Supporting Information

for

Chemoselective and Stereospecific Iodination of Alkynes using Sulfonium Iodate(I) Salt

Dodla Sivanageswara Rao,[a] Thurpu Raghavender Reddy,[a] and

Sudhir Kashyap,*[a,b]

[a]*Department of Chemistry, Malaviya National Institute of Technology (MNIT), Jaipur-302 017, INDIA.*

[b]*INSPIRE Faculty, Department of Science and Technology (DST) and Assistant Professor, Academy of Scientific and Innovative Research (AcSIR), INDIA.*

** Corresponding author. Tel.: +91-9549657150;*

Email: skashyap.chy@mnit.ac.in, skr.kashyap@gmail.com

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Experimental

General Synthesis Information. Reactions were run in screw capped glass vials (4 mL) stirred with Teflon®-coated magnetic stir bars. Moisture and air-sensitive reactions were performed in flame-dried round bottom flasks, fitted with rubber septa or glass gas adapters, under a positive pressure of nitrogen. Moisture and air-sensitive liquids or solutions were transferred via nitrogen-flushed syringe. Concentration of solvents was accomplished by rotary evaporation using a Büchi rotary evaporator at temperatures between 35 °C and 50 °C. Experiments were monitored by thin layer chromatography (TLC).

Materials. Unless otherwise noted, materials were obtained from commercial suppliers and used without purification. Removal of solvent under reduced pressure refers to distillation with a Büchi rotary evaporator attached to a vacuum pump (~3 mmHg). Products obtained as solids or high boiling oils were dried under vacuum (~1 mmHg).

Chromatography. Analytical TLC was performed using Whatman 250 micron aluminum backed UV F254 precoated silica gel flexible plates. Subsequent to elution, ultraviolet illumination at 254 nm allowed for visualization of UV active materials. Staining with p-anisaldehyde, basic potassium permanganate solution, or Molisch's reagents allowed for further visualization. The retardation factor (Rf) is the ratio of the distance traveled by the compound to the distance traveled by the eluent.

Physical Data. Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on Avance 300, 400 or Avance 500 MHz nuclear magnetic resonance spectrometers. Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) relative to tetramethylsilane (δ 0.0) using the residual solvent signal as an internal standard or tetramethylsilane itself: chloroform-d (δ 7.26, singlet). The number of protons (n) for a given resonance is indicated by nH. IR spectra were recorded on Bruker Alpha spectrometer and mass analyses (ESI) were performed on Finnegan MAT 1020 mass spectrometer operating at 70 eV. High resolution mass analyses were performed on a mass spectrometer using ESI-TOF techniques.

Representative procedure for the synthesis of 1-iodoalkyne (A); A preformed solution of Me_3SI (242 mg, 1.1 mmol, 1.1 equiv.) and $PhI(OAc)_2$ (354 mg, 1.1 mmol, 1.1 equiv.) in

acetonitrile (2 mL) was treated with alkyne **1** (**1a**, 102 mg, 110 uL, 1.0 mmol, 1.0 equiv.) at room temperature. After the completion of reaction, the reaction was diluted with EtOAc (10 mL), quenched with saturated $NaHCO_3$ (5 mL), saturated aqueous sodium thiosulfate (2 mL) and extracted with EtOAc (3 X 30 mL). The combined organic layers were washed with brine solution, dried over anhydrous Na_2SO_4, concentrated in vacuo and purified by silica gel column chromatography to obtain the desired 1-iodoalkynes (**2a-2r**). All the products were fully characterised by ^1H and ^{13}C spectroscopy and MS spectrometry and were in complete agreement with the assigned structure and correlated with literature data.

Representative procedure for the synthesis of *(E)*-1,2-diiodoalkenes (B); A preformed solution of Me_3SI (484 mg, 2.2 mmol, 2.2 equiv.) and $PhI(OAc)_2$ (354 mg, 1.1 mmol, 1.1 equiv.) in water (2 mL) was treated with alkyne **1** (**1a**, 102 mg, 110 uL, 1.0 mmol, 1.0 equiv.) at room temperature. After the completion of reaction, the reaction was diluted with EtOAc (10 mL), quenched with saturated $NaHCO_3$ (5 mL), saturated aqueous sodium thiosulfate (2 mL) and extracted with EtOAc (3 X 30 mL). The combined organic layers were washed with brine solution, dried over anhydrous Na_2SO_4, concentrated in vacuo and purified by silica gel column chromatography to obtain the desired *(E)*-1,2-diiodoalkenes (**3a-3v**). All the products were fully characterised by ^1H and ^{13}C spectroscopy and MS spectrometry and were in complete agreement with the assigned structure and correlated with literature data.

Spectroscopic Characterization data.

Iodoethynylbenzene (2a): Following general procedure **A** using ethynylbenzene (**1a,** 93 mg, 0.912 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (204 mg, 0.893 mmol, 98%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, J = 1.9 Hz, 1H), 7.43- 7.42 (m, 1H), 7.33-7.30 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.30, 128.78, 128.21, 123.36, 94.11, 6.10; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[1]

1-Chloro-2-(iodoethynyl)benzene (2b): Following general procedure **A** using 1-chloro-2-ethynylbenzene (**1b,** 125 mg, 0.919 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (238 mg, 0.909 mmol, 99%). Mp. 101-102 °C; (literature: 100-102 °C). ^1H NMR (400 MHz, $CDCl_3$) δ 7.49-7.45 (m,

1H), 7.38 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.28-7.24 (m, 1H), 7.23 (dd, *J* = 4.7, 1.8 Hz, 1H), 7.21-7.18 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.64, 134.12, 129.71, 129.16, 126.31, 123.17, 90.83, 12.27; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[4]

1-Bromo-4-(iodoethynyl)benzene (2c): Following general procedure **A** using 4-bromo-2-ethynylbenzene (**1c,** 100 mg, 0.552 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (168 mg, 0.546 mmol, 99%). Mp. 88-89 °C; (literature: 89-91 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.29(d, *J* = 8.4 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 133.68, 131.48, 123.13, 122.25, 93.01, 8.02; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [1]

1-Fluoro-4-(iodoethynyl)benzene (2d): Following general procedure **A** using 4-fluoro-2-ethynylbenzene (**1d,** 105 mg, 0.873 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (193 mg, 0.786 mmol, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.37 (m, 2H), 7.03-6.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.99, 161.51, 134.28, 134.20, 115.64, 115.42, 92.97, 5.94; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [1]

1-(iodoethynyl)-3-methoxybenzene (2e): Following general procedure **A** using 1-ethynyl-3-methoxybenzene (**1e,** 104 mg, 0.787 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (201 mg, 0.780 mmol, 99%). Mp. 43-44 °C; (literature: 41-42 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24-7.17 (m, 1H), 7.06-6.99 (m, 1H), 6.96 (dd, *J* = 2.5, 1.4 Hz, 1H), 6.91-6.84 (m, 1H), 3.79 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.04, 129.21, 124.74, 124.18, 116.98, 115.44, 93.98, 55.17, 6.48; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[4]

1-(iodoethynyl)-4-methoxybenzene (2f): Following general procedure **A** using 1-ethynyl-4-methoxybenzene (**1f,** 102 mg, 0.771 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (171 mg, 0.663 mmol,

86%). Mp. 63-64 °C; (literature 61-62 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.9 Hz, 2H), 6.83 (d, *J* = 8.9 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.78, 133.65, 115.37, 113.73, 93.90, 55.16, 4.23; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [1]

1-(iodoethynyl)-3-methylbenzene (2g): Following general procedure **A** using 1-ethynyl-3-methylbenzene (**1g,** 90 mg, 0.775 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (176 mg, 0.729 mmol, 94%). Mp. 47-49 °C; (literature 49-51 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.19 (m, 3H), 7.15 (d, *J* = 6.9 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.84, 132.79, 129.65, 129.29, 128.05, 123.05, 94.24, 21.15, 5.81; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [2]

1-(iodoethynyl)-4-methylbenzene (2h): Following general procedure **A** using 1-ethynyl-4-methylbenzene (**1h,** 92 mg, 0.789 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (168 mg, 0.694 mmol, 88%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 7.3 Hz, 2H), 7.11 (d, *J* = 7.5 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.98, 132.16, 128.95, 120.32, 94.21, 21.50, 4.90; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[4]

1-ethyl-4-(iodoethynyl)benzene (2i): Following general procedure **A** using 1-ethynyl-4-ethylbenzene (**1i,** 93 mg, 0.715 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (169 mg, 0.658 mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.2 Hz, 2H), 7.13 (d, *J* = 8.0 Hz,2H), 2.64 (q, *J* = 7.6 Hz, 1H), 1.22 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.26, 132.26, 127.76, 120.56, 94.25, 28.80, 15.26, 4.84; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [3]

1-(tert-butyl)-4-(iodoethynyl)benzene (2j): Following general procedure **A** using 1-(tert-butyl)-4-ethynylbenzene (**1j,** 88 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (151 mg, 0.532 mmol, 96%). Mp. 90-92 °C; (literature 88-90 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.7 Hz,

2H), 7.32 (d, J = 8.7 Hz, 2H), 1.30 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.99, 131.97, 125.15, 120.32, 94.17, 42.35, 34.71, 31.08, 30.92, 13.18; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[4[

1-(iodoethynyl)-2-(trifluoromethyl)benzene (2k): Following general procedure **A** using 1-ethynyl-2-(trifluoromethyl)benzene (**1k,** 122 mg, 0.718 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (195 mg, 0.661 mmol, 92%). Mp. 62-63 °C; (literature 64-65 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.50 (d, J = 9.1 Hz, 1H), 8.20 (d, J = 7.6 Hz, 1H), 8.17 (d, J = 7.6 Hz, 1H), 8.13 (d, J = 9.1 Hz, 1H), 8.09 -8.04 (m, 3H), 8.00 (dd, J = 12.2, 4.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.90, 131.50, 131.11, 130.92, 130.32, 128.60, 128.42, 127.09, 126.24, 125.74, 125.69, 125.15, 124.24, 124.20, 124.14, 117.72, 93.44, 10.94; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[4]

2-(iodoethynyl)-6-methoxynaphthalene (2l): Following general procedure **A** using 2-ethynyl-6-methoxynaphthalene (**1l,** 100 mg, 0.549 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (159 mg, 0.516 mmol, 94%). Mp. 94-96 °C; (literature 95-96 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.69-7.63 (m, 2H), 7.43 (dd, J = 8.4, 1.6 Hz, 1H), 7.15 (dd, J = 8.9, 2.5 Hz, 1H), 7.08 (d, J = 2.3 Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.52, 134.35, 132.34, 129.34, 129.26, 128.17, 126.74, 119.49, 118.25, 105.75, 94.61, 55.34, 5.20; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [5]

4-(iodoethynyl)pyrene (2m): Following general procedure **A** using 4-ethynylpyrene (**1m,** 100 mg, 0.438 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (139 mg, 0.394 mmol, 90%). Mp. 125-127 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.50 (d, J = 9.1 Hz, 1H), 8.20 (d, J = 7.6 Hz, 1H), 8.17 (d, J = 7.6 Hz, 1H), 8.13 (d, J = 9.1 Hz, 1H), 8.09 -8.04 (m, 3H), 8.00 (dd, J = 12.2, 4.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.90, 131.50, 131.11, 130.92, 130.32, 128.60, 128.42, 127.09, 126.24, 125.74, 125.69, 125.15, 124.24, 124.20, 124.14, 117.72, 93.44, 10.94; IR ($CHCl_3$, cm^{-1}): 3050, 2125, 1690, 1500, 980, 750, 550; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_{18}H_{13}IN^+$: 370.00927; found: 370.00985.

3-(iodoethynyl)pyridine (2n): Following general procedure **A** using 3-ethynylpyridine (**1n,** 100 mg, 0.970 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (182 mg, 0.80 mmol, 82%). Mp. 104-106 °C; ^{1}H NMR (400 MHz,) δ 8.68 (s, 1H), 8.55 (d, *J* = 4.9 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (101 MHz,) δ 152.66, 148.58, 139.55, 123.07, 120.74, 90.52, 11.66; IR ($CHCl_3$, cm^{-1}): 3057, 2880, 2100, 1630, 1450, 720, 570; HRMS (ESI-TOF) m/z $[M + H]^+$ calcd. for $C_7H_5IN^+$: 229.94667; found: 229.94498.

1-iododec-1-yne (2o): Following general procedure **A** using dec-1-yne (**1o,** 76.6 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (117 mg, 0.444 mmol, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.35 (t, *J* = 7.1 Hz, 2H), 1.50 (dt, *J* = 7.5, 7.0 Hz, 2H), 1.40-1.26 (m, 10H), 0.88 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 94.83, 31.80, 29.69, 29.13, 29.02, 28.77, 28.47, 22.64, 20.80, 14.10, -7.65; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [7]

(iodoethynyl)cyclopropane (2p): Following general procedure **A** using ethynylcyclopropane (**1p,** 80 mg, 1.207 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (186 mg, 1.050 mmol, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (tdd, *J* = 6.4, 5.5, 3.2 Hz, 1H), 1.02-0.95 (m, 2H), 0.90-0.84 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 124.35, 28.74, 16.95, 12.80; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [5]

(iodoethynyl)cyclopentane (2q): Following general procedure **A** using ethynylcyclopentane (**1q,** 81 mg, 0.863 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (169 mg, 0.768 mmol, 89%). ^{1}H NMR (400 MHz,) δ 2.52 (ddd, *J* = 12.7, 8.9, 3.5 Hz, 1H), 1.77 (dd, *J* = 8.4, 5.6 Hz, 2H), 1.68 (ddd, *J* = 8.8, 8.1, 4.9 Hz, 2H), 1.54-1.38 (m, 2H), 1.33-1.18 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 131.06, 56.48, 34.36, 29.69, 25.28; IR ($CHCl_3$, cm^{-1}): 3250, 2810, 2150, 1700, 620, 520; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_7H_{13}IN^+$: 238.00927; found: 238.00959.

(iodoethynyl)cyclohexane (2r): Following general procedure **A** using ethynylcyclohexane (**1r,** 83 mg, 0.766 mmol) and purified by silica gel column chromatography, eluting with hexanes

afforded the title compound as pale yellow oil (154 mg, 0.659 mmol, 86%). ^{1}H NMR (400 MHz,) δ 2.52 (ddd, *J* = 12.7, 8.9, 3.5 Hz, 1H), 1.77 (ddd, *J* = 8.4, 8.1, 5.6 Hz, 2H), 1.68 (ddd, *J* = 8.8, 8.1, 4.9 Hz, 2H), 1.54-1.36 (m, 3H), 1.35-1.17 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 98.92, 32.39, 31.14, 25.71, 25.69, 24.70, -7.13; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [7]

(E)-(1,2-diiodovinyl)benzene (3a): Following general procedure **B** using ethynylbenzene (**1a,** 93 mg, 0.912 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (308 mg, 0.866 mmol, 95%). Mp. 71-73 °C; (literature 72-74 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.32 (m, 5H), 7.26 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.97, 128.89, 128.44, 128.36, 96.19, 80.83; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-chloro-2-(1,2-diiodovinyl)benzene (3b): Following general procedure **B** using 1-chloro-2-ethynylbenzene (**1b,** 125 mg, 0.919 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as light brown oil (329 mg, 0.845 mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.35 (m, 1H), 7.33 (s, 1H), 7.31-7.25 (m, 2H), 7.21-7.17 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.76, 131.41, 130.06, 130.04, 129.37, 127.12, 92.24, 84.93; IR ($CHCl_3$, cm^{-1}): 3016, 1435, 1215, 744; HRMS (ESI-TOF) m/z $[M + H]^+$ calcd. for $C_8H_6ClI_2^+$: 390.82417; found: 390.82559.

(E)-1-bromo-4-(1,2-diiodovinyl)benzene (3c): Following general procedure **B** using 4-bromo-2-ethynylbenzene (**1c,** 100 mg, 0.552 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (225 mg, 0.52 mmol, 94%). Mp. 63-64 °C; (literature 63-64 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.7 Hz, 1H), 7.28 (s, 1H), 7.22 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.79, 131.60, 130.09, 123.03, 94.57, 81.78; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(1,2-diiodovinyl)-4-fluorobenzene (3d): Following general procedure **B** using 4-fluoro-2-ethynylbenzene (**1d,** 105 mg, 0.873 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale orange oil (293 mg, 0.786 mmol 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 8.2, 5.5 Hz, 1H), 7.25 (s, 1H), 7.04 (dd, *J* = 8.2, 0.5

Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.58, 161.09, 138.94, 138.91, 130.55, 130.46, 115.56, 115.34, 94.91, 81.61; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(1,2-diiodovinyl)-3-methoxybenzene (3e): Following general procedure **B** using 1-ethynyl-3-methoxybenzene (**1e,** 104 mg, 0.787 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (301 mg, 0.78 mmol, 99%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (s, 1H), 7.25 (s, 1H), 6.97-6.92 (m, 1H), 6.89-6.85 (m, 2H), 3.83 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.14, 144.15, 129.45, 120.76, 114.74, 113.75, 95.84, 80.83, 55.33; IR ($CHCl_3$, cm^{-1}): 2932, 1576, 1260, 758; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_9H_{12}I_2NO^+$: 403.90028; found: 403.90212.

(E)-1-(1,2-diiodovinyl)-4-methoxybenzene (3f): Following general procedure **B** using 1-ethynyl-4-methoxybenzene (**1f,** 102 mg, 0.771 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (295 mg, 0.764 mmol, 99%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.9 Hz, 1H), 7.19 (s, 1H), 6.87 (d, *J* = 8.8 Hz, 1H), 3.83 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.70, 135.14, 130.17, 113.60, 96.59, 79.87, 55.29; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(1,2-diiodovinyl)-3-methylbenzene (3g): Following general procedure **B** using 1-ethynyl-3-methylbenzene (**1g,** 90 mg, 0.775 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (252 mg, 0.682 mmol, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 7.3 Hz, 1H), 7.22 (s, 1H), 7.16 -7.10 (m, 1H), 2.36 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.84, 138.07, 129.68, 128.91, 128.21, 125.44, 96.47, 80.55, 21.36; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(1,2-diiodovinyl)-4-methylbenzene (3h): Following general procedure **B** using 1-ethynyl-4-methylbenzene (**1h,** 92 mg, 0.79 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (236 mg, 0.639 mmol, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.2 Hz, 1H), 7.22 (s, 1H), 7.16 (d, *J* = 8.0 Hz, 1H), 2.36 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 140.13, 139.05, 129.05, 128.44, 96.57, 80.17,

77.25, 77.00, 77.00, 76.75, 21.42; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(1,2-diiodovinyl)-4-ethylbenzene (3i): Following general procedure **A** using 1-ethynyl-4-ethylbenzene (**1i,** 93 mg, 0.715 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale orange oil (236 mg, 0.615 mmol, 86%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.2 Hz, 1H), 7.22 (s, 1H), 7.19 (d, *J* = 8.4 Hz, 1H), 2.66 (q, *J* = 7.6 Hz, 1H), 1.26 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.25, 140.24, 128.54, 127.82, 96.66, 80.05, 28.68, 15.12; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [6]

(E)-1-(tert-butyl)-4-(1,2-diiodovinyl)benzene (3j): Following general procedure **B** using 1-(tert-butyl)-4-ethynylbenzene (**1j,** 88 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow solid (187 mg, 0.455 mmol, 82%). Mp. 63-64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.6 Hz, 2H), 7.22 (s, 1H), 1.33 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.07, 131.83, 128.35, 125.23, 96.76, 79.82, 34.78, 31.19; IR ($CHCl_3$, cm^{-1}): 3066, 2954, 2926, 2858, 1607, 1498, 1458, 1150, 829, 777, 597; HRMS (ESI-TOF) m/z $[M + Na]^+$ calcd. for $C_{12}H_{14}I_2Na^+$: 434.90826; found: 434.90542.

(E)-1-(1,2-diiodovinyl)-2-(trifluoromethyl)benzene (3k): Following general procedure **B** using 1-ethynyl-2-(trifluoromethyl)benzene (**1k,** 122 mg, 0.718 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as light brown oil (289 mg, 0.68 mmol, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.9 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 1H), 7.37 (s, 1H), 7.24 (d, *J* = 7.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.26, 132.44, 129.78, 129.01, 126.84, 126.79, 90.40, 85.57; IR ($CHCl_3$, cm^{-1}): 3052, 3002, 2941, 2843, 2165, 1626, 1598, 1480, 1388, 1228, 1172, 1161, 1026, 900, 550, 750; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_9H_9I_2N^+$: 441.87764; found: 441.87903.

(E)-2-(1,2-diiodovinyl)-6-methoxynaphthalene (3l): Following general procedure **B** using 2-ethynyl-6-methoxynaphthalene (**1l,** 100 mg, 0.549 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as white solid (234 mg, 0.538 mmol, 98%). Mp. 117-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 1.5 Hz, 1H), 7.75 (d, *J*

= 9.0 Hz, 1H), 7.72 (d, *J* = 8.6 Hz, 1H), 7.41 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.30 (s, 1H), 7.17 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.13 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.60, 137.99, 134.51, 129.80, 128.09, 127.92, 126.95, 126.51, 119.53, 105.82, 96.78, 80.40, 55.38; IR ($CHCl_3$, cm^{-1}): 3075, 2835, 2300, 2024, 1595, 1490, 1462, 1433, 1254, 1114, 1047, 1023, 751, 550; HRMS (ESI-TOF) m/z $[M + Na]^+$ calcd. for $C_{13}H_{10}I_2Na^+$: 458.87178; found: 458.87929.

(E)-1-(1,2-diiodovinyl)pyrene (3m): Following general procedure **B** using 4-ethynylpyrene (**1m,** 100 mg, 0.438 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (194 mg, 0.403 mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29-8.22 (m, 3H), 8.18 (d, *J* = 7.9 Hz, 2H), 8.14 (d, *J* = 9.3 Hz, 1H), 8.06 (d, *J* = 9.3 Hz, 1H), 8.02 (d, *J* = 7.6 Hz, 1H), 7.88 (d, *J* = 7.9 Hz, 1H), 7.66 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.88, 131.62, 131.30, 131.10, 129.45, 128.20, 128.20, 127.38, 126.47, 125.52, 125.37, 125.09, 124.90, 124.70, 124.37, 95.14, 84.70; IR ($CHCl_3$, cm^{-1}): 3050, 2250, 1690, 1500, 980, 750, 550 HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_{18}H_{14}I_2N^+$: 497.92156; found: 497.92092.

(E)-3-(1,2-diiodovinyl)pyridine (3n): Following general procedure **B** using 3-ethynylpyridine (**1n,** 100 mg, 0.970 mmol) and purified by silica gel column chromatography, eluting with 50:1 hexanes/EtOAc afforded the inseparable mixture of title compound as pale yellow solid (221 mg, 0.621 mmol, 64%) along with **2n**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (s, 1H), 8.55-8.53 (m, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.37 (s, 1H), 7.35-7.27 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.32, 148.06, 136.17, 134.93, 123.27, 107.85, 83.23; IR ($CHCl_3$, cm^{-1}): 3057, 2880, 2100, 1630, 1450, 720, 570; HRMS (ESI-TOF) m/z $[M + H]^+$ calcd. for $C_7H_6I_2N^+$: 357.85841; found: 357.85508.

(E)-1,2-diiododec-1-ene (3o): Following general procedure **B** using dec-1-yne (**1o,** 77 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (178 mg, 0.455 mmol, 82%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.80 (s, 1H), 2.53-2.47 (m, 2H), 1.58-1.49 (m, 4H), 1.36-1.24 (m, 6H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 104.47, 78.95, 44.61, 31.81, 29.36, 29.15, 28.14, 28.12, 22.65, 14.15; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature. [8]

(E)-(1,2-diiodovinyl)cyclopropane (3p): Following general procedure **B** using ethynylcyclopropane (**1p,** 80 mg, 1.207 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (324 mg, 1.014 mmol, 84%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (s, 1H), 1.50 (ttd, *J* = 7.5, 5.2, 0.8 Hz, 1H), 0.82 (dqd, *J* = 3.9, 2.0, 0.7 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 109.78, 23.07, 9.51; IR ($CHCl_3$, cm^{-1}): 2953, 1216, 749; HRMS (ESI-TOF) m/z $[M + Na]^+$ calcd. for $C_5H_6I_2Na^+$: 342.84566; found: 342.84417.

(E)-(1,2-diiodovinyl)cyclopentane (3q): Following general procedure **B** using ethynylcyclopentane (**1q,** 81 mg, 0.863 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (276 mg, 0.794 mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79 (s, 1H), 2.62-2.45 (m, 1H), 1.82-1.71 (m, 4H), 1.69-1.57 (m, 2H), 1.48-1.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 115.46, 77.32, 50.34, 33.11, 25.53; IR ($CHCl_3$, cm^{-1}): 3075, 1421, 951, 748; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_7H_{14}I_2N^+$: 365.92156; found: 365.92247.

(E)-(1,2-diiodovinyl)cyclohexane (3r): Following general procedure **B** using ethynylcyclohexane (**1r,** 83 mg, 0.767 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (249 mg, 0.69 mmol, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 (s, 1H), 2.08 (tt, *J* = 10.8, 3.5 Hz, 1H), 1.80 (ddd, *J* = 10.0, 4.8, 2.0 Hz, 1H), 1.74-1.66 (m, 1H), 1.62-1.52 (m, 1H), 1.50-1.11 (m, 7H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 114.37, 76.42, 48.78, 32.29, 25.46, 25.20; IR ($CHCl_3$, cm^{-1}): 2925, 1729, 772; HRMS (ESI-TOF) m/z $[M + NH_4]^+$ calcd. for $C_8H_{16}I_2N^+$: 379.93721; found: 379.93624; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[16f]

(E)-(1,2-diiodoprop-1-en-1-yl)benzene (3s): Following general procedure **B** using prop-1-yn-1-ylbenzene (**1s,** 93 mg, 0.80 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (236 mg, 0.640 mmol, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.32 (m, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.26 (s, 1H), 7.22 (dd, *J* = 8.2, 1.4 Hz, 1H), 2.80 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.09, 128.43, 128.38, 128.22,

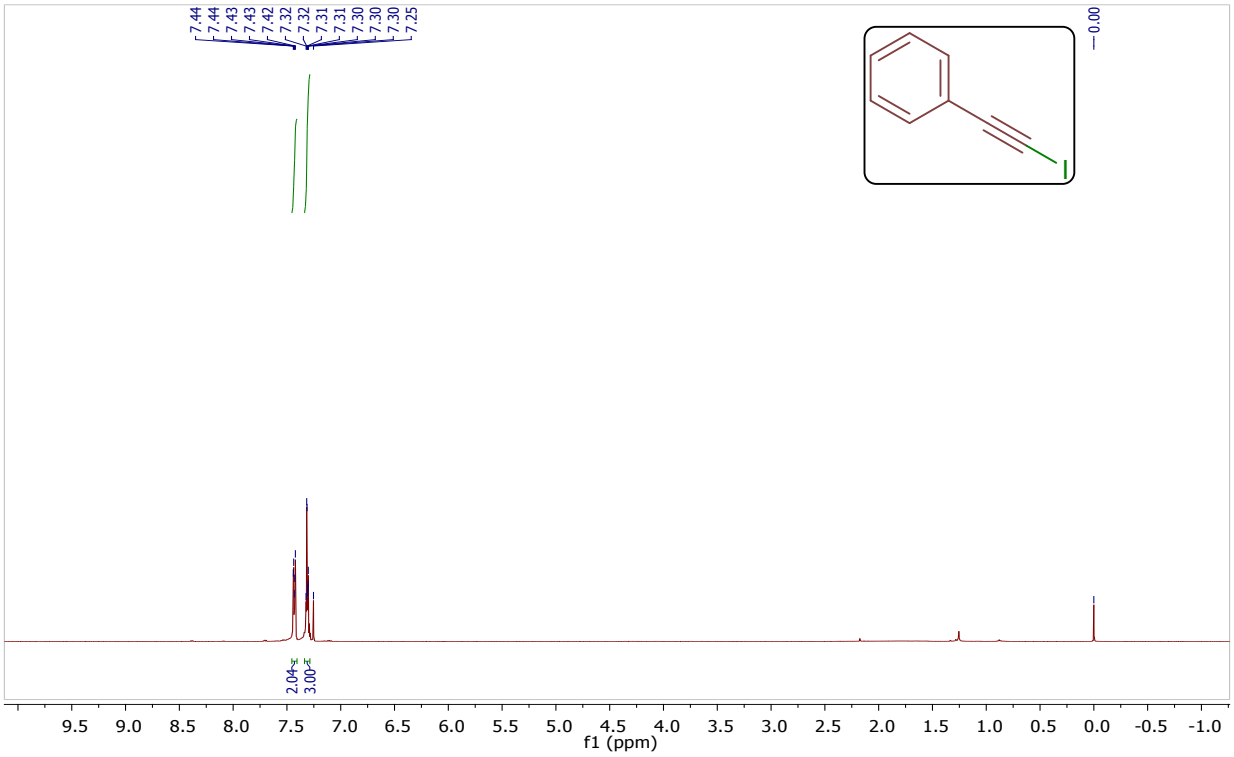
96.30, 95.47, 40.18; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[18b]

(E)-(1,2-diiodobut-1-en-1-yl)benzene (3t): Following general procedure **B** using but-1-yn-1-ylbenzene (**1t,** 92 mg, 0.704 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (243 mg, 0.634 mmol, 90%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (dd, *J* = 10.2, 4.6 Hz, 2H), 7.30-7.26 (m, 1H), 7.22-7.18 (m, 1H), 2.88 (q, *J* = 7.4 Hz, 2H), 1.18 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.96, 128.43, 128.34, 128.13, 106.49, 93.66, 44.83, 12.92; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[18b]

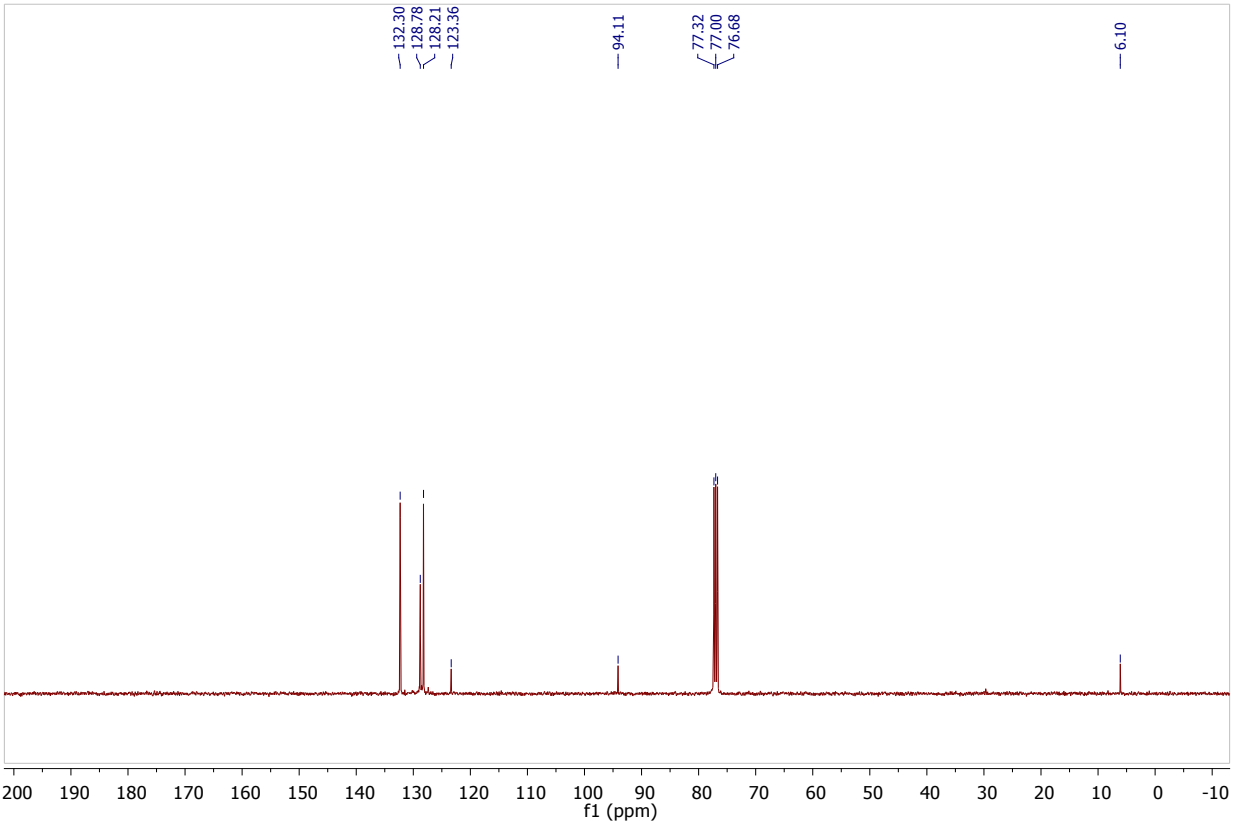
(E)-1,2-diiodo-1,2-diphenylethene (3u): Following general procedure **B** using 1,2-diphenylethyne (**1u,** 100 mg, 0.561 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as white solid (204 mg, 0.472 mmol, 84%). Mp. 149-151 °C; (literature 148-150 °C). ^{1}H NMR (400 MHz,) δ 7.56 -.751 (m, 3H), 7.40-7.31 (m, 7H); ^{13}C NMR (101 MHz,) δ 147.60, 131.59, 128.54, 128.40, 128.33, 128.24, 123.24, 89.34; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[18b]

(E)-3,4-diiodohex-3-ene (3v): Following general procedure **B** using hex-3-yne (**1v,** 72 mg, 0.881 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale yellow oil (266 mg, 0.793 mmol, 90%). Mp. 149-151 °C; (literature 148-150 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.70 (q, *J* = 7.3 Hz, 2H), 1.05 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 102.46, 77.32, 77.00, 77.00, 76.68, 45.05, 12.69; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.[16b]

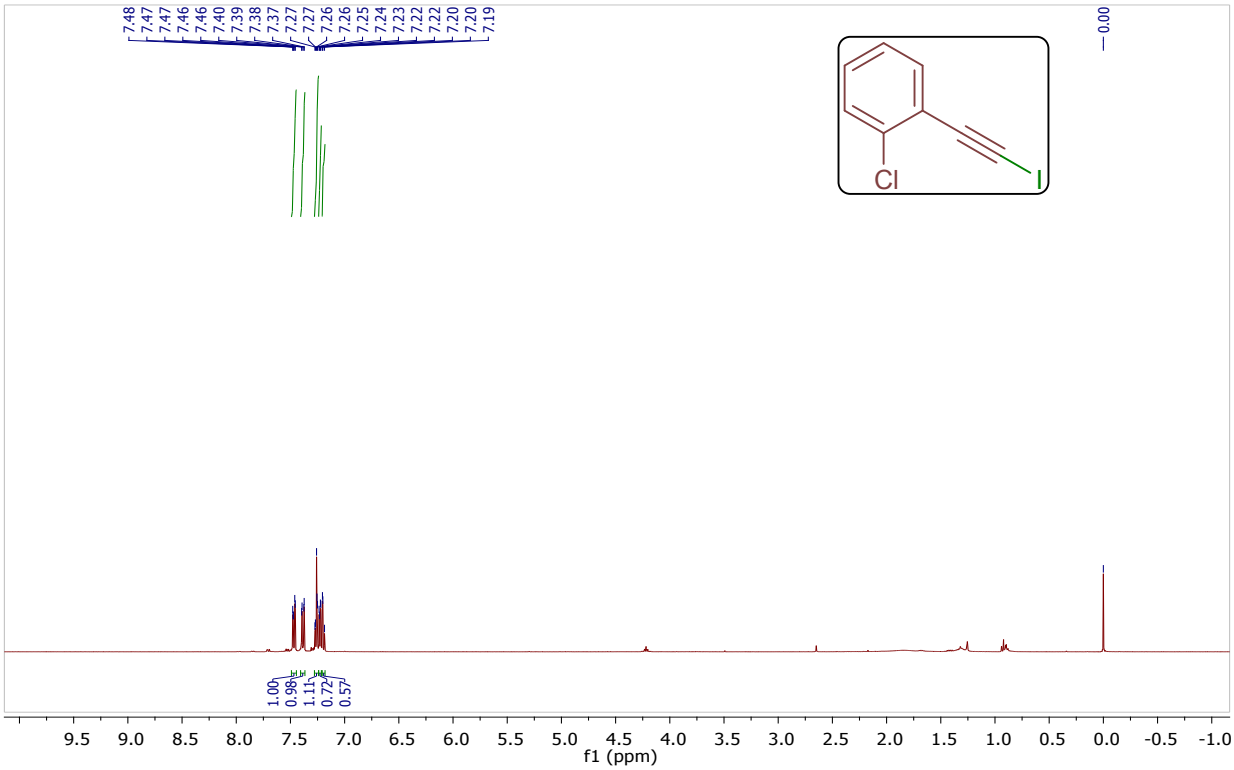
^{1}H NMR Spectrum of compound **2a** in $CDCl_3$



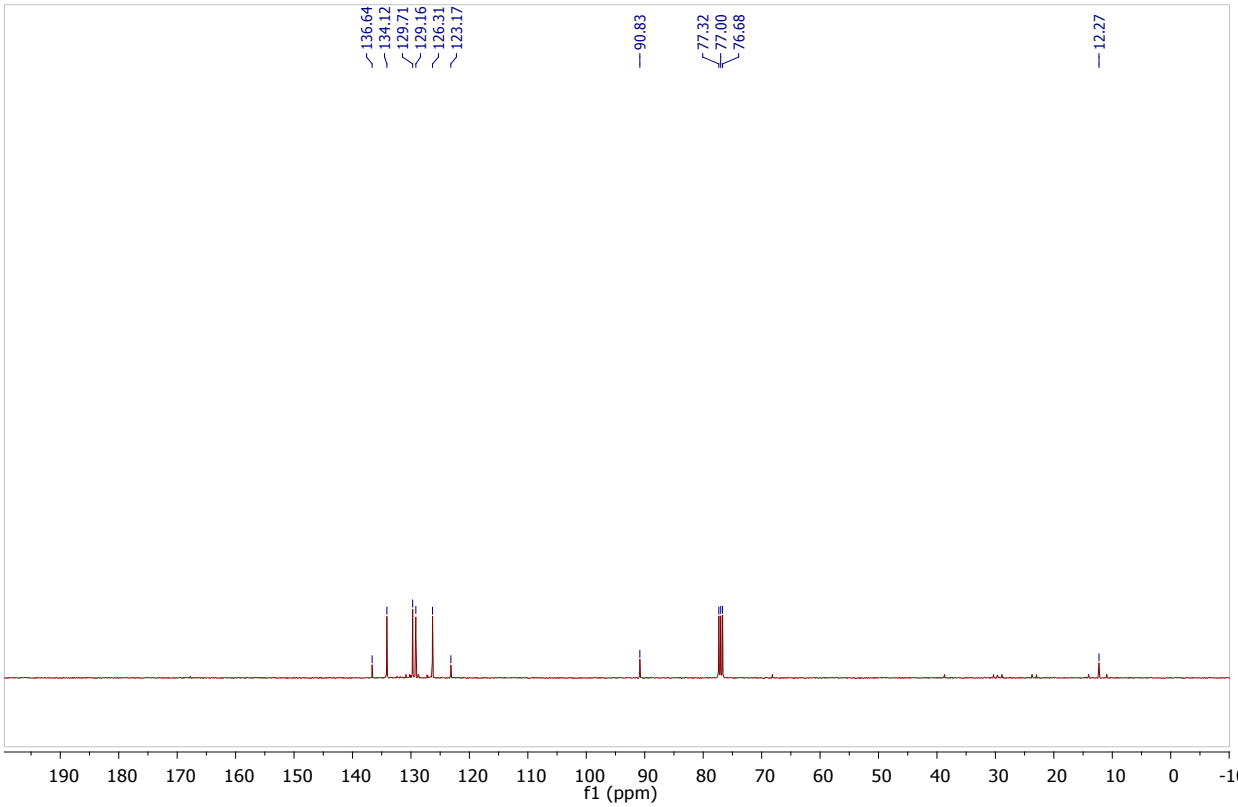
^{13}C NMR Spectrum of compound **2a** in $CDCl_3$



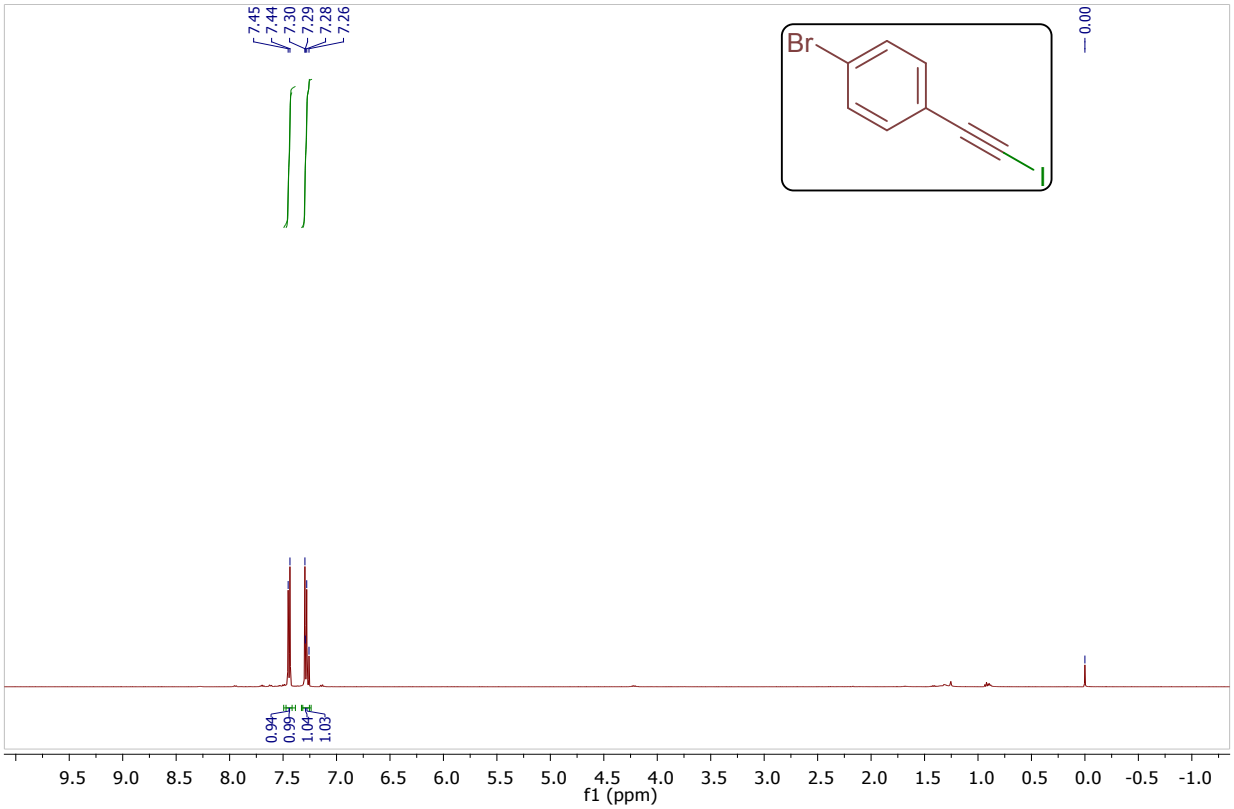
^{1}H NMR Spectrum of compound **2b** in $CDCl_3$



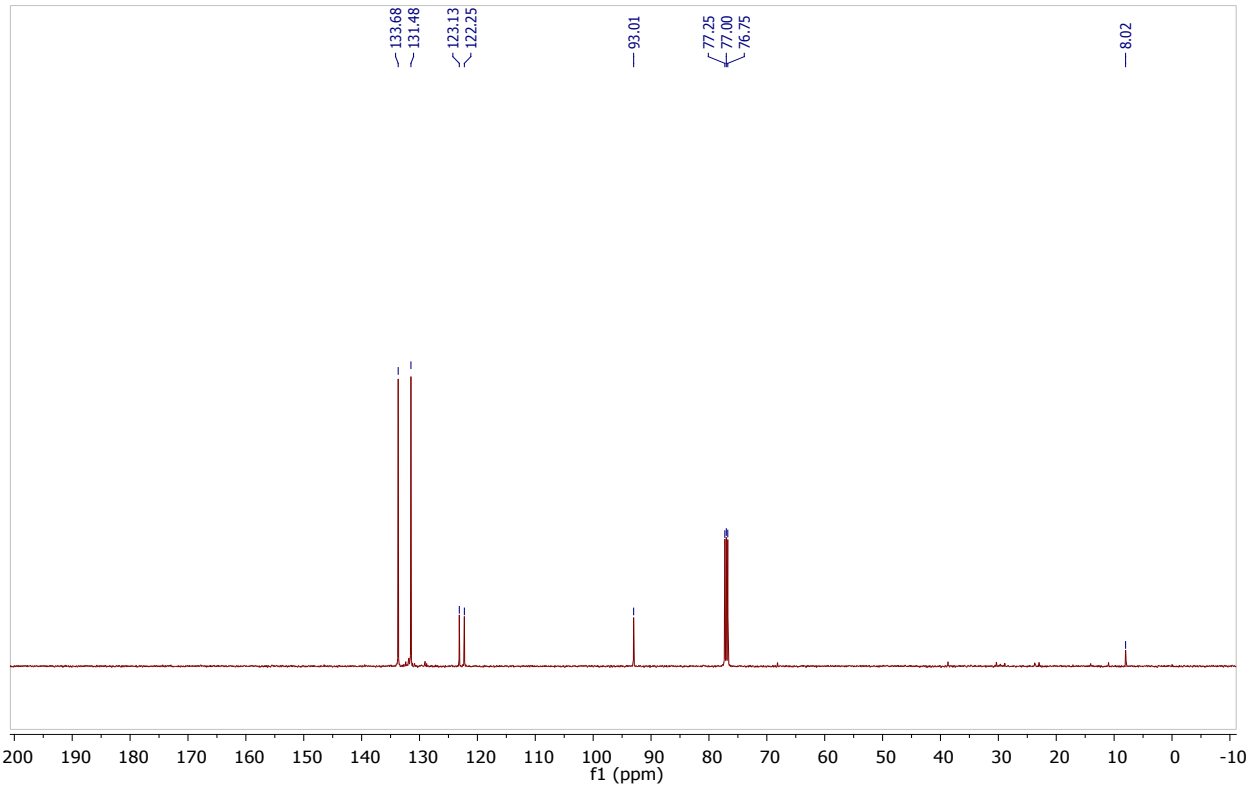
^{13}C NMR Spectrum of compound **2b** in $CDCl_3$



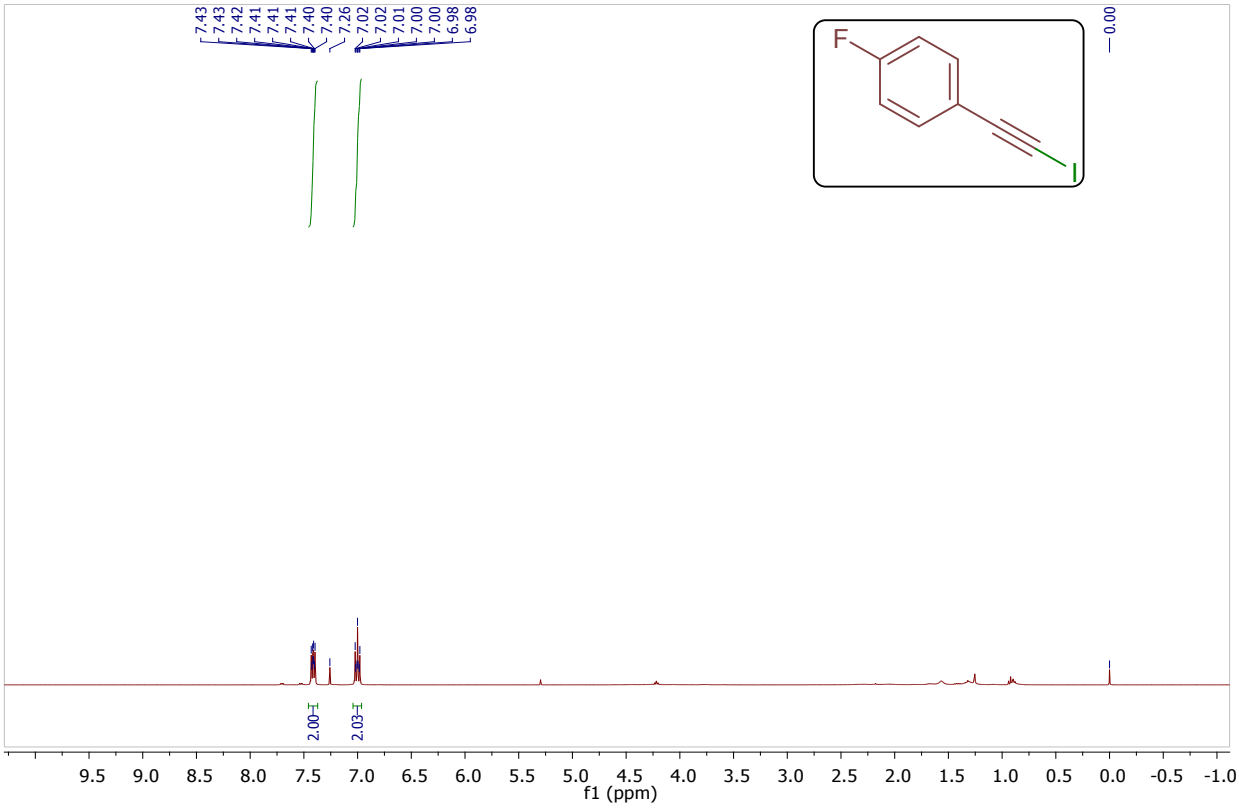
^{1}H NMR Spectrum of compound **2c** in $CDCl_3$



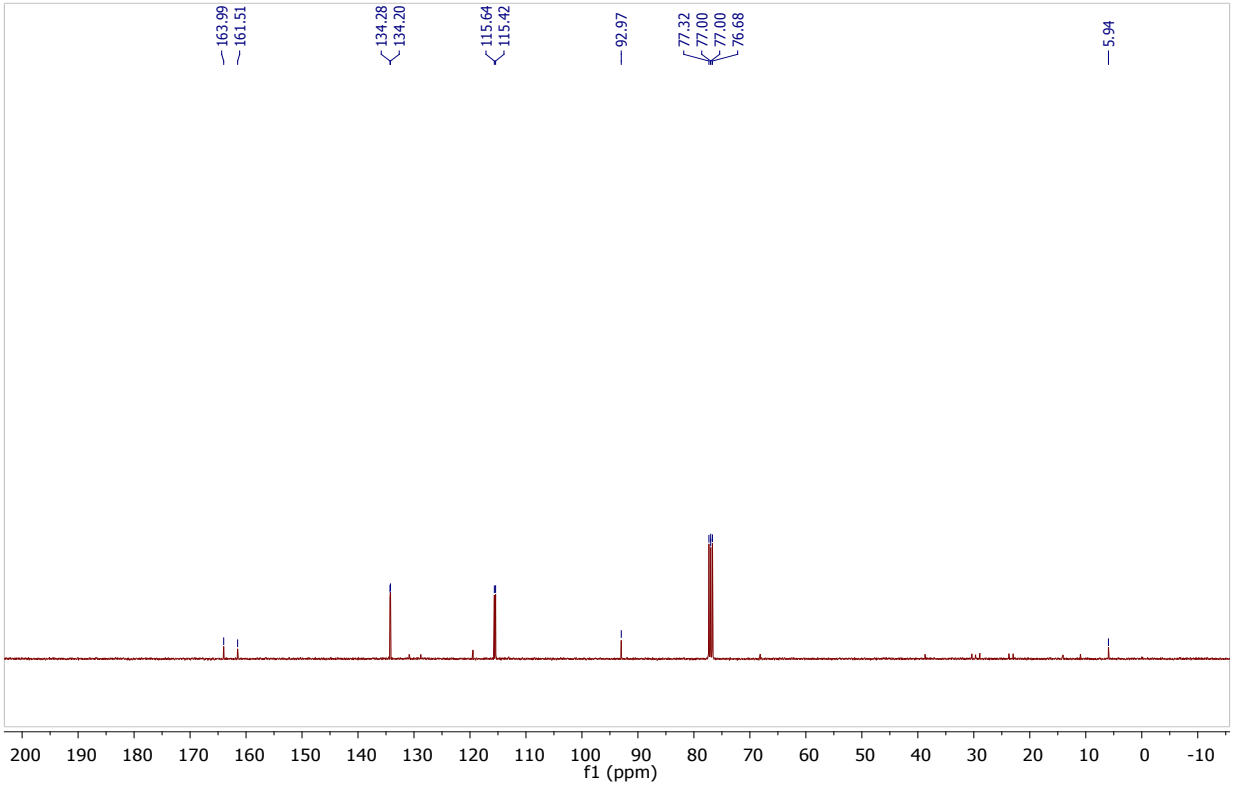
^{13}C NMR Spectrum of compound **2c** in $CDCl_3$



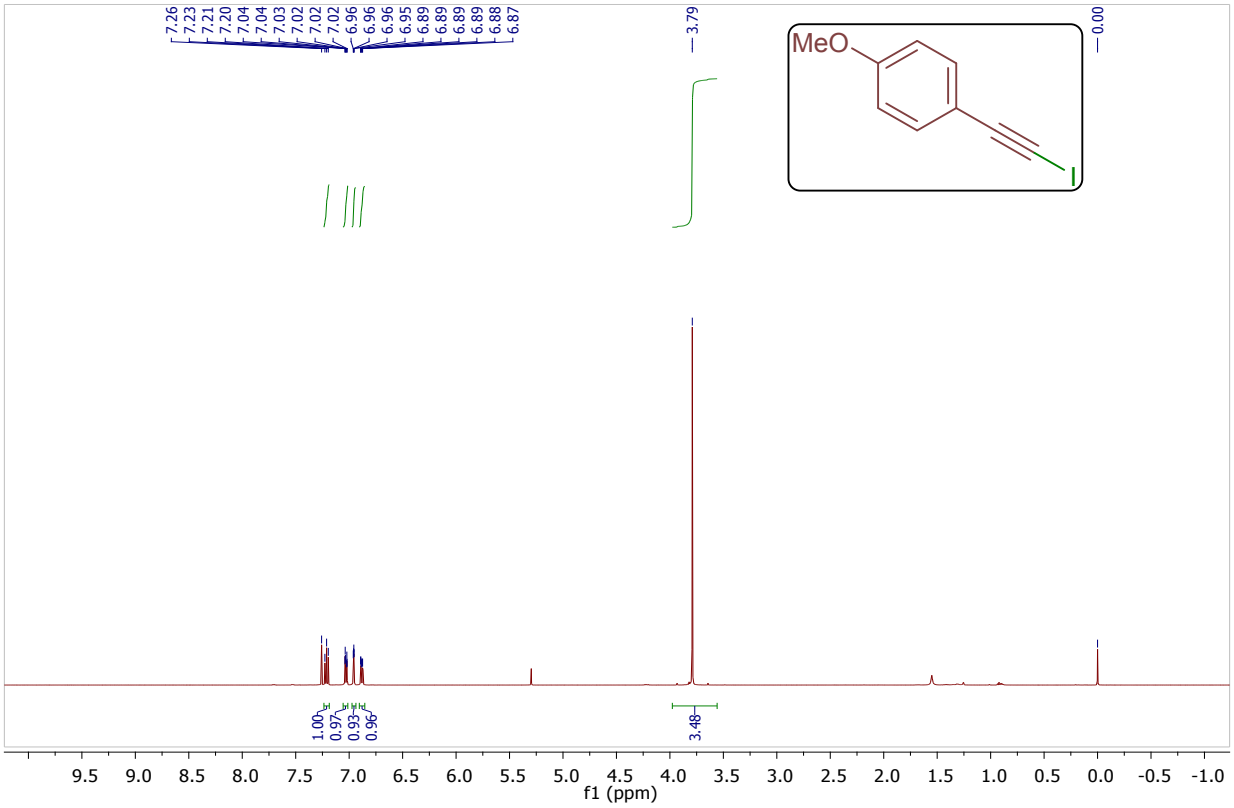
^{1}H NMR Spectrum of compound **2d** in $CDCl_3$



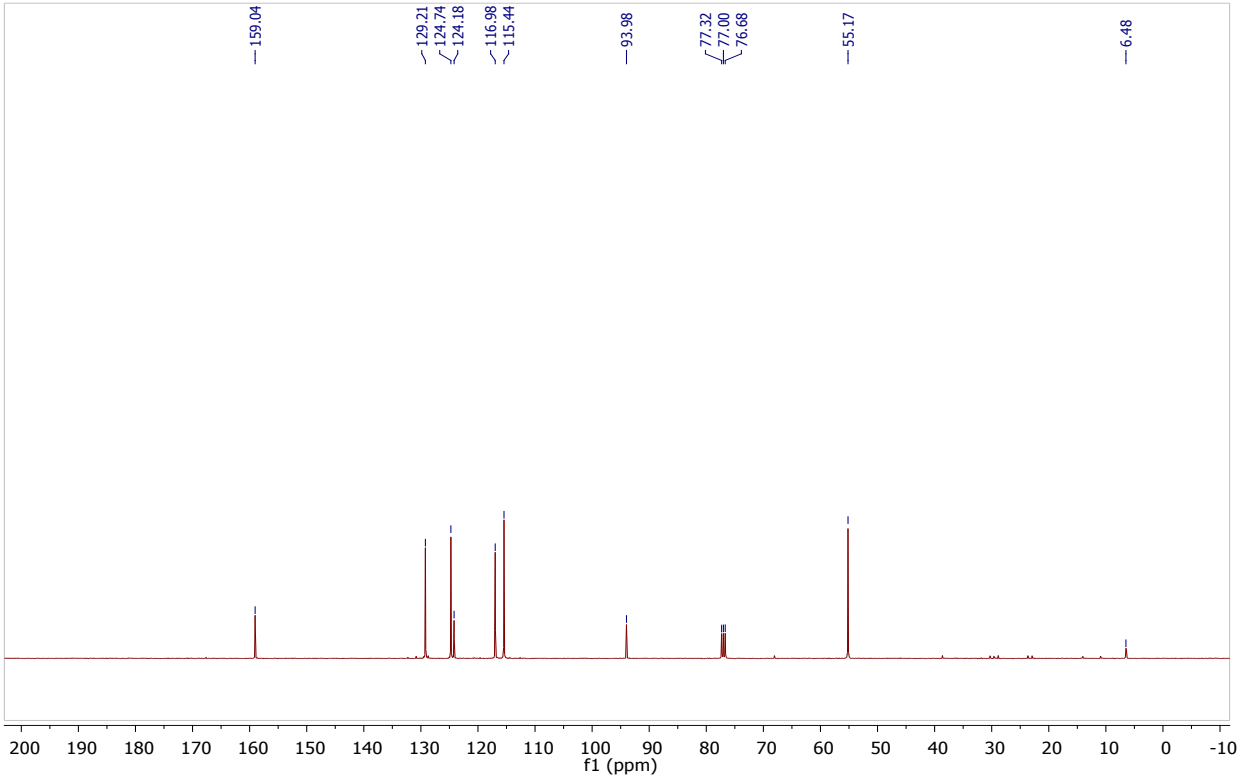
^{13}C NMR Spectrum of compound **2d** in $CDCl_3$



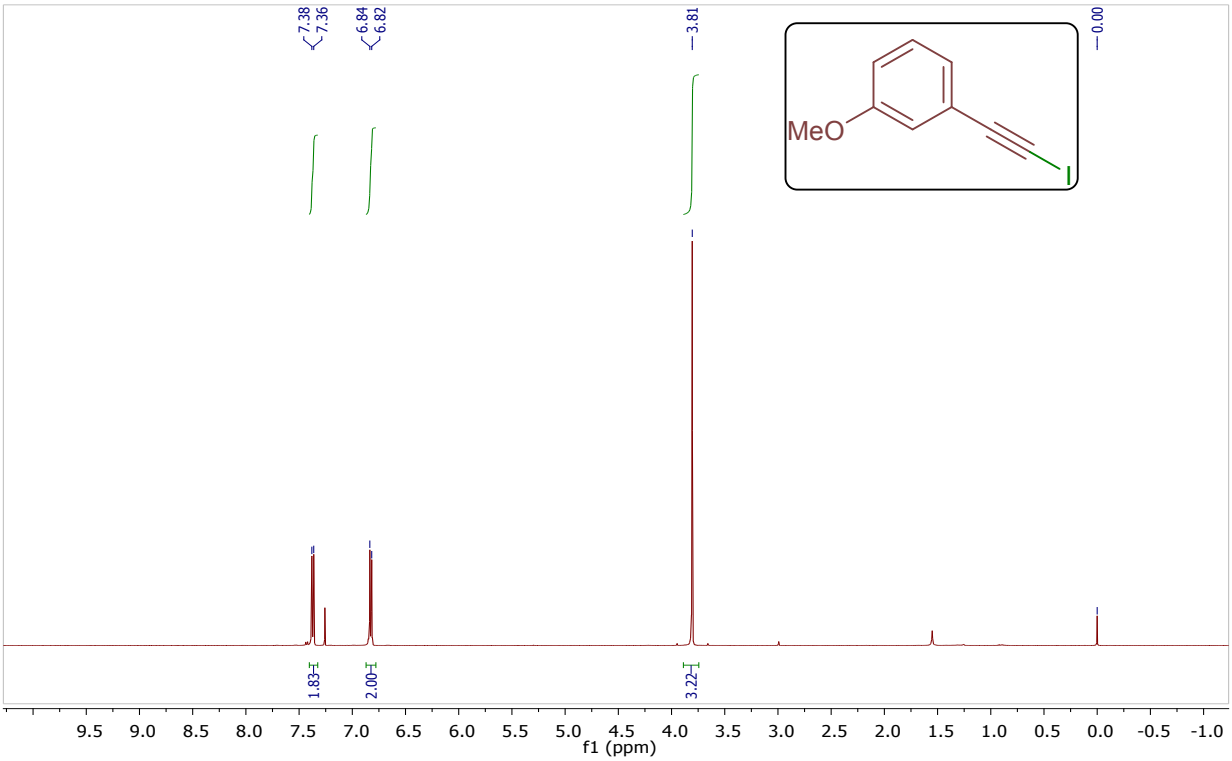
^{1}H NMR Spectrum of compound **2e** in $CDCl_3$



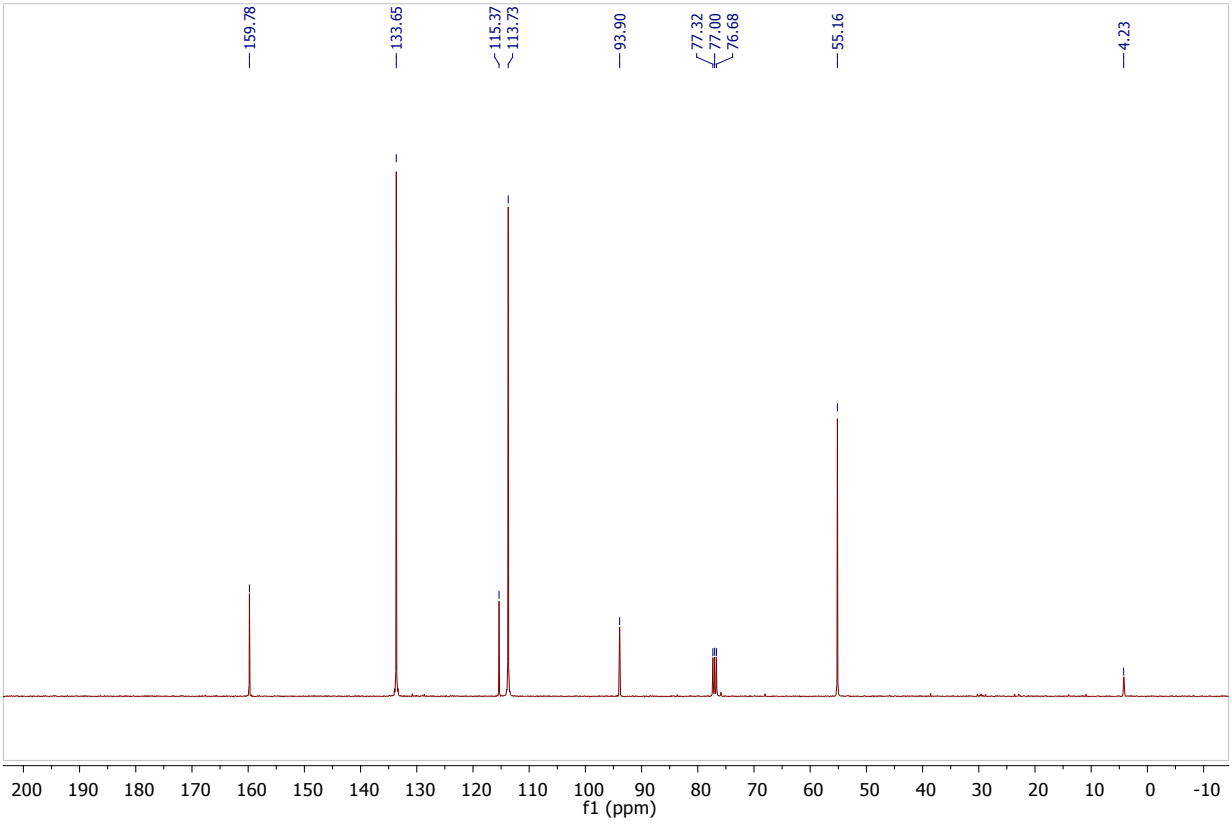
^{13}C NMR Spectrum of compound **2e** in $CDCl_3$



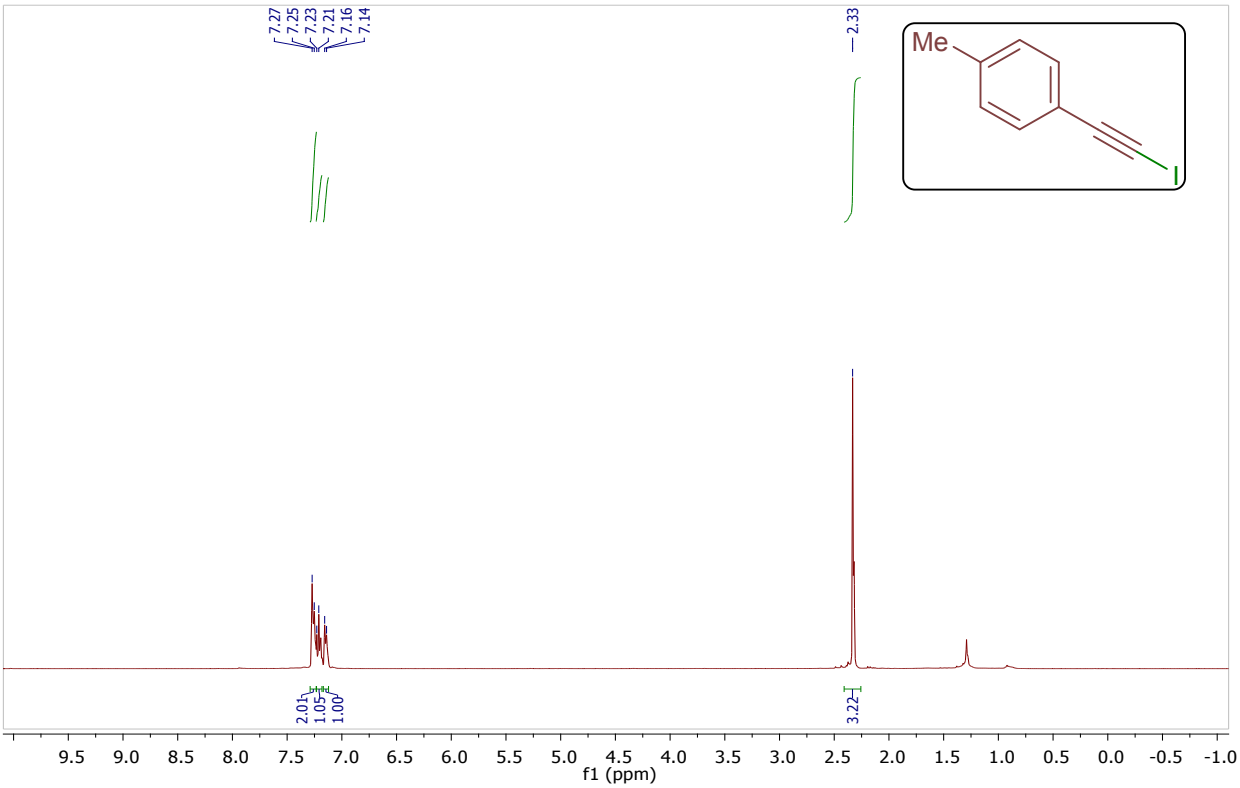
^{1}H NMR Spectrum of compound **2f** in $CDCl_3$



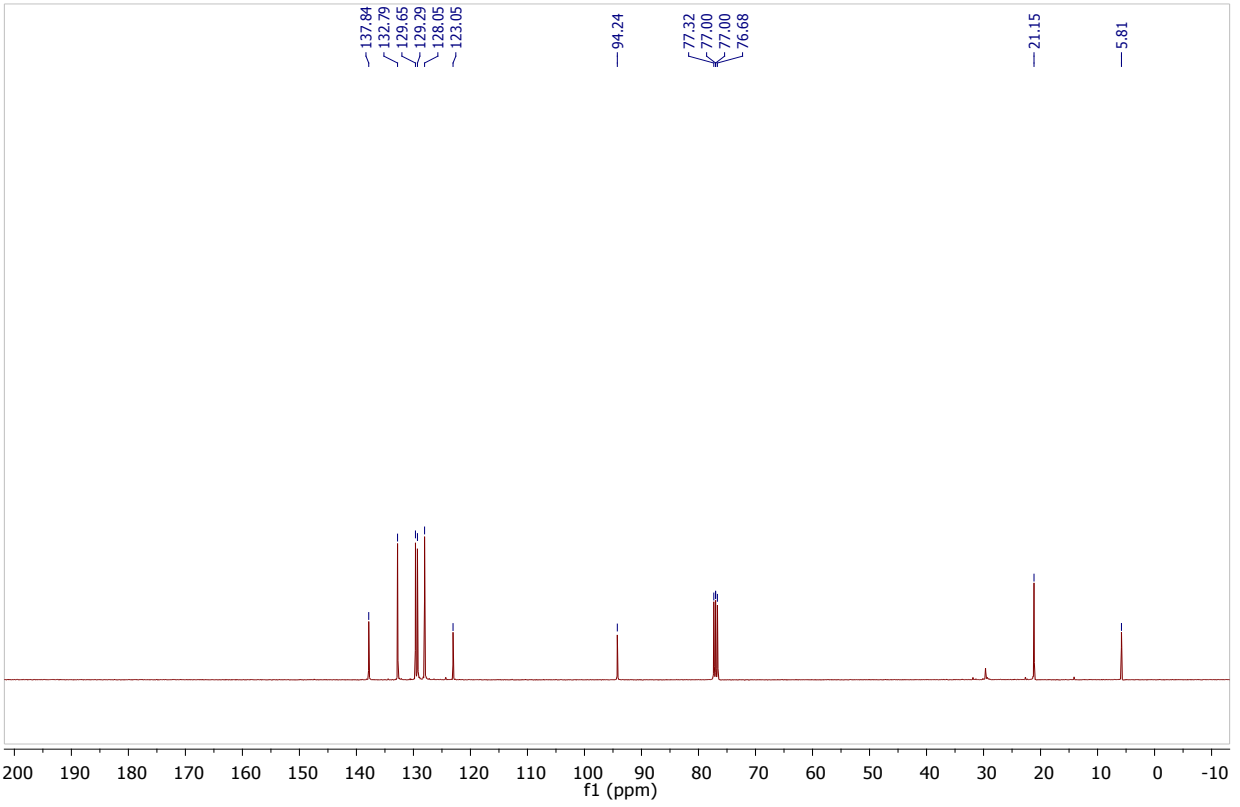
^{13}C NMR Spectrum of compound **2f** in $CDCl_3$



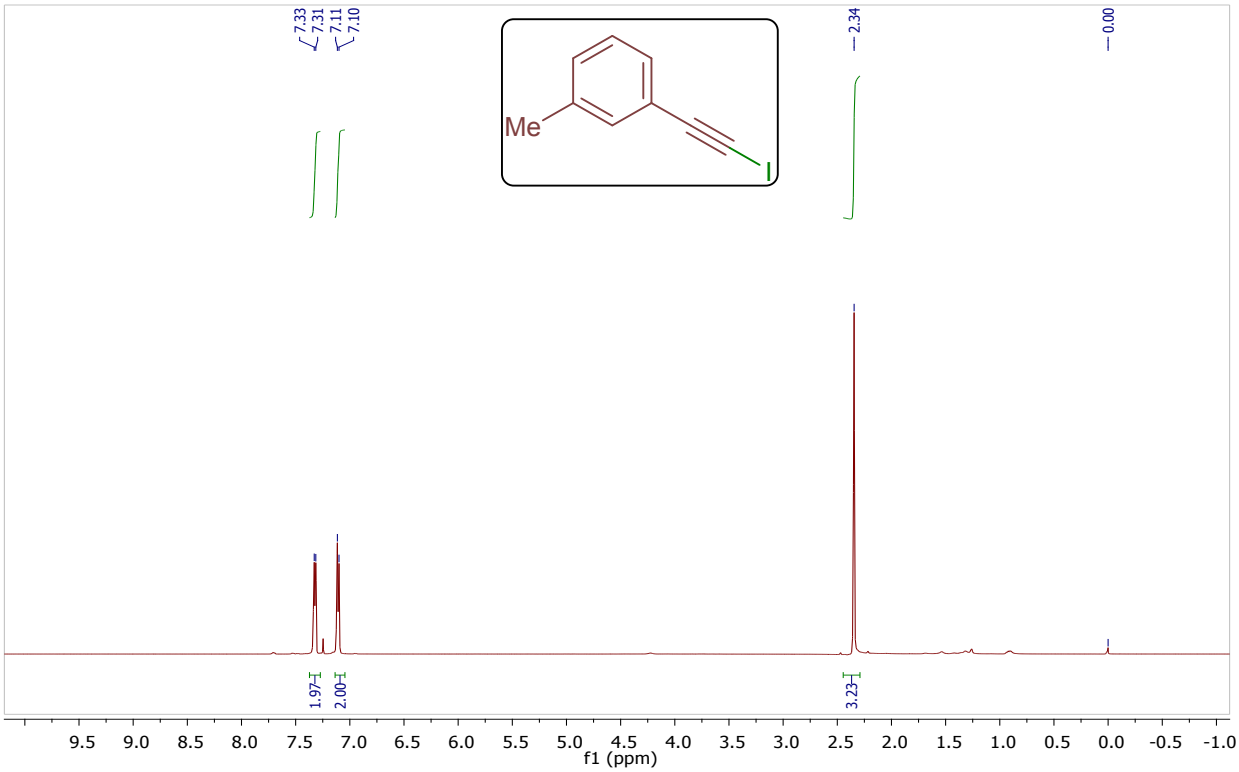
^{1}H NMR Spectrum of compound **2g** in $CDCl_3$



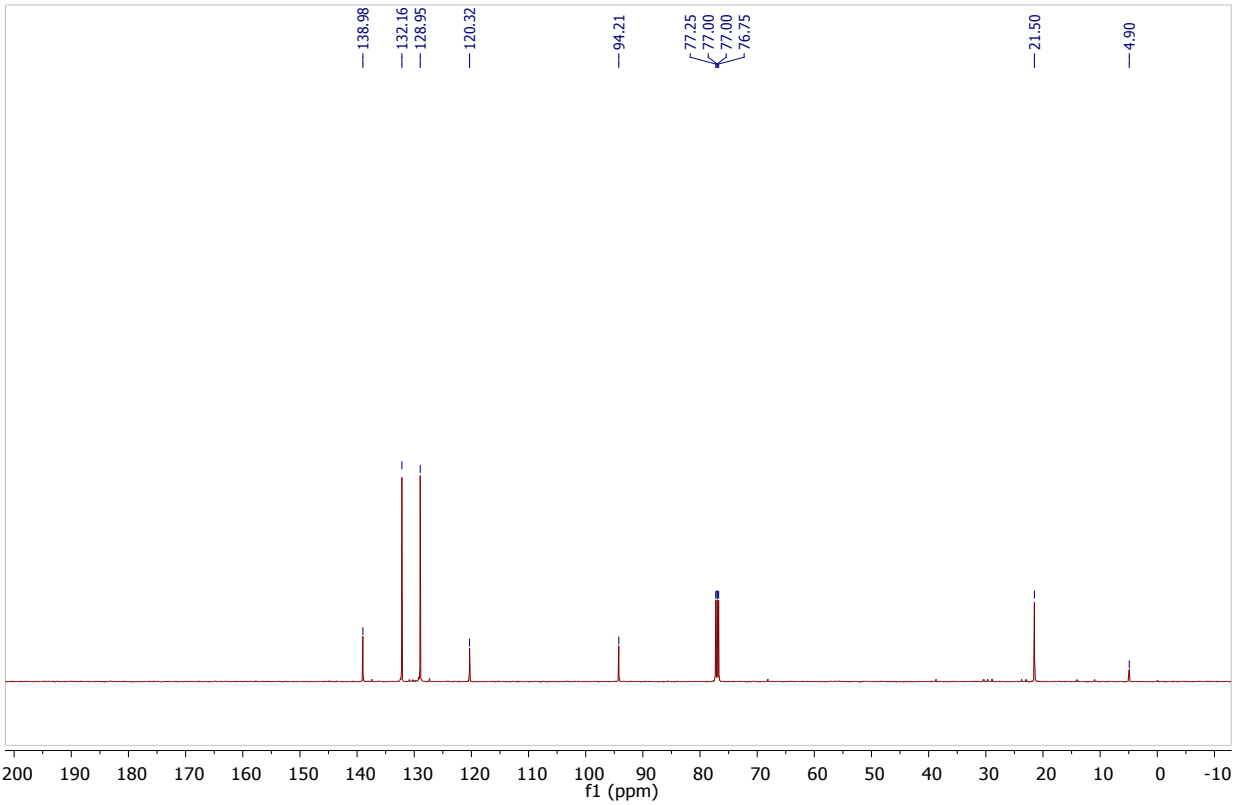
^{13}C NMR Spectrum of compound **2g** in $CDCl_3$



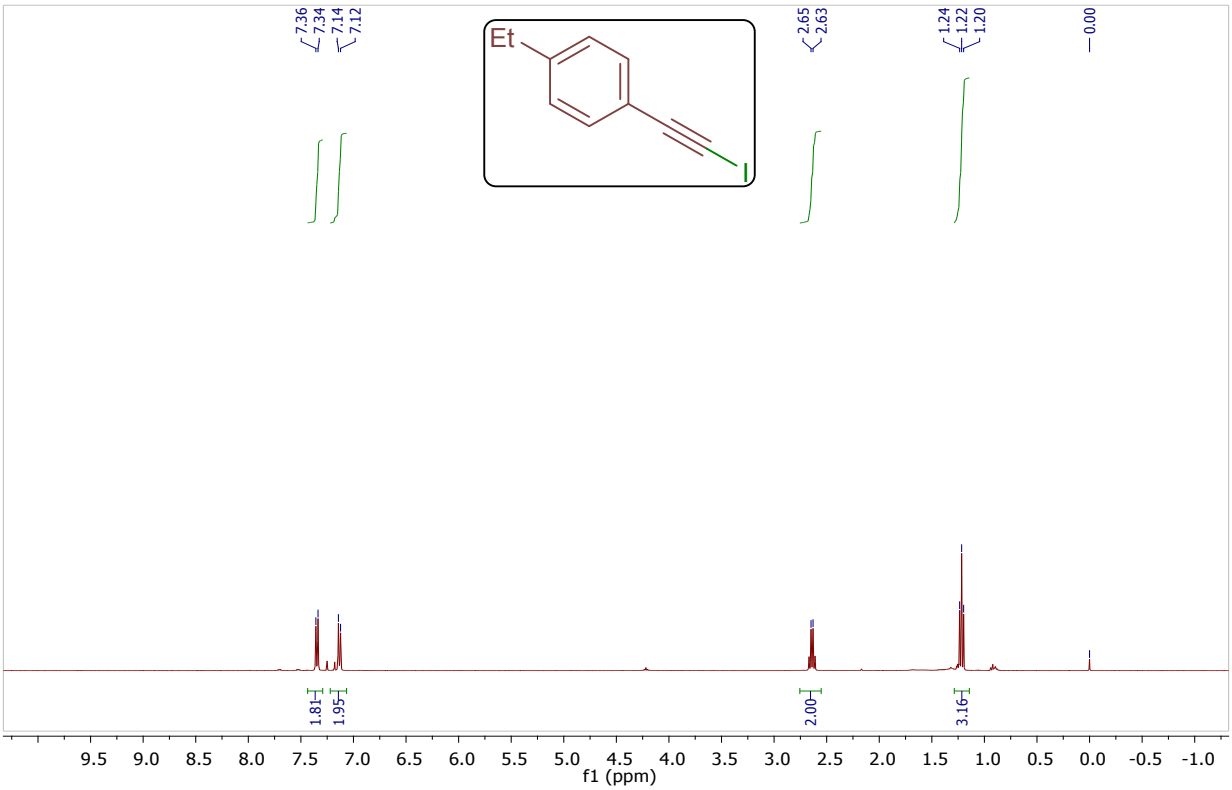
^{1}H NMR Spectrum of compound **2h** in $CDCl_3$



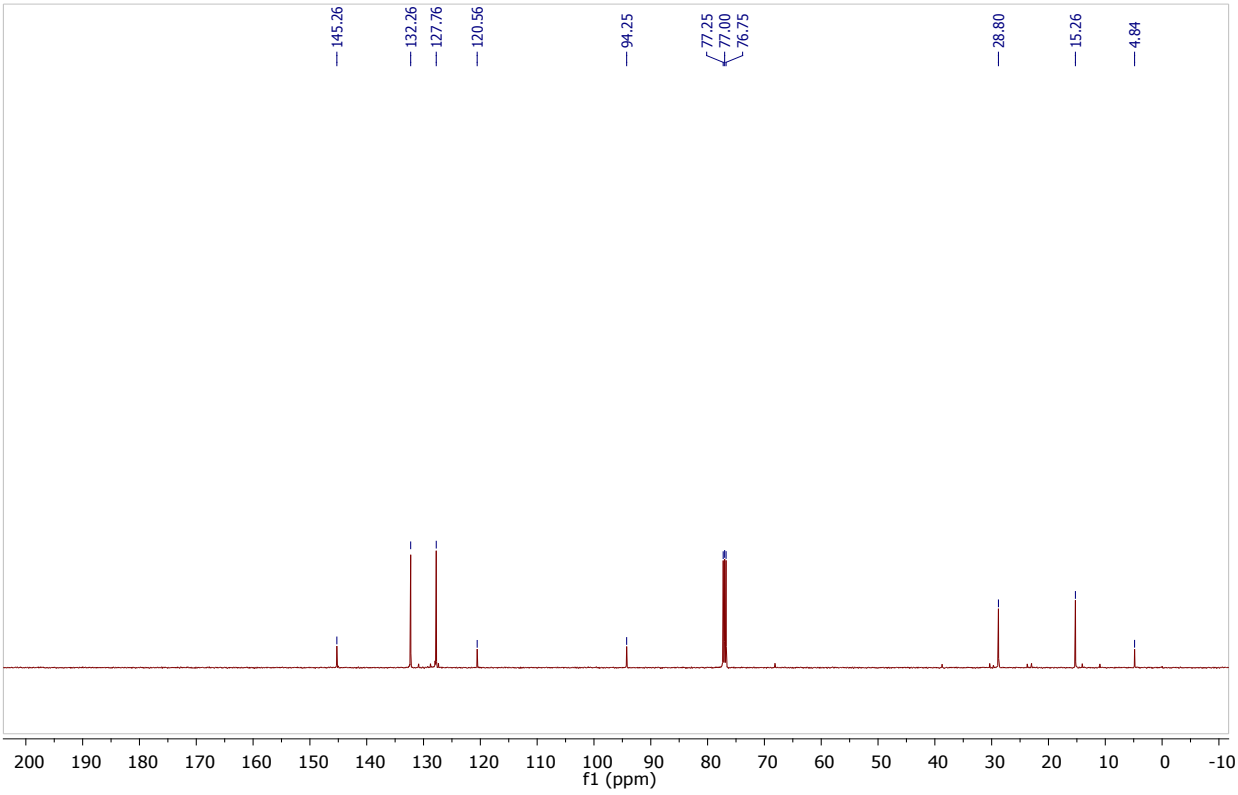
^{13}C NMR Spectrum of compound **2h** in $CDCl_3$



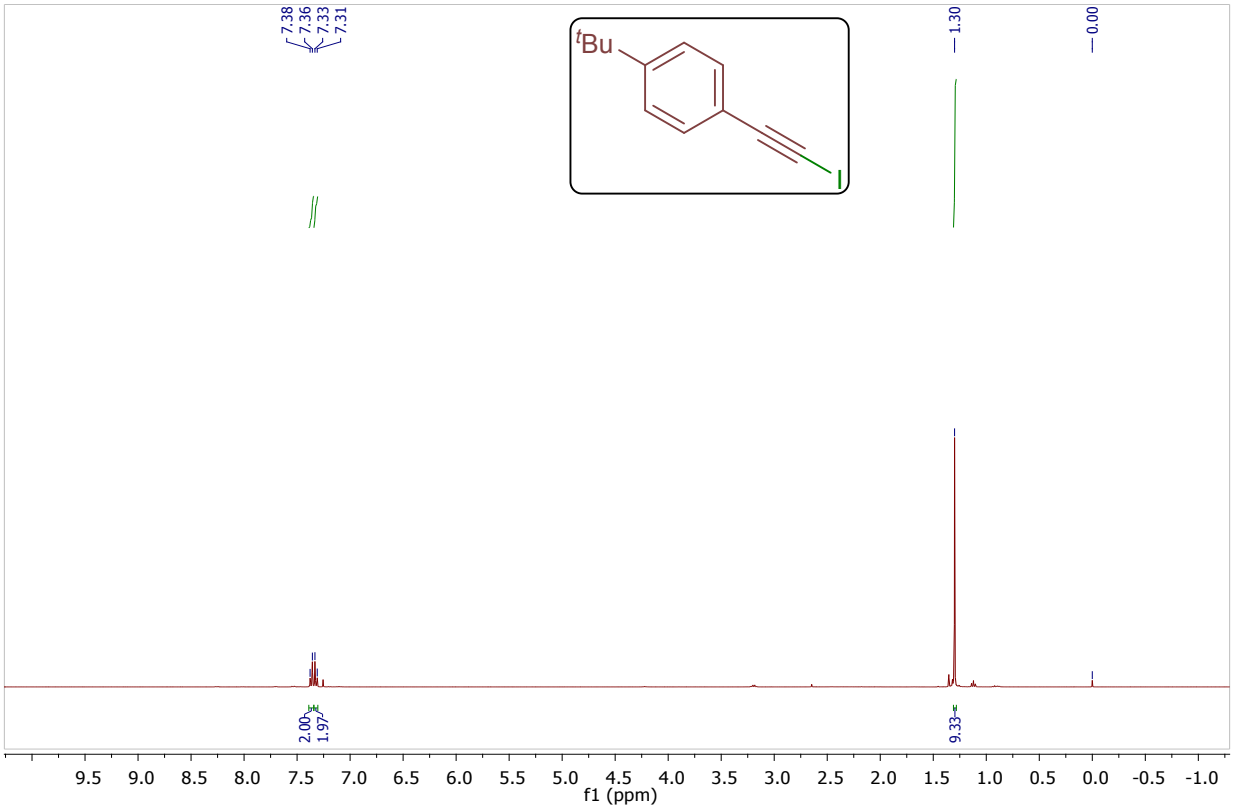
^{1}H NMR Spectrum of compound **2i** in $CDCl_3$



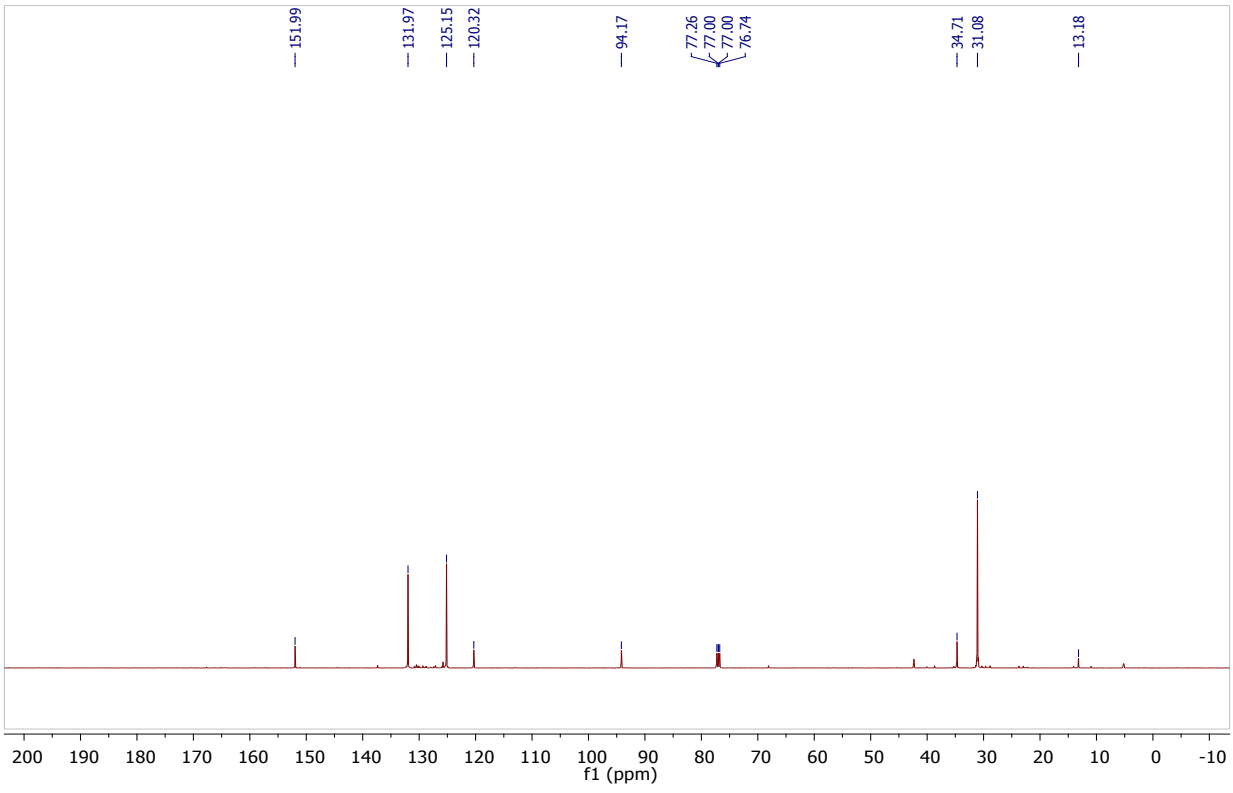
^{13}C NMR Spectrum of compound **2i** in $CDCl_3$



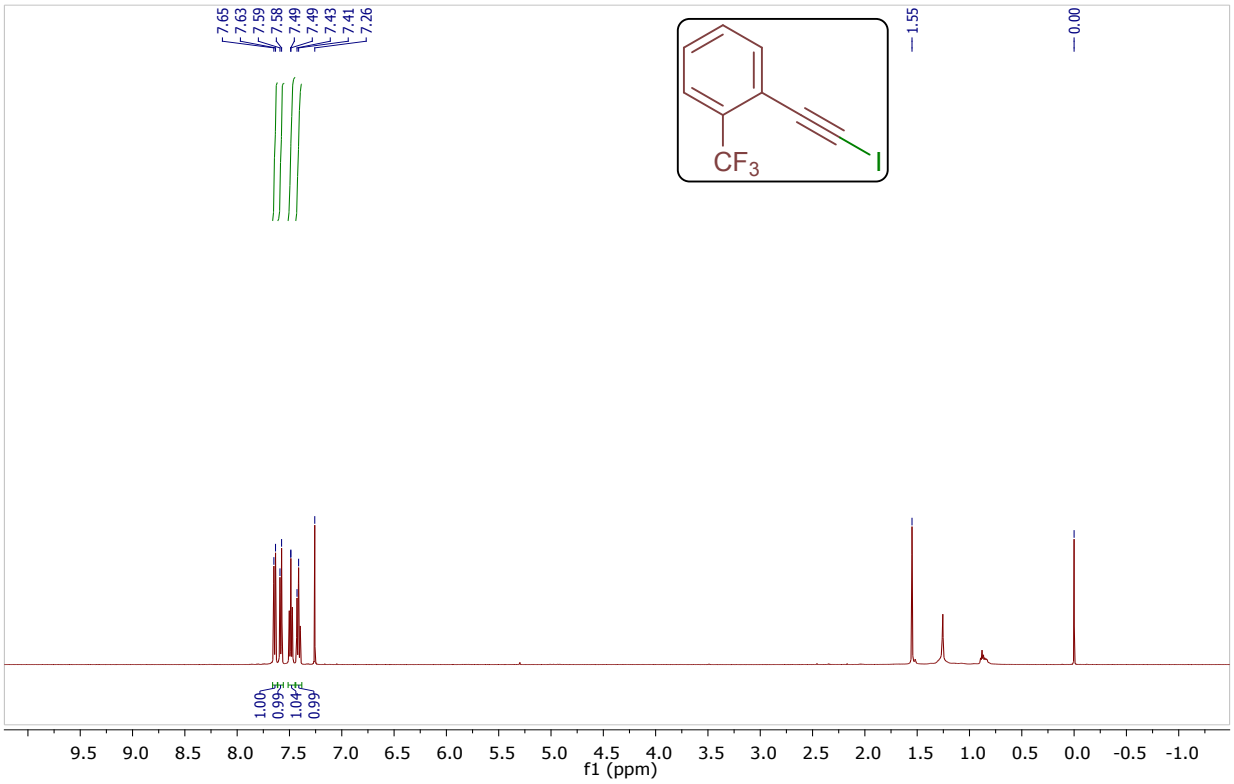
^{1}H NMR Spectrum of compound **2j** in $CDCl_3$



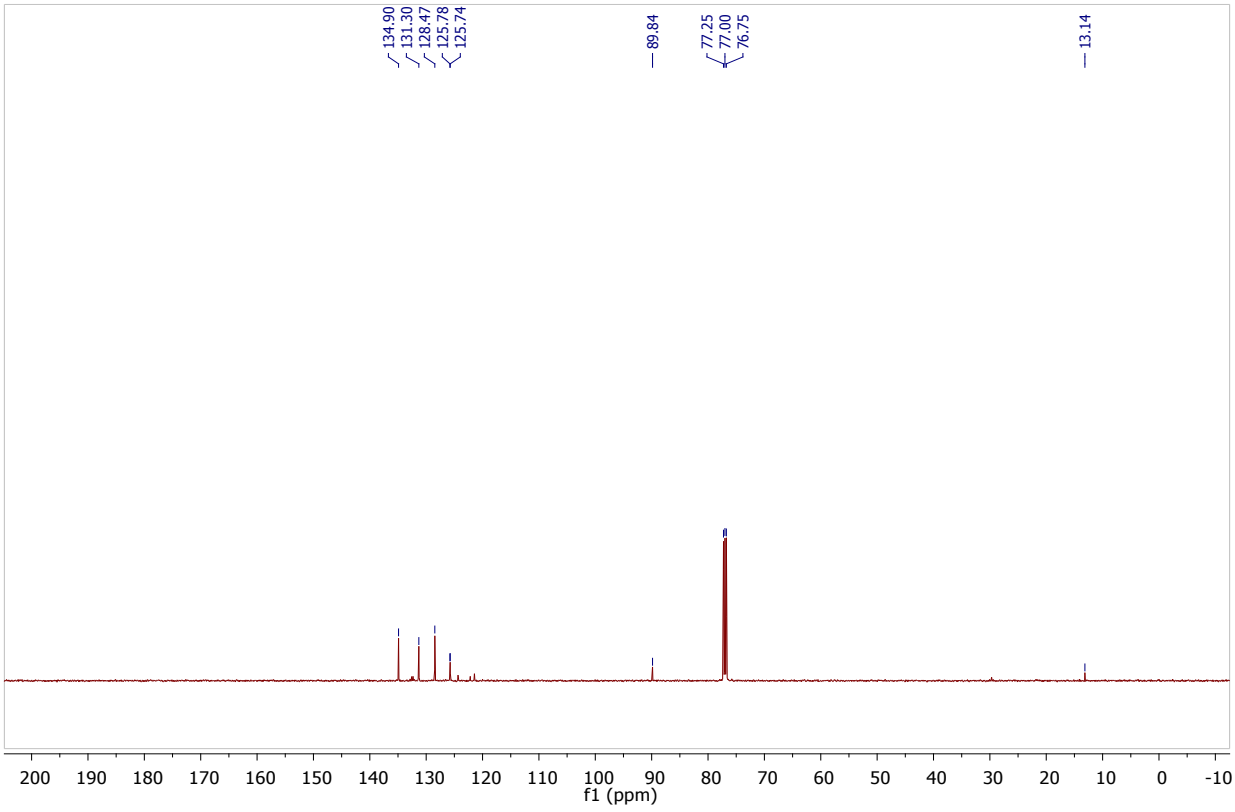
^{13}C NMR Spectrum of compound **2j** in $CDCl_3$



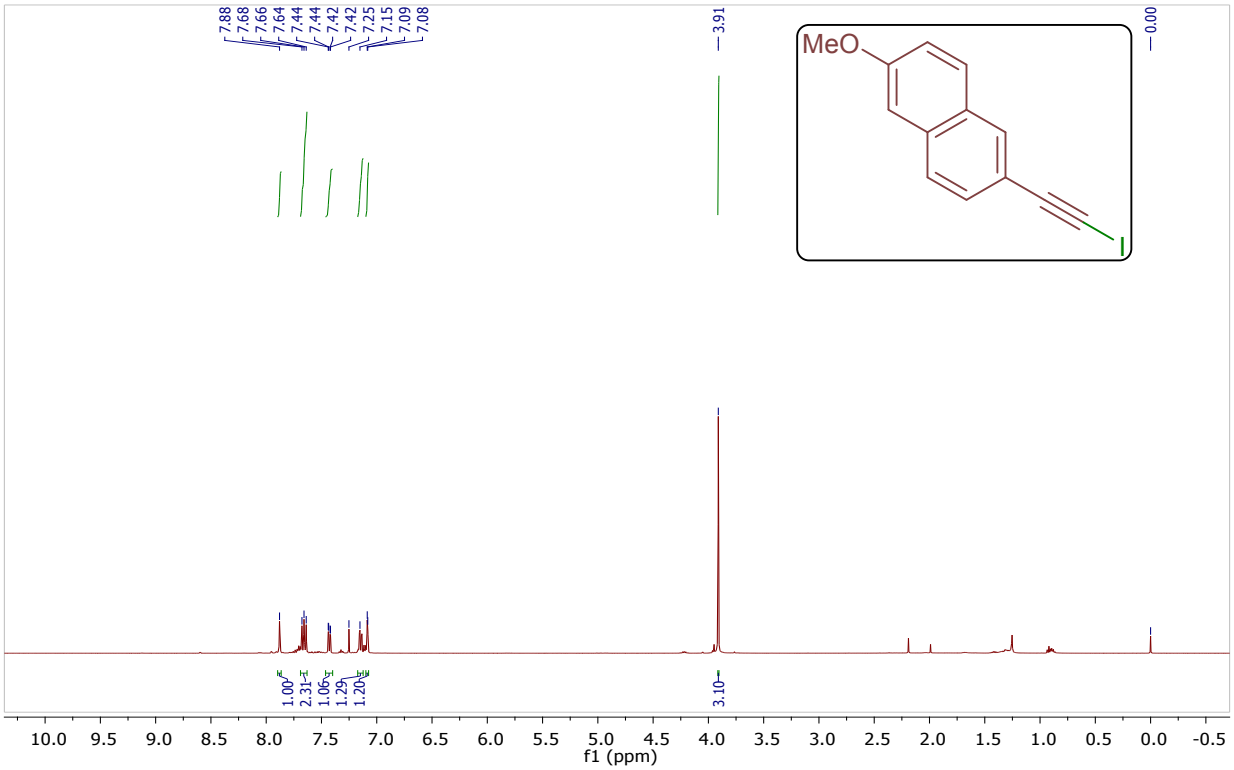
^{1}H NMR Spectrum of compound **2k** in $CDCl_3$



^{13}C NMR Spectrum of compound **2k** in $CDCl_3$



^{1}H NMR Spectrum of compound **2l** in $CDCl_3$



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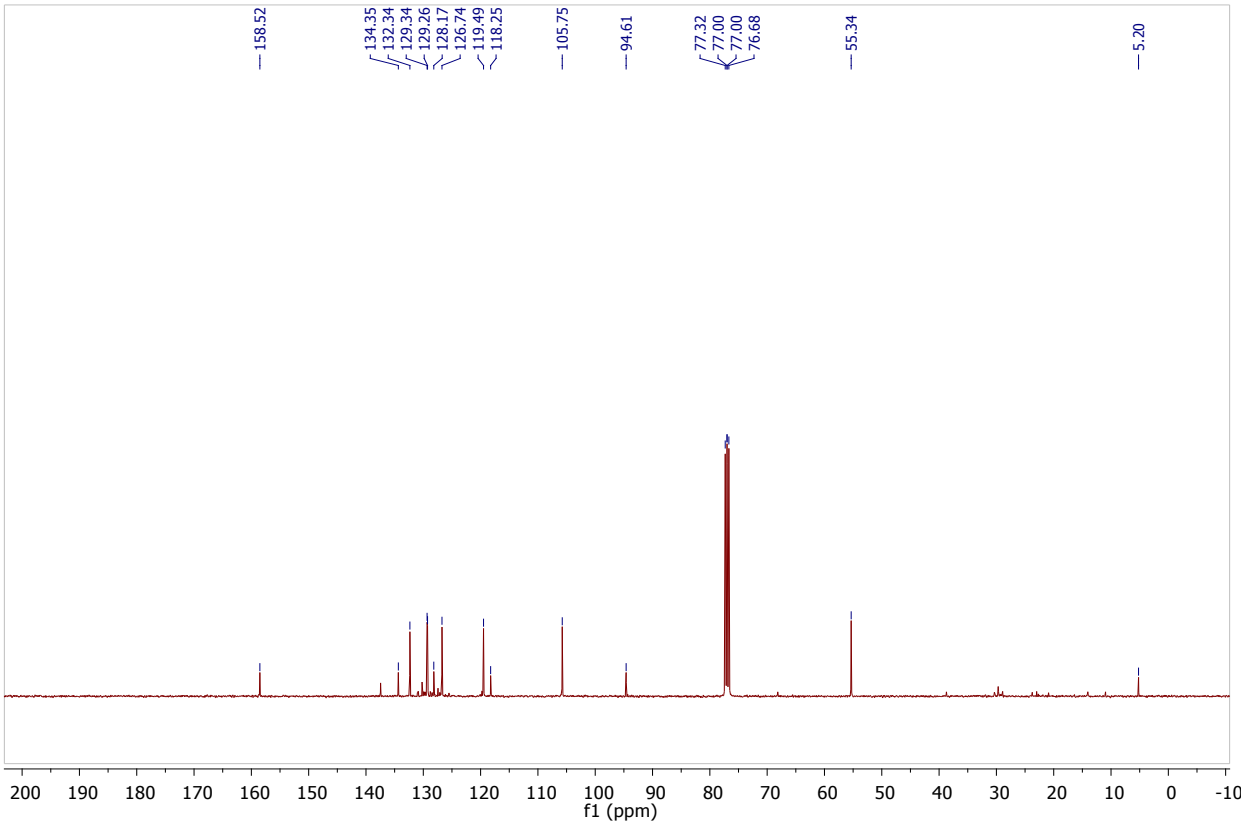
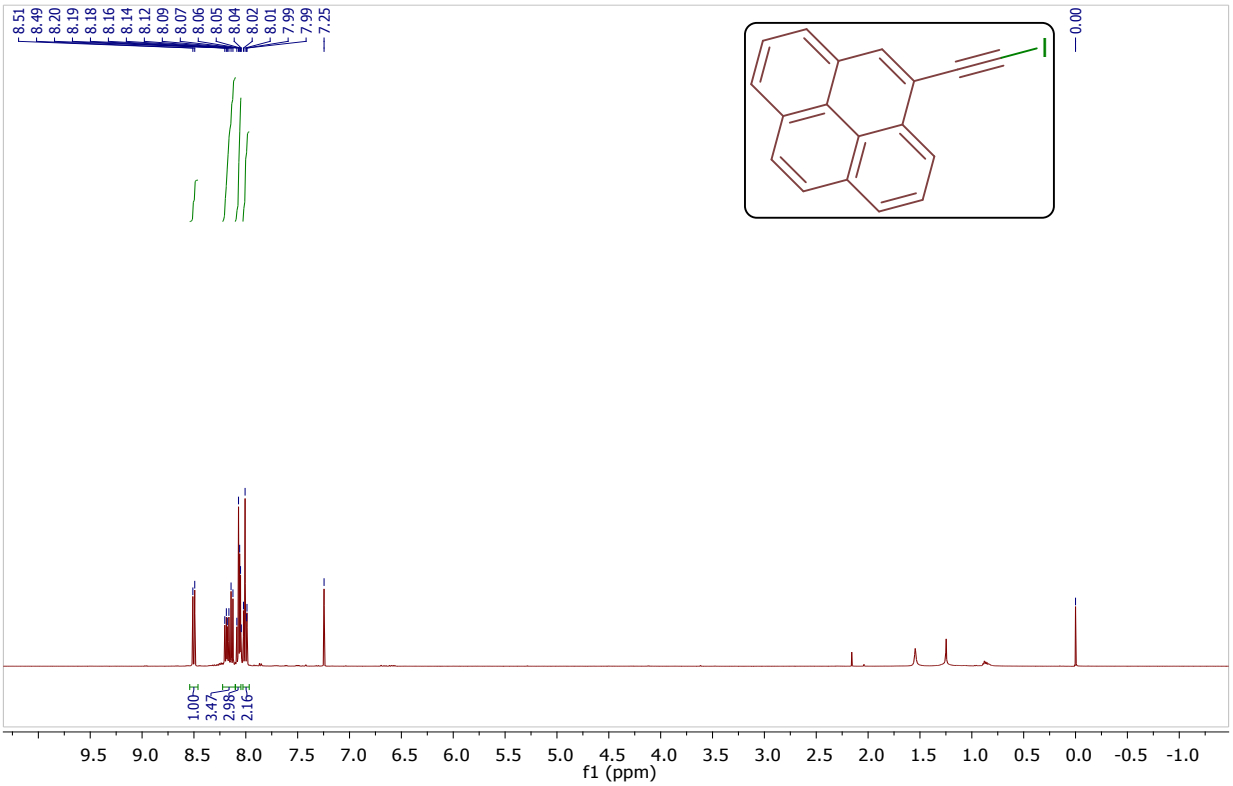
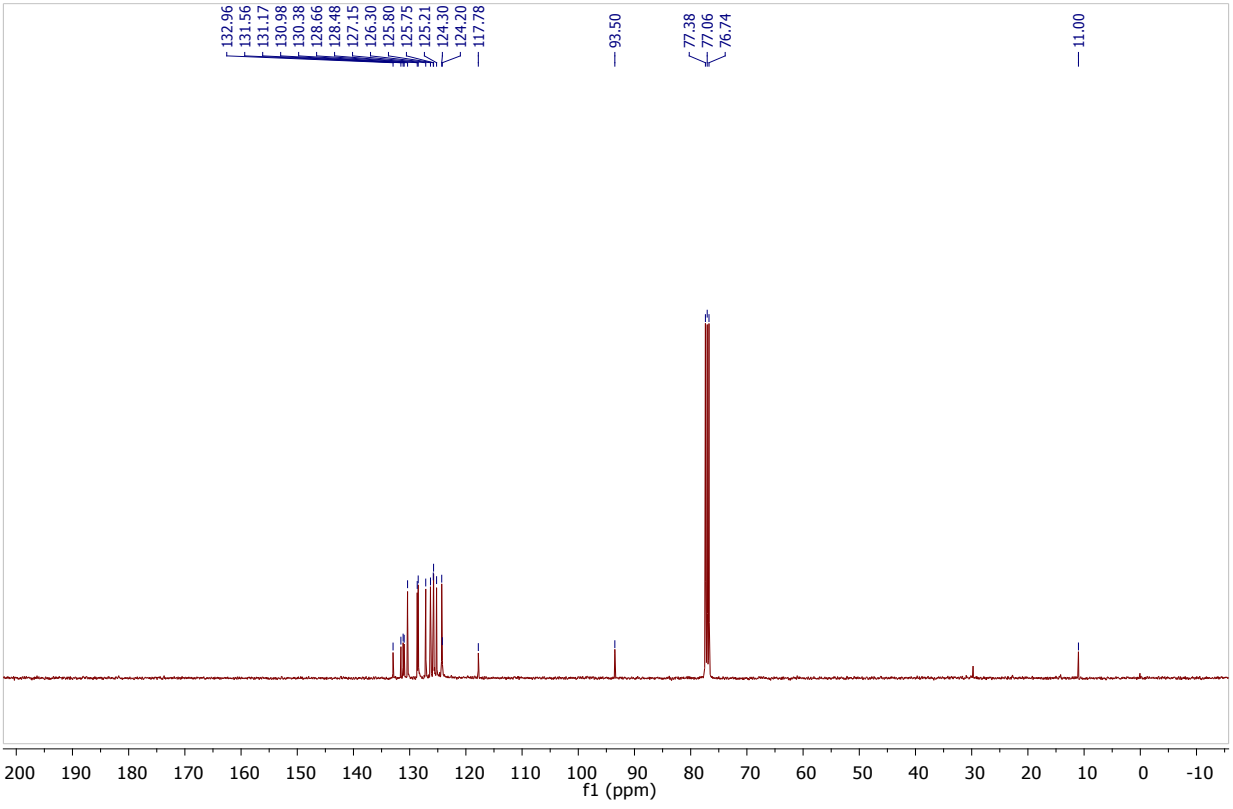


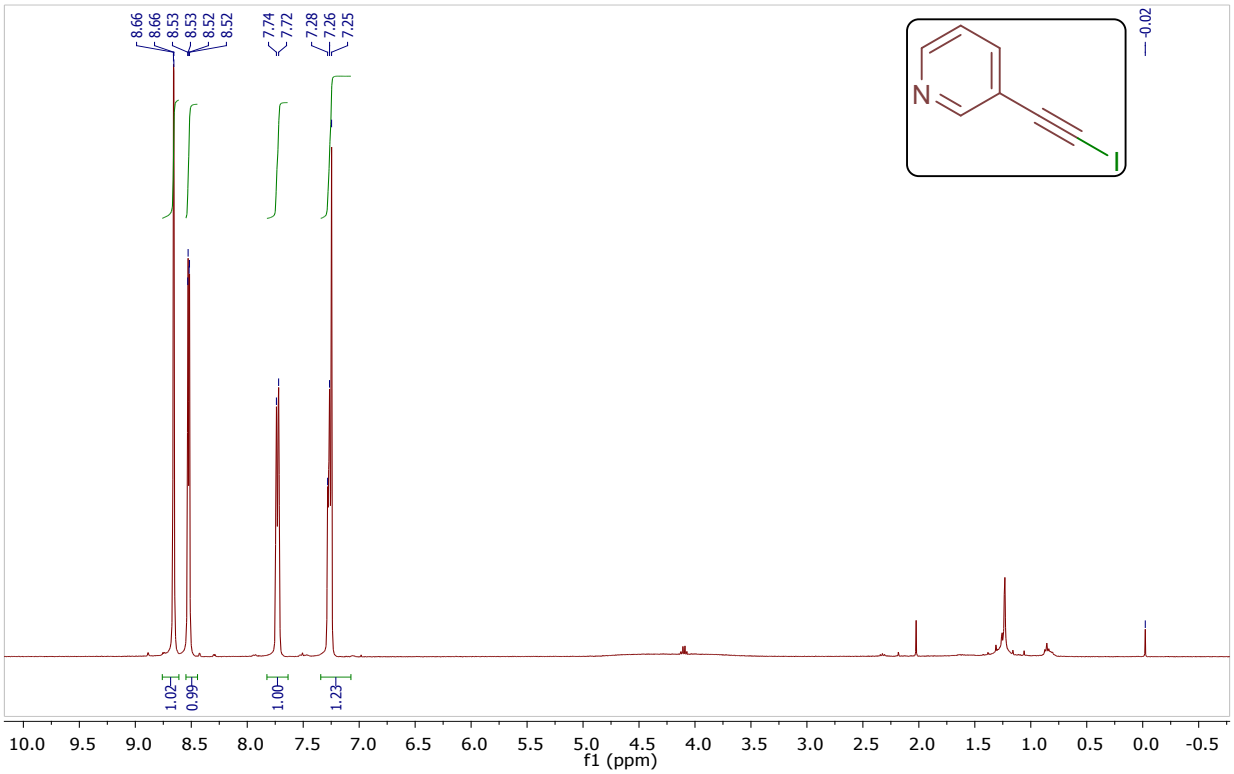
^{1}H NMR Spectrum of compound **2m** in $CDCl_3$



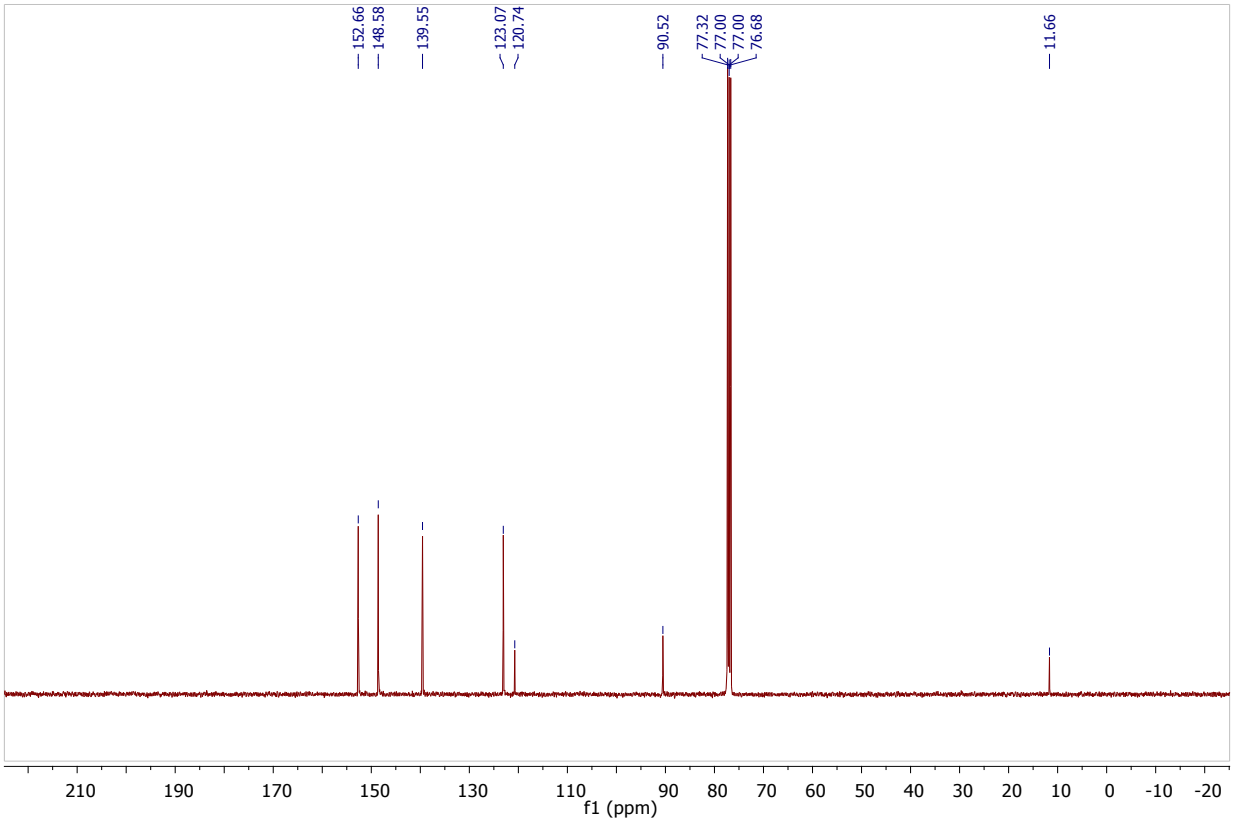
^{13}C NMR Spectrum of compound **2m** in $CDCl_3$



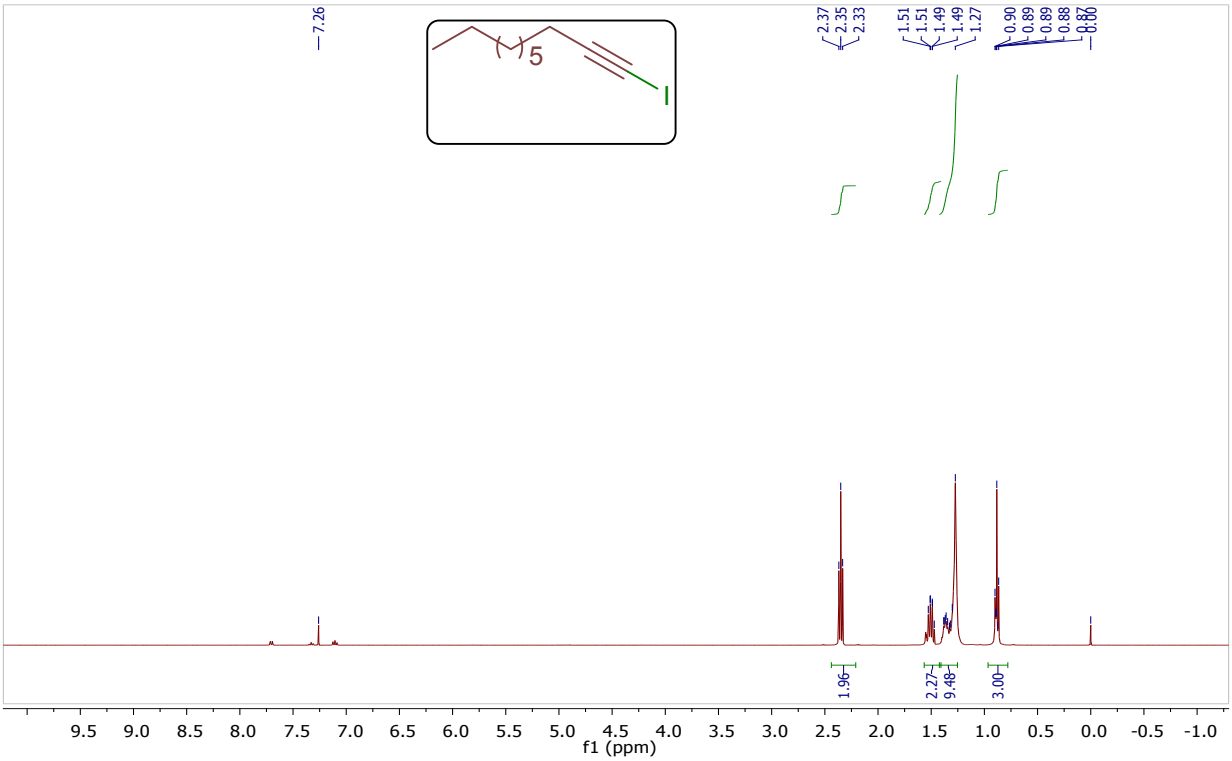
^{1}H NMR Spectrum of compound **2n** in $CDCl_3$



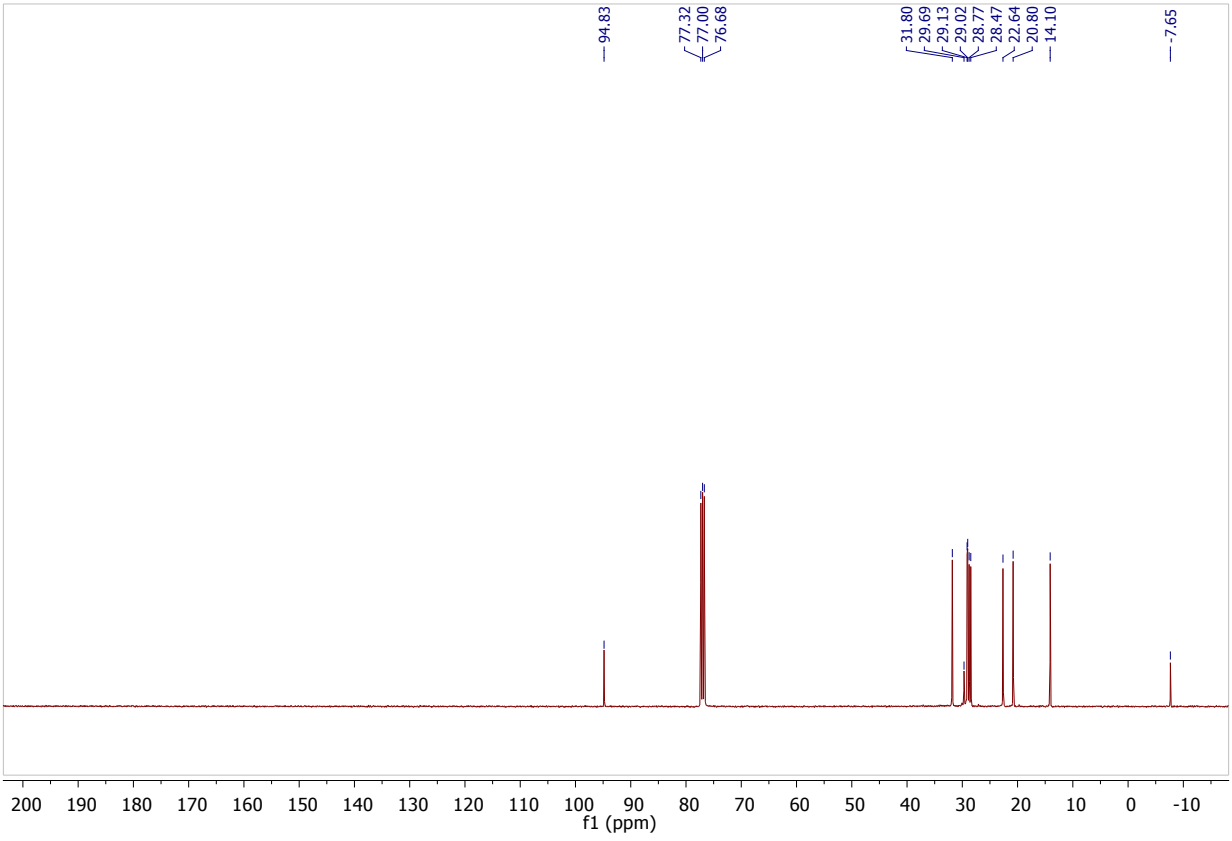
^{13}C NMR Spectrum of compound **2n** in $CDCl_3$



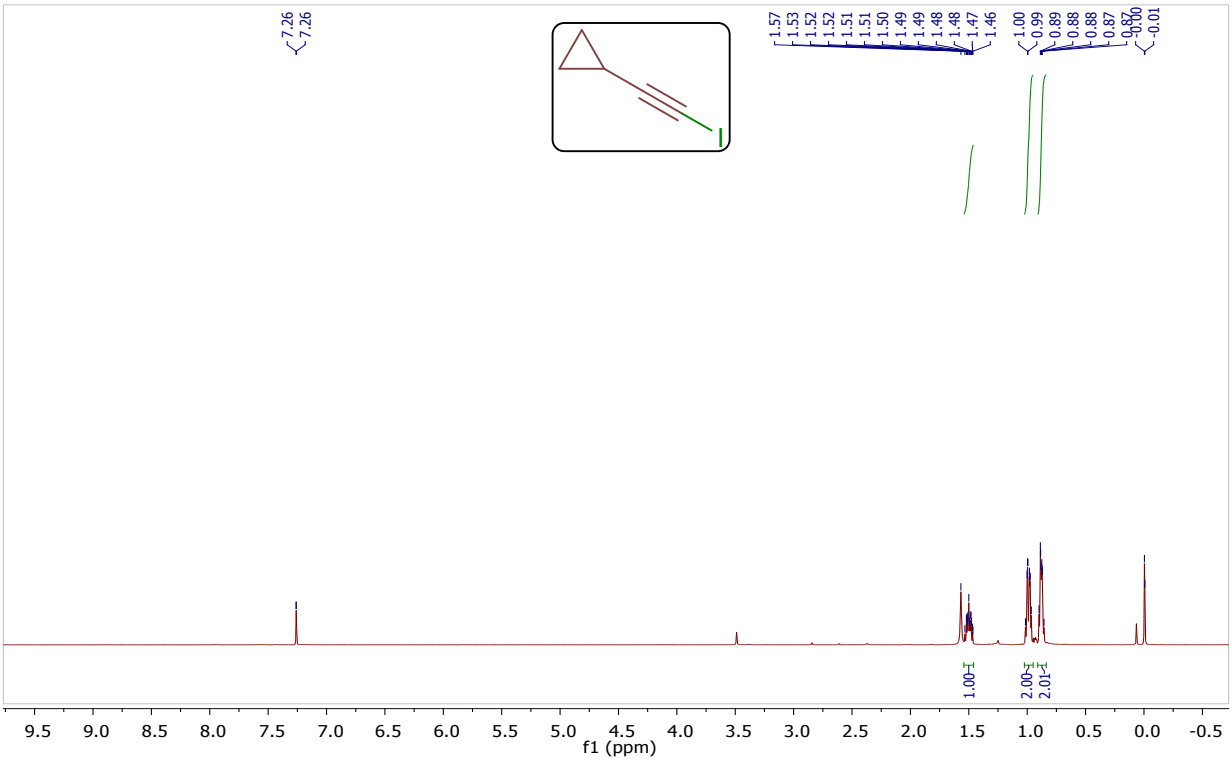
^{1}H NMR Spectrum of compound **2o** in $CDCl_3$



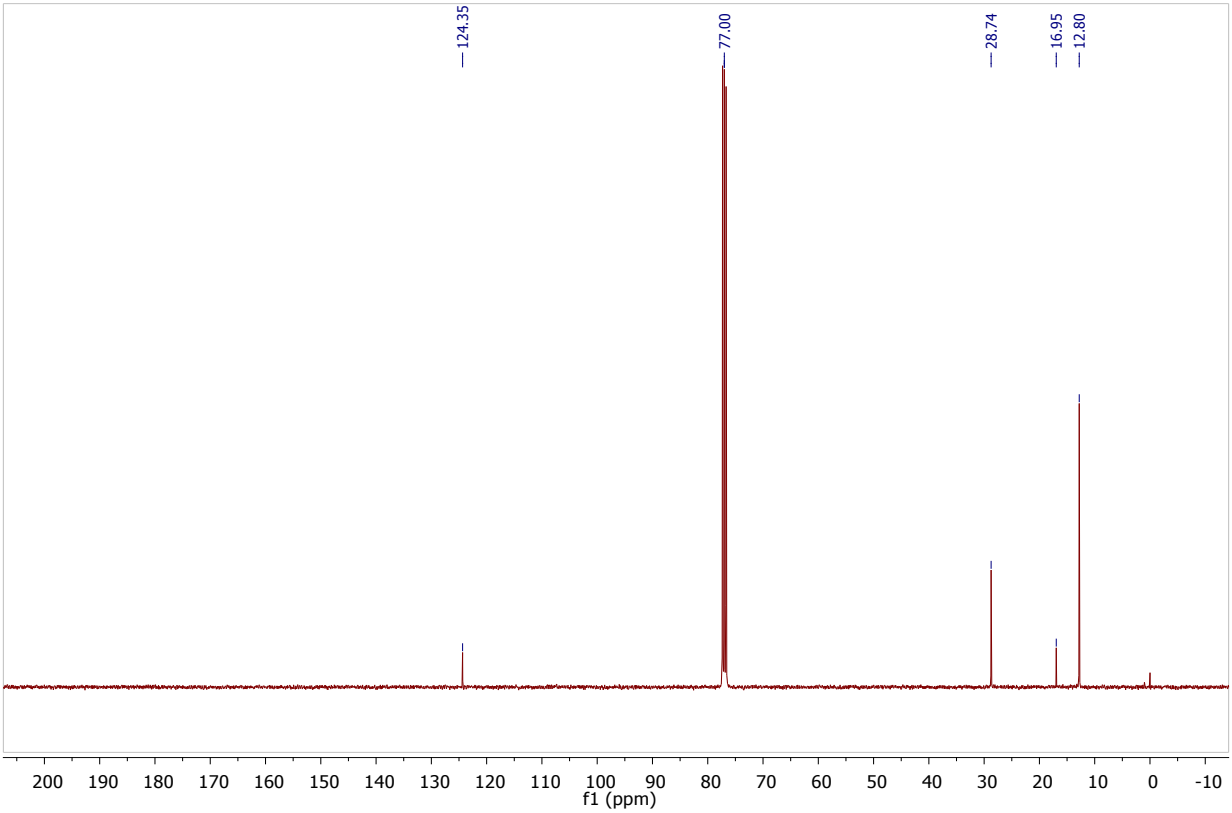
^{13}C NMR Spectrum of compound **2o** in $CDCl_3$



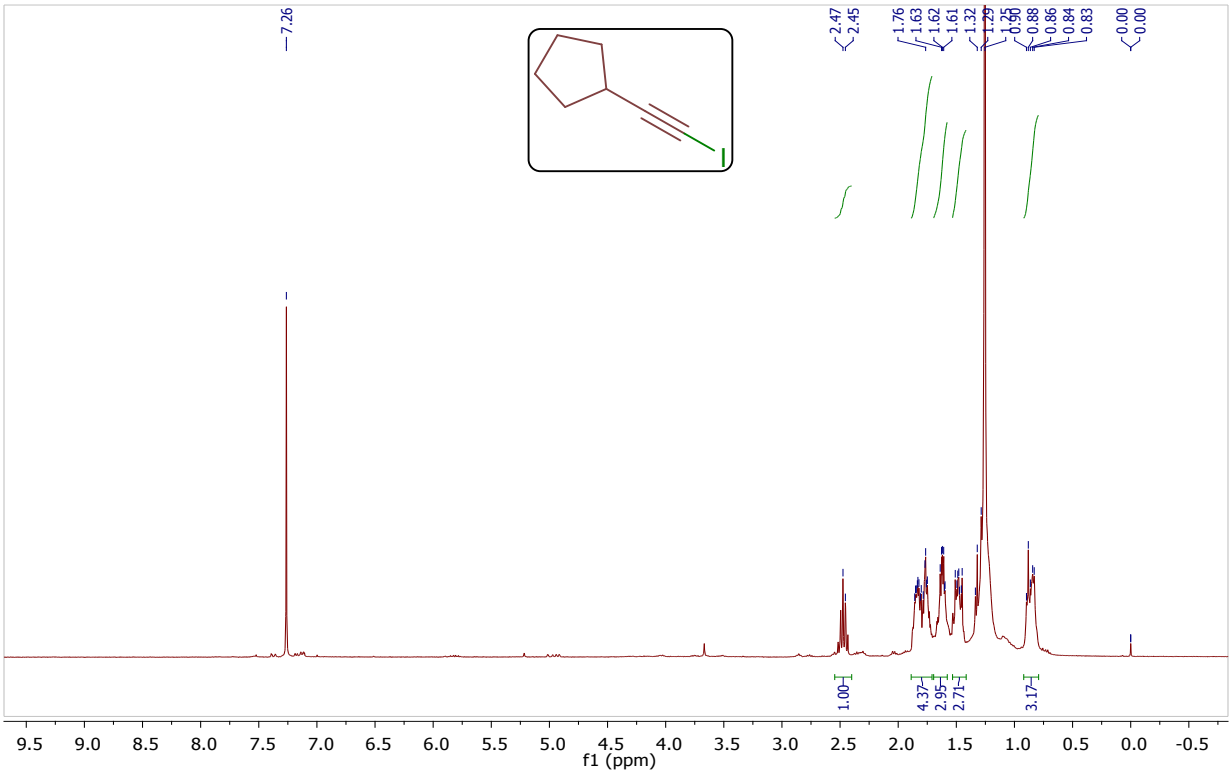
^{1}H NMR Spectrum of compound **2p** in $CDCl_3$



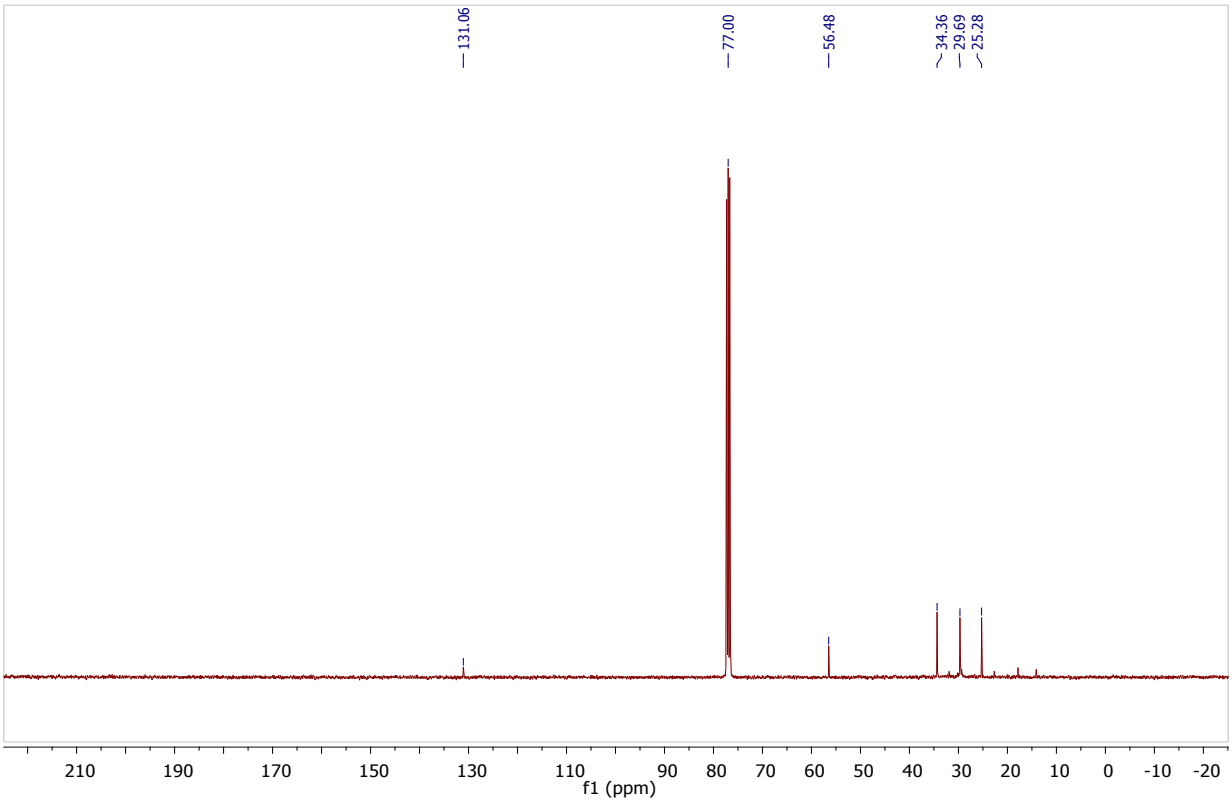
^{13}C NMR Spectrum of compound **2p** in $CDCl_3$



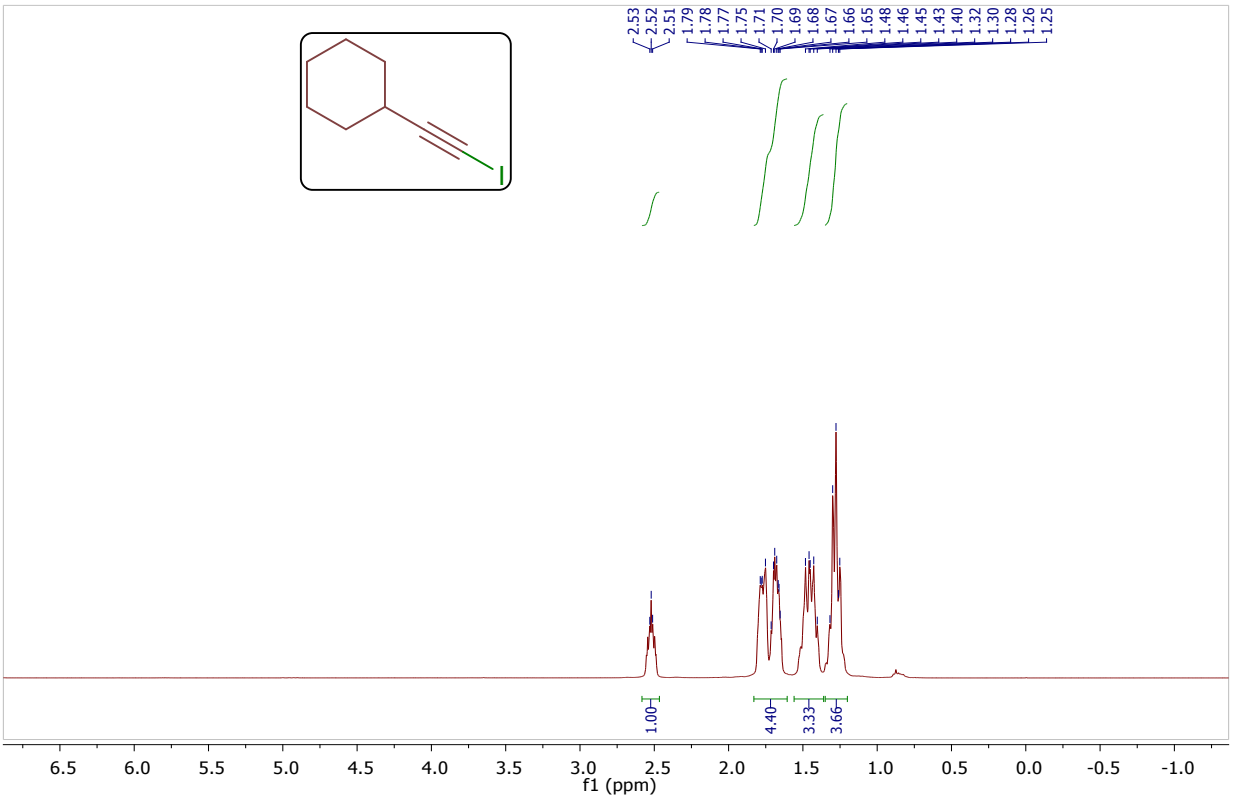
^{1}H NMR Spectrum of compound **2q** in $CDCl_3$



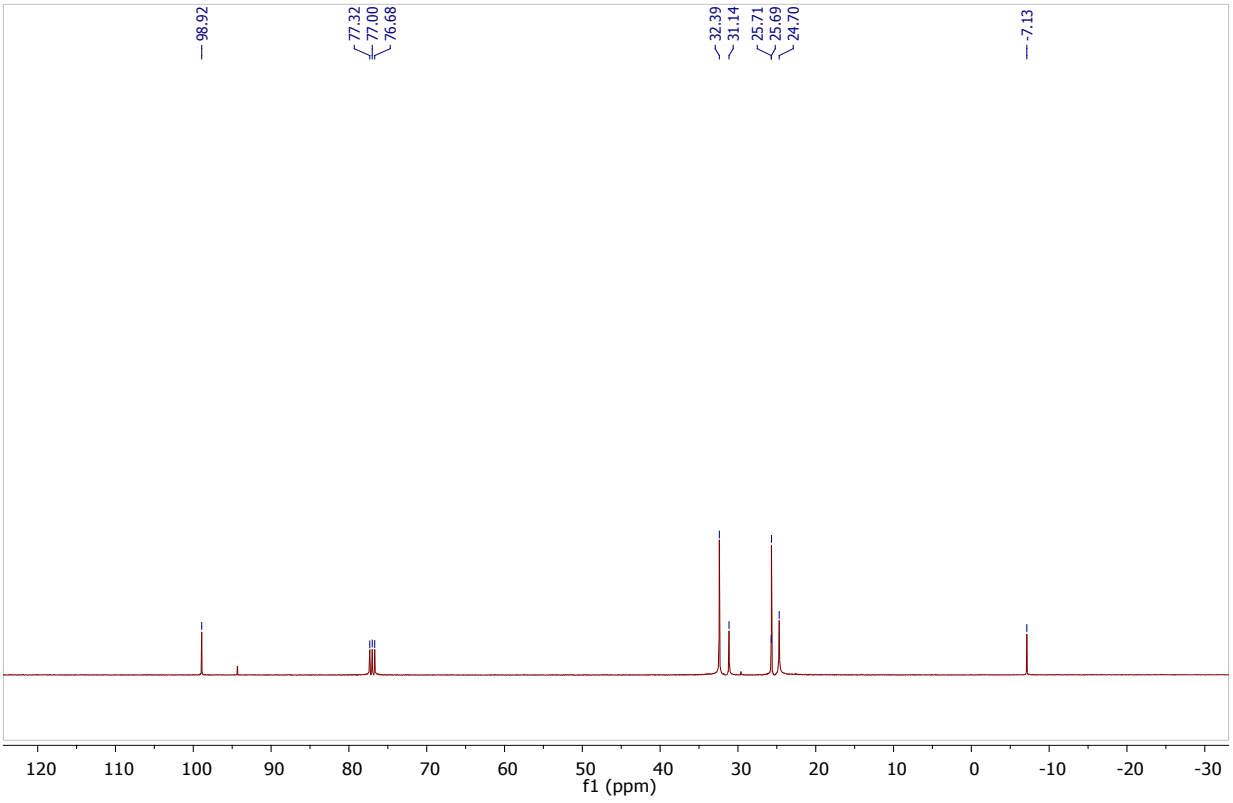
^{13}C NMR Spectrum of compound **2q** in $CDCl_3$



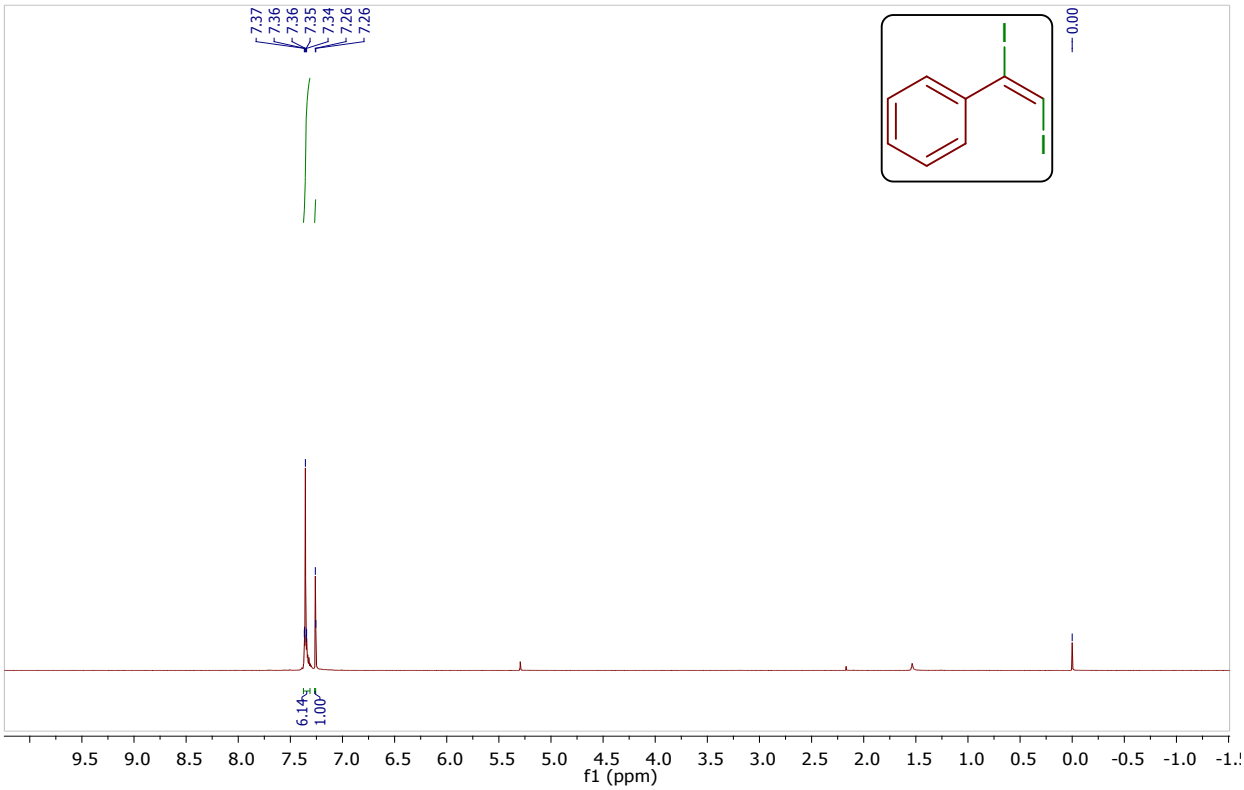
^{1}H NMR Spectrum of compound **2r** in $CDCl_3$



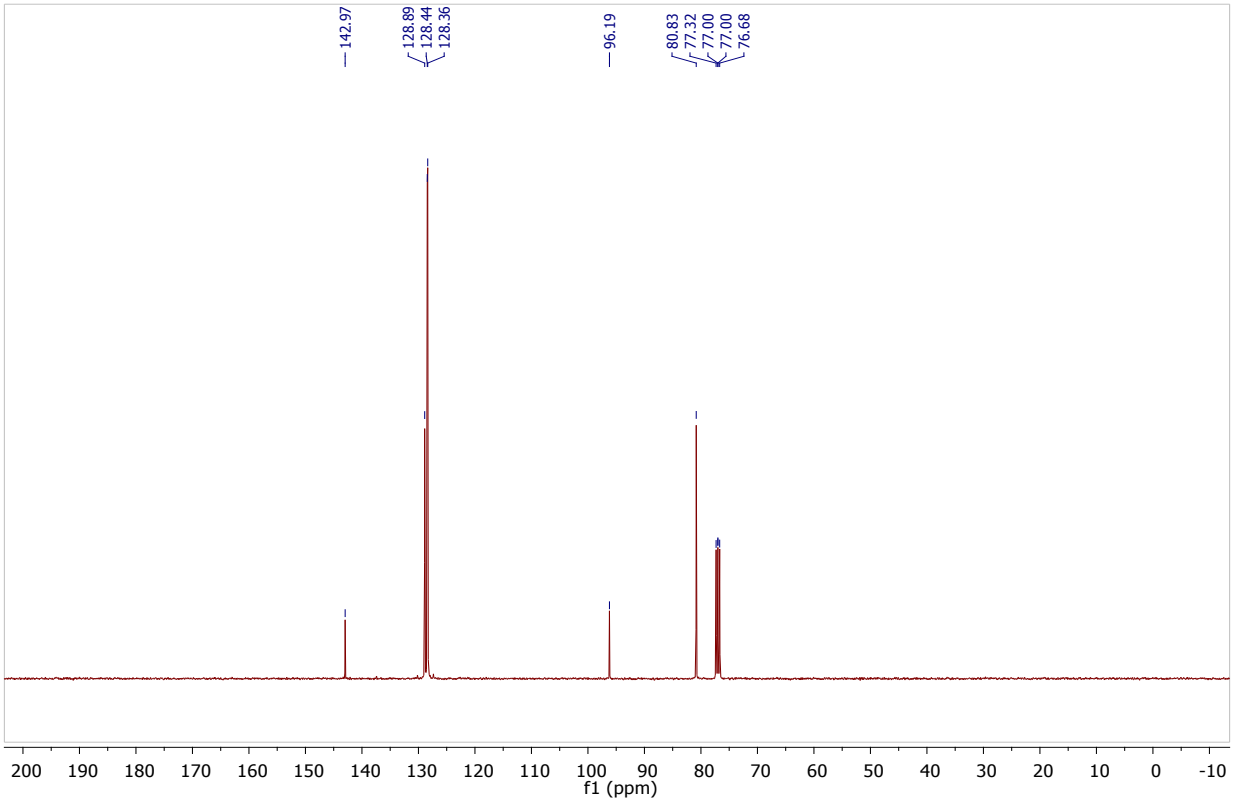
^{13}C NMR Spectrum of compound **2r** in $CDCl_3$



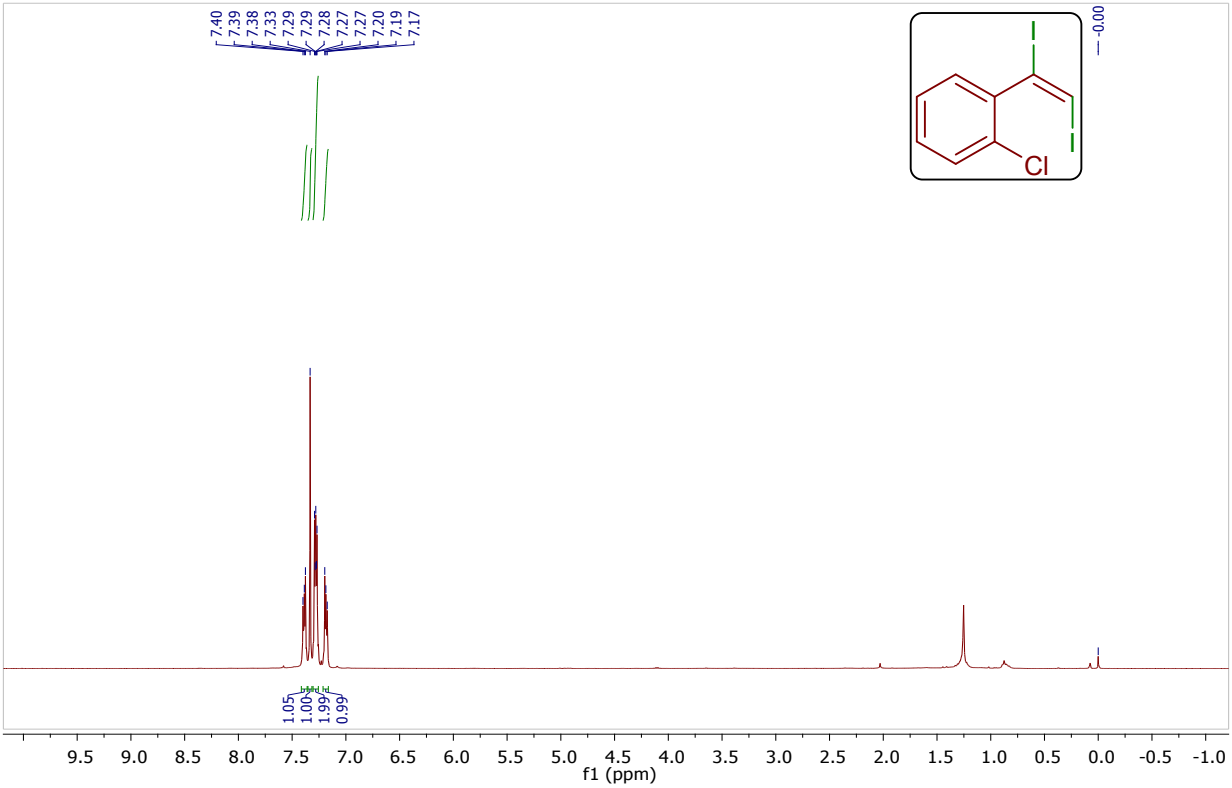
^{1}H NMR Spectrum of compound **3a** in $CDCl_3$



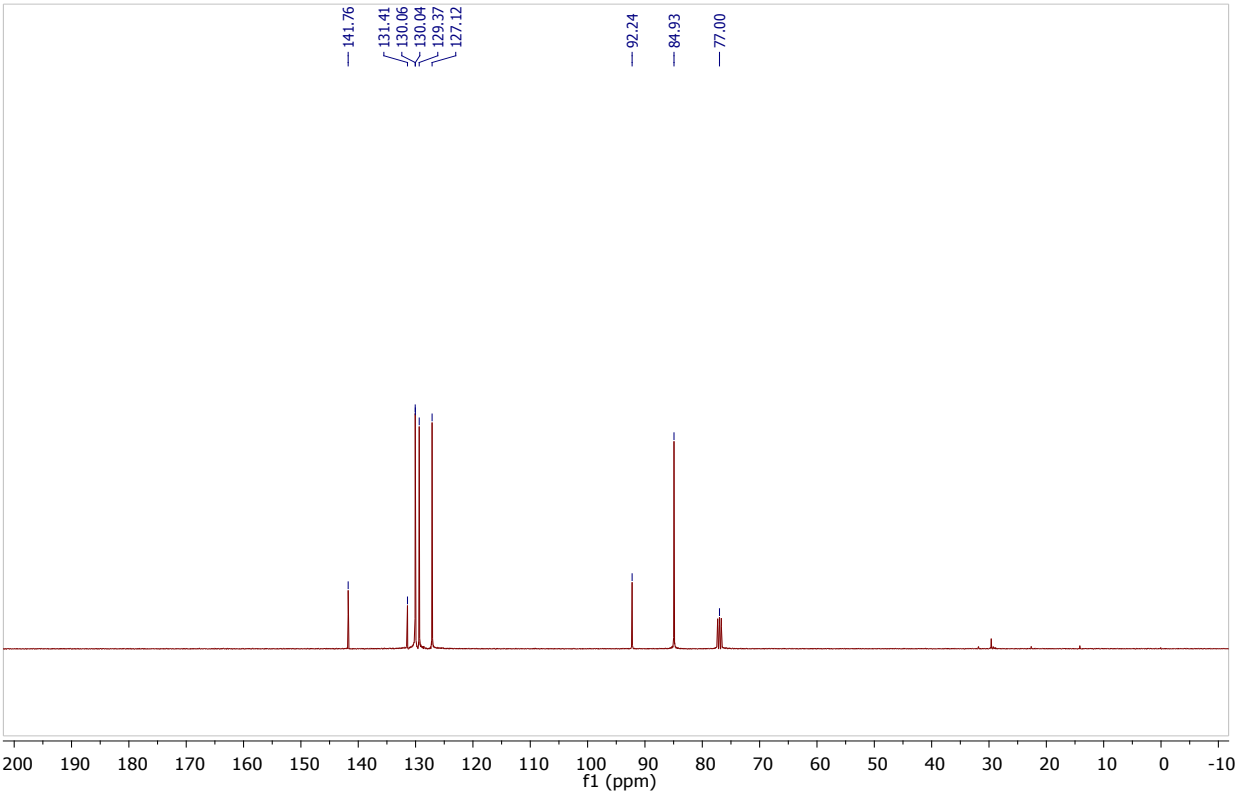
^{13}C NMR Spectrum of compound **3a** in $CDCl_3$



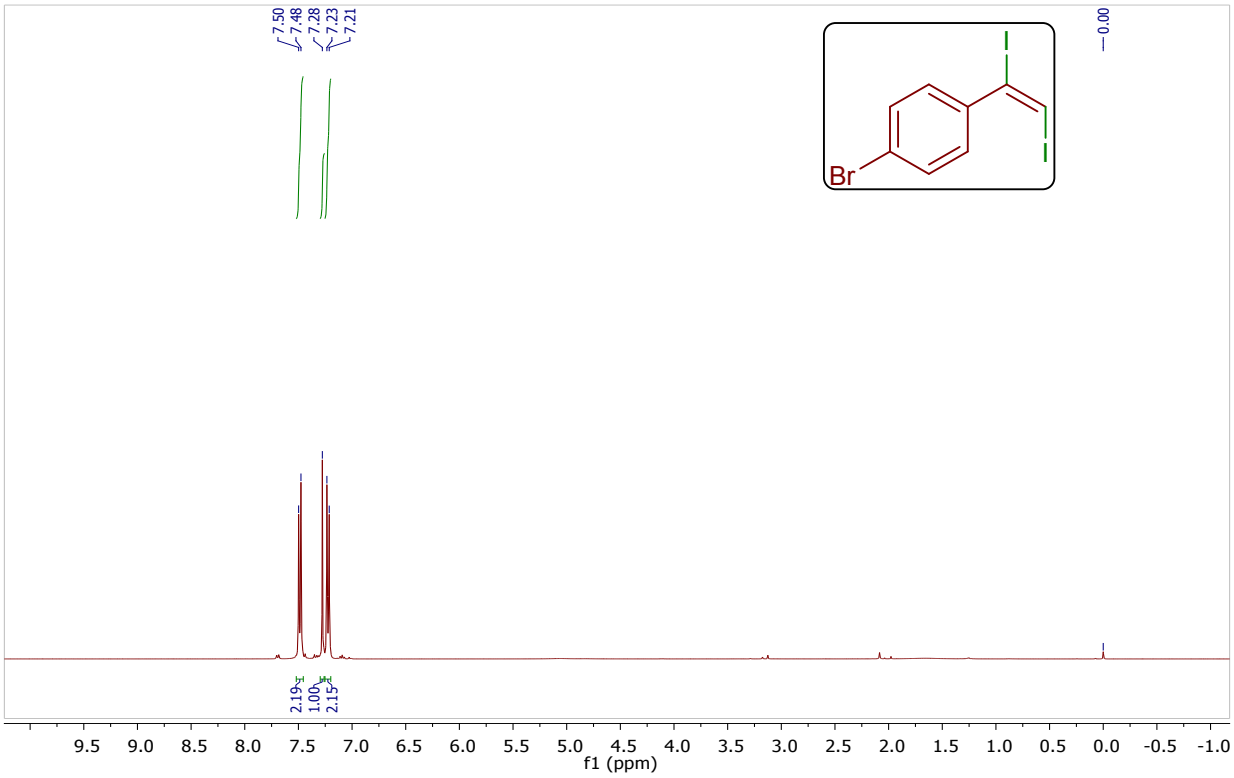
^{1}H NMR Spectrum of compound **3b** in $CDCl_3$



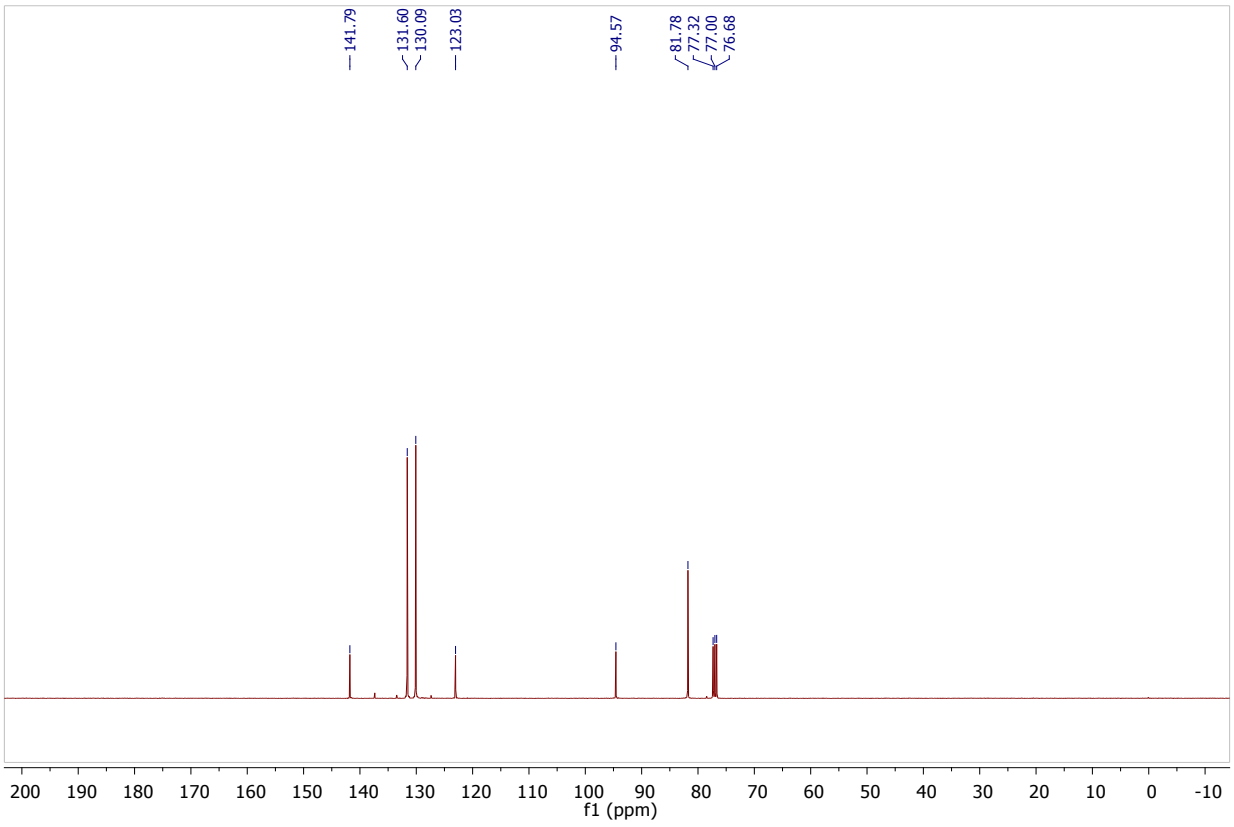
^{13}C NMR Spectrum of compound **3b** in $CDCl_3$



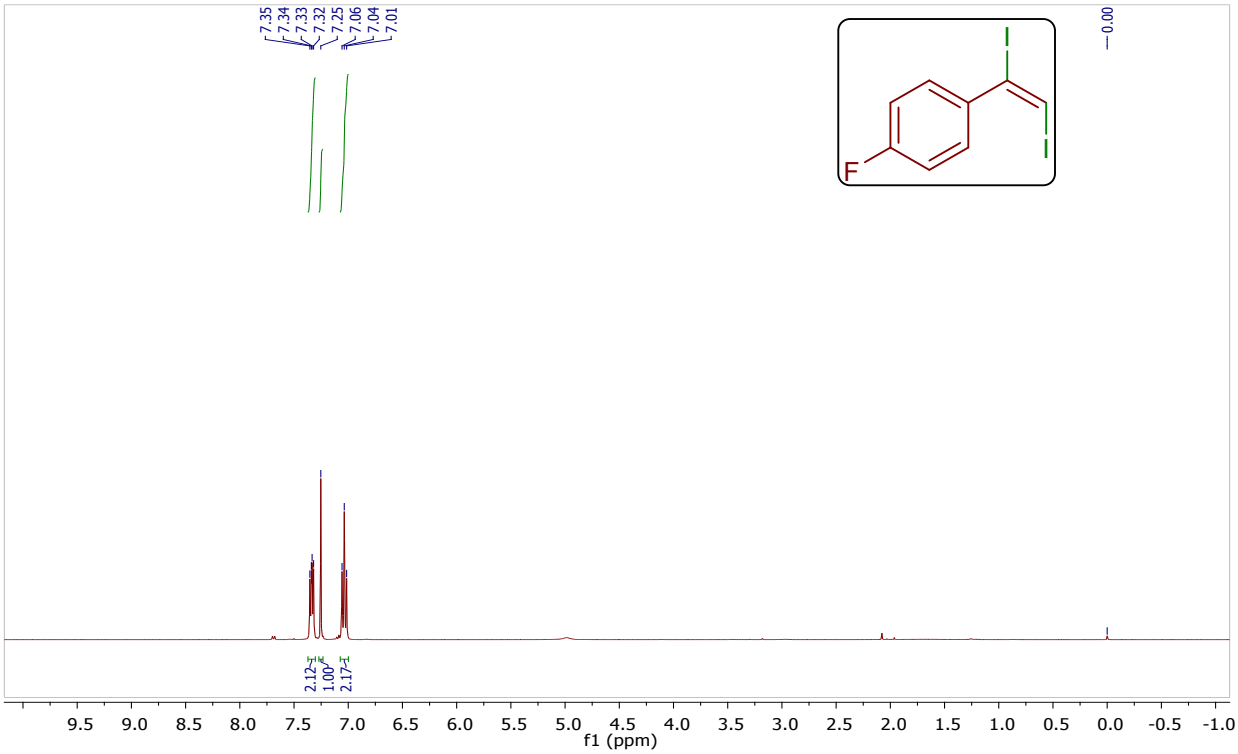
^{1}H NMR Spectrum of compound **3c** in $CDCl_3$



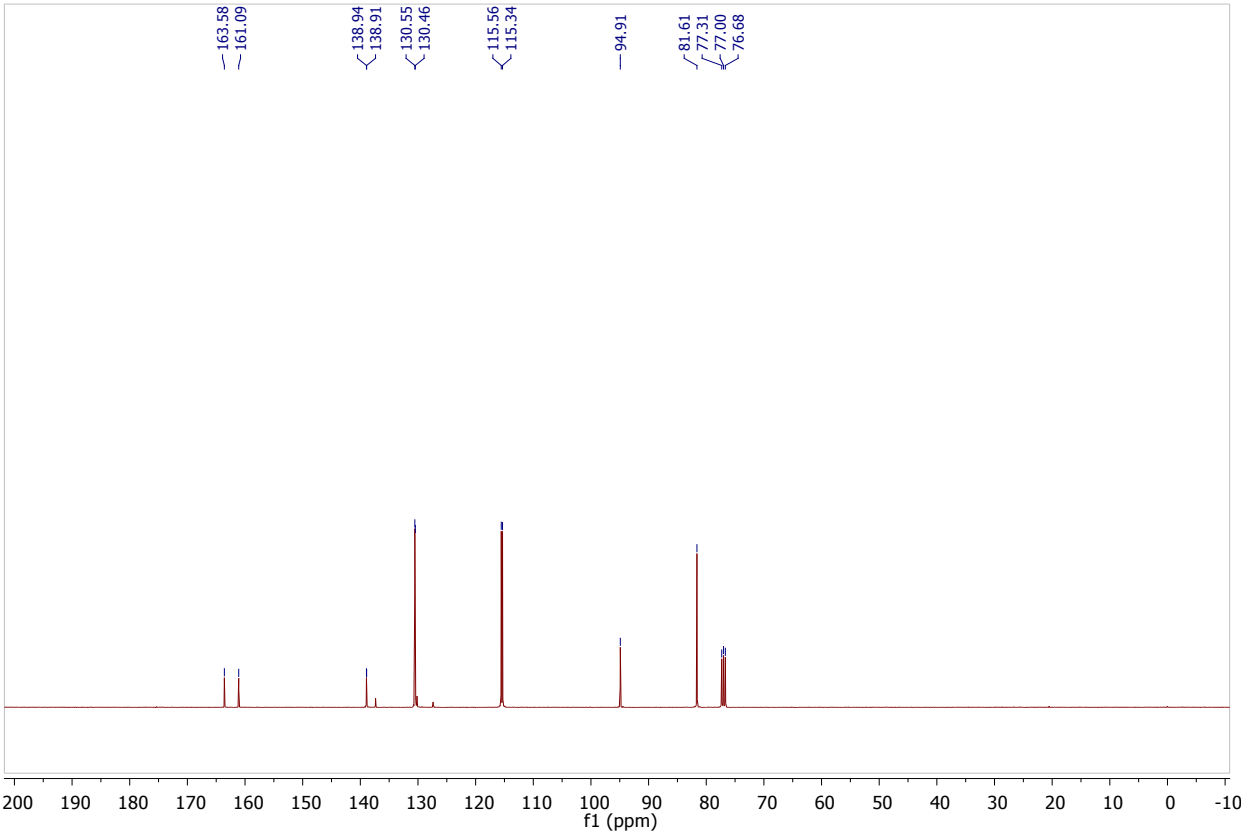
^{13}C NMR Spectrum of compound **3c** in $CDCl_3$



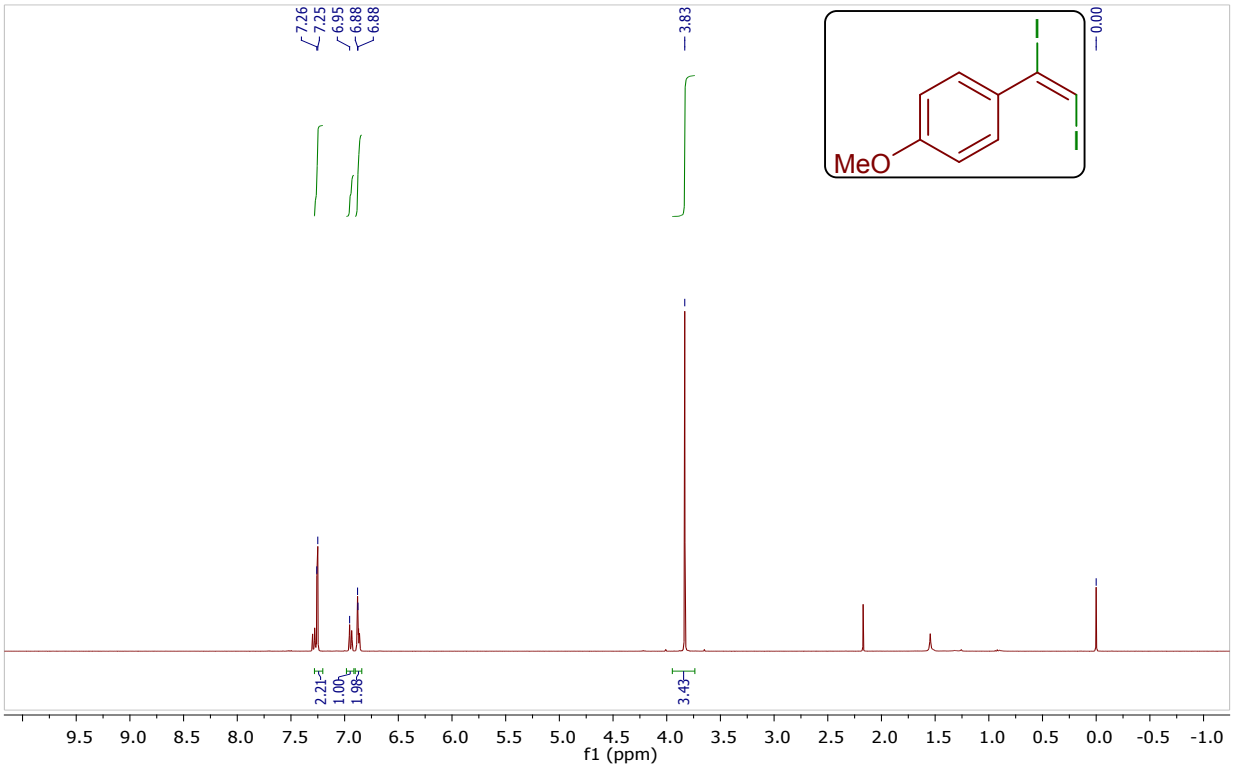
^{1}H NMR Spectrum of compound **3d** in $CDCl_3$



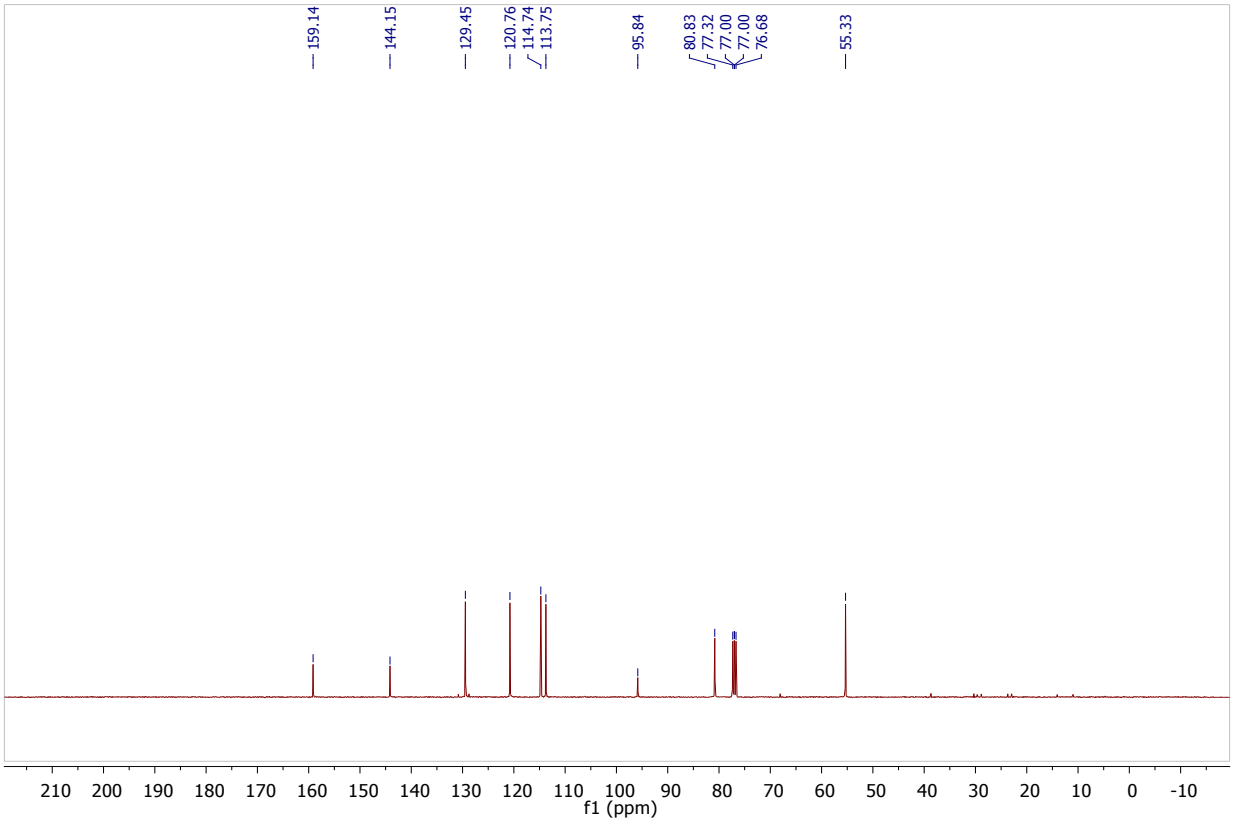
^{13}C NMR Spectrum of compound **3d** in $CDCl_3$



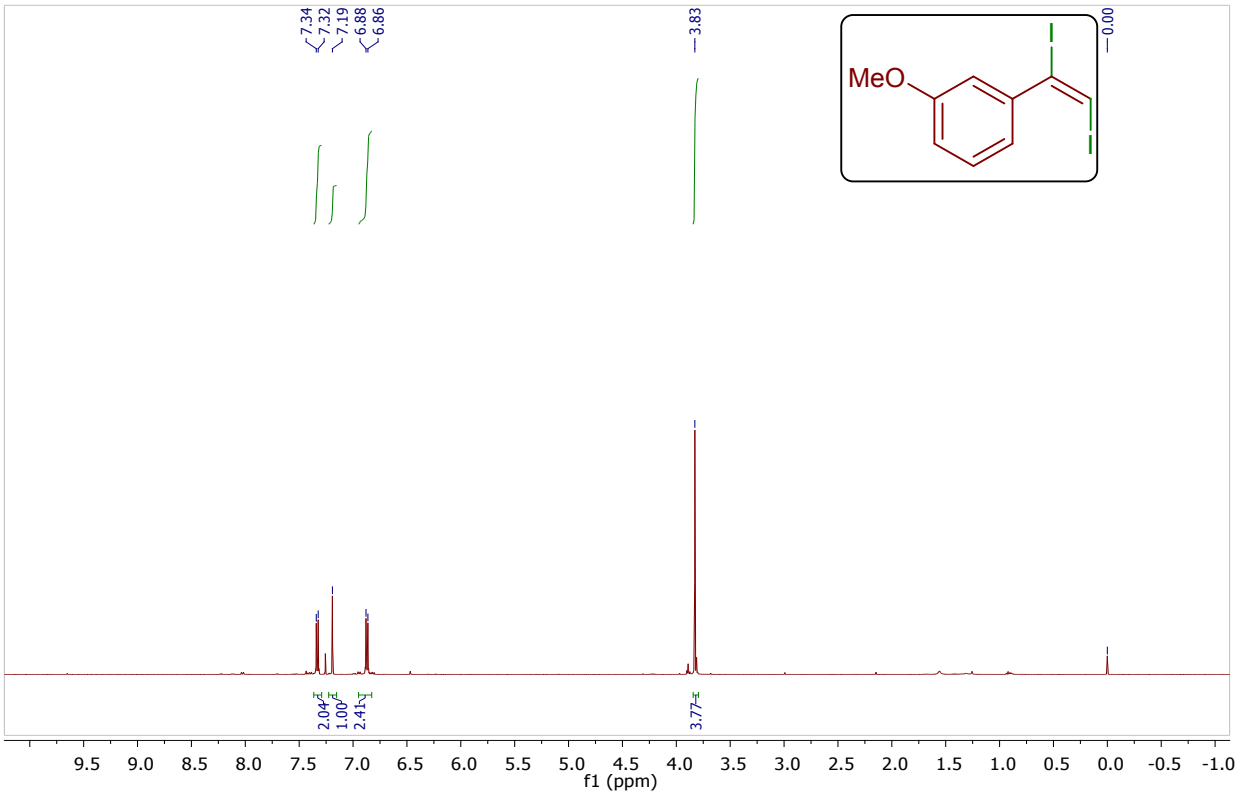
^{1}H NMR Spectrum of compound **3e** in $CDCl_3$



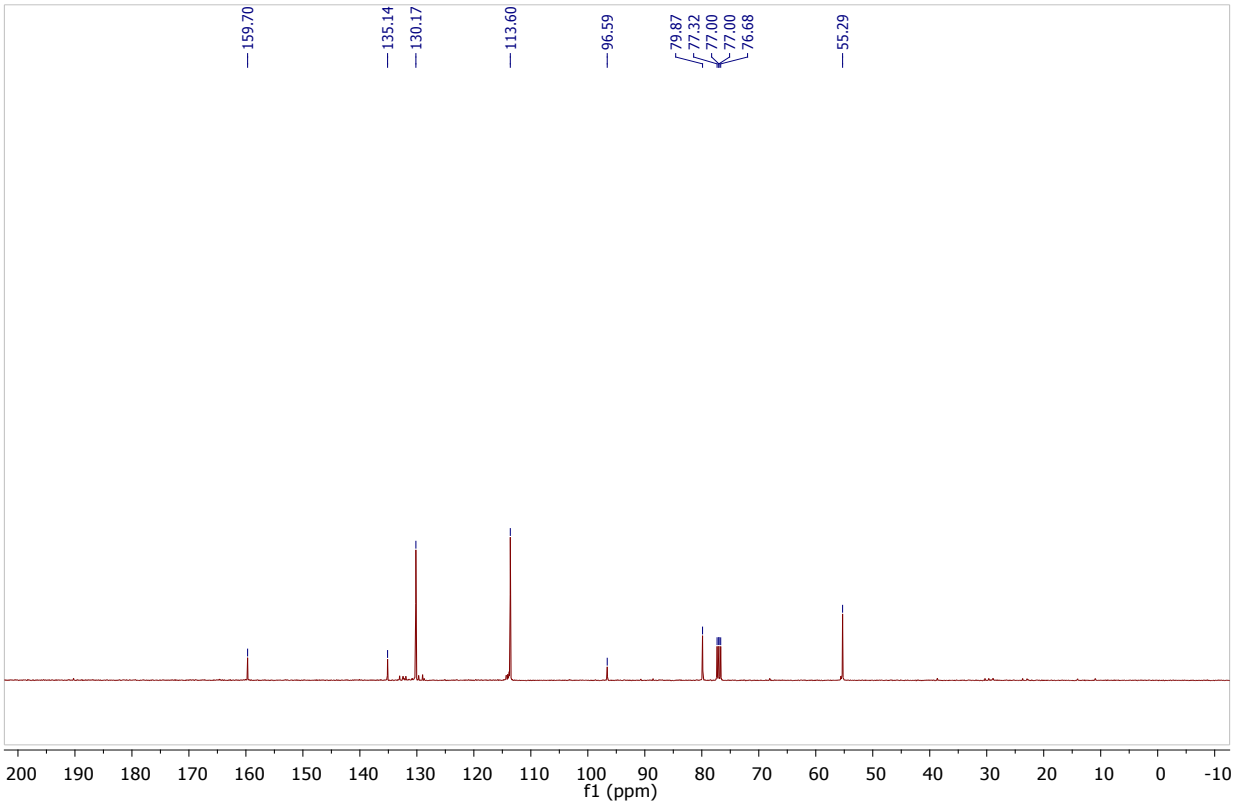
^{13}C NMR Spectrum of compound **3e** in $CDCl_3$



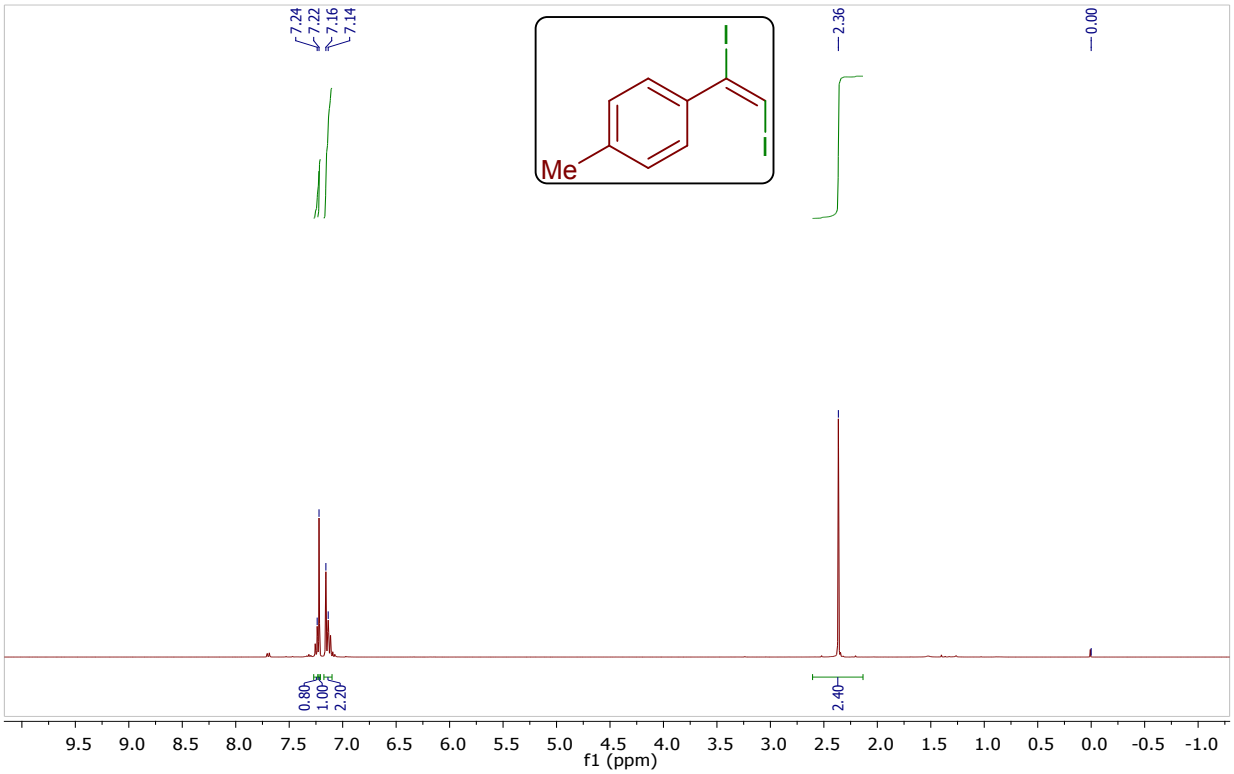
^{1}H NMR Spectrum of compound **3f** in $CDCl_3$



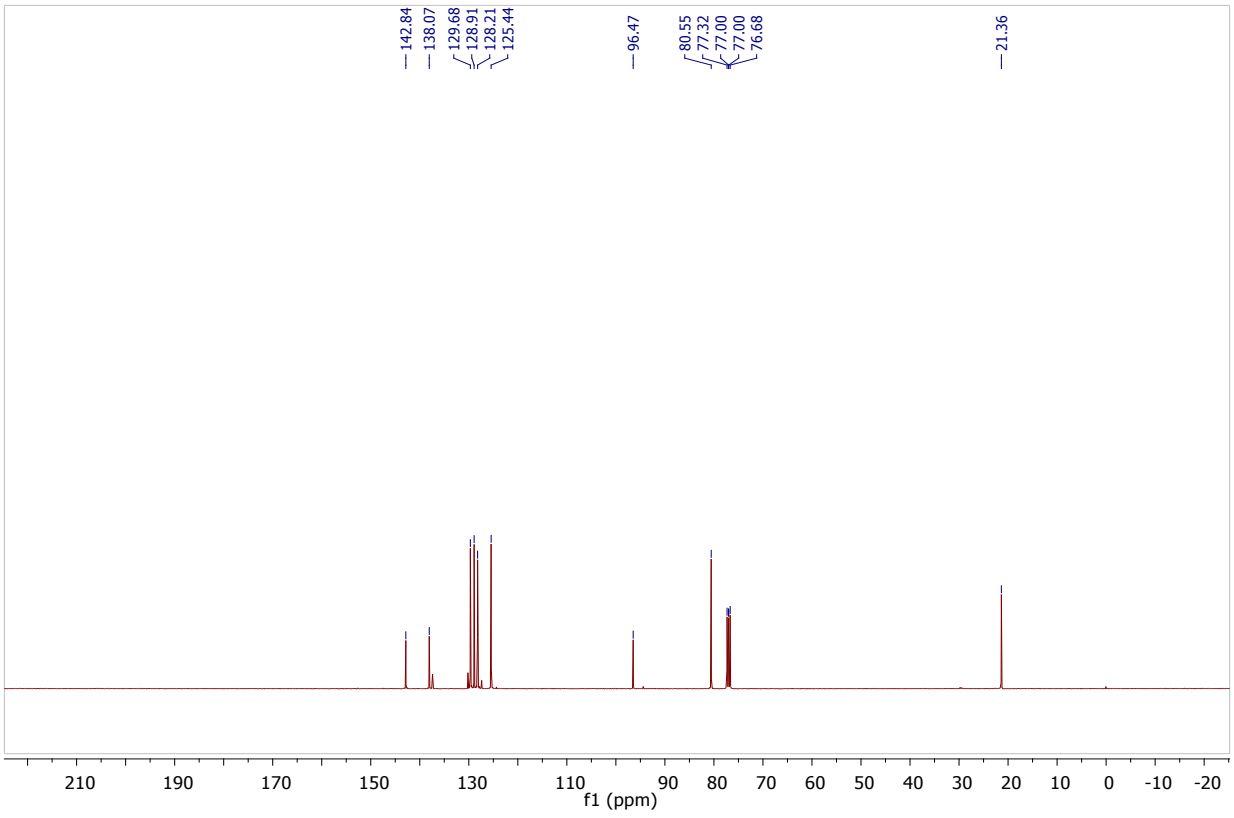
^{13}C NMR Spectrum of compound **3f** in $CDCl_3$



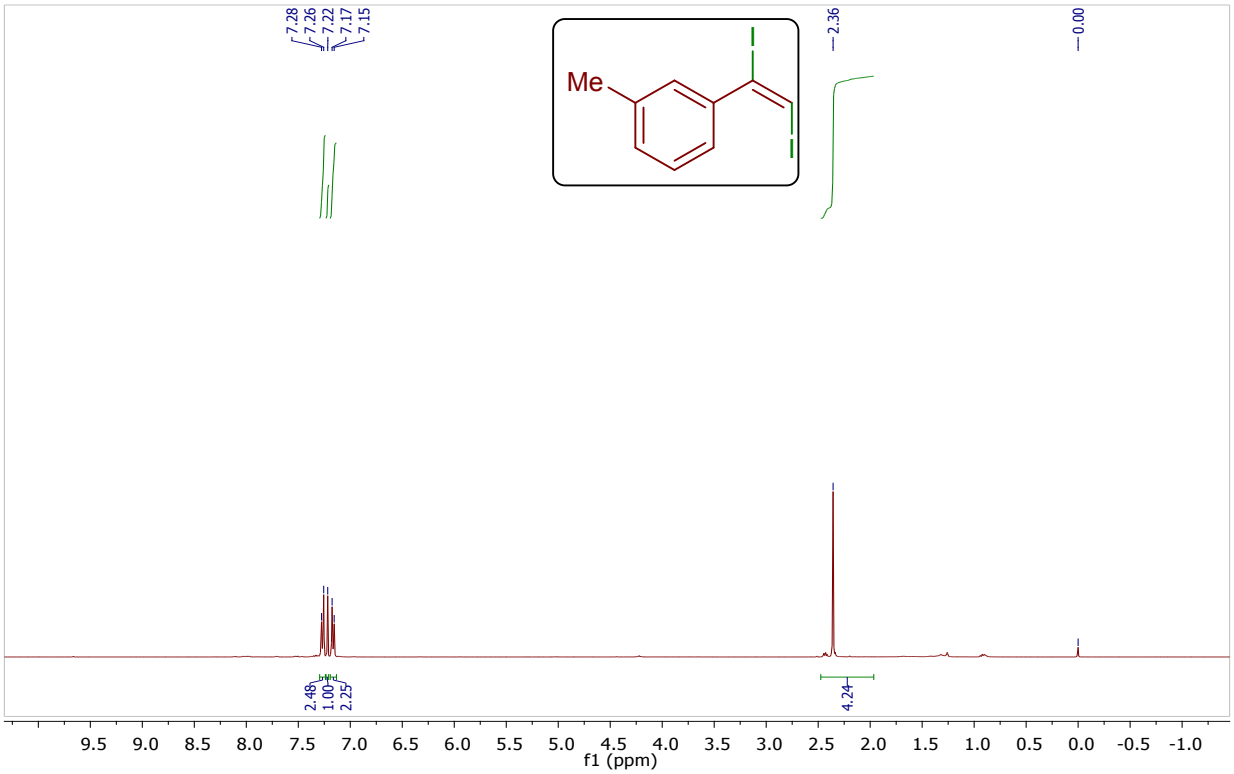
^{1}H NMR Spectrum of compound **3g** in $CDCl_3$



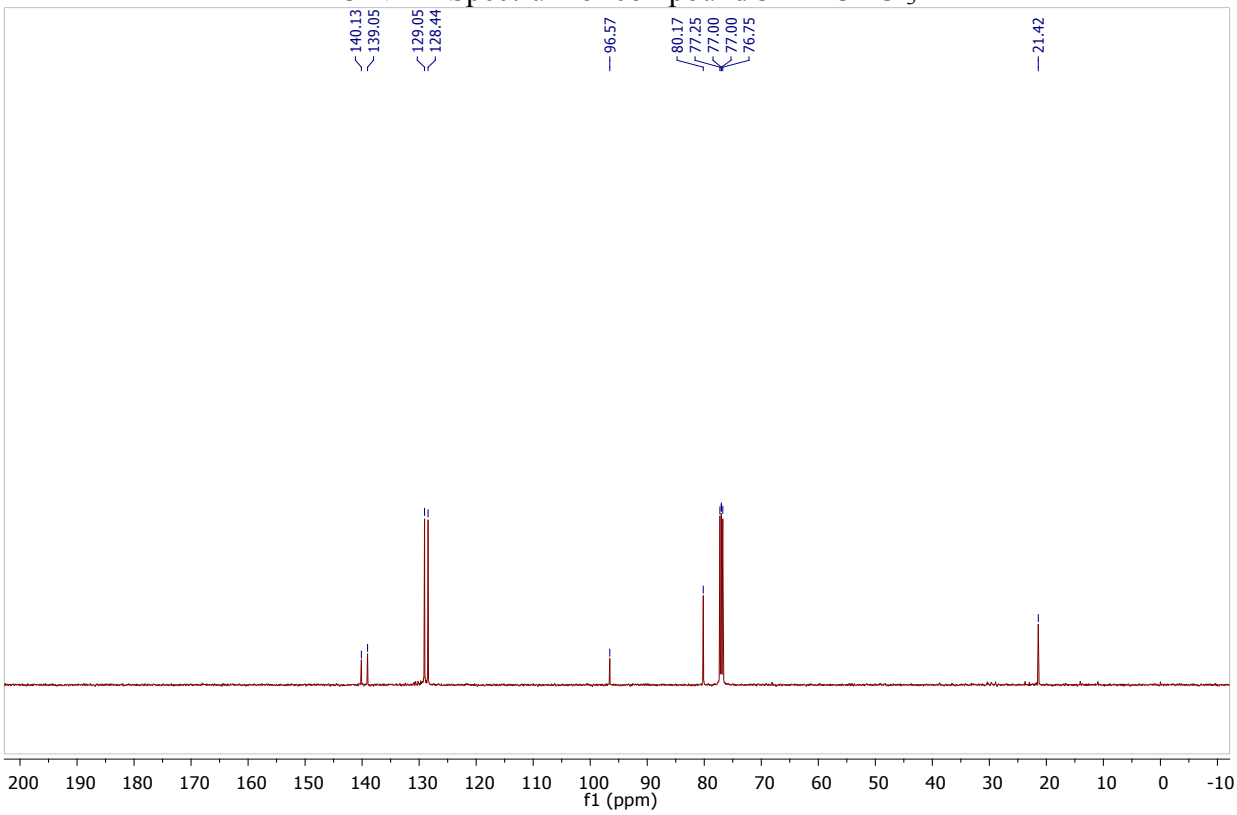
^{13}C NMR Spectrum of compound **3g** in $CDCl_3$



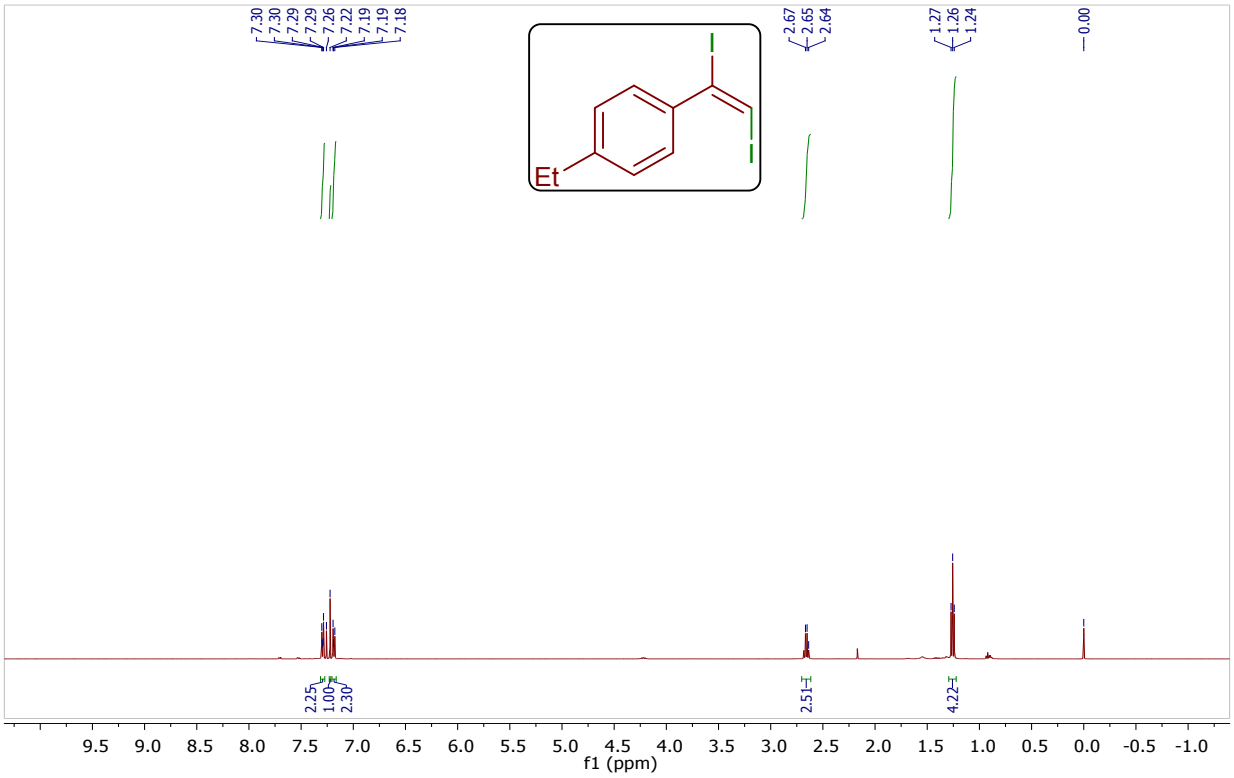
^{1}H NMR Spectrum of compound **3h** in $CDCl_3$



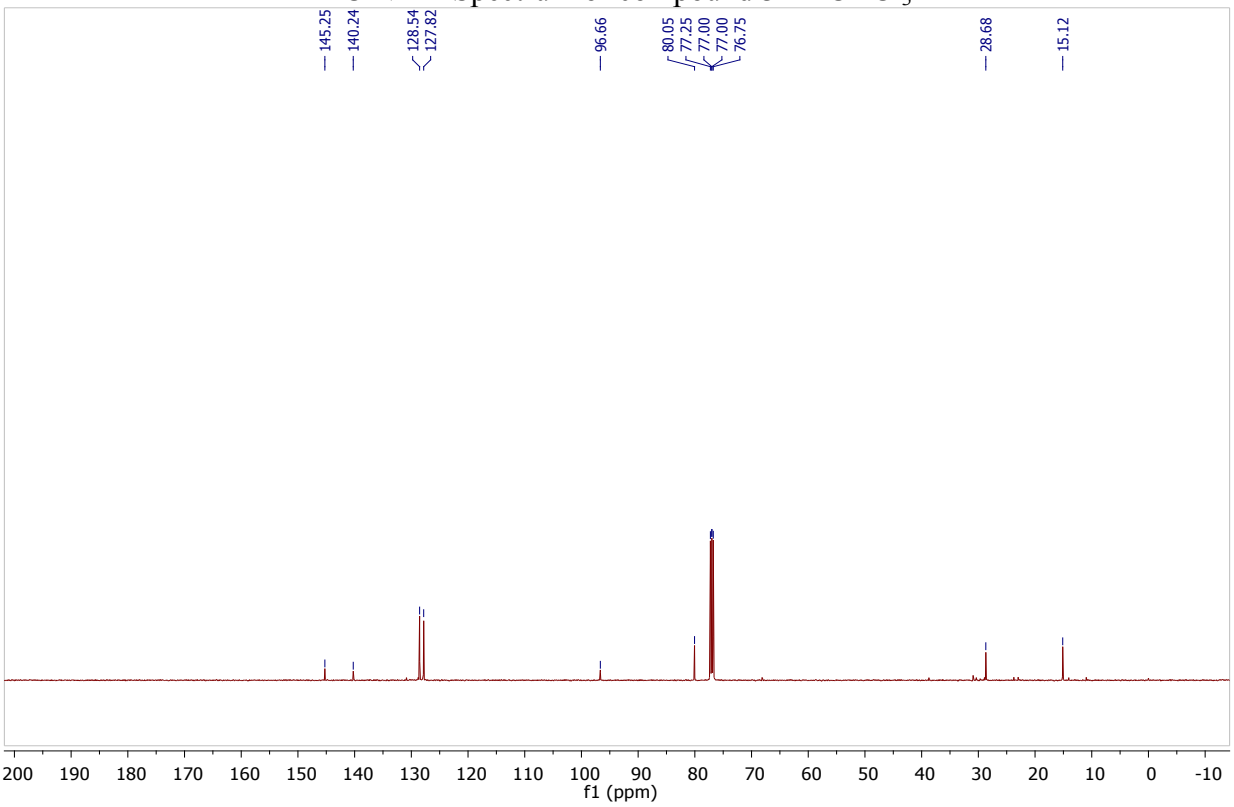
^{13}C NMR Spectrum of compound **3h** in $CDCl_3$



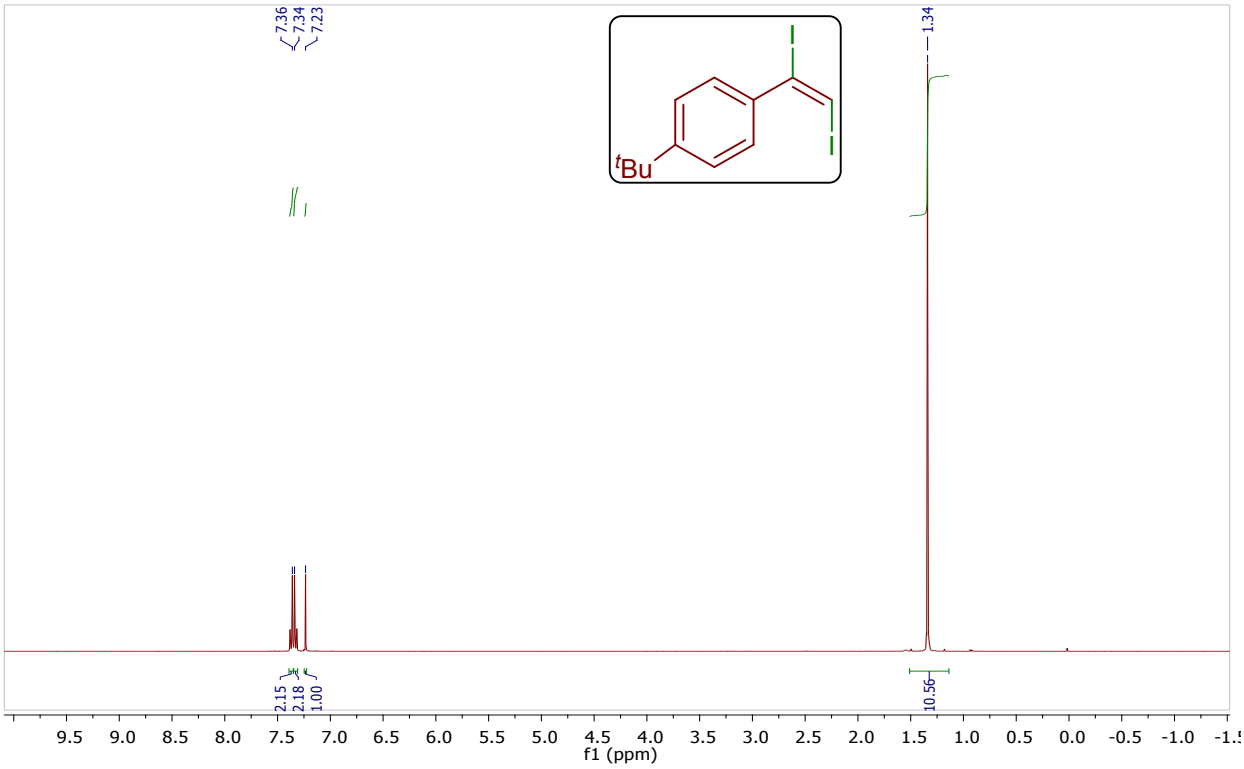
^{1}H NMR Spectrum of compound **3i** in $CDCl_3$



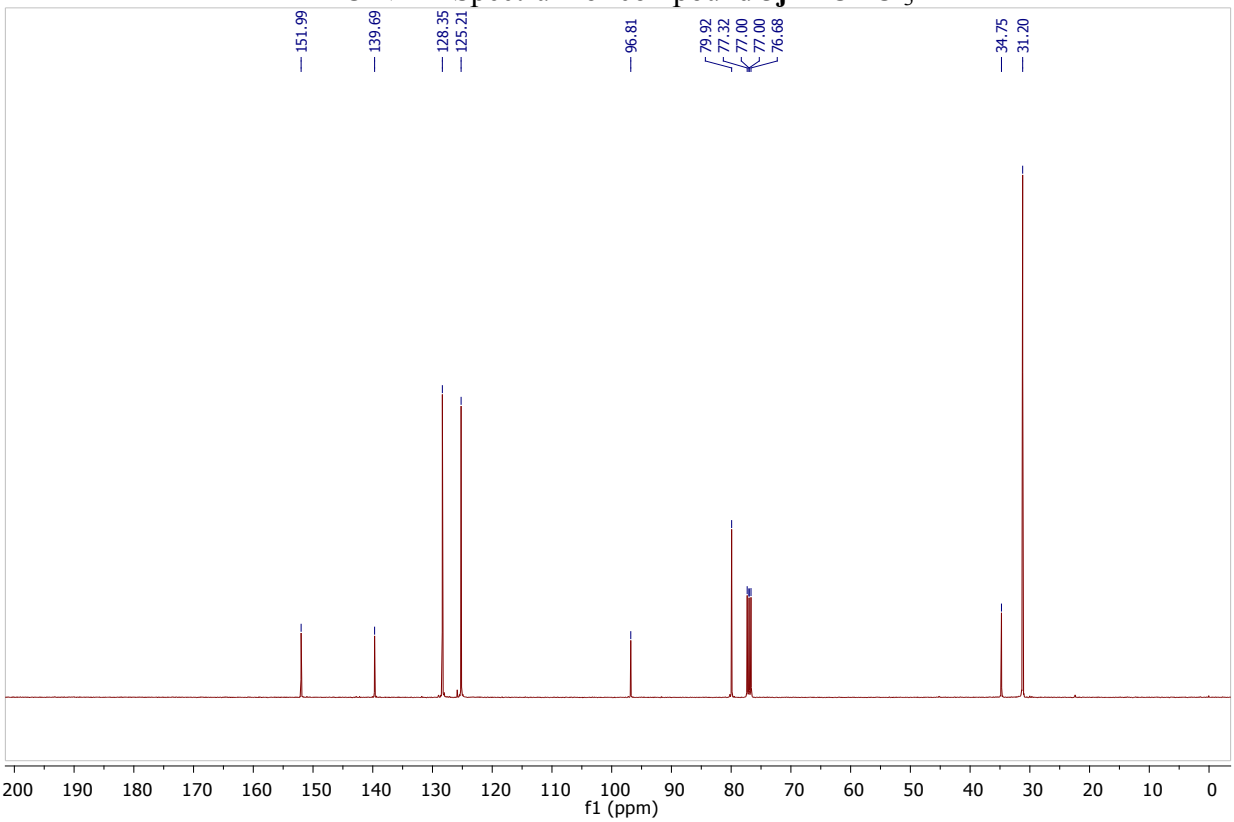
^{13}C NMR Spectrum of compound **3i** in $CDCl_3$



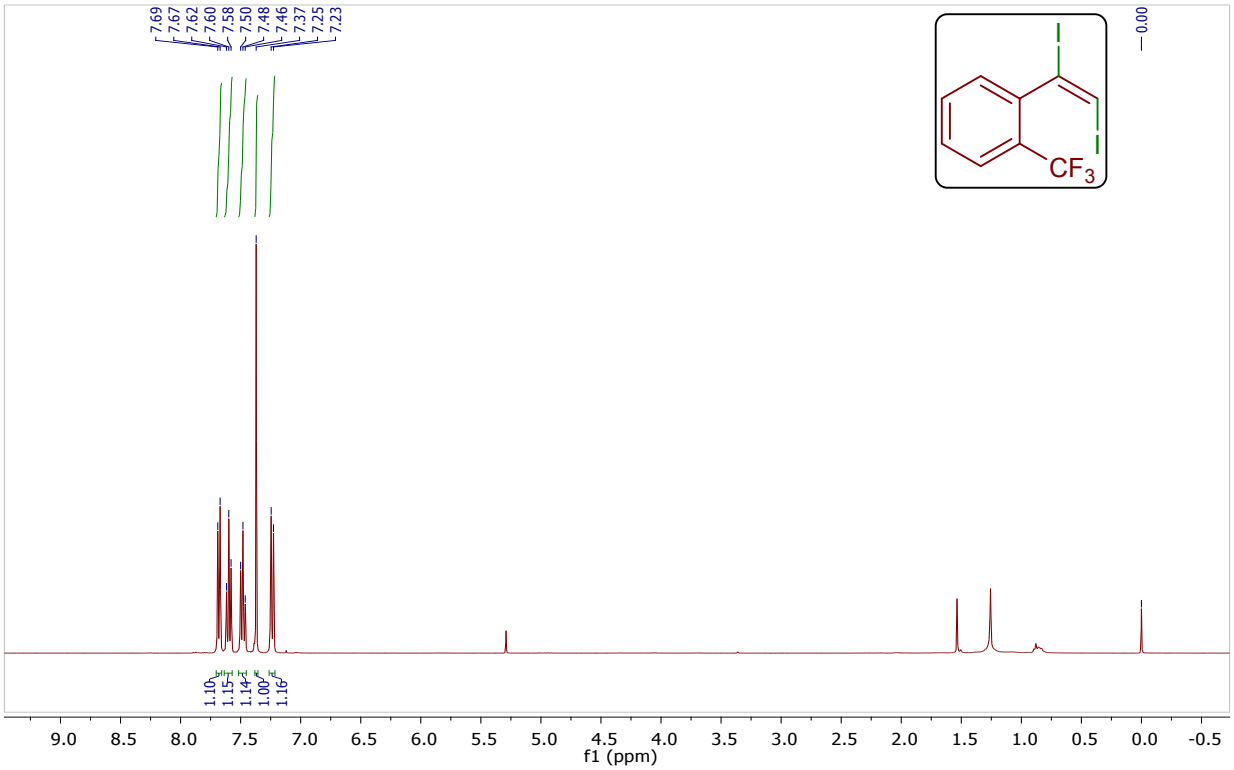
^{1}H NMR Spectrum of compound **3j** in $CDCl_3$



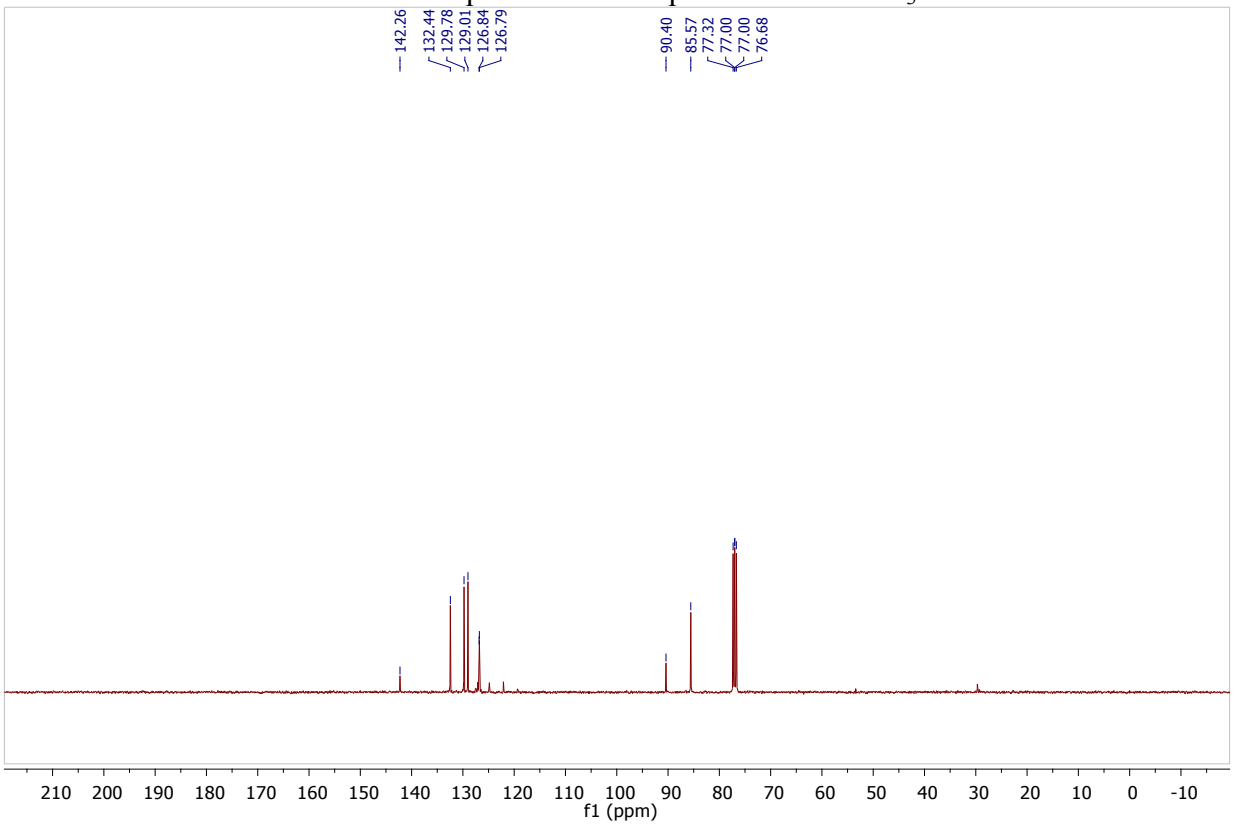
^{13}C NMR Spectrum of compound **3j** in $CDCl_3$



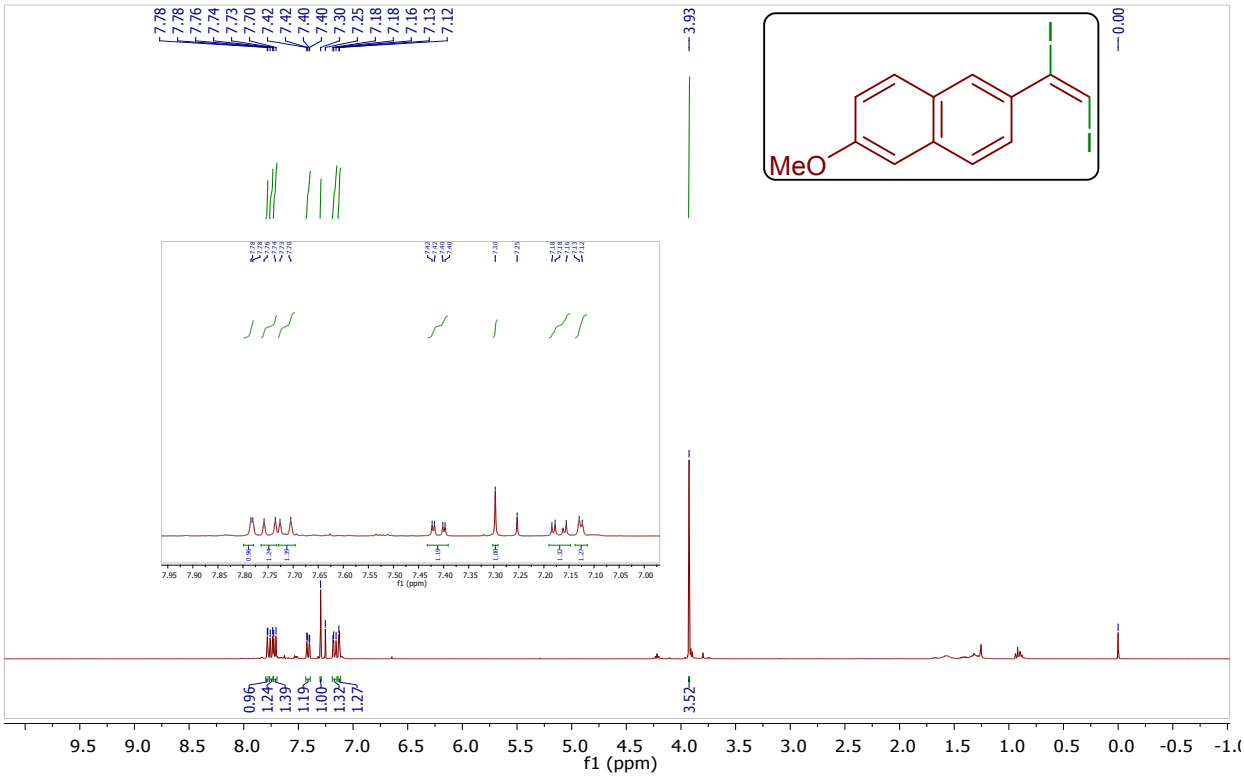
^{1}H NMR Spectrum of compound **3k** in $CDCl_3$



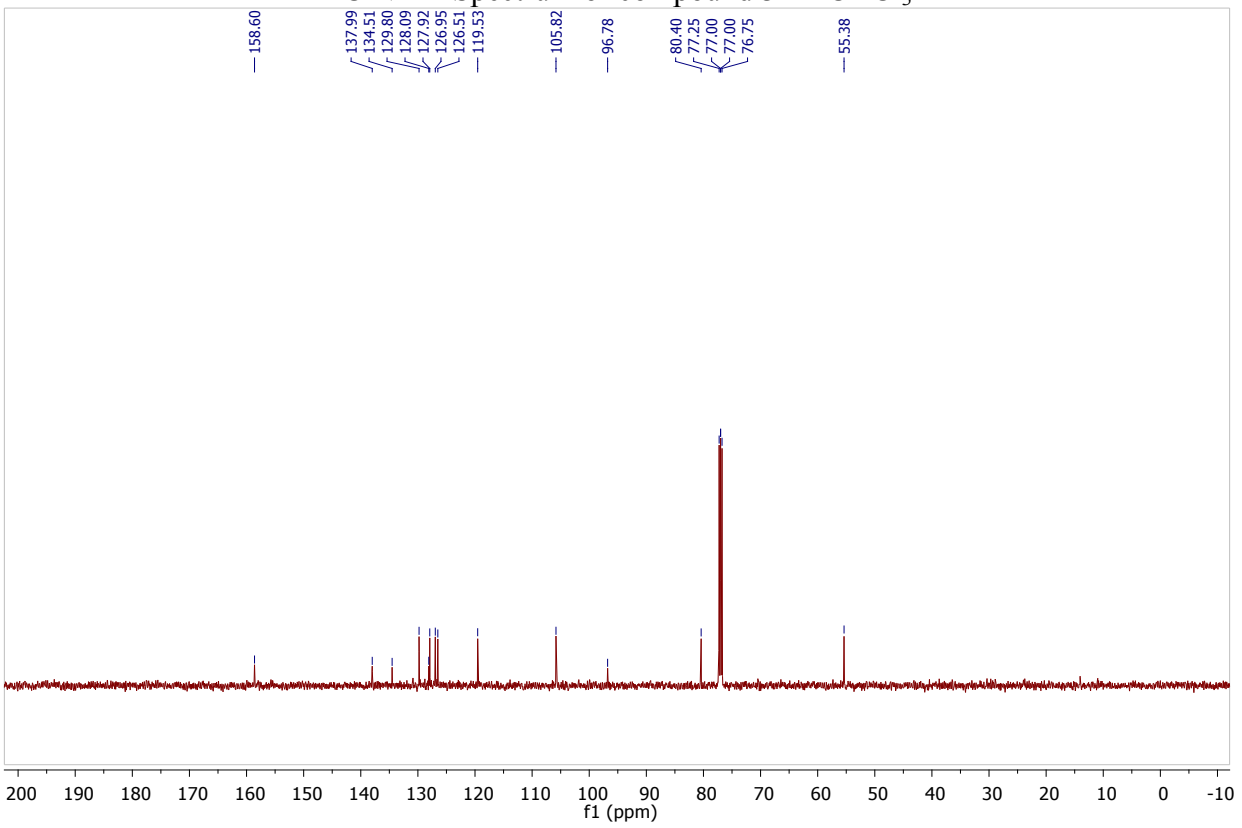
^{13}C NMR Spectrum of compound **3k** in $CDCl_3$



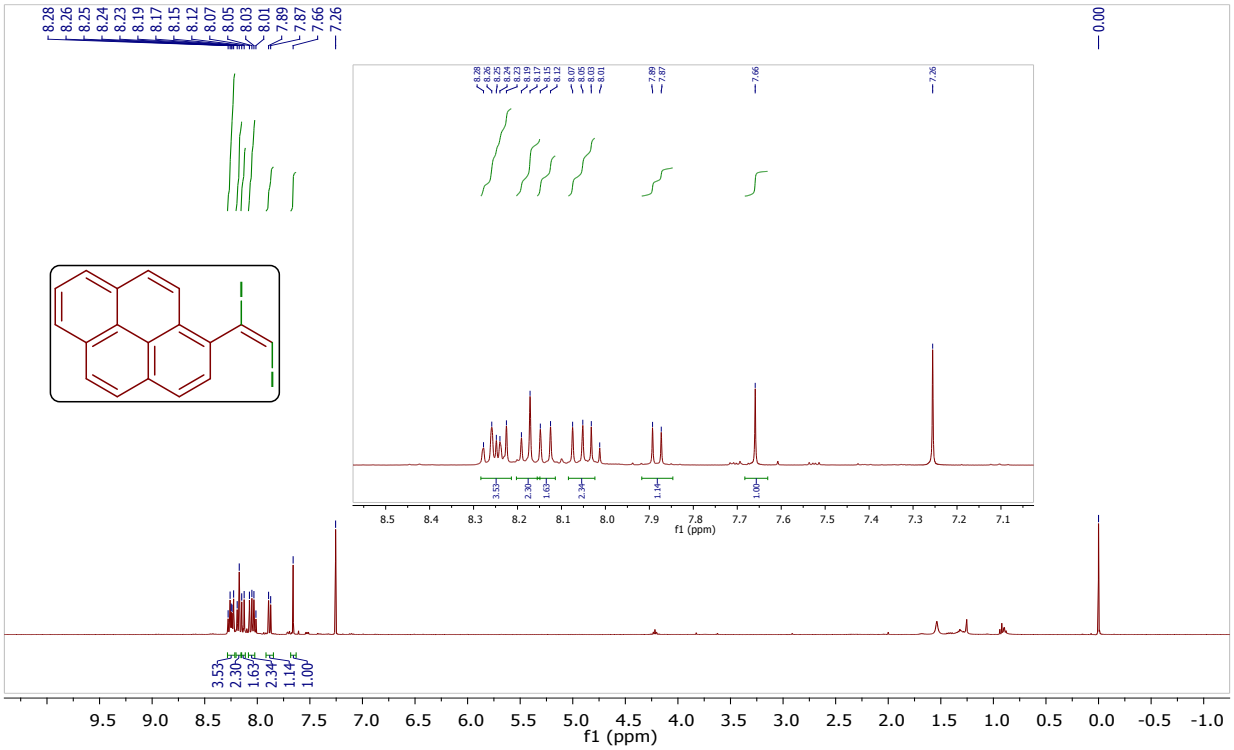
^{1}H NMR Spectrum of compound **3l** in $CDCl_3$



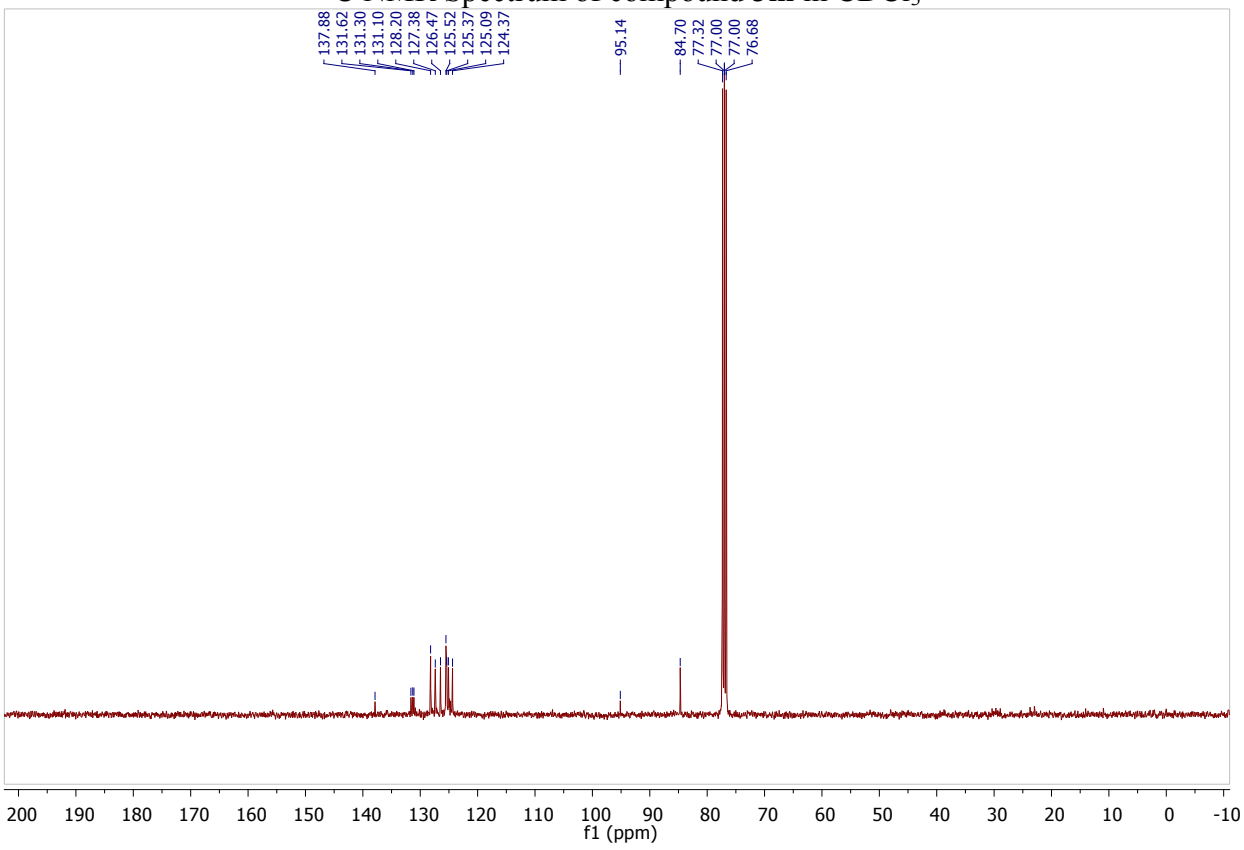
^{13}C NMR Spectrum of compound **3l** in $CDCl_3$



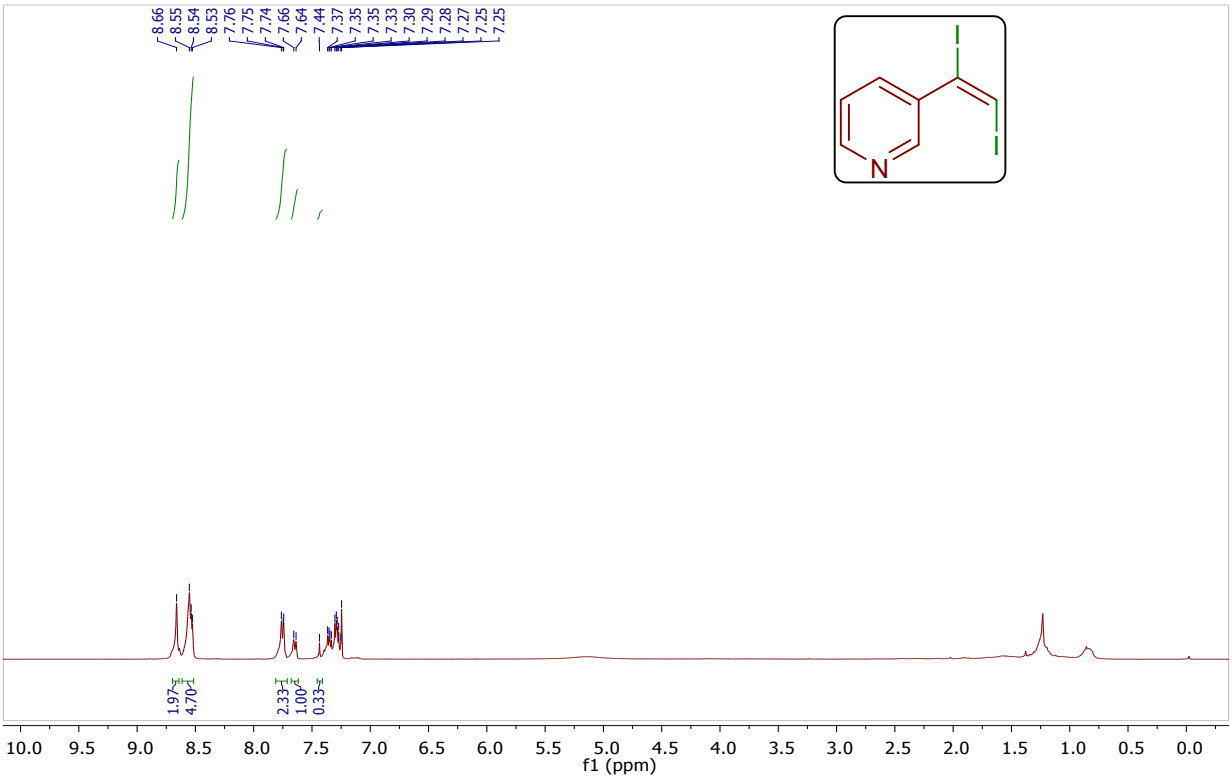
^{1}H NMR Spectrum of compound **3m** in $CDCl_3$



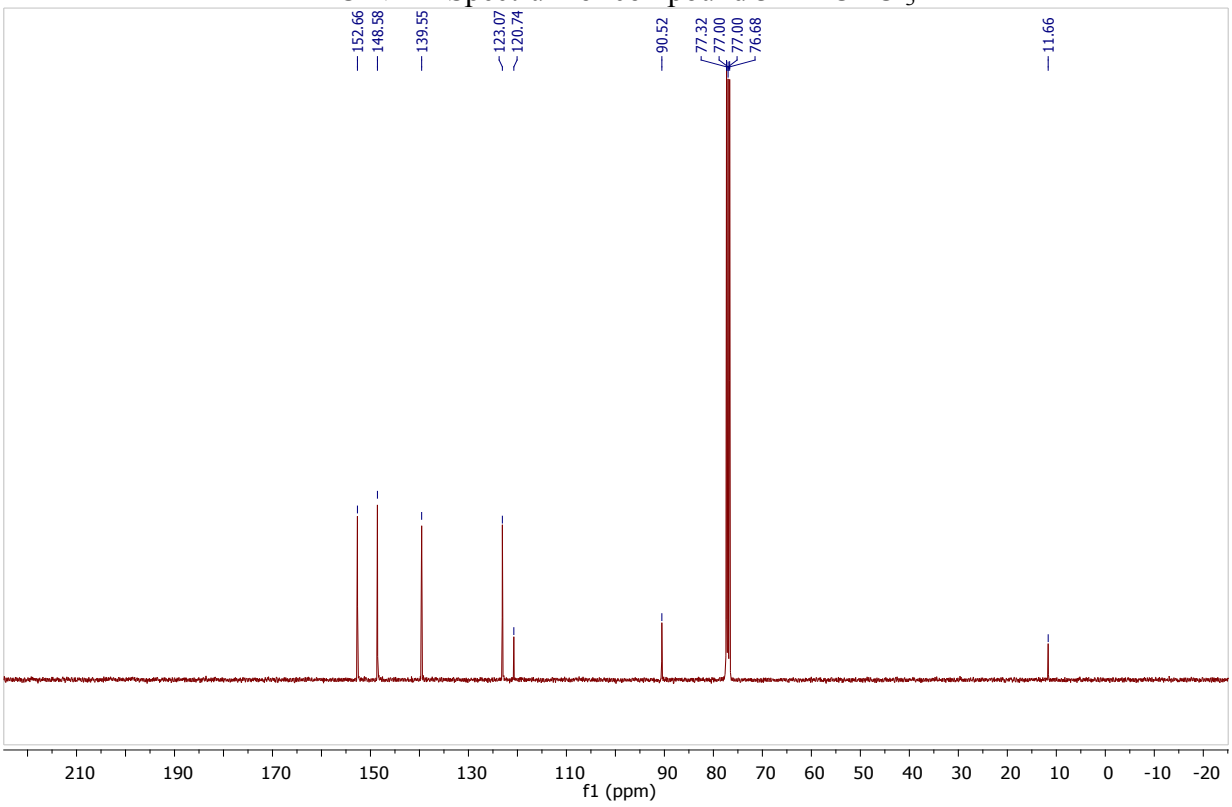
^{13}C NMR Spectrum of compound **3m** in $CDCl_3$



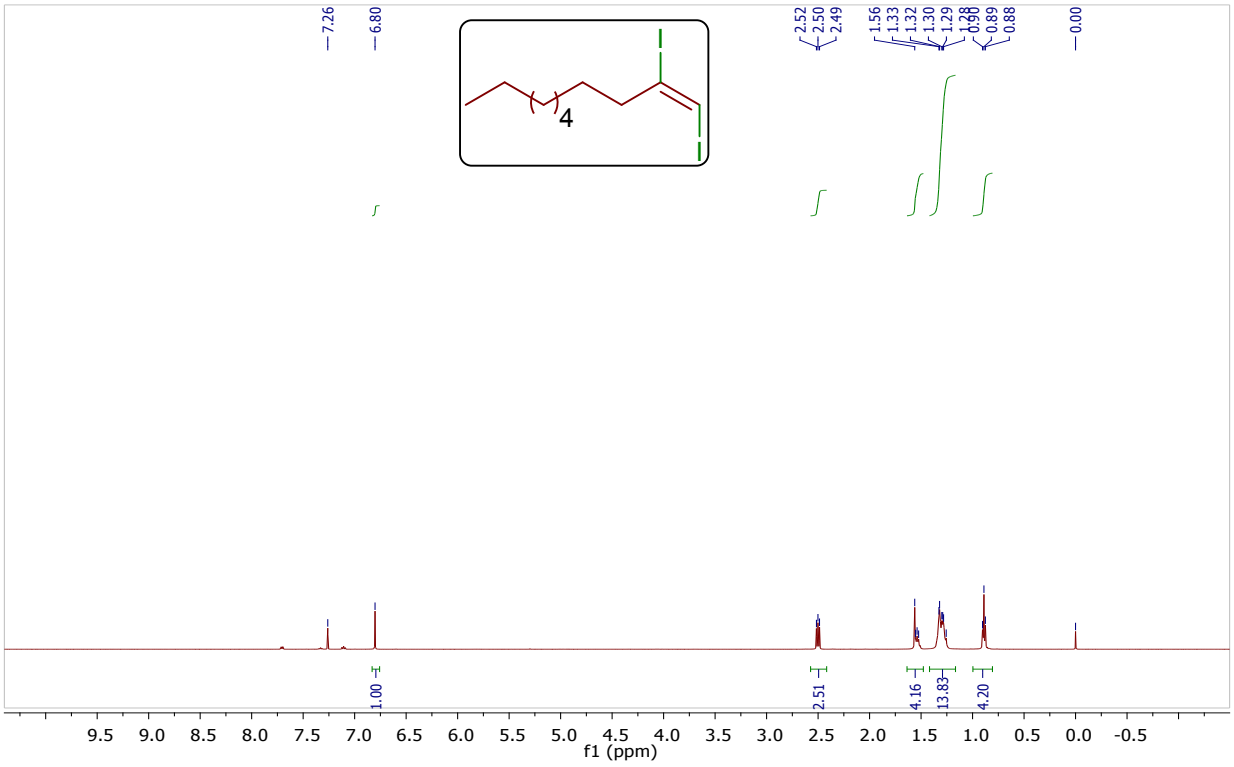
^{1}H NMR Spectrum of compound **3n** in $CDCl_3$



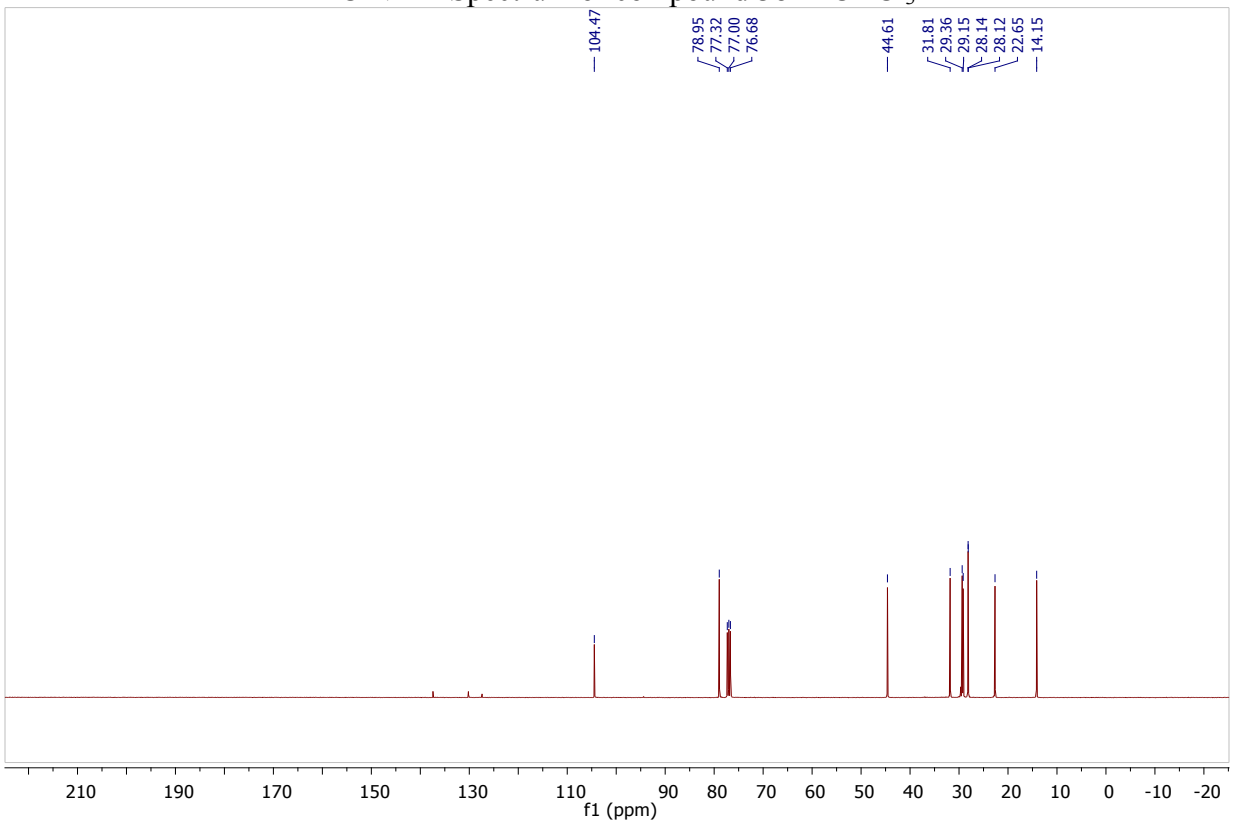
^{13}C NMR Spectrum of compound **3n** in $CDCl_3$



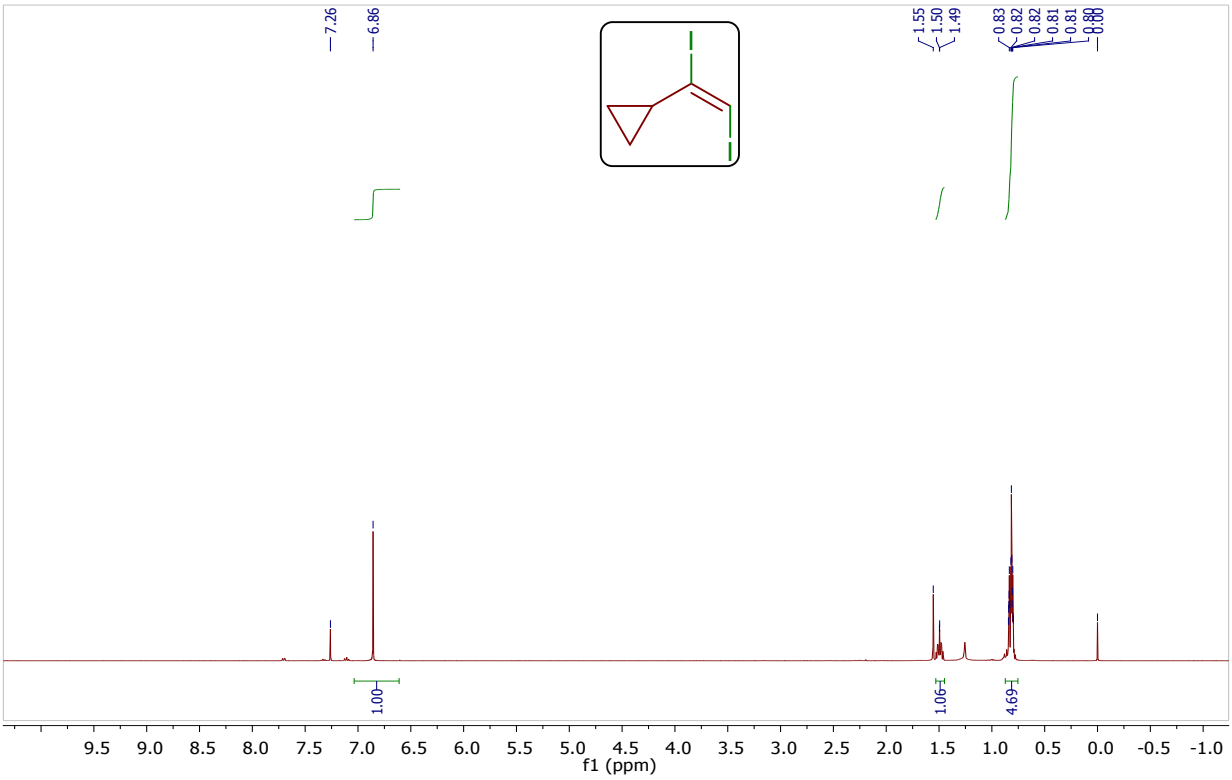
^{1}H NMR Spectrum of compound **3o** in $CDCl_3$



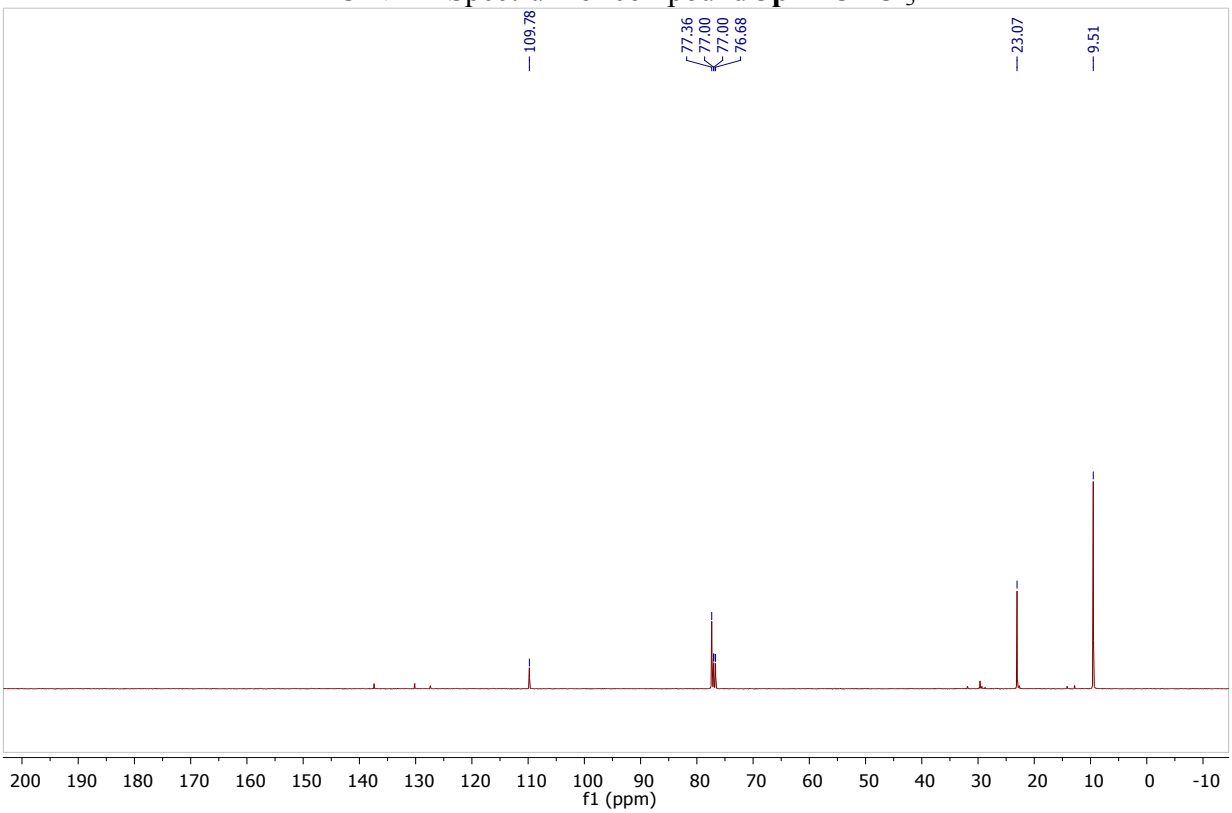
^{13}C NMR Spectrum of compound **3o** in $CDCl_3$



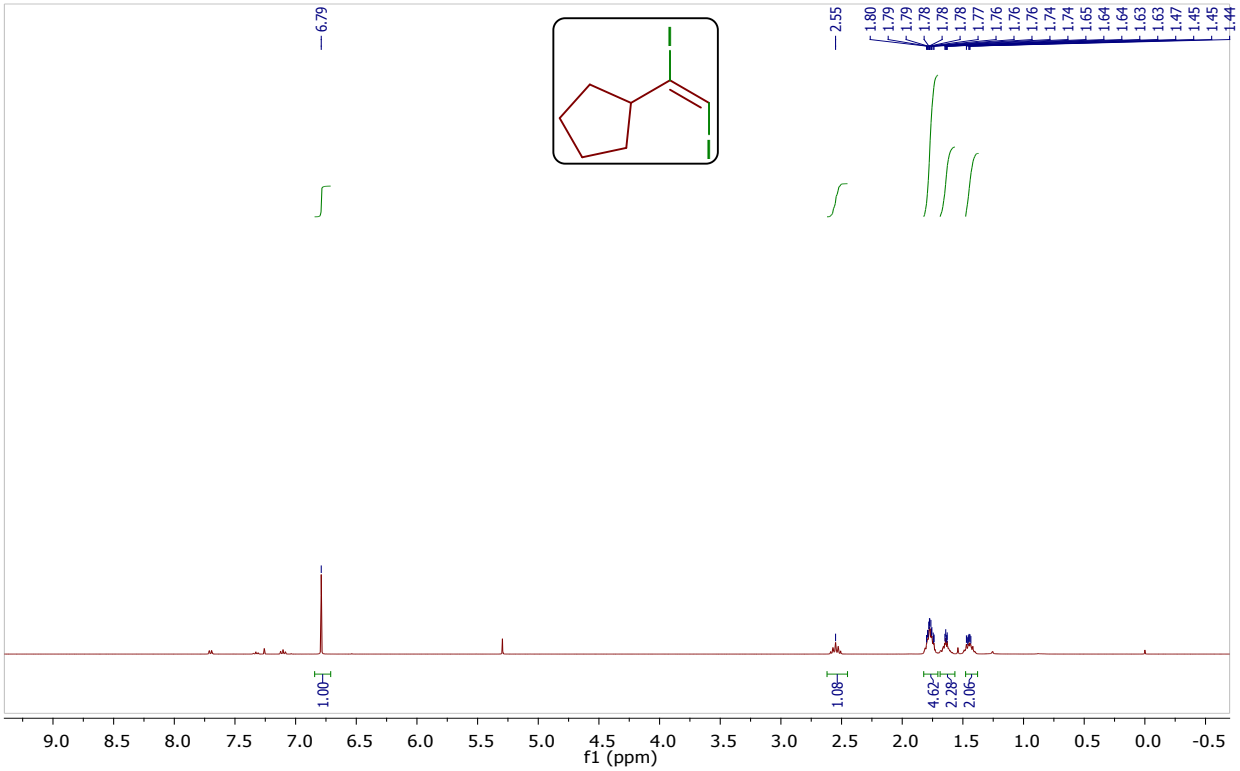
^{1}H NMR Spectrum of compound **3p** in $CDCl_3$



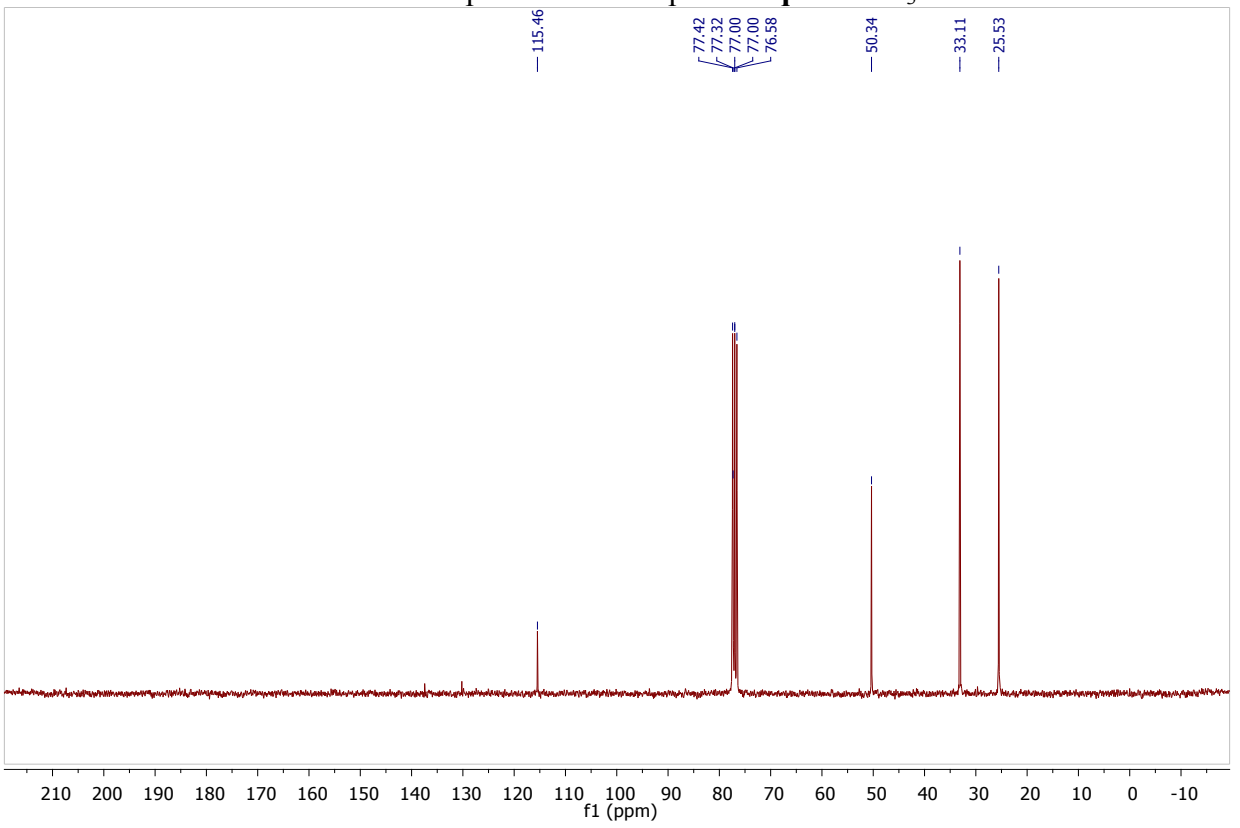
^{13}C NMR Spectrum of compound **3p** in $CDCl_3$



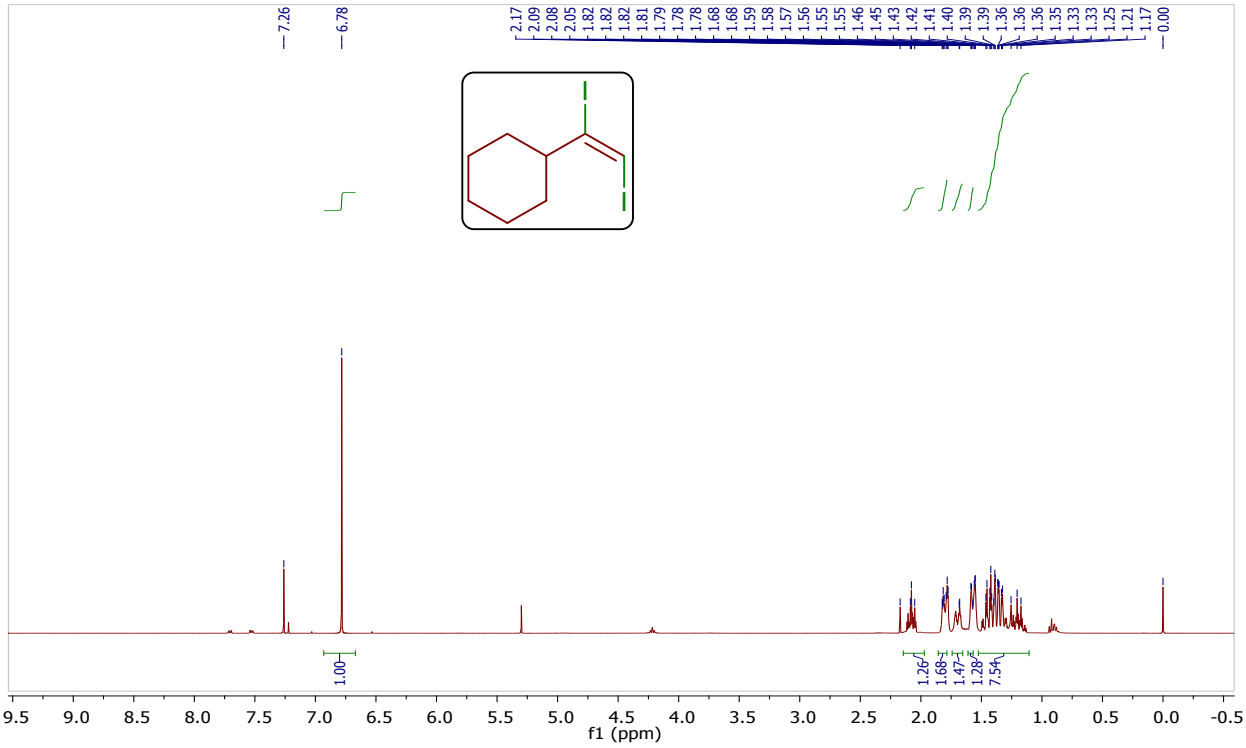
^{1}H NMR Spectrum of compound **3q** in $CDCl_3$



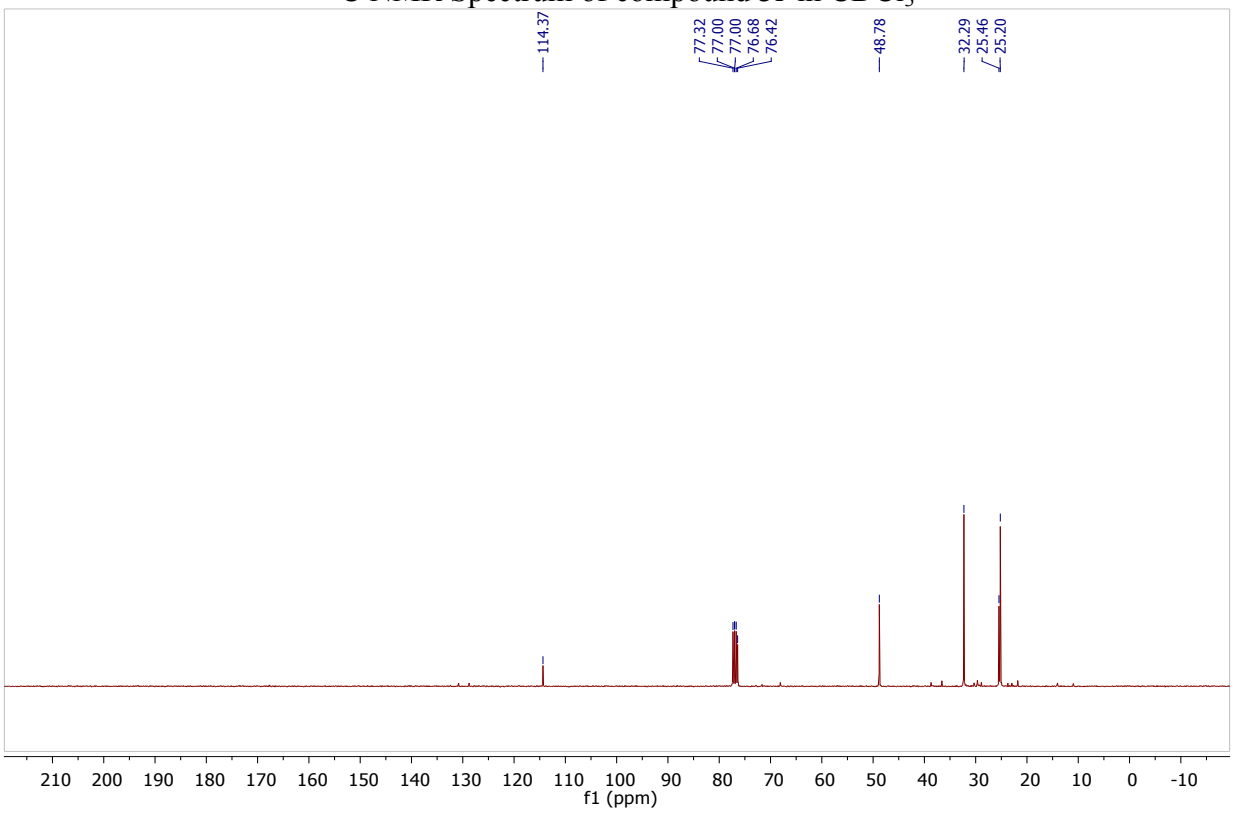
^{13}C NMR Spectrum of compound **3q** in $CDCl_3$



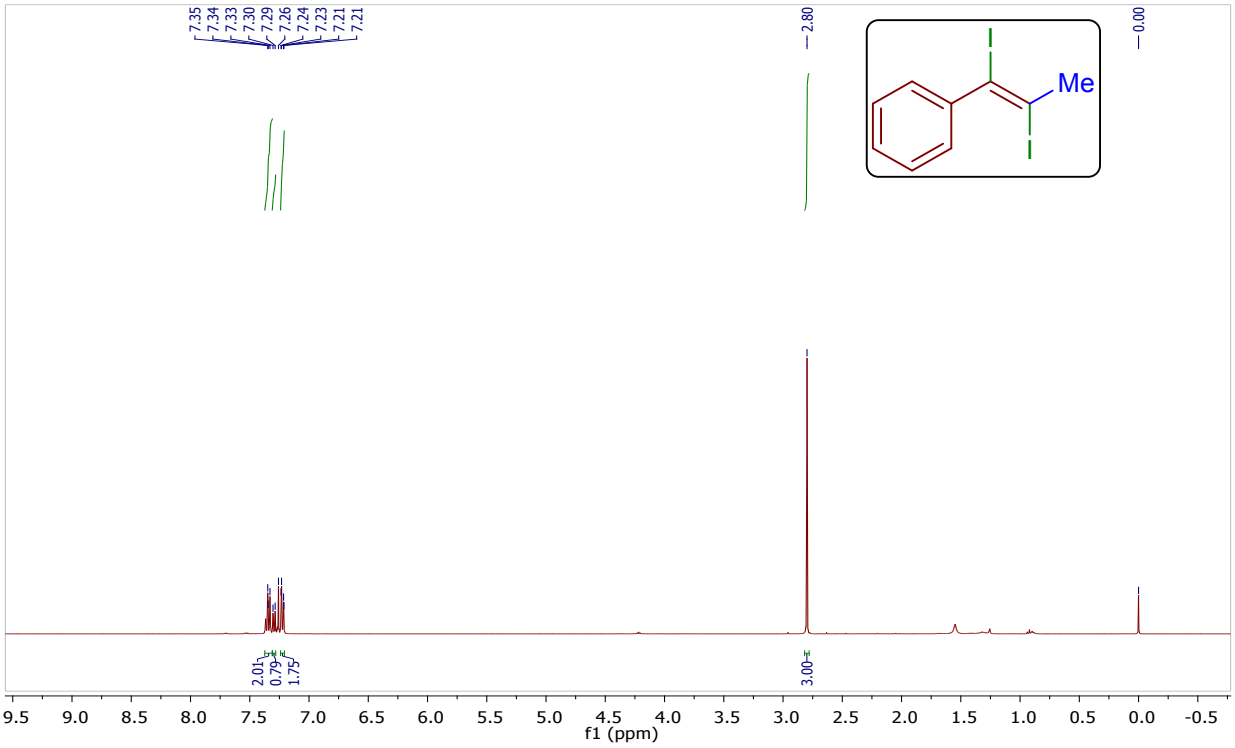
^{1}H NMR Spectrum of compound **3r** in $CDCl_3$



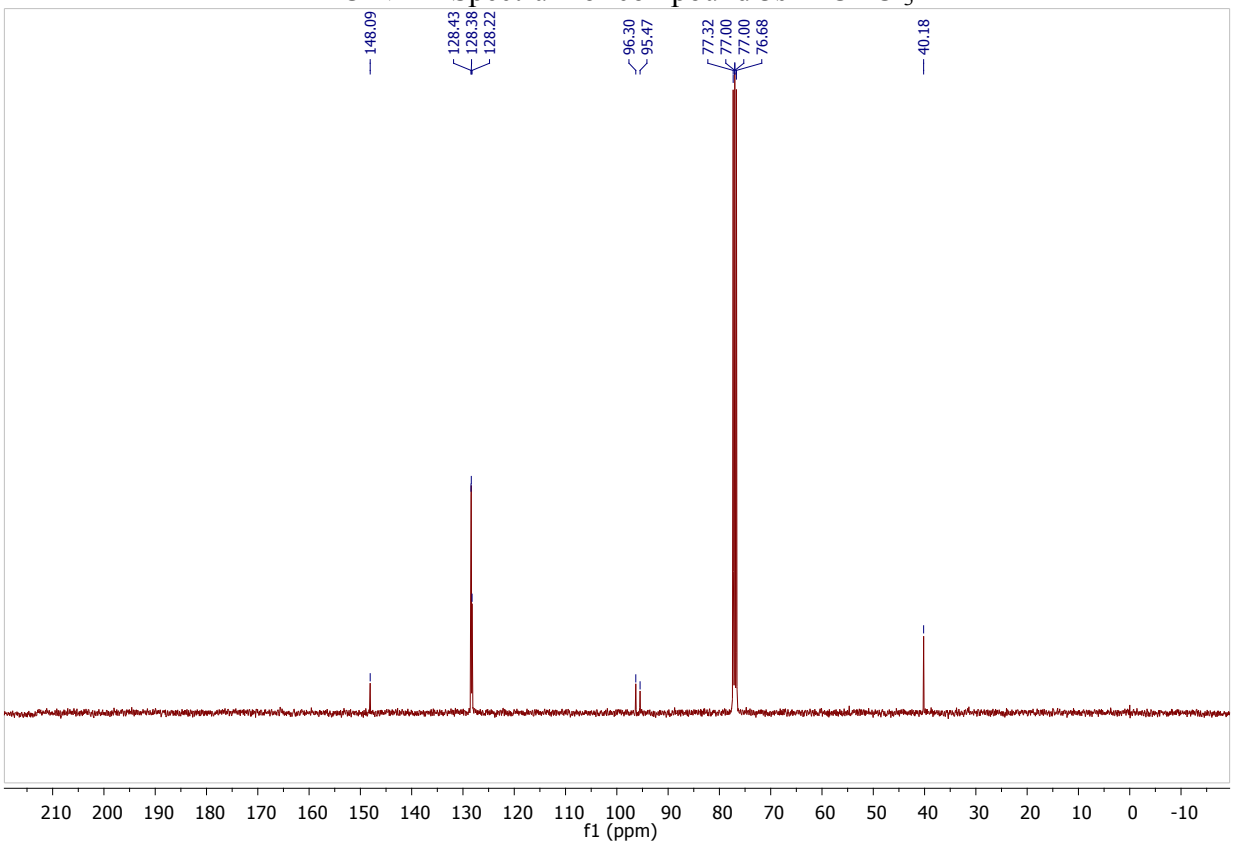
^{13}C NMR Spectrum of compound **3r** in $CDCl_3$



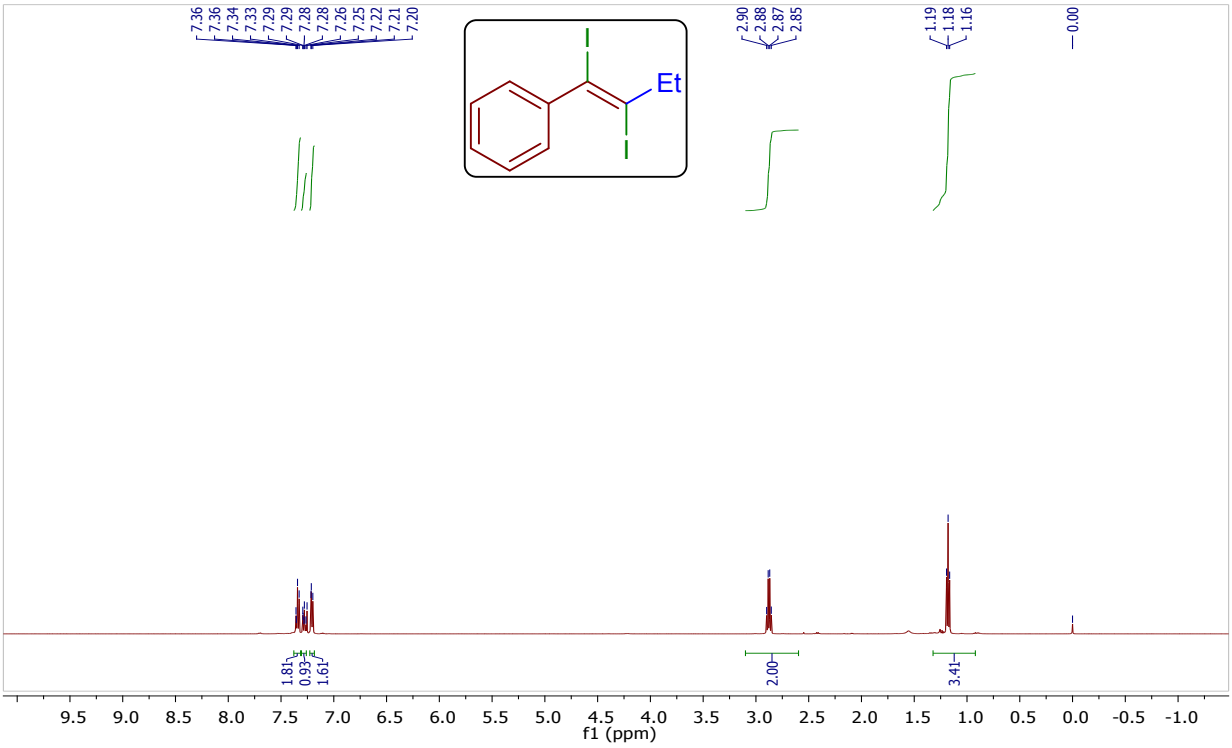
^{1}H NMR Spectrum of compound **3s** in $CDCl_3$



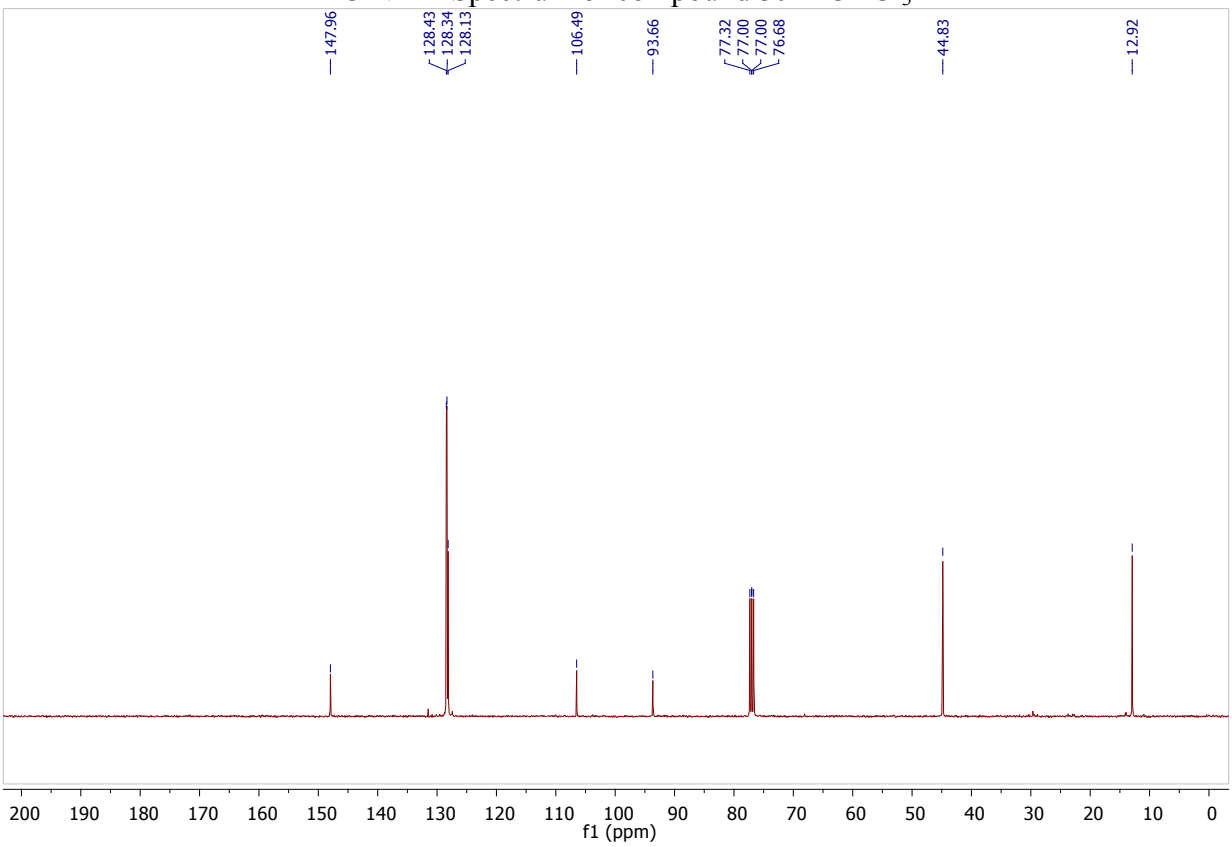
^{13}C NMR Spectrum of compound **3s** in $CDCl_3$



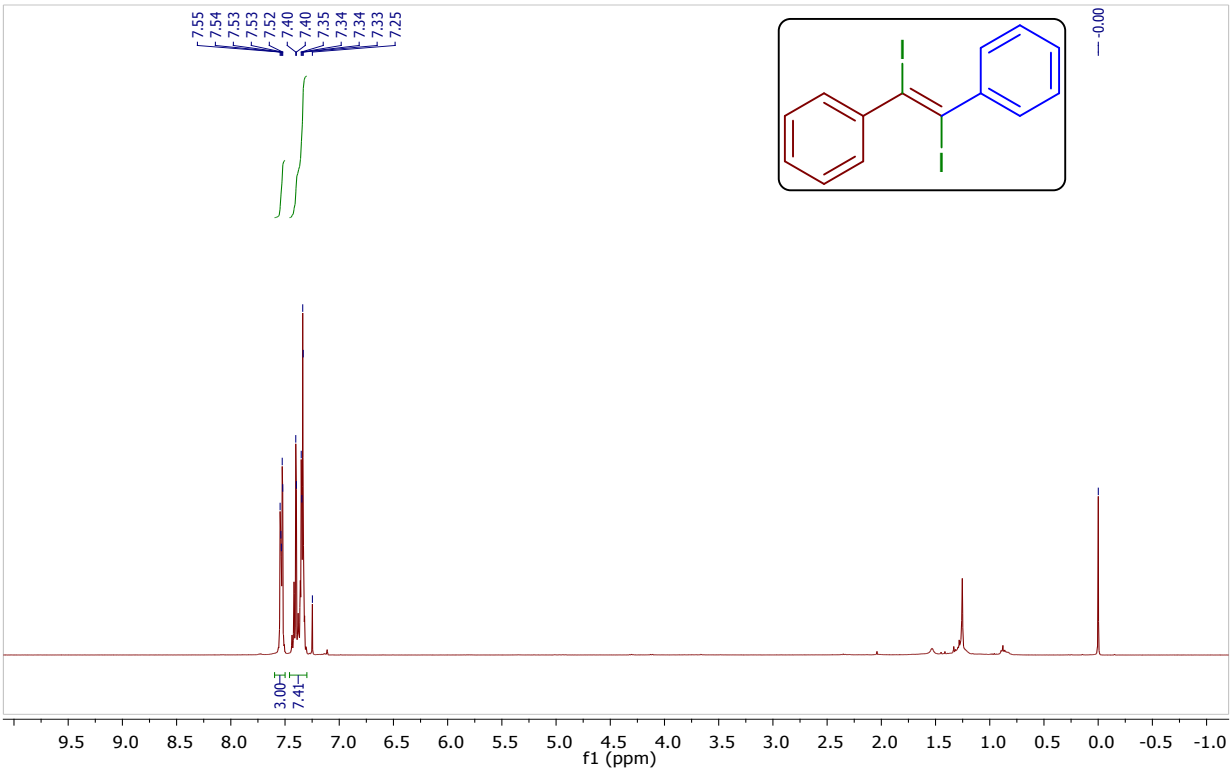
^{1}H NMR Spectrum of compound **3t** in $CDCl_3$



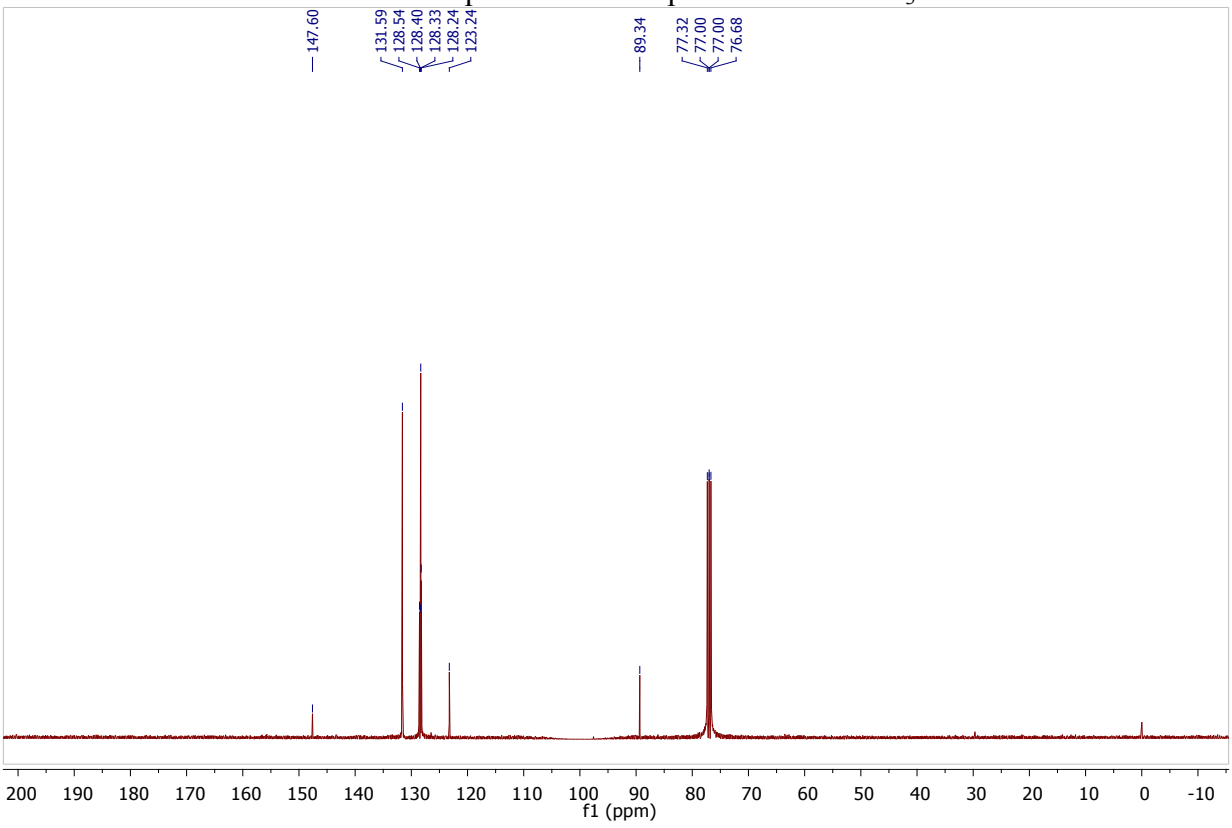
^{13}C NMR Spectrum of compound **3t** in $CDCl_3$



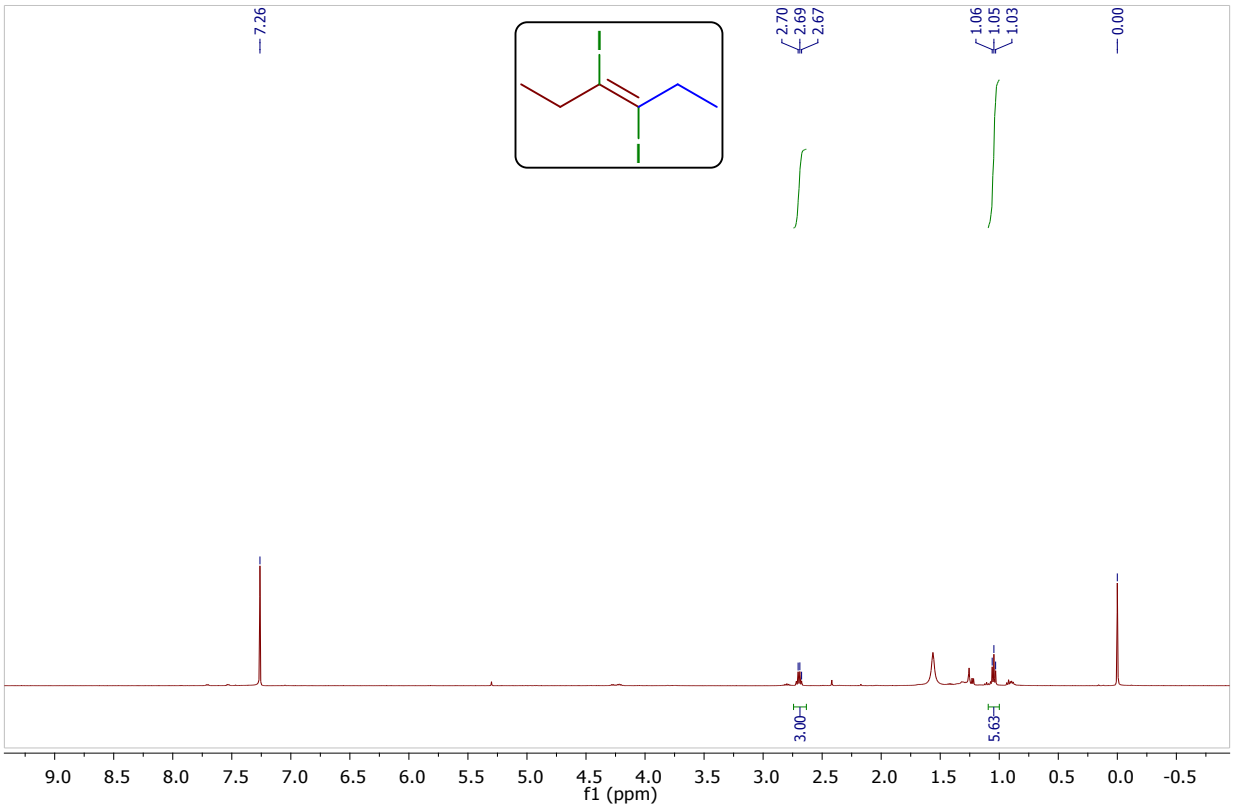
^{1}H NMR Spectrum of compound **3u** in $CDCl_3$



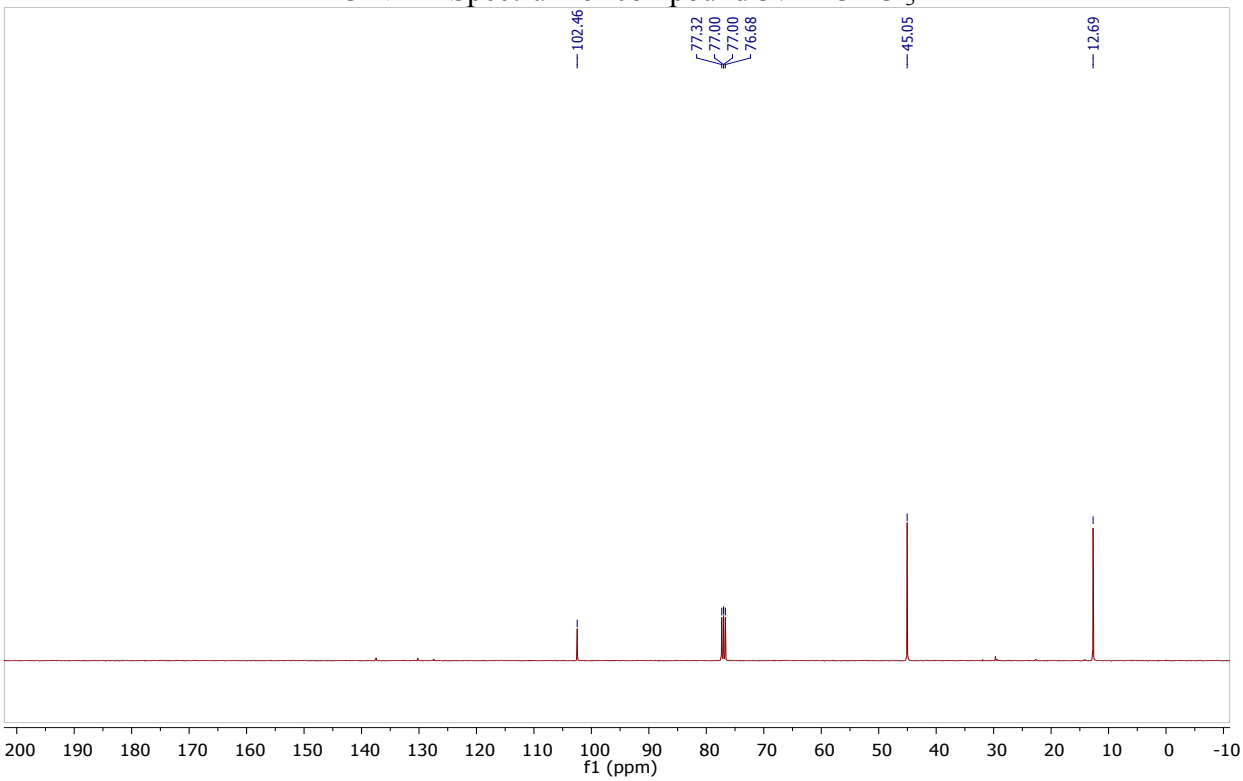
^{13}C NMR Spectrum of compound **3u** in $CDCl_3$



^{1}H NMR Spectrum of compound **3v** in $CDCl_3$



^{13}C NMR Spectrum of compound **3v** in $CDCl_3$



Refrences for Supporting Information

1) K. Srujana, P. Swamy, M. Naresh, C. Durgaiah, B. Rammurthy, G. K. Sai, T. Sony, N. Narender *Chemistry Select,* 2017, **2**, 748-752.

2) K. R. Reddy, M. Venkateshwar, C. U. Maheswari, S. P. Kumar, *Tetrahedron Lett.,* 2010, **51**, 2170-2173.

3) J. Yan, J. Li, D. Cheng, *Synlett,* 2007, 2442-2444.

4) G. Pelletier, S. Lie, J. J. Mousseau, A. B. Charette, *Org. Lett.,* 2012, **14**, 5464-5467.

5) P. Starkov, F. Rota, J. M. D'Oyley, T. D. Sheppard, *Adv. Synth. Catal.,* 2012, **354**, 3217-3224.

6) Q. Jiang J.-Y. Wang, C.-C. Guo, *Synthesis,* 2015, **47**, 2081-2087.

7) S.-N. Chen, T.-T. Hung, *J. Chin. Chem. Soc.,* 2009, ***56***, 1078-1081.

8) A. O. Terent'ev, D. A. Borisov, I. B. Krylov, G. I. Nikishin, *Synth. Commun.* 2007, **37**, 3151-3164.

9) J.-A. Chen, C.-S. Lin, L. K. Liu, *J. Chin. Chem. Soc.,* 1996, **43**, 95-99.