

Supporting Information

Synthesis and Anticholinesterase Activity of 2-Substituted-N-Alkynylindoles

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Materials and Methods

Proton nuclear magnetic resonance spectra (¹H NMR) were obtained on a NMR spectrometer at 400 MHz. Spectra were recorded in CDCl₃ solutions. Chemical shifts

are reported in ppm, referenced to the solvent peak of CDCl₃ or tetramethylsilane (TMS) as the external reference. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (J) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained on a 400 NMR spectrometer at 100 MHz. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), t (triplet), quart (quartet), quint (quintet), sex (sextet), dd (double doublet) and m (multiplet). High resolution mass spectra were recorded on a mass spectrometer using electrospray ionization (ESI). Column chromatography was performed using Silica Gel (230-400 mesh) following the methods described by Still.^[1] Thin layer chromatography (TLC) was performed using Gel GF254, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. Most reactions were monitored by TLC for disappearance of starting material. The following solvents were dried and purified by distillation from the reagents indicated: tetrahydrofuran from sodium with a benzophenone ketyl indicator. All other solvents were ACS or HPLC grade unless otherwise noted. Air- and moisture-sensitive reactions were conducted in flame-dried or oven dried glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen or argon. Reagents and solvents were handled using standard syringe techniques. The *n*-Butyllithium 2.5 M in hexanes purchased from commercial suppliers.

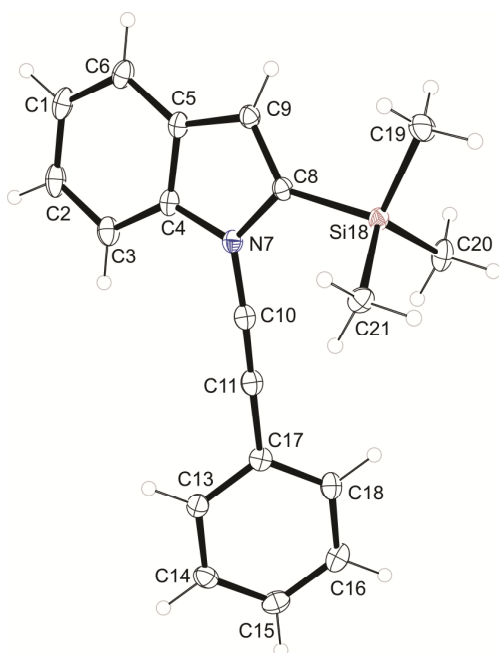


Figure S1. ORTEP structure of compound **3q** (CCDC 1862618).

: 0.102 g Crystal data and structure refinement for **3q**.

	3q
Empirical formula	C ₃₈ H ₃₈ N ₂ Si ₂
Formula weight	578.88
Temperature (K)	110 (2)
Wavelength. λ (Å)	0.71073
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	
<i>a</i> (Å)	11.0160 (4)
<i>b</i> (Å)	11.9919 (4)
<i>c</i> (Å)	13.0752 (5)
α (°)	89.5180 (10)
β (°)	75.5500 (10)
γ (°)	80.3100 (10)
Volume (Å ³)	1647.74 (10)
Z, Calculated density (Mg m ⁻³)	2, 1.167
Absorption coefficient (mm ⁻¹)	0.136
F (000)	616
Crystal size (mm)	0.50 x 0.33x 0.21

Theta range for data collection θ (°)	2.32 a 27.93
Limiting indices	-14 \leq h \leq 14 -13 \leq k \leq 15 -17 \leq l \leq 17
Reflections collected	26553
Reflections unique [R (int)]	7884 [0.0230]
Completeness to theta = 22.61	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9720 and 0.9351
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7884/0/379
Goodness-of-fit on F ²	1.051
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0355 wR2 = 0.0903
R indices (all data)*	R1 = 0.0435 wR2 = 0.0946
Largest diff. peak and hole (e Å ⁻³)	0.308 and -0.295

$$*RI = |F_o - F_c|/|F_o|; wR2 = [w(F_o^2 - F_c^2)^2/(wF_o^2)]^{-1/2}.$$

General procedure for the synthesis of 2-substituted N-alkynylindoles 3. To a Schlenk tube, under an ambient atmosphere, containing 1-(phenylethynyl)-1H-indole derivatives **1** (0.5 mmol) in THF (3mL) were added *n*-BuLi (0.65 mmol, 2.5 M in hexane) was added dropwise over 5 min at -78 °C. The resultant yellow/orange solution was stirred for another 10 min and then warmed to 0 °C over 30 min. The solution was recooled to -78 °C, and the electrophile (0.6 mmol) in 1 mL of THF was added dropwise. The mixture was stirred for 1 h, the room temperature. After quenching with H₂O and extraction with ethyl acetate (3 × 2 mL), the combined organic layers were dried over MgSO₄, and concentrated at reduced pressure to give. The residue was purified by column chromatography over silica gel to provide 2-substituted N-alkynylindoles **3**. **(Phenylethynyl)-1H-indol-2-yl)(p-tolyl)methanol (3a)**. The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a light yellow solid. Yield: 0.101 g (60%); mp 60-63 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.56-7.53 (m, 1H), 7.52-7.49 (m, 1H), 7.43-7.40 (m, 2H), 7.34-7.25 (m, 5H), 7.19-7.10 (m, 4H), 6.43 (s, 1H); 6.09 (s, 1H); 2.73 (s, 1H); 2.32 (s, 3H). ¹³C {¹H} NMR (CDCl₃,

100 MHz): δ (ppm) 143.1, 138.8, 137.9, 137.8, 131.3, 129.1, 128.4, 128.0, 127.3, 126.9, 123.5, 122.4, 122.2, 121.1, 111.1, 103.8, 79.1, 74.1, 69.8, 21.1. MS (EI, 70 eV. m/z (relative intensity)): 338 (26), 337 (100), 219 (21), 204 (95), 90 (10), 77 (3). HRMS (ESI-TOF) m/z calcd for $C_{24}H_{20}NO$ $[M + H]^+$: 338.1545. Found: 338.1552.

Phenyl (phenylethynyl)-1H-indol-2-yl)methanol (3b). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a light yellow solid. Yield: 0.105 g (65%); mp 79-82 °C. 1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.64-7.54 (m, 4H), 7.50-7.33 (m, 9H) 7.29-7.23 (m, 1H), 6.50-6.49 (m, 1H), 6.24 (s, 1H), 2.69 (s, 1H). ^{13}C $\{^1H\}$ NMR ($CDCl_3$, 100 MHz): δ (ppm) 142.9, 140.7, 138.9, 131.4, 128.5, 128.4, 128.2, 128.1, 127.3, 127.0, 123.6, 122.4, 122.3, 121.2, 111.2, 104.2, 79.1, 74.2, 70.0. MS (EI, 70 eV. m/z (relative intensity)): 324 (26), 323 (100), 306 (35), 227 (45), 204 (20), 156 (16), 105 (24). HRMS (ESI-TOF) m/z calcd for $C_{23}H_{18}N$ $[M + H]^+$: 324.1388. Found: 324.1392.

(4-Chlorophenyl)(1-(phenylethynyl)-1H-indol-2-yl)methanol (3c). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a light yellow oil. Yield: 0.118 g (66%). 1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.56-7.52 (m, 1H), 7.51-7.48 (m, 1H), 7.45-7.39 (m, 4H), 7.38-7.26 (m, 6H), 7.23-7.18 (m, 1H), 6.40 (t, $J = 0.8$ Hz, 1H), 6.13 (s, 1H), 2.85 (s, 1H). ^{13}C $\{^1H\}$ NMR ($CDCl_3$, 100 MHz): δ (ppm) 142.3, 139.1, 138.8, 133.9, 131.4, 128.6, 128.5, 128.3, 128.3, 127.1, 123.8, 122.4, 122.1, 121.2, 111.2, 104.2, 78.9, 74.2, 69.2. MS (EI, 70 eV. m/z (relative intensity)): 358 (31), 357 (100), 219 (21), 338 (44), 217 (60), 204 (40), 77 (30). HRMS (ESI-TOF) m/z calcd for $C_{23}H_{17}ClNO$ $[M + H]^+$: 358.0999. Found: 358.1006.

(4-Nitrophenyl)(1-(phenylethynyl)-1H-indol-2-yl)methanol (3d). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as an orange oil. Yield: 0.120 g (65%). 1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22-8.19 (m, 2H), 7.70-7.66 (m, 2H), 7.59-7.52 (m, 2H), 7.47-7.43 (m, 2H), 7.39-7.31 (m, 4H), 7.24-7.20 (m, 1H), 6.39-6.38 (m, 1H), 6.28 (s, 1H), 3.03 (s, 1H). ^{13}C $\{^1H\}$ NMR ($CDCl_3$, 100 MHz): δ (ppm) 147.7, 147.4, 141.3, 138.8, 131.3, 128.5, 128.4, 127.5, 126.9, 124.1, 123.5, 122.6, 121.8, 121.3, 111.2, 104.7, 78.5, 74.4, 68.7. MS (EI, 70 eV. m/z (relative intensity)): 369 (26), 368 (100), 246 (18), 217 (41), 254 (18), 151 (15), 145 (18). HRMS (ESI-TOF) m/z calcd for $C_{23}H_{17}N_2O_3$ $[M + H]^+$: 369.1239. Found: 369.1247.

(2-Fluoro-6-methoxyphenyl)(1-(phenylethynyl)-1*H*-indol-2-yl)methanol (3e). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white solid. Yield: 0.115 g (62%); mp 128-130 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm). 7.63-7.55 (m, 3H), 7.51-7.48 (m, 1H), 7.38-7.24 (m, 5H), 7.18-7.14 (m, 1H), 6.78-6.73 (m, 2H), 6.54 (d, *J* = 10.5 Hz, 1H), 6.35 (s, 1H), 3.85 (s, 1H), 3.83 (s, 3H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 160.7 (d, *J* = 245.9 Hz), 158.7 (d, *J* = 7.4 Hz), 141.9, 139.1, 131.4, 129.8 (d, *J* = 10.8 Hz), 128.3, 127.9, 127.3, 123.5, 122.9, 122.1, 120.9, 116.6 (d, *J* = 15.5 Hz), 111.3, 108.8 (d, *J* = 23.1 Hz), 107.2 (d, *J* = 3.0 Hz), 103.7, 79.1, 73.9, 62.4 (d, *J* = 6.1 Hz), 56.09. MS (EI, 70 eV. *m/z* (relative intensity)): 372 (28), 371 (100), 340 (52), 254 (18), 217 (34), 154 (18), 144 (51), 91(25). HRMS (ESI-TOF) *m/z* calcd for C₂₄H₁₉FNO₂ [M + H]⁺: 372.1400. Found: 372.1411.

(2-Bromo-6-methoxyphenyl)(1-(phenylethynyl)-1*H*-indol-2-yl)methanol (3f). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a yellow oil. Yield: 0.151 g (70%). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.62-7.59 (m, 1H), 7.53-7.49 (m, 3H), 7.46 -7.43 (m, 1H), 7.37 -7.29 (m, 4H), 7.26-7.25 (m 1H), 7.20-7.16 (m, 1H), 6.77 (dd, *J* = 8.7, 3.1 Hz, 1H), 6.49 -6.47 (m, 1H), 6.27 (t, *J* = 0.9 Hz, 1H), 3.77 (s, 3H), 2.75 (d, *J* = 4.5 Hz, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 159.3, 141.5, 140.9, 138.9, 133.36, 131.4, 128.4, 128.1, 127.3, 123.8, 122.5, 122.3, 121.3, 115.5, 114.2, 113.3, 111.3, 104.9, 78.8, 77.3, 68.7, 55.5. HRMS (ESI-TOF) *m/z* calcd for C₂₄H₁₉BrNO₂ [M + H]⁺: 432.0599. Found: 432.0589.

***p*-Tolyl(1-(*p*-tolylethynyl)-1*H*-indol-2-yl)methanol (3g).** The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white solid. Yield: 0.114 g (65%); mp 153-157 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.56-7.66 (m, 2H), 7.36-7.22 (m, 5H), 7.19-7.11 (m, 5H), 6.41 (s, 1H), 6.08 (s, 1H), 2.83 (s, 1H), 2.35 (s, 3H), 2.32 (s, 3H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 143.1, 138.7, 138.2, 137.8, 137.7, 131.3, 129.1, 129.1, 127.2, 126.9, 123.36, 122.1, 121.0, 119.2, 111.1, 103.6, 78.34, 74.02, 69.8, 21.4, 21.1. MS (EI, 70 eV. *m/z* (relative intensity)): 352 (26), 351 (100), 334 (50), 259 (36), 217 (29), 204 (27), 119 (34), 91(33). HRMS (ESI-TOF) *m/z* calcd for C₂₅H₂₂NO [M + H]⁺: 352.1701. Found: 352.1710.

1-(*p*-Tolylethynyl)-1*H*-indole-2-carbaldehyde (3h). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as an orange solid. Yield:

0.158 g (61%); mp 60-62 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 10.12 (s, 1H), 7.76-7.68 (m, 2H), 7.55-7.47 (m, 3H), 7.34 (s, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 2H), 2.38 (s, 3H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 181.0, 140.8, 138.63, 136.3, 131.5, 129.2, 128.0, 126.2, 123.6, 123.4, 119.0, 115.2, 112.3, 77.6, 73.8, 21.5. MS (EI, 70 eV. *m/z* (relative intensity)): 260 (18), 259 (100), 143 (68), 115 (62), 77 (3). HRMS (ESI-TOF) *m/z* calcd for C₁₈H₁₄NO [M + H]⁺: 260.1075. Found: 260.1080.

(1-((4-Chlorophenyl)ethynyl)-1*H*-indol-2-yl)methanol (3j). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white solid. Yield: 0.070 g (50%); mp 80-82 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.61-7.57 (m, 2H), 7.50-7.46 (m, 2H), 7.37-7.32 (m, 3H), 7.25-7.21 (m, 1H), 6.58-6.57 (m, 1H), 4.91 (s, 2H), 1.64 (s, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 139.8, 138.7, 134.1, 132.6, 128.8, 127.4, 123.8, 122.4, 121.2, 120.9, 111.2, 104.8, 79.6, 72.6, 57.2. MS (EI, 70 eV. *m/z* (relative intensity)): 283 (57), 282 (24), 281 (100), 264 (24), 217 (56), 143 (28), 108 (22), 89(19), 75(11). HRMS (ESI-TOF) *m/z* calcd for C₁₇H₁₃ClNO [M + H]⁺: 282.0686. Found: 282.0691.

(1-(Phenylethynyl)-1*H*-indol-2-yl)methanol (3l). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white solid. Yield: 0.069 g (56%); mp 84-87 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.61-7.51 (m, 4H); 7.37-7.28 (m, 4H); 7.22-7.17 (m, 1H), 6.53 (s, 1H), 4.88 (s, 2H), 2.11 (s, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 140.0, 138.8, 131.4, 128.44, 128.2, 127.5, 123.7, 122.4, 122.3, 121.1, 111.2, 104.5, 78.8, 73.7, 57.3. MS (EI, 70 eV. *m/z* (relative intensity)): 247 (100), 230 (29), 217 (44), 144 (16), 115 (17), 89 (18), 63 (10). HRMS (ESI-TOF) *m/z* calcd for C₁₇H₁₄NO [M + H]⁺: 248.1075. Found: 248.1090.

1-(Phenylethynyl)-1*H*-indole-2-carbaldehyde (3m). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as an orange solid. Yield: 0.074 g (60%); mp 80-83 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 10.09 (s, 1H); 7.74-7.69 (m, 2H), 7.63-7.59 (m, 2H), 7.55-7.49 (m, 1H), 7.41-7.22 (m, 5H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 180.9, 140.7, 136.2, 131.5, 128.4, 128.3, 128.1, 126.1, 123.6, 123.5, 122.1, 115.7, 112.2, 78.2, 73.6. MS (EI, 70 eV. *m/z* (relative intensity)): 246 (19), 245 (100), 207 (46), 143 (86), 115 (74), 89 (12), 63 (10). HRMS (ESI-TOF) *m/z* calcd for C₁₇H₁₂NO [M + H]⁺: 246.0919. Found: 246.0924.

Diphenyl (1-(phenylethynyl)-1*H*-indol-2-yl)methanol (3p). The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white solid. Yield: 0.090 g (45%); mp 151-153 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.59-7.56 (m, 1H), 7.47-7.40 (m, 5H), 7.38-7.29 (m, 7H), 7.25-7.17 (m, 4H), 7.04-7.00 (m, 2H), 5.93 (d, *J* = 0.9 Hz, 1H), 3.88 (s, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 145.2, 144.4, 139.4, 132.4, 131.1, 130.0, 128.2, 128.2, 128.1 (2C), 127.7, 127.4 (2C), 126.6, 124.0, 122.4, 121.9, 121.3, 111.1, 108.8, 79.9, 78.6, 75.8. MS (EI, 70 eV. *m/z* (relative intensity)): 400 (26), 399 (77), 294 (81), 281 (100), 207 (83), 105 (40), 77 (38). HRMS (ESI-TOF) *m/z* calcd for C₂₉H₂₂NO [M + H]⁺: 400.1701. Found: 400.1710.

1-(Phenylethynyl)-2-(trimethylsilyl)-1*H*-indole (3q). The product was isolated by column chromatography (hexane was eluent) as a green solid. Yield: 0.130 g (90%); mp 58-61 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 8.10-7.96 (m, 4H), 7.84-7.73 (m, 4H), 7.67-7.60 (m, 1H), 7.18 (s, 1H), 0.90 (s, 9H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 142.8, 141.3, 130.9, 128.5, 128.2, 127.8, 123.7, 123.0, 121.8, 120.9, 114.9, 111.1, 82.4, 71.6, -1.3. MS (EI, 70 eV. *m/z* (relative intensity)): 290 (27), 289 (100), 274 (55), 258 (12), 73 (20). HRMS (ESI-TOF) *m/z* calcd for C₁₉H₂₀NSi [M + H]⁺: 290.1365. Found: 290.1377.

1-(Naphthalen-1-ylethynyl)-2-(trimethylsilyl)-1*H*-indole (3r). The product was isolated by column chromatography (hexane was eluent) as a green oil. Yield: 0.119 g (70%). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 8.73-8.65 (m, 1H), 8.10-7.92 (m, 4H), 7.83-7.61 (m, 4H), 7.59-7.52 (m, 1H), 7.45-7.36 (m, 1H), 6.99 (d, *J* = 0.9 Hz, 1H), 0.68 (s, 9H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 142.9, 141.6, 133.4, 133.2, 130.1, 128.4, 126.8, 126.5, 126.2, 125.4, 123.9, 122.0, 121.0, 120.7, 115.2, 111.2, 86.9, 69.8, -1.2. MS (EI, 70 eV. *m/z* (relative intensity)): 340 (30), 339 (100), 324 (33), 308 (25), 154 (24), 73(35). HRMS (ESI-TOF) *m/z* calcd for C₂₃H₂₂NSi [M + H]⁺: 340.1522. Found: 340.1530.

2-(*tert*-Butyldimethylsilyl)-1-(phenylethynyl)-1*H*-indole (3s). The product was isolated by column chromatography (hexane was eluent) as a green solid. Yield: 0.108 g (65%); mp 81-84 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.65-7.63 (m, 1H), 7.61-7.57 (m, 1H), 7.55-7.51 (m, 2H), 7.39-7.29 (m, 4H), 7.22-7.17 (m, 1H), 6.79 (d, *J* = 0.9 Hz, 1H), 1.01 (s, 9H), 0.46 (s, 6H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 141.6, 140.7, 131.0, 128.5, 128.2, 127.8, 123.7, 123.1, 121.9, 120.8, 116.7, 111.2, 83.1, 71.6,

26.9, 17.5, -5.2. MS (EI. 70 eV. m/z (relative intensity)): 332 (14), 331 (44), 274 (100), 258 (12), 230 (9), 129 (4). HRMS (ESI-TOF) m/z calcd for C₂₂H₂₆NSi [M + H]⁺: 332.1835. Found: 332.1841.

1-(Phenylethynyl)-2-(phenylthio)-1H-indole (3t). The product was isolated by column chromatography (hexane was eluent) as a yellow oil. Yield: 0.081 g (50 %). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.59-7.56 (m, 2H), 7.37-7.13 (m, 12H), 6.93 (d, *J* = 0.8 Hz, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 139.9, 135.4, 131.4, 130.6, 129.2, 128.7, 128.4, 128.0, 127.5, 126.7, 124.7, 122.6, 122.6, 121.1, 114.2, 111.7, 78.9, 75.1. MS (EI. 70 eV. m/z (relative intensity)): 326 (26), 325 (100), 291 (18), 204 (8), 146 (11), 89(9). HRMS (ESI-TOF) m/z calcd for C₂₂H₁₆NS [M + H]⁺: 326.1003. Found: 326.1011.

1-(Phenylethynyl)-2-(phenylselanyl)-1H-indole (3v). The product was isolated by column chromatography (hexane was eluent) as a yellow oil. Yield: 0.102 g (55 %). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.60-7.55 (m, 2H), 7.48-7.44 (m, 2H), 7.40-7.37 (m, 2H), 7.34-7.27 (m, 4H), 7.23-7.18 (m, 4H), 6.91 (d, *J* = 0.8 Hz, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 140.0, 131.4, 131.3, 130.6, 129.3, 128.3, 127.9, 127.9, 127.2, 126.6, 124.2, 122.6, 122.3, 120.8, 114.9, 111.6, 79.6, 74.5. MS (EI. 70 eV. m/z (relative intensity)): 375 (17), 374 (23), 373 (90), 292 (100), 189 (22), 145(49), 89(29). HRMS (ESI-TOF) m/z calcd for C₂₂H₁₆NSe [M + H]⁺: 374.0448. Found: 374.0460.

General procedure for the synthesis of 2-(2,2-dibromovinyl)-1-(phenylethynyl)-1H-indole (4a). To a Schlenck tube, under an ambient atmosphere, containing CBr₄ (2.2 mmol) in CH₂Cl₂ (10 mL) and this solution was cooled to 0 °C. To this solution was added the PPh₃ (4.4 mmol), and the color of the reaction turned orange. After stirring the mixture for 10 minutes at that temperature, the corresponding *N*-alkynyl-1H-indole-2-carbaldehyde (1 mmol) was added dropwise to the solution at 0 °C. After stirring 2 h, the reaction mixture was diluted with pentane, the residue was purified by column chromatography over silica gel to provide 2-(2,2-dibromovinyl)-1-(phenylethynyl)-1H-indole (4a). **2-(2,2-Dibromovinyl)-1-(phenylethynyl)-1H-indole (4a).** Was isolated by column chromatography (hexane was eluent) as a light yellow oil. Yield: 0.299 g (75%). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.71-7.58 (m, 1H), 7.51-7.43 (m, 2H), 7.37-7.32 (m, 1H), 7.30-7.09 (m, 5H), 6.80-6.76 (m, 2H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 137.1, 135.1, 133.0, 132.6, 129.2, 128.9, 128.6, 127.9, 125.6, 124.7,

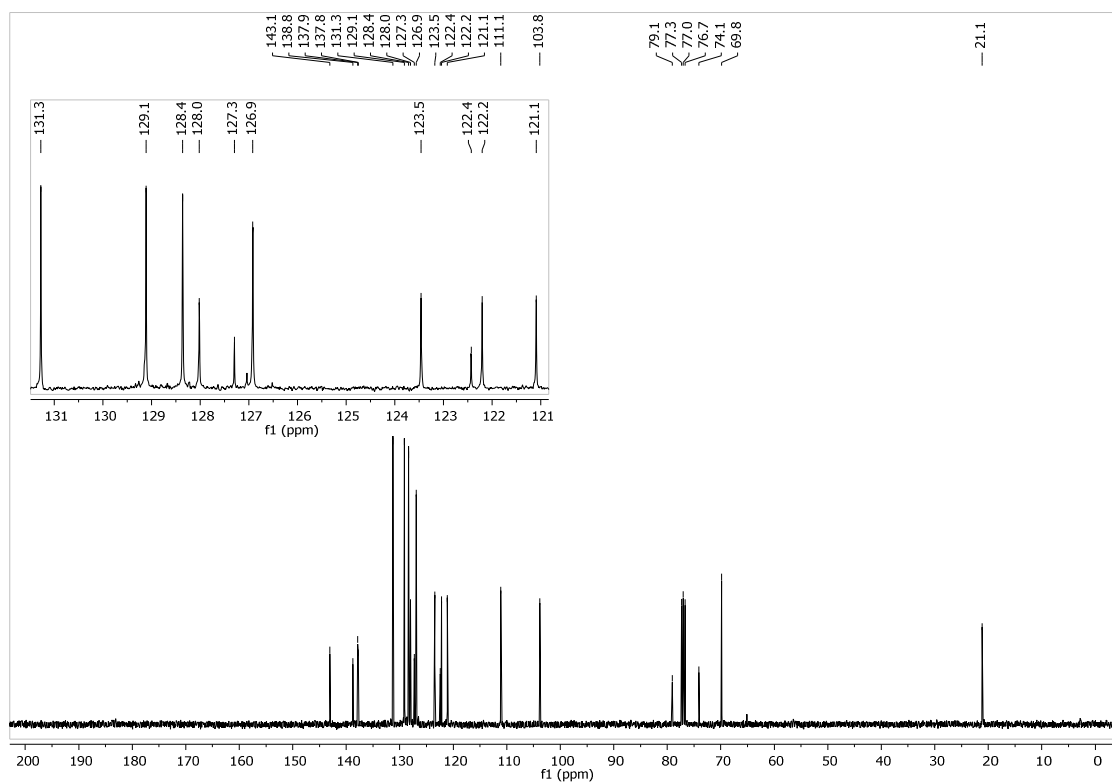
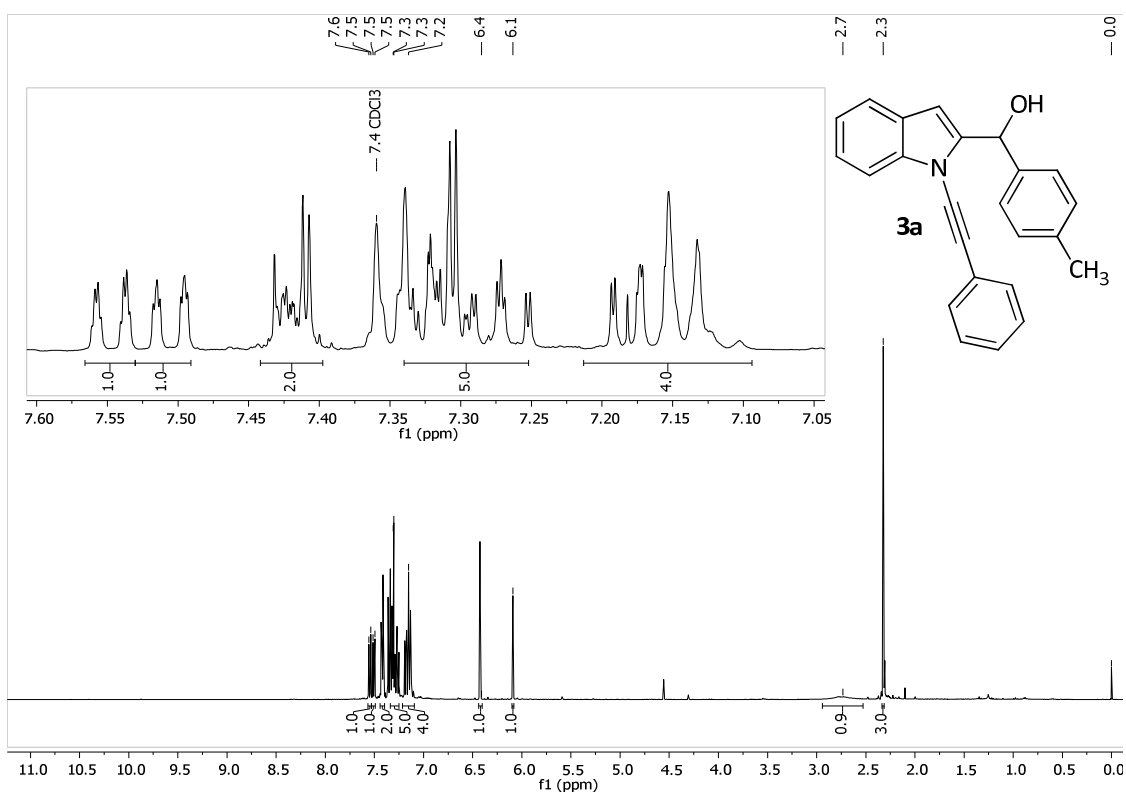
122.45, 121.6, 112.9, 111.2, 107.9, 91.4. HRMS (ESI-TOF) m/z calcd for $C_{18}H_{12}Br_2N$ $[M + H]^+$: 399.9336. Found: 399.9345.

General procedure for the synthesis of 3-phenyl-1-(1-(phenylethynyl)-1*H*-indol-2-yl)prop-2-yn-1-ol (4b). To a solution of terminal alkyne (1.2 equiv) in THF (3 mL), was added *n*-BuLi (1.1 equiv, 2.5 M in hexane) slowly at -78 °C. The reaction mixture was stirred at the same temperature for 30 min, the *N*-alkynyl-1*H*-indole-2-carbaldehyde (1 mmol) was added and the reaction mixture was warmed up to room temperature. After the reaction was completed as monitored by thin-layer chromatography (usually 1 h), the reaction mixture was quenched with H₂O and extraction with ethyl acetate (3 × 2 mL), the combined organic layers were dried over MgSO₄, and concentrated at reduced pressure to give. The residue was purified by column chromatography over silica gel to provide 3-phenyl-1-(1-(phenylethynyl)-1*H*-indol-2-yl)prop-2-yn-1-ol (**4b**). **3-Phenyl-1-(1-(phenylethynyl)-1*H*-indol-2-yl)prop-2-yn-1-ol (4b).** The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a brown solid. Yield: 0.243 g (70%); mp 116-119 °C. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.64-7.57 (m, 2H), 7.56-7.52 (m, 2H), 7.48-7.46 (m, 2H), 7.36-7.26 (m, 7H), 7.23-7.19 (m, 1H), 6.81 (d, *J* = 0.9 Hz, 1H), 6.02 (s, 1H), 2.65 (s, 1H). ¹³C {¹H} NMR (CDCl₃, 100 MHz): δ (ppm) 139.6, 139.2, 131.9, 131.5, 128.8, 128.5, 128.4, 128.2, 127.1, 124.2, 122.5, 121.5, 111.4, 104.6, 86.6, 86.45, 78.8, 74.4, 58.63. HRMS (ESI-TOF) m/z calcd for $C_{25}H_{18}NO$ $[M + H]^+$: 348.1388. Found: 348.1394.

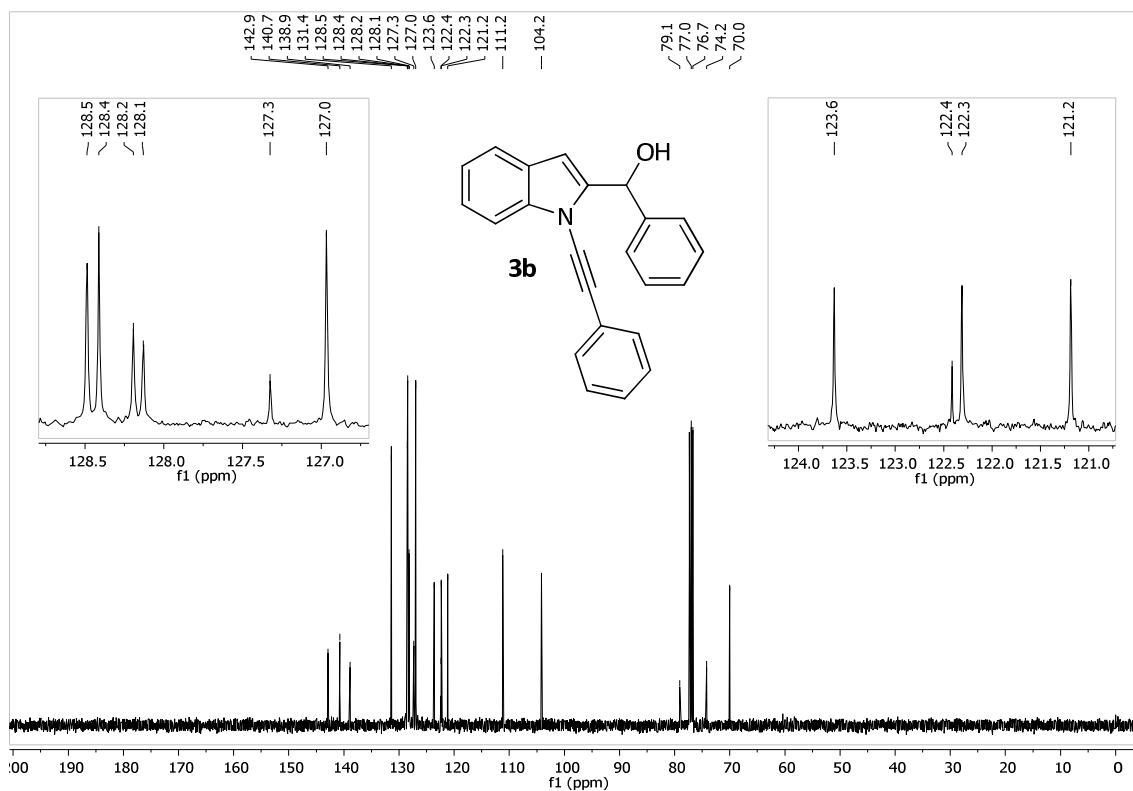
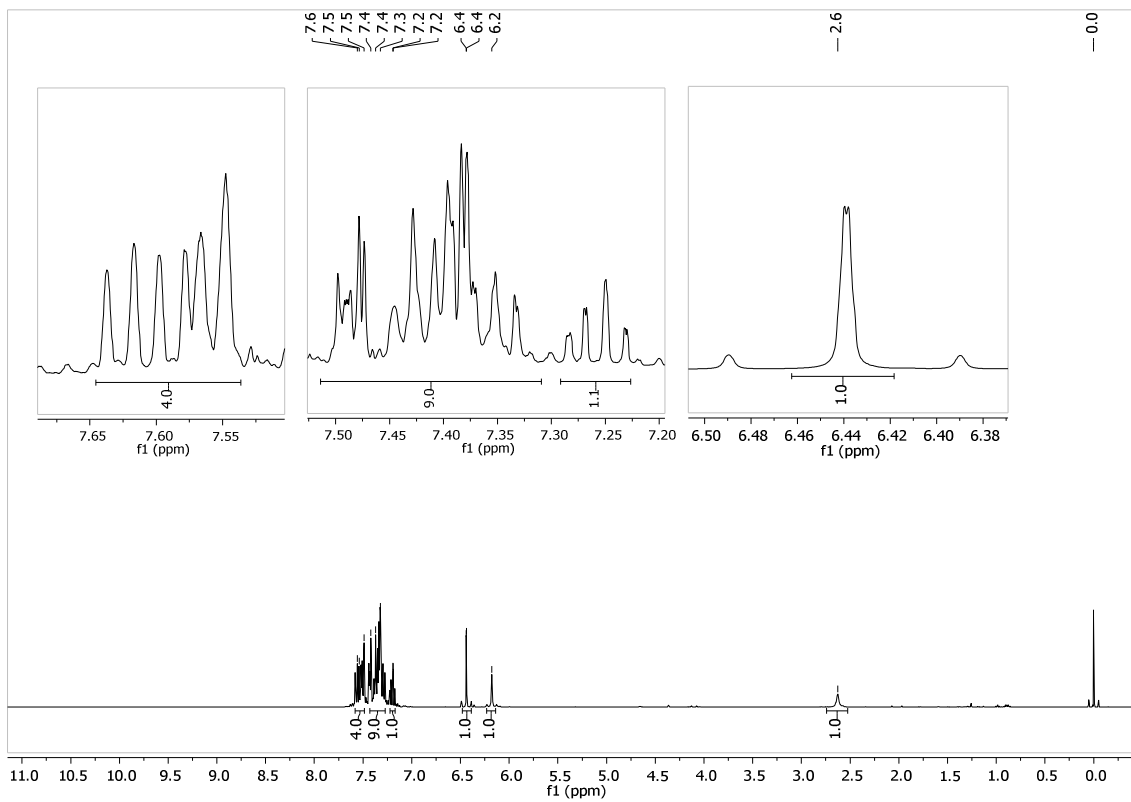
General procedure for the synthesis of (Z)-(1-(2-phenyl-2-(phenyltellanyl)vinyl)-1*H*-indol-2-yl)(*p*-tolyl)methanol (4c). To a solution of (4-chlorophenyl)(1-(phenylethynyl)-1*H*-indol-2-yl)methanol (**3c**) (0.25 mmol) in EtOH (3 mL), was added diorganoil diteluret (0.5 equiv), was added and the reaction mixture NaBH₄ (5 equiv) warmed up to room temperature. The reaction was refluxed for 5 h. The reaction mixture was extracted with NaCl₄ and ethyl acetate (3 × 2 mL), the combined organic layers were dried over MgSO₄, and concentrated at reduced pressure to give. The residue was purified by column chromatography over silica gel to provide (Z)-(1-(2-phenyl-2-(phenyltellanyl)vinyl)-1*H*-indol-2-yl)(*p*-tolyl)methanol (**4c**). **(Z)-(1-(2-Phenyl-2-(phenyltellanyl)vinyl)-1*H*-indol-2-yl)(*p*-tolyl)methanol (4c).** The product was isolated by column chromatography (hexane/ethyl acetate 97:3 as eluent) as a white

solid. Yield: 0.126 g (70%); mp 67-69 °C. ^1H NMR (CDCl_3 , 400 MHz): δ (ppm) 7.67 (d, $J = 8.3$ Hz, 1H), 7.57 -7.52 (m, 2H), 7.38 -7.30 (m, 6H), 7.27- 7.05 (m, 8H), 6.92-6.85 (m, 1H), 6.85 -6.55 (m, 1H), 6.33 (s, 1H), 6.02 (s, 1H), 2.42 (s, 1H), 2.35 (s, 3H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz): δ (ppm) 141.5, 138.1, 137.9, 136.9, 135.9, 129.2, 129.0, 128.7, 128.5, 128.5, 128.2, 127.4, 126.7, 126.7, 126.0, 124.3, 122.9, 122.4, 121.1, 120.8, 111.6, 104.3, 69.7, 21.1. HRMS (ESI-TOF) m/z calcd for $\text{C}_{30}\text{H}_{26}\text{NO}$ $[\text{M} + \text{H}]^+$: 546.1077. Found: 546.1053.

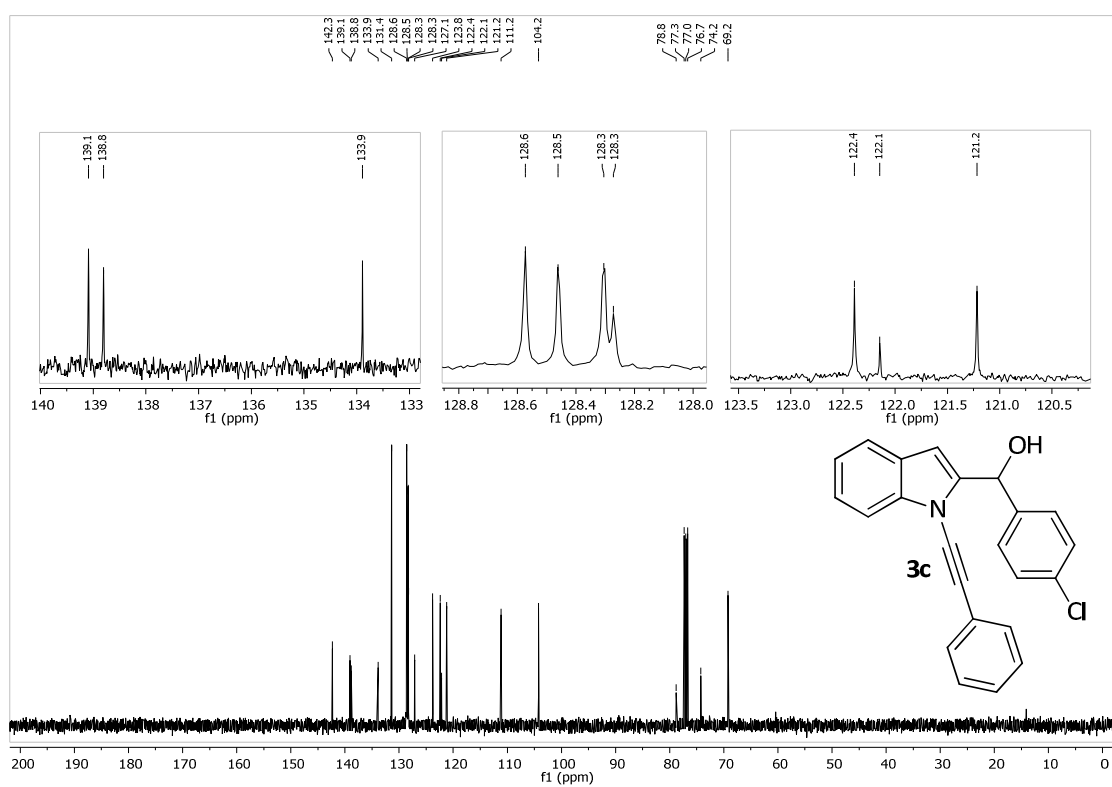
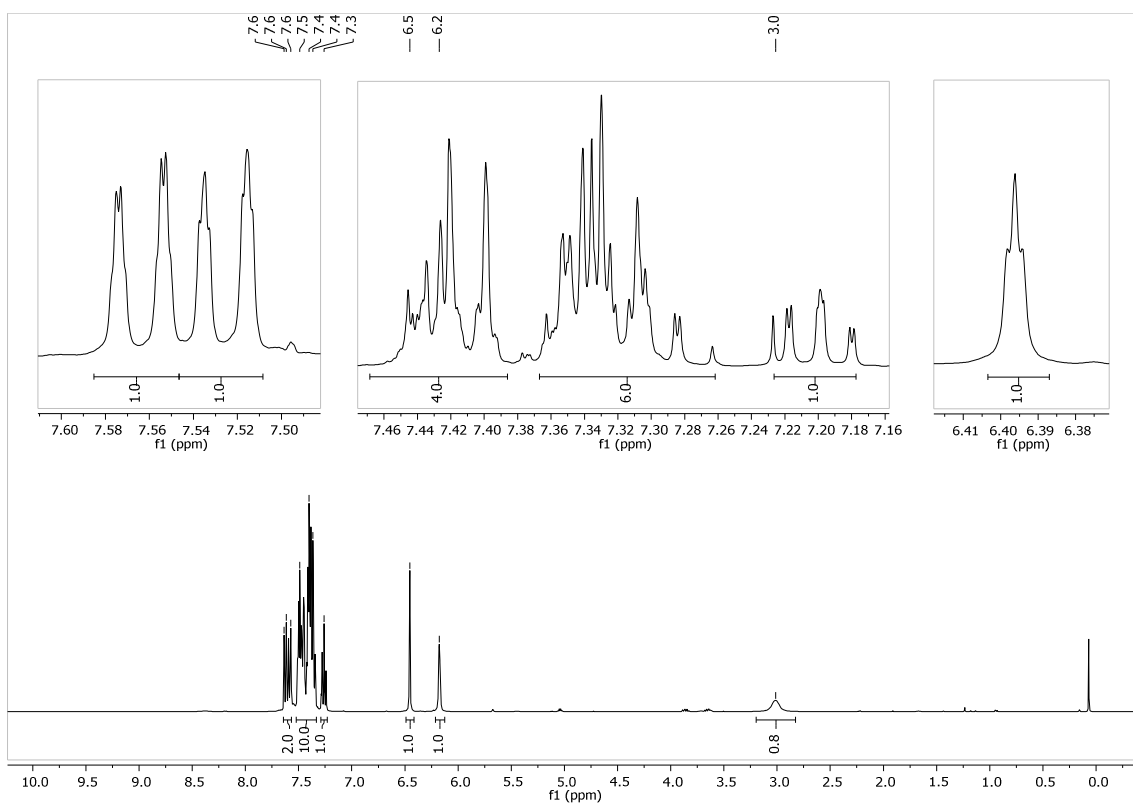
NMR Spectra



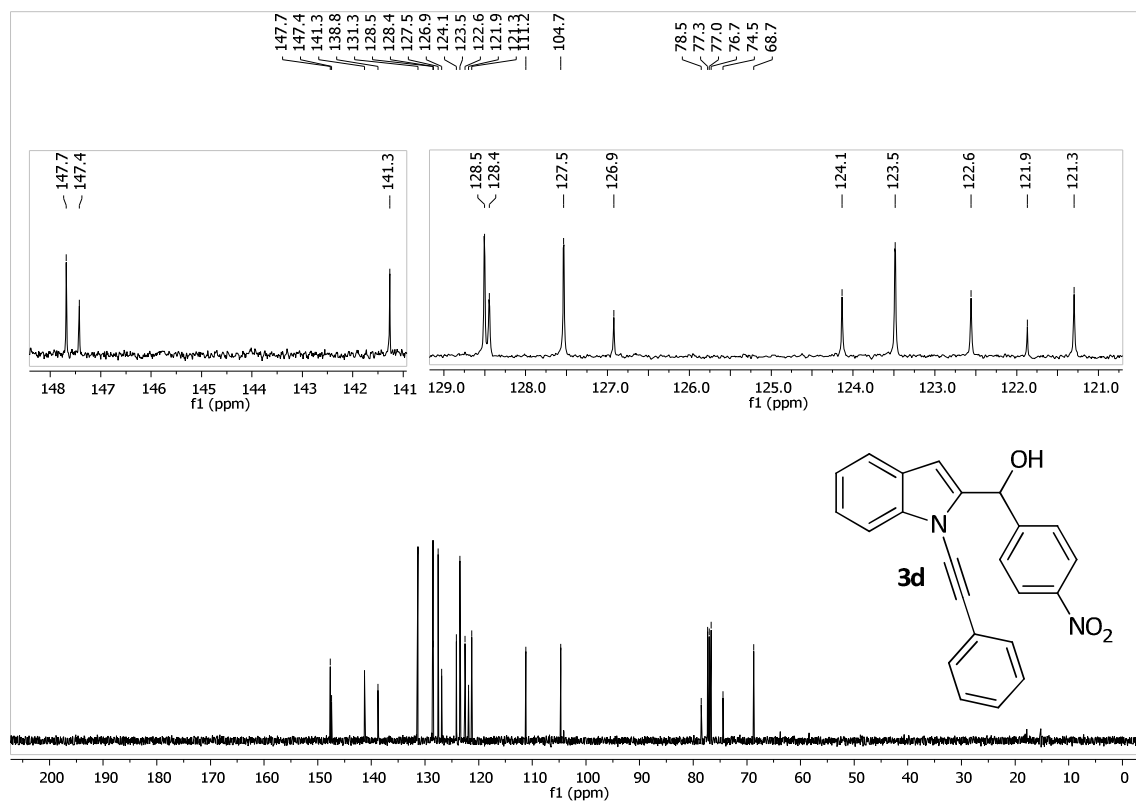
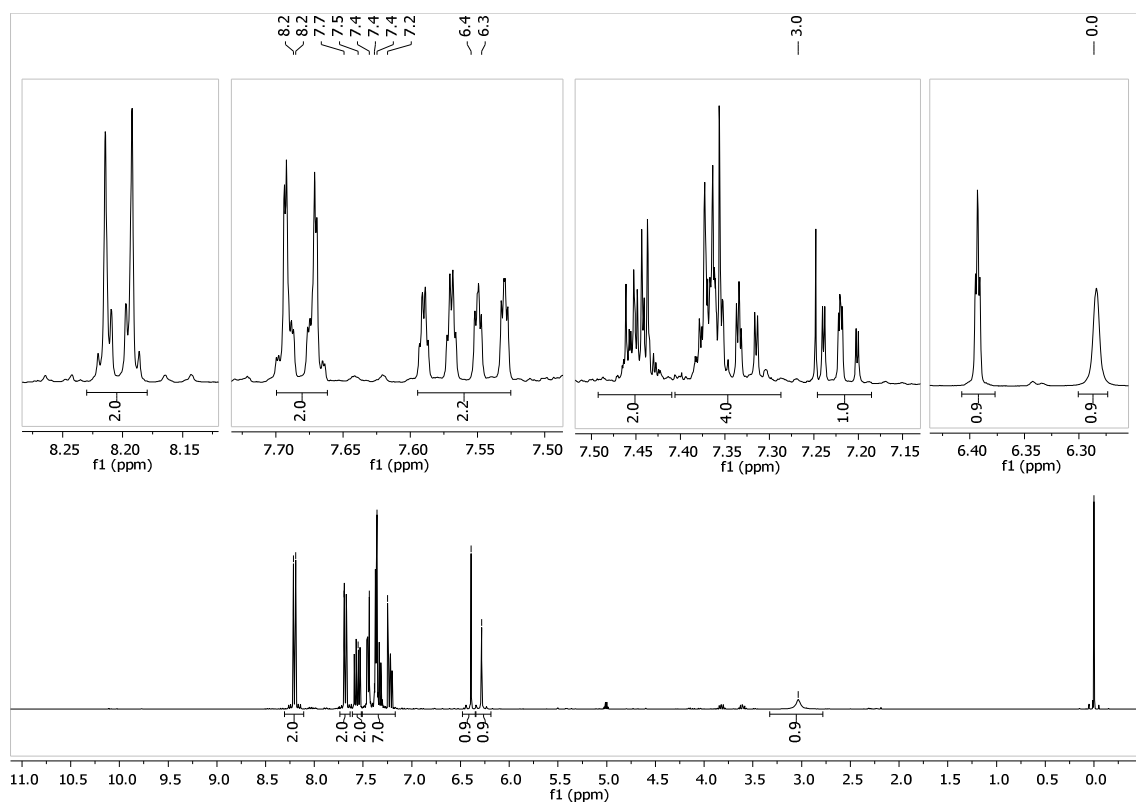
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3a** in CDCl₃.



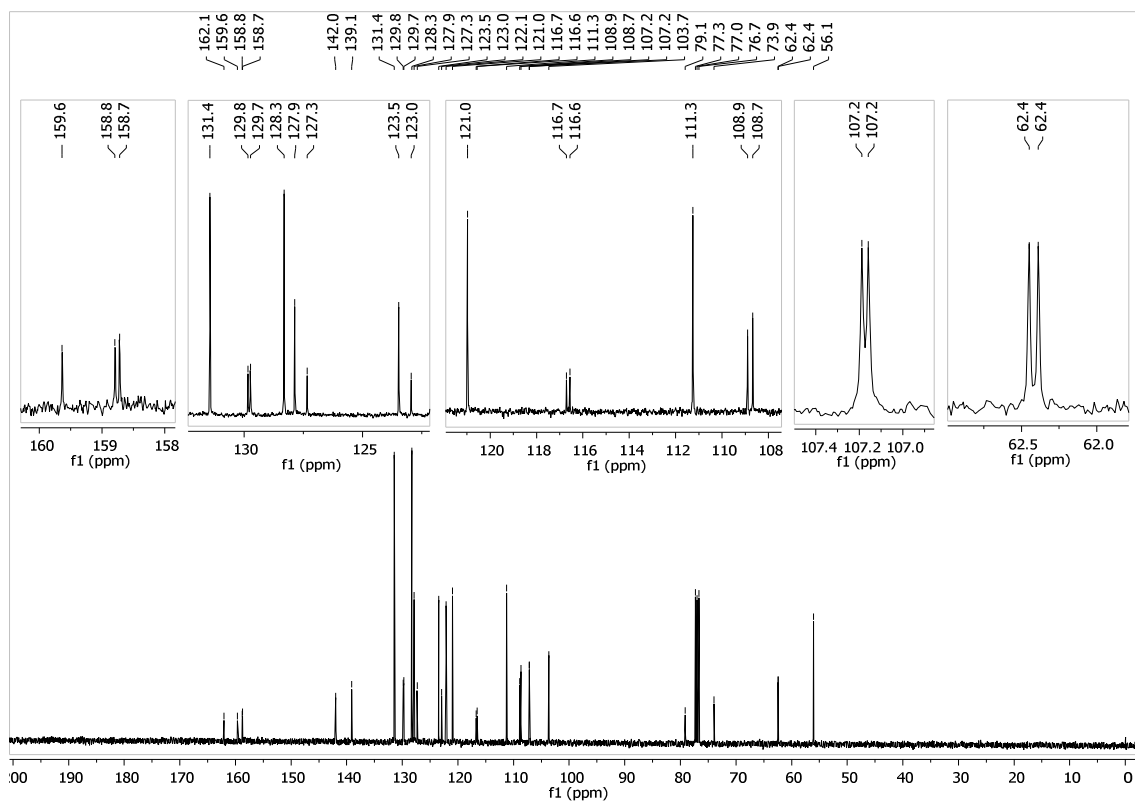
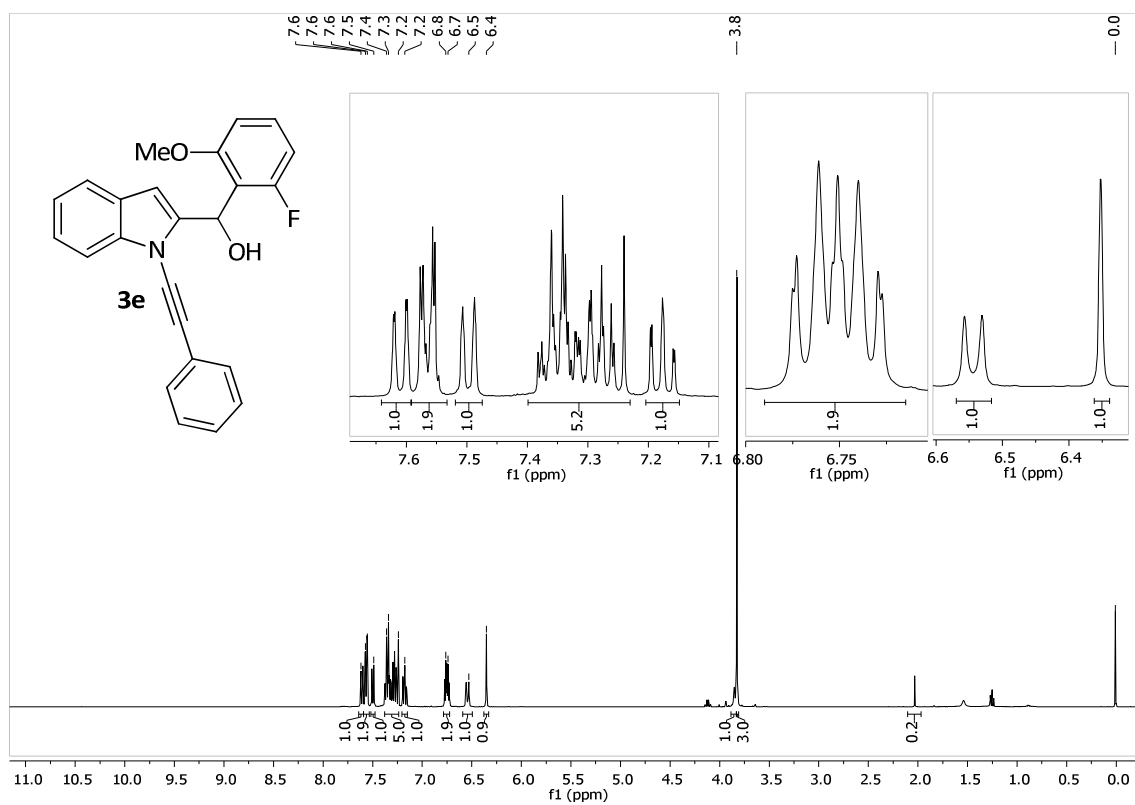
The ^1H (400 MHz) and ^{13}C (100 MHz) spectra of **3b** in CDCl_3 .



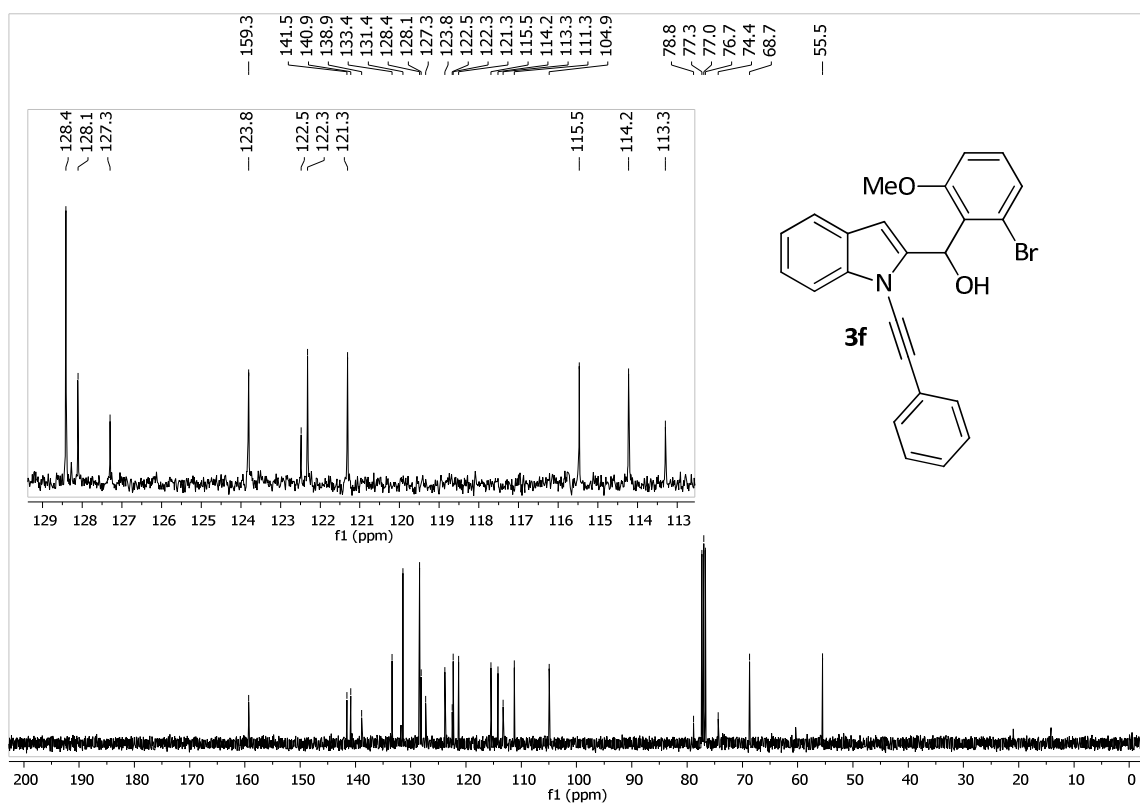
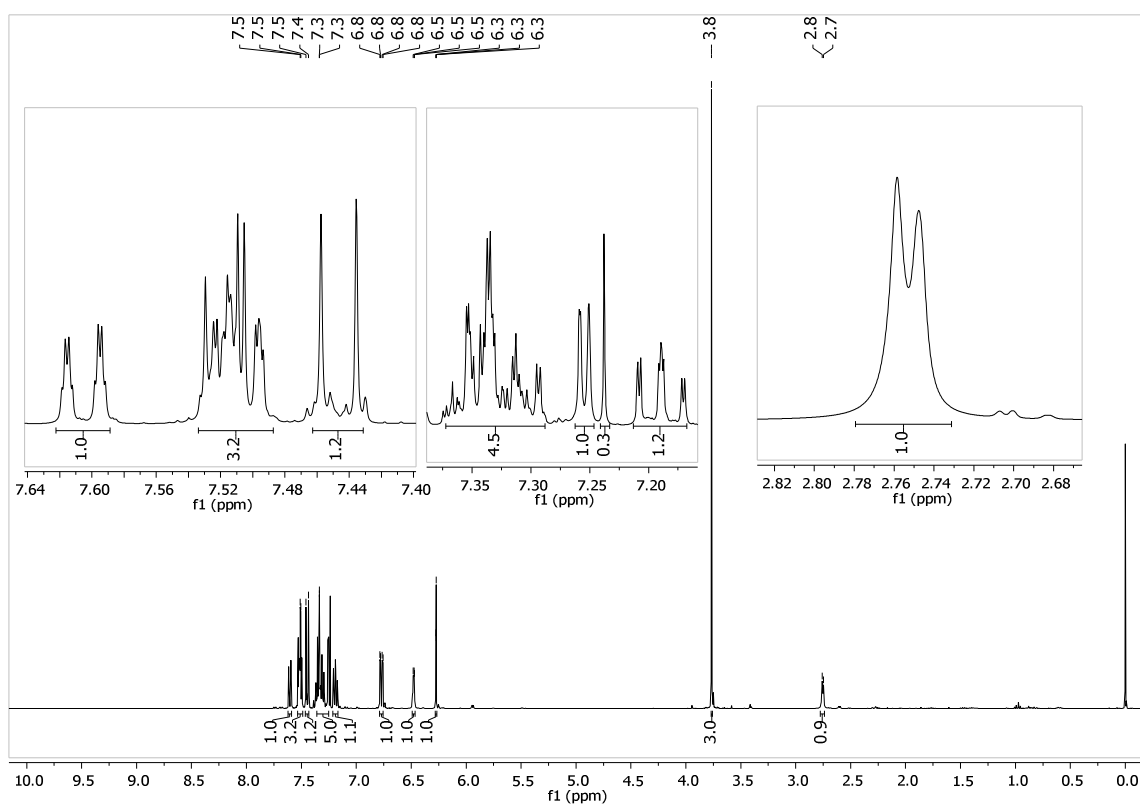
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3c** in CDCl₃.



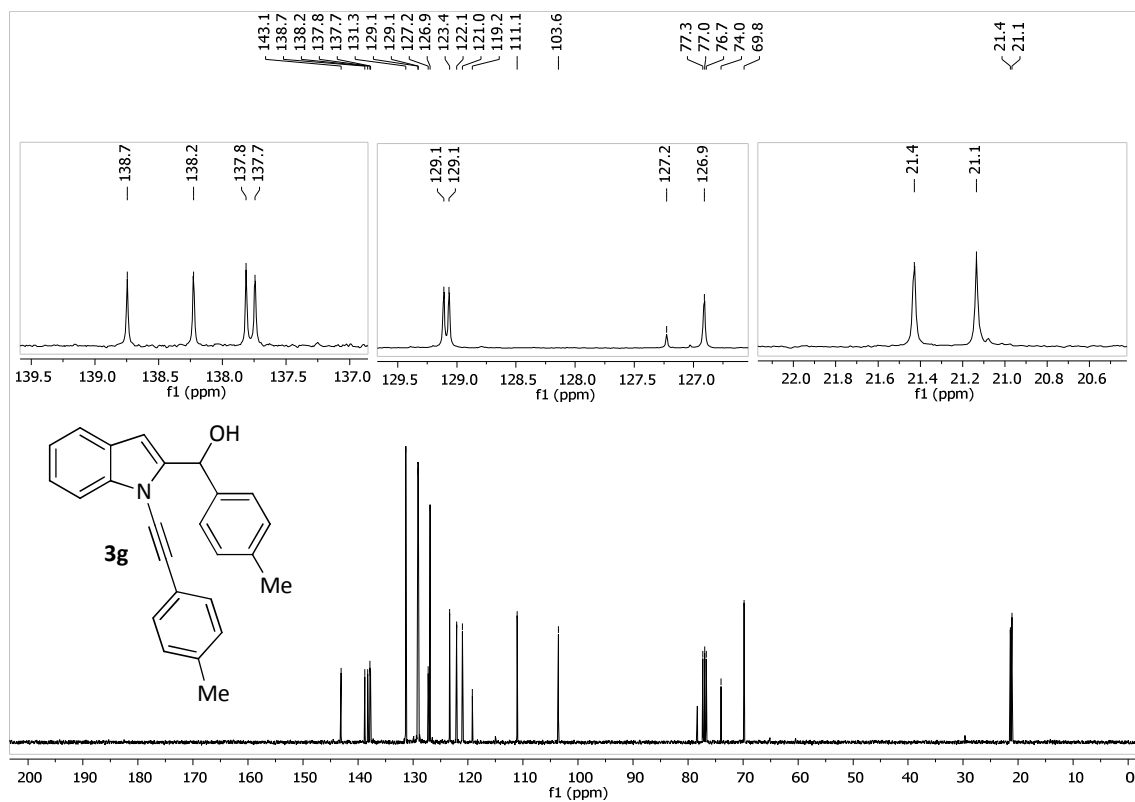
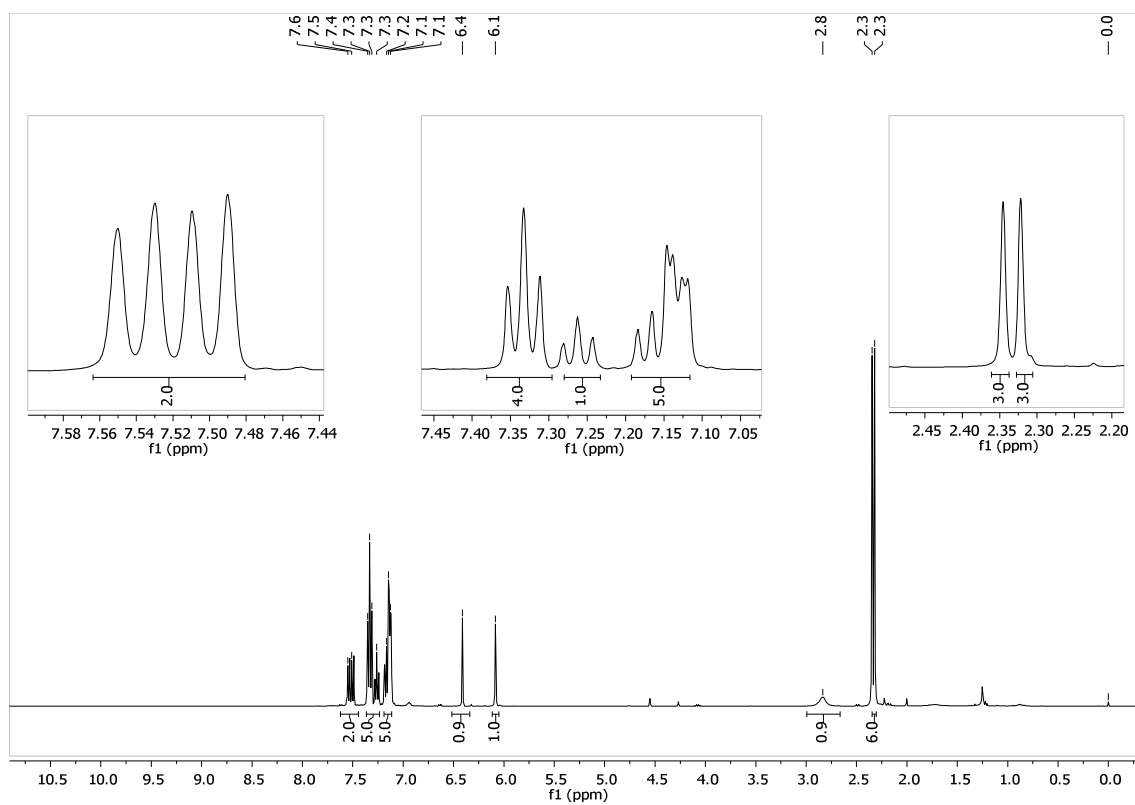
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3d** in CDCl₃.



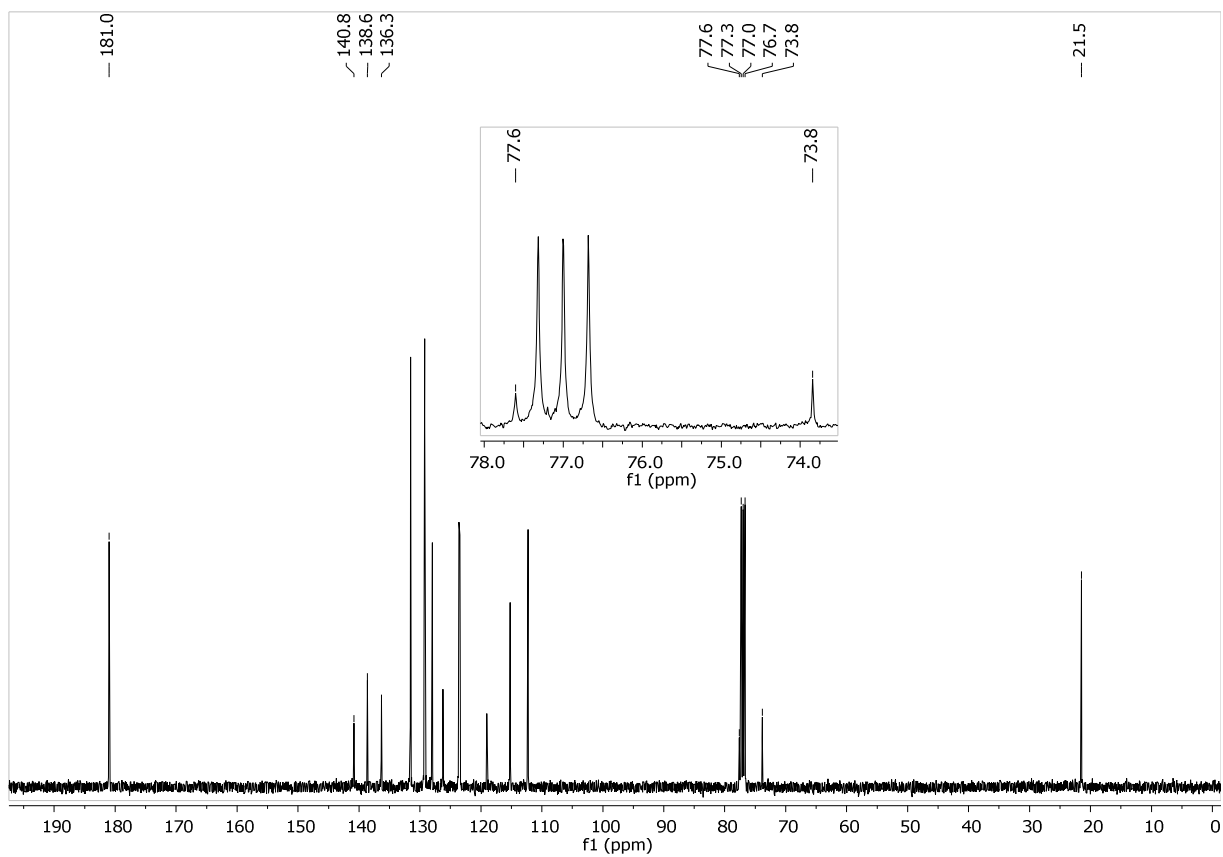
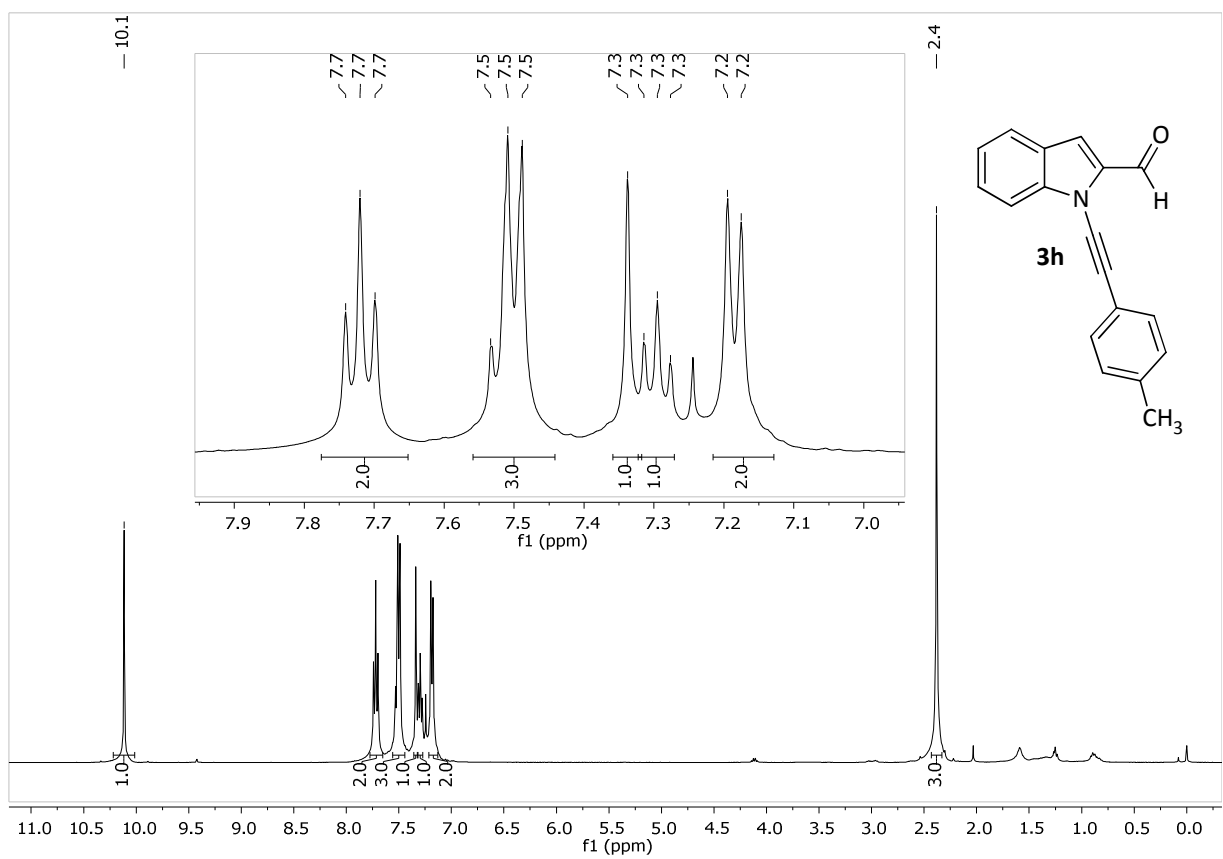
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3e** in CDCl₃.



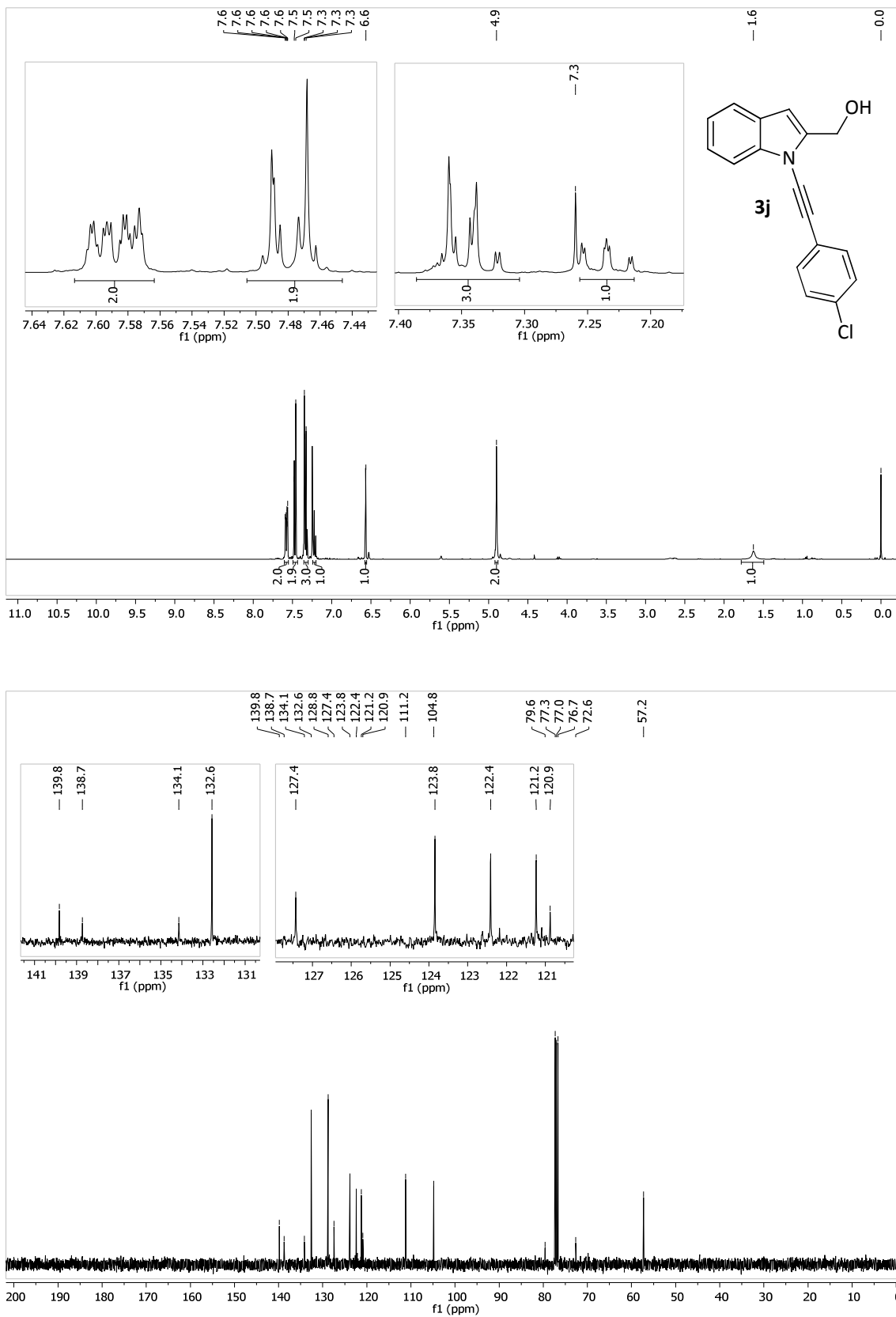
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3f** in CDCl₃.



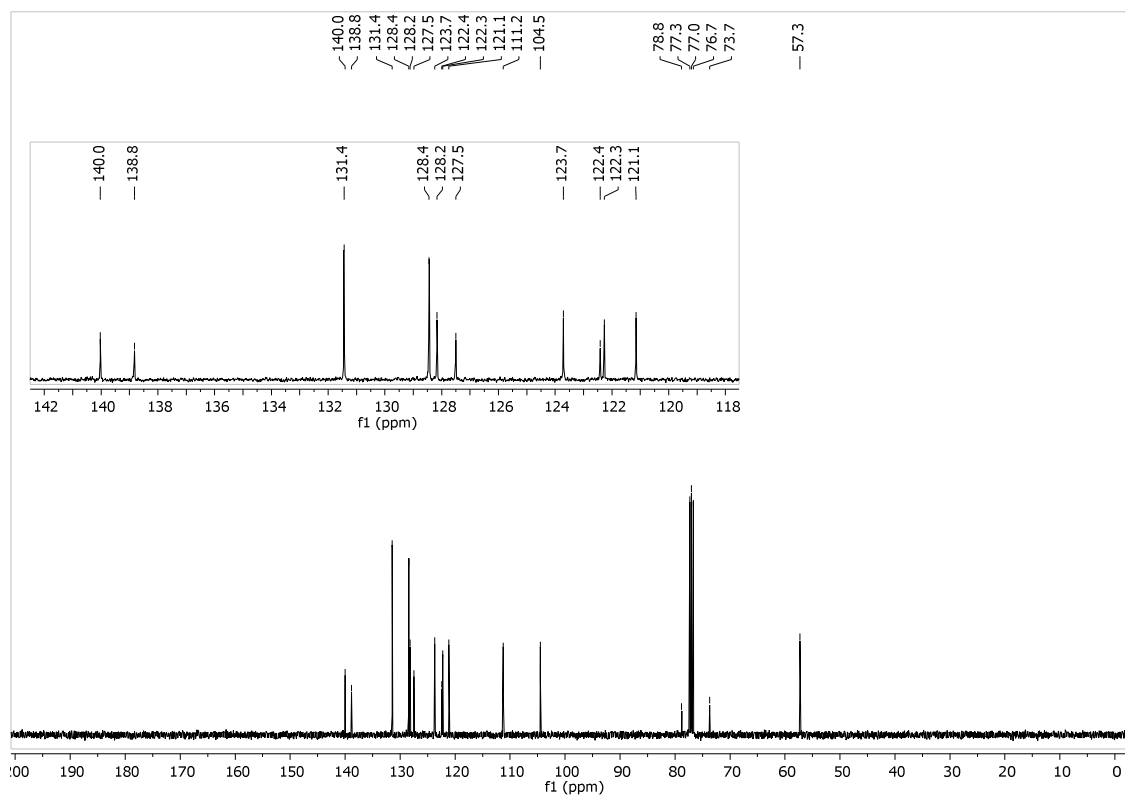
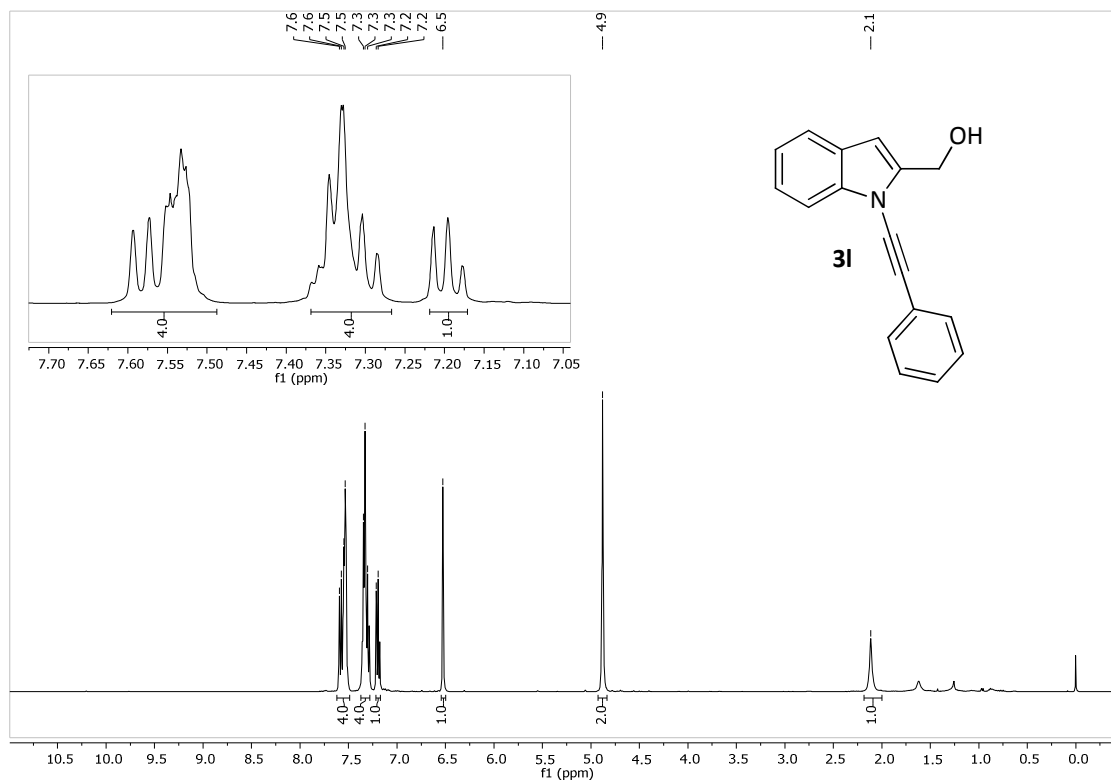
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3g** in CDCl₃.



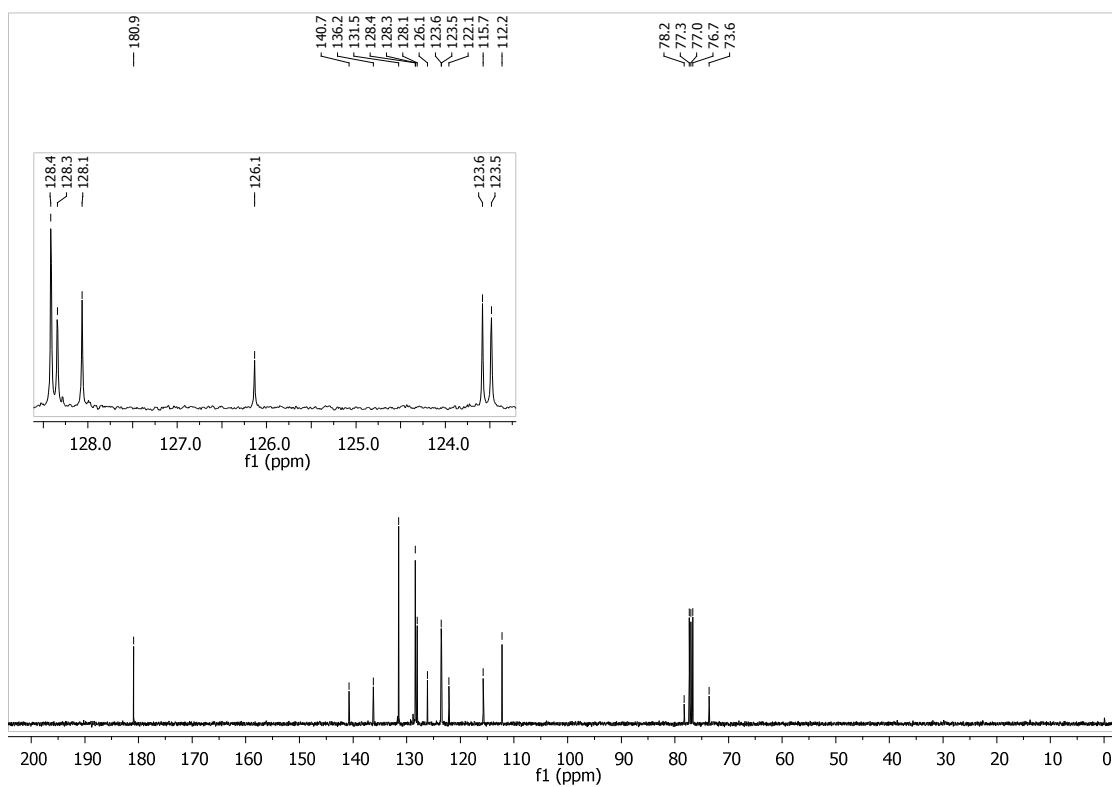
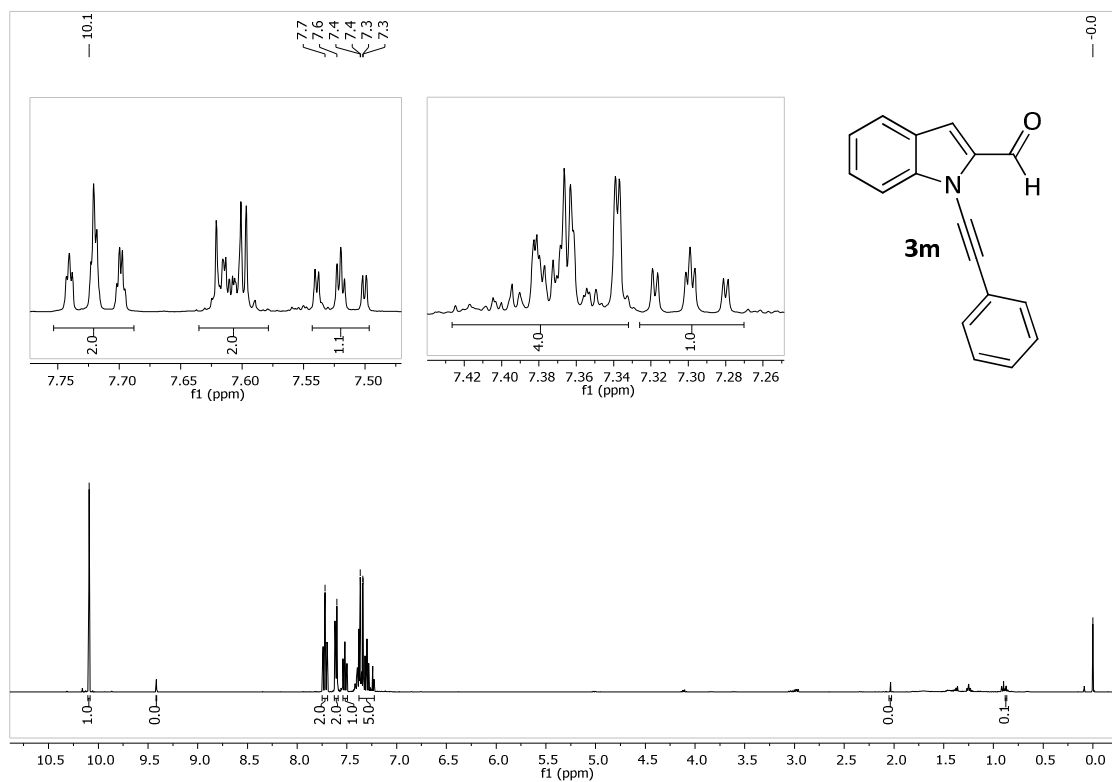
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3h** in CDCl₃.



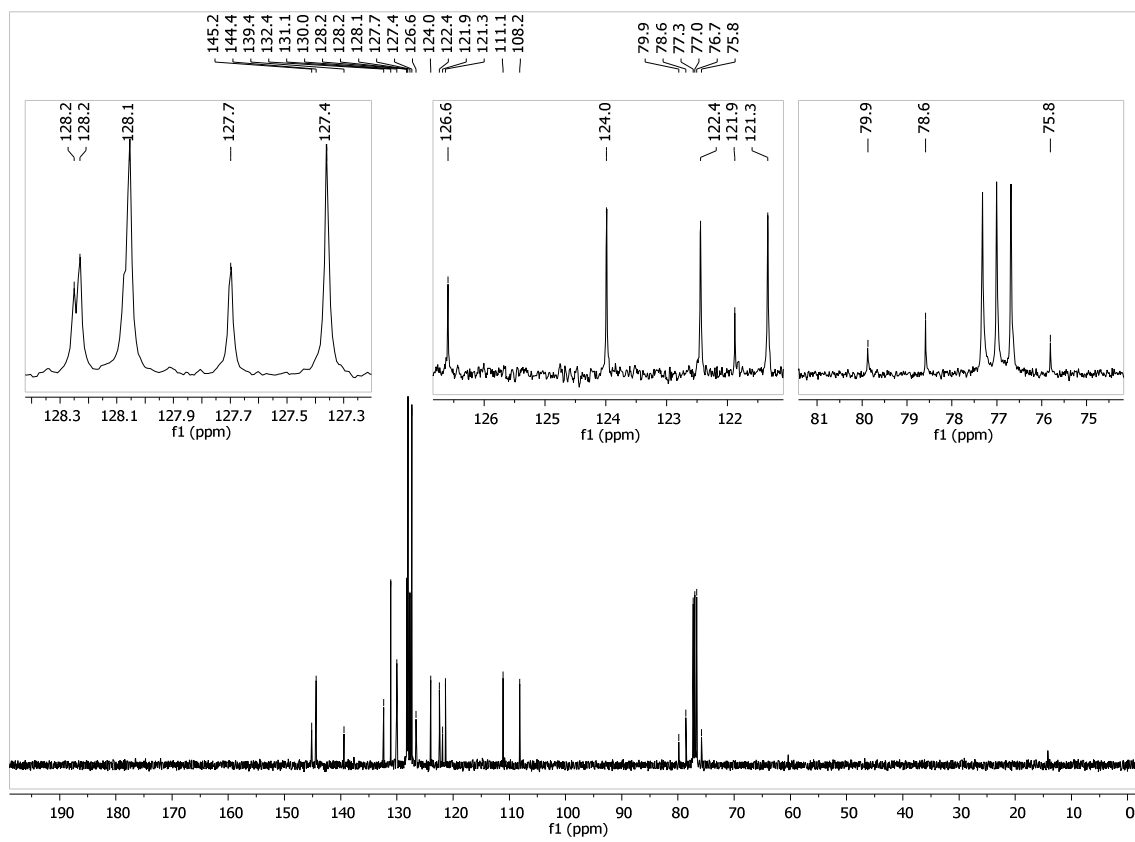
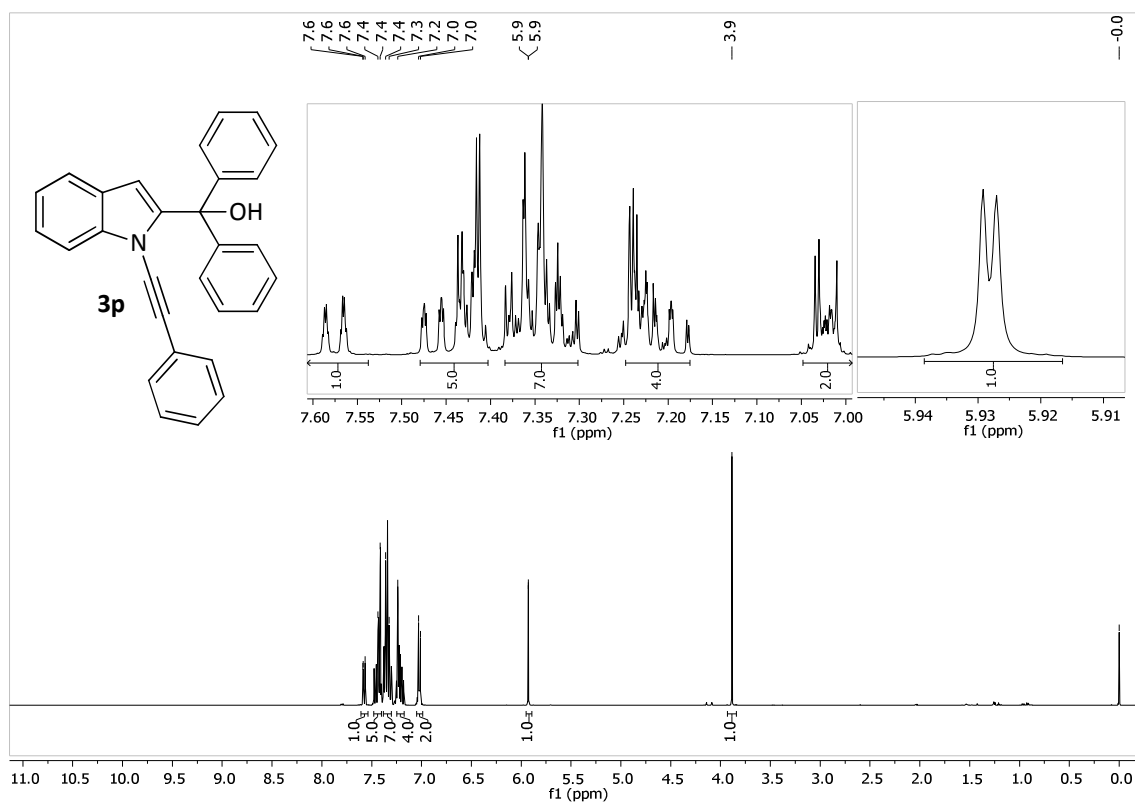
The ^1H (400 MHz) and ^{13}C (100 MHz) spectra of **3j** in CDCl_3 .



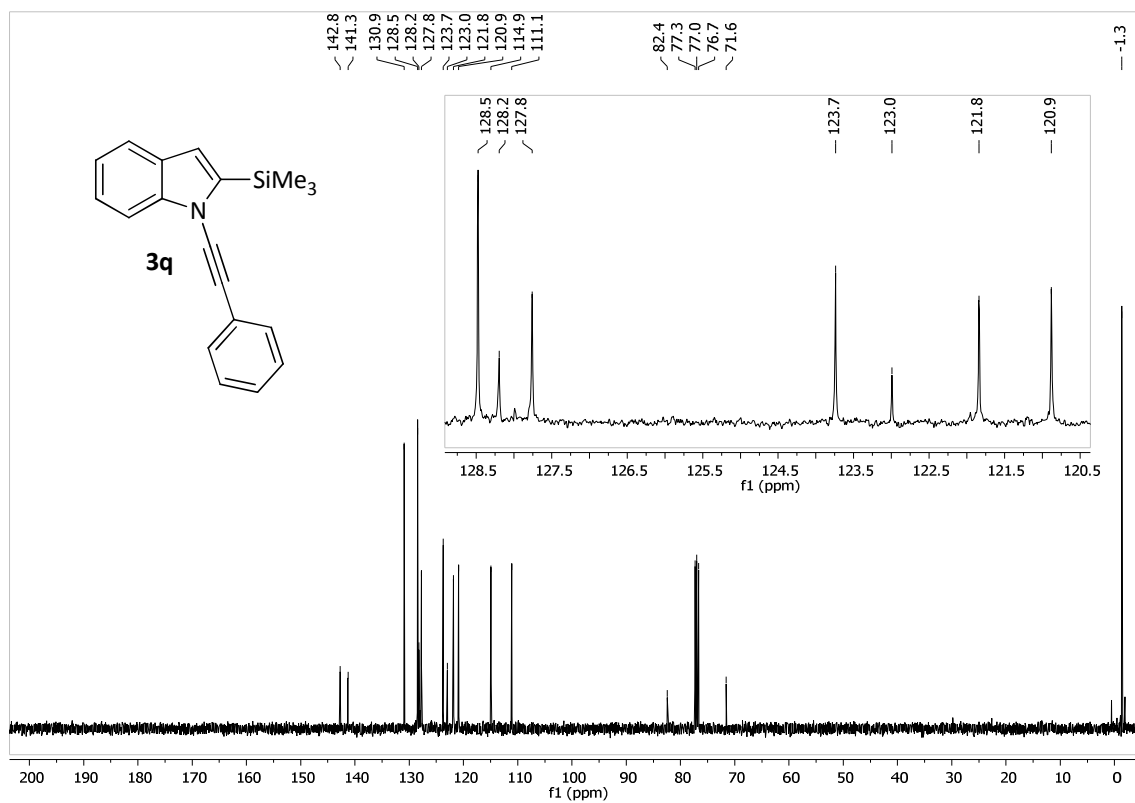
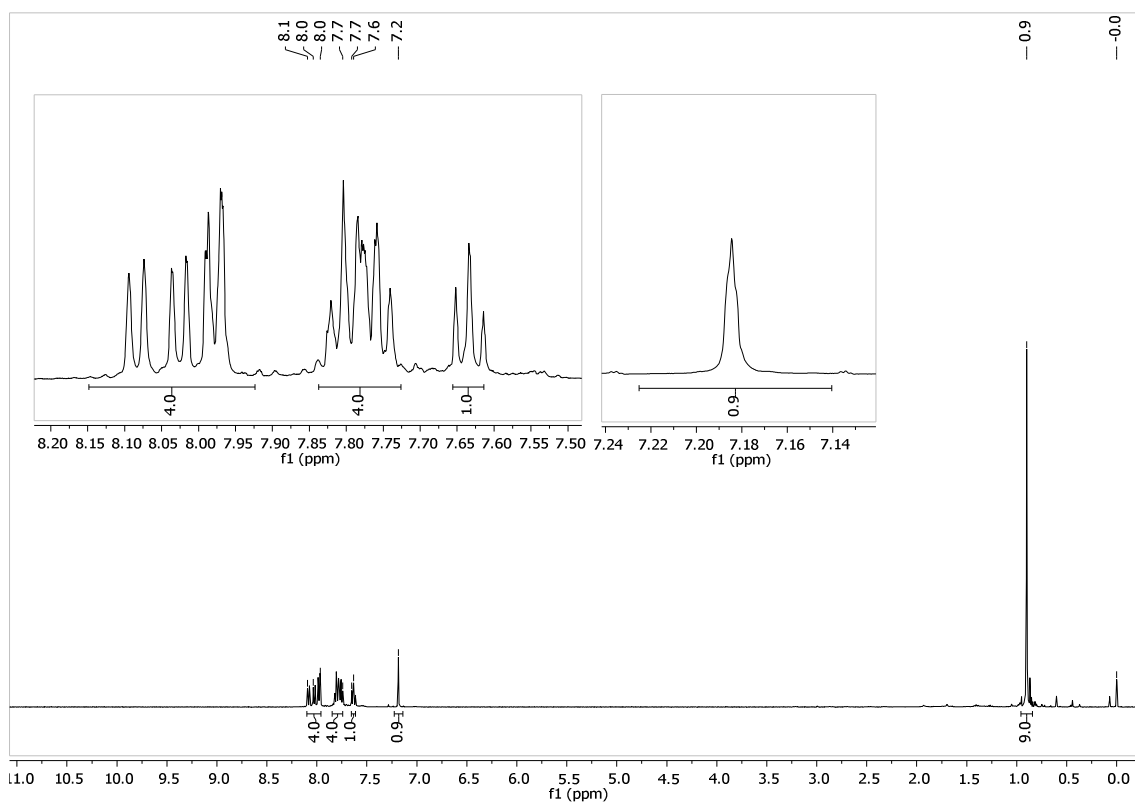
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3I** in CDCl₃.



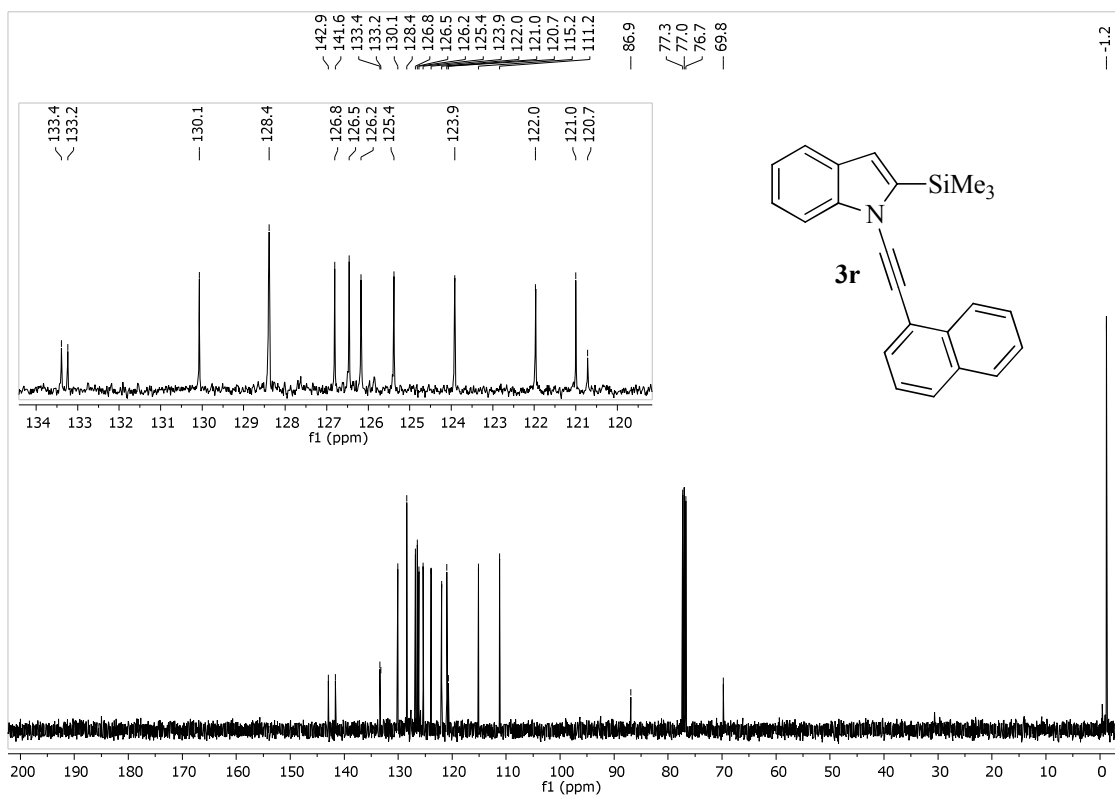
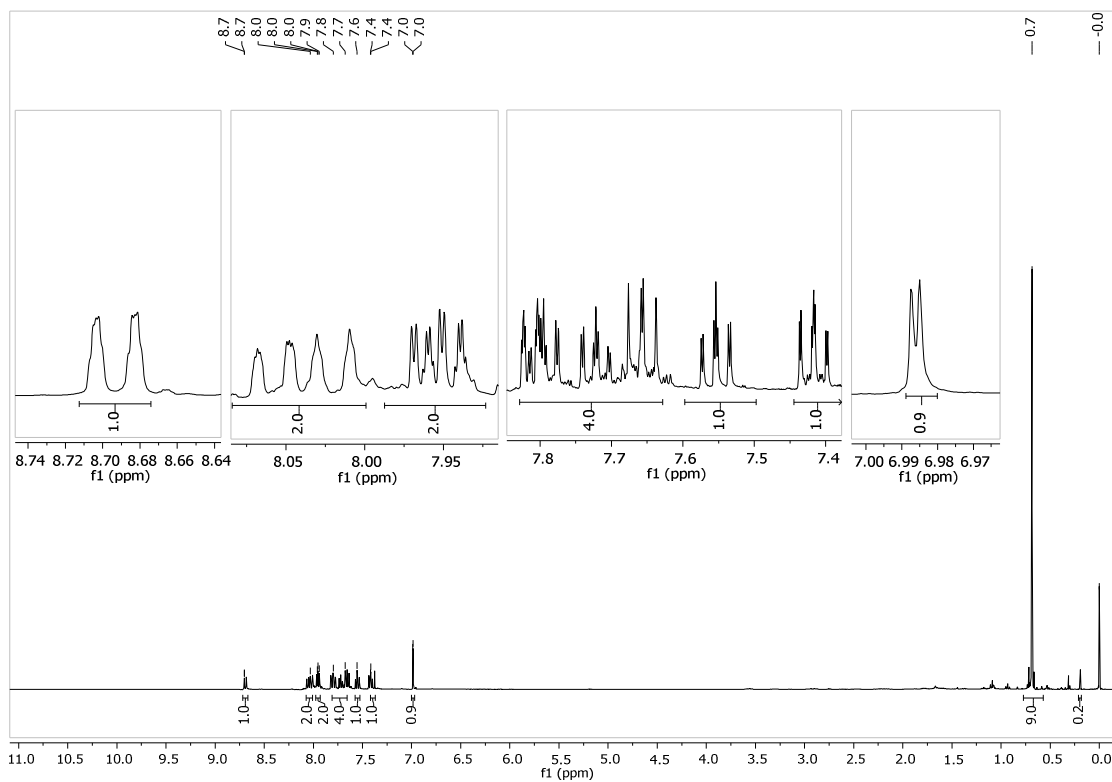
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3m** in CDCl₃.



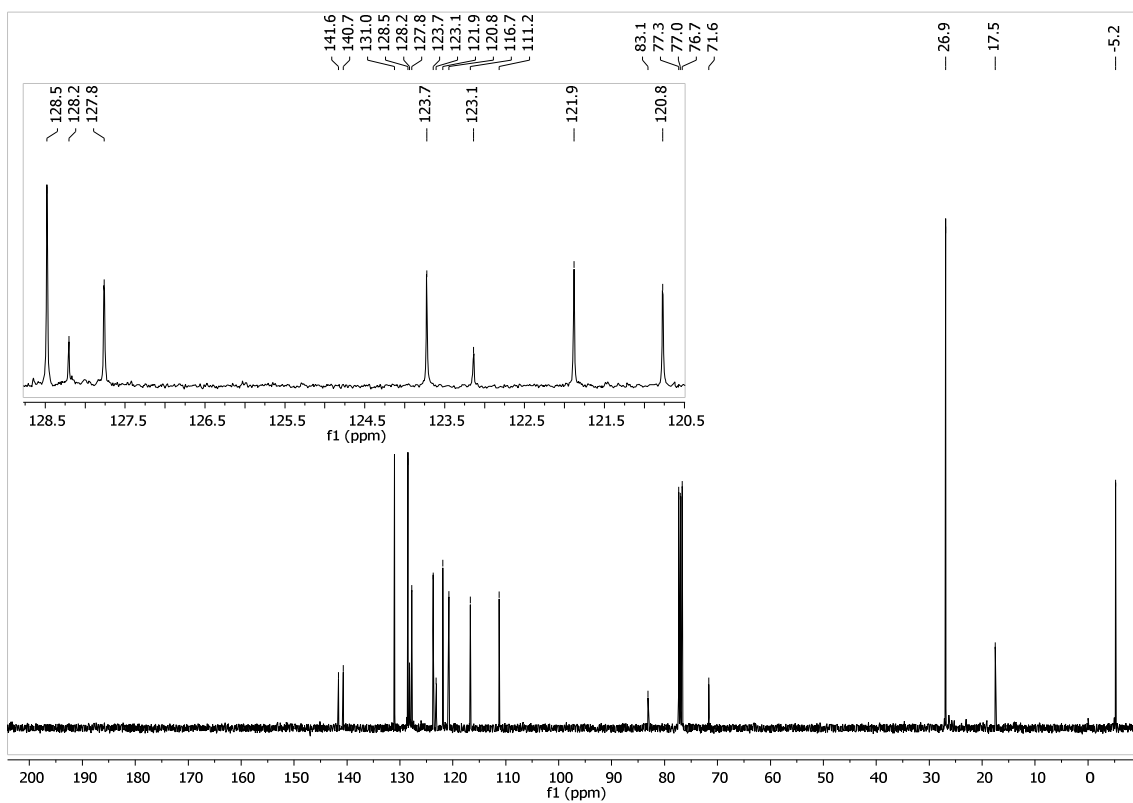
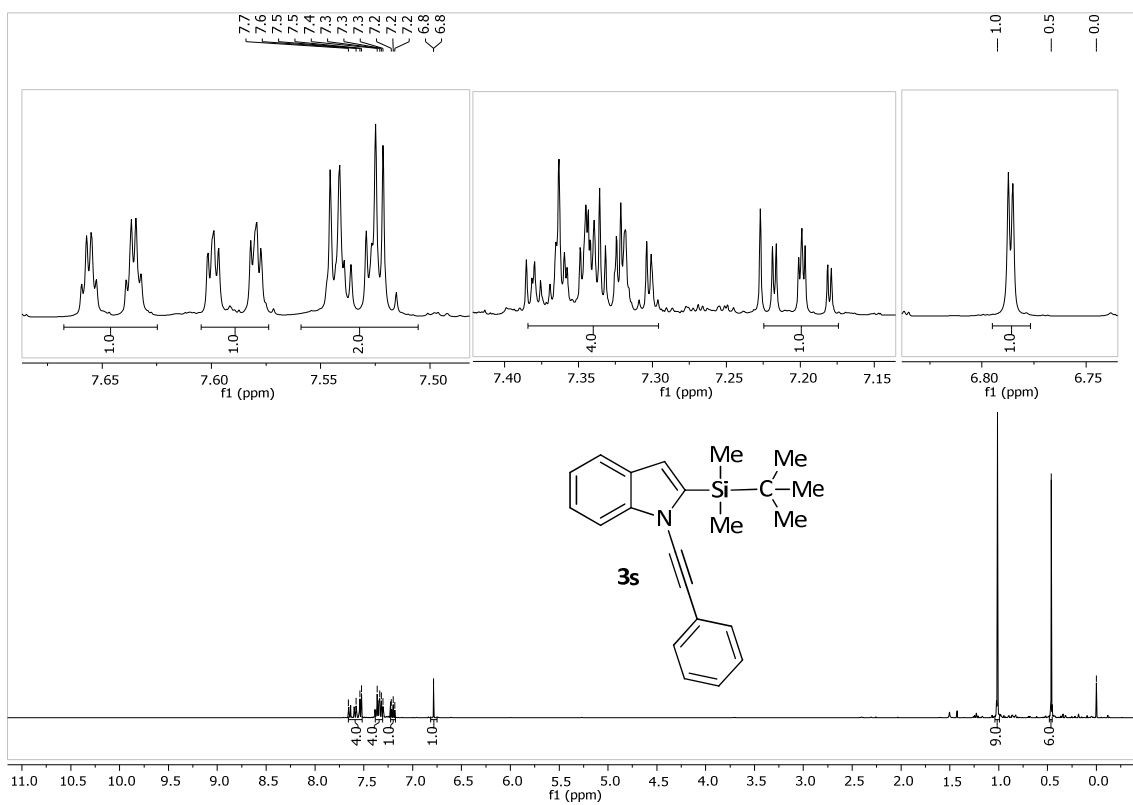
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3p** in CDCl₃.



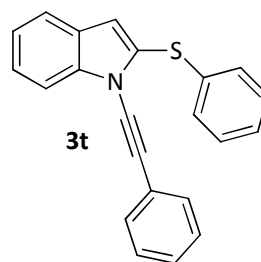
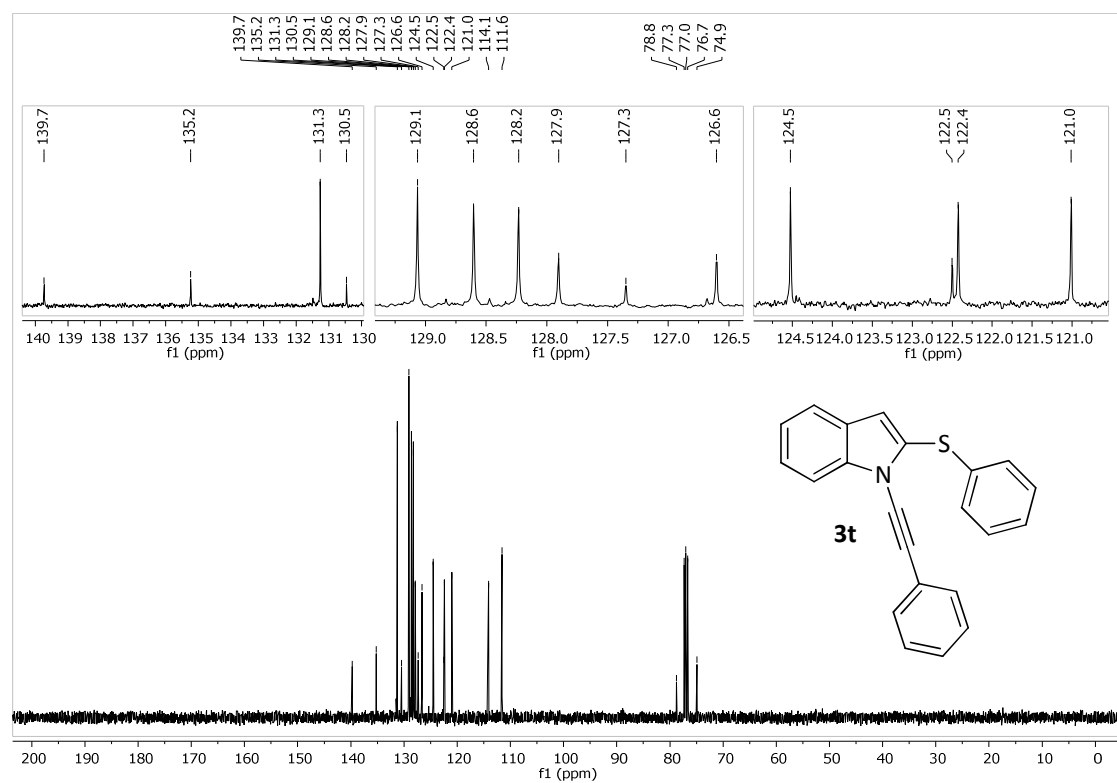
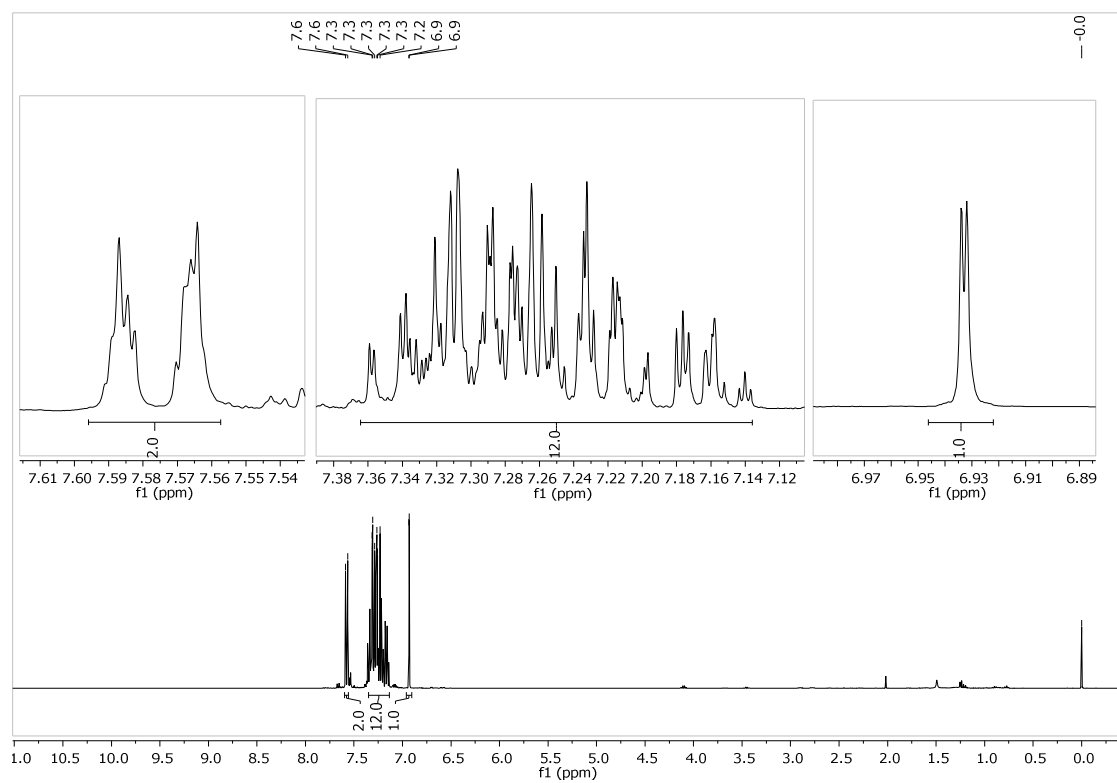
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3q** in CDCl₃.



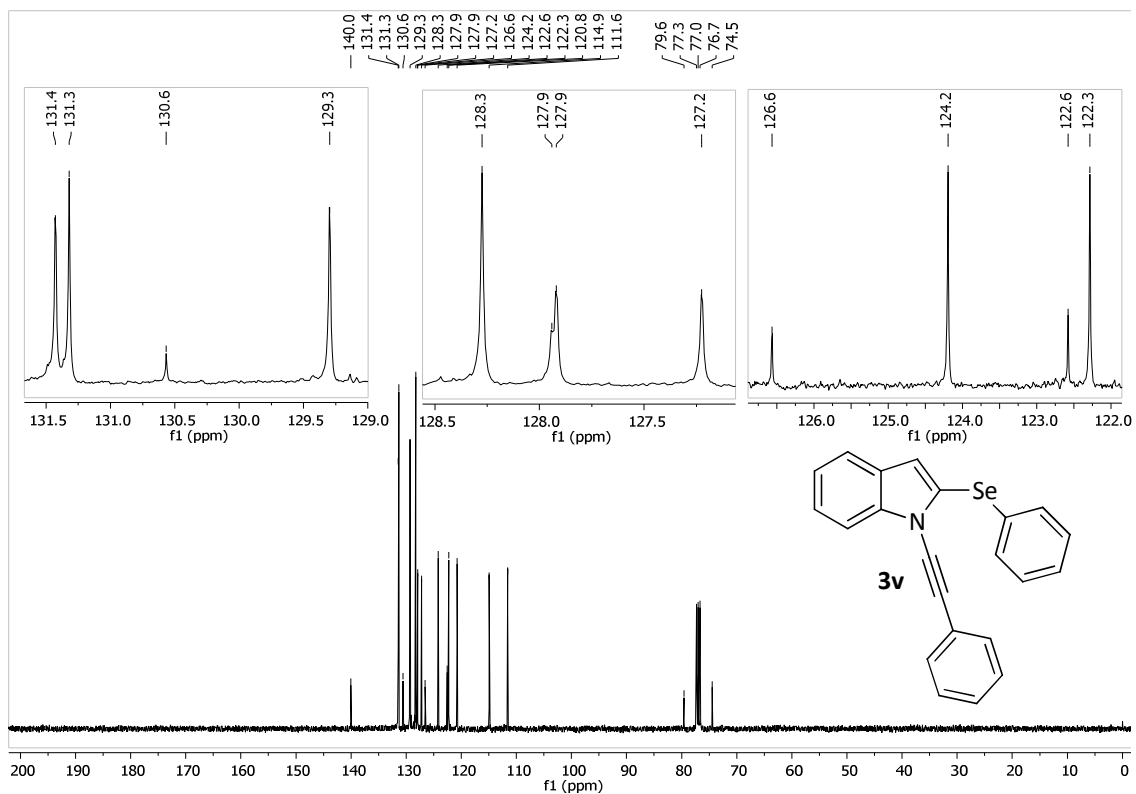
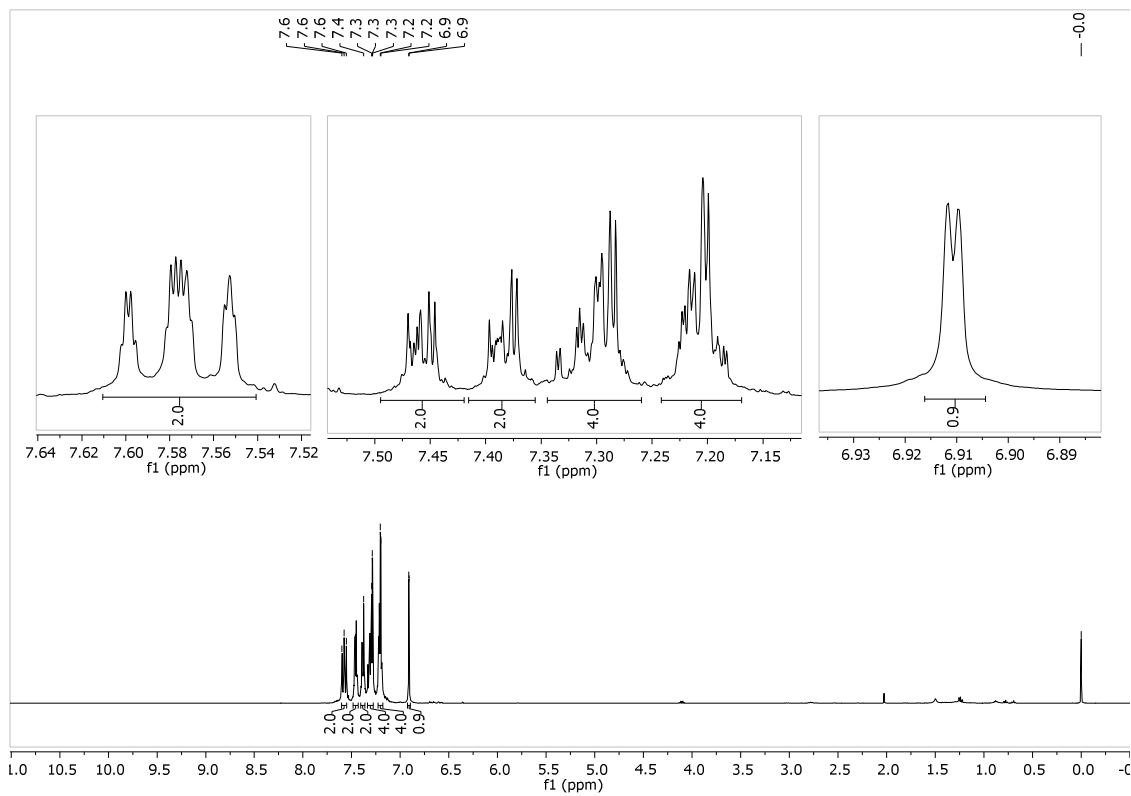
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3r** in CDCl₃.



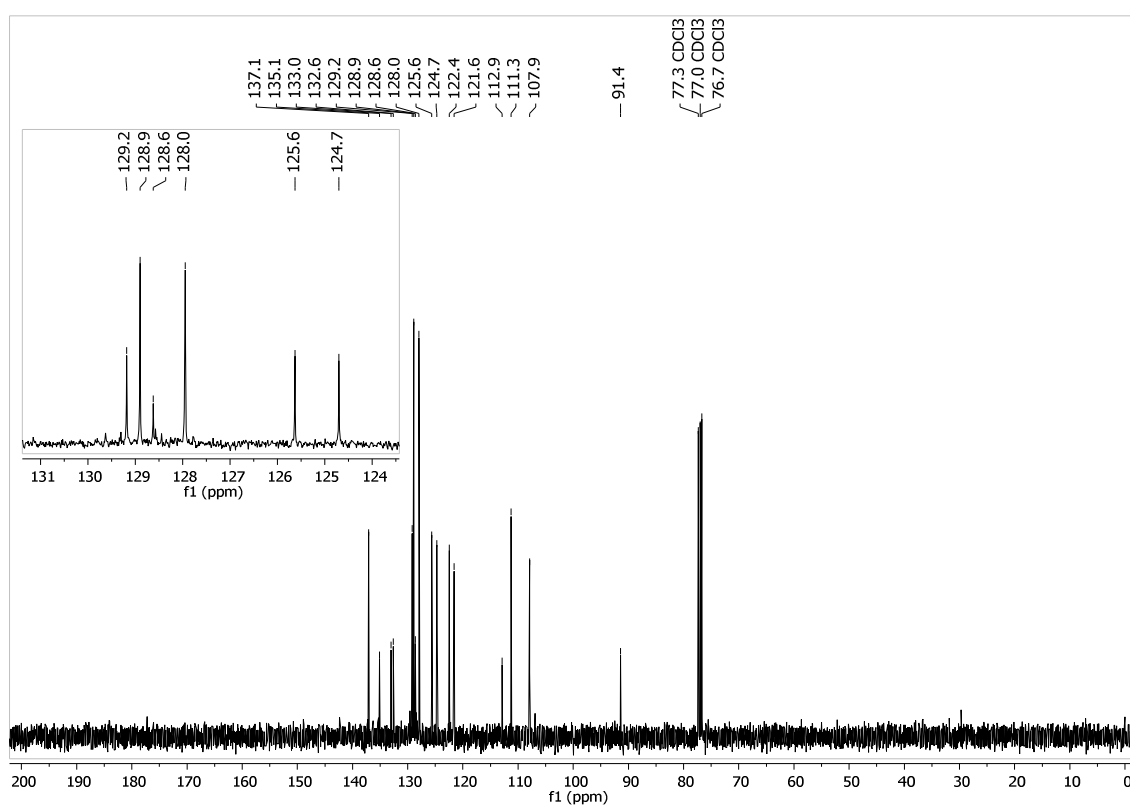
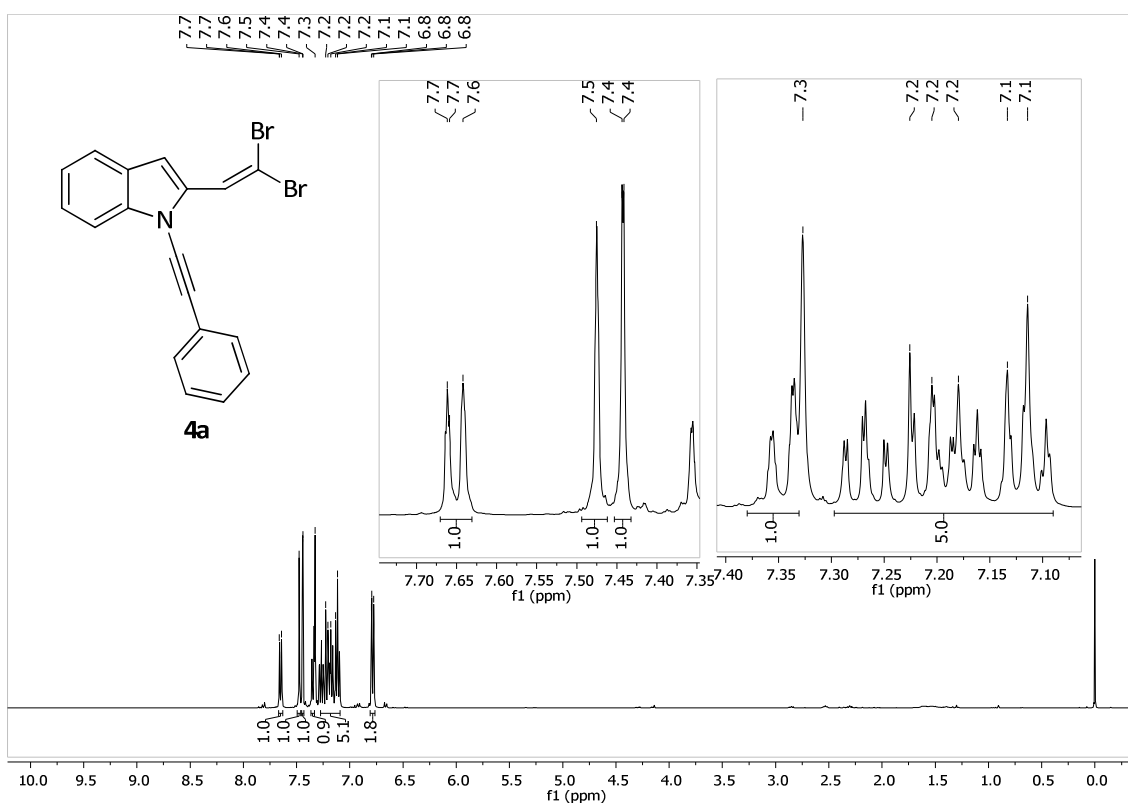
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3s** in CDCl₃.



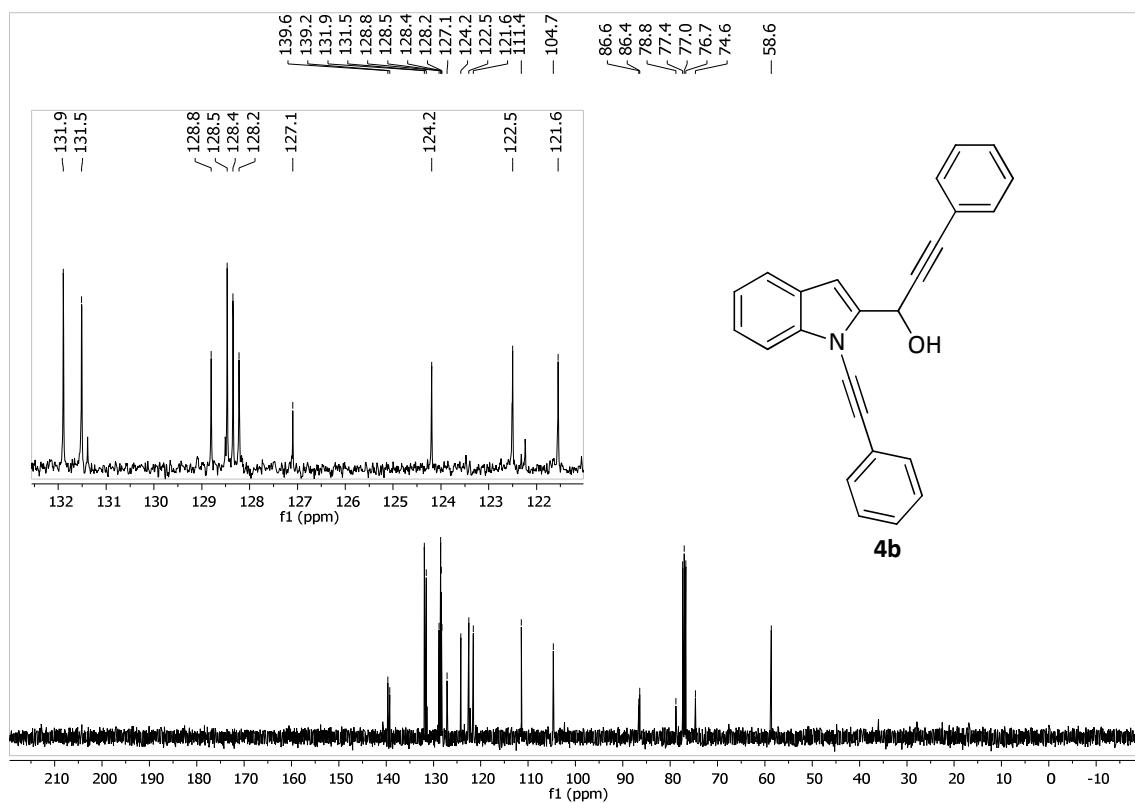
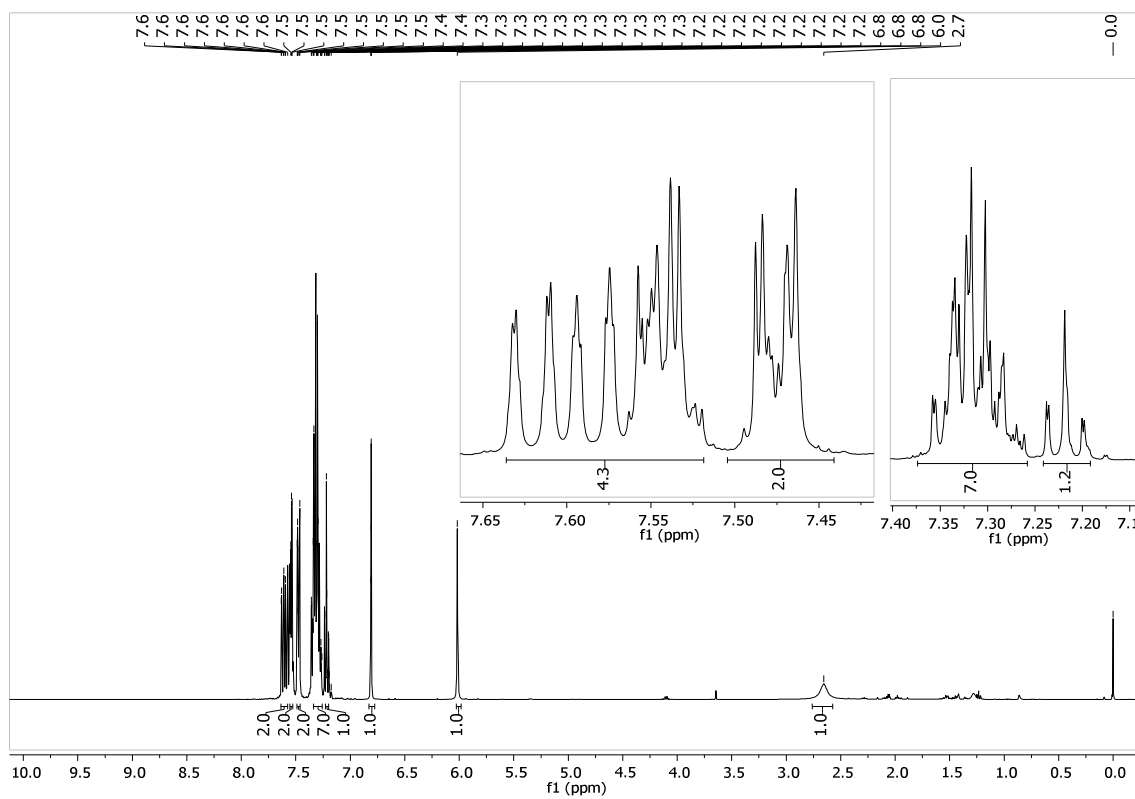
The ^1H (400 MHz) and ^{13}C (100 MHz) spectra of **3t** in CDCl_3 .



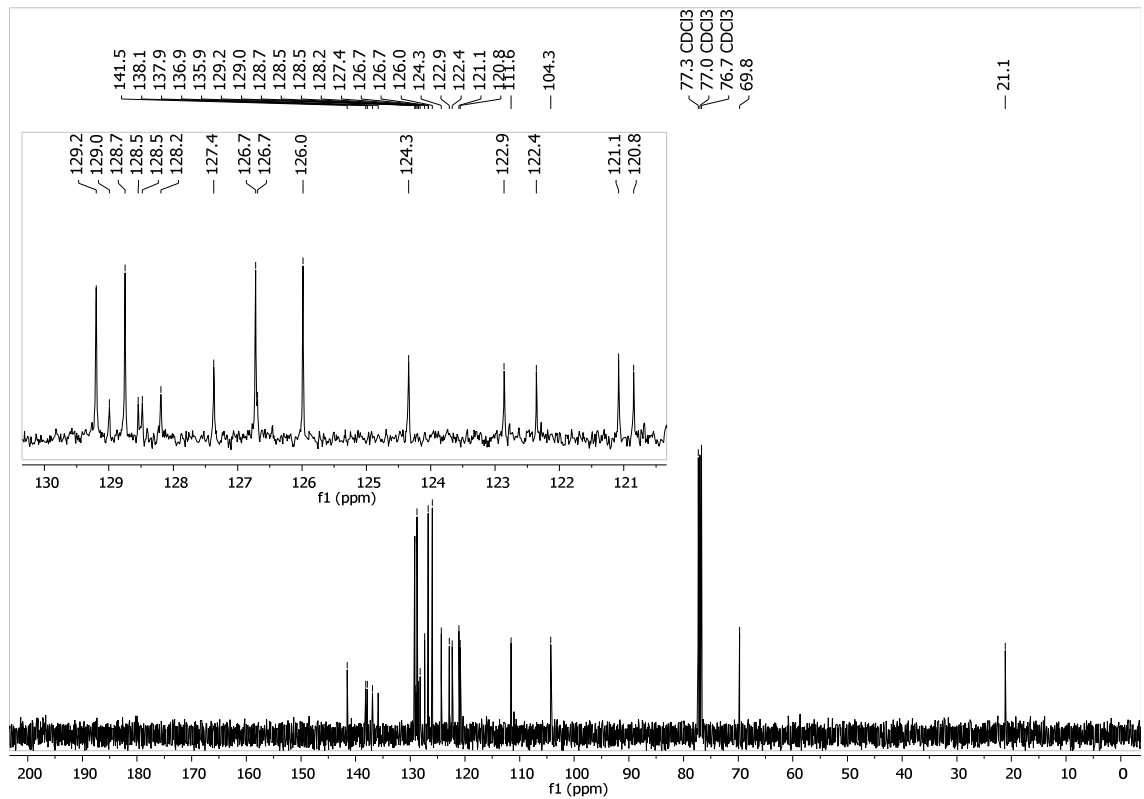
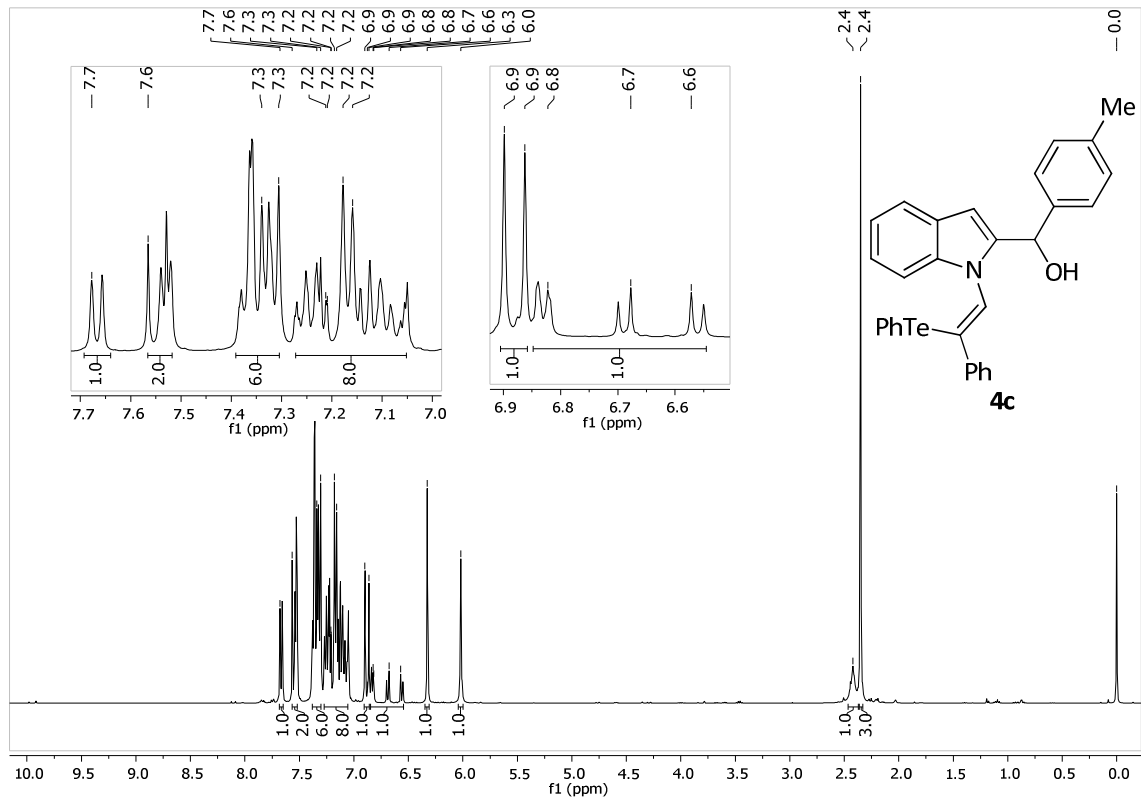
The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **3v** in CDCl₃.



The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **4a** in CDCl₃.



The ^1H (400 MHz) and ^{13}C (100 MHz) spectra of **4b** in CDCl_3 .



The ¹H (400 MHz) and ¹³C (100 MHz) spectra of **4c** in CDCl₃.

