224.

Methyl 4-(2-mesitylethynyl)benzoate. (2jh)

Pale yellow solid, mp 106–107 °C, yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01 (d, J=8.4 Hz, 2H), 7.57 (d, J=8.0 Hz, 2H), 6.90 (s, 2H), 3.93 (s, 3H), 2.48 (s, 6H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 139.4, 137.4, 130.1, 128.5, 128.1, 127.7, 126.7, 118.4, 95.3, 89.5, 51.2, 20.4, 20.0; HRMS: $[M+H]^+$ calcd. for $C_{19}H_{19}O_2^+$, m/z 279.1380, Found: 279.1382.

Methyl 4-((4-methoxyphenyl)ethynyl)benzoate.(2ji) [7]

Pale yellow solid, mp 145–146 °C, yield: 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 (d, J=8.4 Hz, 2H), 7.56 (d, J=8.0 Hz, 2H), 7.48 (d, J=8.4 Hz, 2H), 6.89 (d, J=8.0 Hz, 2H), 3.92 (s, 3H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 159.0, 132.2, 130.3, 128.5, 128.1, 127.4, 113.8, 113.1, 91.6, 86.5, 54.3, 51.2.

4-(2-(2,6-Dimethylphenyl)ethynyl)benzonitrile. (2kg)

Pale yellow solid, mp 82–83 °C, yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62 (q, J=7.8 Hz, 4H), 7.20–7.15 (m, 1H), 7.10–7.07 (m, 2H), 2.50 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.6, 131.1, 130.8, 127.7, 127.7, 125.9, 120.9, 117.6, 110.3, 95.0, 90.7, 20.1; HRMS: $[M+H]^+$ calcd. for $C_{17}H_{14}N^+$, m/z 232.1121, Found: 232.1112.

4-(2-Mesitylethynyl)benzonitrile. (2kh)

Pale yellow, solid, mp 128–129 °C, yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64–7.57 (m, 4H), 6.91 (s, 2H), 2.47 (s, 6H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.5, 137.9, 131.0, 130.7, 128.0, 126.8, 118.0, 117.7, 110.0, 94.4, 91.1, 20.4, 19.9; HRMS: $[M+H]^+$ calcd. for $C_{18}H_{16}N^+$, m/z 246.1277, Found: 246.1268.

4-(2-(4-Methoxyphenyl)ethynyl)benzonitrile. (2ki)

Pale yellow solid, mp 118–119 °C, yield: 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63–7.56 (m, 4H), 7.48 (d, J=8.4 Hz, 2H), 6.90 (d, J=8.4 Hz, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.3, 132.3, 131.0, 130.8, 127.6, 117.6, 113.3, 113.2, 110.0, 93.1, 85.7, 54.3; HRMS: $[M+H]^+$ calcd. for $C_{16}H_{12}NO^+$, m/z 234.0913, Found: 234.0903.

4-(2-(2,6-Dimethylphenyl)ethynyl)benzaldehyde. (2lg)

White solid, mp 41–42 °C, yield: 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.02 (s, 1H), 7.86 (d, J=8.4 Hz, 2H), 7.70 (d, J=8.4 Hz, 2H), 7.19–7.14 (m, 1H), 7.09–7.07 (m, 2H), 2.52 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.4, 139.6, 134.3, 130.8, 129.1, 128.6, 127.5, 125.9, 121.2, 95.9, 90.4, 20.1; HRMS: $[M+H]^+$ calcd. for $C_{17}H_{15}O^+$, m/z 235.1117, Found: 235.1117.

1-Nitro-4-(phenylethynyl)benzene. (2am) [6]

Yellow solid, mp 110–112 °C, yield: 99% (Scheme 5) and 97% (Scheme 8); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.8 Hz, 2H), 7.67 (d, J=8.4 Hz, 2H), 7.58–7.55 (m, 2H), 7.41–

7.38 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.2, 133.5, 133.1, 131.5, 130.5, 129.8, 124.8, 133.3, 95.9, 88.8.

1-(2-(4-Nitrophenyl)ethynyl)naphthalene. (2an)

Yellow solid, mp 119–120 ^{o}C, yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.41 (d, *J*=8.4 Hz, 1H), 8.29 (d, *J*=8.4 Hz, 2H), 7.96–7.91 (m, 2H), 7.85–7.79 (m, 3H), 7.69–7.64 (m, 1H), 7.60 (t, *J*=7.4 Hz, 1H), 7.52 (t, *J*=7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.0, 132.2, 132.1, 131.3, 130.1, 129.3, 128.9, 127.5, 126.2, 125.7, 124.8, 124.3, 122.7, 118.7, 91.9, 91.3; HRMS: $[M+H]^+$ calcd. for $C_{18}H_{12}NO_2^+$, m/z 274.0863, Found: 274.0861.

1-(2-(4-Nitrophenyl)ethynyl)-4-vinylbenzene. (2ao)

Yellow solid, mp 149–151 ^{o}C, yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, *J*=8.8 Hz, 2H), 7.66 (d, *J*=8.8 Hz, 2H), 7.52 (d, *J*=8.0 Hz, 2H), 7.42 (d, *J*=8.0 Hz, 2H), 6.73 (dd, *J*=17.6 Hz, 10.8 Hz, 1H), 5.82 (d, *J*=17.6 Hz, 1H), 5.34 (d, *J*=10.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.0, 138.5, 136.1, 132.3, 132.1, 130.3, 126.4, 123.7, 121.3, 115.5, 94.8, 88.2; HRMS: $[M+H]^+$ calcd. for $C_{16}H_{12}NO_2^+$, m/z 250.0863, Found: 250.0864.

1-(2-(4-Nitrophenyl)ethynyl)-2-vinylbenzene. (2ap)

Yellow solid, mp 90–91 ^{o}C, yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.8 Hz, 2H), 7.68–7.61 (m, 3H), 7.54 (d, *J*=6.8 Hz, 1H), 7.37 (t, *J*= 7.4 Hz, 1H), 7.29–7.21 (m, 2H), 5.87 (d, *J*=17.2 Hz, 1H), 5.43 (d, *J*=11.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.0, 139.5, 134.5, 132.8, 132.2, 130.3, 129.5, 127.7, 124.9, 123.7, 120.7, 116.4, 93.2, 92.0; HRMS: $[M+H]^+$ calcd. for $C_{16}H_{12}NO_2^+$, m/z 250.0863, Found: 250.0861.

1-Nitro-4-((4-(trifluoromethyl)phenyl)ethynyl)benzene. (2aq) [8]

Yellow solid, mp 110–111 ^{o}C, yield: 99%;^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, *J*=8.8 Hz, 2H), 7.71–7.63 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.4, 131.5, 131.1, 129.9 (q, *J*=32.7 Hz), 128.4, 124.9, 124.5 (q, *J*=3.8 Hz), 122.7 (q, *J*=270.8 Hz), 122.7, 91.8, 88.5.

Methyl 4-((4-(trifluoromethyl)phenyl)ethynyl)benzoate. (2jq) [9]

Pale yellow solid, mp 136–137 ^{o}C, yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.80–7.76 (m, 1H), 8.58 (d, *J*=8.0 Hz, 1H), 8.04 (d, *J*=8.4 Hz, 2H), 7.82 (d, *J*=7.6 Hz, 1H), 7.61 (d, *J*=8.4 Hz, 2H), 7.32–7.28 (m, 1H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.4, 130.9, 130.6, 129.4 (q, *J*=32.2 Hz), 129.0, 128.6, 126.2, 125.5, 124.4 (q, *J*=3.8 Hz), 122.8 (q, *J*=270.5 Hz), 89.8, 89.6, 51.3.

4-((4-(Trifluoromethyl)phenyl)ethynyl)benzonitrile. (2kq)

Pale yellow solid, mp 114–115 ^{o}C, yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68–7.61 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 131.2, 131.1, 131.0, 129.8 (q, *J*=32.8 Hz), 126.4, 125.0, 124.4 (q, *J*=3.8 Hz), 122.8 (q, *J*=270.7 Hz), 117.3, 111.1, 91.0, 88.7; HRMS: $[M+H]^+$ calcd. for $C_{16}H_9F_3N^+$, m/z 272.0682, Found: 272.0674.

4-((4-(Trifluoromethyl)phenyl)ethynyl)benzaldehyde. (2lq) [10]

Pale yellow solid, mp 93–94 ^{o}C, yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.4 (s, 1H), 7.89 (d, *J*=8.0 Hz, 2H), 7.71–7.62 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.3, 134.8, 131.3, 131.0, 129.6 (q, *J*=32.9 Hz), 128.6, 127.7, 125.3, 124.4 (q, *J*=3.8 Hz), 122.8 (q, *J*=270.6 Hz), 90.6, 89.6.

2-(2-(4-Nitrophenyl)ethynyl)thiophene. (2ar)

Yellow solid, mp 137–138 °C, yield: 69%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.0 Hz, 2H), 7.64 (d, J=8.4 Hz, 2H), 7.40–7.36 (m, 2H), 7.08–7.04 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.9, 132.2, 130.9, 128.9, 127.8, 126.4, 122.7, 121.0, 90.3, 87.1; HRMS: $[M+H]^+$ calcd. for $C_{12}H_8NO_2S^+$, m/z 230.0270, Found: 230.0269.

3-(2-(4-Nitrophenyl)ethynyl)pyridine. (2as) [11]

Yellow solid, mp 125–126 °C, yield: 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.80 (s, 1H), 8.61 (d, J=4.0 Hz, 1H), 8.25 (d, J=8.8 Hz, 2H), 7.85 (d, J=8.0 Hz, 1H), 7.70 (d, J=8.8 Hz, 2H), 7.36–7.32 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.4, 148.4, 146.3, 137.7, 131.4, 128.3, 122.7, 122.2, 118.3, 89.9, 89.5.

Methyl 4-(2-(thiophen-2-yl)ethynyl)benzoate. (2jr)

Yellow solid, mp 104–105 °C, yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, J=8.4 Hz, 2H), 7.57 (d, J=8.4 Hz, 2H), 7.35–7.31 (m, 2H), 7.04 (t, J=4.4 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.5, 131.5, 130.2, 128.5, 128.5, 127.0, 126.6, 126.2, 121.7, 91.3, 84.6, 51.2; HRMS: $[M+H]^+$ calcd. for $C_{14}H_{11}O_2S^+$, m/z 243.0474, Found: 243.0474.

Methyl 4-(2-(pyridin-3-yl)ethynyl)benzoate. (2js) [12]

Pale yellow solid, mp 113–114 °C, yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.04 (d, J=8.0 Hz, 2H), 7.66–7.59 (m, 6H), 3.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.4, 151.3, 148.0, 137.5, 130.6, 129.0, 128.6, 126.1, 122.1, 118.9, 90.7, 87.7, 51.3.

4-(2-(Thiophen-2-yl)ethynyl)benzonitrile. (2kr) [13]

Pale yellow solid, mp 134–135 °C, yield: 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65–7.57 (m, 4H), 7.38–7.33 (m, 2H), 7.05 (t, J=4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 132.0, 131.1, 130.7, 127.5, 126.9, 126.3, 121.1, 117.5, 110.5, 90.4, 86.1.

1,3-Dimethyl-2-(o-tolylethynyl)benzene. (2gd) [14]

White solid, mp 57–58 °C, yield: 38% in Scheme 5 and 78% in Scheme 6; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, J=7.2 Hz, 1H), 7.27–7.09 (m, 6H), 2.56 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.2, 139.8, 131.9, 129.6, 128.2, 127.7, 126.8, 125.7, 123.7, 123.3, 97.0, 91.1, 21.3, 21.1.

1,2-Bis(2,5-dimethylphenyl)ethyne. (2ff) [15]

White solid, mp 116–117 °C, yield: 67%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.31 (s, 2H), 7.09 (d, J=7.6 Hz, 2H), 7.01 (d, J=7.6 Hz, 2H), 2.46 (s, 6H), 2.29 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 135.9, 134.1, 131.3, 128.4, 128.1, 122.2, 91.2, 19.8, 19.5.

1,2-Bis(4-(trifluoromethyl)phenyl)ethyne. (2qq) [16]

White solid, mp 107–108 °C, yield: 85%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67–7.61 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 131.0, 129.5 (q, J=32.6 Hz), 125.4, 124.4 (q, J=3.8 Hz), 122.8 (q, J=272.3 Hz), 89.1.

1-methyl-2-(phenylethynyl)benzene. (2dm) [17]

Colorless oil, yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54–7.47 (m, 3H), 7.32–7.30 (m, 3H), 7.21–7.12 (m, 3H), 2.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.3, 131.9, 131.6, 129.6, 128.5, 128.4, 128.3, 125.7, 123.7, 123.1, 93.5, 88.5, 20.8.

1,3-dimethyl-2-(phenylethynyl)benzene. (2gm) [17]

Colorless oil, yield: 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55–7.52 (m, 2H), 7.34–7.29 (m, 3H), 7.12–7.03 (m, 3H), 2.51 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.3, 131.5, 128.4, 128.2, 127.9, 126.8, 123.9, 123.1, 97.9, 87.2, 21.2.

2-(phenylethynyl)thiophene. (2rm) [18]

Pale yellow solid, mp 49–50 ^{o}C, yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53–7.49 (m, 2H), 7.35–7.32 (m, 3H), 7.28 (d, J=4.4 Hz, 2H), 7.00 (t, J=4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 130.4, 127.4, 127.3, 126.2, 126.1, 122.2, 121.9, 92.0, 81.6.

3-(phenylethynyl)pyridine. (2sm) [18]

Colorless oil, yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.77 (s, 1H), 8.55 (d, J=4.8 Hz, 1H), 7.81 (d, J=8.0 Hz, 1H), 7.56–7.54 (m, 2H), 7.37 (t, J=3.2 Hz, 3H), 7.31–7.27 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.3, 148.6, 138.5, 131.7, 128.8, 128.5, 123.1, 122.5, 120.5, 92.6, 85.9.

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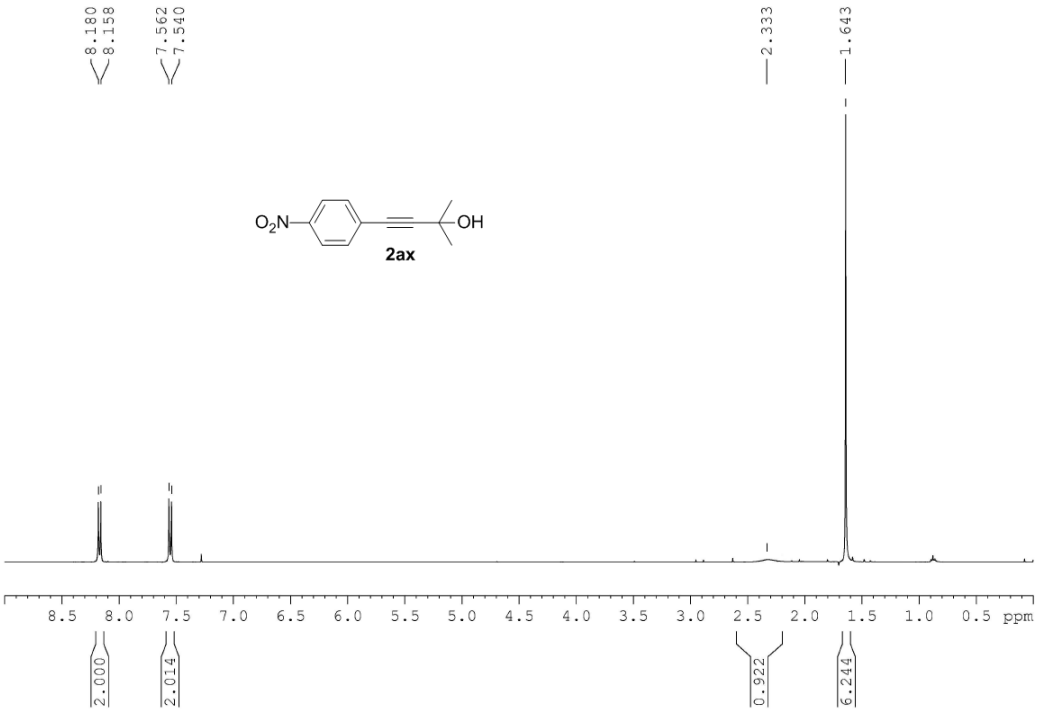
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OH
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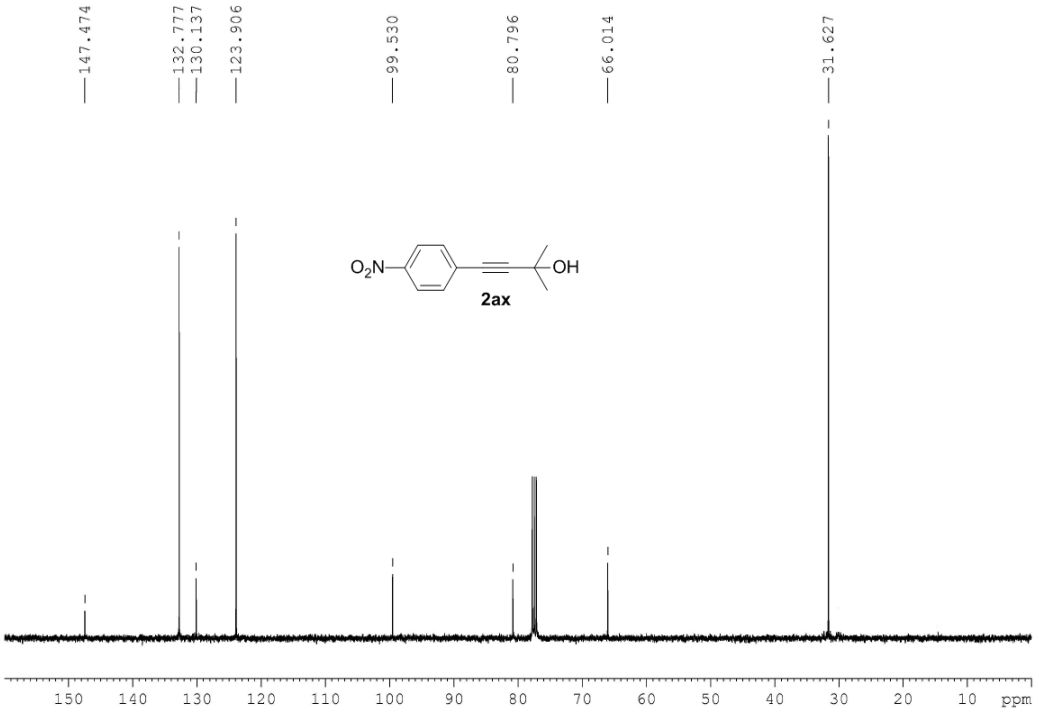
6.244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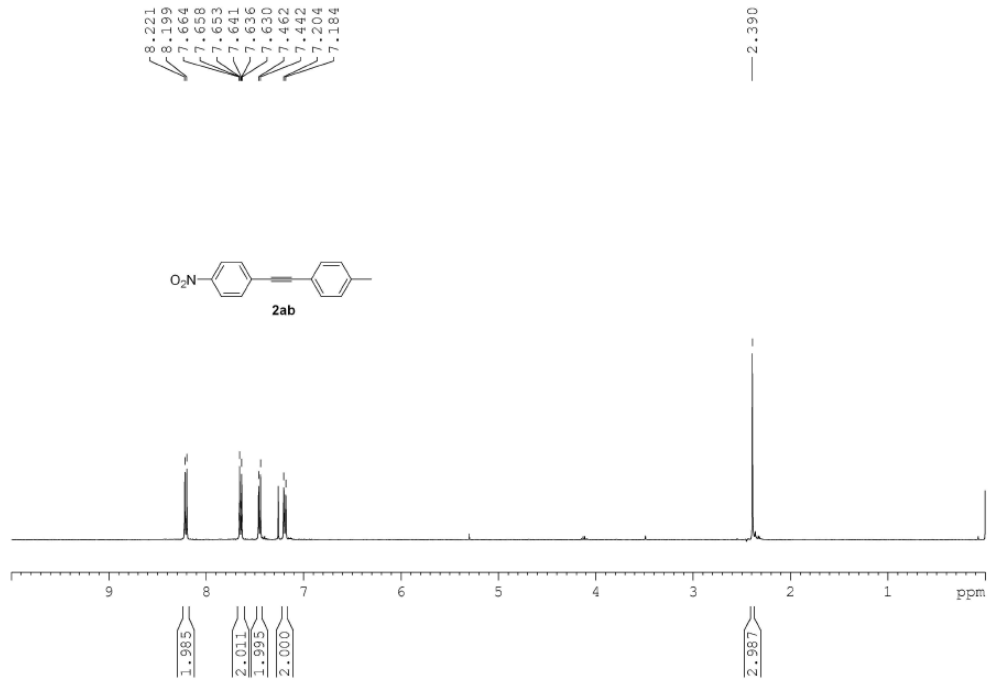
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2.390
O2N
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6
5
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3
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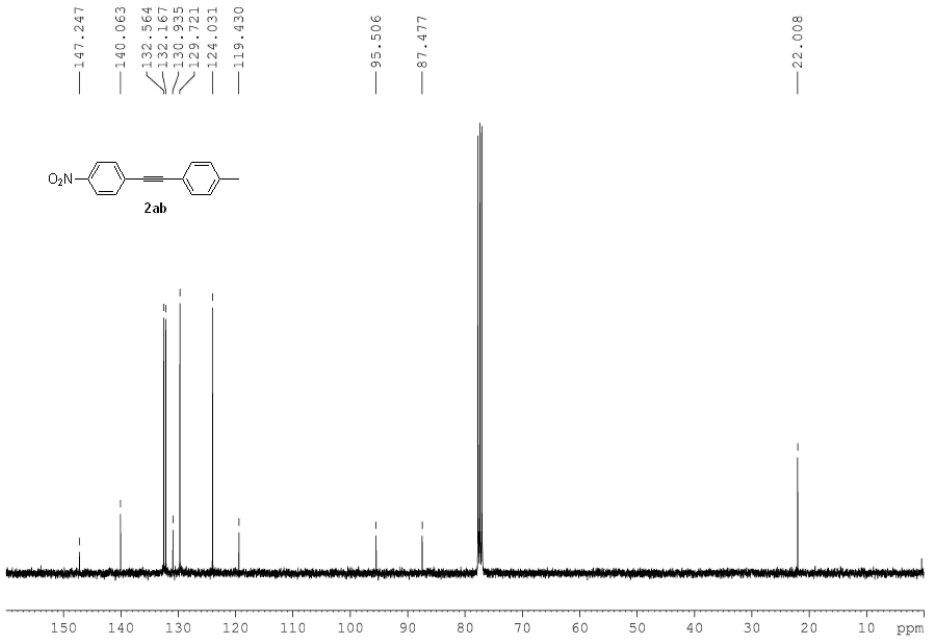
2.987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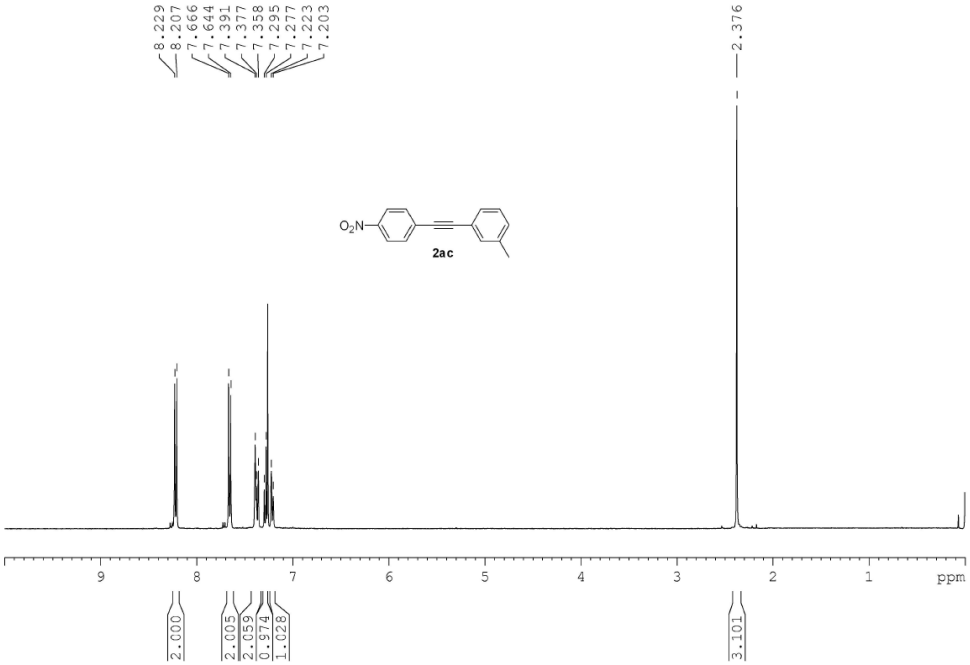
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2ac
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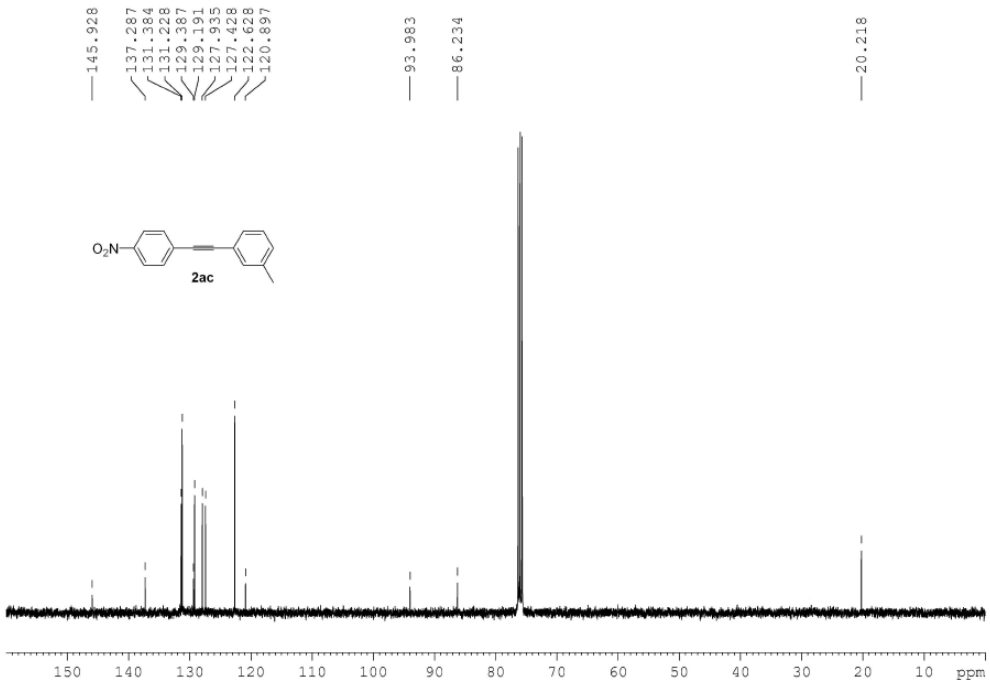
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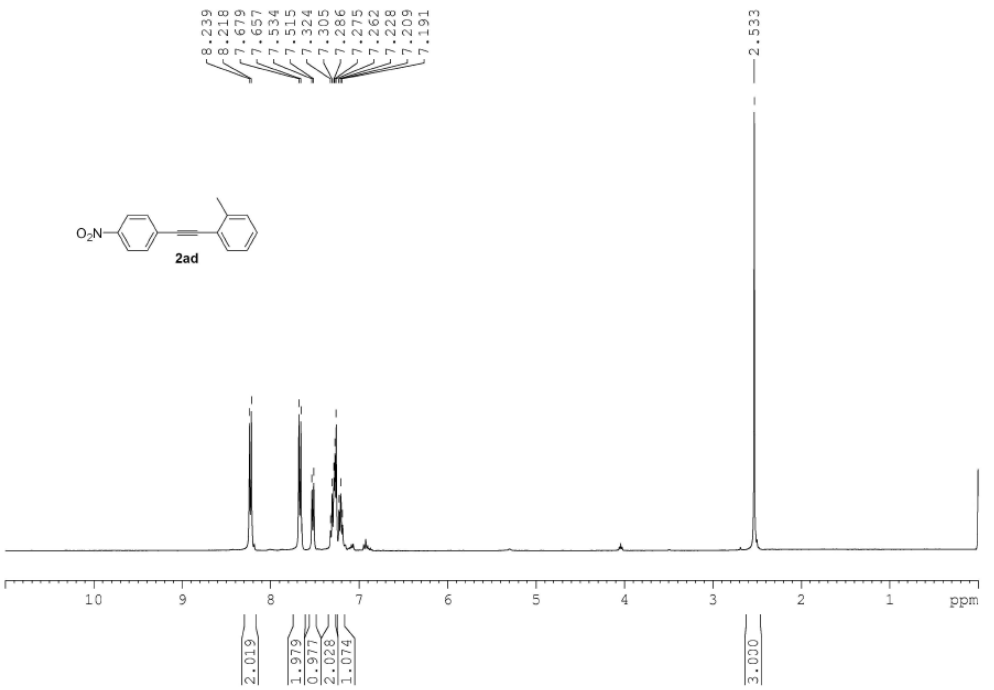
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7.191
2.533
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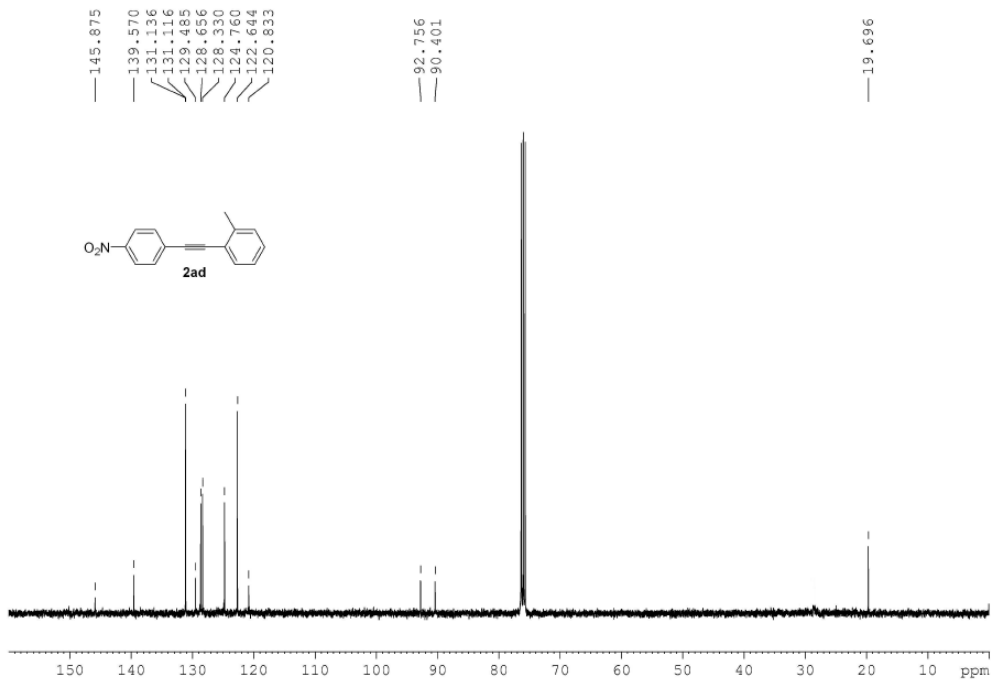
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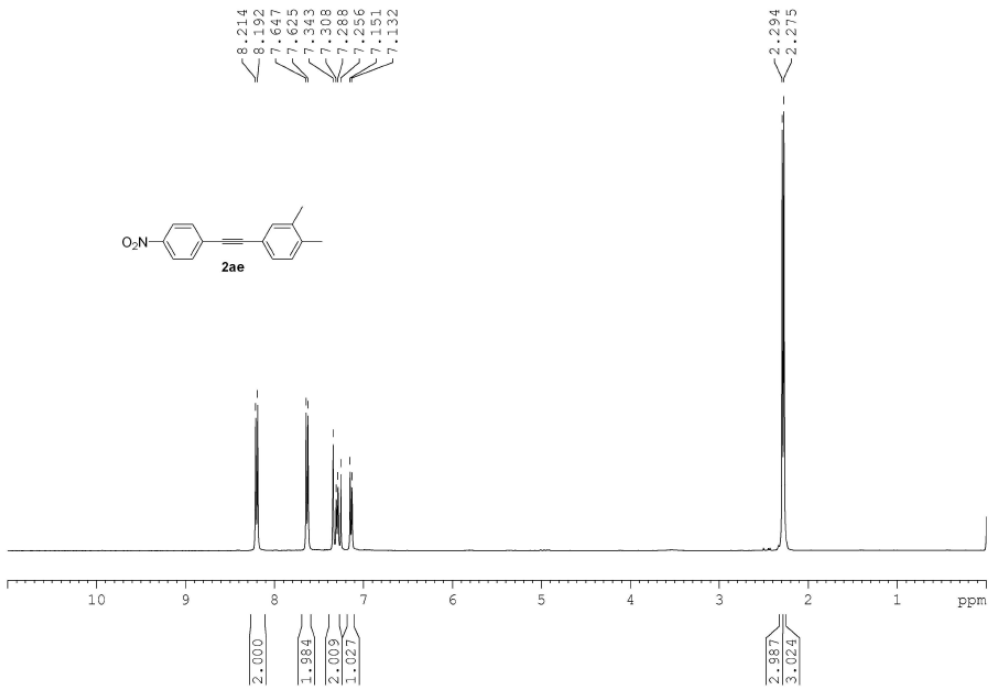
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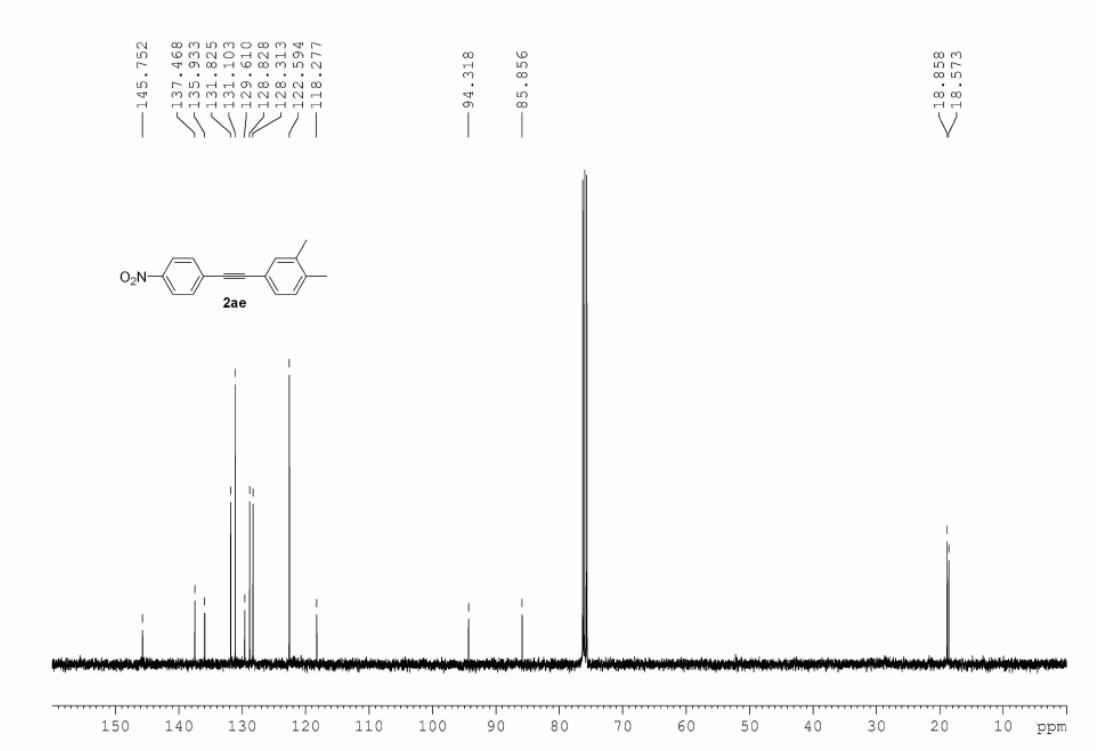
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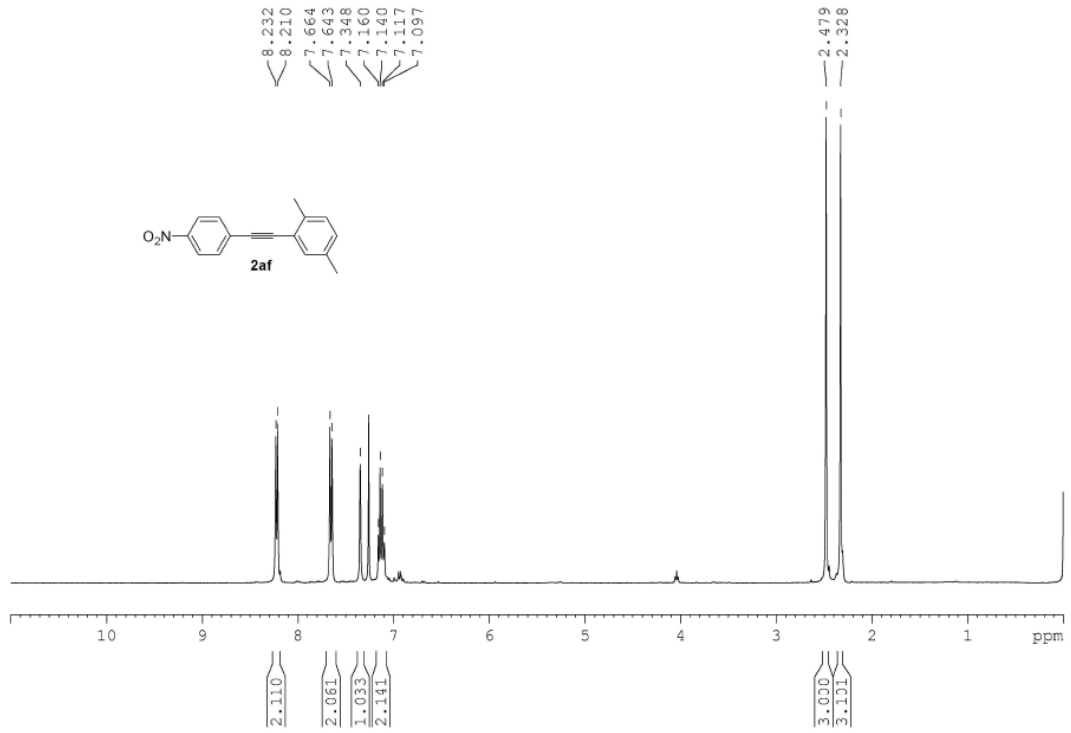
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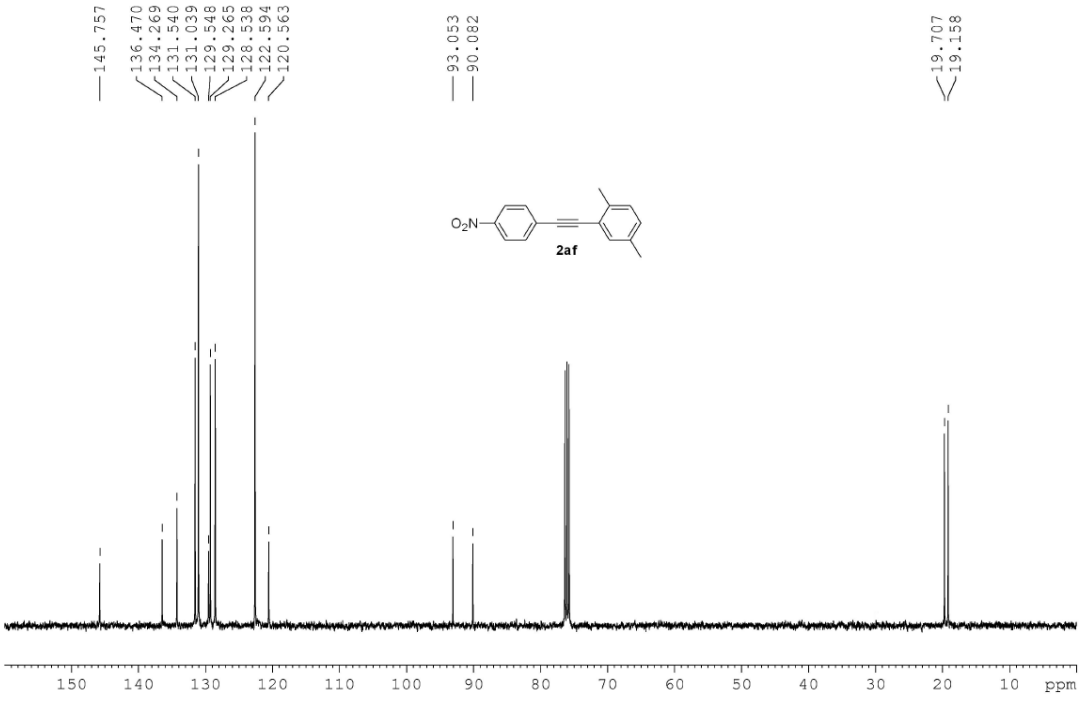
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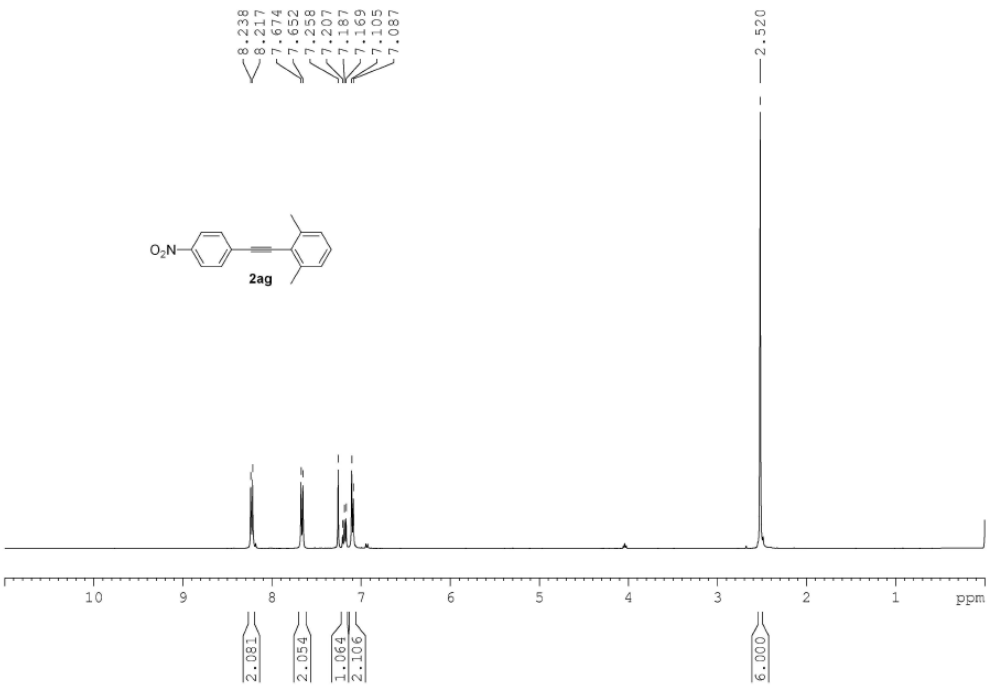
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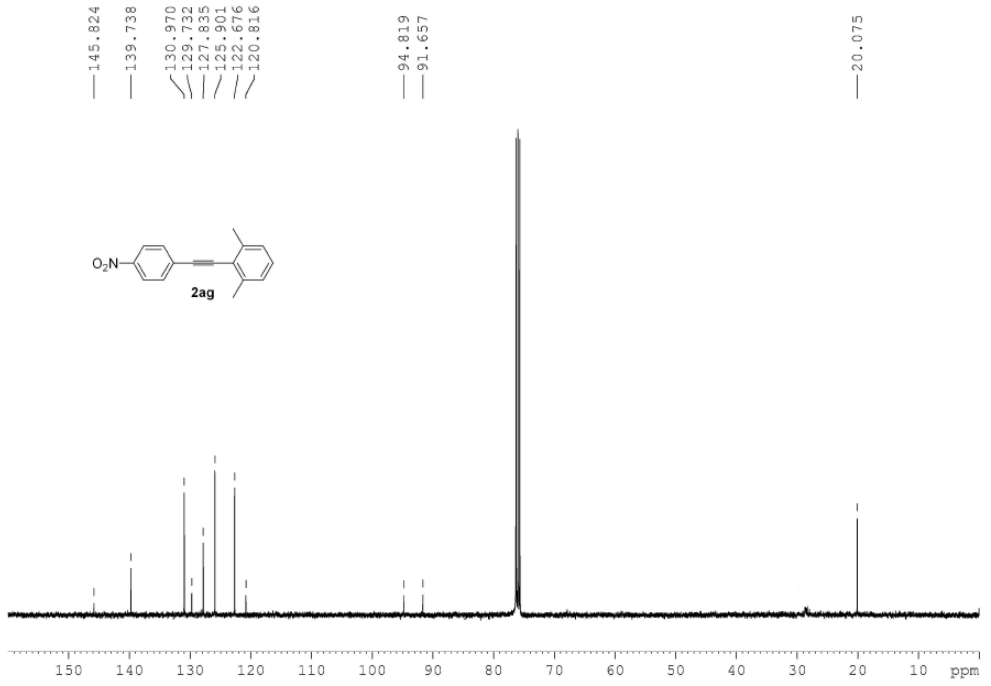
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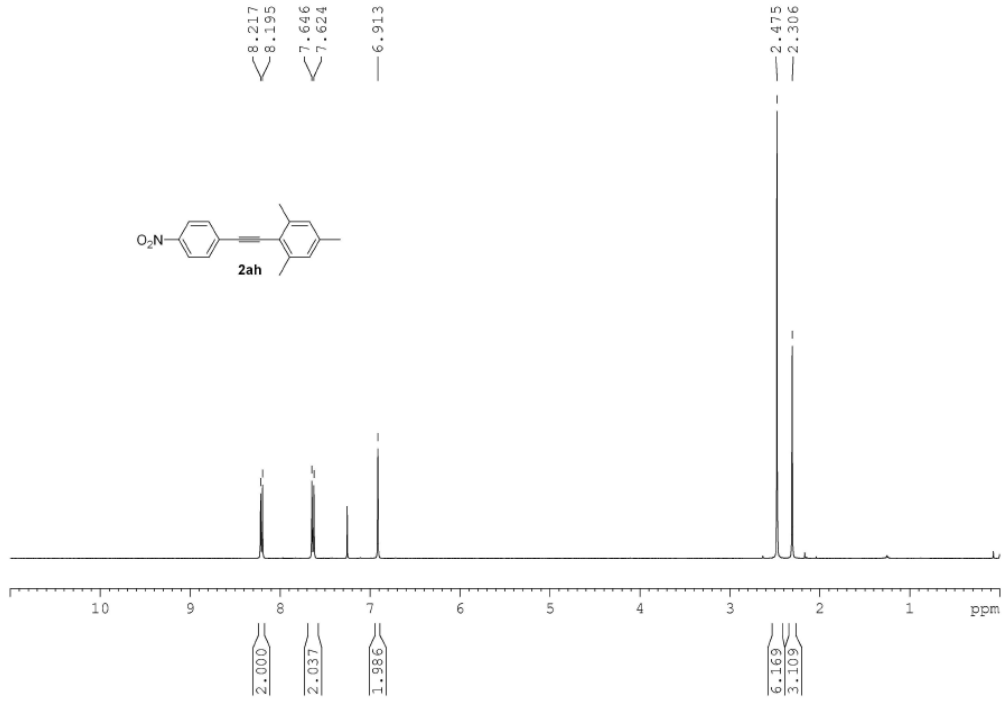
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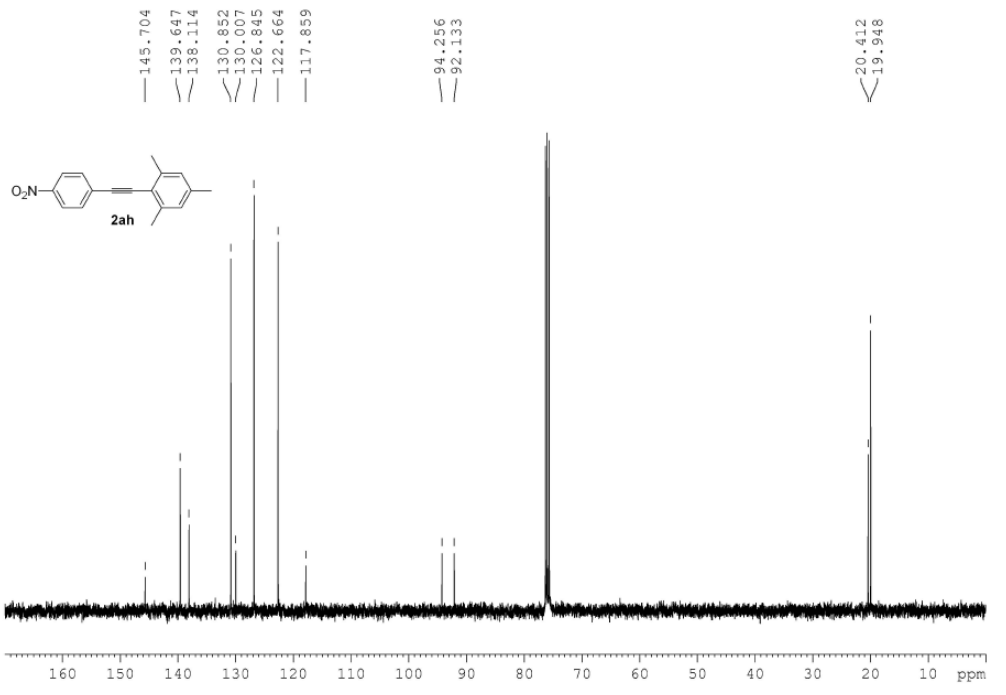
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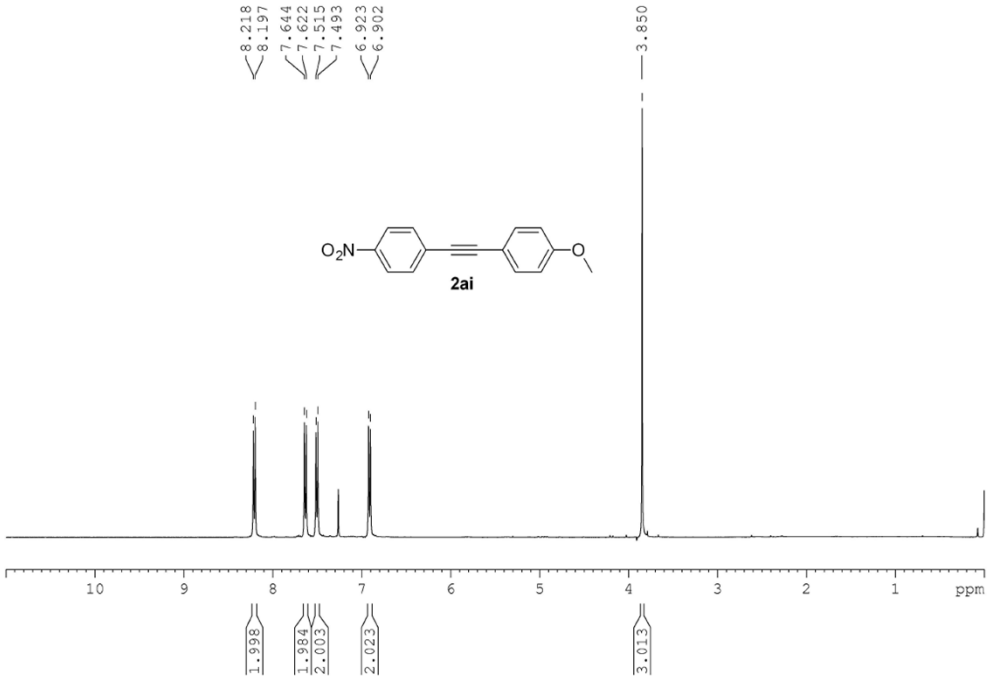
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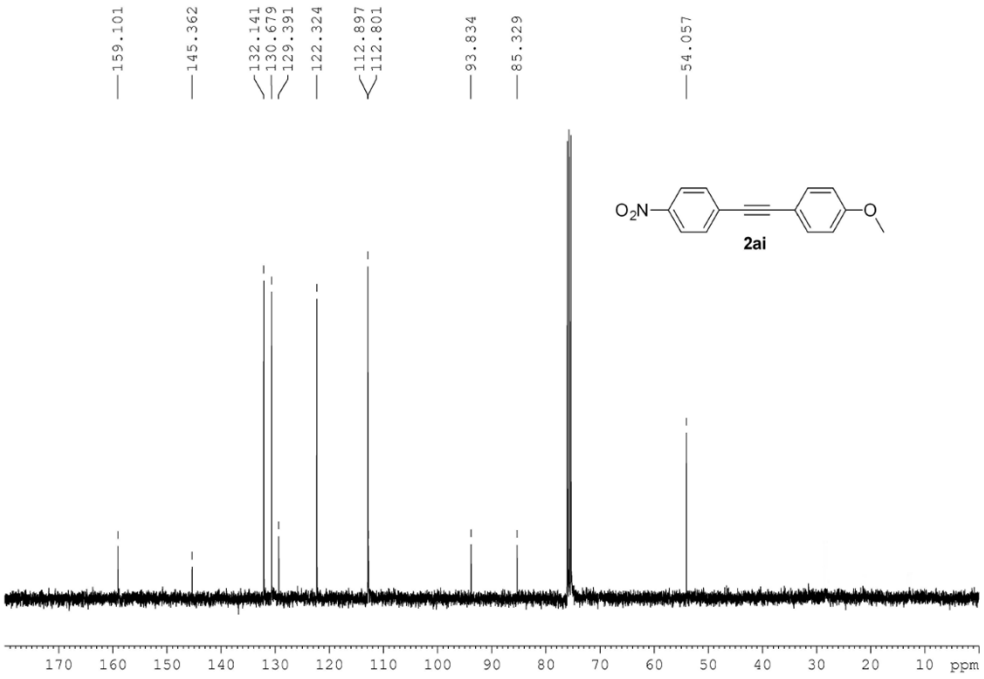
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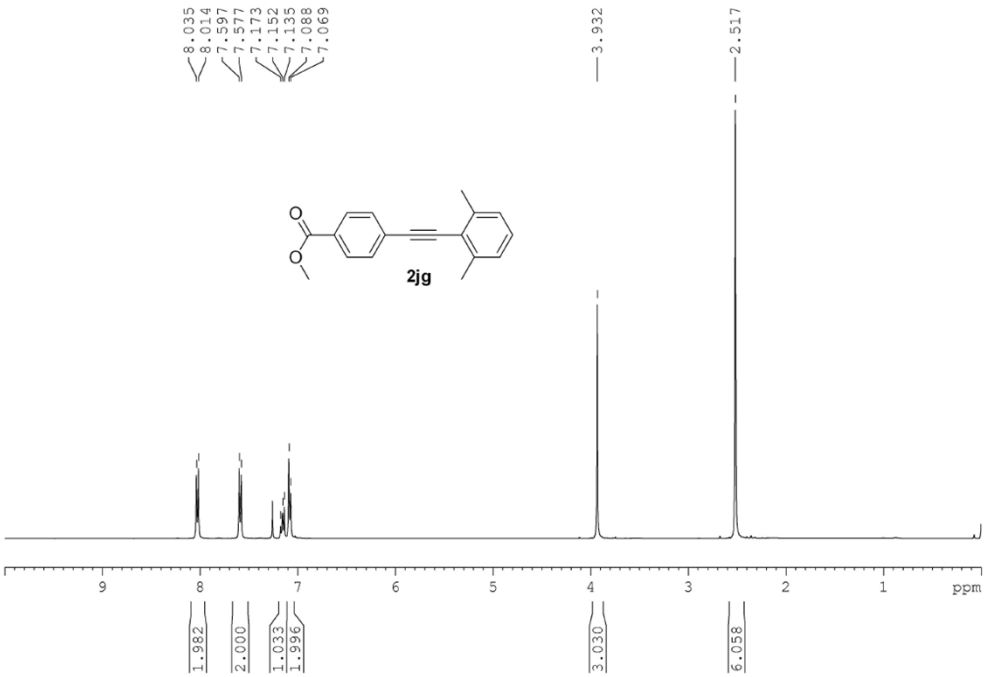
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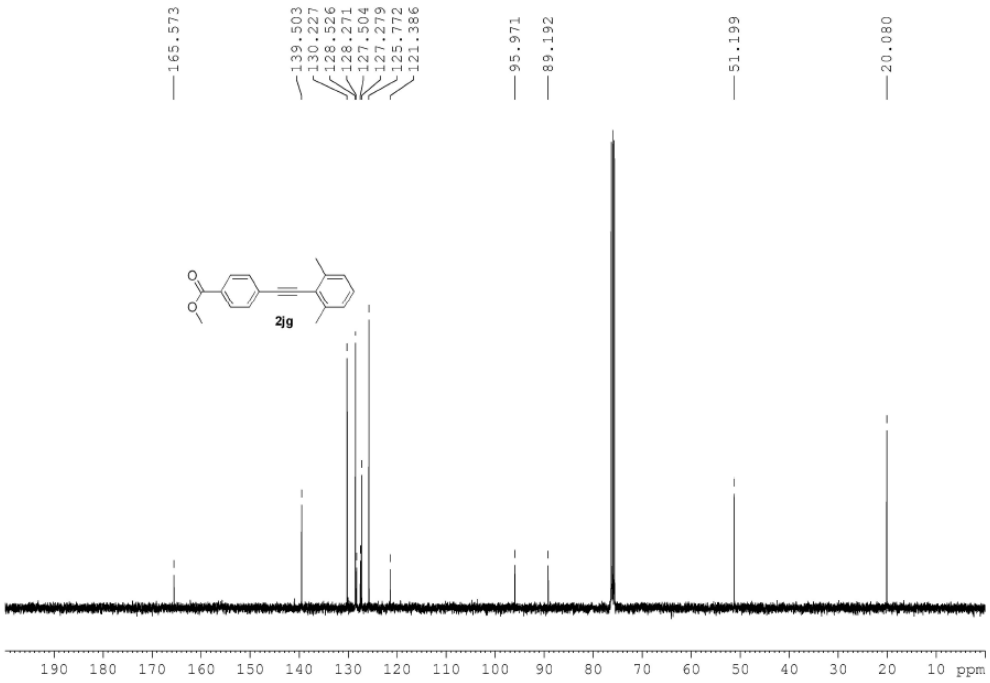
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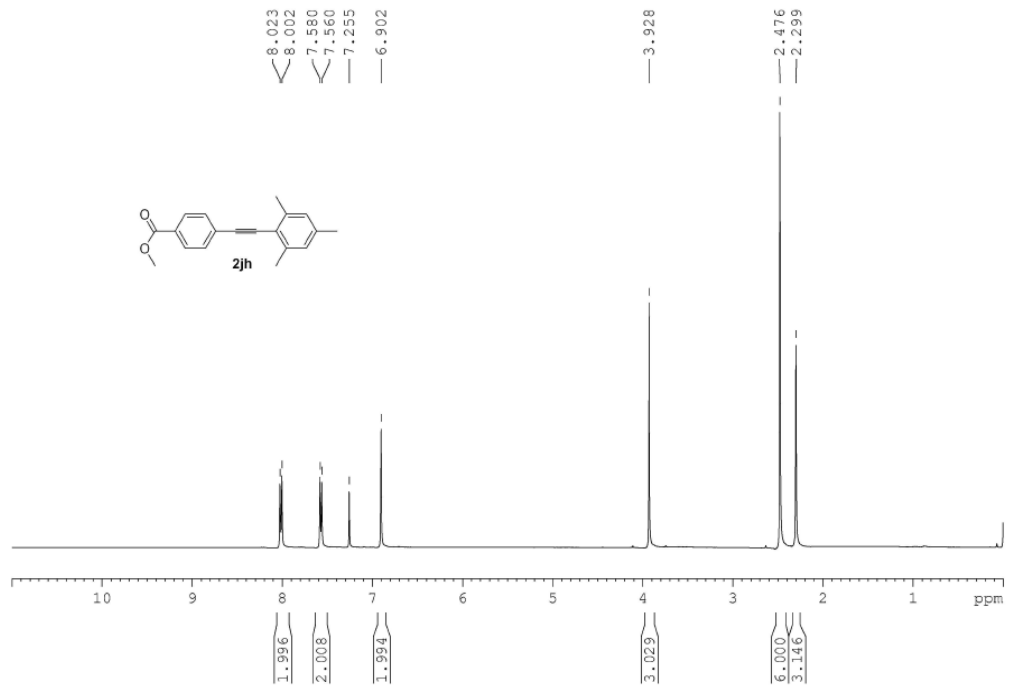
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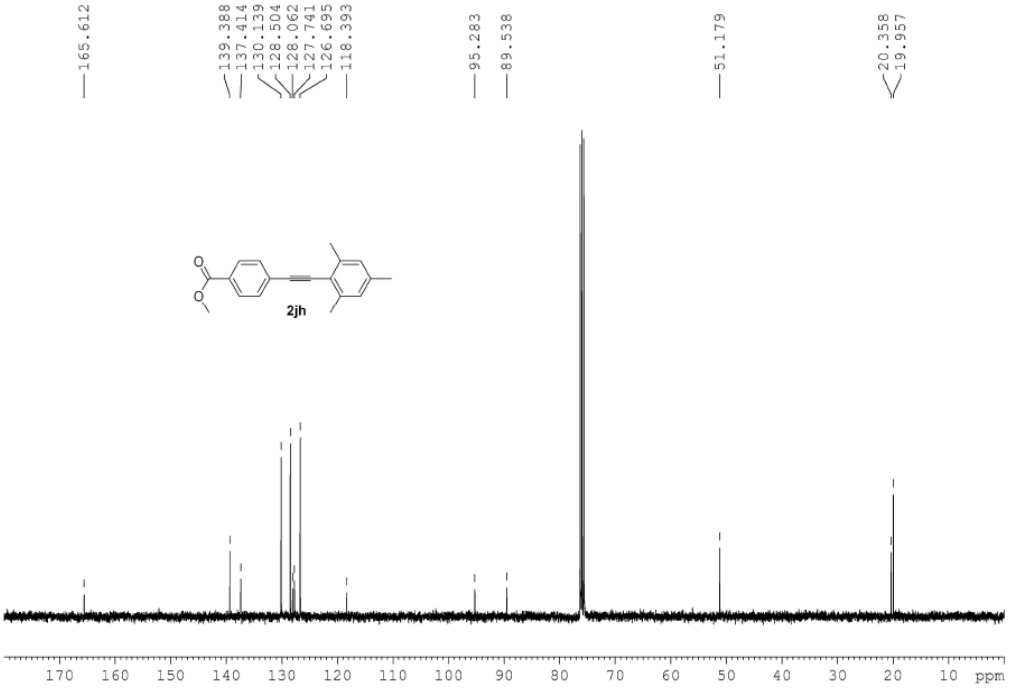
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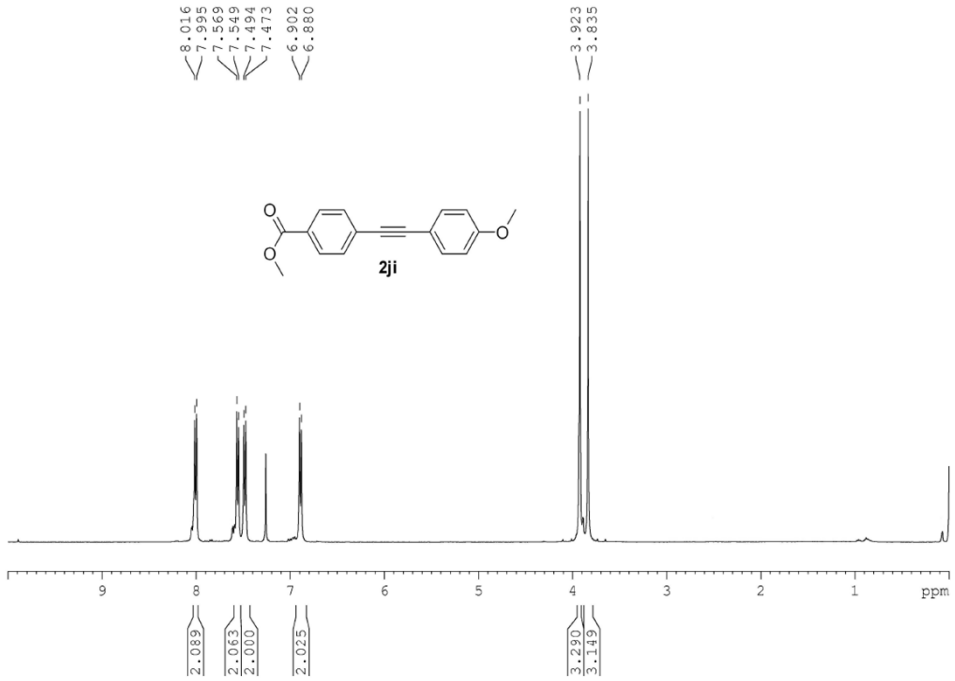
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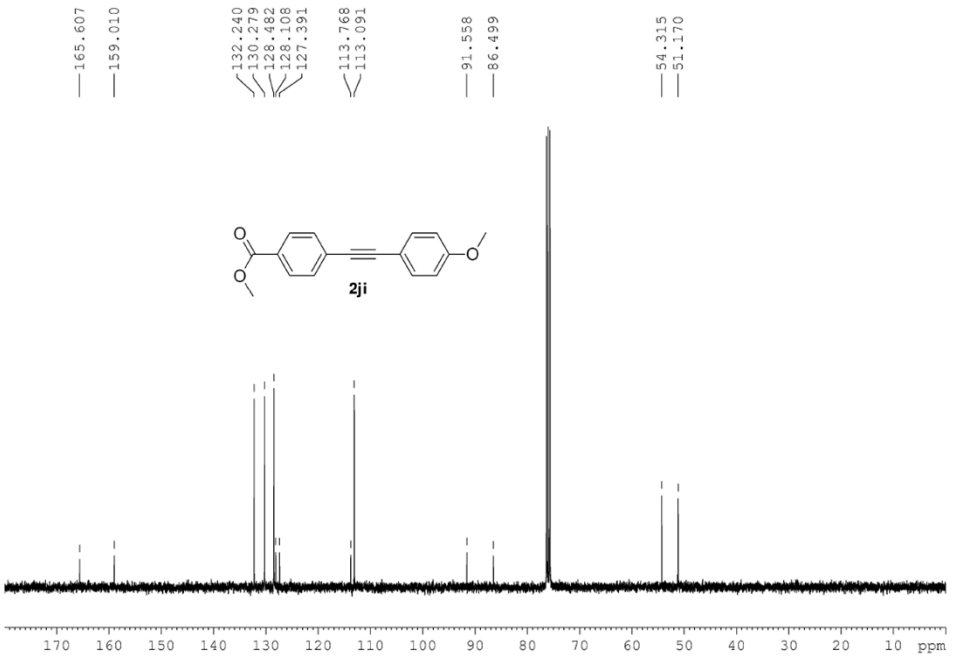
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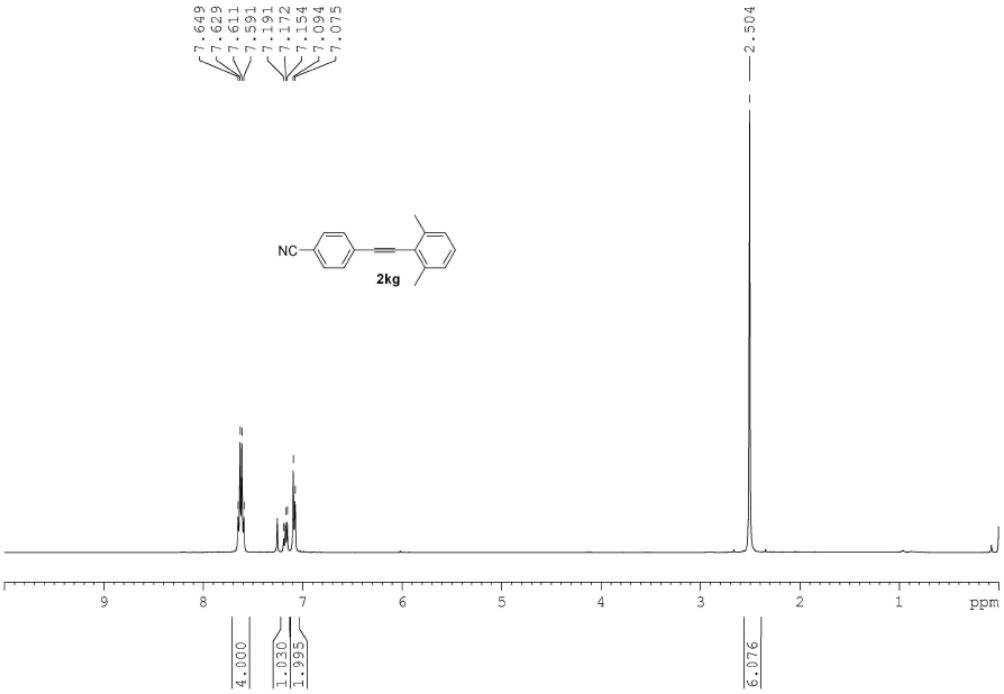
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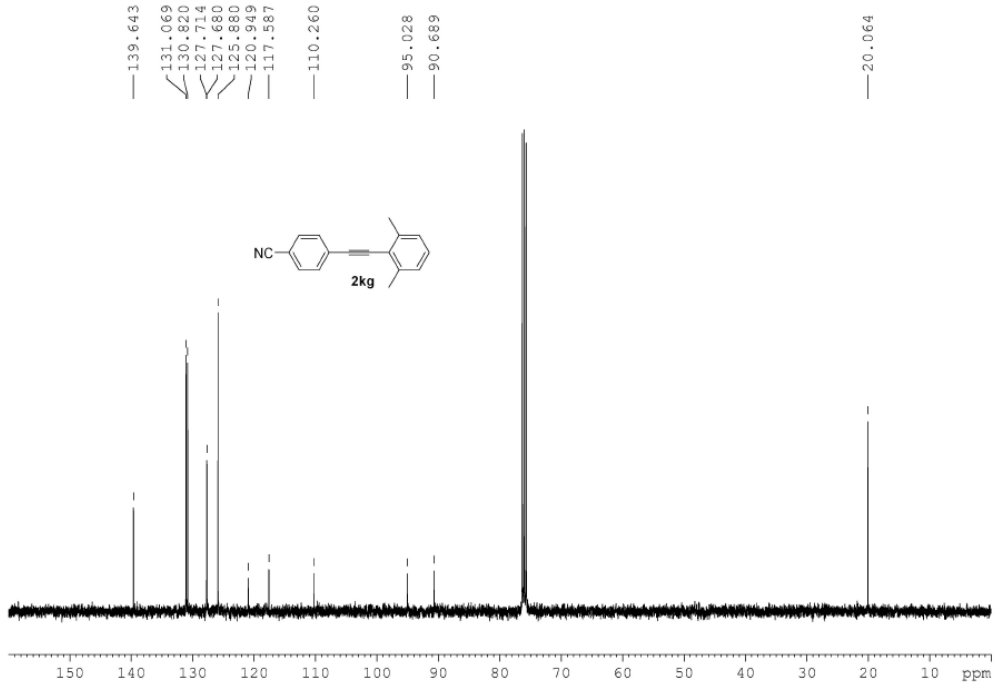
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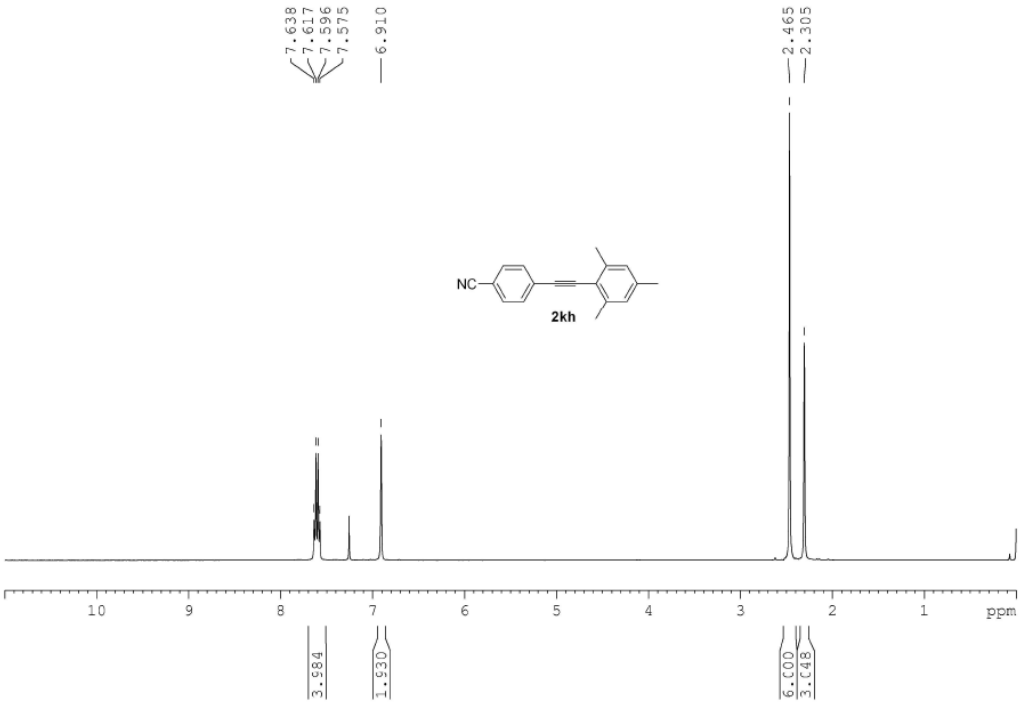
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2.305
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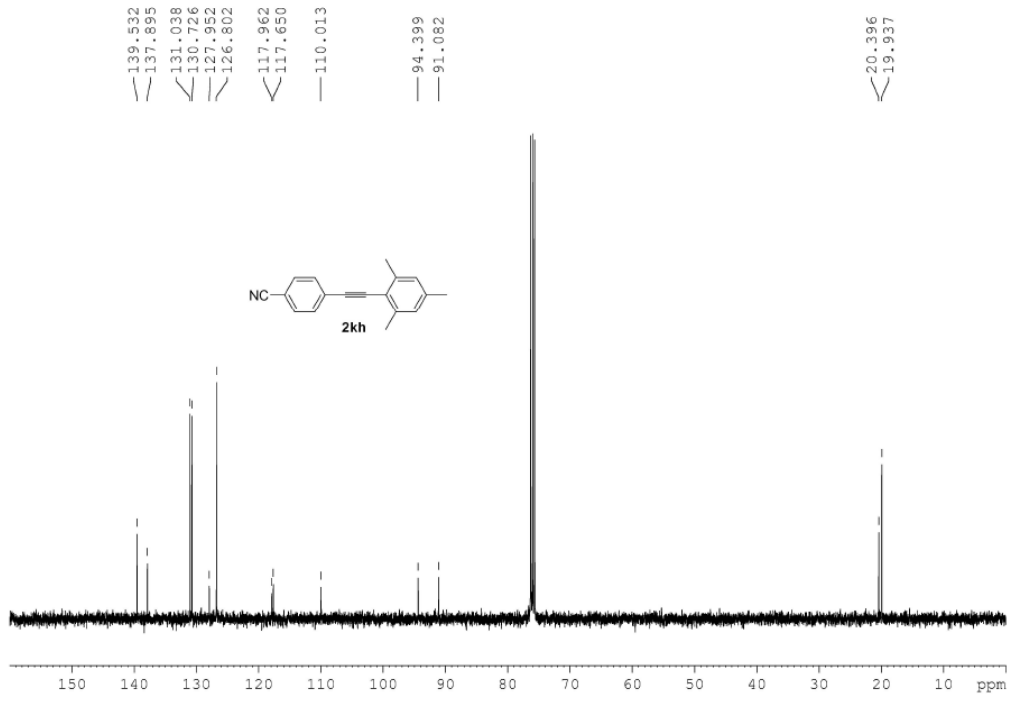
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117.650
110.013
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91.082
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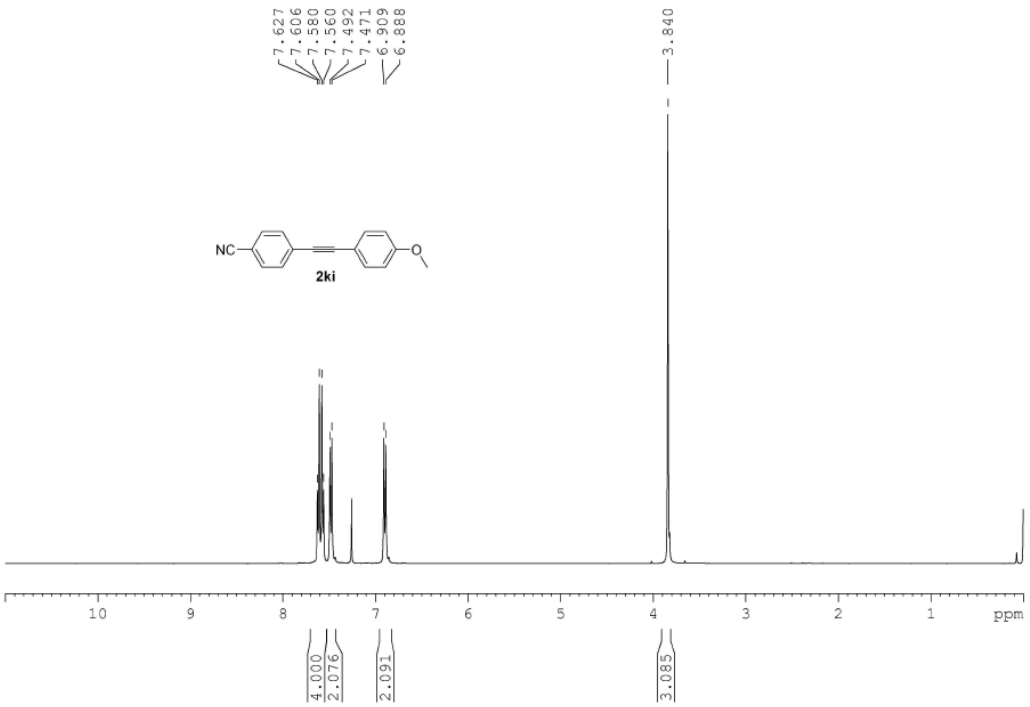
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6.888
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3
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1
ppm
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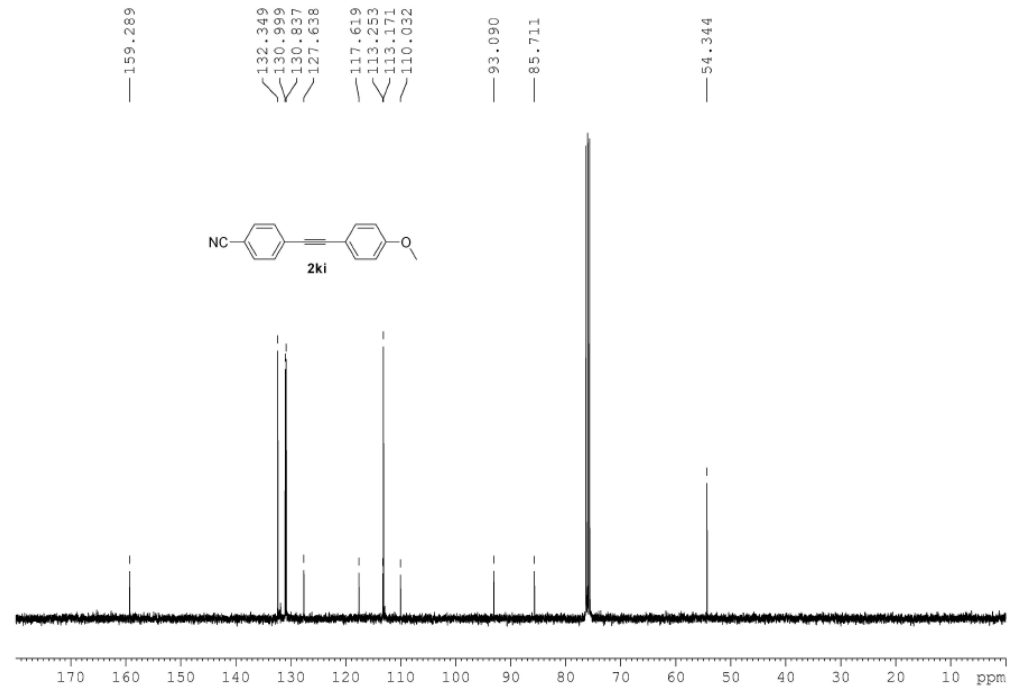
2.091
3.085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NC
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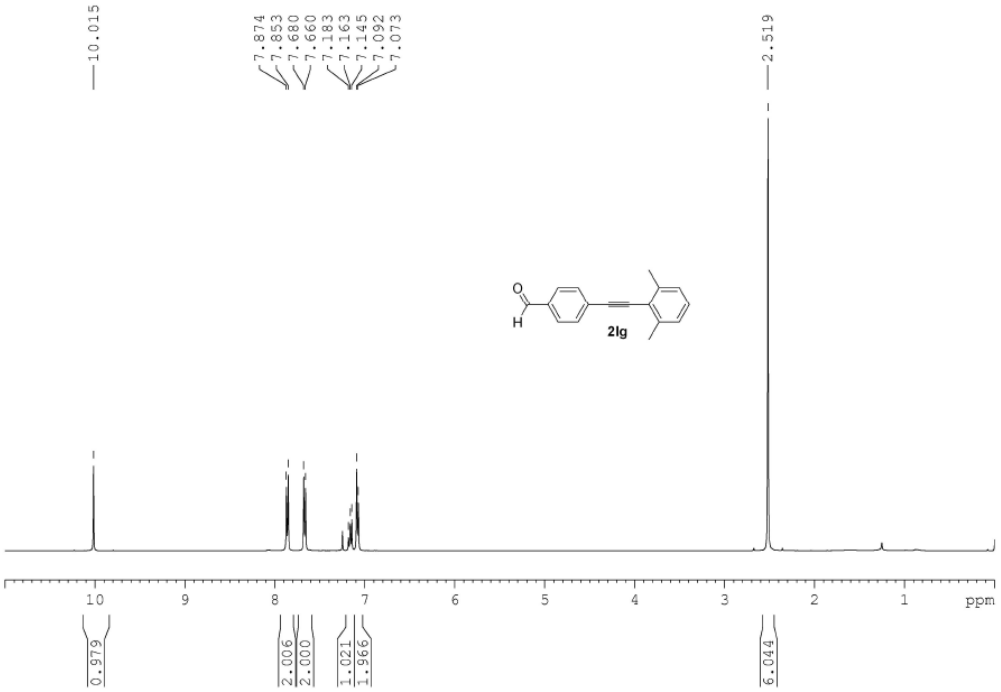
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O
H
2lg
10
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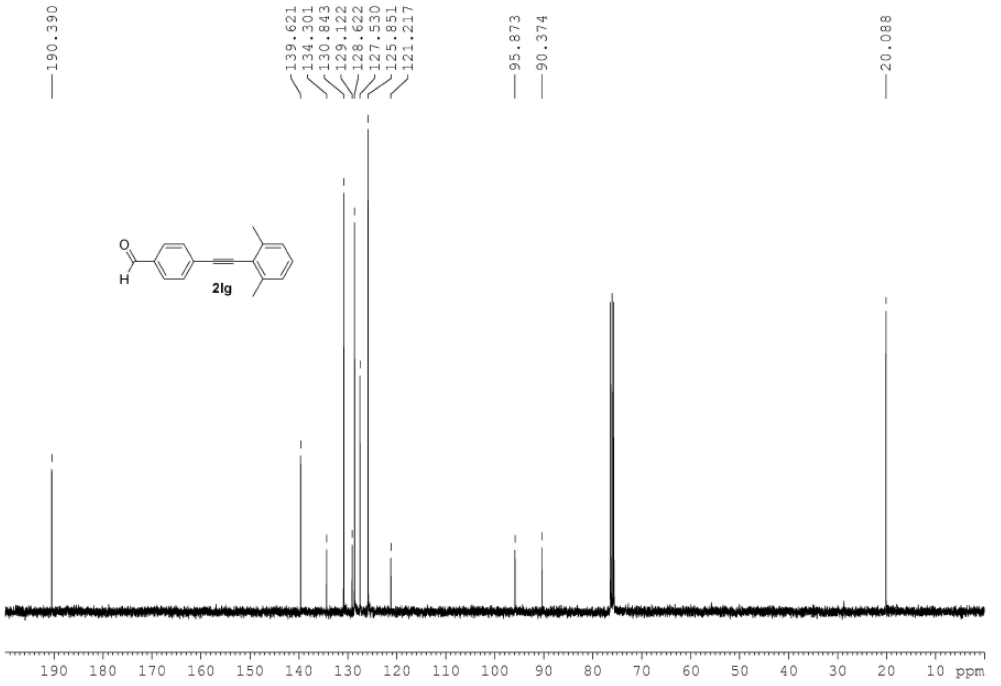
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125.851
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90.374
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O
H
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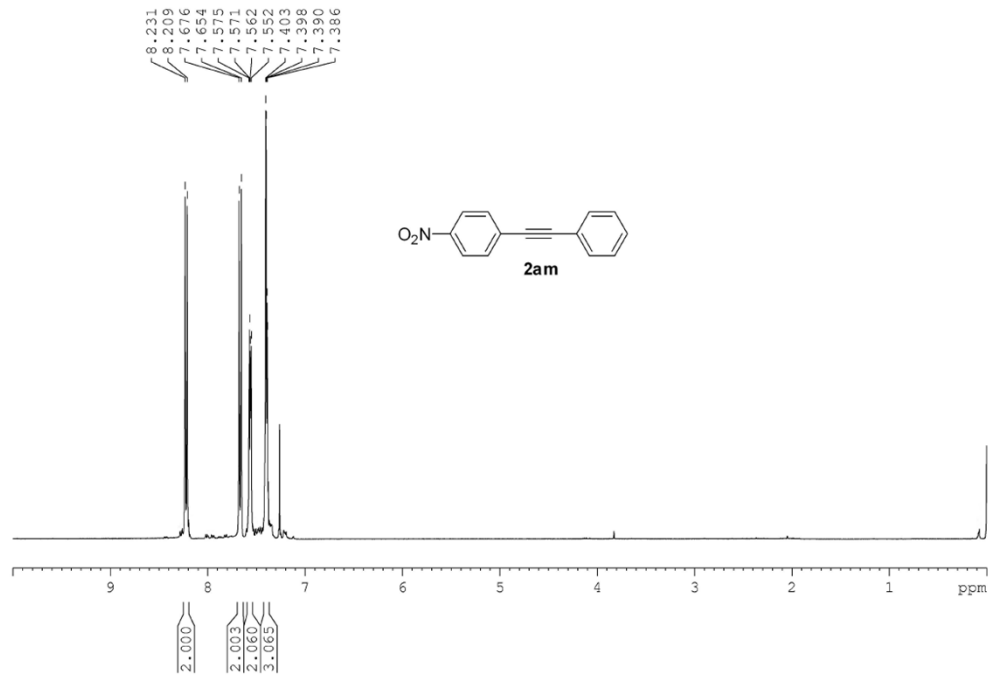
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7.386
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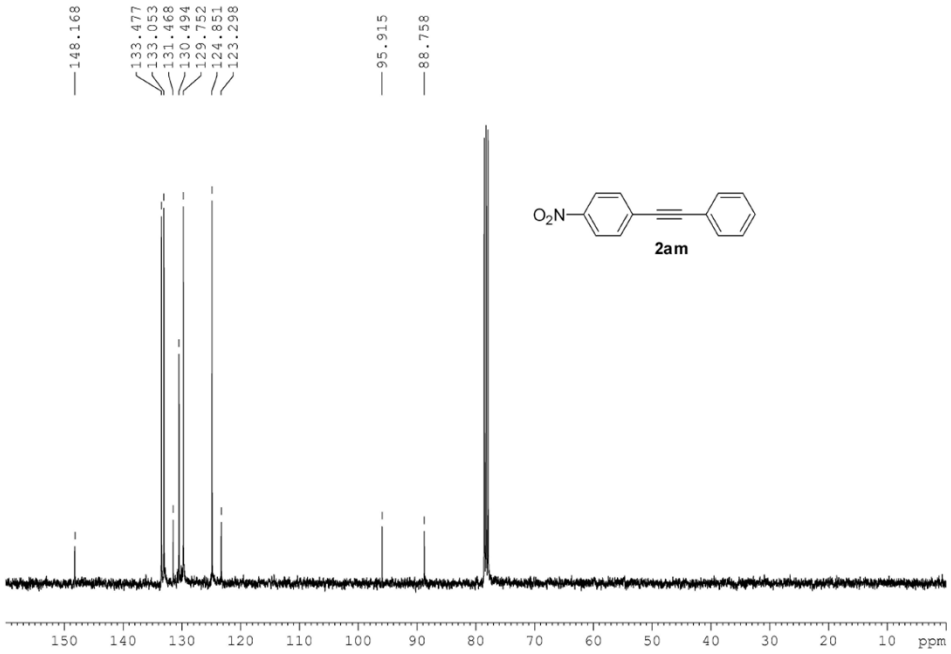
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123.298
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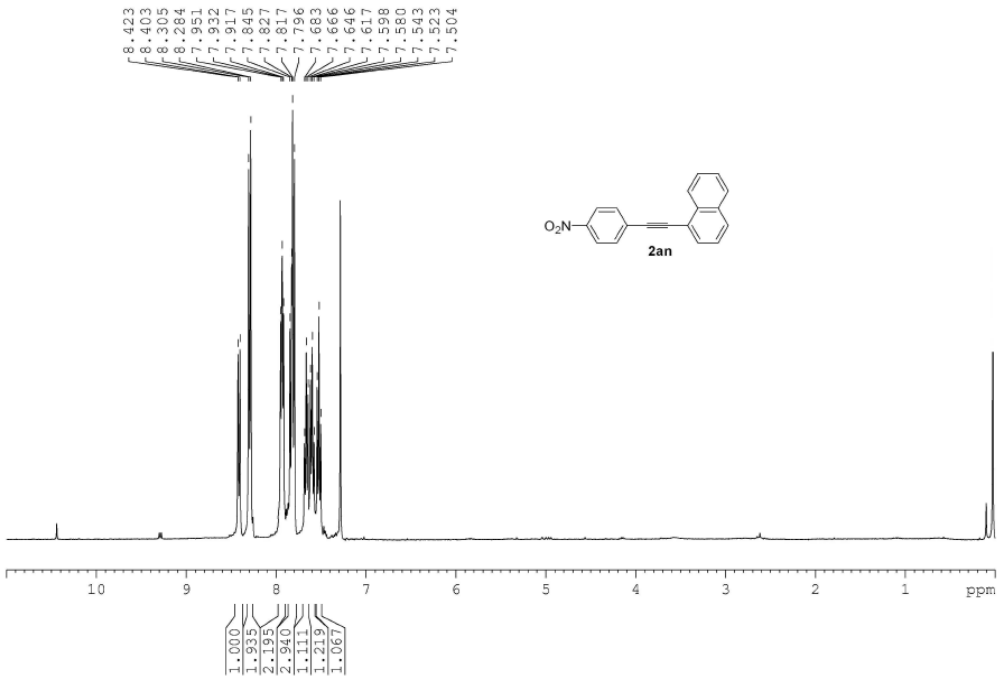
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7.917
7.845
7.827
7.817
7.796
7.683
7.666
7.646
7.617
7.598
7.580
7.543
7.523
7.504
O2N
2an
10
9
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2.195
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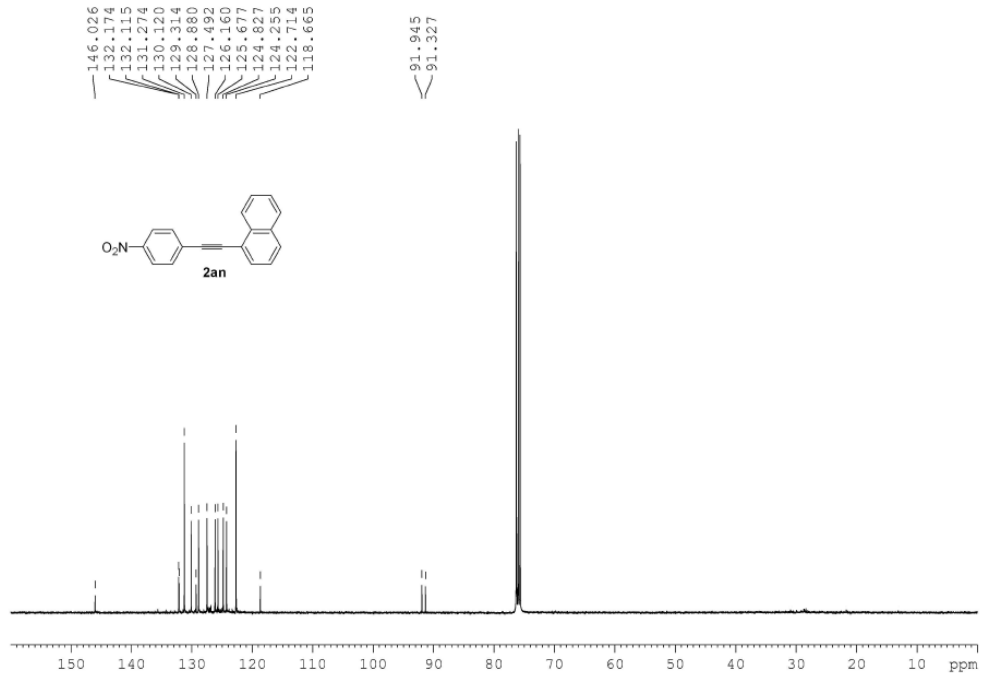
1.067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127.492
126.160
125.677
124.827
124.255
122.714
118.665
91.945
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70
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30
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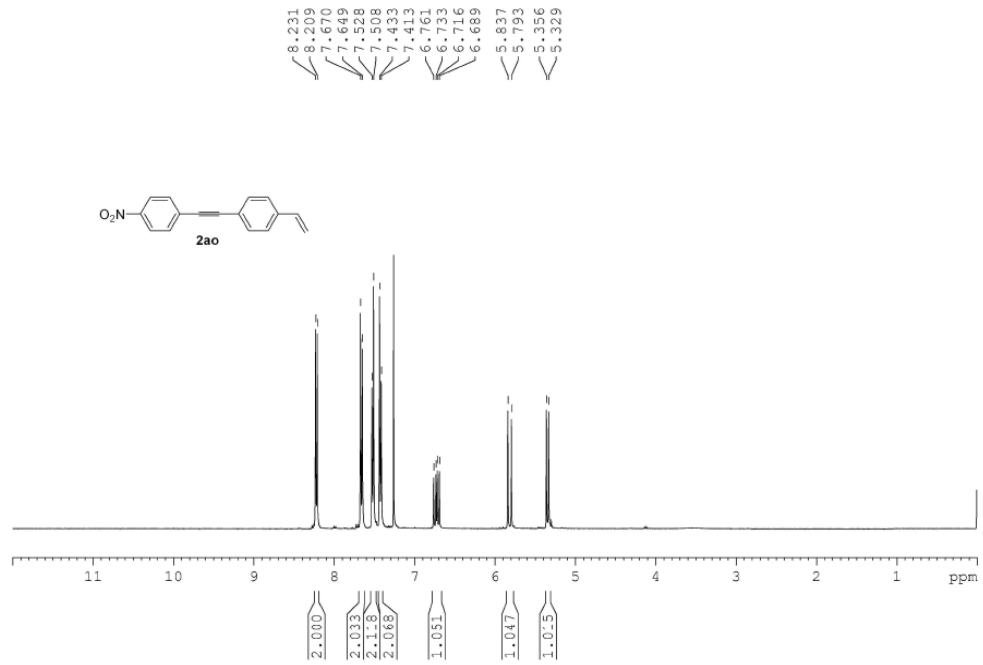
10
ppm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6.716
6.689
5.837
5.793
5.356
5.329
O2N
2ao
11
10
9
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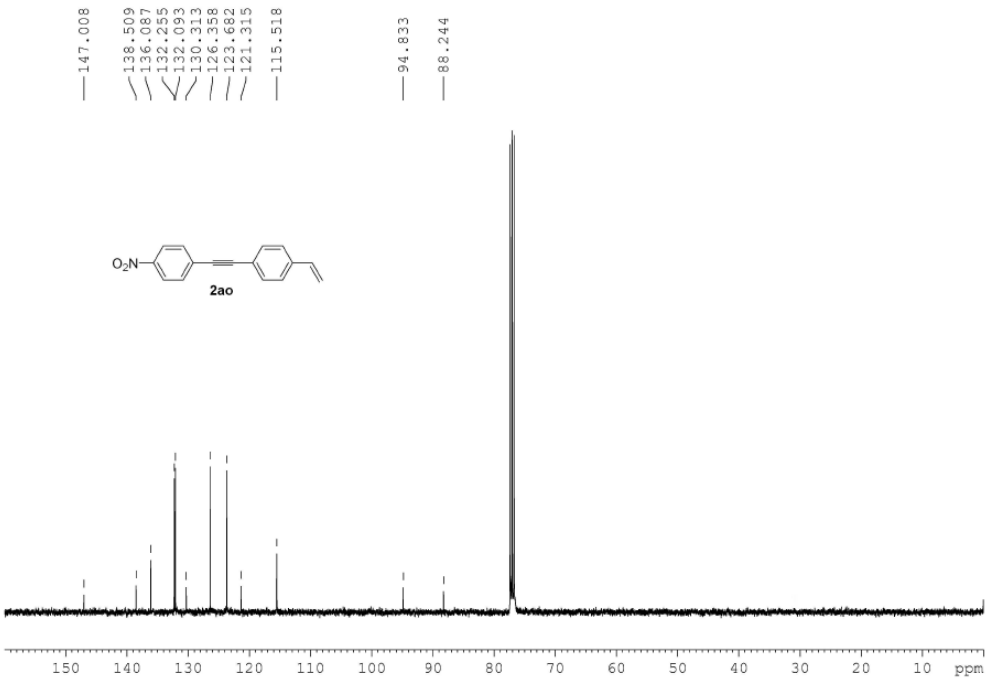
1.051
1.047
1.015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115.518
94.833
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140
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70
60
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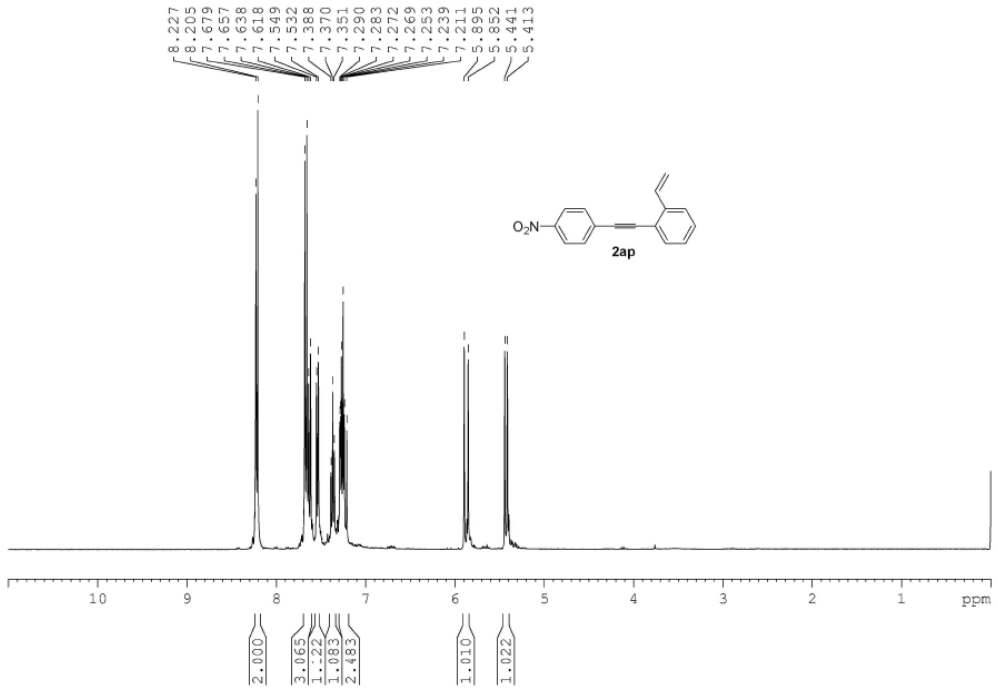
30
20
10
ppm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7.532
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7.351
7.290
7.283
7.272
7.269
7.253
7.239
7.211
5.895
5.852
5.441
5.413
O2N
2ap
10
9
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3
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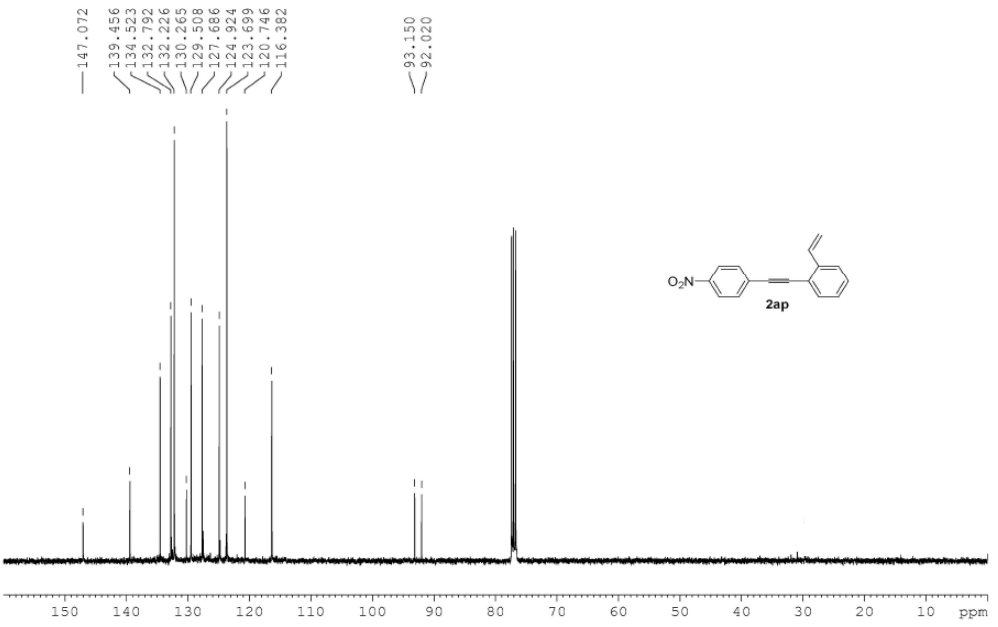
1.010
1.022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124.924
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120.746
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93.150
92.020
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140
130
120
110
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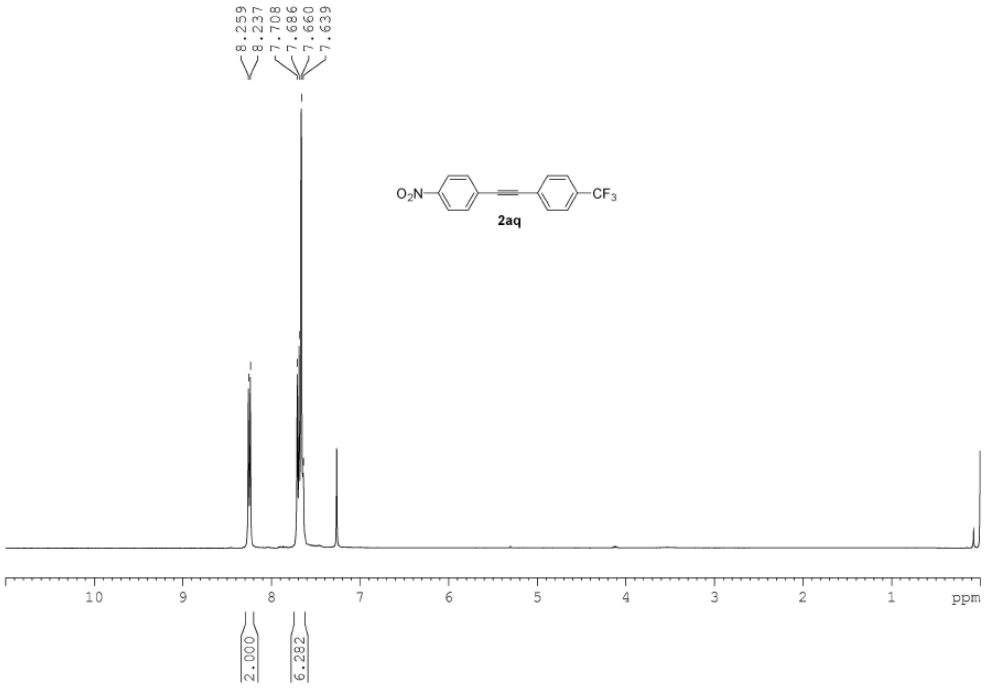
10
ppm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7.686
7.660
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CF3
2aq
10
9
8
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4
3
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1
ppm
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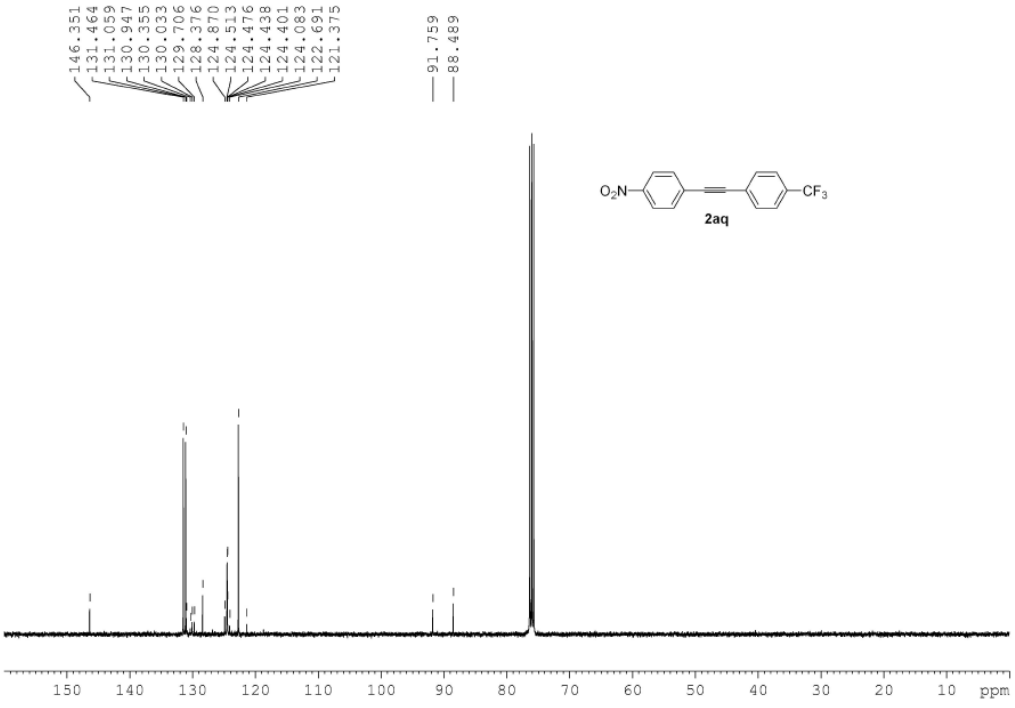
6.282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124.083
122.691
121.375
91.759
88.489
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70
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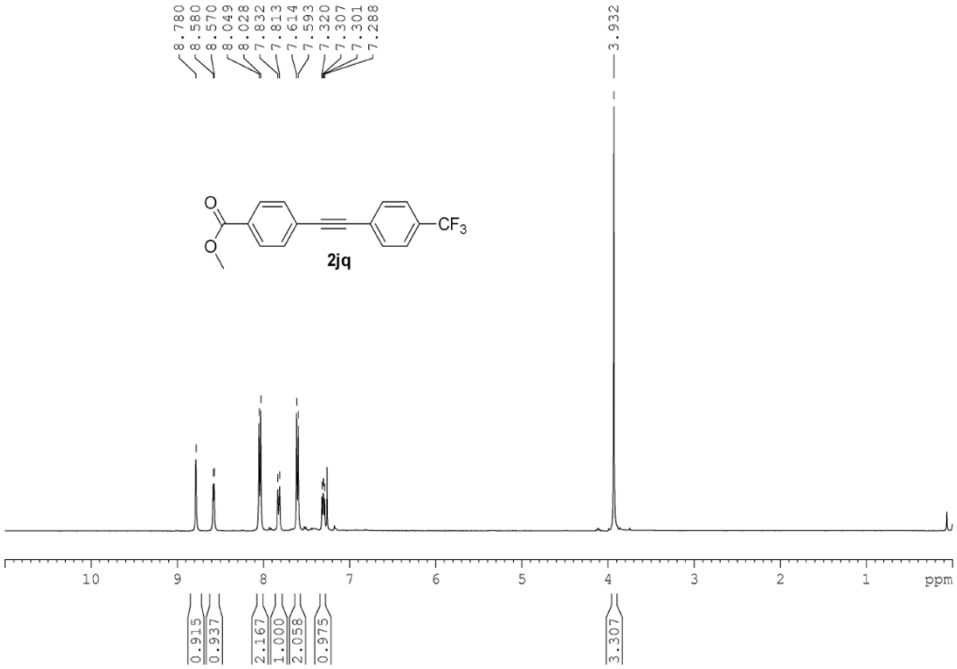
ppm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7.301
7.288
3.932
O
O
CF3
2jq
10
9
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1.000
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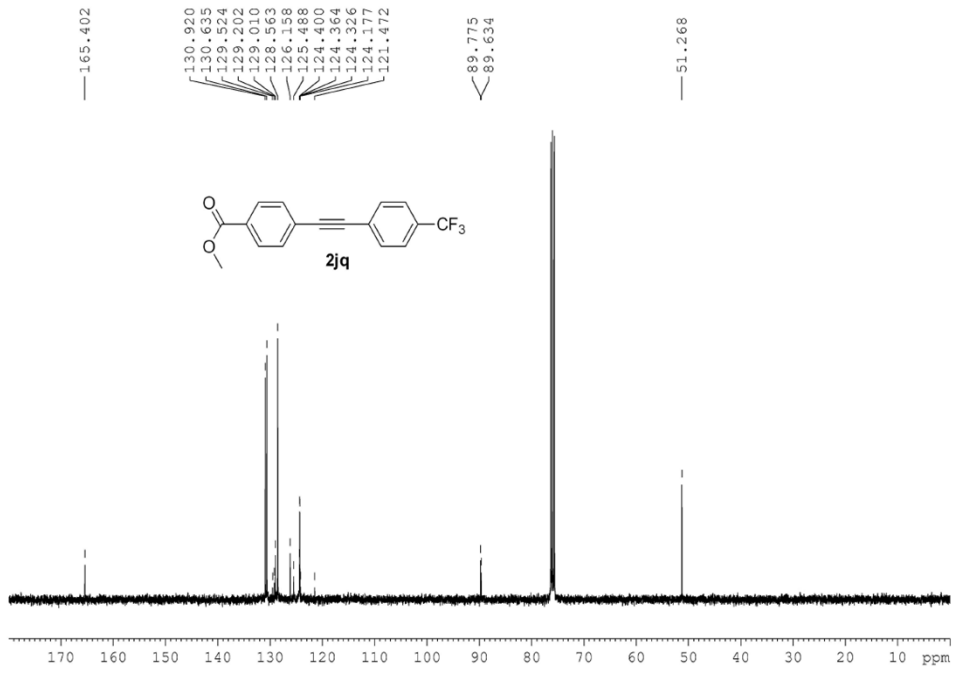
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124.326
124.177
121.472
89.775
89.634
51.268
O
O
CF3
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150
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30
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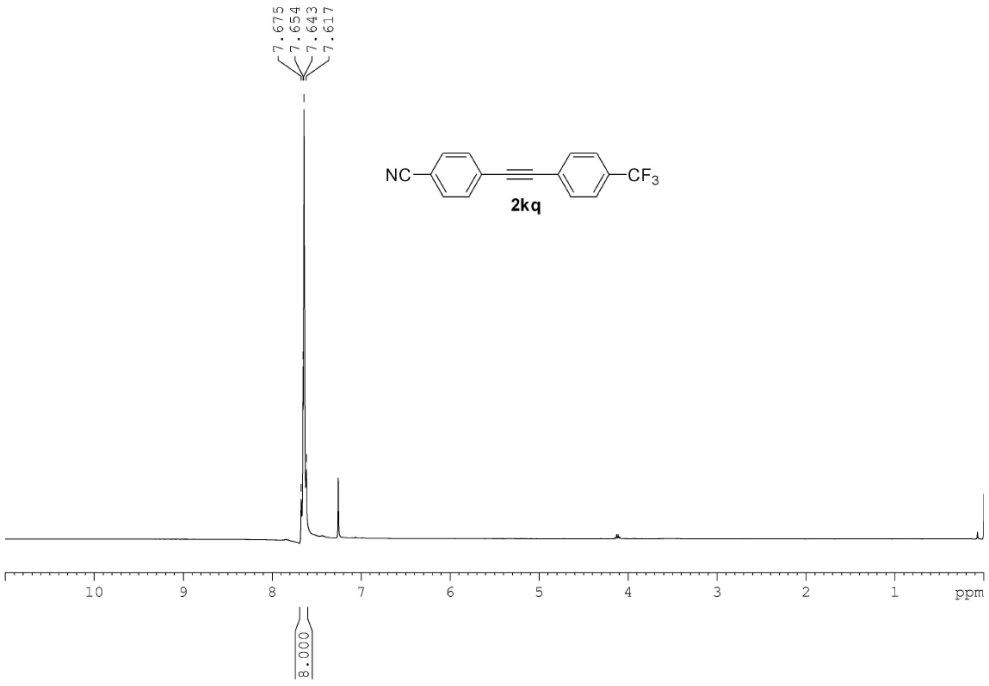
10
ppm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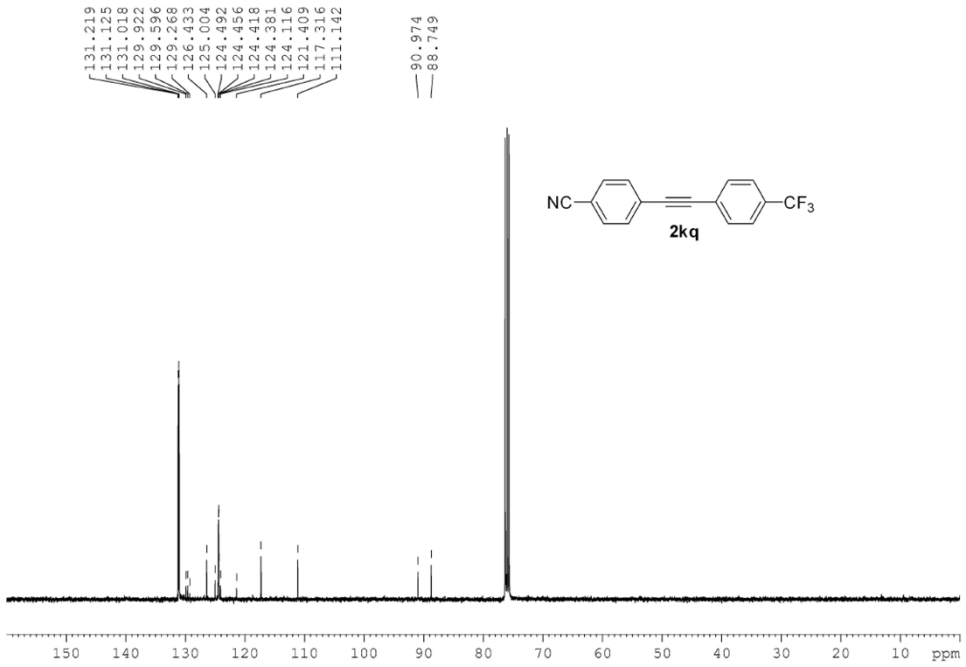
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124.418
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124.116
121.409
117.316
111.142
90.974
88.749
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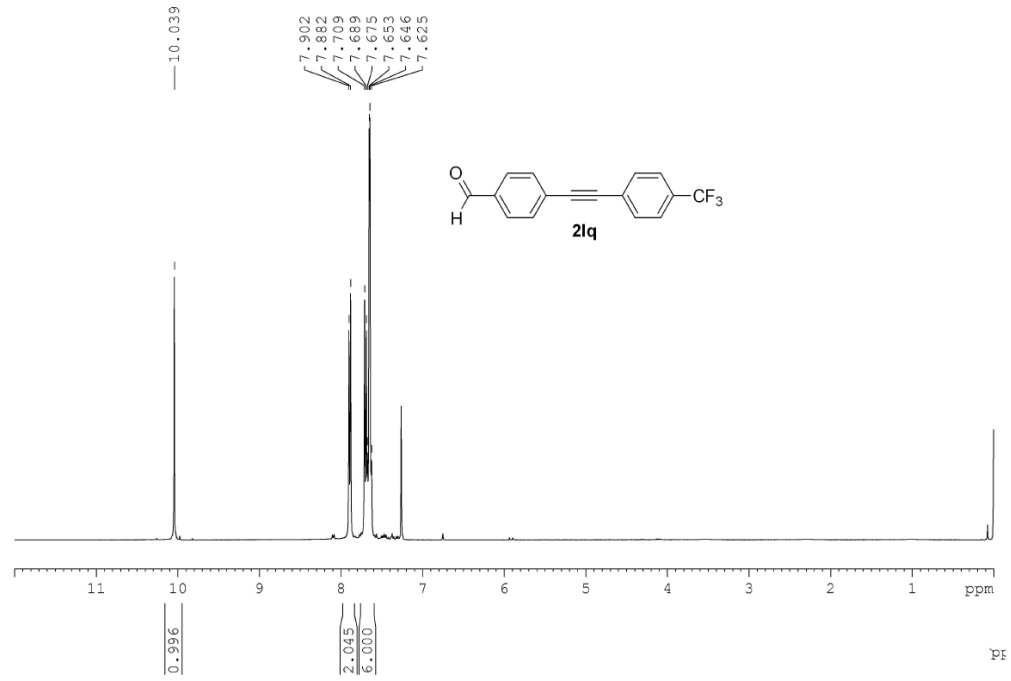
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7.646
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H
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10
9
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6
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2
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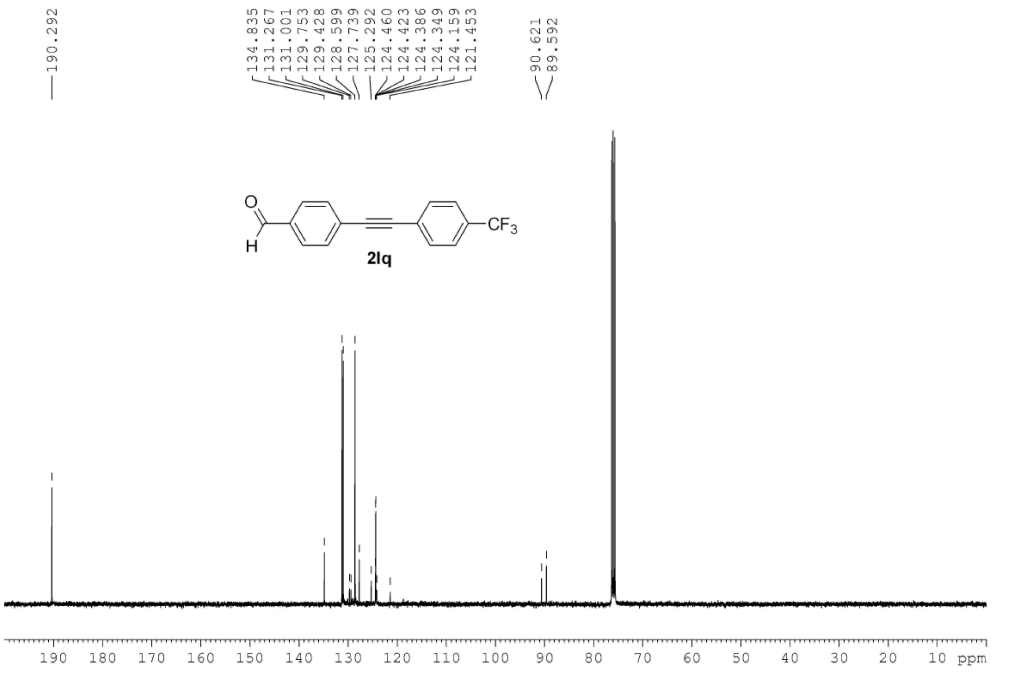
6.000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124.386
124.349
124.159
121.453
90.621
89.592
O
H
CF3
2lq
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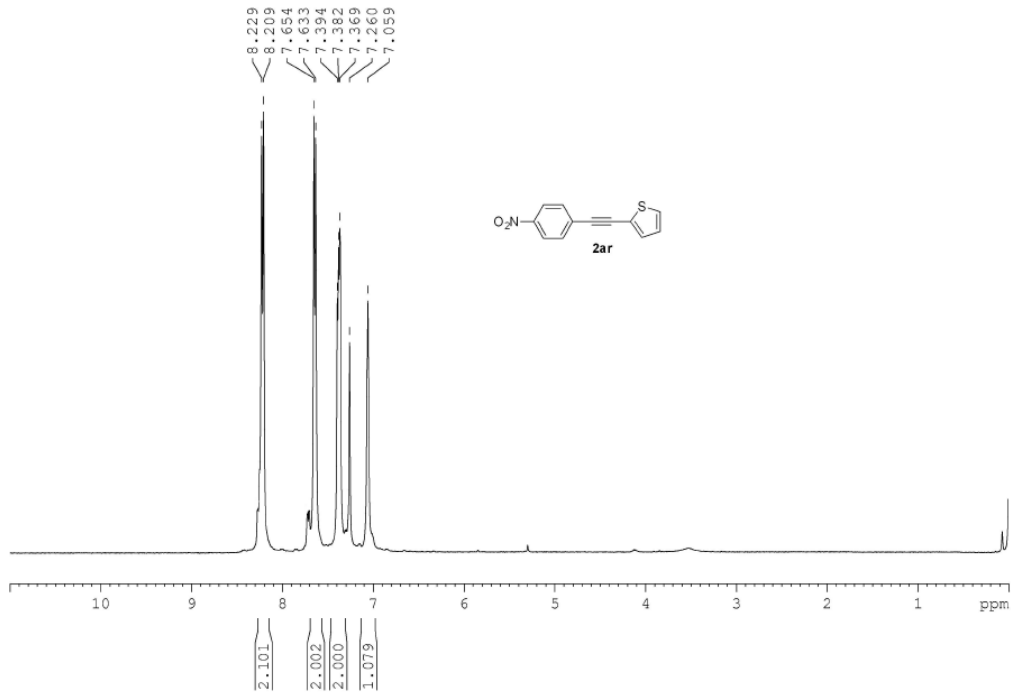
30
20
10
ppm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7.633
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7.382
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7.260
7.059
O2N
S
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8
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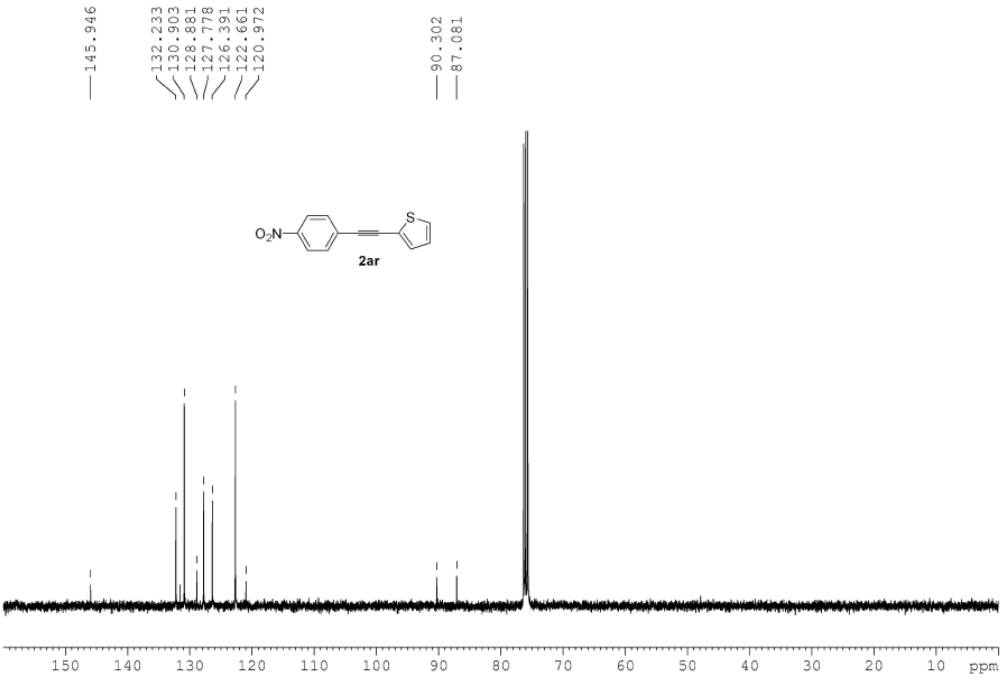
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87.081
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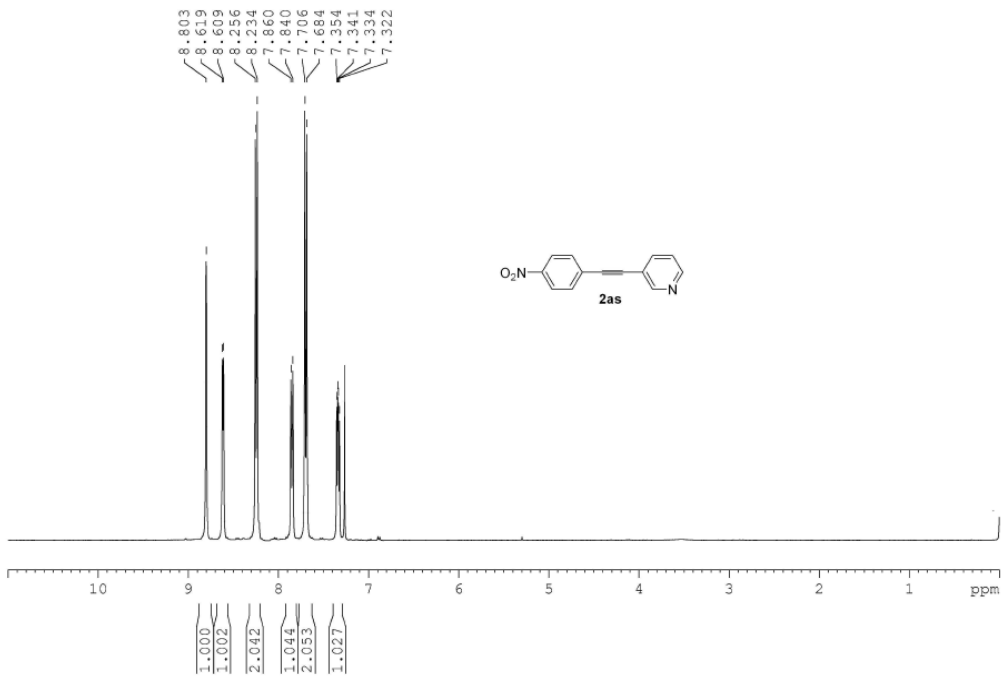
10
ppm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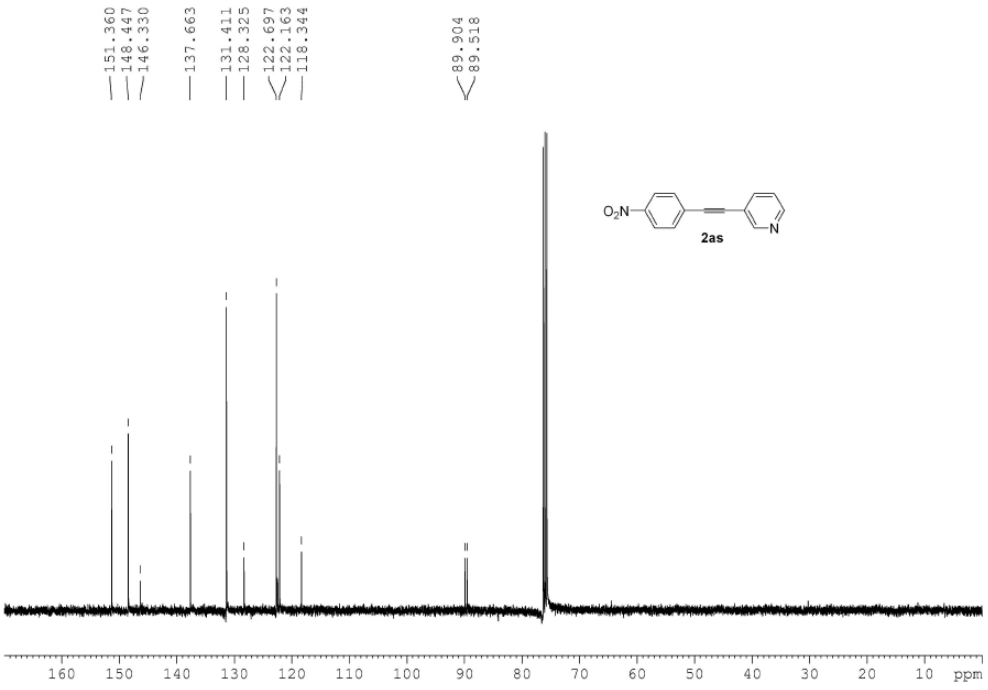
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122.163
118.344
89.904
89.518
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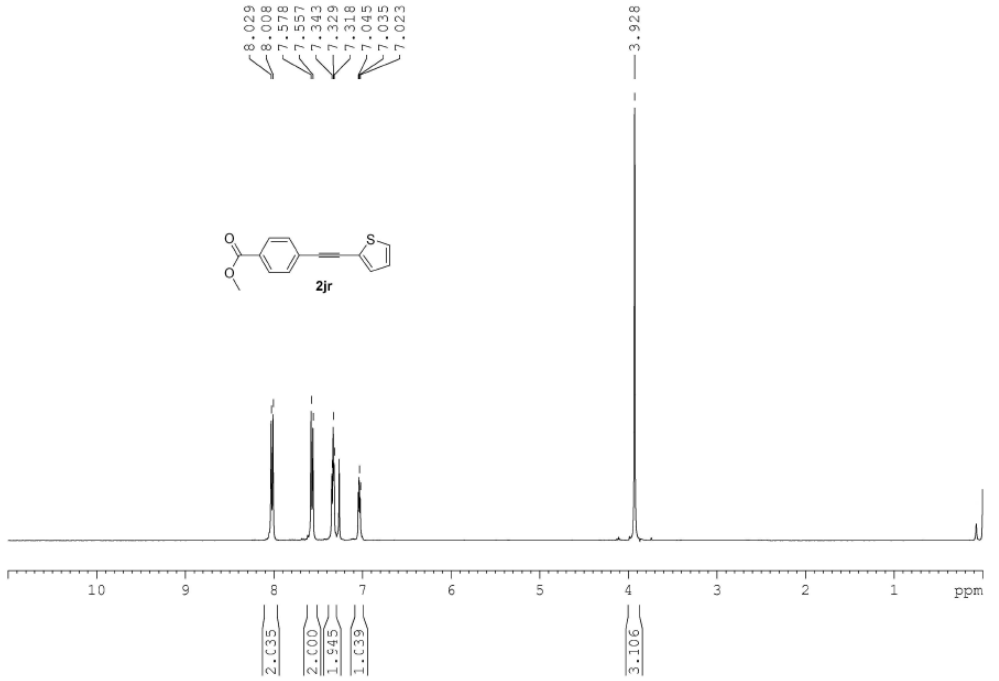
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7.023
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O
O
S
2jr
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6
5
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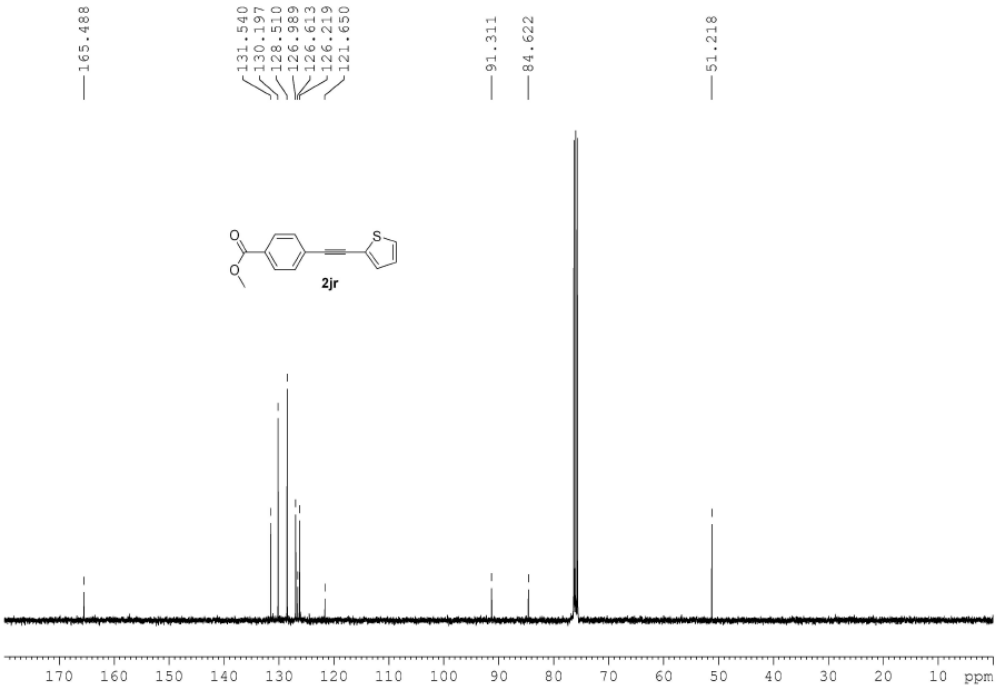
3.106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O
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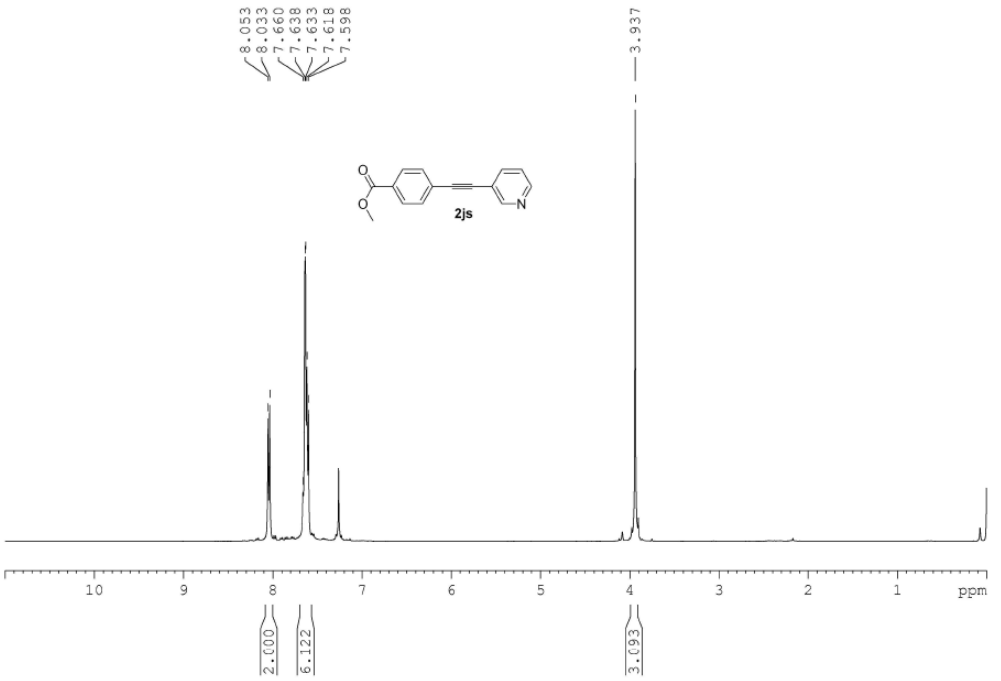
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2
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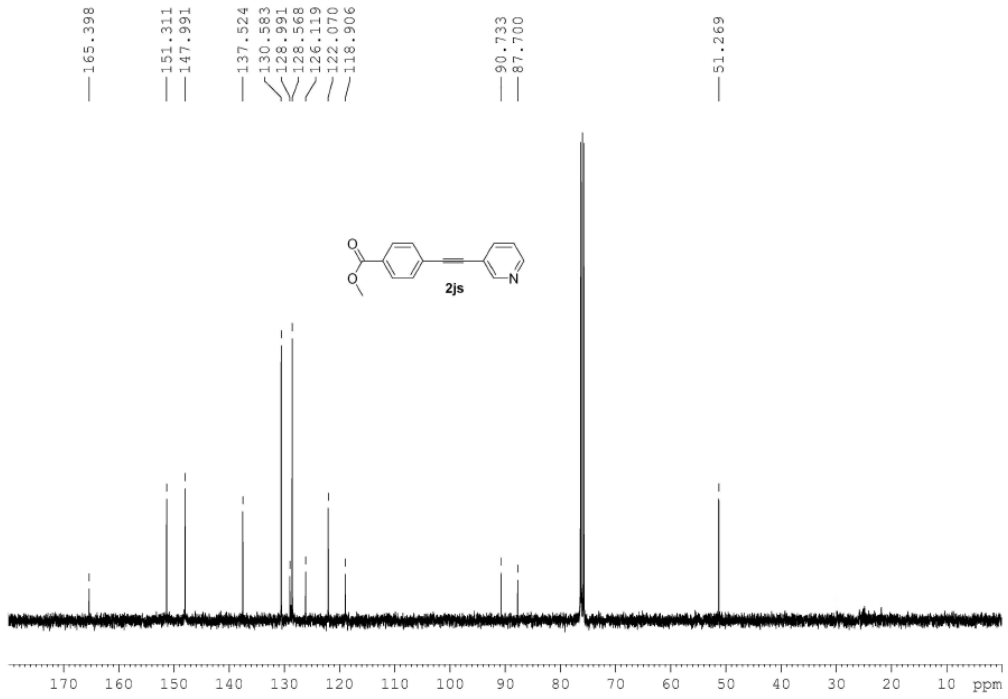
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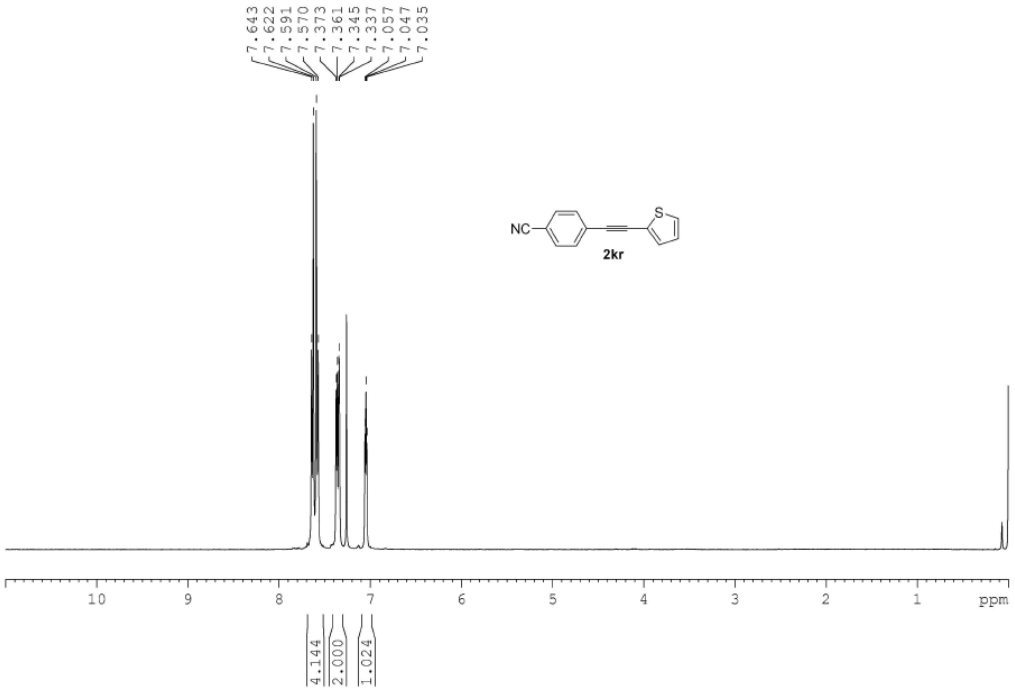
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7.570
7.373
7.361
7.345
7.337
7.057
7.047
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9
8
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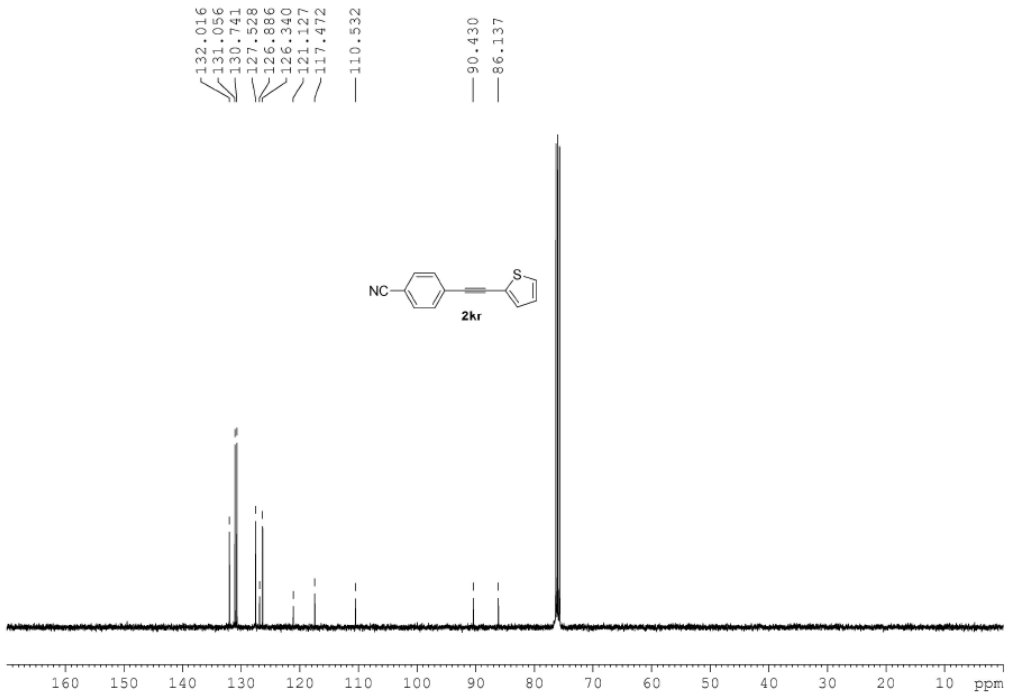
1.024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90
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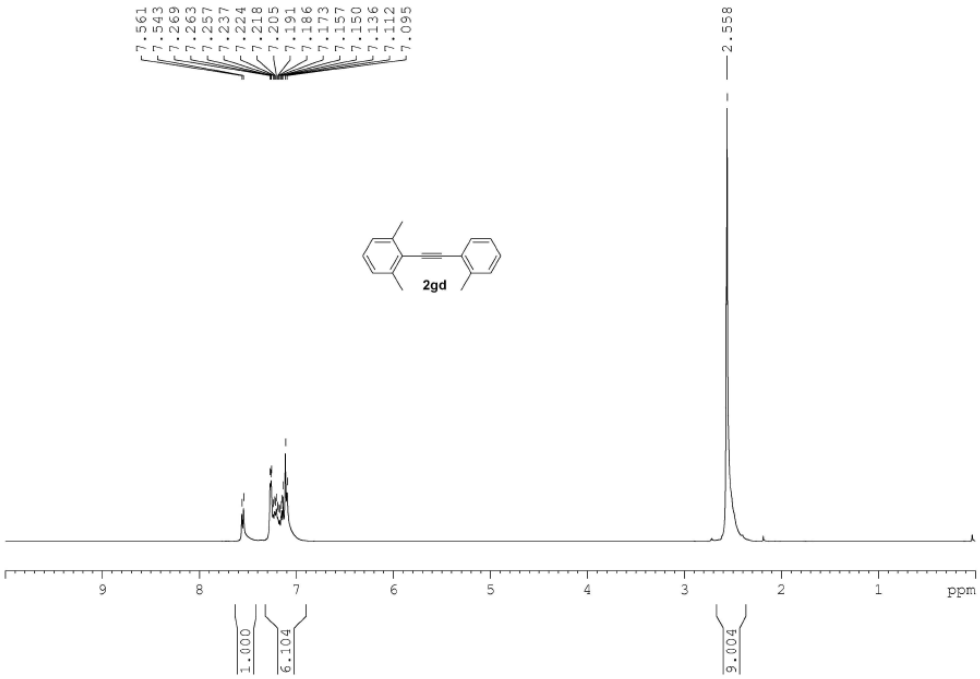
50
40
30
20
10
ppm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7.237
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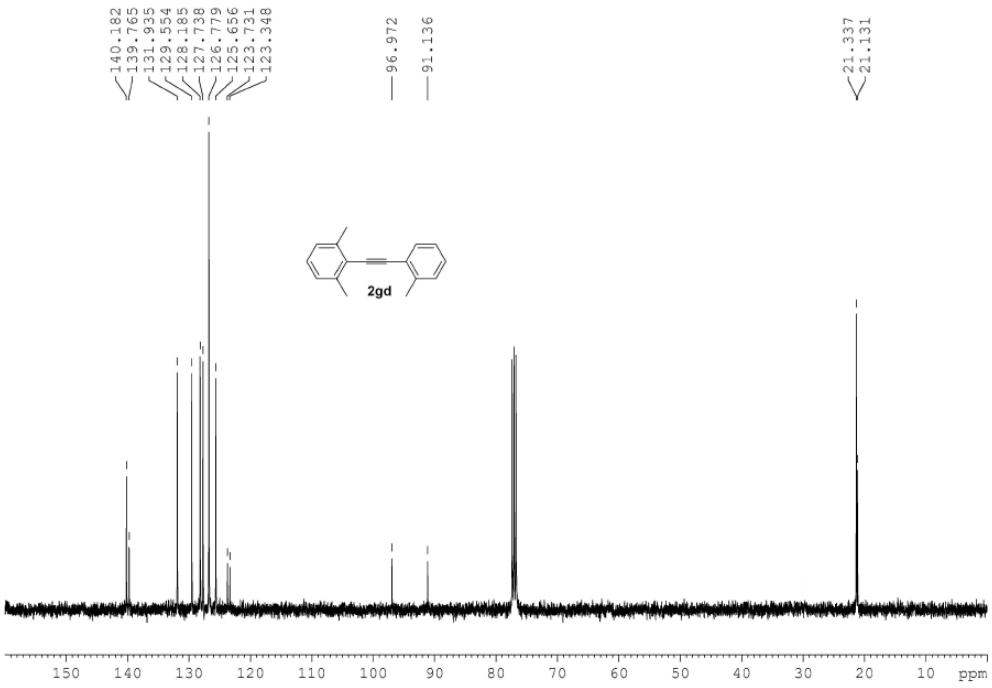
7.218
7.205
7.191
7.186
7.173
7.157
7.150
7.136
7.112
7.095
2.558
2gd
9
8
7
6
5
4
3
2
1
ppm
1.000
6.104
9.004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139.765
131.935
129.554
128.185
127.738
126.779
125.656
123.731
123.348
96.972
91.136
21.337
21.131
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150
140
130
120
110
100
90
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70
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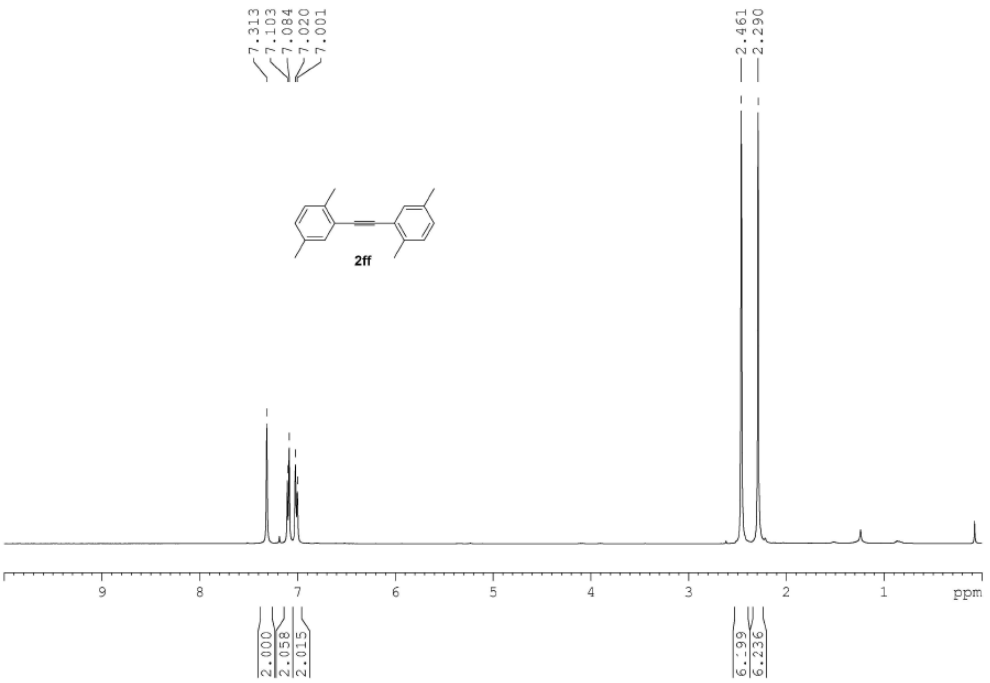
50
40
30
20
10
ppm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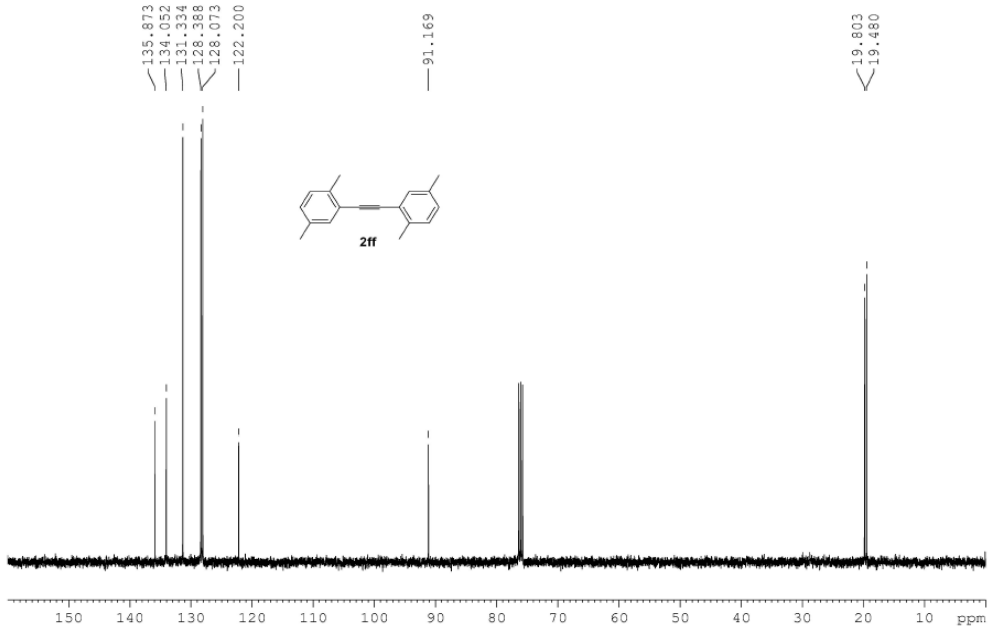
7.103
7.084
7.020
7.001
2.461
2.290
2ff
9
8
7
6
5
4
3
2
1
ppm
2.000
2.058
2.015
6.99
6.236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134.052
131.334
128.388
128.073
122.200
91.169
19.803
19.480
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130
120
110
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90
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70
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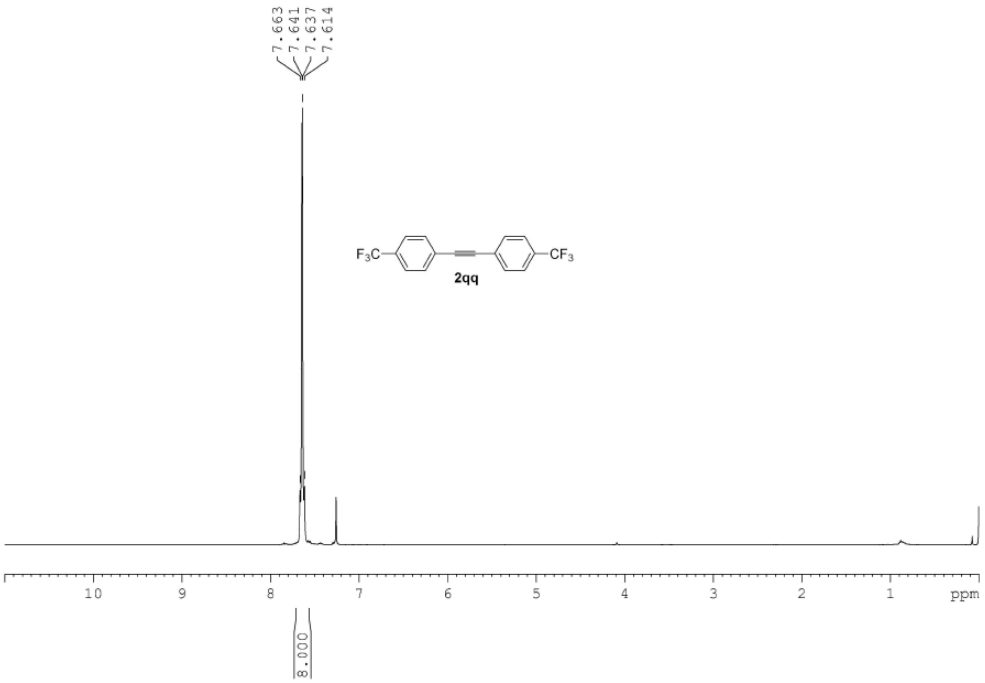
50
40
30
20
10
ppm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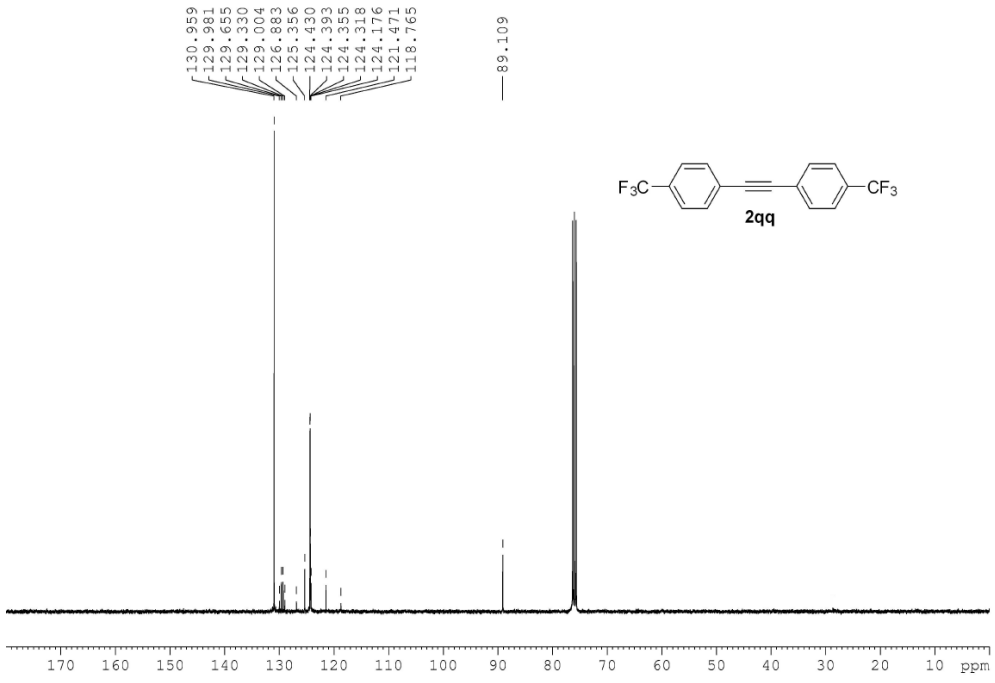
7.641
7.637
7.614
F3C
CF3
2qq
10
9
8
7
6
5
4
3
2
1
ppm
8.000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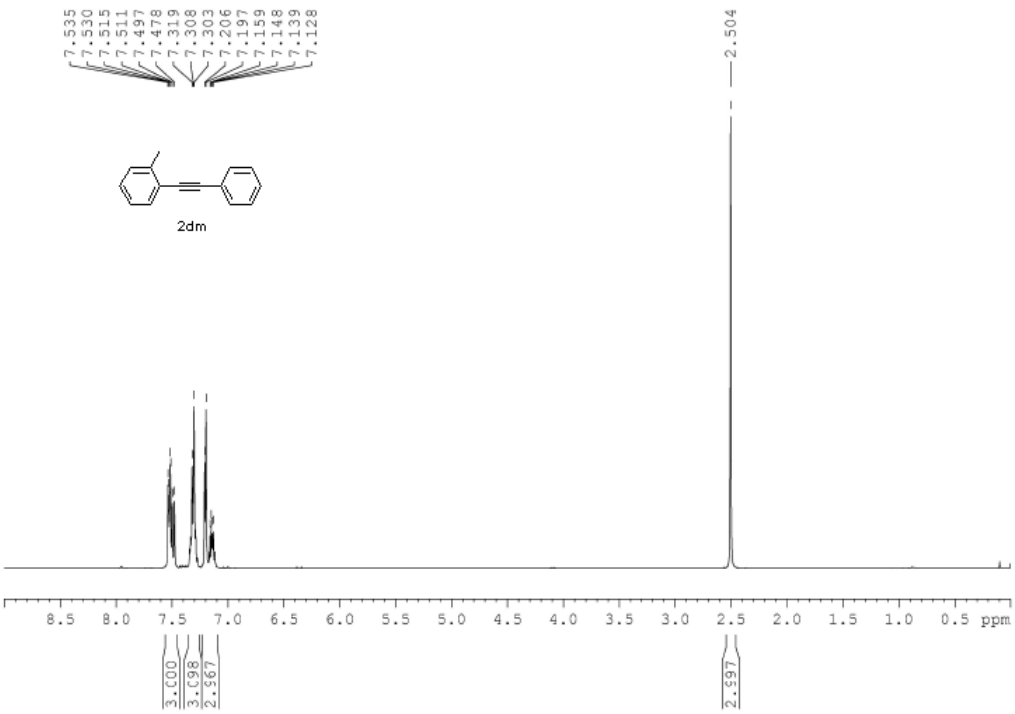
129.981
129.655
129.330
129.004
126.883
125.356
124.430
124.393
124.355
124.318
124.176
121.471
118.765
89.109
F3C
CF3
2qq
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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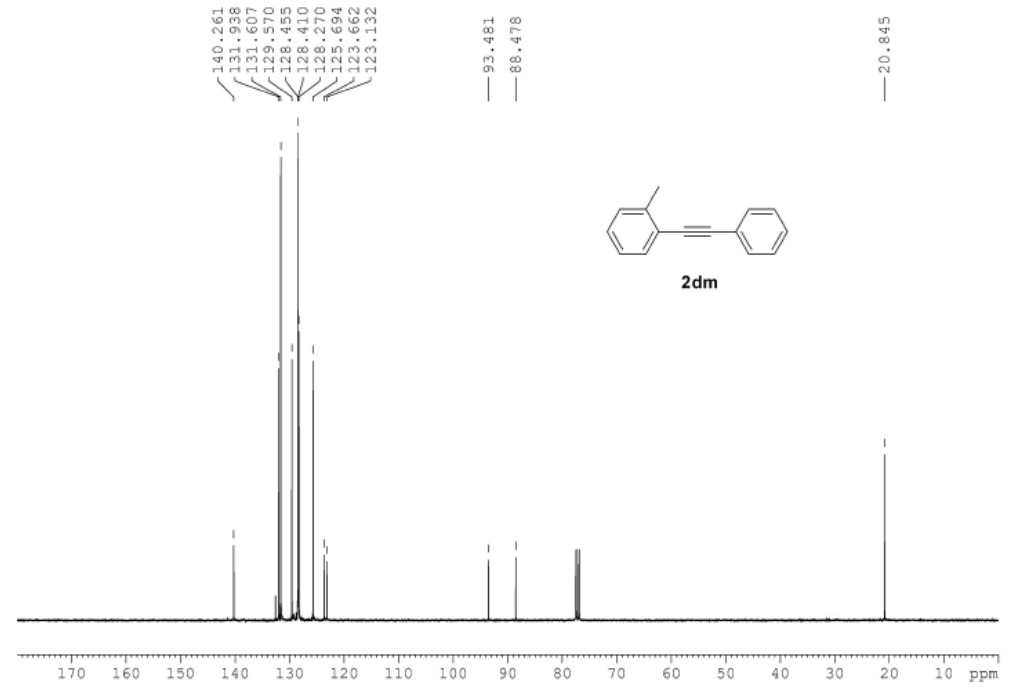
7.515
7.511
7.497
7.478
7.319
7.308
7.303
7.206
7.197
7.159
7.148
7.139
7.128
2.504
2dm
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
3.000
3.098
2.967
2.697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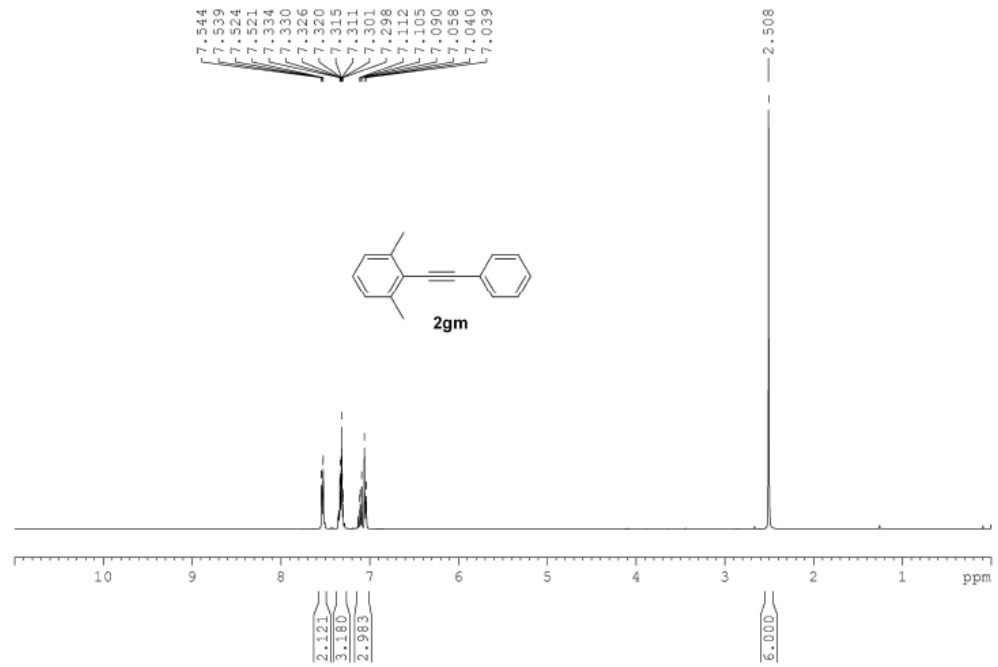
131.607
129.570
128.455
128.410
128.270
125.694
123.662
123.132
93.481
88.478
20.845
2dm
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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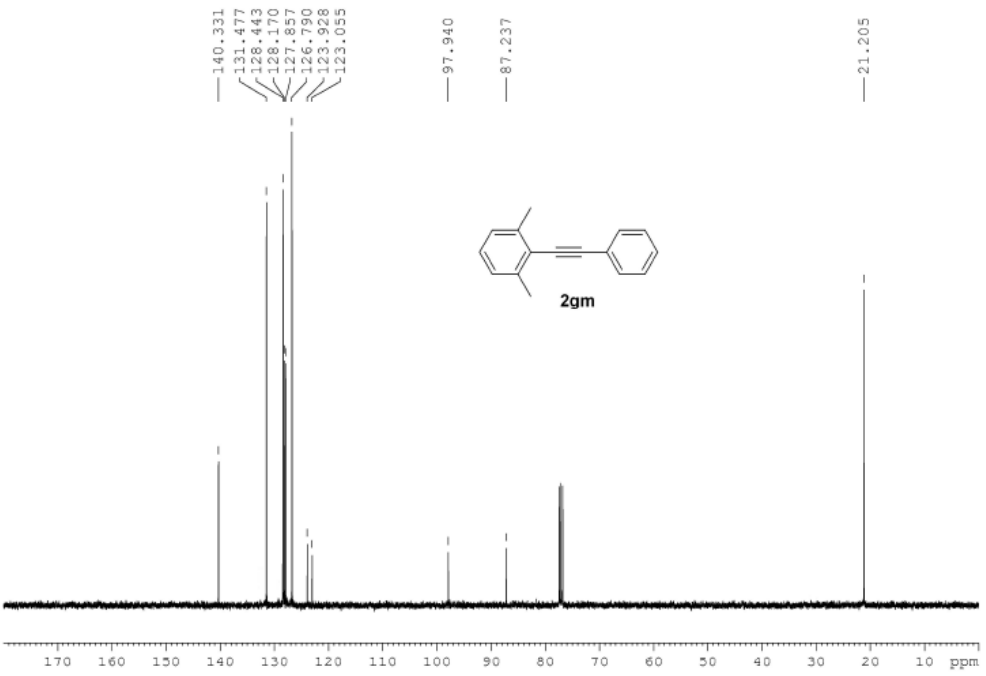
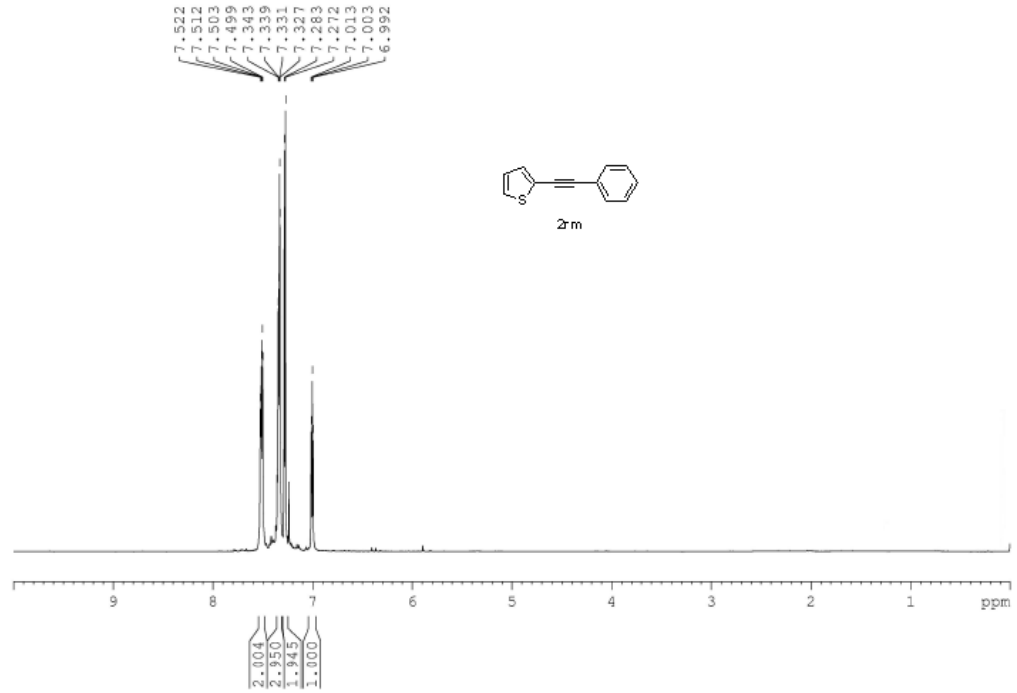
7.539
7.524
7.521
7.334
7.330
7.326
7.320
7.315
7.311
7.301
7.298
7.112
7.105
7.090
7.058
7.040
7.039
2.508
2gm
10
9
8
7
6
5
4
3
2
1
ppm
2.121
3.180
2.983
6.000


140.331
131.477
128.443
128.170
127.857
126.790
123.928
123.055
97.940
87.237
21.205
2gm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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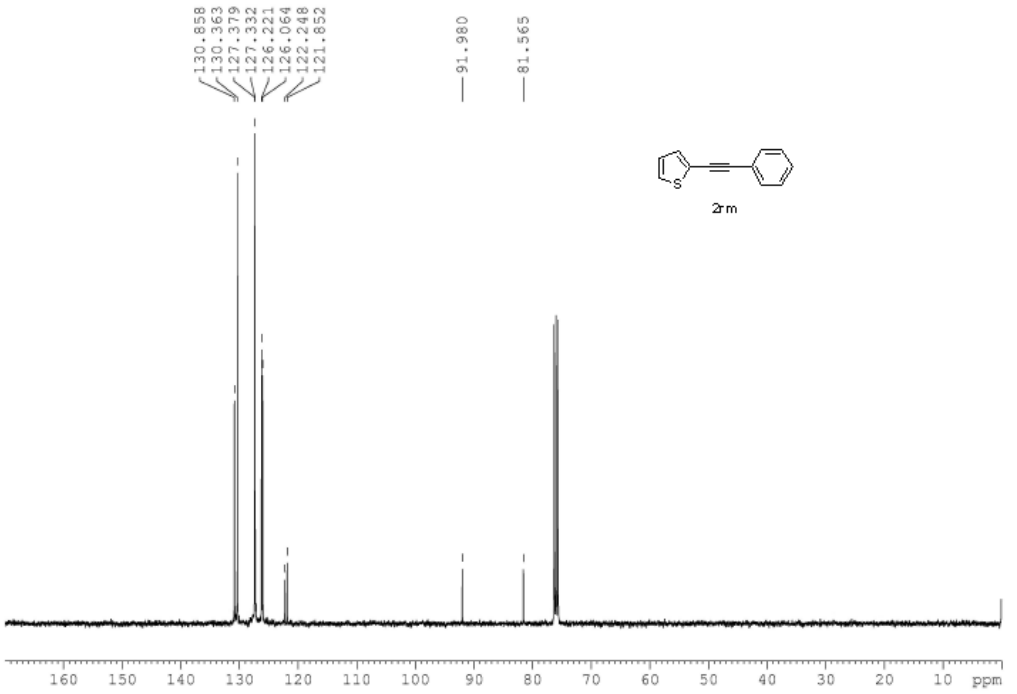
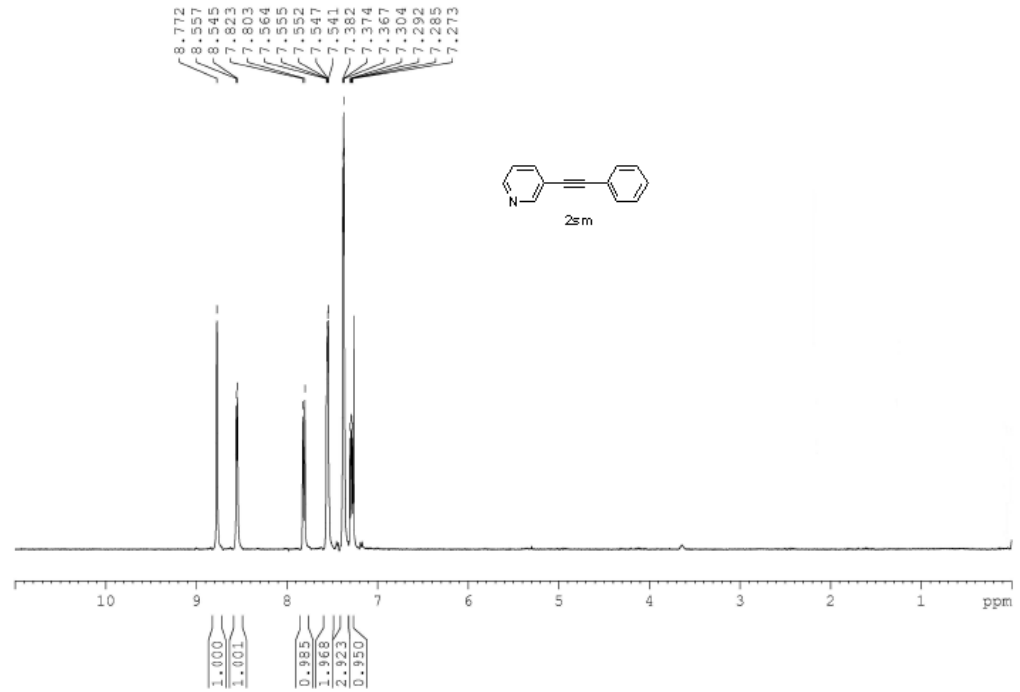
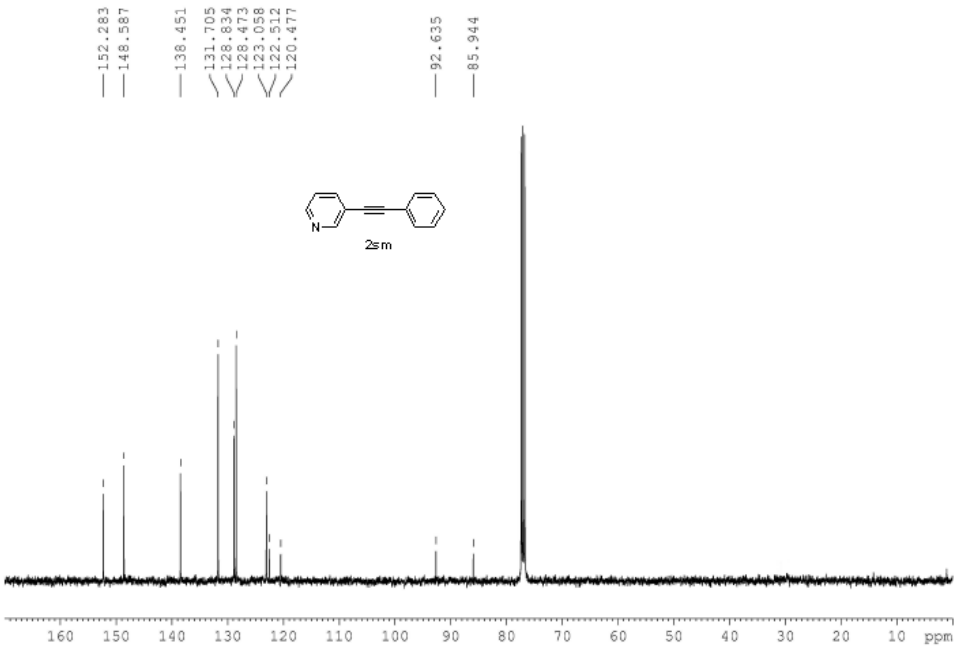
7.512
7.503
7.499
7.343
7.339
7.331
7.327
7.283
7.272
7.013
7.003
6.992
2rm
9
8
7
6
5
4
3
2
1
ppm
2.004
2.950
1.945
1.000


130.858
130.363
127.379
127.332
126.221
126.064
122.248
121.852
91.980
81.565
2rm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.772
8.557
8.545
7.823
7.803
7.564
7.555
7.552
7.547
7.541
7.382
7.374
7.367
7.304
7.292
7.285
7.273
2sm
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.001
0.985
1.968
2.923
0.950


152.283
148.587
138.451
131.705
128.834
128.473
123.058
122.512
120.477
92.635
85.944
2sm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm