Supporting Information

Palladium-Catalyzed Double Coupling Reaction of Terminal Alkynes with Isonitriles: A Direct Approach to Symmetrical *N*-Aryl Dialkynylimines

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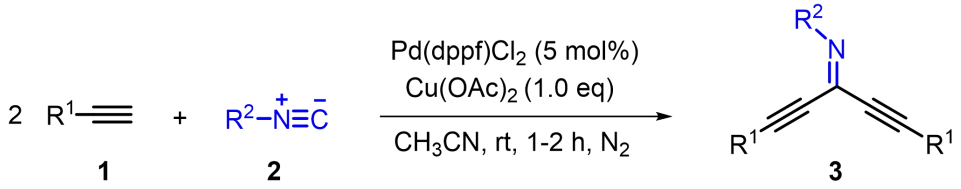
Experimental

General methods and materials. Proton nuclear magnetic resonance spectra (^{1}H NMR) and carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at 400 MHz and 100 MHz or 500 MHz and 125 MHz, respectively, using $CDCl_3$ as reference standard (δ 7.26 ppm) for ^{1}H NMR and (δ 77.04 ppm) for ^{13}C NMR. HRMS were recorded using ESI. Melting points were uncorrected. Precoated silica gel plates GF-254 were used for thin-layer analytical chromatography. Column chromatography was performed on silica gel (300-400 mesh). Unless otherwise noted, all reactions were carried out under the atmosphere of nitrogen. Solvents if necessary were dried and distilled according to standard methods prior to use. All reagents were purchased from commercial sources (Aladdin, Macklin, Adamas, and Guoyao) and used as received without further purification.

General procedure for the synthesis of *N*-aryl dialkynylimines 3.

A mixture of terminal alkynes **1** (0.2 mmol), isonitriles **2** (0.2 mmol), $Pd(dppf)Cl_2$ (0.01 mmol, 5 mol%), $Cu(OAc)_2$ (0.2 mmol, 1.0 eq) and 3.0 mL CH_3CN was stirred at rt for 1-2 h under the atmosphere of nitrogen. The progress of the reaction was monitored by thin-layer chromatography. Upon completion, the mixture was evaporated under reduced pressure, and the residue was separated by column chromatography (ethyl acetate/petroleum ether = 1:50 to 1:10) to give the pure products **3**.



1 2 3

Figure S1 X-ray data for compound 3g (CCDC 1960026)

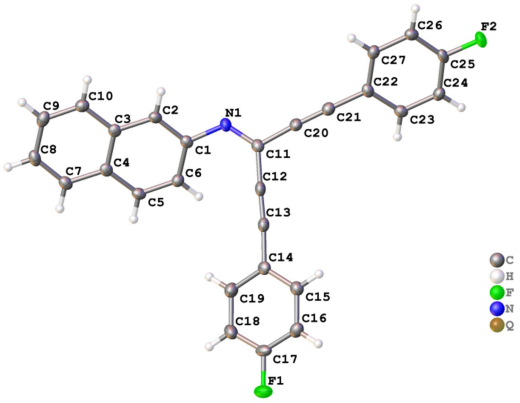


Table S1. Crystal data and structure refinement for **3g**

Compound	**3g**
Empirical formula	$C_{27}H_{15}F_2N$
Formula weight	391.40
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.8348(3)
b/Å	14.7954(9)
c/Å	21.5626(13)
α/°	90
β/°	92.427(5)
γ/°	90
Volume/Å^3	1859.79(19)
Z	4
ρ_{calc}g/cm^3	1.398
μ/mm^{-1}	0.095
F(000)	808.0
Crystal size/mm^3	0.13 × 0.12 × 0.11
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.678 to 50
Index ranges	$-6 \le h \le 6$, $-13 \le k \le 17$, $-25 \le l \le 19$
Reflections collected	7922
Independent reflections	3261 [R_{int} = 0.0239, R_{sigma} = 0.0344]
Data/restraints/parameters	3261/0/271
Goodness-of-fit on F^2	1.050
Final R indexes [I>=2σ (I)]	R_1 = 0.0389, wR_2 = 0.0847
Final R indexes [all data]	R_1 = 0.0498, wR_2 = 0.0907
Largest diff. peak/hole / e Å^{-3}	0.17/-0.20

Figure S2 X-ray data for compound 5 (CCDC 2032367)

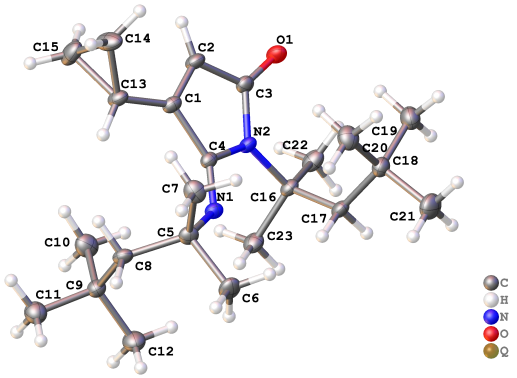


Table S2. Crystal data and structure refinement for **5**

Compound	**5**
Empirical formula	$C_{23}H_{40}N_2O$
Formula weight	360.57
Temperature/K	100.01(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.2053(5)
b/Å	9.9516(6)
c/Å	14.8729(13)
α/°	79.118(6)
β/°	88.843(6)
γ/°	68.605(6)
Volume/Å^3	1108.87(14)
Z	2
ρ_{calc}g/cm^3	1.080
μ/mm^{-1}	0.065
F(000)	400.0
Crystal size/mm^3	0.13 × 0.12 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.482 to 49.998
Index ranges	-9 ≤ h ≤ 9, -11 ≤ k ≤ 11, -17 ≤ l ≤ 12
Reflections collected	7160
Independent reflections	3897 [R_{int} = 0.0277, R_{sigma} = 0.0522]
Data/restraints/parameters	3897/0/245
Goodness-of-fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0497, wR_2 = 0.1070
Final R indexes [all data]	R_1 = 0.0654, wR_2 = 0.1161
Largest diff. peak/hole / e Å^{-3}	0.23/-0.24

Figure S3 X-ray data for compound 6c (CCDC 1960024)

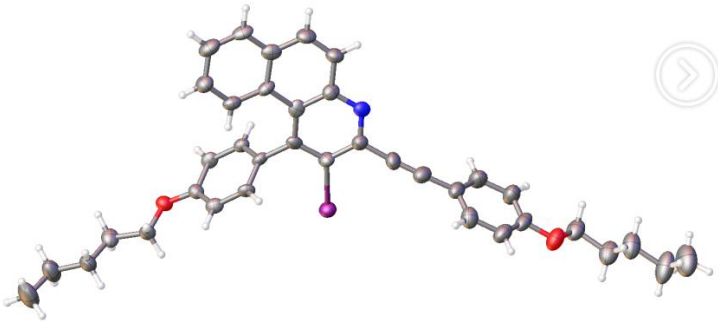
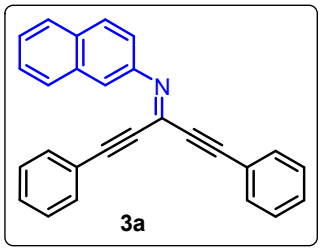


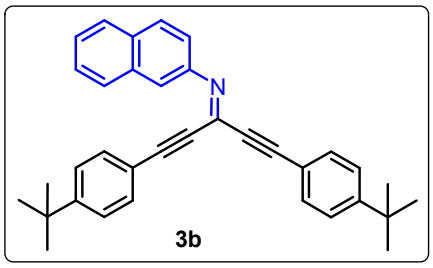
Table S3. Crystal data and structure refinement for **6c**

Compound	**6c**
Empirical formula	$C_{37}H_{34.22}INO_2$
Formula weight	651.77
Temperature/K	150.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	5.8087(3)
b/Å	11.2608(7)
c/Å	47.001(2)
α/°	93.058(5)
β/°	90.358(4)
γ/°	92.480(5)
Volume/Å^3	3067.0(3)
Z	4
ρ_{calc}g/cm^3	1.412
μ/mm^{-1}	8.455
F(000)	1329.0
Crystal size/mm^3	0.12 × 0.11 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	3.766 to 147.928
Index ranges	-7 ≤ h ≤ 4, -13 ≤ k ≤ 13, -58 ≤ l ≤ 53
Reflections collected	21375
Independent reflections	11868 [R_{int} = 0.0960, R_{sigma} = 0.1392]
Data/restraints/parameters	11868/26/808
Goodness-of-fit on F^2	1.087
Final R indexes [I>=2σ (I)]	R_1 = 0.0948, wR_2 = 0.2186
Final R indexes [all data]	R_1 = 0.1384, wR_2 = 0.2497
Largest diff. peak/hole / e Å^{-3}	1.72/-1.80

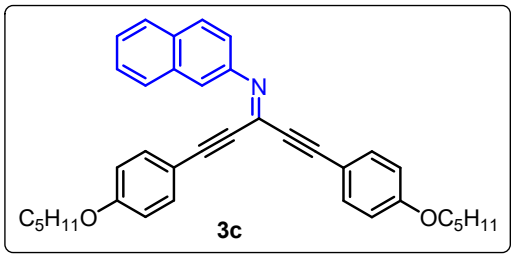
Spectral data of all compounds



***N*-(naphthalen-2-yl)-1,5-diphenylpenta-1,4-diyn-3-imin e (3a):** brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 (dd, J = 11.1, 8.3 Hz, 3H), 7.74 (s, 1H), 7.67 (dd, J = 7.6, 1.6 Hz, 2H), 7.52–7.28 (m, 9H), 7.27–7.20 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.6, 133.9, 133.6, 132.7, 132.5, 132.0, 130.2, 130.0, 128.6, 128.6, 128.4, 128.2, 127.8, 126.4, 125.8, 121.9, 121.3, 120.8, 118.9, 95.2, 90.4, 88.7, 84.5 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{18}N$ 356.1434; Found 356.1423.

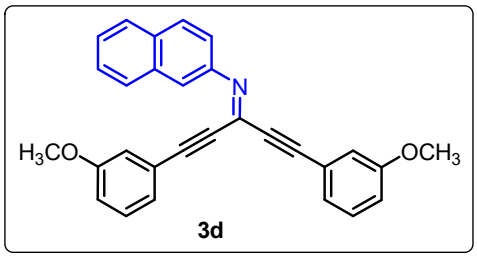


***N*-(1,5-*bis*(4-*tert*-butylphenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3b):** pale brown solid, m.p. 99.0-102.2 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (t, J = 7.9 Hz, 3H), 7.79 (s, 1H), 7.67 (d, J = 8.3 Hz, 2H), 7.55–7.45 (m, 5H), 7.31 (q, J = 9.0 Hz, 4H), 1.38 (s, 9H), 1.31 (s, 9H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.8, 153.4, 147.7, 134.3, 133.7, 132.5, 132.3, 131.9, 128.3, 128.2, 127.8, 126.3, 125.6, 125.5, 122.0, 118.8, 118.3, 117.8, 95.6, 90.8, 88.4, 84.3, 35.0, 35.0, 31.2, 31.1 ppm; HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{35}H_{34}N$ 468.2686; Found 468.2676.



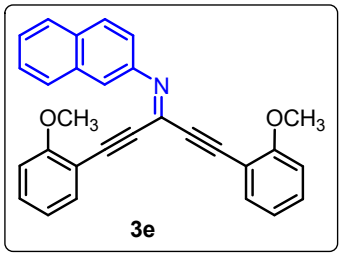
***N*-(1,5-*bis*(4-(pentyloxy)phenyl)penta-1,4 -diyn-3-ylidene)naphthalen-2-amine (3c):** pale brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 (t, J = 8.2 Hz, 3H), 7.72 (s, 1H), 7.61 (d, J = 8.7 Hz, 2H), 7.50–7.41 (m, 3H), 7.28–7.20 (m, 3H), 6.90 (d, J = 8.7 Hz, 2H), 6.76 (d, J = 8.7 Hz, 2H), 3.98 (t, J = 6.6 Hz, 2H), 3.91 (t, J = 6.6 Hz, 2H), 1.78–1.73 (m, 4H), 1.47–1.31 (m, 9H), 0.95–0.87 (m, 6H) ppm; **^{13}C NMR** (100MHz, $CDCl_3$): δ 160.7, 160.5, 147.8, 134.4, 134.4, 134.3, 133.7, 131.8, 128.2, 128.1, 127.8, 126.3, 125.4, 122.2, 118.7, 114.7,

113.0, 112.4, 96.0, 90.9, 88.1, 84.2, 68.2, 68.2, 28.9, 28.8, 28.2, 28.1, 22.5, 22.4, 14.1, 14.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{37}H_{38}O_2N$ 528.2897; Found 528.2883.



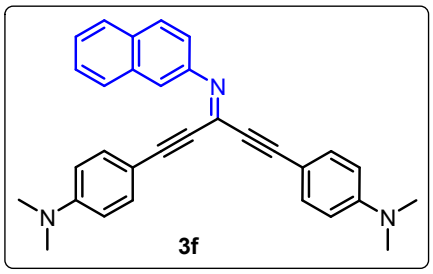
3d

***N*-(1,5-*bis*(3-methoxyphenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3d):** pale brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.84–7.74 (m, 3H), 7.65 (d, J = 1.6 Hz, 1H), 7.44–7.35 (m, 3H), 7.27–7.19 (m, 2H), 7.15–7.07 (m, 2H), 6.95–6.90 (m, 1H), 6.88–6.80 (m, 2H), 6.65 (d, J = 1.3 Hz, 1H), 3.77 (s, 3H), 3.56 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.4, 159.3, 147.7, 134.0, 133.6, 131.9, 129.6, 129.6, 128.3, 128.1, 127.8, 126.4, 125.7, 125.2, 124.9, 122.2, 121.9, 121.6, 118.6, 117.3, 117.1, 116.9, 116.7, 95.2, 90.3, 88.1, 84.2, 55.4, 55.2 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{29}H_{22}O_2N$ 416.1645; Found 416.1636.

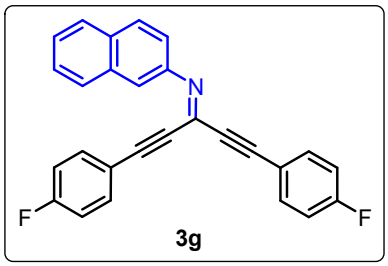


3e

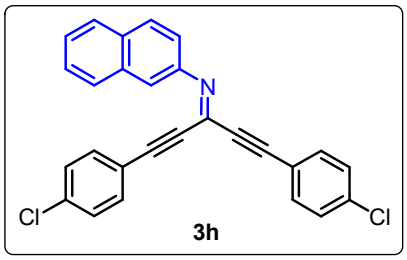
***N*-(1,5-*bis*(2-methoxyphenyl)penta-1,4-diyn-3-ylidene) naphthalen-2-amine (3e):** reddish brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.89–7.80 (m, 4H), 7.65 (dd, J = 7.6, 1.6 Hz, 1H), 7.55 (dd, J= 8.7, 2.0 Hz, 1H), 7.52–7.38 (m, 3H), 7.37–7.29 (m, 2H), 7.05–6.91 (m, 2H), 6.86 (t, J = 7.5 Hz, 1H), 6.79 (d, J = 8.6 Hz, 1H), 3.98 (s, 3H), 3.55 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 161.2, 161.1, 147.7, 134.5, 134.4, 134.2, 133.7, 131.9, 131.8, 131.4, 128.3, 128.1, 127.7, 126.0, 125.4, 122.3, 120.5, 120.4, 118.7, 110.9, 110.8, 110.7, 110.2, 92.7, 92.3, 88.6, 87.1, 55.9, 55.5 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{29}H_{22}O_2N$ 416.1645; Found 416.1635.



***N*-(1,5-*bis*(4-(dimethylamino)phenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3f):** black solid, m.p. 85.7-88.4 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 (dd, J = 8.5, 3.6 Hz, 3H), 7.56 (d, J = 8.9 Hz, 2H), 7.52–7.40 (m, 4H), 7.20–7.14 (m, 2H), 6.67 (d, J = 9.0 Hz, 2H), 6.52 (d, J = 9.0 Hz, 2H), 3.02 (s, 6H), 2.96 (s, 6H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 151.1, 151.0, 148.2, 134.9, 134.1, 134.1, 133.7, 131.6, 128.0, 128.0, 127.7, 126.0, 125.1, 122.6, 118.7, 111.6, 111.5, 107.7, 107.0, 98.0, 92.6, 88.5, 84.8, 40.1, 40.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{31}H_{28}N_3$ 442.2278; Found 442.2267.

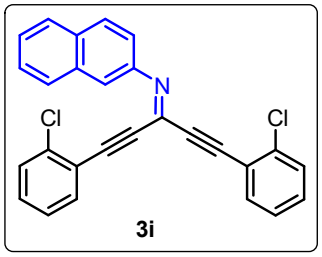


***N*-(1,5-*bis*(4-fluorophenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3g):** pale brown solid, m.p. 129.1-132.1 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.86–7.71 (m, 3H), 7.69–7.53 (m, 3H), 7.45–7.33 (m, 3H), 7.26–7.17 (m, 2H), 7.02 (t, J = 8.7 Hz, 2H), 6.89 (t, J = 8.7 Hz, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 163.6 (d, $^{1}J_{CF}$ = 252 Hz), 163.5 (d, $^{1}J_{CF}$ = 251 Hz), 147.5, 134.7 (d, $^{3}J_{CF}$ = 8.0 Hz), 134.6 (d, $^{3}J_{CF}$ = 8.0 Hz), 133.6, 133.57, 132.0, 128.4, 128.1, 127.8, 126.5, 125.8, 121.7, 118.7, 117.3 (d, $^{4}J_{CF}$ = 3.0 Hz), 116.8 (d, $^{4}J_{CF}$ = 3.0 Hz), 116.0 (d, $^{2}J_{CF}$ = 22.0 Hz), 115.9 (d, $^{2}J_{CF}$ = 22.0 Hz), 94.1, 89.3, 88.2, 84.2 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{16}NF_2$ 392.1245; Found 392.1238.



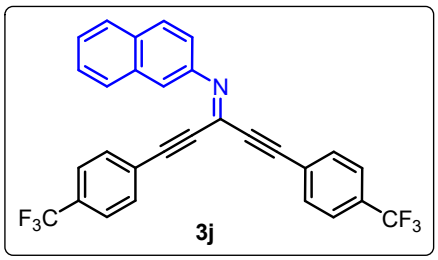
***N*-(1,5-*bis*(4-chlorophenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3h):** pale brown solid, m.p. 129.8-131.4 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.91–7.81 (m, 3H), 7.71 (d, J = 1.7 Hz, 1H), 7.64–7.57 (m, 2H), 7.52–7.43 (m, 3H), 7.41–7.36 (m, 2H), 7.27–7.22 (m, 4H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.3, 136.6, 136.2, 133.8, 133.7, 133.5, 133.3, 132.0, 129.0, 128.9, 128.4, 128.1, 127.8, 126.5, 125.9,

121.7, 119.7, 119.1, 118.8, 93.9, 89.2, 89.1, 85.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{16}NCl_2$ 424.0654; Found 424.0644.



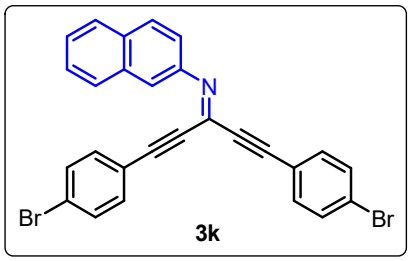
3i

***N*-(1,5-*bis*(2-chlorophenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3i):** pale brown solid, m.p. 89.2-91.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88–7.79 (m, 4H), 7.69 (dd, J = 7.6, 1.6 Hz, 1H), 7.56–7.41 (m, 4H), 7.39–7.23 (m, 5H), 7.16 (td, J = 7.5, 1.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.3, 137.2, 134.5, 134.3, 133.6, 133.1, 132.2, 131.1, 130.9, 129.6, 129.5, 128.5, 128.3, 127.8, 126.7, 126.6, 126.4, 125.8, 121.9, 121.5, 121.1, 118.8, 92.9, 91.7, 88.5, 87.2 ppm; HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{16}NCl_2$ 424.0654; Found 424.0646.



3j

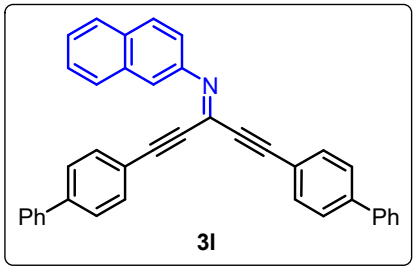
***N*-(1,5-*bis*(4-(trifluoromethyl)phenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3j):** pale brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.87 (ddd, J = 8.6, 7.3, 6.2 Hz, 3H), 7.79 (d, J = 8.1 Hz, 2H), 7.74 (d, J = 1.8 Hz, 1H), 7.68 (d, J = 8.2 Hz, 2H), 7.57–7.40 (m, 7H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.2, 133.5, 132.8, 132.7, 132.2, 131.9, 131.8, 131.6, 131.4, 128.5, 128.2, 127.8, 126.6, 126.1, 125.50 (q), 124.9 (q), 124.4, 122.4, 122.2, 121.5, 118.9, 93.1, 89.8, 88.6, 85.6 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{29}H_{16}NF_6$ 492.1181; Found 492.1169.



3k

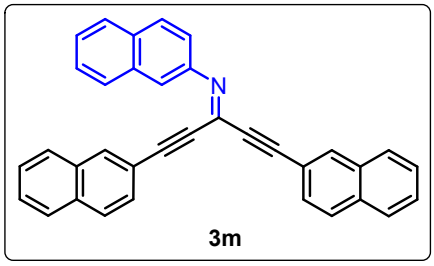
***N*-(1,5-bis(4-bromophenyl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3k):** pale brown solid, m.p. 95.8-98.5 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.86–7.72 (m, 3H), 7.64 (d, J = 1.8 Hz, 1H), 7.53–7.44 (m, 4H), 7.43–7.38 (m, 2H), 7.37–7.32 (m, 2H), 7.19 (s, 1H), 7.16–7.03 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.3, 133.9, 133.8, 133.5, 133.3, 132.1, 131.9, 131.9, 128.4, 128.1, 127.8, 126.5, 125.9,

125.0, 124.6, 121.6, 120.1, 119.6, 118.8, 94.0, 89.2, 85.1, 77.2 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{16}NBr_2$ 513.9629; Found 513.9613.



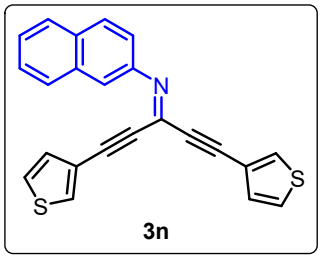
3l

1,5-Di([1,1'-biphenyl]-4-yl)-N-(naphthalen-2-yl) penta-1,4-diyn-3-imine (3l): pale brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85–7.77 (m, 3H), 7.69 (d, J = 8.3 Hz, 3H), 7.60–7.53 (m, 4H), 7.49–7.30 (m, 14H), 7.18 (s, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.6, 142.9, 142.7, 140.1, 139.9, 133.9, 133.6, 133.1, 133.0, 132.0, 129.0, 129.0, 128.3, 128.2, 128.1, 128.0, 127.8, 127.2, 127.2, 127.2, 127.1, 126.4, 125.7, 121.9, 120.1, 119.5, 118.9, 95.2, 90.4, 89.3, 85.2 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{39}H_{26}N$ 508.2060; Found 508.2047.



3m

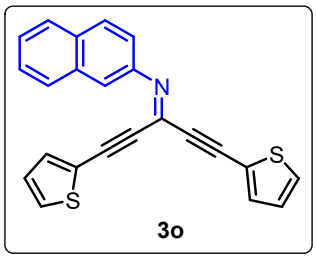
***N*-(1,5-di(naphthalen-2-yl)penta-1,4-diyn-3-yli dene)naphthalen-2-amine (3m):** pale brown solid, m.p. 129.6-131.6 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.27 (s, 1H), 7.97–7.66 (m, 12H), 7.60–7.45 (m, 7H), 7.35 (dd, J = 8.5, 1.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.7, 133.9, 133.7, 133.6, 133.6, 133.5, 132.9, 132.7, 132.1, 128.5, 128.4, 128.3, 128.2, 128.2, 128.1, 128.1, 127.9, 127.8, 127.7, 127.6, 126.9, 126.9, 126.4, 125.7, 122.0, 118.9, 118.6, 117.9, 95.8, 90.9, 88.9, 84.9 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{35}H_{22}N$ 456.1747; Found 456.1738.



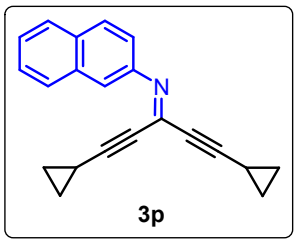
3n

***N*-(1,5-di(thiophen-3-yl)penta-1,4-diyn-3-ylidene)napht halen-2-amine (3n):** brown solid, m.p. 131.8-133.6 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.88–7.84 (m, 3H), 7.77 (dd, J = 2.8, 1.2 Hz, 1H), 7.73 (d, J = 1.9 Hz, 1H), 7.52–7.43 (m, 4H), 7.36–7.32 (m, 2H), 7.23 (dd, J = 5.0, 3.0 Hz, 1H), 6.98 (dd, J = 5.0, 1.0 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.5, 133.8, 133.6, 132.4, 132.1, 131.9, 130.2, 129.8, 128.3, 128.1, 127.8, 126.4,

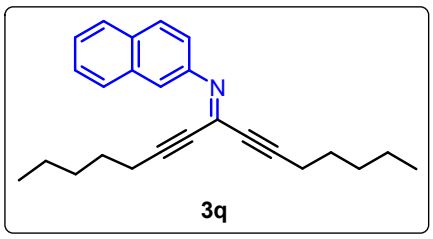
125.9, 125.8, 125.7, 121.9, 120.5, 119.9, 118.7, 90.6, 88.3, 85.7, 84.4 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{14}NS_2$ 368.0562; Found 368.0562.



***N*-(1,5-di(thiophen-2-yl)penta-1,4-diyn-3-ylidene)naphthalen-2-amine (3o):** brown solid, m.p. 66.1-68.9 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.87–7.73 (m, 3H), 7.67 (d, J = 1.6 Hz, 1H), 7.46–7.36 (m, 4H), 7.31 (dd, J = 5.1, 1.0 Hz, 1H), 7.19 (s, 1H), 7.14 (dd, J = 3.7, 1.0 Hz, 1H), 7.01 (dd, J = 5.1, 3.7 Hz, 1H), 6.90 (dd, J = 5.1, 3.7 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.3, 135.1, 135.1, 133.6, 132.9, 132.1, 130.7, 130.0, 128.4, 128.2, 127.8, 127.5, 127.5, 126.4, 125.8, 122.0, 121.2, 120.6, 118.9, 92.2, 89.2, 88.6, 84.3 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{14}NS_2$ 368.0562; Found 368.0555.

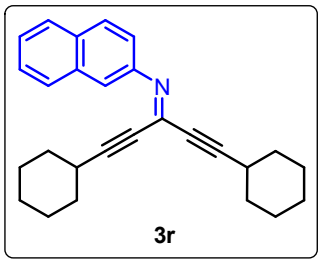


***N*-(1,5-dicyclopropylpenta-1,4-diyn-3-ylidene)naphthalen-2-amine (3p):** pale brown solid, m.p. 86.4-89.1 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.81 (dt, J = 7.8, 5.6 Hz, 3H), 7.56–7.39 (m, 3H), 7.30 (dd, J = 8.5, 1.8 Hz, 1H), 1.61–1.42 (m, 1H), 1.25 (ddd, J = 9.8, 6.6, 4.1 Hz, 1H), 1.01–0.88 (m, 4H), 0.83–0.73 (m, 2H), 0.65–0.54 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.6, 134.4, 133.4, 131.4, 127.9, 127.7, 127.6, 126.1, 125.1, 121.6, 117.9, 101.3, 95.6, 76.1, 72.0, 9.2, 9.1, 0.1, 0.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{18}N$ 284.1434; Found 284.1424.

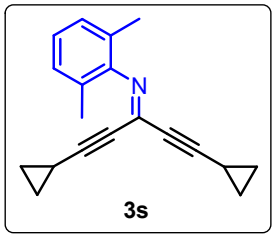


***N*-(pentadeca-6,9-diyn-8-ylidene)naphthalen-2-amine (3q):** yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.79 (t, J = 7.5 Hz, 3H), 7.51 (d, J = 1.7 Hz, 1H), 7.43 (pd, J = 6.9, 1.4 Hz, 2H), 7.29 (dd, J = 8.7, 2.0 Hz, 1H), 2.45 (t, J = 7.2 Hz, 2H), 2.20 (t, J = 7.0 Hz, 2H), 1.71 – 1.61 (m, 2H), 1.50–1.30 (m, 6H), 1.07 (td, J = 7.6, 5.0 Hz, 4H), 0.93 (t, J = 7.2 Hz, 3H), 0.71 (dd, J = 9.2, 4.7 Hz, 3H) ppm; **^{13}C NMR** (100

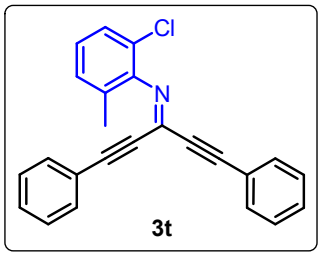
MHz, $CDCl_3$): δ 147.8, 134.9, 133.6, 131.6, 128.2, 127.9, 127.7, 126.1, 125.2, 121.6, 117.8, 97.9, 92.4, 80.8, 77.4, 31.2, 30.7, 27.8, 27.3, 22.2, 22.0, 19.4, 19.3, 13.9, 13.7 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{30}N$ 344.2373; Found 344.2367.



***N*-(1,5-dicyclohexylpenta-1,4-diyn-3-ylidene)naphthalen-2-amine (3r):** yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.79 (dd, J = 10.3, 7.8 Hz, 3H), 7.53 (d, J = 1.7 Hz, 1H), 7.46–7.39 (m, 2H), 7.30 (dd, J = 8.6, 2.0 Hz, 1H), 2.75–2.54 (m, 1H), 2.44 (tt, J = 7.7, 3.7 Hz, 1H), 1.92 (dd, J = 9.6, 3.6 Hz, 2H), 1.77 (dt, J = 9.7, 4.8 Hz, 2H), 1.65–1.54 (m, 5H), 1.47–1.28 (m, 8H), 1.21–1.08 (m, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.9, 135.2, 133.6, 131.6, 128.1, 127.9, 127.7, 126.1, 125.1, 121.7, 117.9, 101.3, 96.0, 80.9, 77.4, 32.0, 31.4, 29.7, 29.2, 25.8, 25.6, 24.9, 24.1 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{30}N$ 368.2373; Found 368.2363.

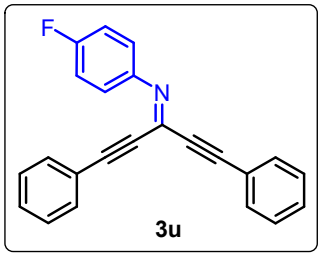


***N*-(1,5-dicyclopropylpenta-1,4-diyn-3-ylidene)-2,6-dimethylbenzenamine (3s):** yellow solid, m.p. 68.3-71.3 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.97 (d, J = 7.5 Hz, 2H), 6.91–6.85 (m, 1H), 2.02 (s, 6H), 1.52–1.40 (m, 1H), 1.15–1.05 (m, 1H), 0.97–0.88 (m, 4H), 0.70 (tt, J = 4.0, 2.7 Hz, 2H), 0.42–0.29 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.2, 136.5, 127.4, 126.1, 123.5, 101.3, 94.8, 75.0, 71.4, 17.7, 9.4, 8.9, -0.0, -0.3 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{20}N$ 262.1590; Found 262.1584.

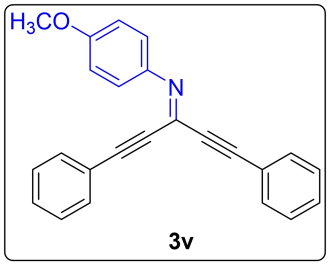


2-Chloro-*N*-(1,5-diphenylpenta-1,4-diyn-3-ylidene)-6-methylbenzenamine (3t): yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.75–7.63 (m, 2H), 7.45–7.23 (m, 7H), 7.19–7.11 (m, 3H), 7.01 (t, J = 7.8 Hz, 1H), 2.19 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.4, 138.4, 132.8, 132.6, 130.3, 130.1, 129.4, 128.6, 128.6, 128.5, 127.1, 125.0, 123.6, 121.0, 120.5,

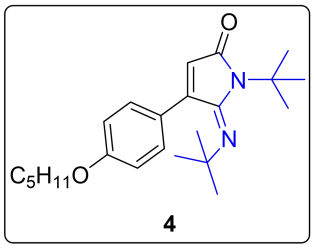
96.0, 91.1, 87.2, 83.8, 18.3 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{17}NCl$ 354.1044; Found 354.1036.



***N*-(1,5-diphenylpenta-1,4-diyn-3-ylidene)-4-fluorobenzenamine (3u):** yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 (dd, J = 7.9, 1.5 Hz, 2H), 7.44–7.25 (m, 10H), 7.16–7.06 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 161.1 (d, $^1J_{CF}$ = 244 Hz), 145.9 (d, $^4J_{CF}$ = 3.0 Hz), 133.9, 133.9, 132.6, 132.5, 130.3, 129.9, 128.6 (d, $^3J_{CF}$ = 8.0 Hz), 123.3 (d, $^3J_{CF}$ = 8.0 Hz), 121.2, 120.7, 115.4 (d, $^2J_{CF}$ = 23.0 Hz), 95.3, 90.4, 88.3, 84.1 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{15}NF$ 324.1183; Found 324.1175.

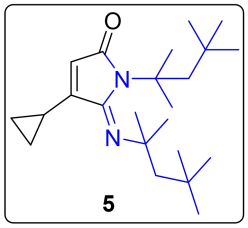


***N*-(4-methoxyphenyl)-1,5-diphenylpenta-1,4-diyn-3-imine (3v)**: yellow solid, m.p. 176.9-179.2 °C; 1**H NMR** (500 MHz, $CDCl_3$): δ 7.65 (d, *J* = 6.5 Hz, 2H), 7.47–7.34 (m, 10H), 6.95 (d, *J* = 8.9 Hz, 2H), 3.85 (s, 3H) ppm; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 158.5, 142.8, 132.5, 132.4, 131.3, 130.0, 129.7, 128.6, 128.5, 124.0, 121.5, 121.1, 113.8, 94.6, 89.6, 88.9, 84.8, 55.5 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{18}NO$ 336.1383; Found 336.1385.

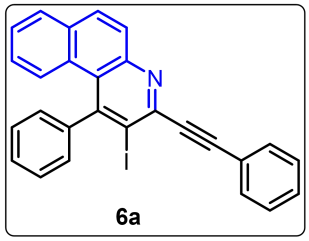


(*E*)-1-(*tert*-butyl)-5-(*tert*-butylimino)-4-(4-(pentyloxy)phenyl)-1,5-dihydro-2*H*-pyrrol-2-one (4): yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.18 (s, 1H), 3.97 (t, *J* = 6.5 Hz, 2H), 1.83–1.78 (m, 2H), 1.66 (s, 9H), 1.46–1.37 (m, 4H), 1.07 (s, 9H), 0.94 (t, *J* = 7.0 Hz, 3H) ppm; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 169.9, 159.4, 147.7, 142.8, 133.3, 129.9, 127.3, 114.0, 68.1, 57.3, 56.0, 32.1, 29.8, 28.9, 28.2, 22.5, 14.1 ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 159.4, 147.7, 142.8, 133.3, 129.9,

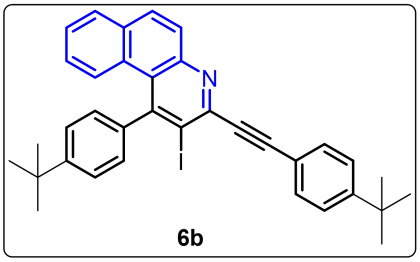
127.3, 114.0, 68.1, 57.3, 56.0, 32.1, 29.8, 28.9, 28.2, 22.5, 14.1 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{35}N_2O_2$ 371.2693; Found 371.2699.



(*Z*)-4-cyclopropyl-1-(2,4,4-trimethylpentan-2-yl)-5-((2,4,4-trimethylpentan-2-yl)imino)-1,5-dihydro-2*H*-pyrrol-2-one (5): yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.78 (d, *J* = 0.8 Hz, 1H), 1.99 (s, 2H), 1.94 (s, 2H), 1.92–1.84 (m, 1H), 1.70 (s, 6H), 1.58 (s, 6H), 1.10–1.04 (m, 2H), 1.01 (s, 9H), 0.92 (s, 9H), 0.77–0.70 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.3, 149.3, 146.1, 126.0, 60.9, 59.2, 55.1, 50.4, 33.1, 32.1, 31.9, 31.7, 31.6, 31.2, 12.1, 12.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{41}N_2O$ 361.3213; Found 361.3219.

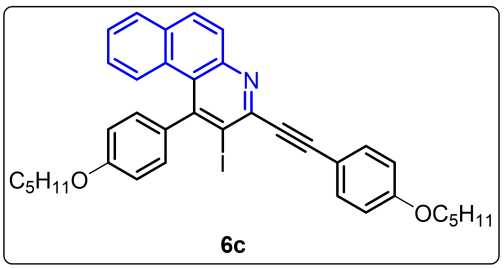


2-Iodo-1-phenyl-3-(2-phenylethynyl)benzo[f]quinoline (6a): yellow solid, m.p. 84.5-85.3 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.08 (m, 2H), 7.89 (t, J = 9.2 Hz, 1H), 7.78 (d, J = 3.1 Hz, 1H), 7.71–7.56 (m, 4H), 7.56–7.37 (m, 5H), 7.34–7.29 (m, 2H), 7.18 (d, J = 7.3 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 162.3, 154.2, 153.2, 148.7, 146.0, 133.1, 132.3, 129.9, 129.5, 129.0, 128.9, 128.7, 128.5, 128.5, 128.4, 128.3, 127.4, 126.5, 124.5, 122.0, 105.7, 101.0, 91.9 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{17}IN$ 482.0400; Found 482.0402.



1-(4-*Tert*-butylphenyl)-3-(2-(4-tert-butylphenyl)ethynyl)-2-iodobenzo[f]quinoline (6b): yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15–7.98 (m, 2H), 7.90–7.83 (m, 1H), 7.79–7.59 (m, 3H), 7.52 (dd, J = 33.4, 10.2 Hz, 4H), 7.29–7.05 (m, 4H), 1.50 (s, 9H), 1.38 (s, 9H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 162.5, 148.9, 143.1, 142.8, 142.5, 133.5, 132.6, 132.1, 129.2, 128.9, 128.6, 128.5, 128.3, 128.2, 127.1, 126.6,

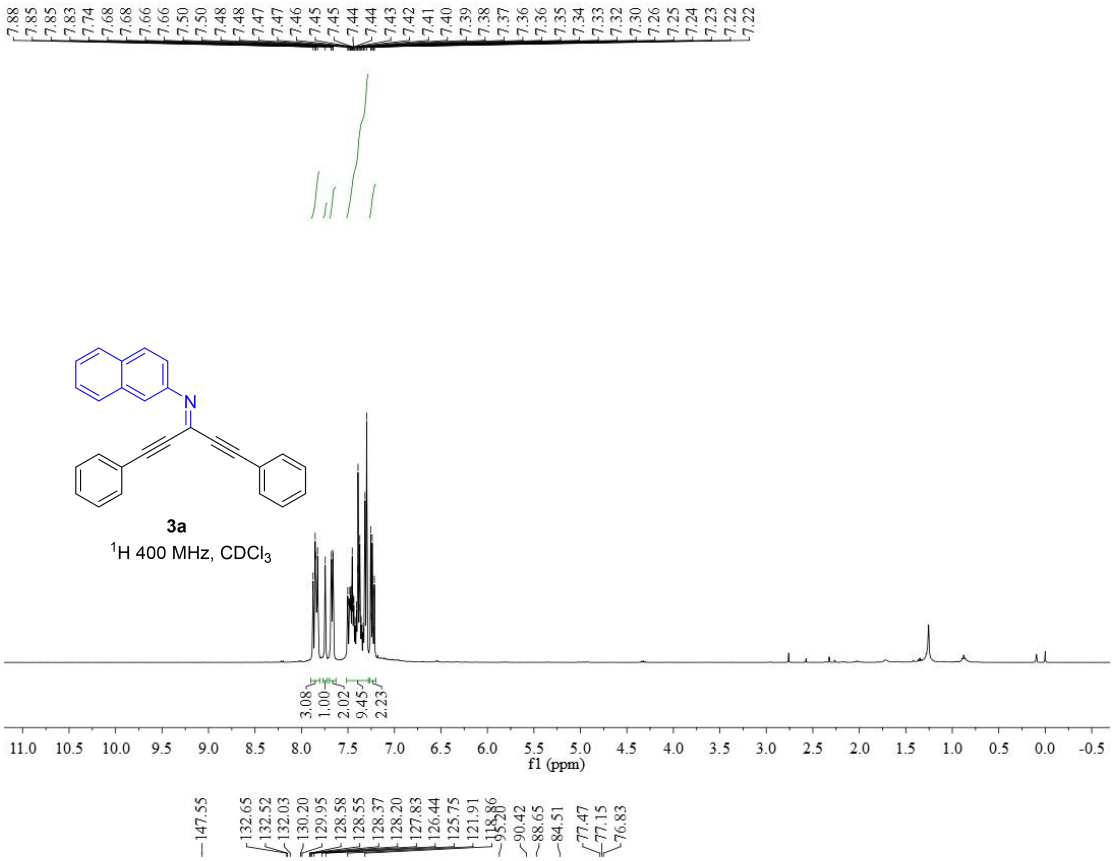
126.2, 125.5, 125.3, 119.1, 105.9, 101.4, 91.7, 34.9, 34.8, 31.5, 31.3 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{35}H_{33}IN$ 594.1652; Found 594.1655.

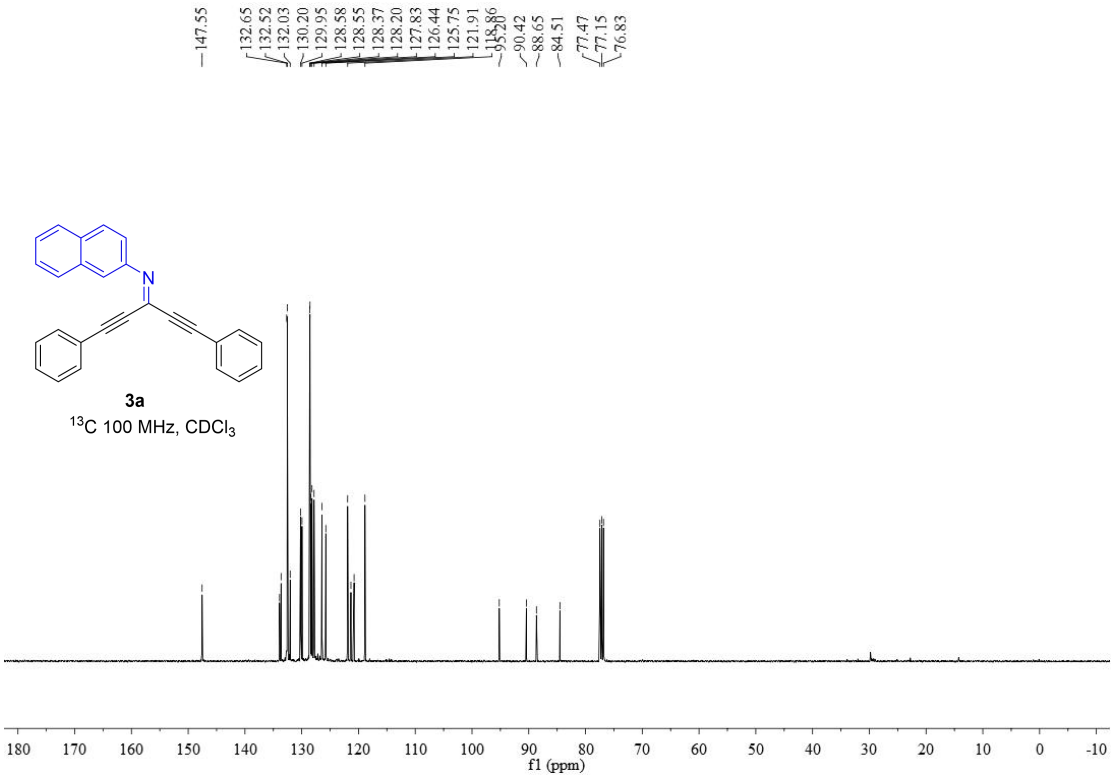


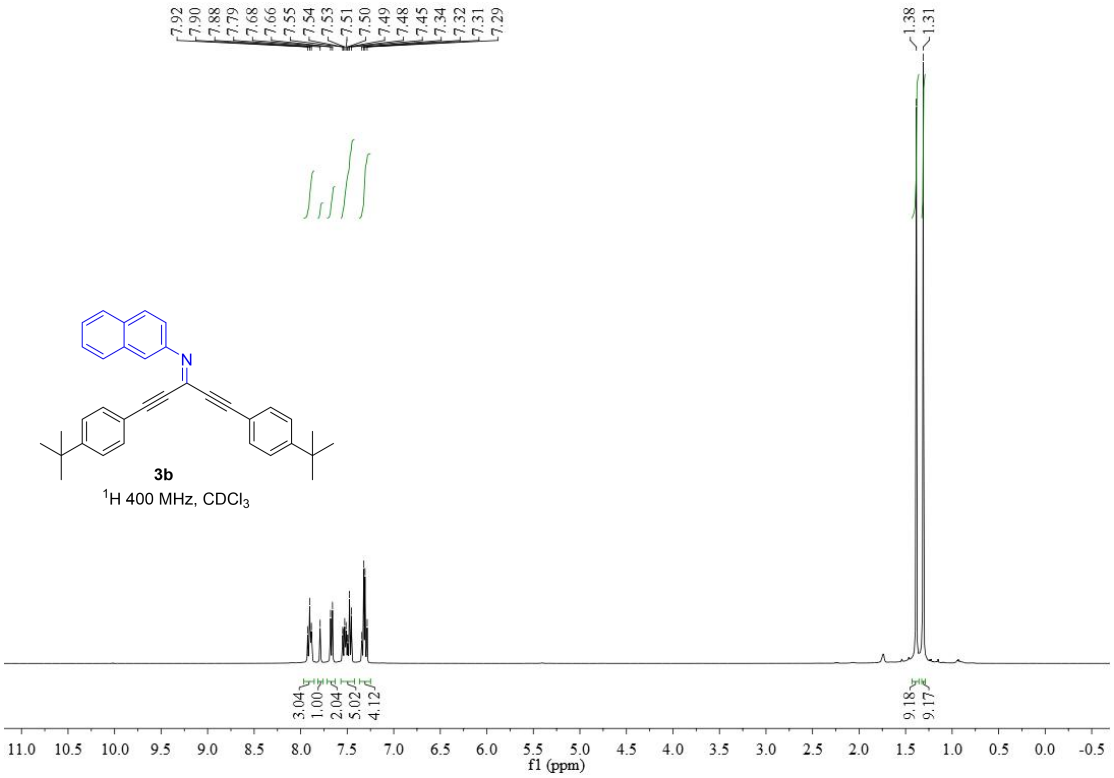
6c

2-Iodo-1-(4-(pentyloxy)phenyl)-3-(2-(4-(pentyloxy)phenyl)ethynyl)benzo[f]quinoline (6c): yellow solid, m.p. 88.2-90.3 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (dt, J = 14.8, 8.9 Hz, 2H), 7.78 (t, J = 7.0 Hz, 1H), 7.60 (d, J = 8.7 Hz, 1H), 7.55–7.30 (m, 3H), 7.21–7.03 (m, 5H), 6.92–6.74 (m, 2H), 4.03 (t, J = 6.5 Hz, 2H), 3.92 (m, 2H), 1.87–1.78 (m, 2H), 1.72 (m, 2H), 1.50–1.30 (m, 8H), 0.88 (m, 6H) ppm; **^{13}C NMR** (100MHz, $CDCl_3$) δ 160.4, 159.6, 153.7, 138.0, 134.0, 133.2, 132.6, 130.7, 130.1, 129.8, 129.0, 128.5, 128.2, 127.3, 126.6, 124.7, 115.8, 114.7, 114.2, 113.6, 106.9, 102.2, 91.3, 68.3, 68.2, 29.0, 28.9, 28.3, 28.2, 22.6, 22.5, 14.1, 14.0 ppm; **HRMS** (ESI) m/z: $[M+H]^+$ Calcd for $C_{37}H_{37}INO_2$ 654.1863; Found 654.1858.

Copies of ^{1}H and ^{13}C NMR spectra of all compounds






7.92
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3b
1H 400 MHz, CDCl3
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1.00
2.04
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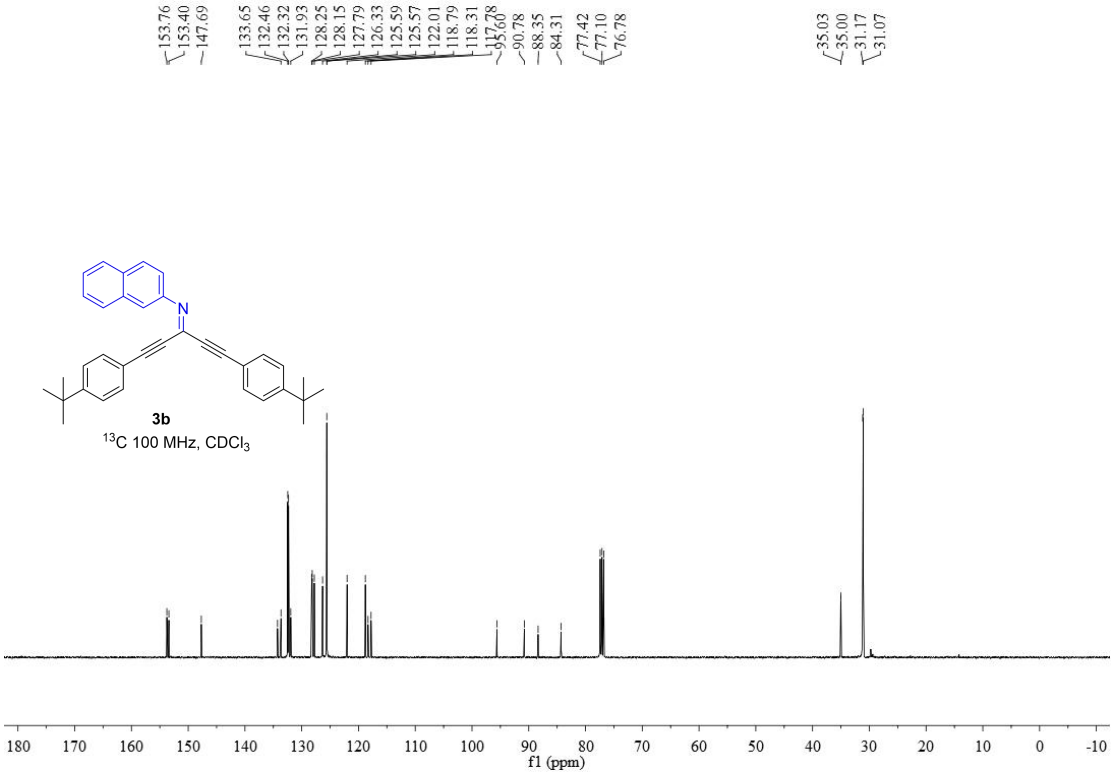
4.12
9.18
9.17
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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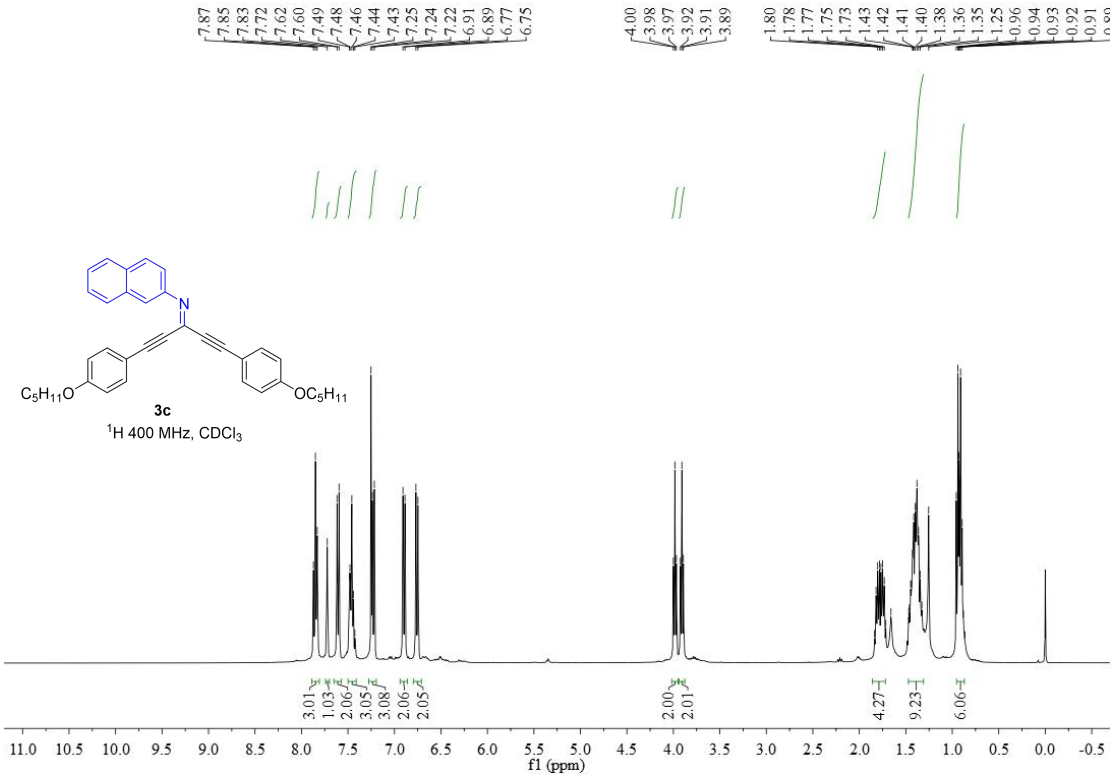
153.40
147.69
133.65
132.46
132.32
131.93
128.25
128.15
127.79
126.33
125.59
125.57
122.01
118.79
118.31
117.78
95.60
90.78
88.35
84.31
77.42
77.10
76.78
35.03
35.00
31.17
31.07
N
3b
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.85
7.83
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7.48
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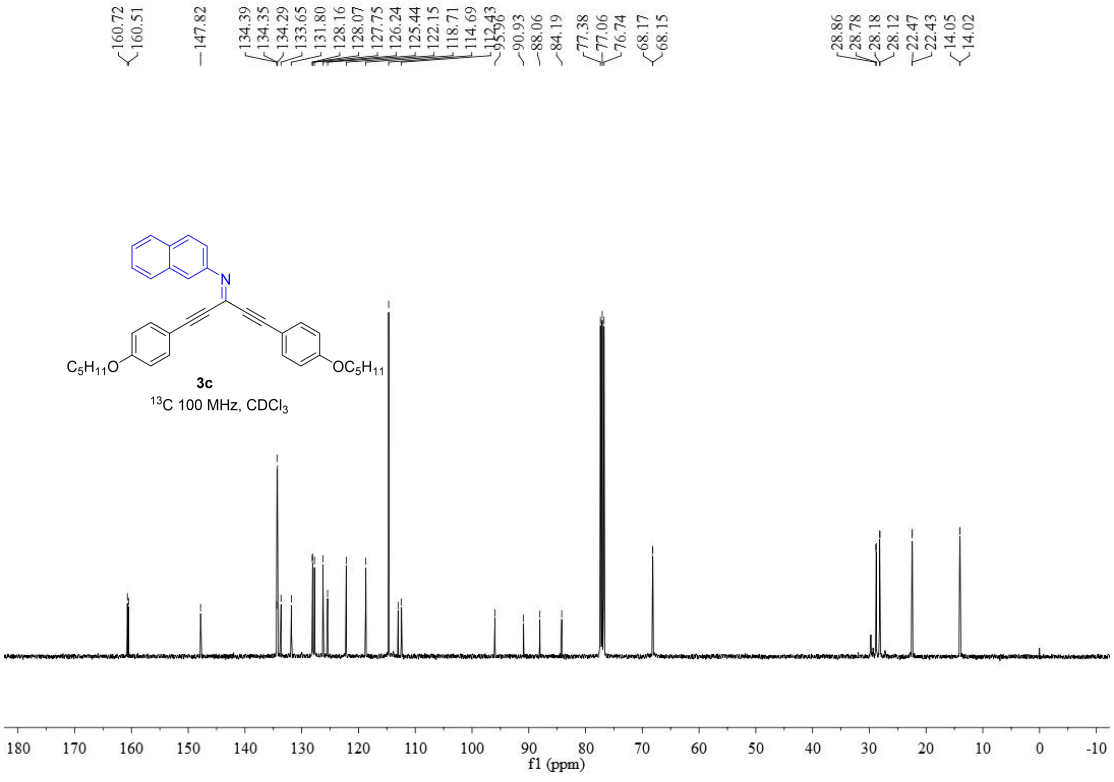
7.43
7.25
7.24
7.22
6.91
6.89
6.77
6.75
4.00
3.98
3.97
3.92
3.91
3.89
1.80
1.78
1.77
1.75
1.73
1.43
1.42
1.41
1.40
1.38
1.36
1.35
1.25
0.96
0.94
0.93
0.92
0.91
N
C5H11O
OC5H11
3c
1H 400 MHz, CDCl3
3.01
1.03
2.06
3.05
3.08
2.06
2.05
2.00
2.01
4.27
9.23
6.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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134.29
133.65
131.80
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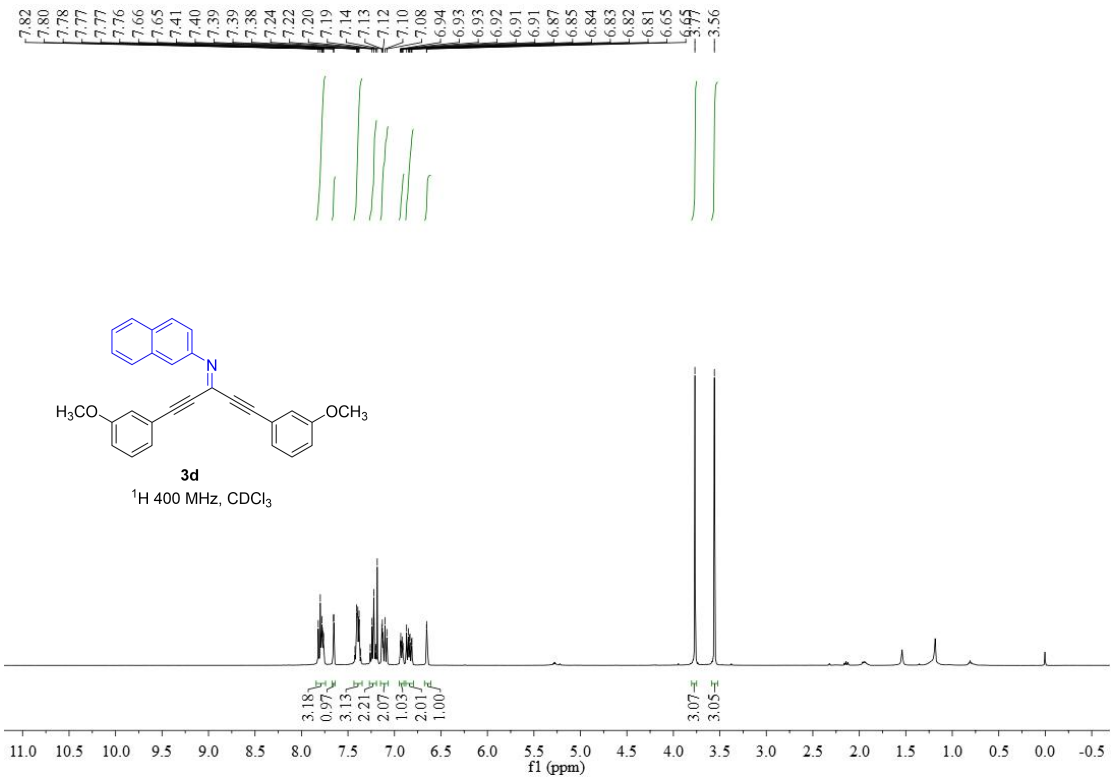
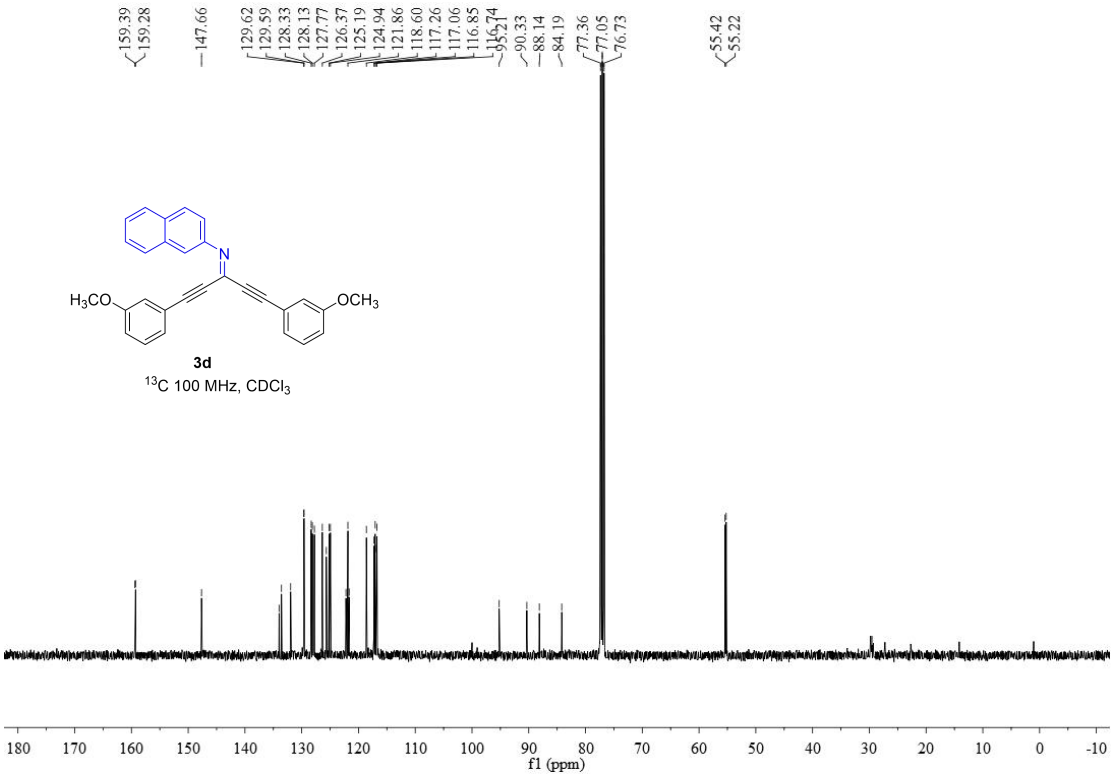
128.07
127.75
126.24
125.44
122.15
118.71
114.69
112.43
95.96
90.93
88.06
84.19
77.38
77.06
76.74
68.17
68.15
28.86
28.78
28.18
28.12
22.47
22.43
14.05
14.02
N
C5H11O
OC5H11
3c
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.82
7.80
7.78
7.77
7.77
7.76
7.66
7.65
7.41
7.40
7.39
7.39
7.38
7.24
7.22
7.20
7.19
7.14
7.13
7.12
7.10
7.08
6.94
6.93
6.93
6.92
6.91
6.91
6.87
6.85
6.84
6.83
6.82
6.81
6.65
3.77
3.56
N
H3CO
OCH3
3d
1H 400 MHz, CDCl3
3.18
0.97
3.13
2.21
2.07
1.03
2.01
1.00
3.07
3.05
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)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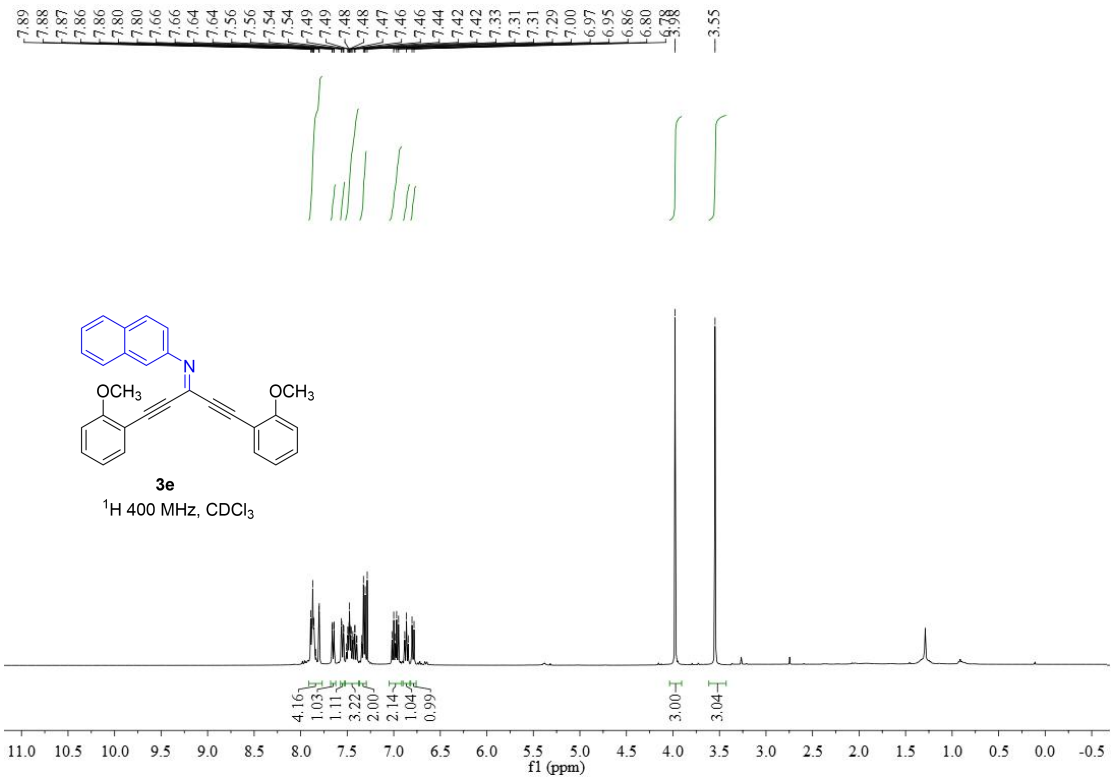
159.28
147.66
129.62
129.59
128.33
128.13
127.77
126.37
125.19
124.94
121.86
118.60
117.26
117.06
116.85
116.74
95.21
90.33
88.14
84.19
77.36
77.05
76.73
55.42
55.22
N
H3CO
OCH3
3d
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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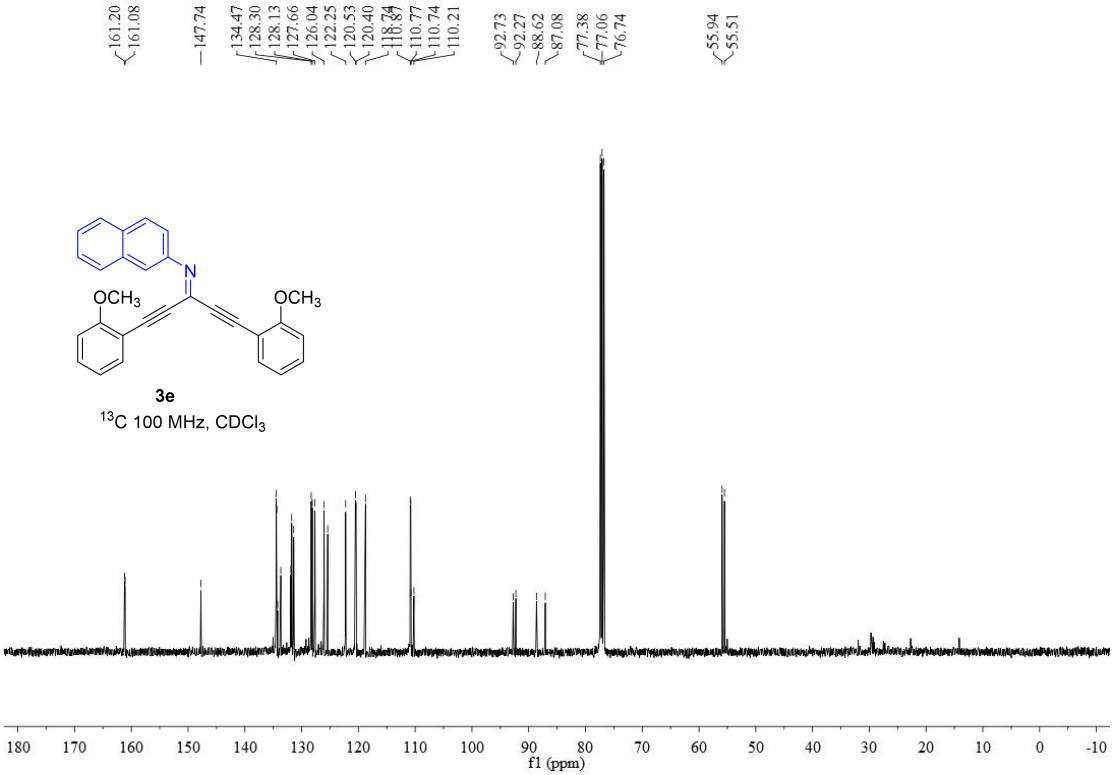
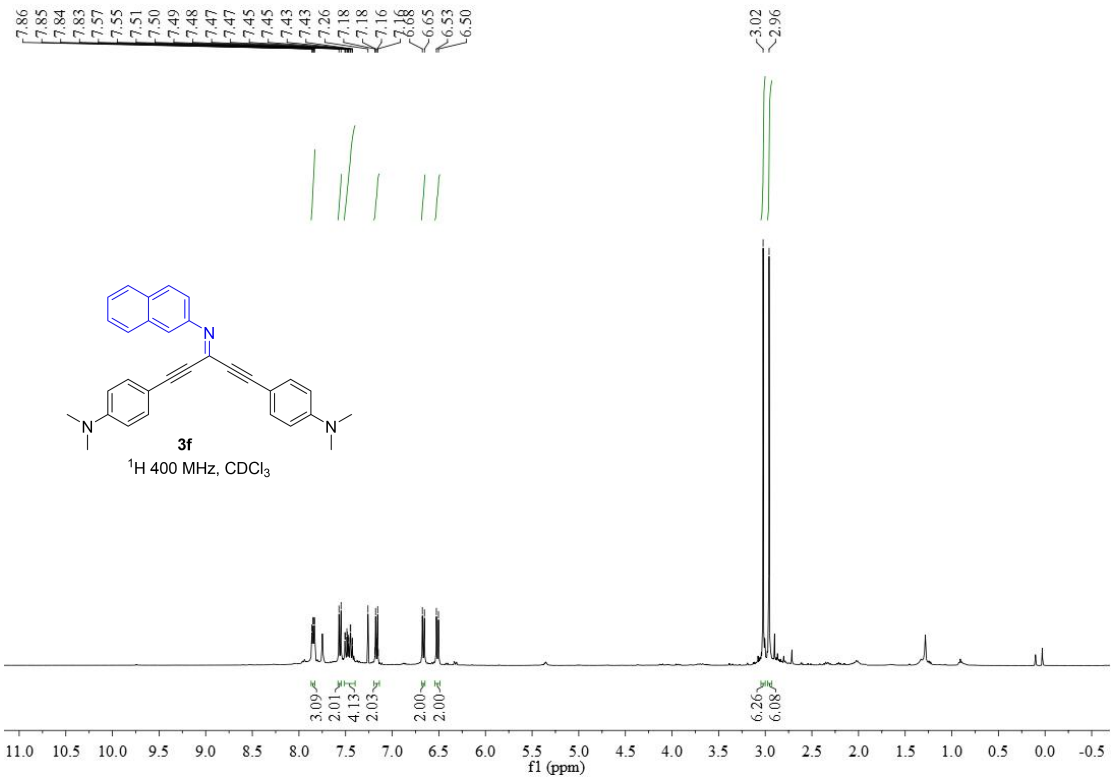
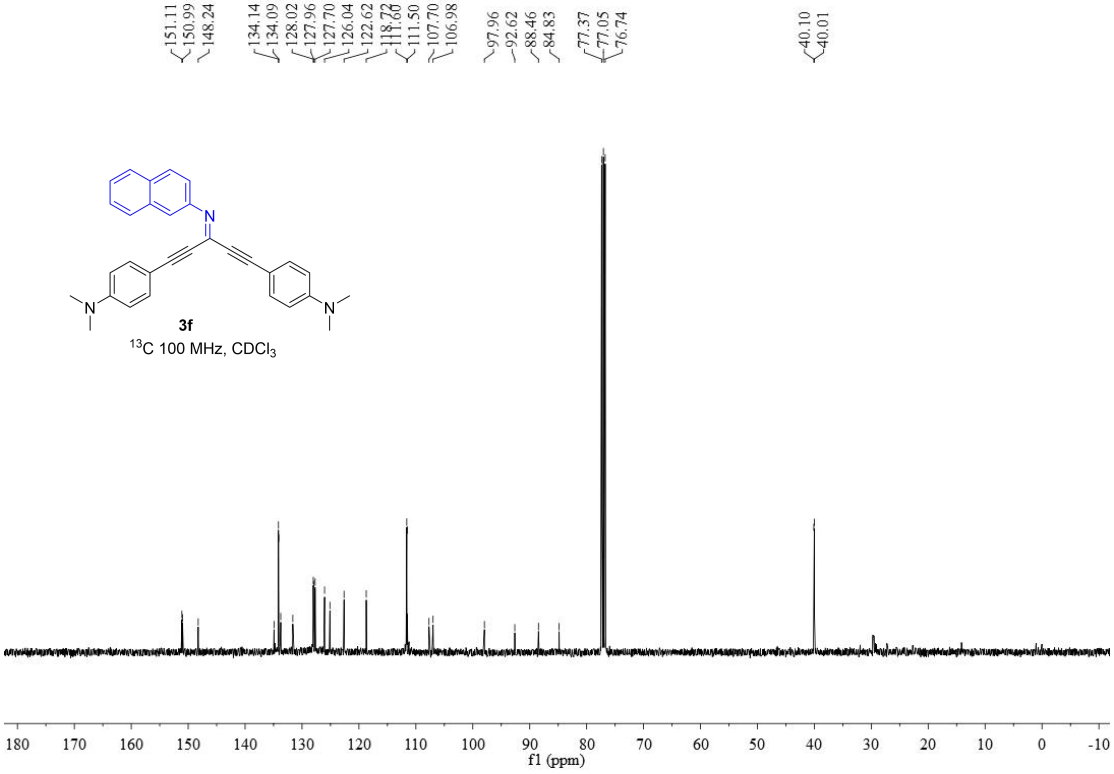
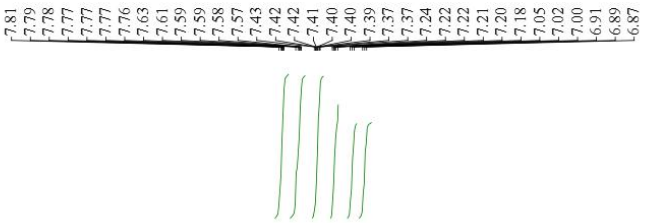
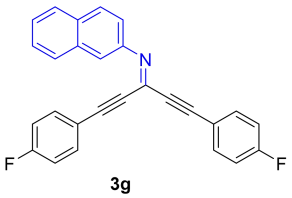
7.88
7.87
7.86
7.86
7.80
7.80
7.66
7.66
7.64
7.64
7.56
7.56
7.54
7.54
7.49
7.49
7.48
7.48
7.47
7.46
7.46
7.44
7.42
7.42
7.33
7.31
7.31
7.29
7.00
6.97
6.95
6.86
6.80
6.78
3.98
3.55
N
OCH3
OCH3
3e
1H 400 MHz, CDCl3
4.16
1.03
1.11
3.22
2.00
2.14
1.04
0.99
3.00
3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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 0.0 -0.5
f1 (ppm)


161.20
161.08
147.74
134.47
128.30
128.13
127.66
126.04
122.25
120.53
120.40
110.87
110.77
110.74
110.21
92.73
92.27
88.62
87.08
77.38
77.06
76.74
55.94
55.51
N
OCH3
OCH3
3e
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.86
7.85
7.84
7.83
7.57
7.55
7.51
7.50
7.49
7.48
7.47
7.47
7.45
7.45
7.43
7.43
7.26
7.18
7.18
7.16
7.16
6.68
6.65
6.53
6.50
3.02
2.96
3.09
2.01
4.13
2.03
2.00
2.00
6.26
6.08
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)
3f
1H 400 MHz, CDCl3

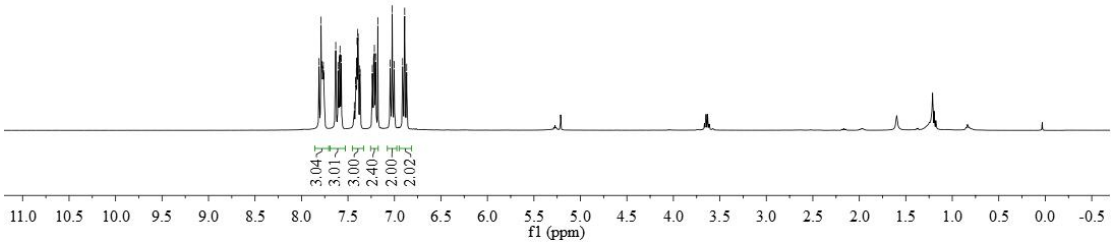

151.11
150.99
148.24
134.14
134.09
128.02
127.96
127.70
126.04
122.62
118.72
111.60
111.50
107.70
106.98
97.96
92.62
88.46
84.83
77.37
77.05
76.74
40.10
40.01
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
3f
13C 100 MHz, CDCl3

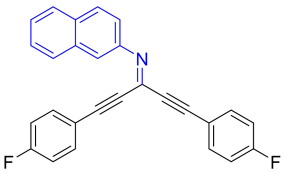




3g

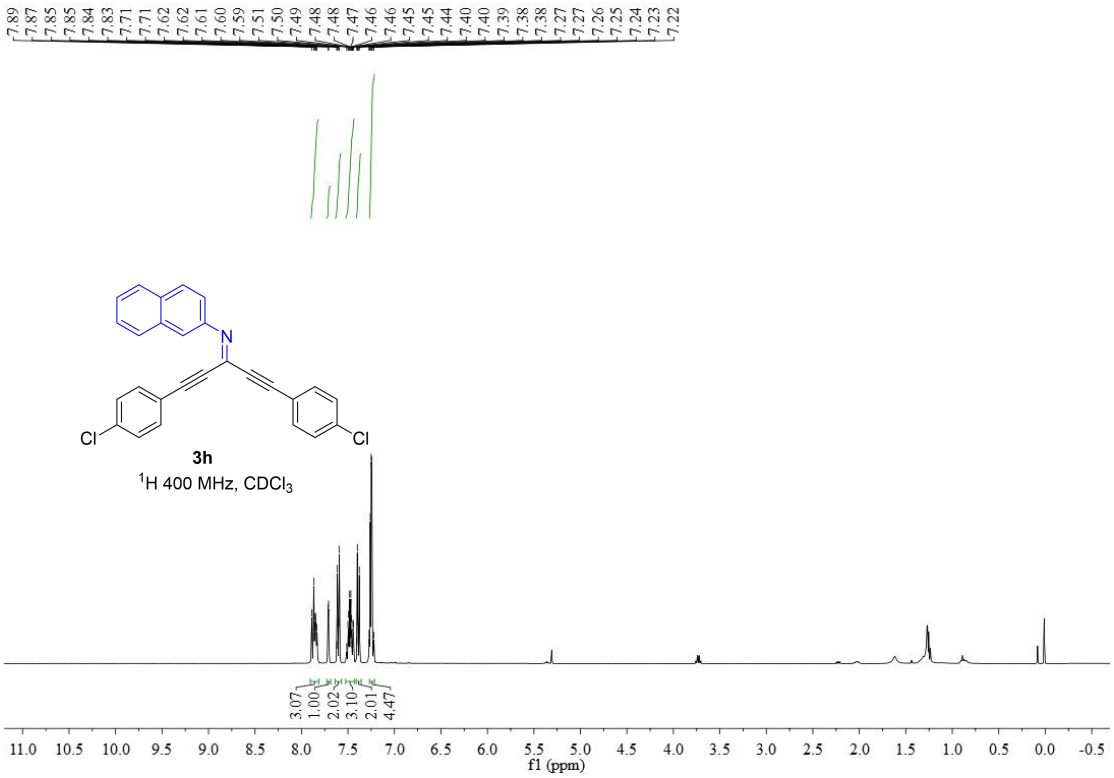
^{1}H 400 MHz, $CDCl_3$

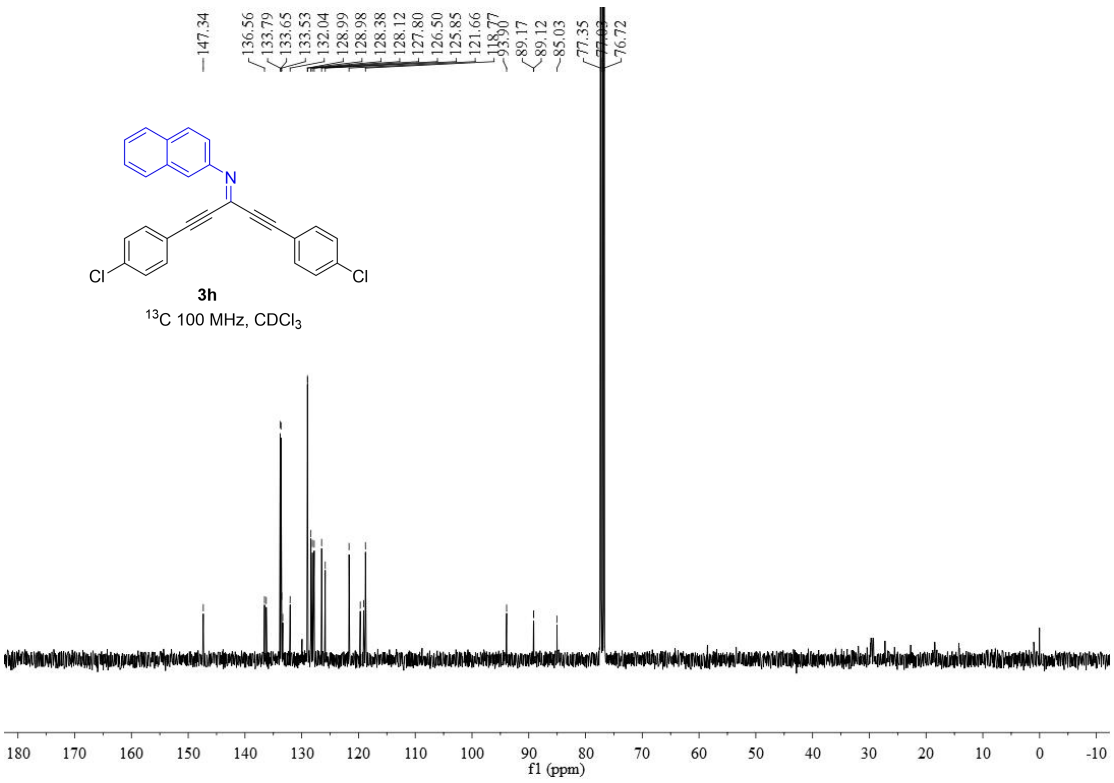


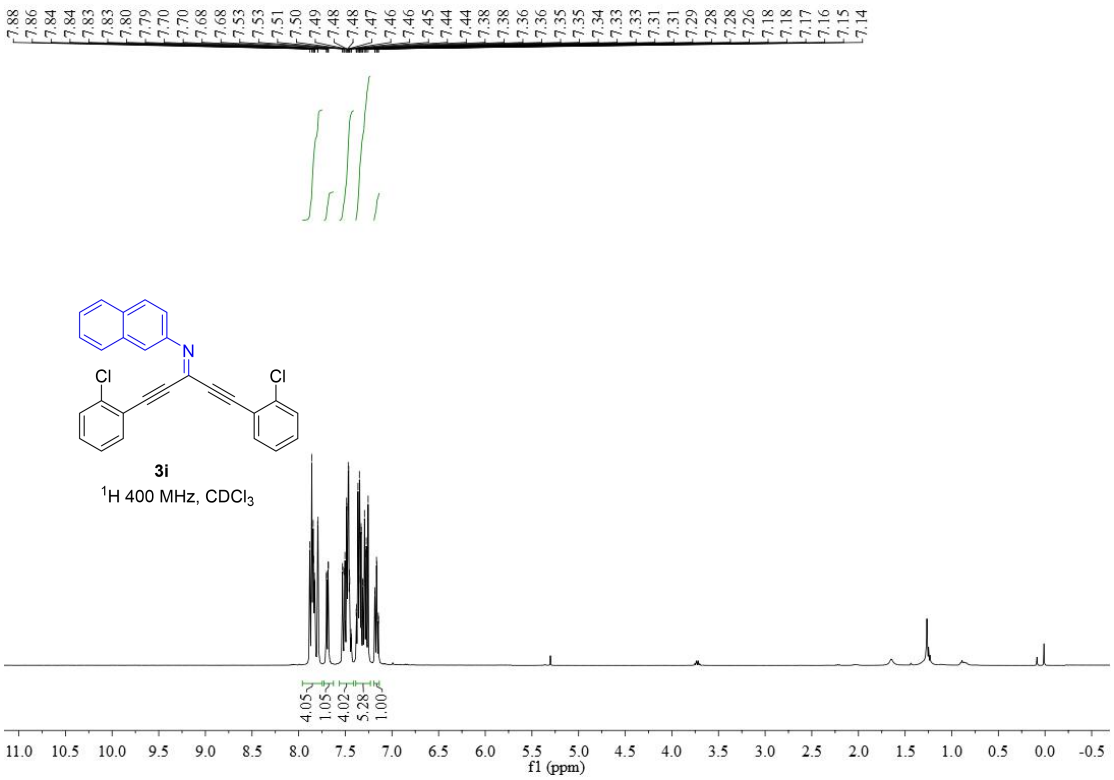


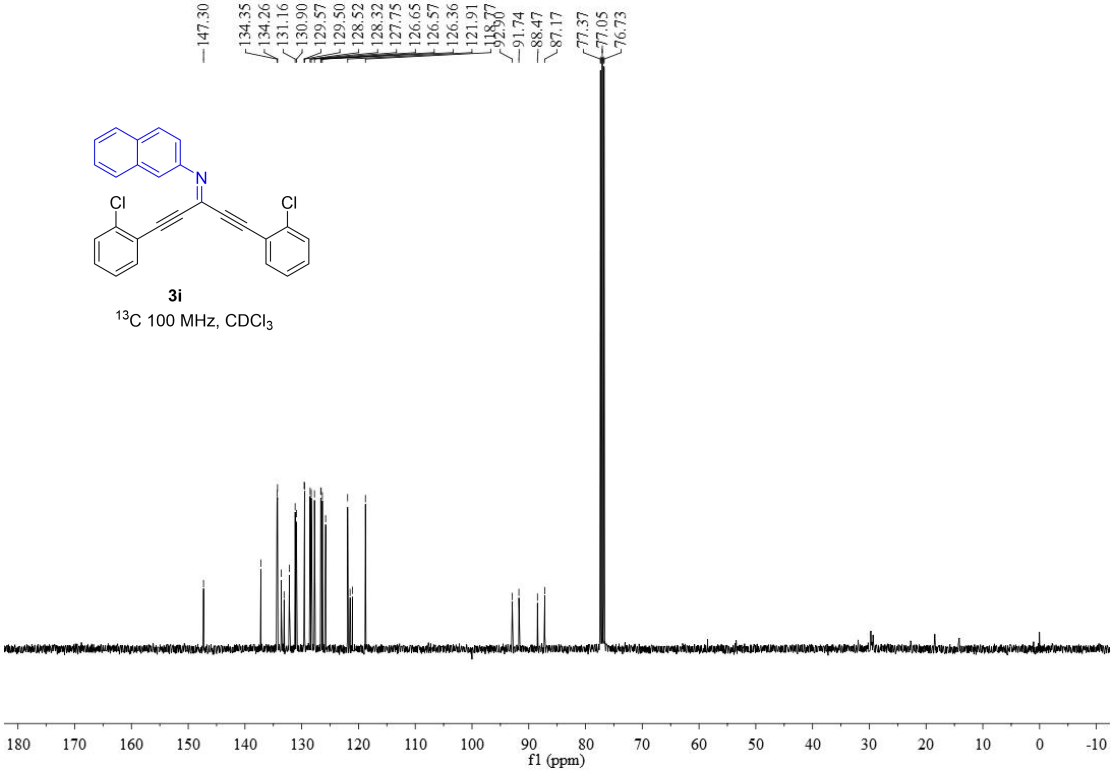
3g

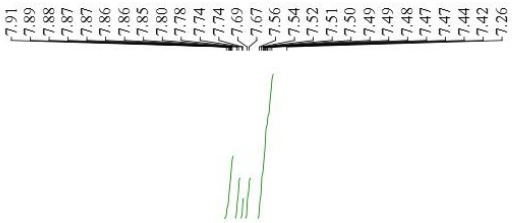
^{13}C 100 MHz, $CDCl_3$

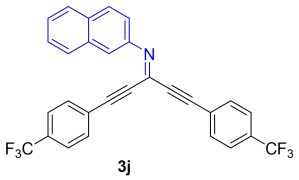

7.89 7.87 7.85 7.85 7.84 7.83 7.71 7.71 7.62 7.62 7.61 7.60 7.59 7.51 7.50 7.49 7.48 7.48 7.47 7.46 7.46 7.45 7.45 7.44 7.40 7.40 7.39 7.38 7.38 7.27 7.27 7.26 7.25 7.24 7.23 7.22
N
Cl
Cl
3h
1H 400 MHz, CDCl3
3.07 1.00 2.02 3.10 2.01 4.47
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


147.34 136.56 133.79 133.65 133.53 132.04 128.99 128.98 128.38 128.12 127.80 126.50 125.85 121.66 118.77 93.00 89.17 89.12 85.03 77.35 76.72
N
Cl
Cl
3h
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.88 7.86 7.84 7.84 7.83 7.83 7.80 7.79 7.70 7.70 7.68 7.68 7.53 7.53 7.51 7.50 7.49 7.48 7.48 7.47 7.46 7.46 7.45 7.44 7.44 7.38 7.38 7.36 7.36 7.35 7.35 7.34 7.33 7.33 7.31 7.31 7.29 7.28 7.28 7.26 7.18 7.18 7.17 7.16 7.15 7.14
N
Cl
Cl
3i
1H 400 MHz, CDCl3
4.05 1.05 4.02 5.28 1.00
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)

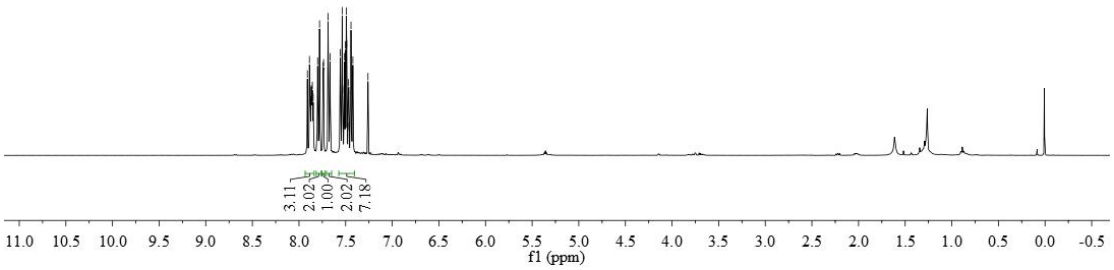

147.30 134.35 134.26 131.16 130.90 129.57 129.50 128.52 128.32 127.75 126.65 126.57 126.36 121.91 118.77 92.90 91.74 88.47 87.17 77.37 77.05 76.73
N
Cl
Cl
3i
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

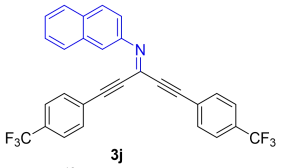




3j

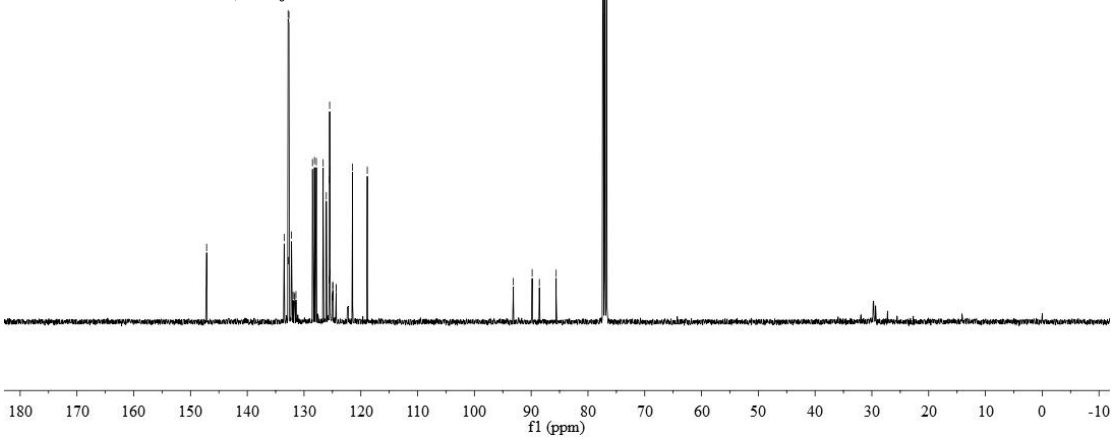
^{1}H 400 MHz, $CDCl_3$

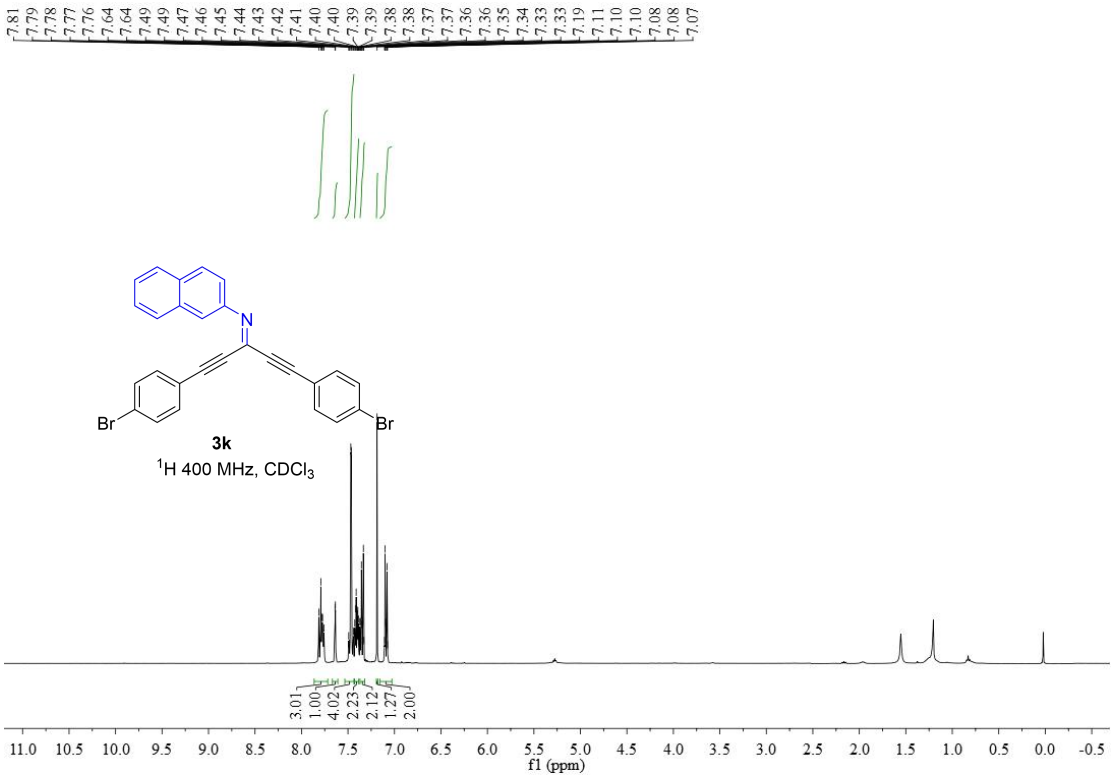


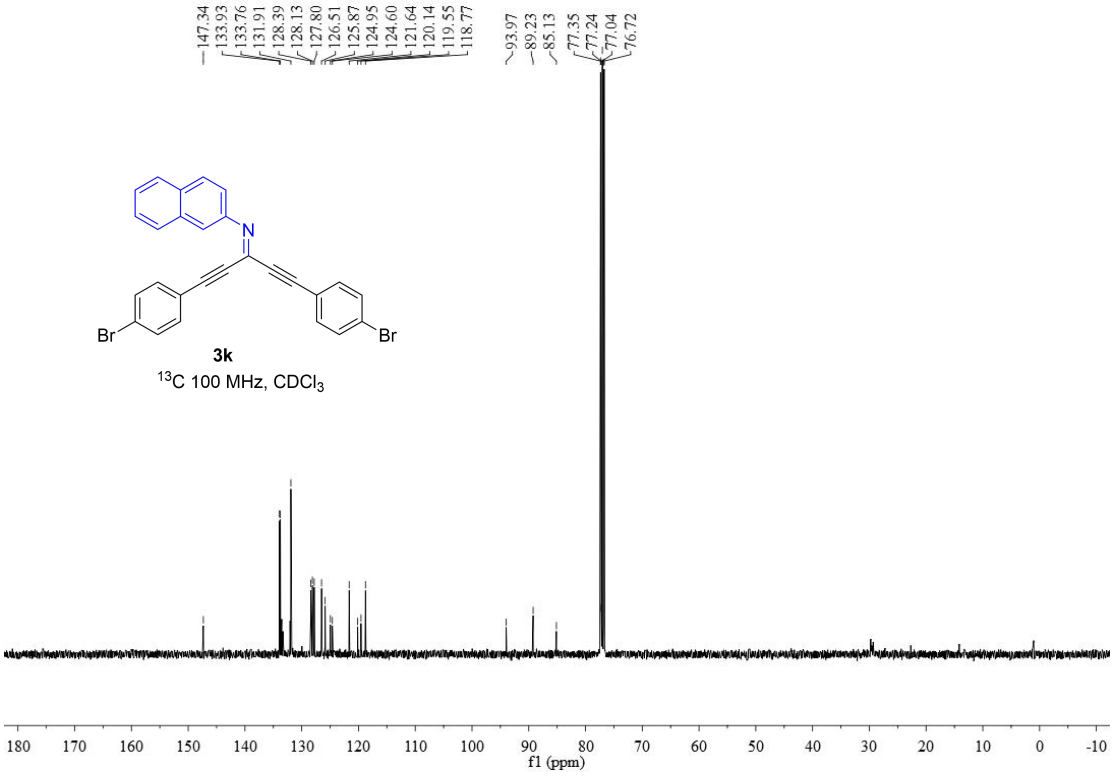


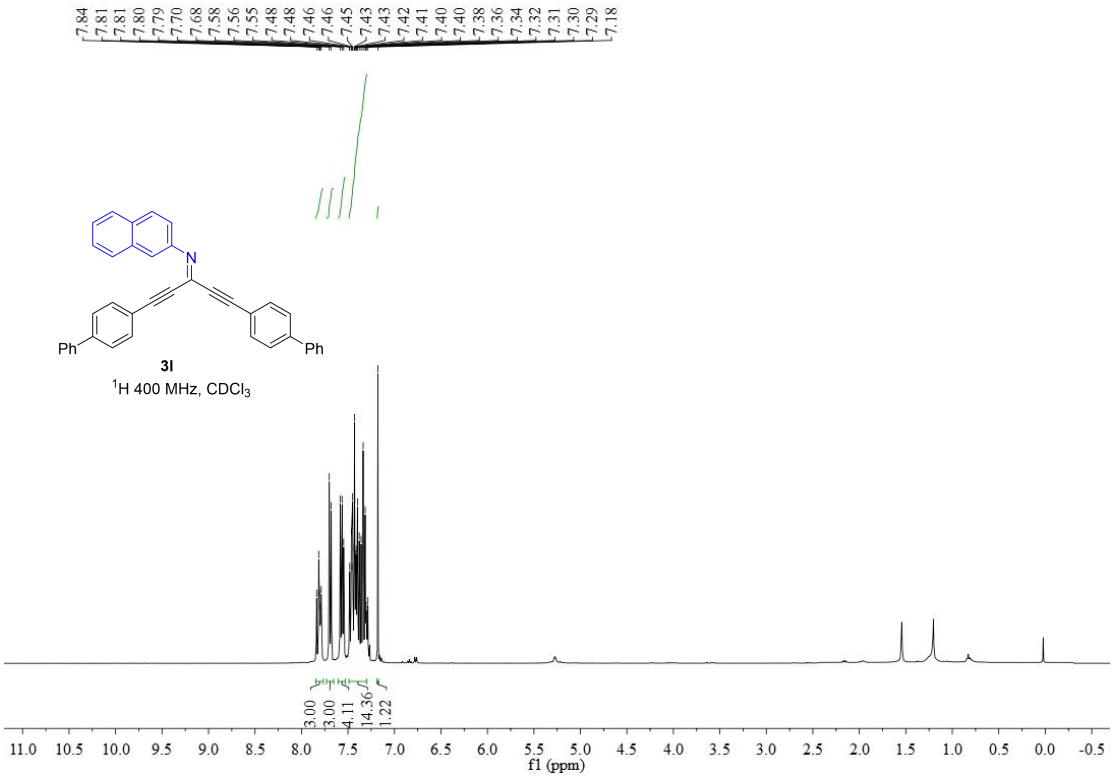
3j

^{13}C 100 MHz, $CDCl_3$



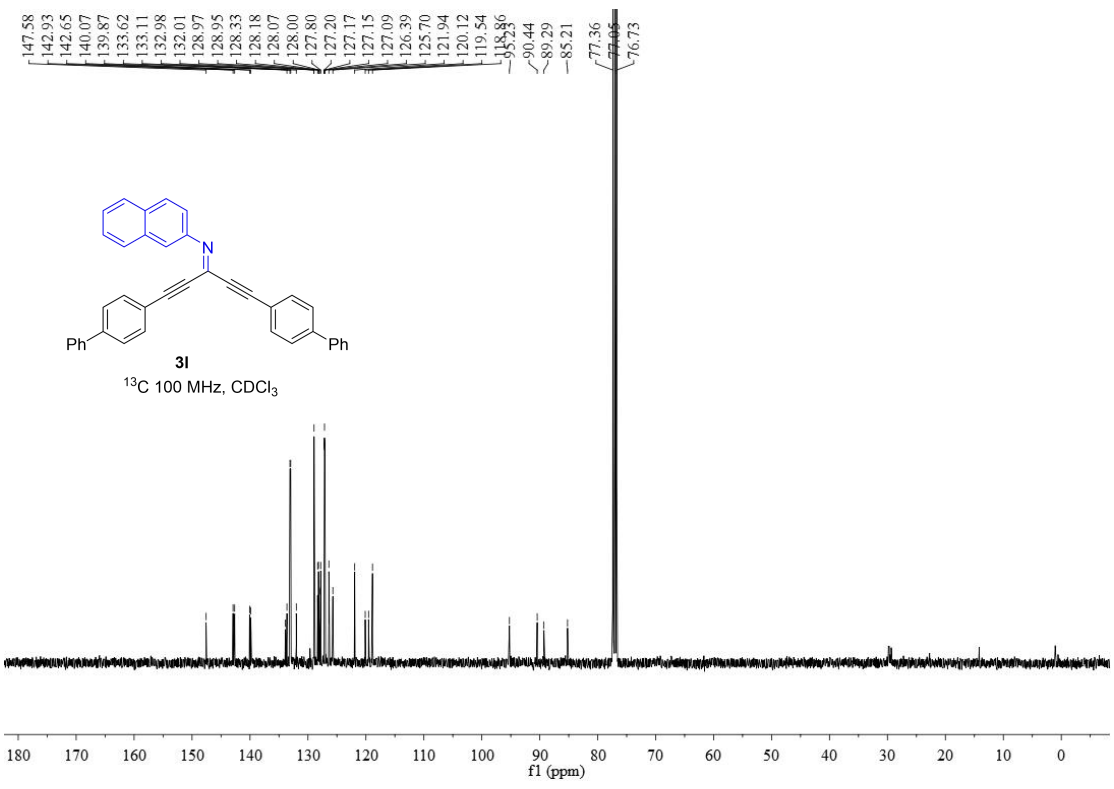

7.81 7.79 7.78 7.77 7.76 7.64 7.64 7.49 7.49 7.47 7.46 7.45 7.44 7.43 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.35 7.34 7.33 7.33 7.19 7.11 7.10 7.10 7.08 7.08 7.07
N
Br
Br
3k
1H 400 MHz, CDCl3
3.01 1.00 4.02 2.23 2.12 1.27 2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


147.34 133.93 133.76 131.91 128.39 128.13 127.80 126.51 125.87 124.95 124.60 121.64 120.14 119.55 118.77
93.97 89.23 85.13 77.35 77.24 77.04 76.72
N
Br
Br
3k
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



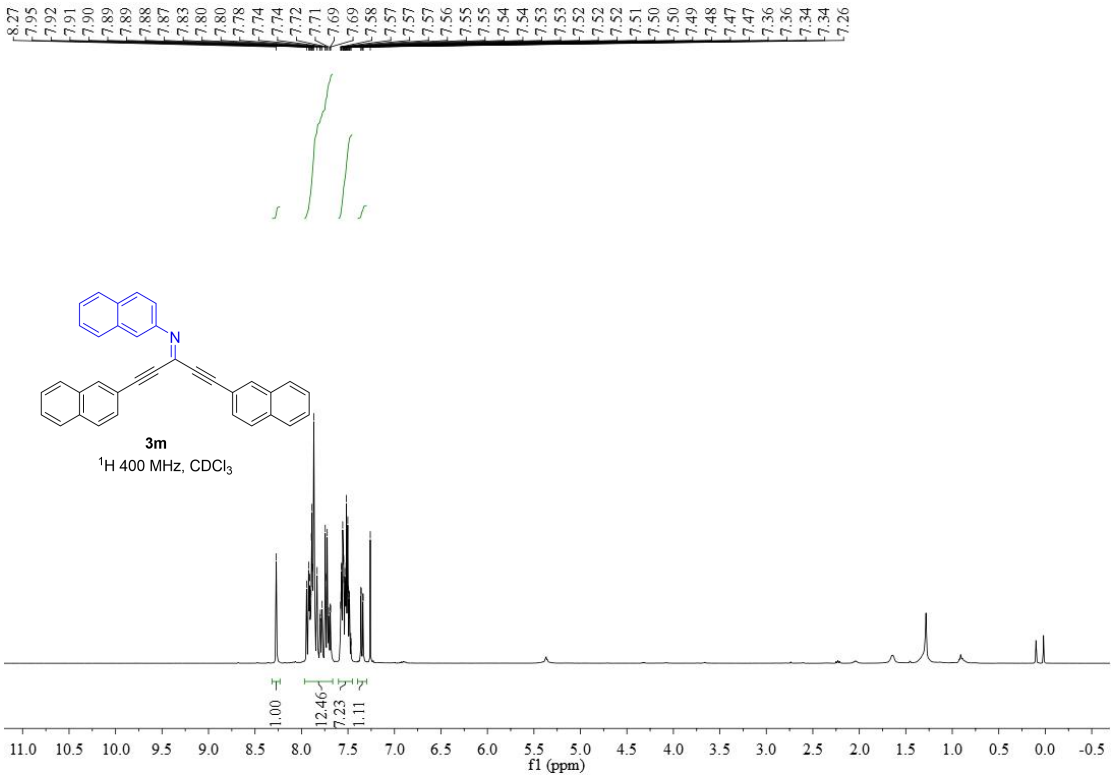
3l

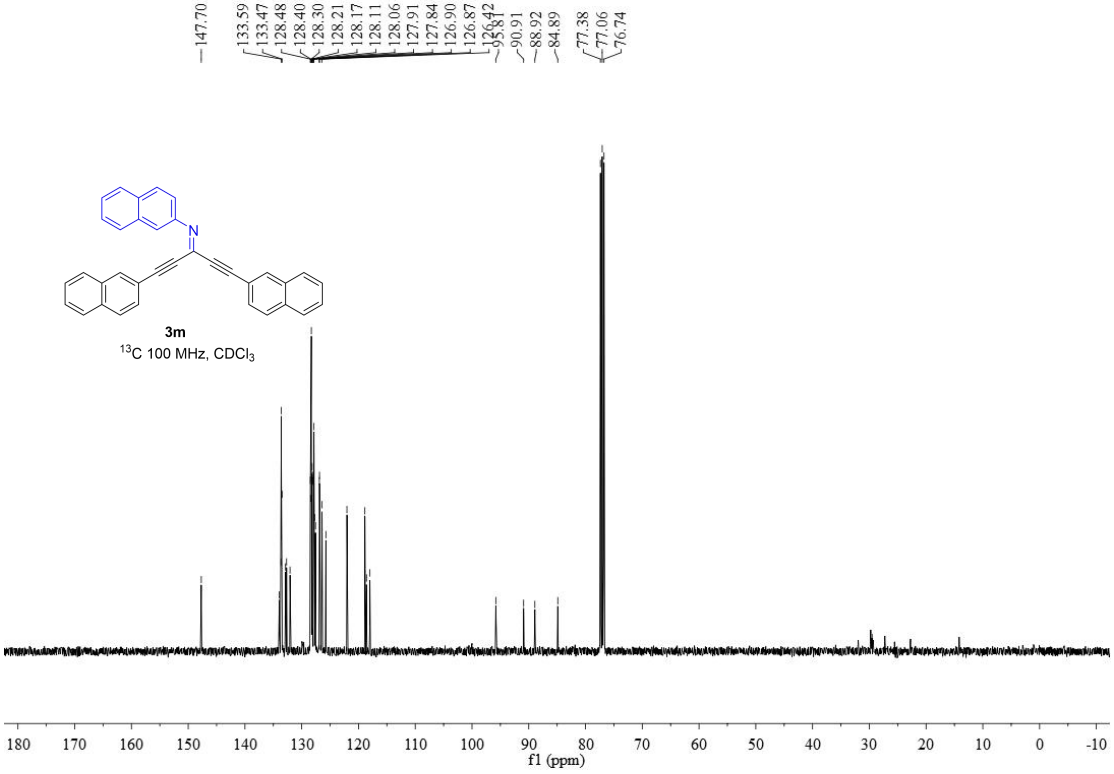
^{1}H 400 MHz, $CDCl_3$

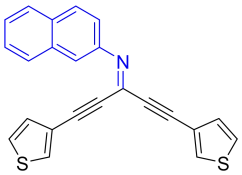


3l

^{13}C 100 MHz, $CDCl_3$

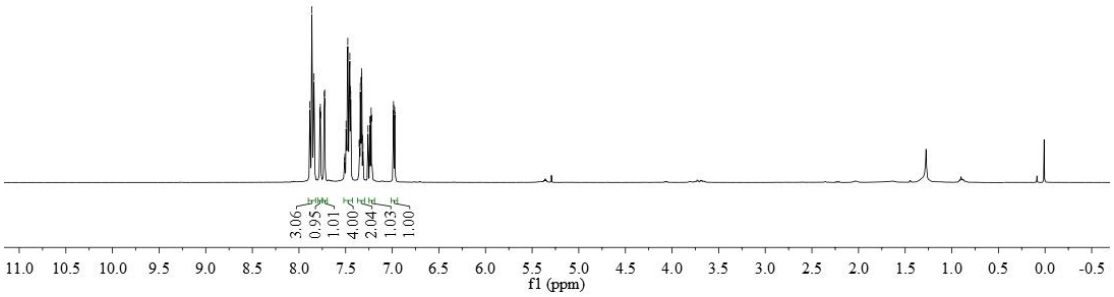

8.27
7.95
7.92
7.91
7.90
7.89
7.89
7.88
7.87
7.83
7.80
7.80
7.78
7.74
7.74
7.72
7.71
7.69
7.69
7.58
7.57
7.57
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.52
7.52
7.51
7.50
7.50
7.49
7.48
7.47
7.47
7.36
7.36
7.34
7.34
7.26
N
3m
1H 400 MHz, CDCl3
1.00
12.46
7.23
1.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 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 0.0 -0.5
f1 (ppm)

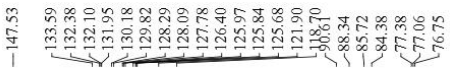

147.70
133.59
133.47
128.48
128.40
128.30
128.21
128.17
128.11
128.06
127.91
127.84
126.90
126.87
126.42
95.81
90.91
88.92
84.89
77.38
77.06
76.74
N
3m
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

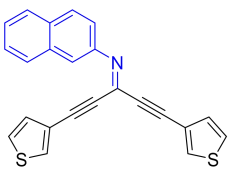


3n

^{1}H 400 MHz, $CDCl_3$

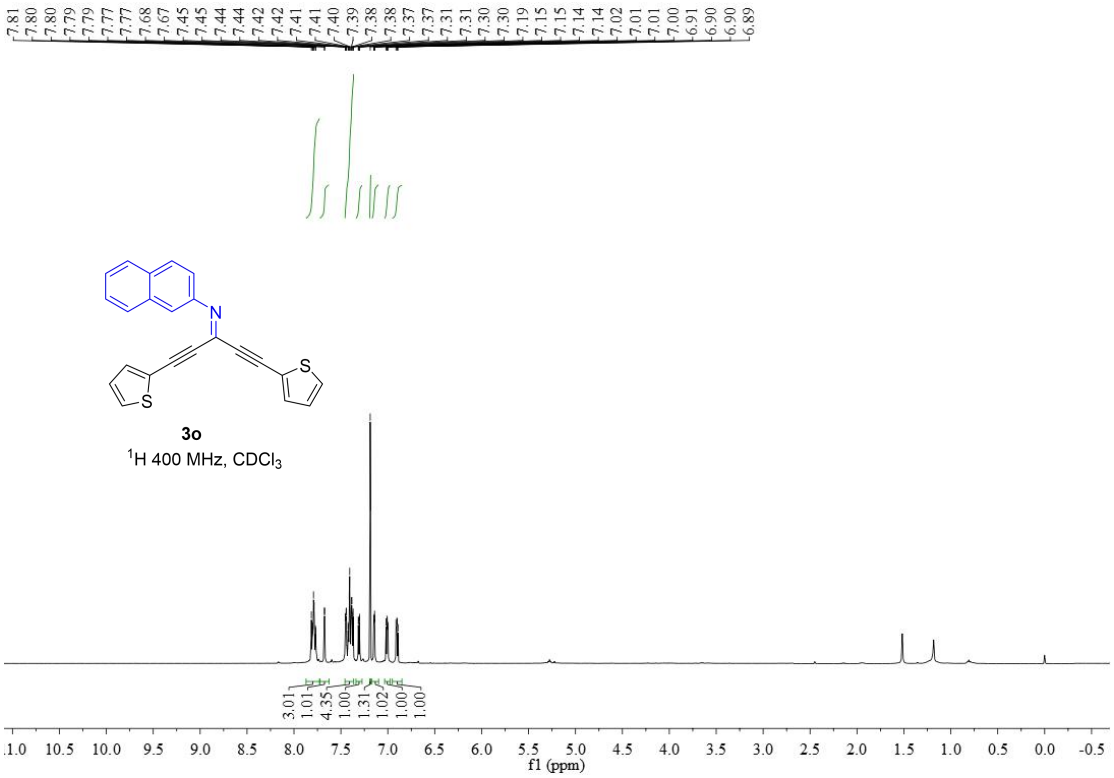


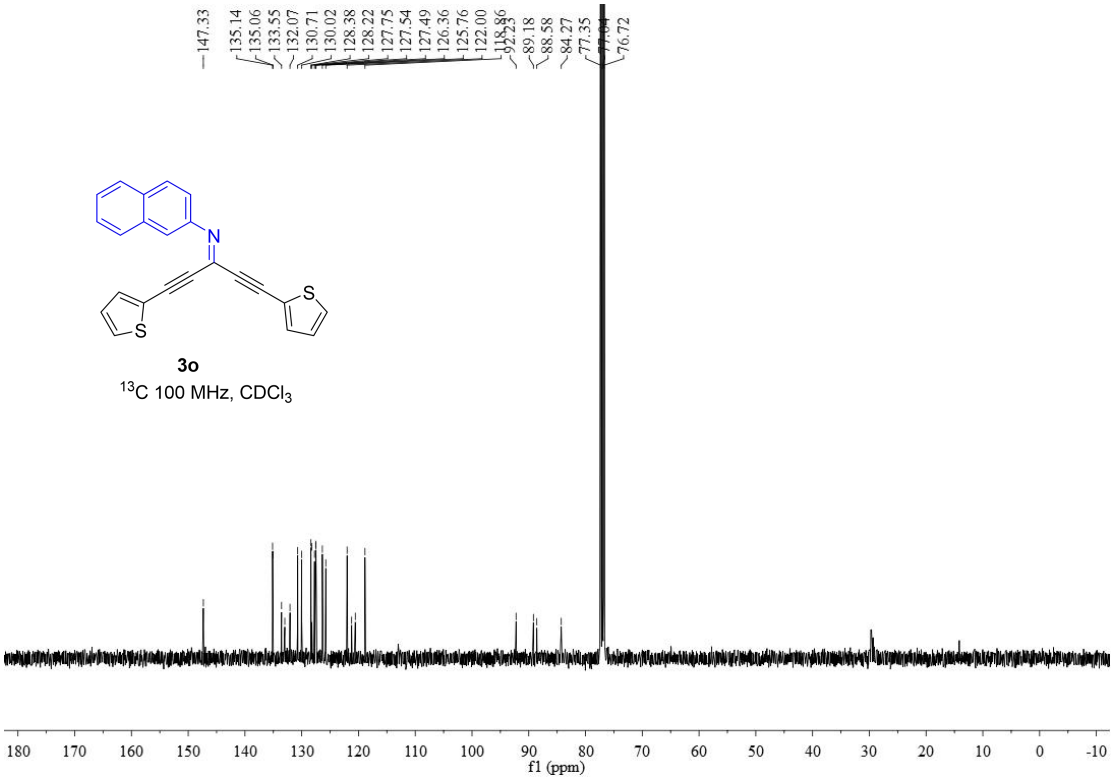


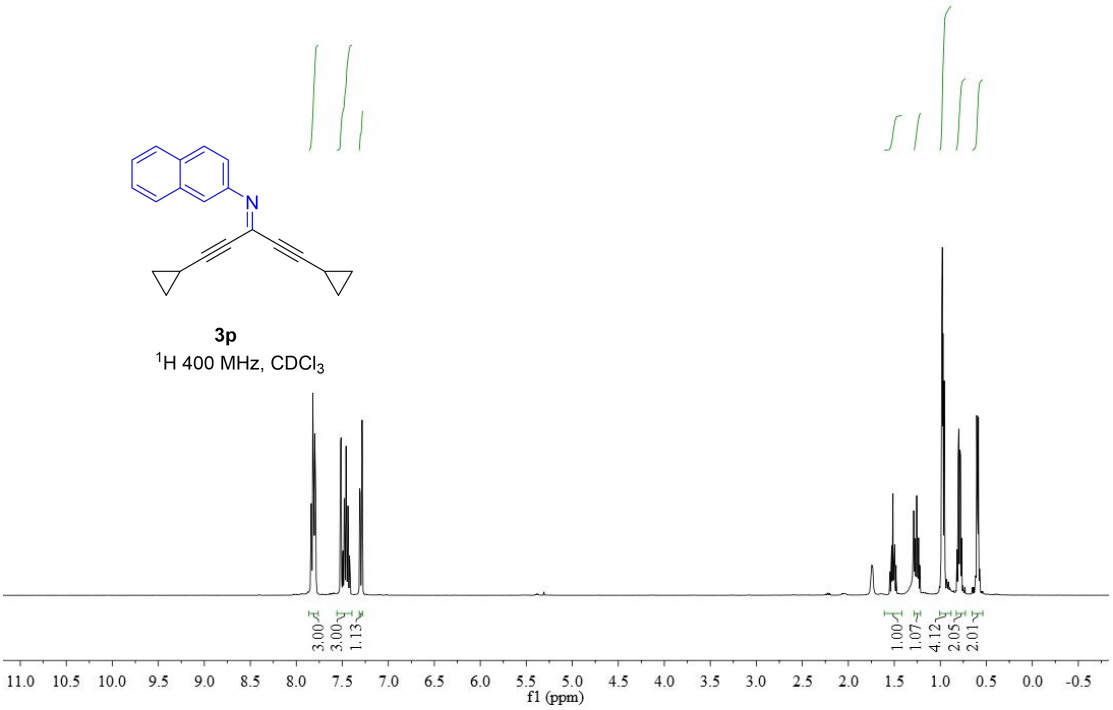


3n

^{13}C 100 MHz, $CDCl_3$

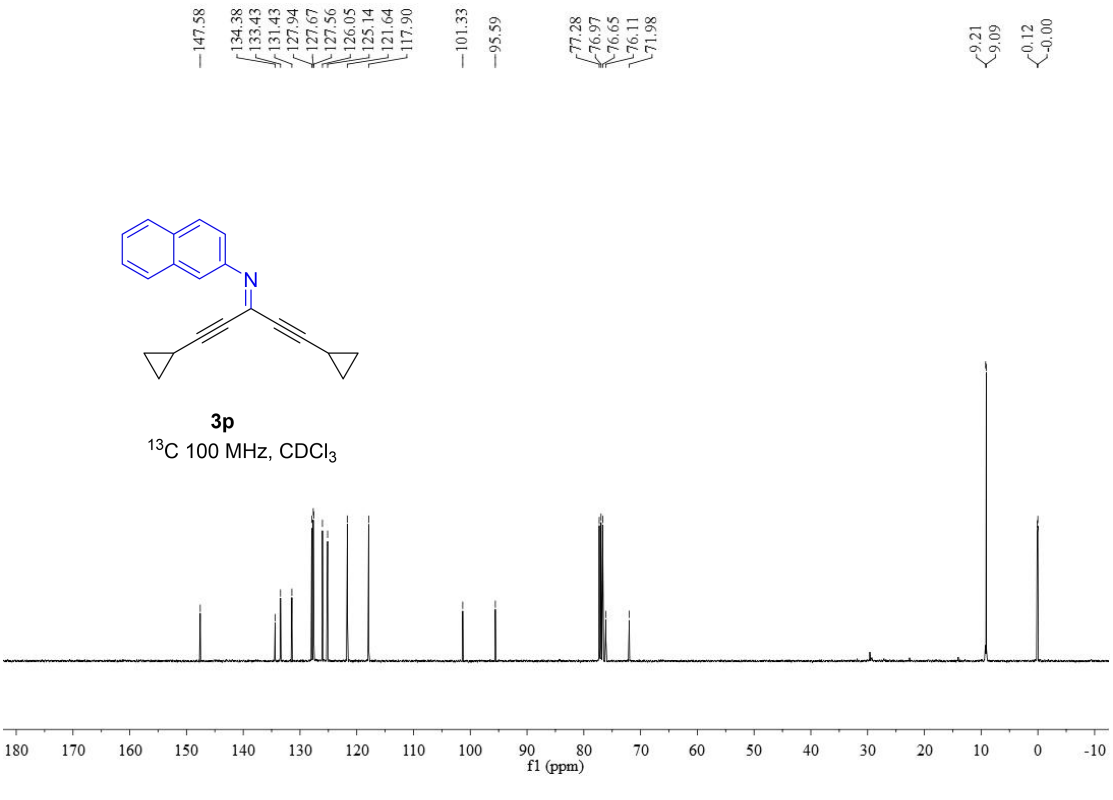

7.81 7.80 7.80 7.79 7.79 7.77 7.77 7.68 7.67 7.45 7.45 7.44 7.44 7.42 7.42 7.41 7.41 7.40 7.39 7.38 7.38 7.37 7.37 7.31 7.31 7.30 7.30 7.19 7.15 7.15 7.14 7.14 7.02 7.01 7.01 7.00 6.91 6.90 6.90 6.89
N
S
S
3o
1H 400 MHz, CDCl3
3.01 1.01 4.35 1.00 1.31 1.02 1.00 1.00
f1 (ppm)


147.33 135.14 135.06 133.55 132.07 130.71 130.02 128.38 128.22 127.75 127.54 127.49 126.36 125.76 122.00 118.86 92.23 89.18 88.58 84.27 77.35 76.72
N
S
S
3o
13C 100 MHz, CDCl3
f1 (ppm)



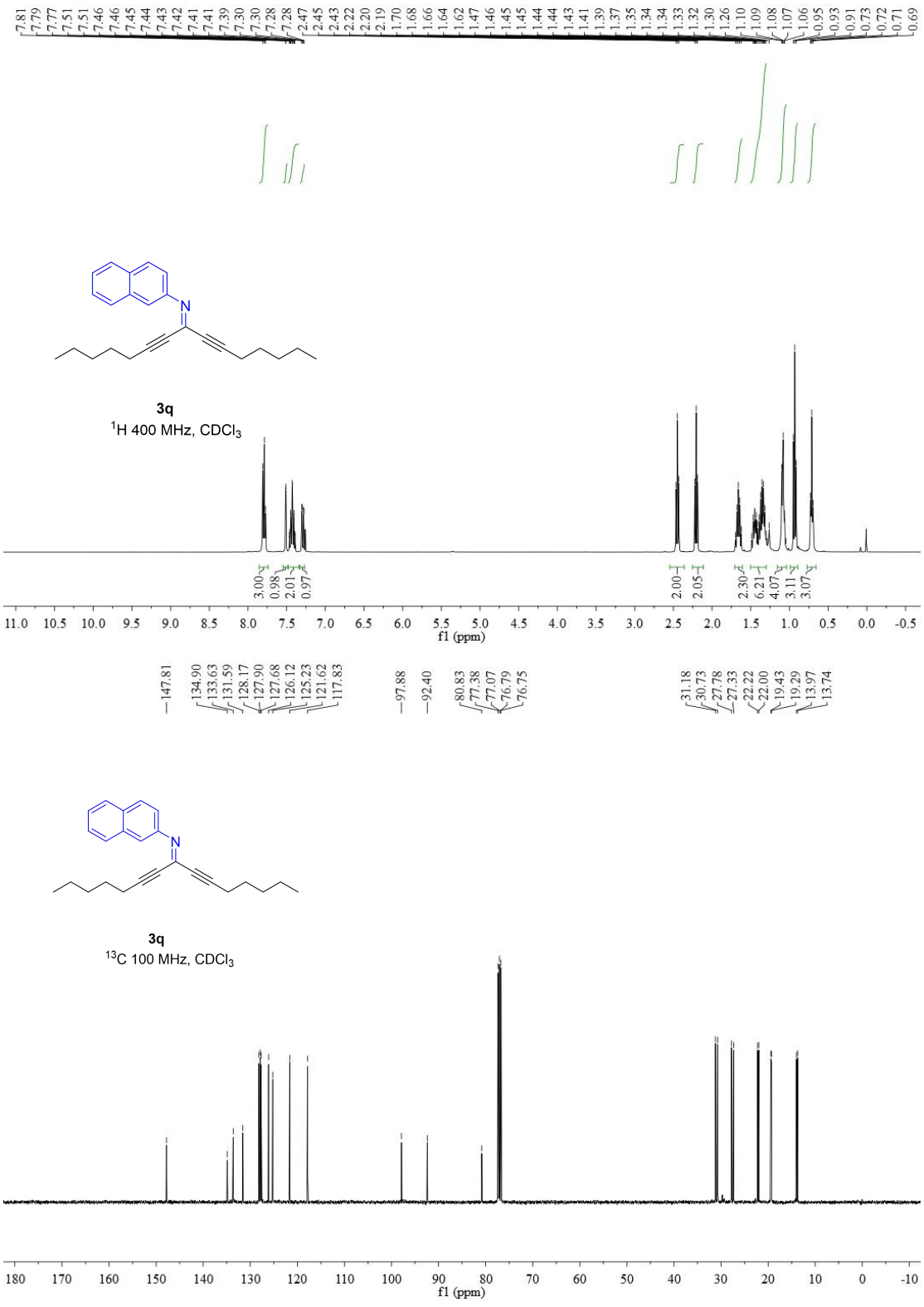
3p

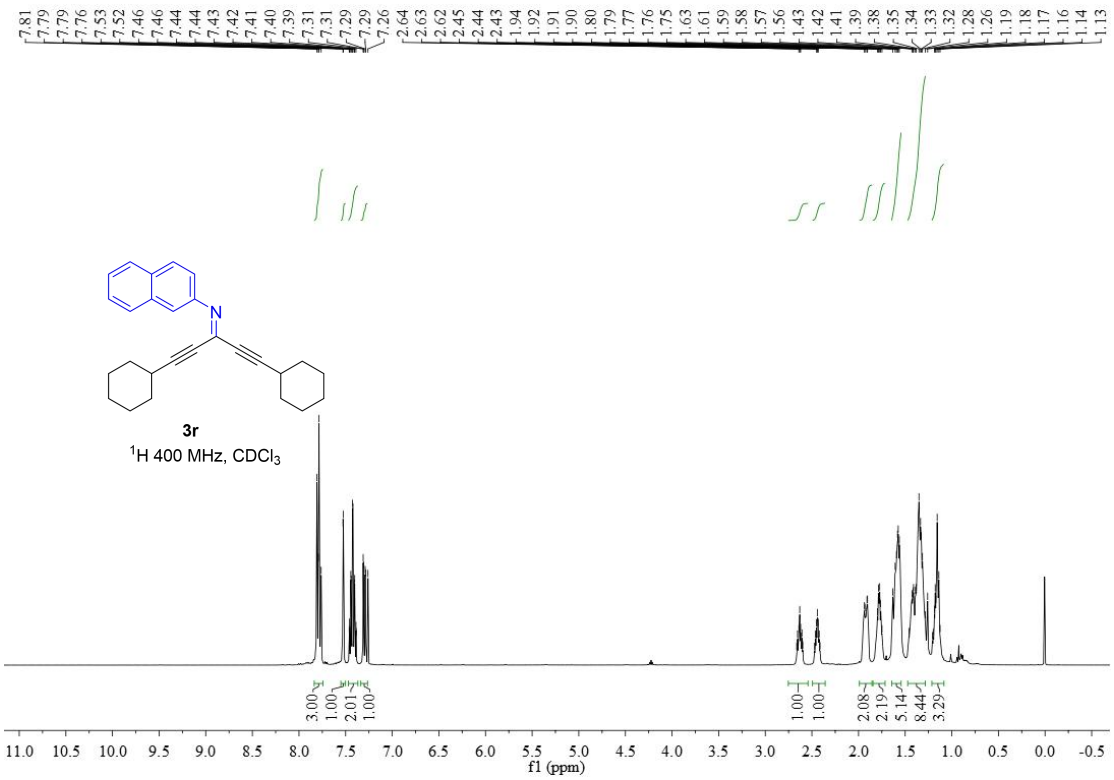
^{1}H 400 MHz, $CDCl_3$

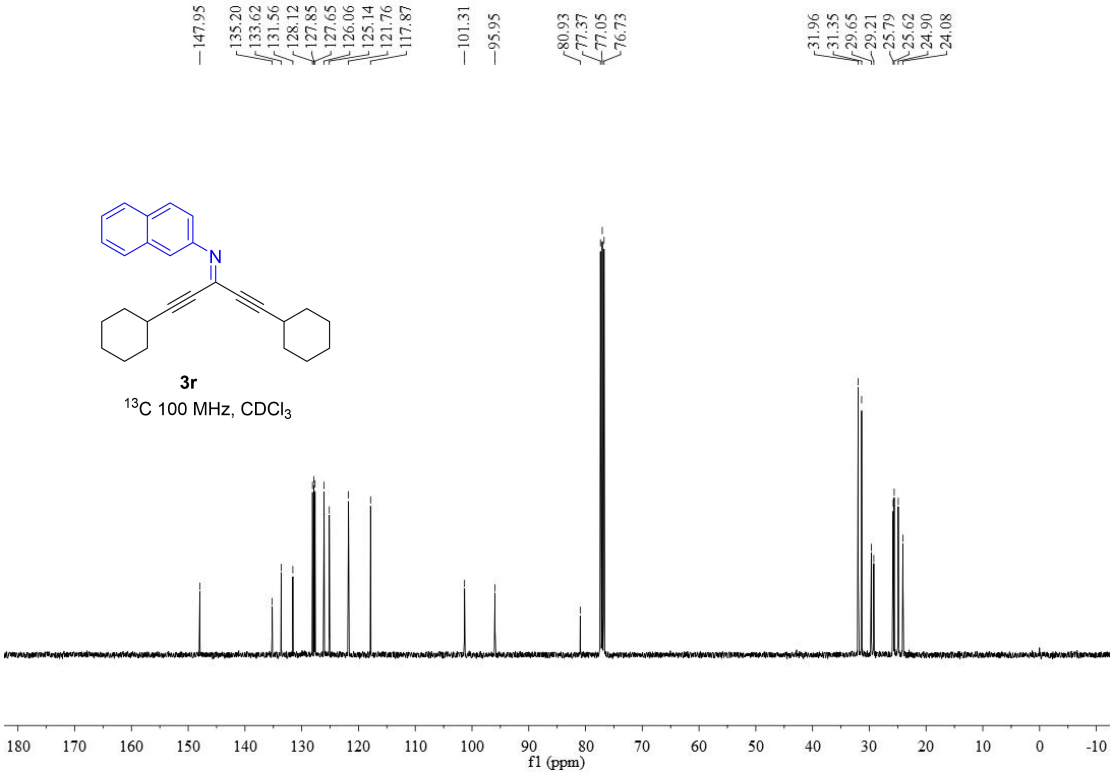


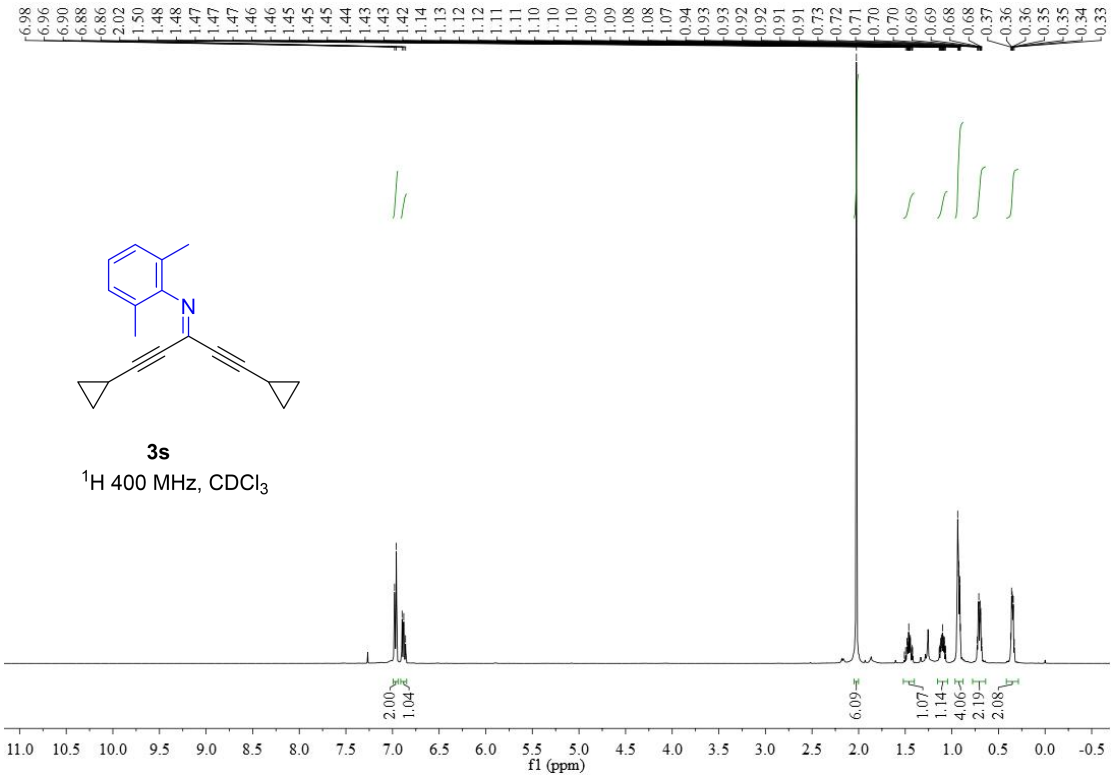
3p

^{13}C 100 MHz, $CDCl_3$


7.81 7.79 7.77 7.51 7.51 7.46 7.46 7.45 7.44 7.43 7.42 7.41 7.41 7.39 7.30 7.30 7.28 7.28 2.47 2.45 2.43 2.22 2.20 2.19 1.70 1.68 1.66 1.64 1.62 1.47 1.46 1.45 1.45 1.44 1.44 1.43 1.41 1.39 1.37 1.35 1.34 1.34 1.33 1.32 1.30 1.26 1.10 1.09 1.08 1.07 1.06 0.95 0.93 0.91 0.73 0.72 0.71 0.69
N
3q
1H 400 MHz, CDCl3
3.00 0.98 2.01 0.97 2.00 2.05 2.30 6.21 4.07 3.11 3.07
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)
147.81 134.90 133.63 131.59 128.17 127.90 127.68 126.12 125.23 121.62 117.83 97.88 92.40 80.83 77.38 77.07 76.79 76.75 31.18 30.73 27.78 27.33 22.22 22.00 19.43 19.29 13.97 13.74
N
3q
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

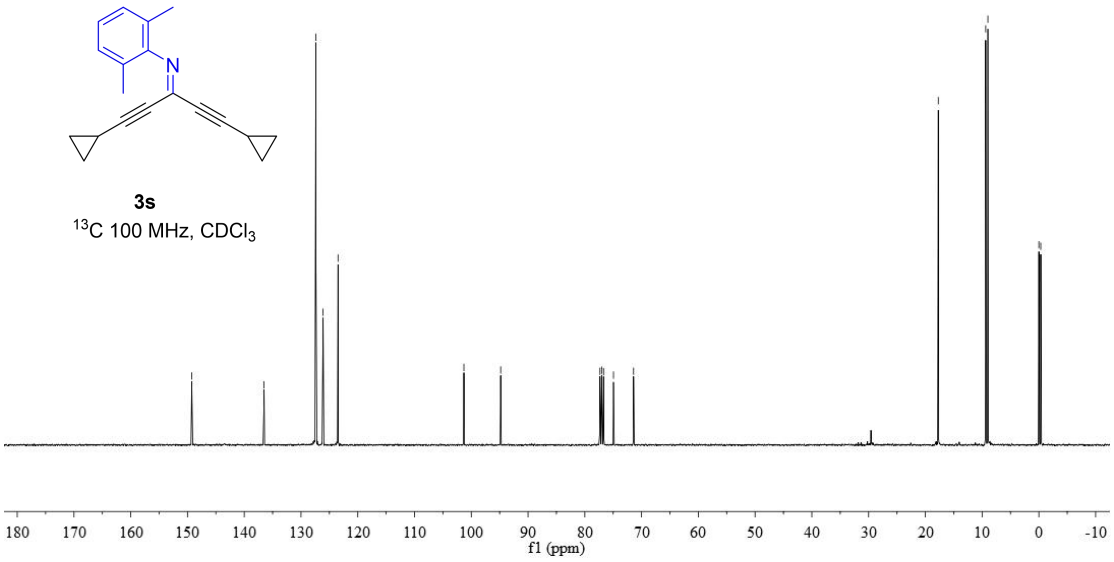

7.81 7.79 7.79 7.76 7.53 7.52 7.46 7.46 7.44 7.44 7.43 7.42 7.41 7.40 7.39 7.31 7.31 7.29 7.29 7.26
2.64 2.63 2.62 2.45 2.44 2.43 1.94 1.92 1.91 1.90 1.80 1.79 1.77 1.76 1.75 1.63 1.61 1.59 1.58 1.57 1.56 1.43 1.42 1.41 1.39 1.38 1.35 1.34 1.33 1.32 1.28 1.26 1.19 1.18 1.17 1.16 1.14 1.13
N
3r
1H 400 MHz, CDCl3
3.00 1.00 2.01 1.00 1.00 1.00 2.08 2.19 5.14 8.44 3.29
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)


147.95 135.20 133.62 131.56 128.12 127.85 127.65 126.06 125.14 121.76 117.87 101.31 95.95 80.93 77.37 77.05 76.73 31.96 31.35 29.65 29.21 25.79 25.62 24.90 24.08
N
3r
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



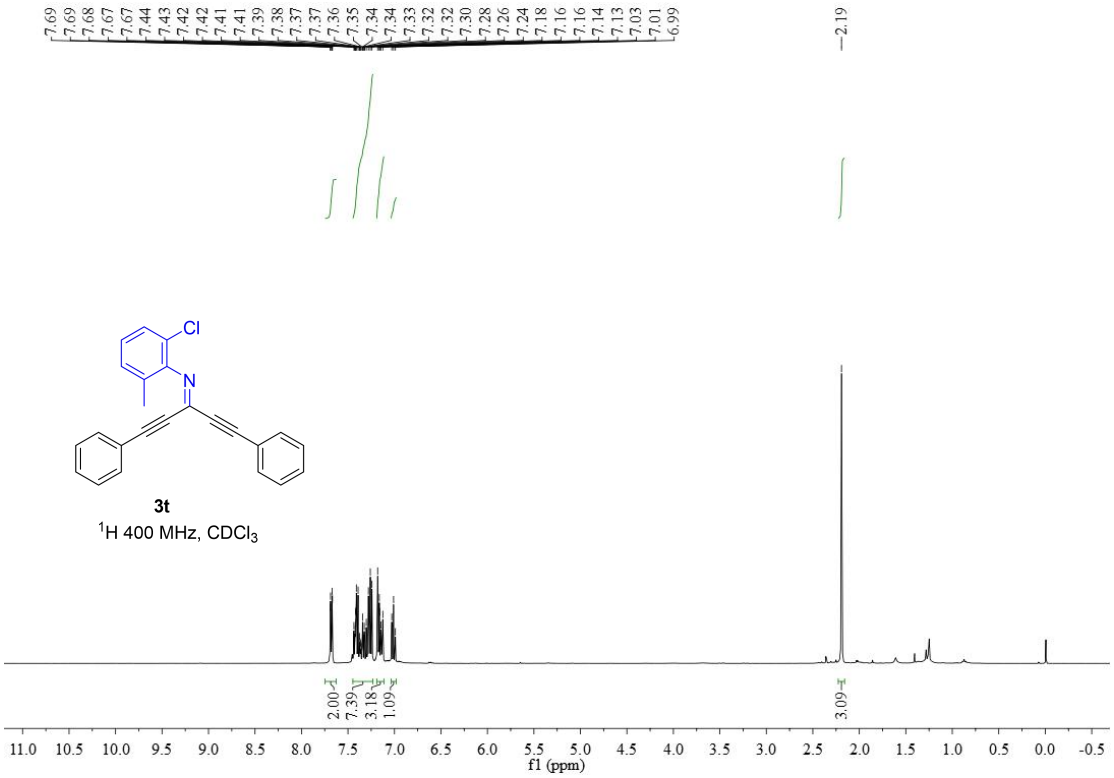
3s

^{1}H 400 MHz, $CDCl_3$



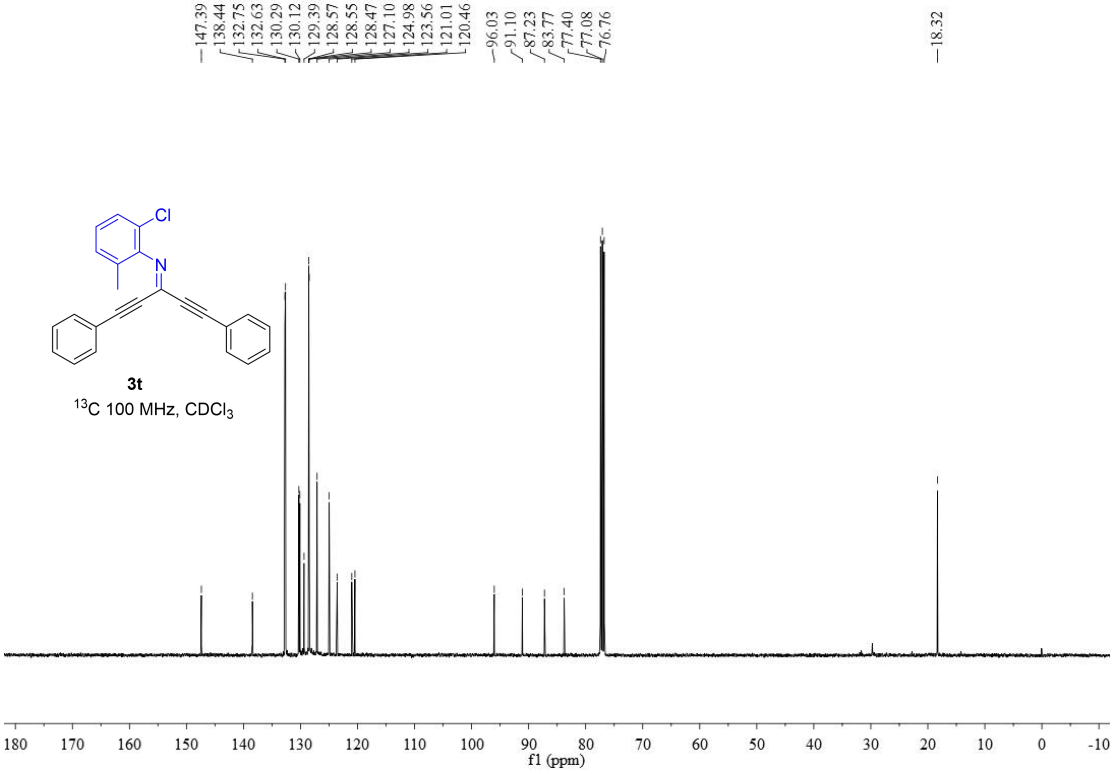
3s

^{13}C 100 MHz, $CDCl_3$



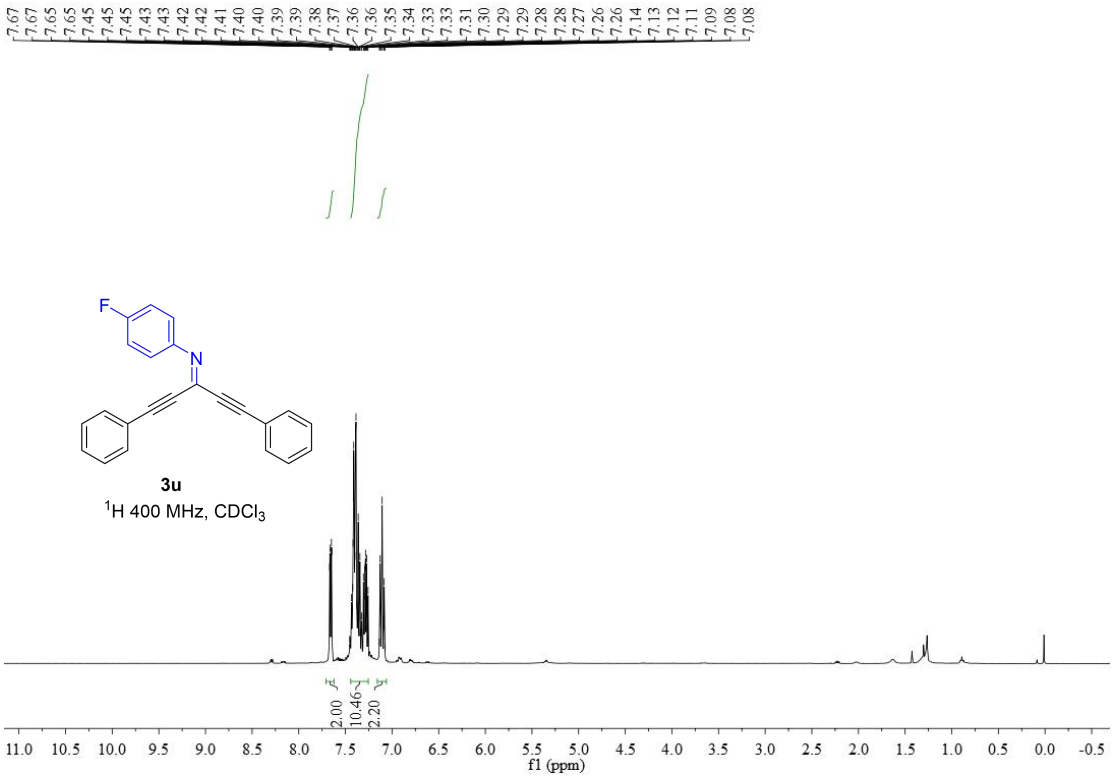
3t

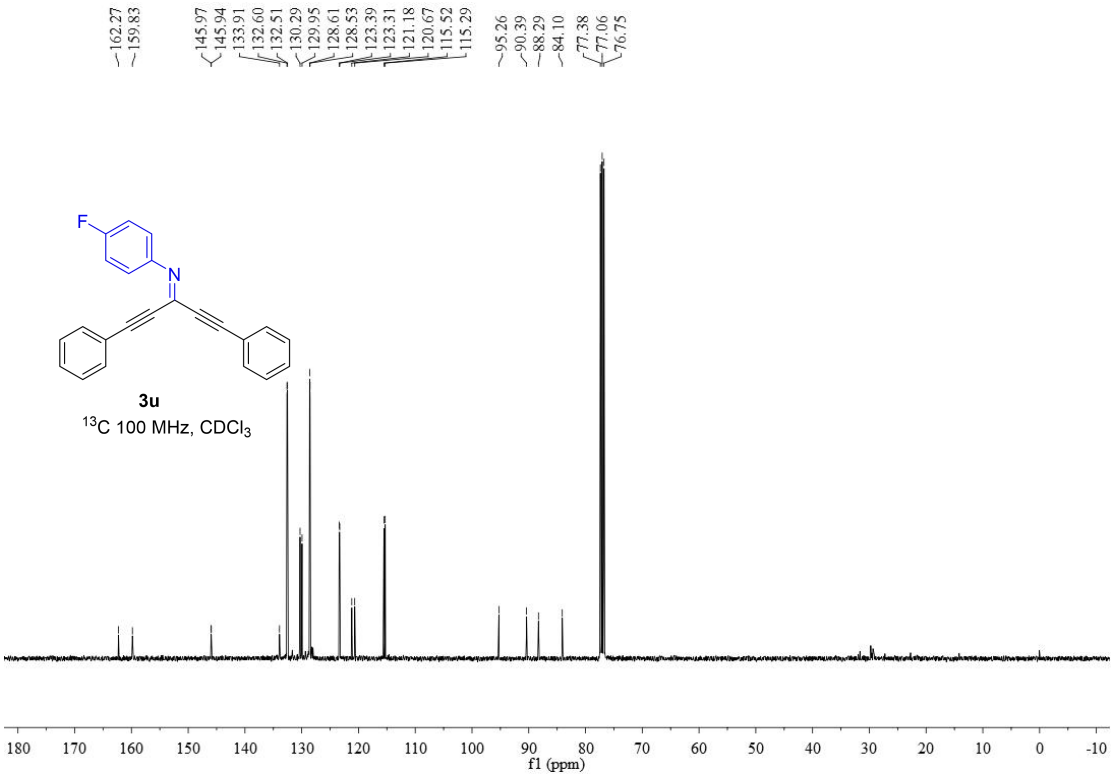
^{1}H 400 MHz, $CDCl_3$

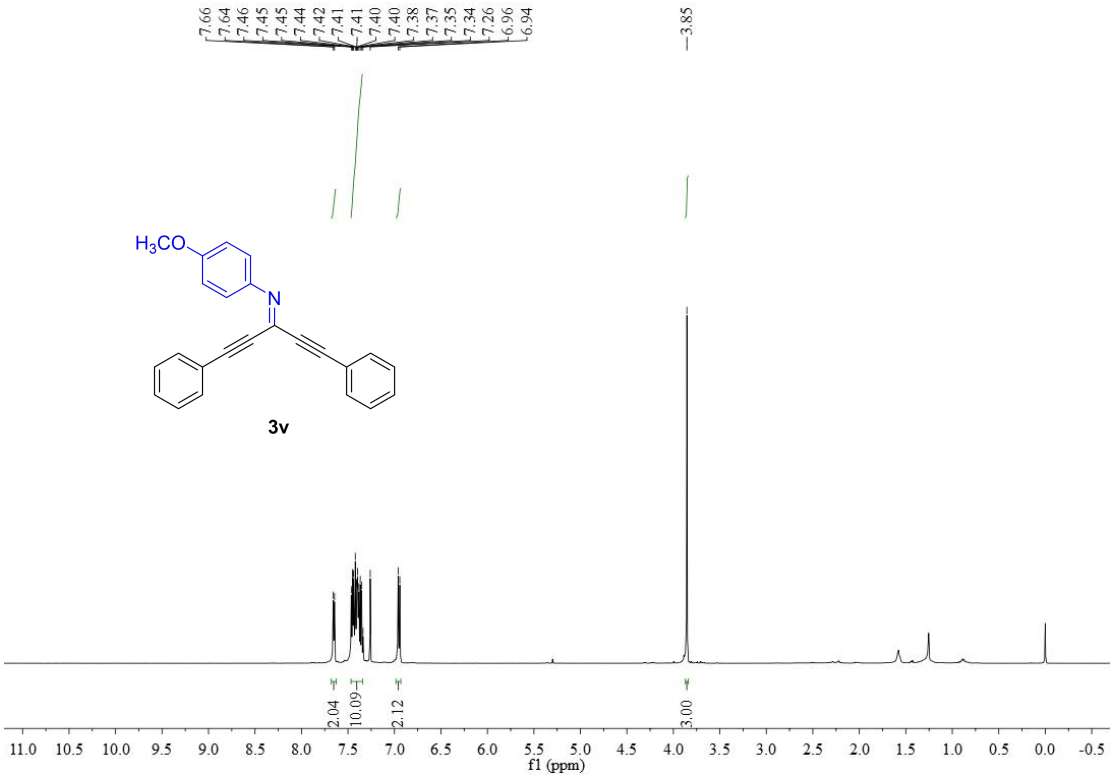


3t

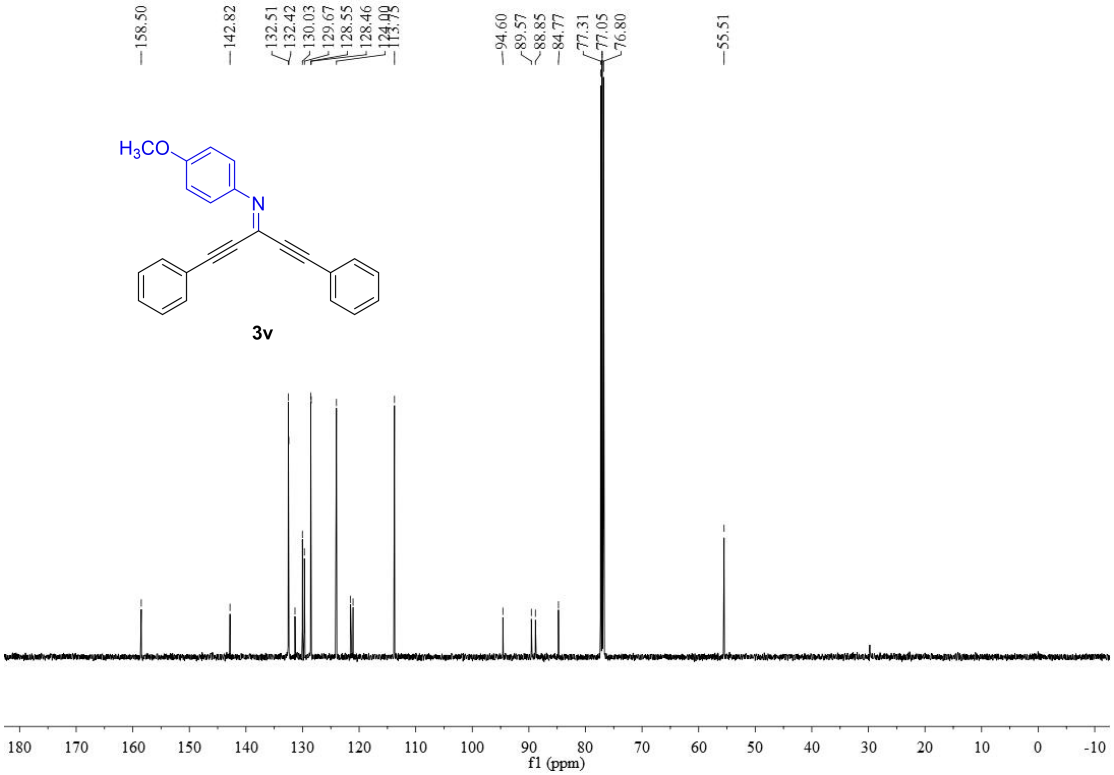
^{13}C 100 MHz, $CDCl_3$


7.67 7.67 7.65 7.65 7.45 7.45 7.45 7.43 7.43 7.42 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.36 7.36 7.35 7.34 7.33 7.33 7.31 7.30 7.29 7.29 7.28 7.28 7.27 7.26 7.26 7.14 7.13 7.12 7.11 7.09 7.08 7.08
F
N
3u
1H 400 MHz, CDCl3
2.00
10.46
2.20
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)

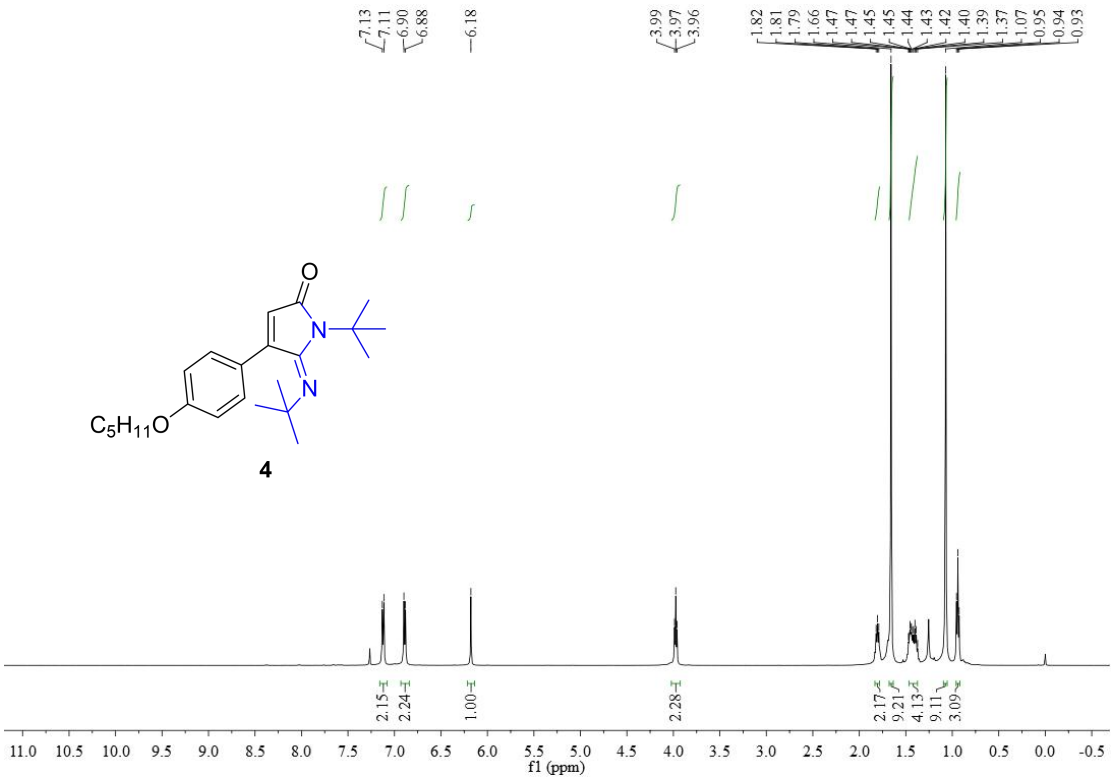

162.27 159.83 145.97 145.94 133.91 132.60 132.51 130.29 129.95 128.61 128.53 123.39 123.31 121.18 120.67 115.52 115.29 95.26 90.39 88.29 84.10 77.38 77.06 76.75
F
N
3u
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

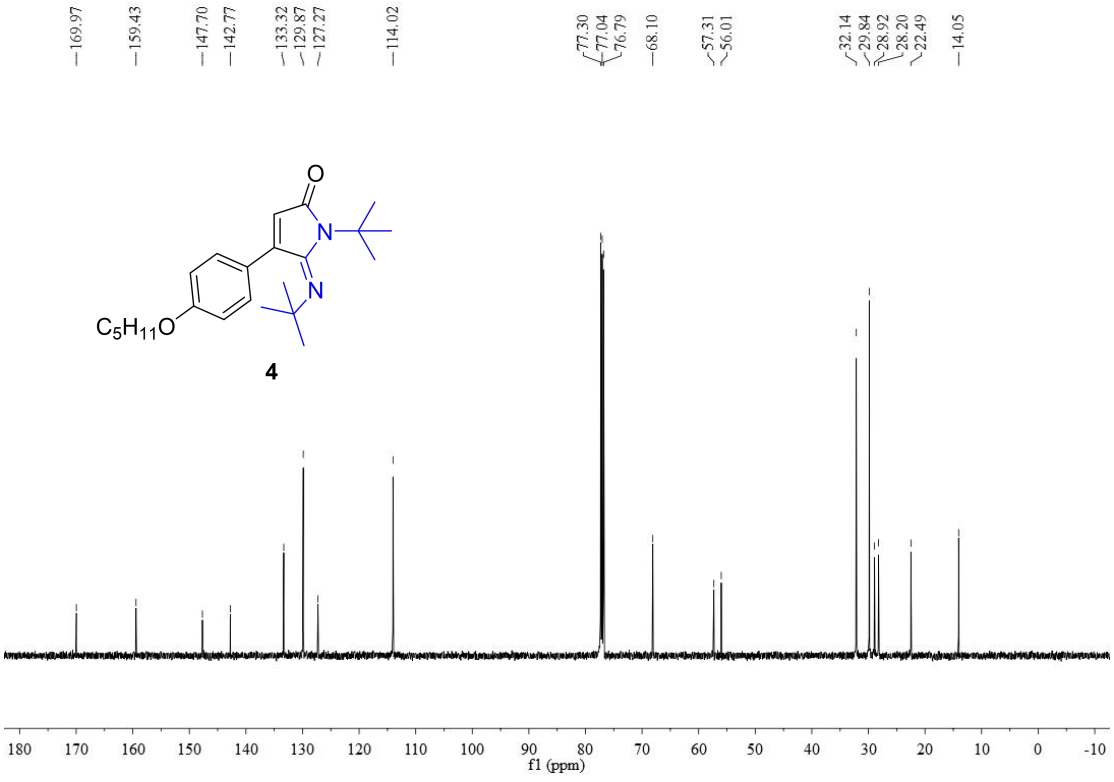

7.66
7.64
7.46
7.45
7.45
7.44
7.42
7.41
7.41
7.40
7.40
7.38
7.37
7.35
7.34
7.26
6.96
6.94
3.85
H3CO
N
2.04
10.09
2.12
3.00
f1 (ppm)

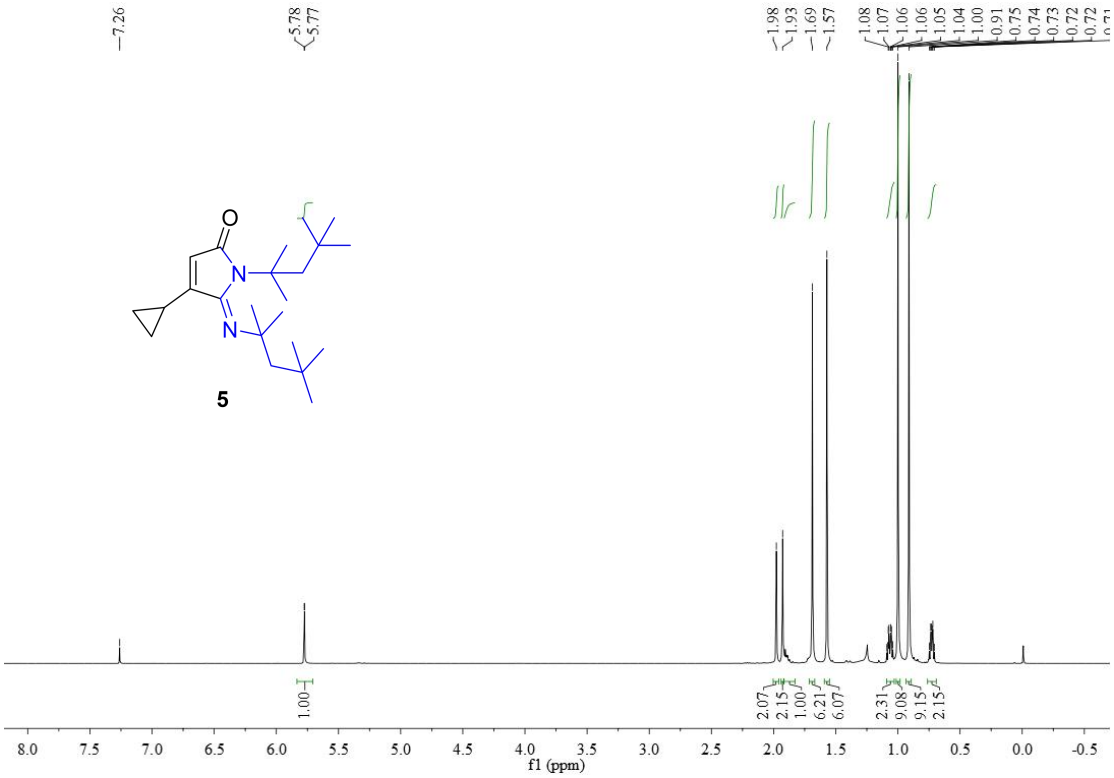
3v

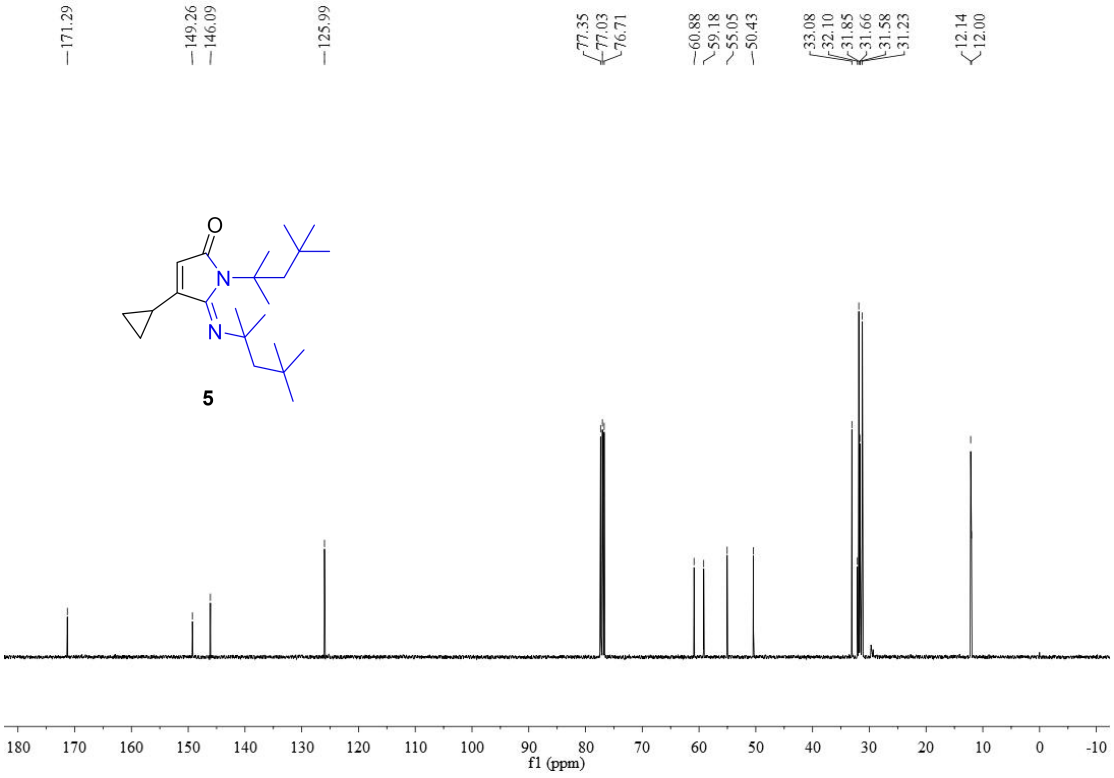

158.50
142.82
132.51
132.42
130.03
129.67
128.55
128.46
124.00
113.75
94.60
89.57
88.85
84.77
77.31
77.05
76.80
55.51
H3CO
N
f1 (ppm)

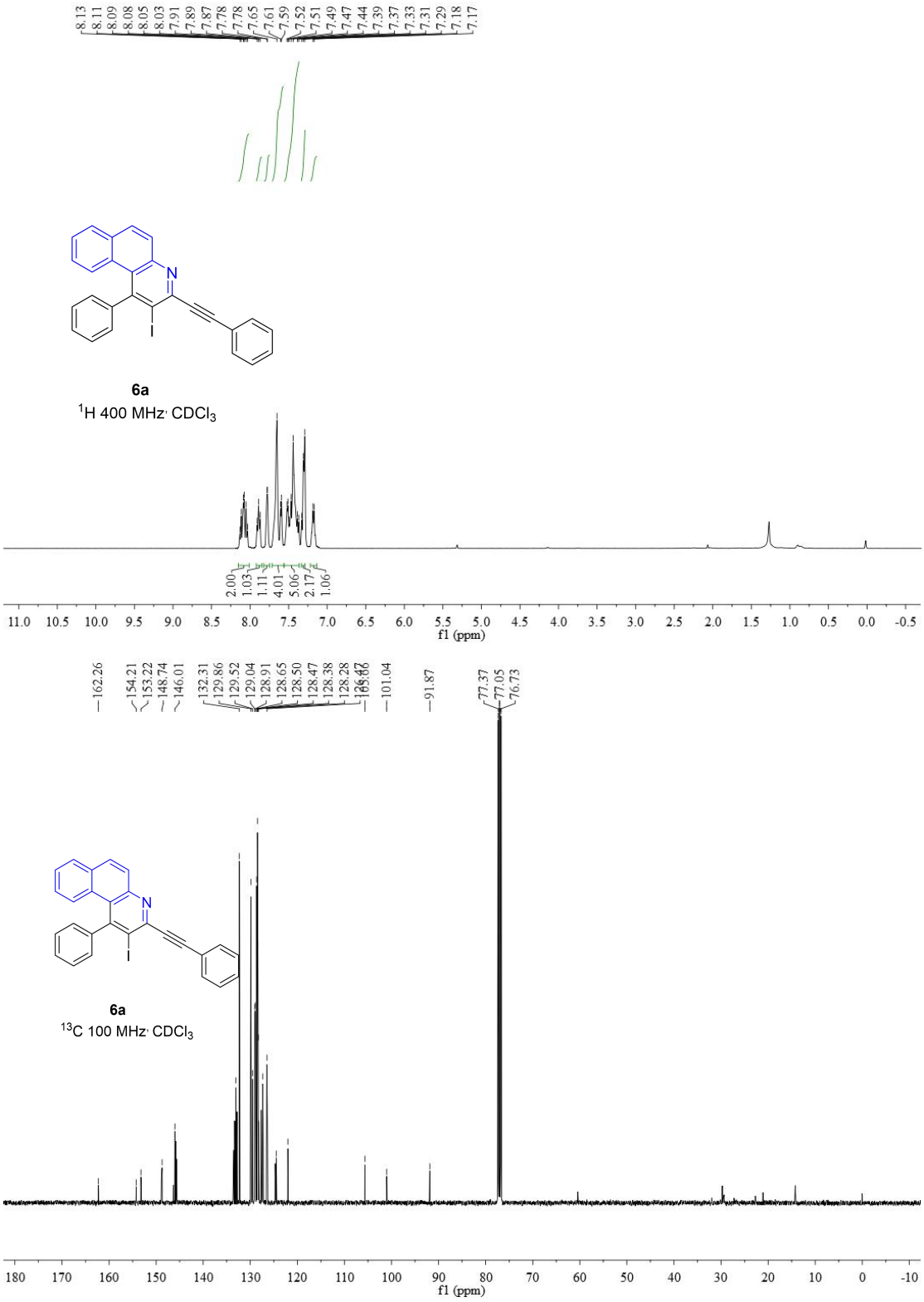
3v

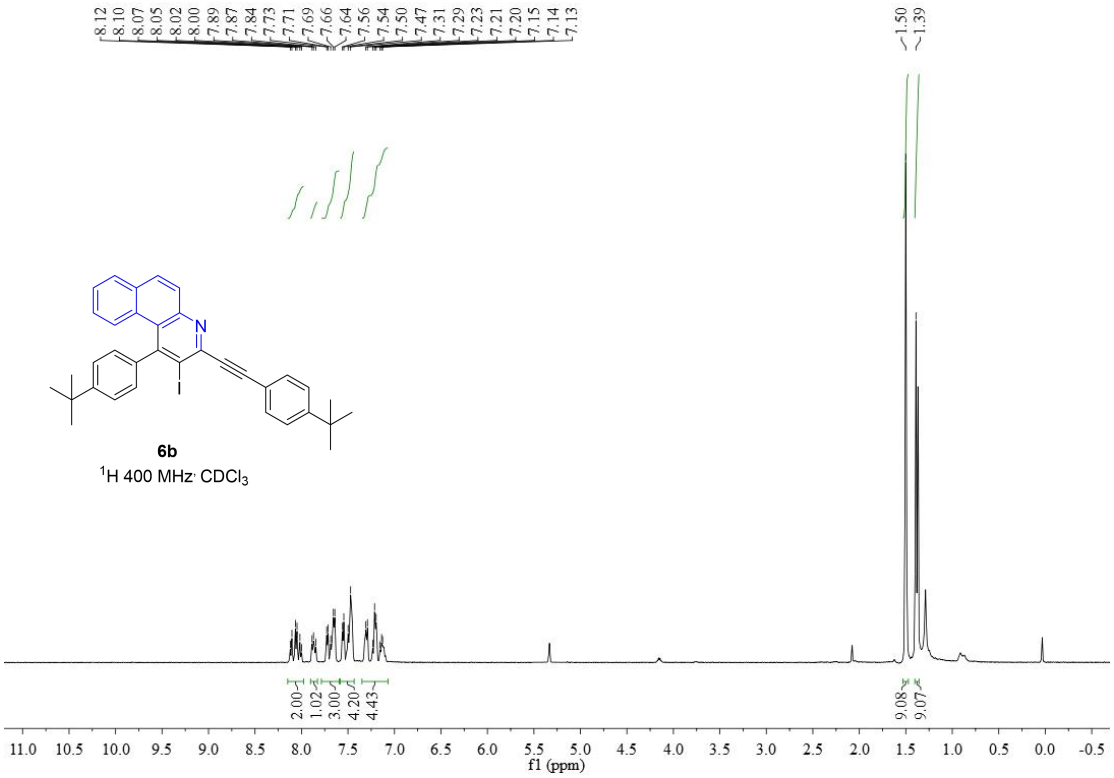

7.13
7.11
6.90
6.88
6.18
3.99
3.97
3.96
1.82
1.81
1.79
1.66
1.47
1.47
1.45
1.45
1.44
1.43
1.42
1.40
1.39
1.37
1.07
0.95
0.94
0.93
O
N
N
C5H11O
4
2.15
2.24
1.00
2.28
2.17
9.21
4.13
9.11
3.09
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


169.97
159.43
147.70
142.77
133.32
129.87
127.27
114.02
77.30
77.04
76.79
68.10
57.31
56.01
32.14
29.84
28.92
28.20
22.49
14.05
O
N
N
C5H11O
4
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.26
5.78
5.77
1.98
1.93
1.69
1.57
1.08
1.07
1.06
1.06
1.05
1.04
1.00
0.91
0.75
0.74
0.73
0.72
0.72
O
N
N
5
1.00
2.07
2.15
1.00
6.21
6.07
2.31
9.08
9.15
2.15
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
0.0
-0.5
f1 (ppm)

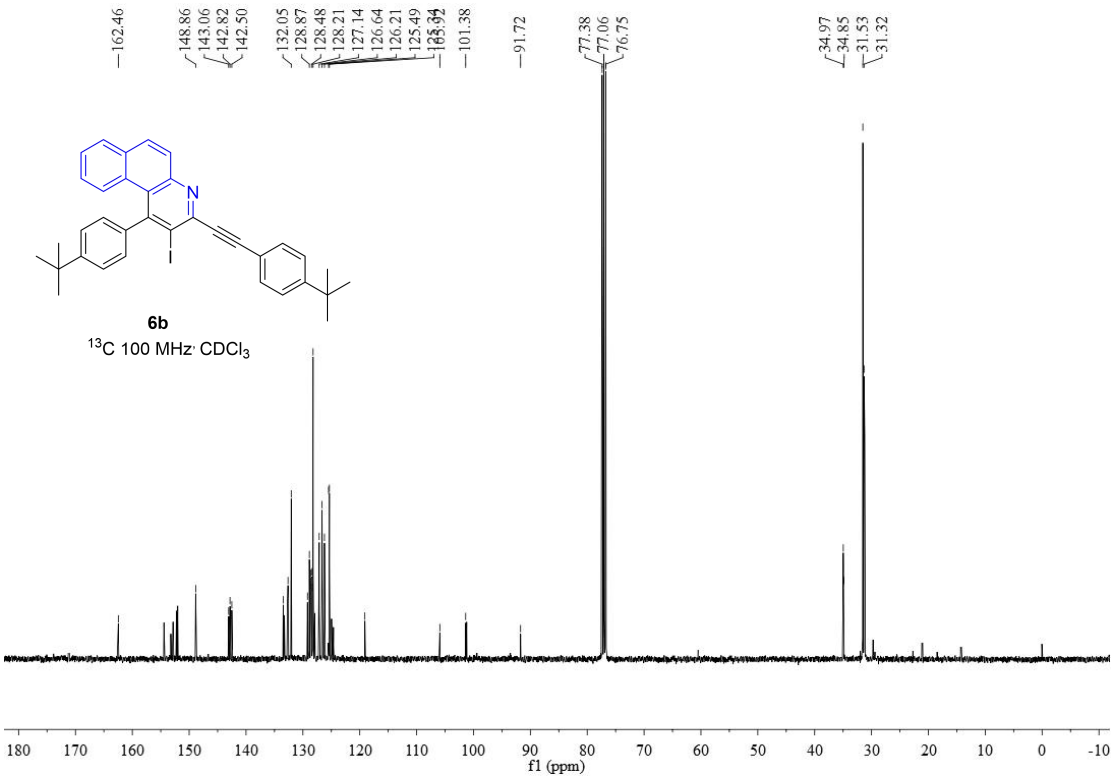

171.29
149.26
146.09
125.99
77.35
77.03
76.71
60.88
59.18
55.05
50.43
33.08
32.10
31.85
31.66
31.58
31.23
12.14
12.00
O
N
N
5
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.13
8.11
8.09
8.08
8.05
8.03
7.91
7.89
7.87
7.78
7.78
7.65
7.61
7.59
7.52
7.51
7.49
7.47
7.44
7.39
7.37
7.33
7.31
7.29
7.18
7.17
N
I
6a
1H 400 MHz, CDCl3
2.00
1.03
1.11
4.01
5.06
2.17
1.06
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)
162.26
154.21
153.22
148.74
146.01
132.31
129.86
129.52
129.04
128.91
128.65
128.50
128.47
128.38
128.28
101.04
91.87
77.37
77.05
76.73
N
I
6a
13C 100 MHz, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



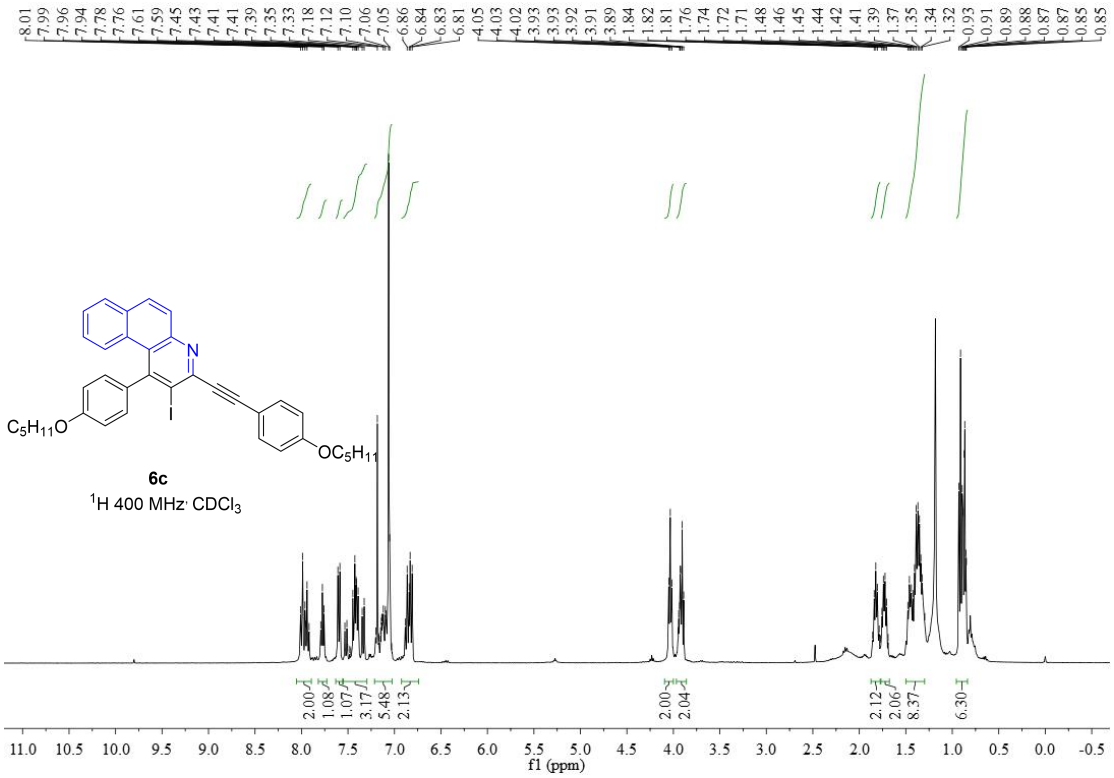
6b

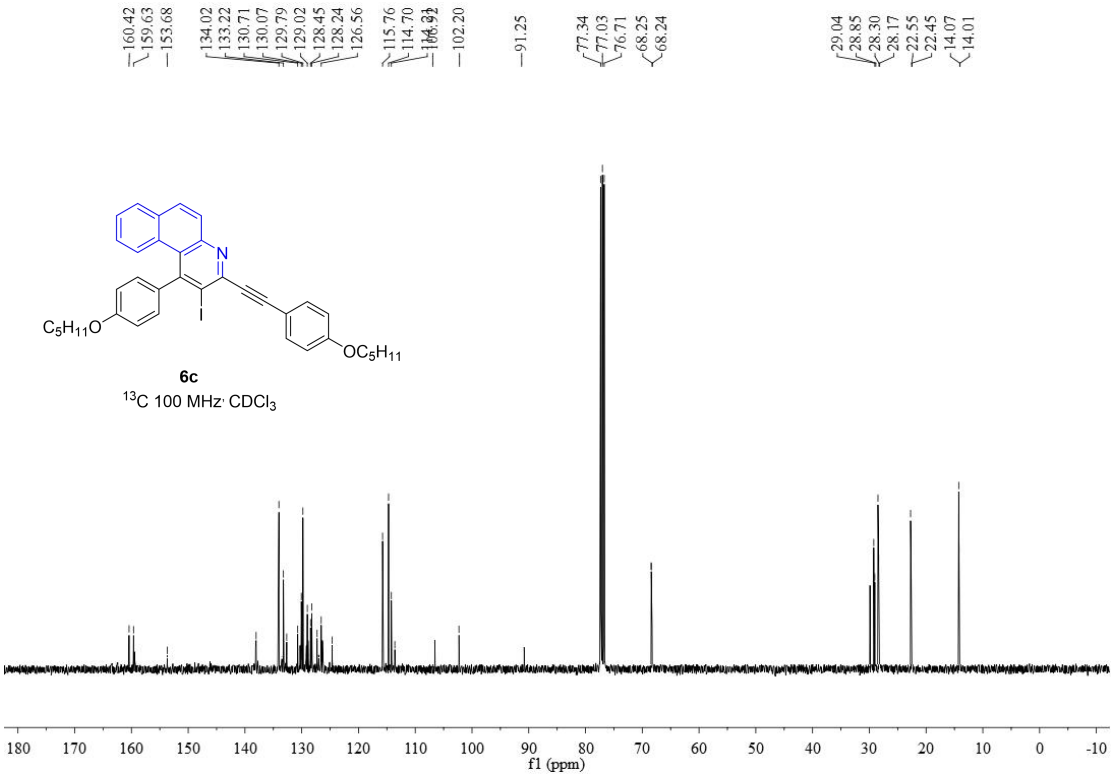
^{1}H 400 MHz· $CDCl_3$



6b

^{13}C 100 MHz· $CDCl_3$


8.01 7.99 7.96 7.94 7.78 7.76 7.61 7.59 7.45 7.43 7.41 7.41 7.39 7.35 7.33 7.18 7.12 7.10 7.06 7.05 6.86 6.84 6.83 6.81
4.05 4.03 4.02 3.93 3.93 3.92 3.91 3.89 1.84 1.82 1.81 1.76 1.74 1.72 1.71 1.48 1.46 1.45 1.44 1.42 1.41 1.39 1.37 1.35 1.34 1.32 0.93 0.91 0.89 0.88 0.87 0.87 0.85 0.85
N
I
C5H11O
OC5H11
6c
1H 400 MHz· CDCl3
2.00 1.08 1.07 3.17 5.48 2.13 2.00 2.04 2.12 2.06 8.37 6.30
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)


160.42 159.63 153.68 134.02 133.22 130.71 130.07 129.79 129.02 128.45 128.24 126.56 115.76 114.70 102.20 91.25 77.34 77.03 76.71 68.25 68.24 29.04 28.85 28.30 28.17 22.55 22.45 14.07 14.01
N
I
C5H11O
OC5H11
6c
13C 100 MHz· CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)