

Supporting Information

Visible-Light Activated Metal Catalyst-Free Vicinal Diazidation of Olefins with Sulfonium Iodate(I) Species

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Experimental

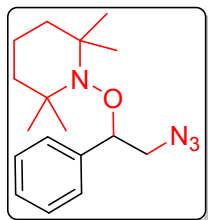
General Synthesis Information. Reactions were run in screw capped glass vials (4 mL) stirred with Teflon®-coated magnetic stir bars. Moisture and air-sensitive reactions were performed in flame-dried round bottom flasks, fitted with rubber septa or glass gas adapters, under a positive pressure of nitrogen. 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). Melting points were obtained in open capillary tubes using a micro melting point apparatus and were uncorrected.

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.

Physical Data. Proton and Carbon nuclear magnetic resonance spectra (¹H, ¹³C NMR) were recorded on Avance 300, 400 or 500 MHz and ECS 4000 MHz (JEOL) nuclear magnetic resonance spectrometers. The proton resonances are annotated as: chemical shift (δ) relative to tetramethylsilane (δ 0.0) using the residual solvent signal as an internal standard or tetramethylsilane itself: chloroform-d (δ 7.26, singlet), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad), coupling constant (J , Hz), and number of protons for a given resonance is indicated by nH. The chemical shifts of ¹³C NMR are reported in ppm relative to the central line of the triplet at 77.00 ppm for CDCl₃. IR spectra were recorded on a Perkin-Elmer FT-IR spectrometer and wave numbers of maximum absorption peaks are presented in cm⁻¹. Mass analyses (ESI-MS) and HRMS were performed on Xevo G2-S QTOF (Waters, USA) Spectrometer.

Synthesis and Spectroscopic Characterization data of all products.



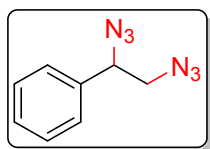
1-(2-azido-1-phenylethoxy)-2,2,6,6-tetramethylpiperidine (2): A preformed solution of $\text{Me}_3\text{Si}(\text{OAc})_2$ (354 mg, 1.1 mmol, 1.1 equiv.), NaN_3 (162 mg, 2.5 mmol, 2.5 equiv.) and 2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) (171 mg, 1.1 mmol, 1.1 equiv.) in DMF (2 mL) was treated with styrene (**1a**, 104 mg, 1.0 mmol, 1.0 equiv.) at room temperature. The mixture was stirred under nitrogen atmosphere and irradiated with visible light (27 W white compact fluorescent lamp, household CFL). The distance between the light source and the reaction flask was approximately 3-4 cm, resulted in the temperature increasing up to 36 °C. For safety reasons, the reaction was carried out behind an antiblast shield. The reaction was stirred until the completion of starting material, typically for 12h (adjudged by TLC). The reaction mixture was diluted with EtOAc (10 mL), quenched with saturated NaHCO_3 (5 mL) and saturated aqueous sodium thiosulfate (2 mL) and extracted with EtOAc (3 × 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 using hexanes as eluent to afford the compound **2** as pale-yellow oil (277 mg, 0.92 mmol, 92%). The product was characterised by ^1H and ^{13}C spectroscopy and MS spectrometry and were in complete agreement with the assigned structure and correlated with literature data.^[1]

^1H NMR (400 MHz, CDCl_3) δ 7.28-7.20 (m, 5H), 4.75 (dd, $J = 6.9, 4.6$ Hz, 1H), 3.66 (dd, $J = 12.4, 4.6$ Hz, 1H), 3.57 (dd, $J = 12.4, 6.9$ Hz, 1H), 1.33 (m, $J = 43.2, 25.3$ Hz, 15H), 0.87-0.71 (m, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 140.65, 128.17, 127.84, 127.52, 84.91, 60.01, 55.19, 40.37, 34.36, 33.96, 29.68, 22.67, 20.27, 17.06.

Representative procedure for Diazidation:

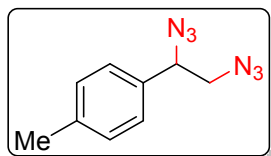
A preformed solution of $\text{Me}_3\text{Si}(\text{OAc})_2$ (1.1 equiv.) and NaN_3 (2.5 equiv.) in DMF (2 mL) was treated with alkene (**1**, 1.0 equiv.) at room temperature under nitrogen atmosphere and irradiated with visible light (27 W white compact fluorescent lamp, household CFL). The reaction was

stirred for 12h or until the completion of starting material (adjudged by TLC). The reaction mixture was diluted with EtOAc (10 mL), quenched with saturated NaHCO₃ (5 mL) and saturated aqueous sodium thiosulfate (2 mL) and extracted with EtOAc (3 × 30 mL). The combined organic layers were washed with brine solution, dried over anhydrous Na₂SO₄, concentrated *in vacuo* and purified by silica gel column chromatography to obtain the desired diazides (**3-33**). All the products were fully characterised by ¹H and ¹³C spectroscopy and MS spectrometry and were in complete agreement with the assigned structure and correlated with literature data.



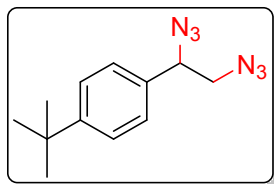
(1,2-diazidoethyl)benzene (3): Following general procedure using styrene (**1a**, 104 mg, 1.0 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (180 mg, 0.96 mmol, 96%).

¹H NMR (400 MHz, CDCl₃) δ 7.48-7.26 (m, 5H), 4.66 (dd, *J* = 8.3, 5.0 Hz, 1H), 3.49 (dd, *J* = 12.7, 8.3 Hz, 1H), 3.43 (dd, *J* = 12.7, 5.0 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 136.26, 129.01, 128.97, 126.88, 65.44, 55.87; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2-6]



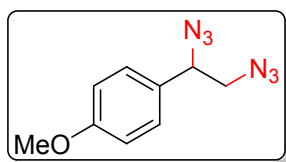
1-(1,2-diazidoethyl)-4-methylbenzene (4): Following general procedure using 1-methyl-4-vinylbenzene (**1b**, 90 mg, 0.762 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (149 mg, 0.739 mmol, 97%).

¹H NMR (400 MHz, CDCl₃) δ 7.28-7.10 (m, 4H), 4.62 (dd, *J* = 8.3, 5.0 Hz, 1H), 3.48 (dd, *J* = 12.7, 8.3 Hz, 1H), 3.40 (dd, *J* = 12.7, 4.9 Hz, 1H), 2.36 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 138.89, 133.17, 129.66, 126.80, 65.23, 55.79, 21.09; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[4]

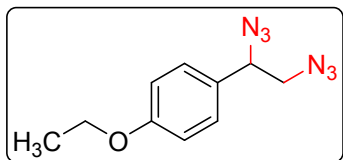


1-(tert-butyl)-4-(1,2-diazidoethyl) benzene (5): Following general procedure using 1-(tert-butyl)-4-vinylbenzene (**1c**, 88 mg, 0.55 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil. (110 mg, 0.451 mmol, 82%).

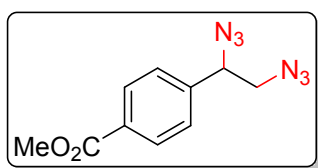
^1H NMR (500 MHz, CDCl_3) δ 7.42 (d, $J = 8.2$ Hz, 2H), 7.25 (d, $J = 8.2$ Hz, 2H), 4.64 (dd, $J = 8.5, 4.7$ Hz, 1H), 3.50 (dd, $J = 12.8, 8.5$ Hz, 1H), 3.43 (dd, $J = 12.8, 4.7$ Hz, 1H), 1.32 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3) δ 152.10, 133.26, 126.60, 125.97, 65.31, 55.94, 34.65, 31.23; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,4]



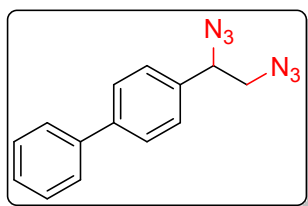
1-(1,2-diazidoethyl)-4-methoxybenzene (6): Following general procedure using 1-methoxy-4-vinylbenzene (**1d**, 100 mg, 0.746 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (126 mg, 0.581 mmol, 78%). ^1H NMR (500 MHz, CDCl_3) δ 7.26 (d, $J = 8.7$ Hz, 2H), 6.94 (d, $J = 8.7$ Hz, 2H), 4.62 (dd, $J = 8.2, 5.0$ Hz, 1H), 3.82 (s, 3H), 3.49 (dd, $J = 12.7, 8.3$ Hz, 1H), 3.41 (dd, $J = 12.7, 5.0$ Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 160.02, 128.23, 128.23, 114.40, 64.99, 55.82, 55.27; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,4]



1-(1,2-diazidoethyl)-4-ethoxybenzene (7): Following general procedure using 1-methoxy-4-vinylbenzene (**1e**, 99 mg, 0.668 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (133 mg, 0.574 mmol, 86%). IR (Film): 2199, 1724, 1435, 1220, 1245, 746 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.17 (d, $J = 8.7$ Hz, 2H), 6.84 (d, $J = 6.4$ Hz, 2H), 4.54 (dd, $J = 8.2, 5.0$ Hz, 1H), 3.96 (q, $J = 6.9$ Hz, 2H), 3.41 (dd, $J = 12.7, 8.4$ Hz, 1H), 3.33 (dd, $J = 12.7, 5.0$ Hz, 1H), 1.34 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 159.41, 128.21, 128.00, 114.87, 65.03, 63.49, 55.82; HRMS (ESI) m/z [$\text{M} + \text{H}$] $^+$ calculated for $[\text{C}_{10}\text{H}_{13}\text{N}_6\text{O}]^+$: 233.11265; found 233.11263.

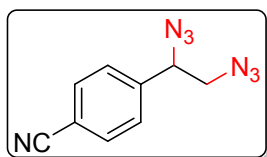


methyl 4-(1,2-diazidoethyl)benzoate (8): Following general procedure using methoxy-4-vinylbenzene (**1f**, 100 mg, 0.617 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (113 mg, 0.462 mmol, 75%). ^1H NMR (400 MHz, CDCl_3) δ 8.03 (d, $J = 8.2$ Hz, 2H), 7.45 (d, $J = 8.2$ Hz, 2H), 4.94 (dd, $J = 7.3, 5.8$ Hz, 1H), 3.92 (s, 3H), 3.47 (d, $J = 6.0$ Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 166.70, 145.38, 130.09, 129.96, 125.87, 57.94, 52.21; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[4]



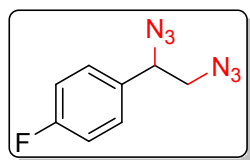
4-(1,2-diazidoethyl)-1,1'-biphenyl (9): Following general procedure using methoxy-4-vinylbenzene (**1g**, 100 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (134 mg, 0.510 mmol, 92%). ^1H NMR (400 MHz, CDCl_3) δ 7.70-7.66 (d, $J = 8.4$ Hz, 2H), 7.66-7.61 (d, $J = 8.4$ Hz, 2H), 7.61-7.56 (d, $J = 8.4$ Hz, 2H), 7.42 (m, 3H), 4.72 (dd, $J = 8.2, 5.0$ Hz, 1H), 3.55 (dd, $J = 12.7, 8.2$ Hz, 1H), 3.48 (dd, $J = 12.8, 5.0$ Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.98, 140.21, 138.89,

128.99, 128.85, 128.45, 127.77, 127.67, 127.38, 127.30, 65.26, 55.93; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[4]



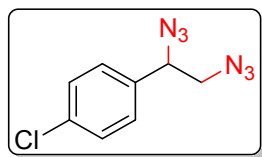
4-(1,2-diazidoethyl)benzonitrile (10): Following general procedure using 4-vinylbenzonitrile (**1h**, 107 mg, 0.829 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (141 mg, 0.663 mmol, 80%).

¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, *J* = 8.5 Hz, 2H), 7.41 (dd, *J* = 8.2 Hz, 2H), 4.67 (dd, *J* = 7.2, 5.6 Hz, 1H), 3.45 (dd, *J* = 11.2, 5.7 Hz, 1H), 3.41 (dd, *J* = 11.1, 3.8 Hz, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 141.56, 132.78, 127.69, 118.11, 112.88, 64.69, 55.69; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,3]



1-(1,2-diazidoethyl)-4-fluorobenzene (11): Following general procedure using 1-fluoro-4-vinylbenzene (**1i**, 102 mg, 0.836 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (160 mg, 0.777 mmol, 93%).

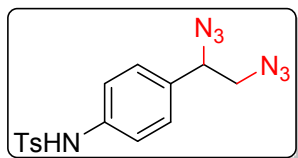
¹H NMR (400 MHz, CDCl₃) δ 7.34-7.29 (m, 2H), 7.14-7.07 (m, 2H), 4.66 (dd, *J* = 7.9, 5.1 Hz, 1H), 3.49 (dd, *J* = 12.7, 7.9 Hz, 1H), 3.42 (dd, *J* = 12.7, 5.1 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 164.54, 161.26, 132.16, 128.80, 128.69, 116.24, 115.95, 64.73, 55.91; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,4]



1-chloro-4-(1,2-diazidoethyl)benzene (12): Following general procedure using 1-chloro-4-vinylbenzene (**1j**, 115 mg, 0.833 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (166 mg, 0.749 mmol, 90%).

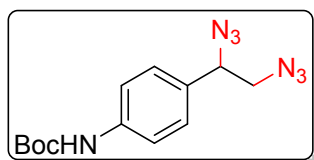
¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 4.65 (dd, *J* =

7.9, 5.1 Hz, 1H), 3.51-3.45 (dd, $J = 12.7, 8.1$ Hz, 1 H), 3.43 (dd, $J = 12.7, 5.0$ Hz, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 134.93, 134.85, 129.28, 128.28, 64.75, 55.84; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2-4]



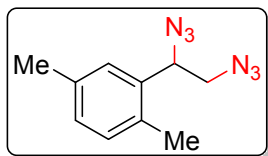
***N*-(4-(1,2-diazidoethyl)phenyl)-4-methylbenzenesulfonamide (13)**: Following general procedure using 4-methyl-*N*-(4-vinylphenyl)benzenesulfonamide (**1k**, 100 mg, 0.366 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as white crystals (100 mg, 0.281 mmol, 77%).

Mp: 98-99 °C; IR (Film): 3455, 2195, 1447, 1217, 1190, 746 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, $J = 8.5$ Hz, 2H), 7.36-7.31 (m, 4H), 7.08 (d, $J = 8.5$ Hz, 2H), 4.70 (dd, $J = 7.3, 5.5$ Hz, 1H), 3.53-3.44 (m, 2H), 2.48 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 145.21, 138.53, 136.37, 132.22, 129.66, 128.55, 127.65, 64.96, 56.03, 21.74, HRMS (ESI) m/z $[\text{M} + \text{NH}_4]^+$ calculated for $[\text{C}_{15}\text{H}_{19}\text{N}_9\text{O}_2\text{S}]^+$: 375.43164; found 375.43135.



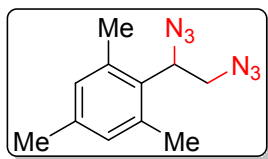
***N*-(4-(1,2-diazidoethyl)phenyl)-1-(oxoboranyl)methanimine (14)**: Following general procedure using 1-(oxoboranyl)-*N*-(4-vinylphenyl)methanimine (**1l**, 100 mg, 0.456 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as white solid (127 mg, 0.419 mmol, 92%).

Mp: 162-164 °C; IR (Film): 3350, 2180, 1720, 1436, 1219, 1150, 746 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.34 (d, $J = 8.5$ Hz, 2H), 7.18 (d, $J = 8.5$ Hz, 2H), 6.57 (s, 1H), 4.55 (dd, $J = 8.2, 5.0$ Hz, 1H), 3.40 (dd, $J = 12.7, 8.4$ Hz, 1H), 3.32 (dd, $J = 12.7, 4.9$ Hz, 1H), 1.44 (s, 9 H); ^{13}C NMR (101 MHz) δ 152.55, 139.06, 130.53, 127.68, 118.75, 80.83, 65.01, 55.79, 28.24; HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ calculated for $[\text{C}_{13}\text{H}_{18}\text{N}_7\text{O}_2]^+$: 304.33121; found 304.33104.



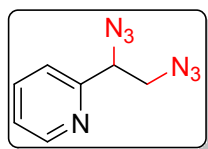
2-(1,2-diaziidoethyl)-1,4-dimethylbenzene (15): Following general procedure using 1,4-dimethyl-2-vinylbenzene (**1m**, 90 mg, 0.681 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (117 mg, 0.544 mmol, 80%).

IR (Film): 2924, 2192, 1695, 1442, 1250, 743 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.08 (s, 1H), 7.01 (d, $J = 7.8$ Hz, 1H), 6.98 (d, $J = 7.7$ Hz, 1H) 4.80 (dd, $J = 8.7, 4.4$ Hz, 1H), 3.39 (dd, $J = 13.7, 8.7$ Hz, 1H), 3.29 (dd, $J = 13.7, 5.2$ Hz, 1H), 2.25 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (101 MHz) δ 136.27, 134.13, 132.11, 130.85, 129.39, 126.92, 62.10, 55.19, 20.99, 18.66; HRMS (ESI) m/z $[\text{M}]^+$ calculated for $[\text{C}_{10}\text{H}_{12}\text{N}_6]^+$: 216.11260; found 216.11263.



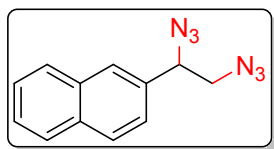
2-(1,2-diaziidoethyl)-1,3,5-trimethylbenzene (16): Following general procedure using 1,3,5-trimethyl-2-vinylbenzene (**1n**, 90 mg, 0.616 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (133 mg, 0.579 mmol, 94%).

^1H NMR (500 MHz, CDCl_3) δ 6.87 (s, 2H), 5.18 (dd, $J = 9.5, 4.9$ Hz, 1H), 3.70 (dd, $J = 12.8, 9.5$ Hz, 1H), 3.34 (dd, $J = 12.8, 4.9$ Hz, 1H), 2.41 (s, 6H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 138.29, 136.63, 130.49, 129.04, 62.09, 53.38, 20.78, 20.70; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2]



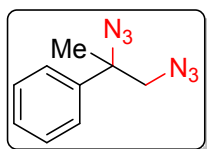
2-(1,2-diaziidoethyl)pyridine (17): Following general procedure using 2-vinylpyridine (**1o**, 98 mg, 0.933 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (132 mg, 0.699 mmol, 75%).

^1H NMR (400 MHz, CDCl_3) δ 8.62 (d, $J = 4.1$ Hz, 1H), 7.77-7.74 (td, $J = 8.0, 2.2$ Hz, 1H), 7.42 (d, $J = 7.9$ Hz, 1H), 7.32-7.28 (m, 1H), 4.69 (dd, $J = 7.1, 4.9$ Hz, 1 H), 3.79 (dd, $J = 12.7, 4.1$ Hz, 1 H), 3.66 (dd, $J = 12.7, 7.7$ Hz, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 155.78, 149.73, 137.23, 123.59, 121.96, 65.45, 54.46; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,4]



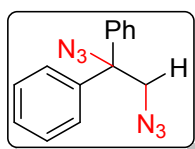
2-(1,2-diazidoethyl)naphthalene (18): Following general procedure using 2-vinylnaphthalene (**1p**, 100 mg, 0.649 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (120 mg, 0.506 mmol, 78%).

^1H NMR (400 MHz, CDCl_3) δ 7.91-7.78 (m, 4H), 7.55-7.49 (m, 2H), 7.40 (dd, $J = 8.4, 1.8$ Hz, 1H), 4.82 (dd, $J = 8.3, 4.9$ Hz, 1H), 3.58 (dd, $J = 12.7, 8.3$ Hz, 1H), 3.50 (dd, $J = 12.7, 4.9$ Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 133.60, 133.37, 133.09, 129.09, 128.01, 127.75, 126.69, 126.52, 123.93, 65.67, 55.83; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[3,4]

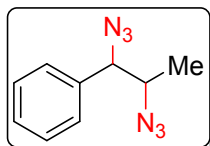


(1,2-diazidopropan-2-yl)benzene (19): Following general procedure using prop-1-en-2-ylbenzene (**1q**, 91 mg, 0.771 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (130 mg, 0.647 mmol, 84%).

^1H NMR (400 MHz, CDCl_3) δ 7.49-7.29 (m, 5H), 3.49 (d, $J = 12.6$ Hz, 1H), 3.40 (d, $J = 12.6$ Hz, 1H), 1.77 (s, 3H); ^{13}C NMR (101 MHz) δ 140.54, 128.83, 128.28, 125.74, 66.53, 60.97, 22.28; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2-6]

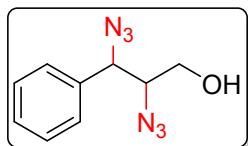


(1,2-diazidoethane-1,1-diyl)dibenzene (20): Following general procedure using ethene-1,1-diyldibenzene (**1r**, 102 mg, 0.566 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (140 mg, 0.532 mmol, 94%). ^1H NMR (500 MHz, CDCl_3) δ 7.57-7.06 (m, 10H), 4.03 (s, 2H); ^{13}C NMR (126 MHz, CDCl_3) δ 140.14, 128.63, 128.32, 127.34, 72.44, 59.35, the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[3]

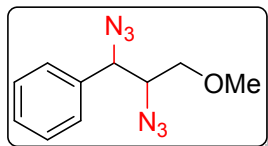


(1,2-diazidopropyl) benzene (21): Following general procedure using (*E*)-but-2-en-2-ylbenzene (**1s**, 91 mg, 0.689 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as colourless oil (118 mg, 0.585 mmol, 85%, 3.5:1 *dr*).

^1H NMR (500 MHz, CDCl_3) δ 7.43-7.27 (m, 5H), 4.36 (m, $J = 7.8$ Hz, 1H), 1.10 (d, $J = 6.7$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 136.25, 128.96, 128.82, 127.53, 70.76, 61.49, 16.74, *Syn* isomer; ^1H NMR (500 MHz, CDCl_3) δ 7.43-7.27 (m, 5H), 4.52 (d, $J = 5.8$ Hz, 1H), 1.25 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 136.01, 128.96, 128.78, 127.53, 69.61, 61.01, 15.02; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,4,6]

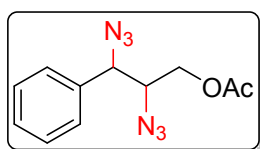


2,3-diazido-3-phenylpropan-1-ol (22): Following general procedure using (*E*)-3-phenylprop-2-en-1-ol (**1t**, 104 mg, 0.776 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (135 mg, 0.620 mmol, 80%, 1:1 *dr*). Both isomers; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.33 (m, 10H), 4.83 (d, $J = 6.4$ Hz, 1H), 4.81 (d, $J = 6.0$ Hz, 1H), 3.80 (d, $J = 1.6$ Hz, 1H), 3.79 (d, $J = 2.1$ Hz, 1H), 3.68-3.60 (m, 3H), 3.54 (dd, $J = 12.5, 7.0$ Hz, 1H); ^{13}C NMR (101 MHz,) δ 140.23, 140.11, 128.71, 128.70, 128.48, 126.45, 126.34, 74.80, 74.60, 68.85, 67.39, 62.59, 62.35; HRMS (ESI) m/z $[\text{M} + \text{NH}_4]^+$ calculated for $[\text{C}_9\text{H}_{14}\text{N}_7\text{O}]^+$: 236.26235; found 236.26226.



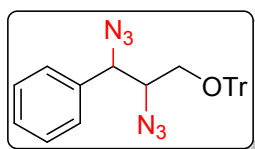
(1,2-diazido-3-methoxypropyl)benzene (23): Following general procedure using (*E*)-(3-methoxyprop-1-en-1-yl)benzene (**1u**, 100 mg, 0.675 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (128 mg, 0.553 mmol, 82%, 4:1 *dr*).

IR (Film): 2160, 2146, 1725, 1437, 1220, 772 cm^{-1} ; Major isomer; ^1H NMR (400 MHz, CDCl_3) δ 7.45-7.23 (m, 5H), 4.85 (d, $J = 5.5$ Hz, 1H), 3.74-3.67 (m, 1H), 3.62-3.50 (m, 2H), 3.34 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 140.20, 128.59, 128.27, 126.39, 74.72, 72.11, 65.35, 59.31; HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ calculated for $[\text{C}_{10}\text{H}_{11}\text{N}_6\text{O}]^+$: 233.25602; found 233.25632.



2,3-diazido-3-phenylpropyl acetate (24): Following general procedure using cinnamyl acetate (**1v**, 100 mg, 0.568 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (123 mg, 0.477 mmol, 84%, 1.2:1 *dr*).

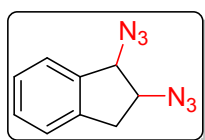
IR (Film): 2160, 1720, 1467, 1240, 646 cm^{-1} ; Both isomers; ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.23 (m, 10H), 4.70 (d, $J = 6.4$ Hz, 1H), 4.63 (d, $J = 6.4$ Hz, 1H), 4.21 (m, 2H), 4.08 (dd, $J = 11.7, 3.9$ Hz, 1H), 3.92 (dd, $J = 11.4, 7.8$ Hz, 1H), 3.78 (m, 2H), 2.02 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.89, 170.58, 139.54, 139.48, 128.84, 128.73, 128.60, 126.45, 126.38, 73.99, 73.59, 66.11, 65.29, 63.92, 63.47, 20.72, 20.66; HRMS (ESI) m/z $[\text{M}]^+$ calculated for $[\text{C}_{11}\text{H}_{12}\text{N}_6\text{O}_2]^+$: 260.26210; found 260.26203.



((2,3-diazido-3-phenylpropoxy)methanetriyl)tribenzene (25): Following general procedure using ((cinnamyloxy)methanetriyl)tribenzene (**1w**, 100 mg, 0.265 mmol) and purified by silica

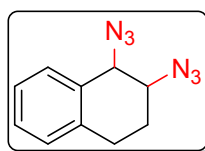
gel column chromatography, eluting with hexanes afforded the title compound as colourless semi solid (108 mg, 0.235 mmol, 89%, 1:1 *dr*).

IR (Film): 2153, 1699, 1547, 1245, 747 cm^{-1} ; Both isomers; ^1H NMR (400 MHz, CDCl_3) δ 7.47-7.40 (m, 10H), 7.34-7.18 (m, 30H), 4.82 (dd, $J = 6.0, 3.7$ Hz, 1H), 4.77 (dd, $J = 6.0, 3.4$ Hz, 1H), 3.64 (dd, $J = 11.2, 5.3$ Hz, 1H), 3.56 (m, 2H), 3.39-3.28 (m, 3H), 3.16 (dd, $J = 10.1, 6.2$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 143.35, 143.34, 140.02, 139.97, 128.53, 128.42, 128.39, 128.12, 127.95, 127.90, 127.20, 127.16, 126.41, 126.27, 87.57, 87.36, 74.40, 73.85, 67.75, 66.24, 63.82, 63.26; HRMS (ESI) m/z $[\text{M}]^+$ calculated for $[\text{C}_{28}\text{H}_{24}\text{N}_6\text{O}]^+$: 460.54410; found 460.54402.



1,2-diazido-2,3-dihydro-1H-indene (26): Following general procedure using 1,2-dihydronaphthalene (**1x**, 100 mg, 0.862 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (124 mg, 0.620 mmol, 72%, 24:1 *dr*).

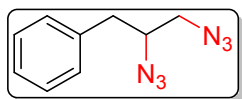
^1H NMR (500 MHz, CDCl_3) δ 7.57-7.10 (m, 4H), 4.84 (d, $J = 5.6$ Hz, 1H), 4.29 (dd, $J = 12.4, 6.6$ Hz, 1H), 3.31-3.00 (m, 2H); ^{13}C NMR (126 MHz, CDCl_3) δ 139.70, 137.50, 129.72, 127.70, 125.37, 124.91, 66.96, 64.09, 35.57; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,3,5,6]



1,2-diazido-1,2,3,4-tetrahydronaphthalene (27): Following general procedure using 1,2-dihydronaphthalene (**1y**, 100 mg, 0.769 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (126 mg, 0.592 mmol, 77%, 24:1 *dr*).

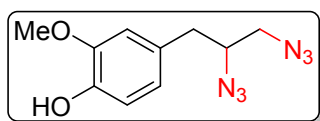
^1H NMR (400 MHz, CDCl_3) δ 7.35-7.16 (m, 4H), 4.65 (d, $J = 3.1$ Hz, 1H), 3.80 (dt, $J = 11.4, 3.4$ Hz, 1H), 3.06 (ddd, $J = 17.5, 6.1, 3.4$ Hz, 1H), 2.89 (ddd, $J = 17.4, 10.6, 6.5$ Hz, 1H), 2.31-2.15 (m, 1H), 2.11-2.02 (m, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 135.33, 131.64, 129.57, 129.28,

129.09, 126.55, 62.47, 59.68, 27.34, 22.79; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,5,6]



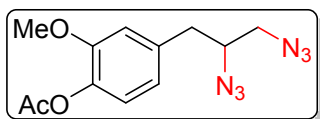
(2,3-diazidopropyl)benzene (28): Following general procedure using allylbenzene (**1z**, 89 mg, 0.754 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as yellow oil (140 mg, 0.693 mmol, 92%).

¹H NMR (400 MHz, CDCl₃) δ 7.38-7.31 (m, 2H), 7.30-7.26 (m, 1H), 7.24-7.18 (m, 2H), 3.72 (m, 1H), 3.40 (dd, *J* = 12.7, 4.0 Hz, 1H), 3.29 (dd, *J* = 12.7, 6.9 Hz, 1H), 2.88 (d, *J* = 7.1 Hz, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 136.30, 129.23, 128.79, 127.15, 62.87, 53.88, 37.99; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[5]



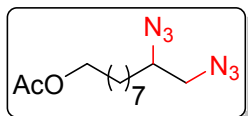
4-(2,3-diazidopropyl)-2-methoxyphenol (29): Following the procedure employed for compound using 4-allyl-2-methoxyphenol (106 mg, 0.646 mmol, 1.0 equiv.) purified by silica gel column chromatography afforded the title compound as pale-yellow oil (150 mg, 0.607 mmol, 94%).

¹H NMR (400 MHz,) δ 6.86 (d, *J* = 7.8 Hz, 1H), 6.72-6.66 (m, 2H), 5.67 (s, 1H), 3.87 (s, 3H), 3.66 (qd, *J* = 7.1, 4.0 Hz, 1H), 3.38 (dd, *J* = 12.8, 3.9 Hz, 1H), 3.26 (dd, *J* = 12.7, 6.8 Hz, 1H), 2.79 (d, *J* = 7.1 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 146.58, 144.65, 128.00, 121.92, 114.56, 111.60, 62.96, 55.87, 53.70, 37.53; HRMS (ESI-TOF) *m/z* [M + Na]⁺ calculated for [C₁₀H₁₂N₆NaO₂]⁺: 271.24432; found; 271.24401.



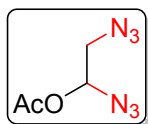
4-(2,3-diazidopropyl)-2-methoxyphenyl acetate (30): Following general procedure using 4-allyl-2-methoxyphenyl acetate (**1aa**, 100 mg, 0.485 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as yellow oil; (109 mg, 0.378 mmol, 78%).

IR (Film): 2920, 2161, 1726, 1451, 1246, 752 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 6.98 (d, $J = 8.0$ Hz, 1H), 6.82-6.75 (m, 2H), 3.82 (s, 3H), 3.70 (qd, $J = 6.9, 4.1$ Hz, 1H), 3.41 (dd, $J = 12.8, 3.9$ Hz, 1H), 3.29 (dd, $J = 12.6, 6.6$ Hz, 1H), 2.84 (d, $J = 6.9$ Hz, 2H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 169.01, 151.07, 138.70, 135.18, 122.91, 121.28, 113.23, 62.55, 55.82, 53.70, 37.65, 20.57; HRMS (ESI-TOF) m/z $[\text{M} + \text{Na}]^+$ calculated for $[\text{C}_{12}\text{H}_{14}\text{N}_6\text{NaO}_3]^+$: 313.27105; found; 313.27113.



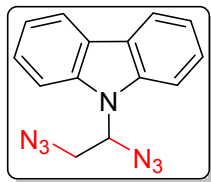
10,11-diazidoundecyl acetate (31): Following general procedure using undec-10-en-1-yl acetate (**1ab**, 100 mg, 0.505 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (105 mg, 0.373 mmol, 74%).

IR (Film): 2196, 2089, 1752, 1465, 1243, 781 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 4.01 (t, $J = 6.8$ Hz, 2H), 3.42 (ddd, $J = 11.0, 7.1, 3.7$ Hz, 1H), 3.35 (dd, $J = 12.7, 4.0$ Hz, 1H), 3.27 (dd, $J = 12.6, 7.3$ Hz, 1H), 2.00 (s, 3H), 1.62-1.54 (m, 2H), 1.50 (dd, $J = 14.7, 7.3$ Hz, 2H), 1.28-1.22 (s, 12H); ^{13}C NMR (101 MHz, CDCl_3) δ 171.06, 64.43, 61.91, 54.67, 31.59, 29.19, 29.14, 29.09, 29.02, 28.42, 25.72, 25.70, 20.85; HRMS m/z $[\text{M}]^+$ calculated for $[\text{C}_{13}\text{H}_{24}\text{N}_6\text{O}_2]^+$: 296.38321; found: 295.38334.



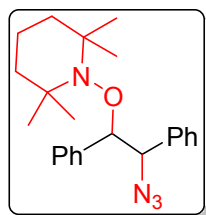
1,2-diazidoethyl acetate (32): Following general procedure using vinyl acetate (**1ac**, 93 mg, 1.081 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as pale-yellow oil (156 mg, 0.918 mmol, 85%).

IR (Film): 2106, 1720, 1642, 1242, cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 6.04-5.94 (m, 1H), 3.41 (dd, $J = 13.2, 5.8$ Hz, 1H), 3.35 (dd, $J = 13.1, 5.0$ Hz, 1H), 2.18 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.07, 83.20, 52.37, 20.68; HRMS m/z $[\text{M} + \text{H}]^+$ calculated for $[\text{C}_4\text{H}_7\text{N}_6\text{O}_2]^+$: 171.14904; found: 171.14913.



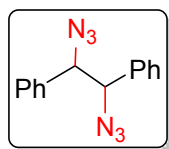
9-(1,2-diazidoethyl)-9H-carbazole (33): Following general procedure using 9-vinylcarbazole (**1ad**, 100 mg, 0.518 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as brown colour semi-solid (126 mg, 0.455 mmol, 88%).

IR (Film): 2923, 2102, 1549, 1450, 1219, 772 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 8.10 (d, $J = 7.8$ Hz, 2H), 7.60 (d, $J = 8.2$ Hz, 2H), 7.49 (t, $J = 7.2$ Hz, 2H), 7.32 (t, $J = 7.2$ Hz, 2H), 6.29 (t, $J = 6.9$ Hz, 1H), 3.87 (dd, $J = 12.9, 6.9$ Hz, 1H), 3.76 (dd, $J = 12.9, 6.8$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 138.84, 127.35, 126.34, 124.05, 120.70, 110.03, 71.22, 51.88, 27.75; HRMS m/z $[\text{M} + \text{H}]^+$ calculated for $[\text{C}_{14}\text{H}_{11}\text{N}_7]^+$: 278.30223; found: 278.30201.



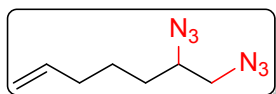
1-(2-azido-1,2-diphenylethoxy)-2,2,6,6-tetramethylpiperidine (34): Following the procedure employed for compound **2**, using *trans*-stilbene (**1ae**, 100 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as yellow oil (199 mg, 0.527 mmol, 95%, 1.3:1 *dr*).

^1H NMR (400 MHz, CDCl_3) δ 7.14-7.01 (m, 6H), 6.95-6.82 (m, 4H), 5.03 (d, $J = 8.0$ Hz, 1H), 4.91 (d, $J = 8.0$ Hz, 1H), 4.80 (d, $J = 3.4$ Hz, 1H), 1.66-1.22 (m, 15H), 0.23 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 138.69, 137.05, 136.93, 136.64, 130.66, 129.51, 129.03, 128.34, 127.96, 127.92, 127.71, 127.64, 127.54, 127.27, 127.21, 127.15, 126.89, 91.42, 88.99, 69.14, 67.85, 60.10, 40.80, 40.56, 35.09, 34.11, 20.22, 17.02; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[1,2]



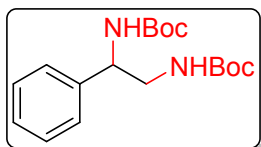
1,2-diazido-1,2-diphenylethane (35): Following general procedure for diazidation using *trans*-stilbene (**1af**, 100 mg, 0.555 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as yellow oil (134 mg, 0.510 mmol, 92%, 2.6:1 *dr*).

^1H NMR (400 MHz, CDCl_3) δ 7.34-7.27 (m, $J = 4.7, 1.7$ Hz, 4H), 7.21-7.12 (m, 10H), 7.03-6.95 (m, 6H), 4.69 (s, 1H), 4.54 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 135.71, 129.90, 129.01, 128.94, 128.67, 128.65, 128.53, 127.92, 127.63, 70.69, 69.61; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[2,6]



6,7-diazidohept-1-ene (36): Following general procedure for diazidation using 1,6-heptadiene (**1ag**, 71 mg, 100 μL , 0.738 mmol) and purified by silica gel column chromatography, eluting with hexanes afforded the title compound as clear liquid (63 mg, 0.354 mmol, 48%).

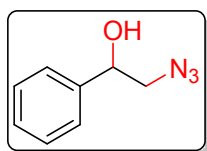
^1H NMR (400 MHz,) δ 5.90-5.68 (m, 1H), 5.10-4.93 (m, 2H), 3.53-3.43 (m, 1H), 3.43-3.36 (m, 1H), 3.32 (dd, $J = 12.6, 7.4$ Hz, 1H), 2.18-2.04 (m, 2H), 1.73-1.35 (m, 4H); ^{13}C NMR (101 MHz,) δ 137.72, 115.32, 61.89, 54.79, 33.21, 31.09, 25.01; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[3e]



di-tert-butyl (1-phenylethane-1,2-diyl)dicarbamate (37): A preformed solution of PPh_3 (306 mg, 1.170 mmol, 2.2 equiv.) and H_2O (48 μL 2.659 mmol, 5 equiv.) in THF (5 mL) was treated with compound **3** (100 mg, 0.531 mmol, 1. equiv., prepared by diazidation of styrene) at 0 $^\circ\text{C}$. The reaction was stirred at room temperature until the complete conversion of starting material as observed by TLC. Subsequently, a solution of di-*t*-butylpyrocarbonate (Boc anhydride) (366 μL 1.595 mmol, 3.0 equiv.) in THF (2 mL) was added to the above reaction mixture dropwise at room temperature. The resulting mixture was then stirred for additional 12 h or until diamine intermediates were fully consumed (monitored by TLC). The reaction mixture was concentrated *in vacuo*, the residue was subsequently purified by column chromatography (Hexane/EtOAc:

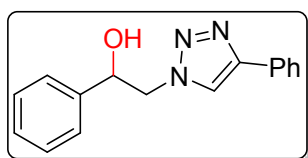
from 10:1 to 2:1) to afford the desire *N*-Boc protected diamine product **37** as white solid (151 mg, 0.451 mmol, 85%).

Mp:144-146 °C; IR (Film): 3350, 2196, 1720, 1456, 1150, 1225, 766 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.30 (m, 2H), 7.29-7.24 (m, 3H), 5.54 (s, 1H), 4.89 (m, 1H), 4.74 (m, 1H), 3.51-3.31 (m, 2H), 1.84 (s, 1H), 1.43 (s, 9H); ¹³C NMR (101 MHz, CDCl₃) δ 156.80, 155.76, 140.14, 128.67, 127.54, 126.31, 79.69, 55.90, 45.79, 28.30; HRMS (ESI-TOF) m/z [M + Na]⁺ calculated for [C₁₈H₂₈N₂NaO₄]⁺: 359.42122; found; 359.42133.



2-azido-1-phenylethan-1-ol (38): Following a slightly modified reported procedure, a solution of 1-(2-azido-1-phenylethoxy)-2,2,6,6-tetramethylpiperidine (**2**, 100 mg, 0.331 mmol, 1.1 equiv.) in H₂O/AcOH (3 mL:1 mL) was treated with Zn-dust (129 mg, 6.0 equiv.) at room temperature. Additional Zn-dust (129 mg, 6.0 equiv.) was added the above reaction mixture after 3h and continued to stirred over night at room temperature. The reaction as the treated with diluted sodium hydroxide solution to adjust the pH to 14. The resulting mixture was extracted with CH₂Cl₂ (3 x 30 mL), The combined organic layers were washed with brine solution, dried over anhydrous Na₂SO₄. The solvent was evaporated under reduced pressure and the crude product was purified by column chromatography on silica gel by using a mixture of EtOAc/hexane as an eluent to provide analytical pure product **38** as oily liquid (50 mg, 0.311 mmol 94%).

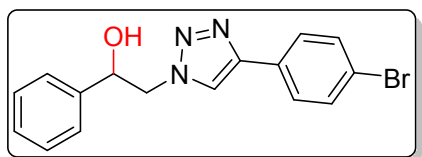
¹H NMR (400 MHz, CDCl₃) δ 7.48-7.29 (m, 5H), 4.88 (dd, *J* = 7.9, 4.1 Hz, 1H), 3.48 (dd, *J* = 12.6, 7.9 Hz, 1H), 3.44 (dd, *J* = 12.6, 4.1 Hz, 1H), 2.39 (s, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 140.51, 128.69, 128.36, 125.88, 73.44, 58.09; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[1,7]



1-phenyl-2-(4-phenyl-1H-1,2,3-triazol-1-yl)ethan-1-ol (39): Following a slightly modified reported procedure, compound 2-azido-1-phenylethan-1-ol (**38**, 100 mg, 0.613 mmol, 1.0 equiv.) was treated with phenylacetylene (84 μL, 0.736 mmol, 1.2 equiv.), CuI (400 mg, 1.5 equiv.) and

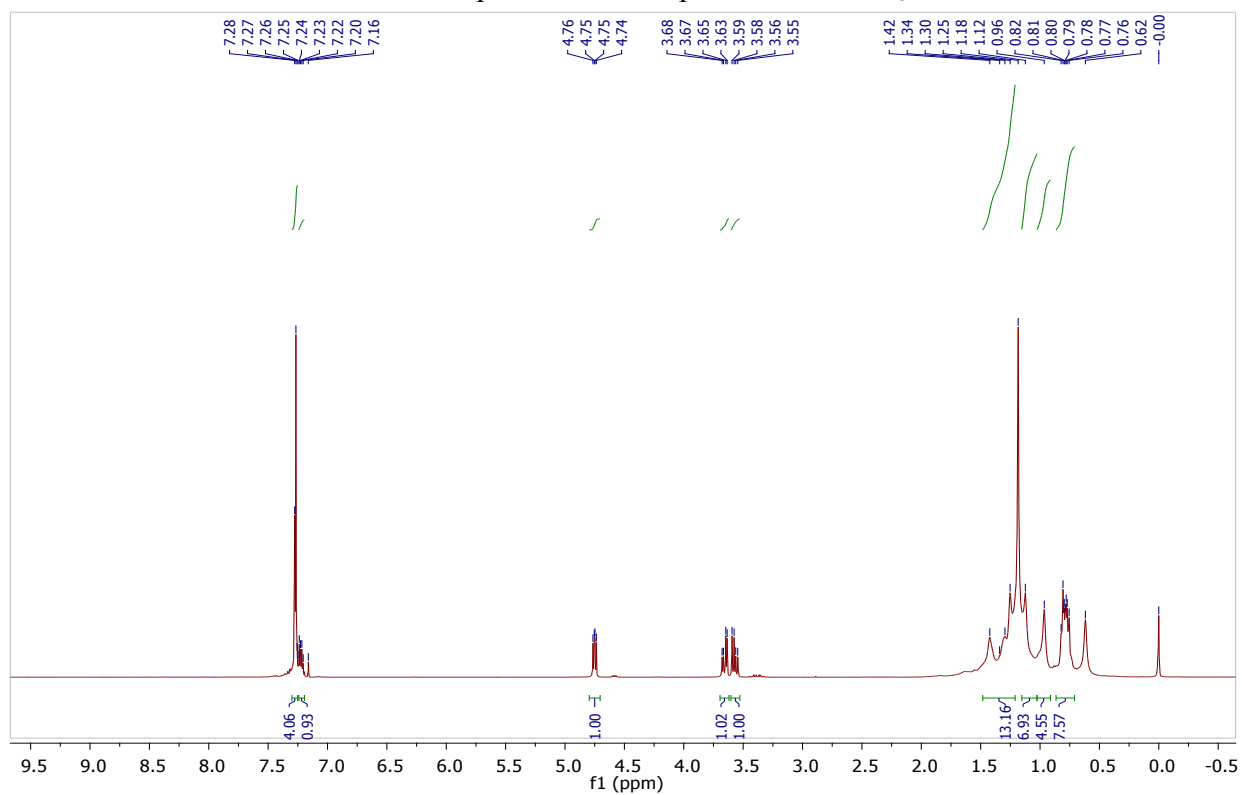
N,N-diisopropylethylamine (319 μL , 1.84 mmol, 3.0 equiv.) in CH_3CN (2 mL) and stirred at room temperature for 3h or till the consumption of starting material. The resulting residues after usual work-up were purified by silica gel column chromatography, eluting with EtOAc/hexanes afforded the title compound as white solid (146 mg, 0.551 mmol, 90%).

Mp. 105-107 $^\circ\text{C}$; IR (CHCl_3 , cm^{-1}): 3307, 2922, 2852, 1712, 1462, 1221, 1065, 770, 693; ^1H NMR (500 MHz, CDCl_3) δ 7.79 (s, 1H), 7.78-7.70 (m, 2H), 7.46-7.27 (m, 8H), 5.24 (dd, $J = 8.8$, 3.2 Hz, 1H), 4.65 (dd, $J = 14.0$, 3.2 Hz, 1H), 4.45 (dd, $J = 14.0$, 8.9 Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 147.36, 140.14, 130.29, 128.79, 128.47, 128.10, 125.87, 125.61, 121.20, 72.88, 57.51; the overall spectroscopic data are in complete agreement with assigned structures and consistent with literature.^[1,7]

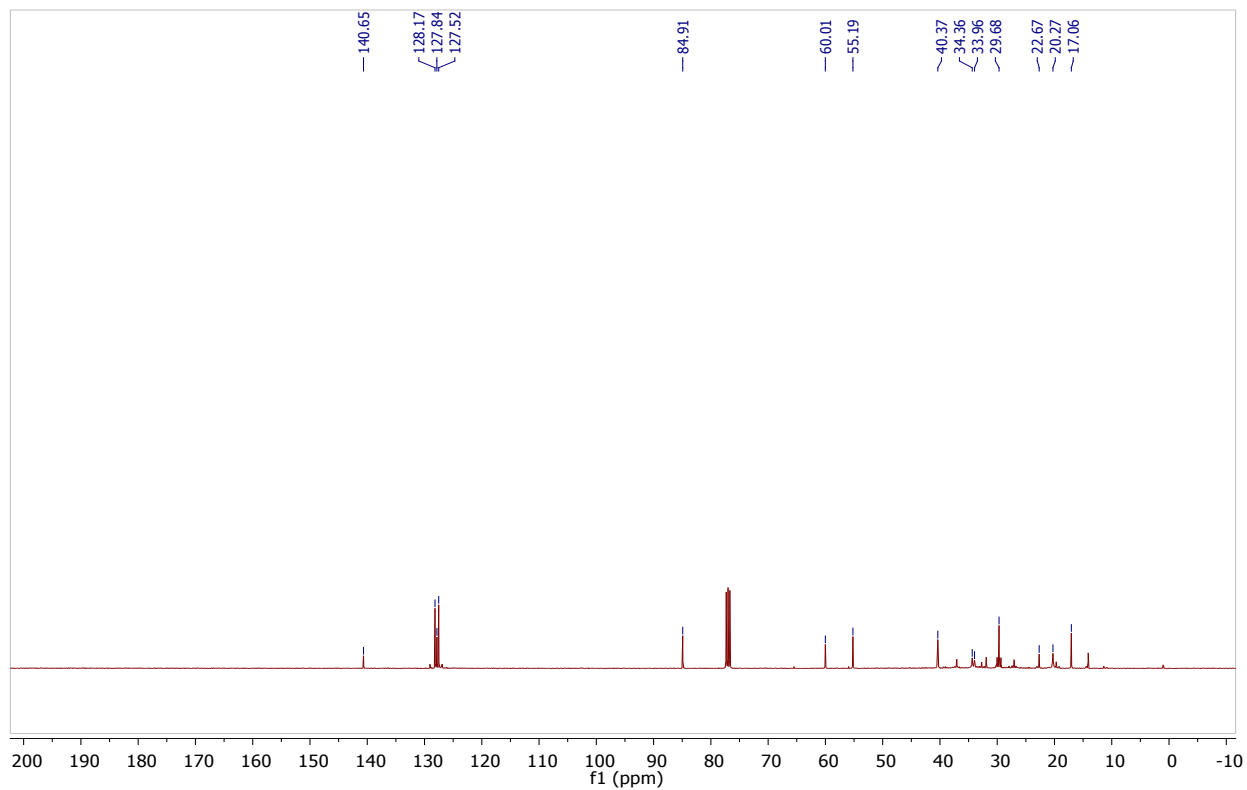


2-(4-(4-bromophenyl)-1H-1,2,3-triazol-1-yl)-1-phenylethan-1-ol (40): Following the procedure employed for compound **39** using 2-azido-1-phenylethan-1-ol (**38**, 100 mg, 0.613 mmol, 1.0 equiv.) and *p*-bromophenylacetylene (133 mg, 0.736 mmol, 1.2 equiv.), purified by silica gel column chromatography afforded the title compound as white solid (194 mg, 0.563 mmol, 92%). Mp. 128-130 $^\circ\text{C}$; IR (Film): 3500, 2199, 1650, 1546, 1448, 1222, 771, 655 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.74 (s, 1H), 7.52 (d, $J = 8.5$ Hz, 2H), 7.43 (d, $J = 8.2$ Hz, 2H), 7.39-7.25 (m, 5H), 5.18 (dd, $J = 8.8$, 2.6 Hz, 1H), 4.59 (dd, $J = 14.0$, 3.2 Hz, 1H), 4.36 (dd, $J = 14.0$, 8.9 Hz, 1H), 3.49 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 146.42, 139.99, 131.96, 129.34, 128.90, 128.65, 127.12, 125.85, 122.02, 121.28, 73.00, 57.44; HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ calculated for $[\text{C}_{16}\text{H}_{15}\text{BrN}_3\text{O}]^+$: 345.22421; found 345.22435.

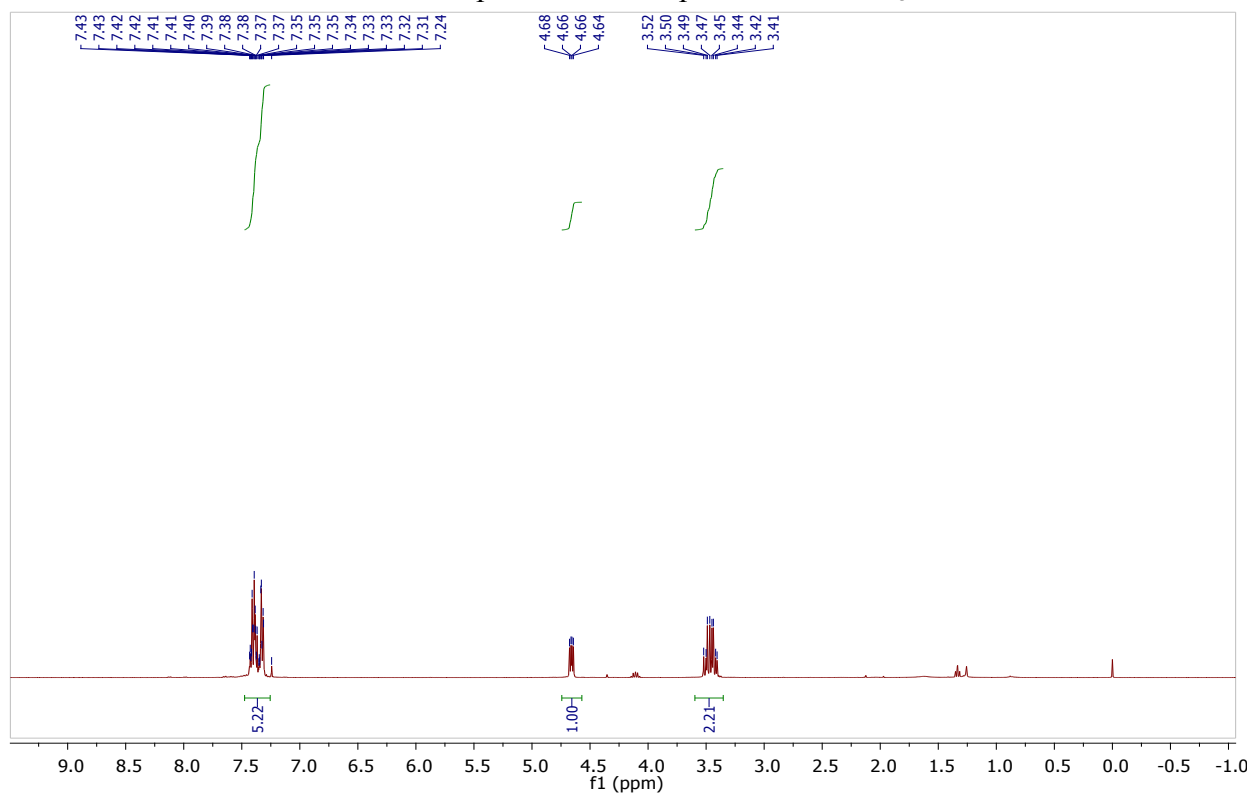
¹H NMR Spectrum of compound **2** in CDCl₃



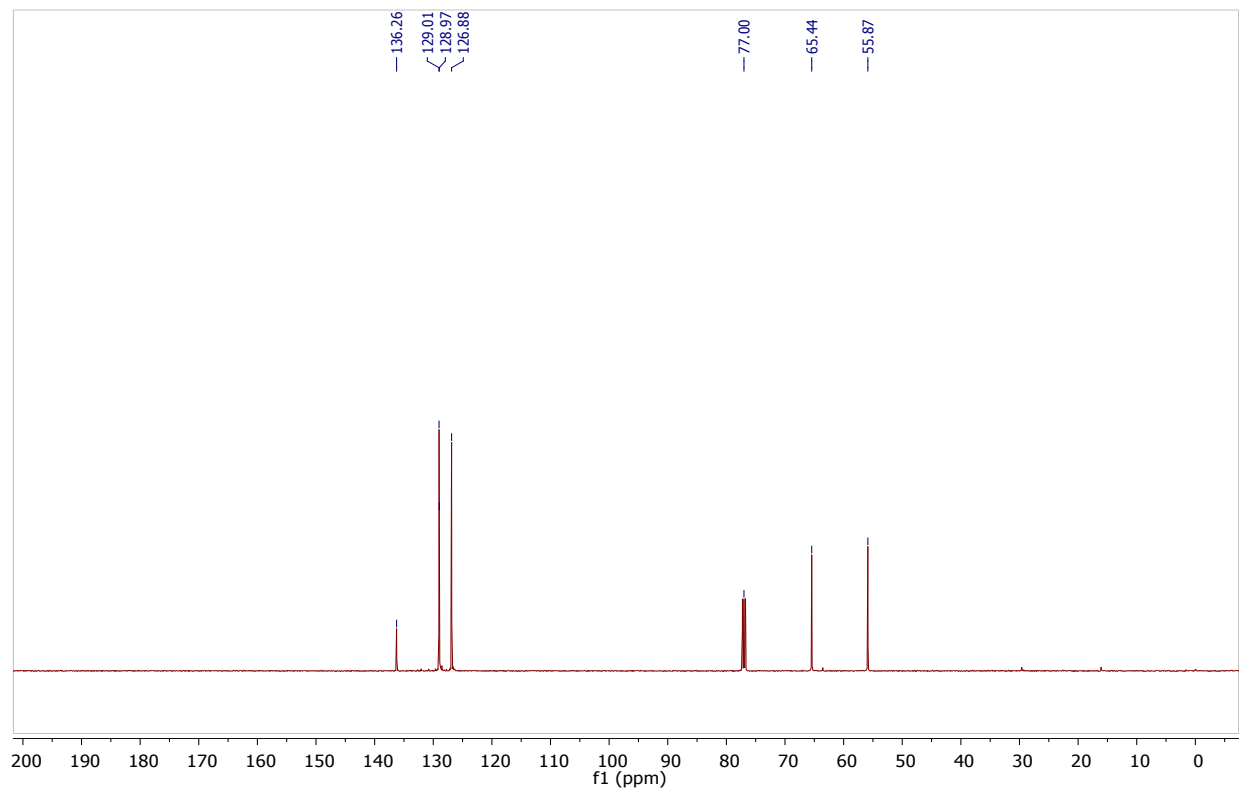
¹³C NMR Spectrum of compound **2** in CDCl₃



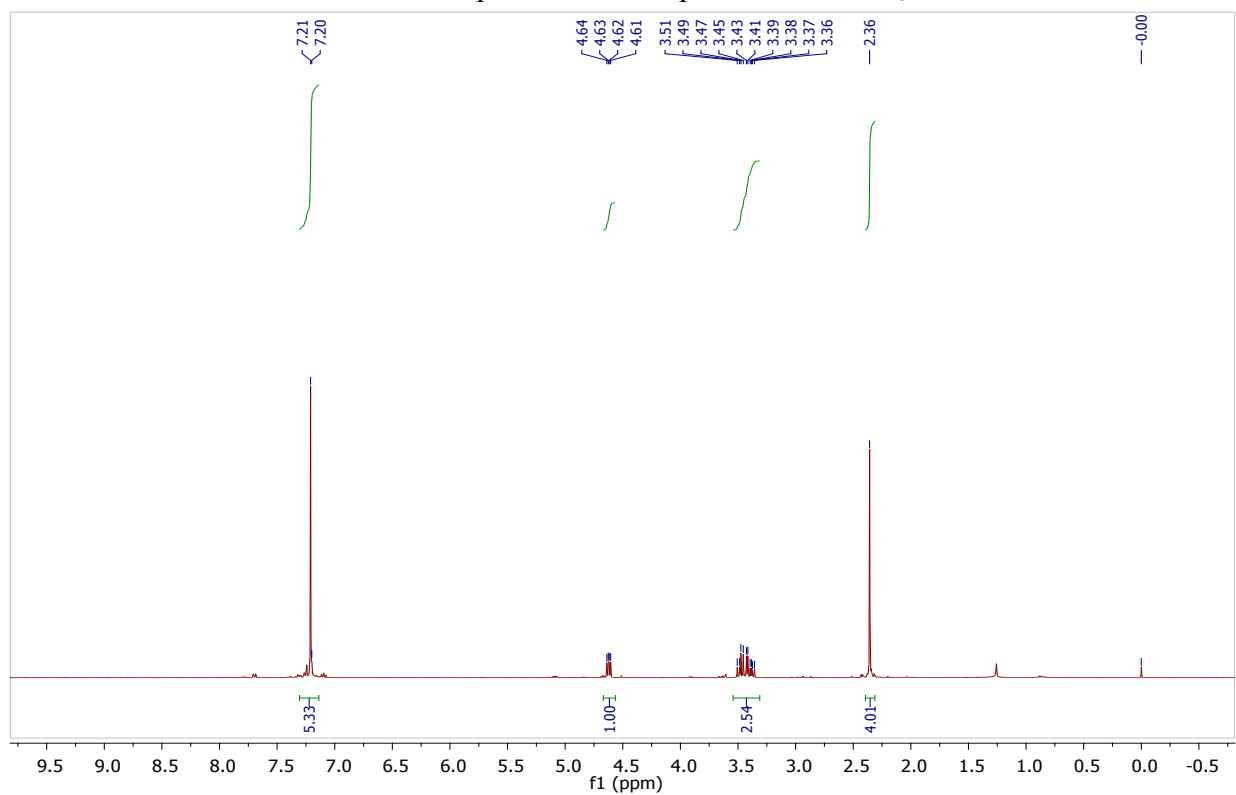
¹H NMR Spectrum of compound **3** in CDCl₃



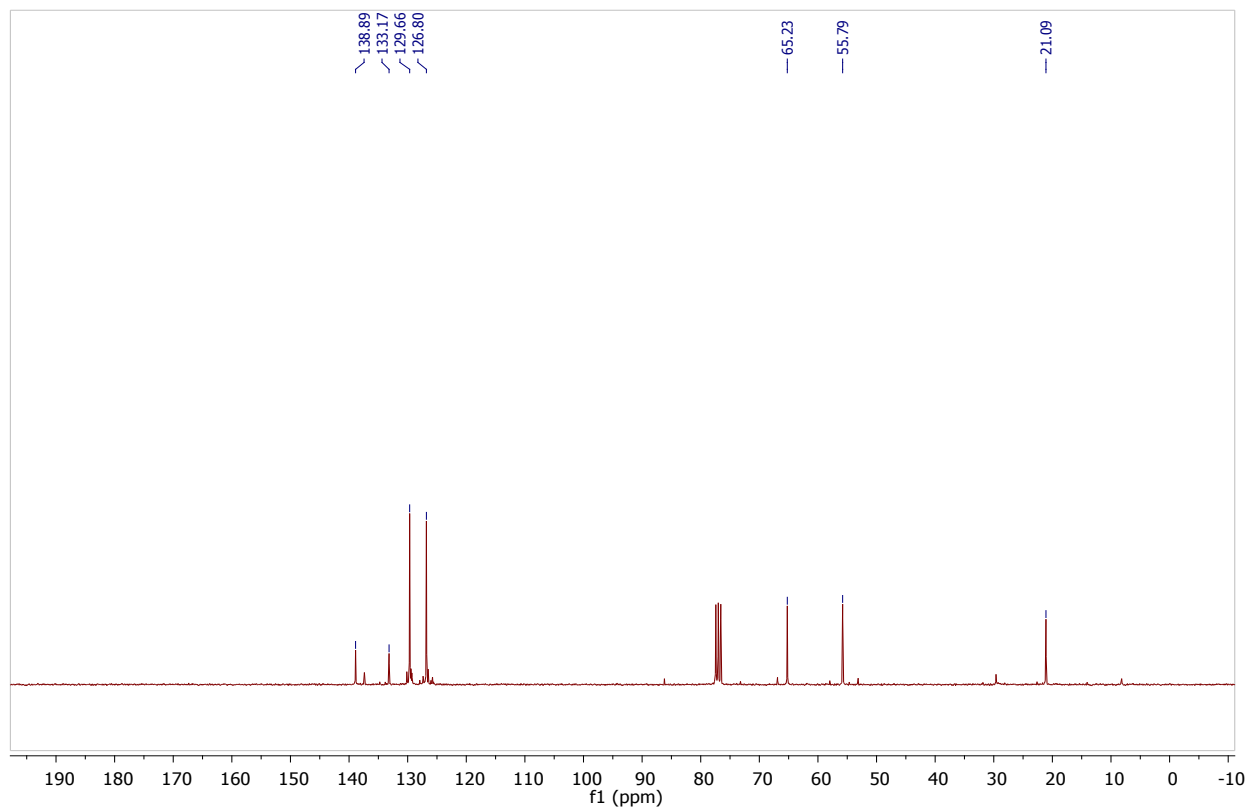
¹³C NMR Spectrum of compound **3** in CDCl₃



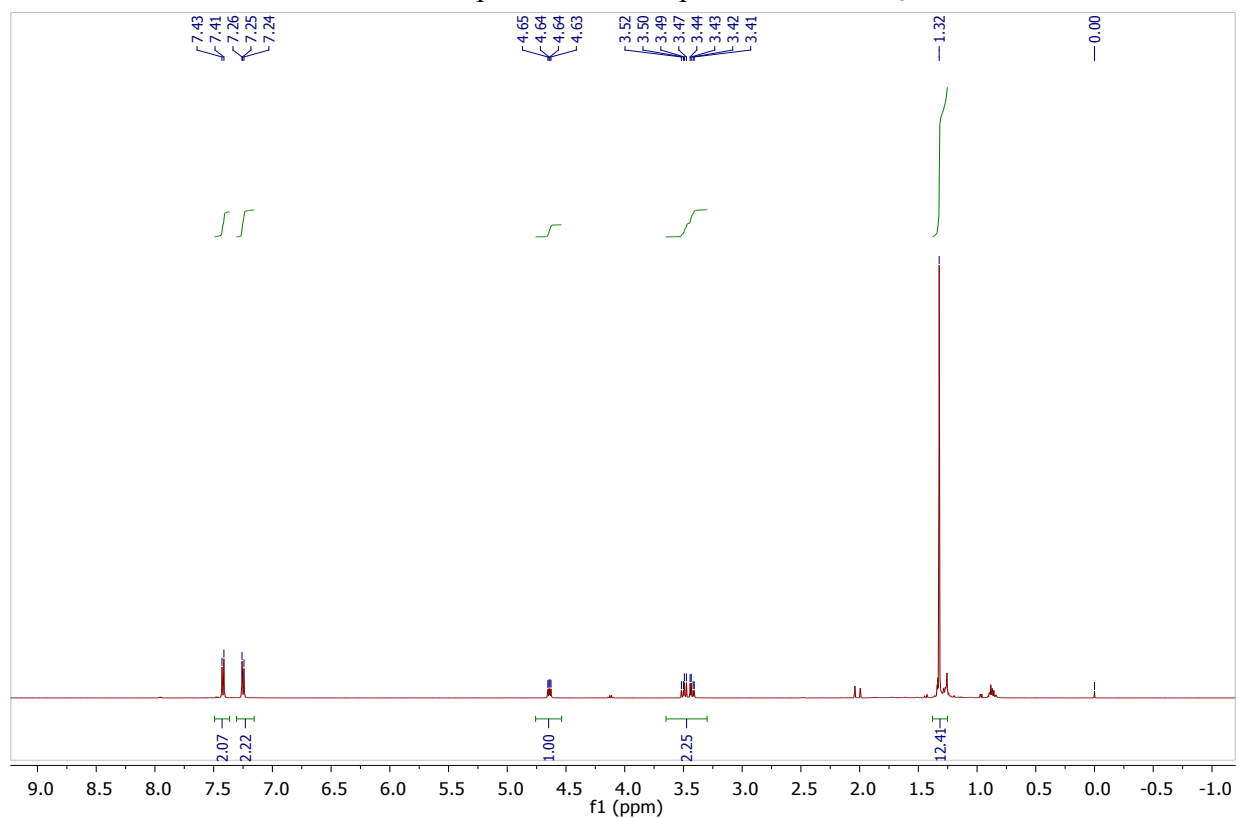
¹H NMR Spectrum of compound **4** in CDCl₃



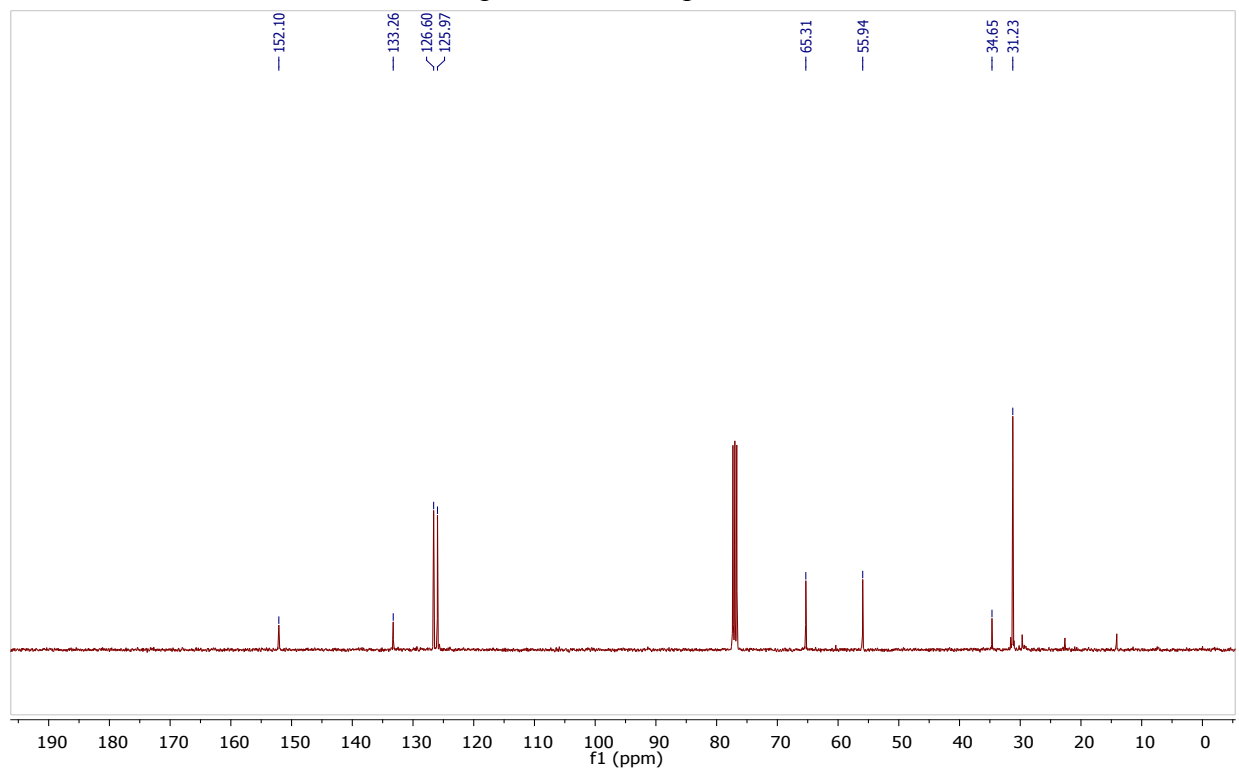
¹³C NMR Spectrum of compound **4** in CDCl₃



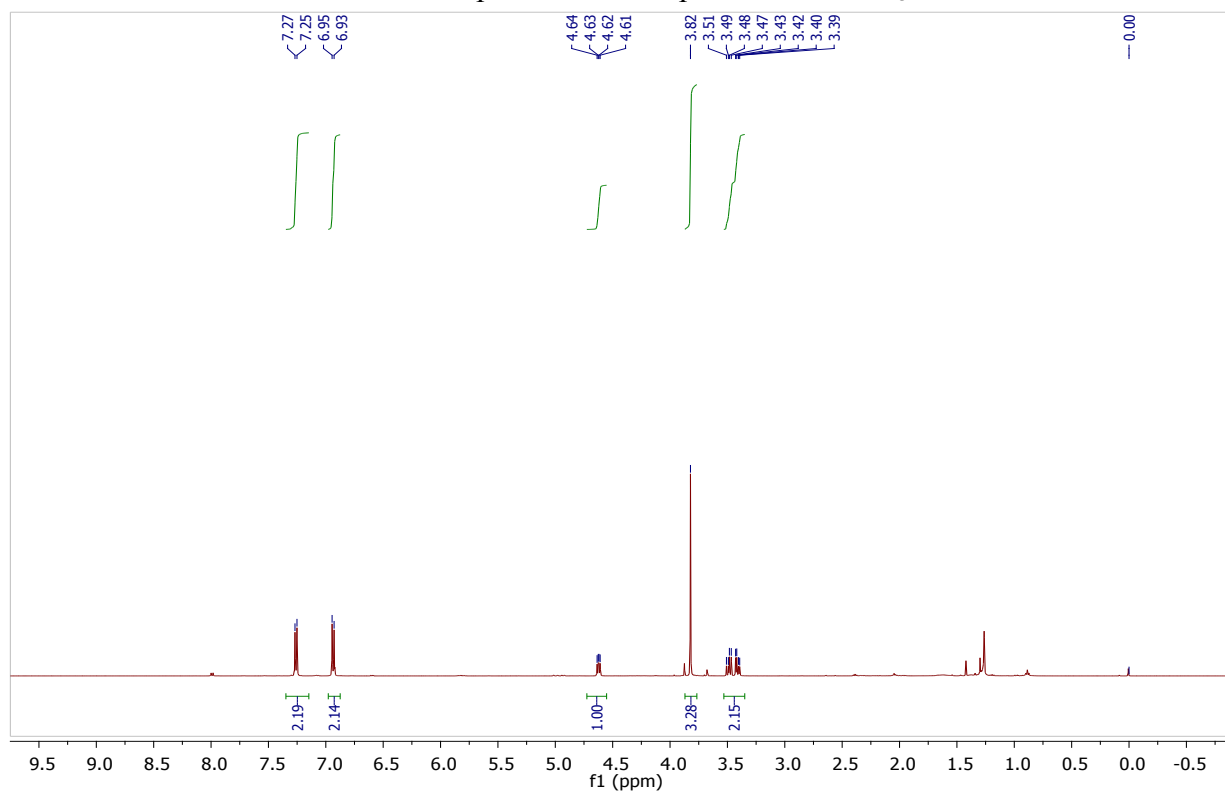
^1H NMR Spectrum of compound **5** in CDCl_3



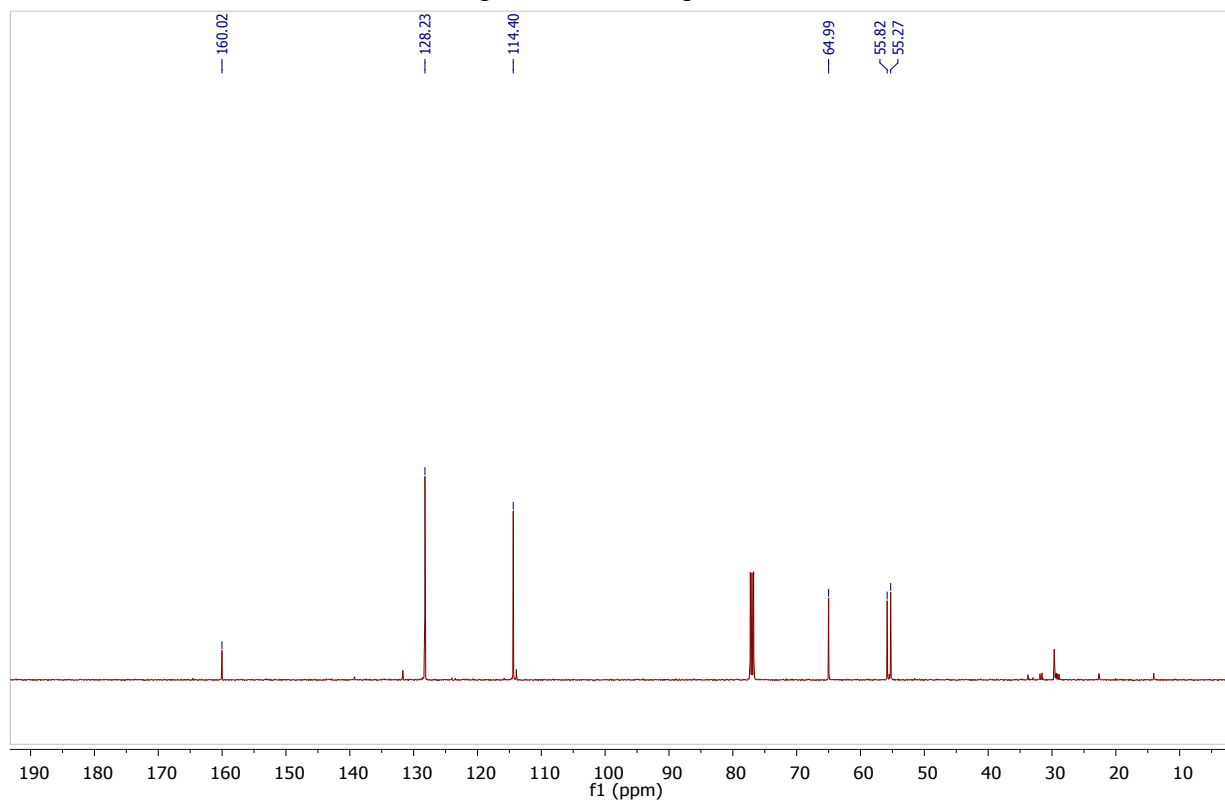
^{13}C NMR Spectrum of compound **5** in CDCl_3



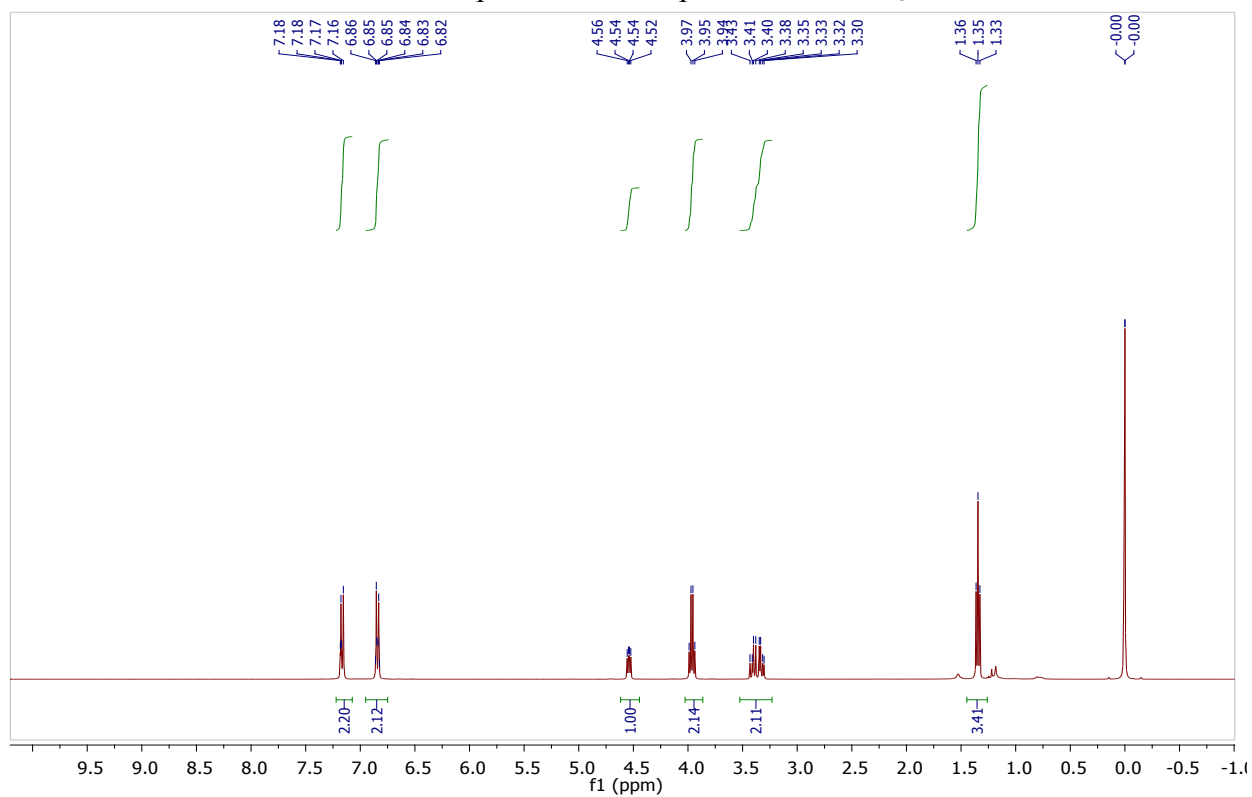
¹H NMR Spectrum of compound **6** in CDCl₃



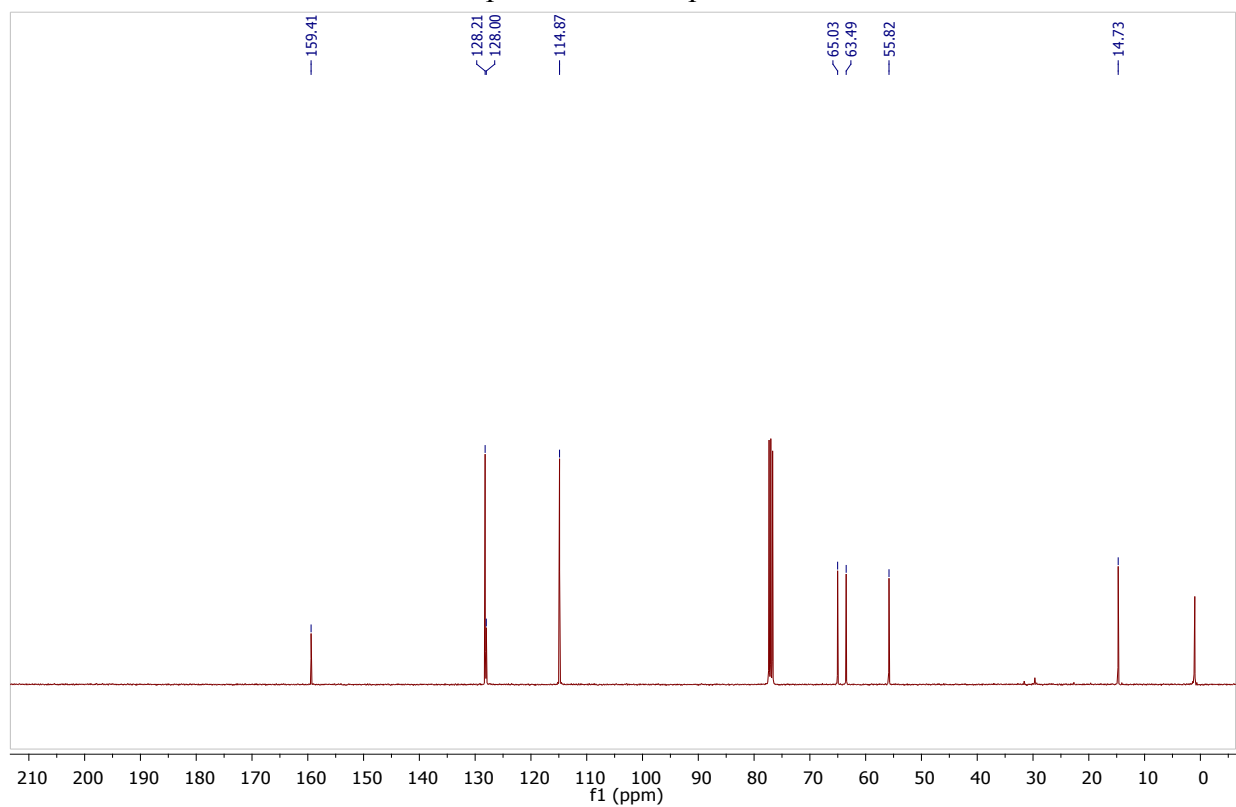
¹³C NMR Spectrum of compound **6** in CDCl₃



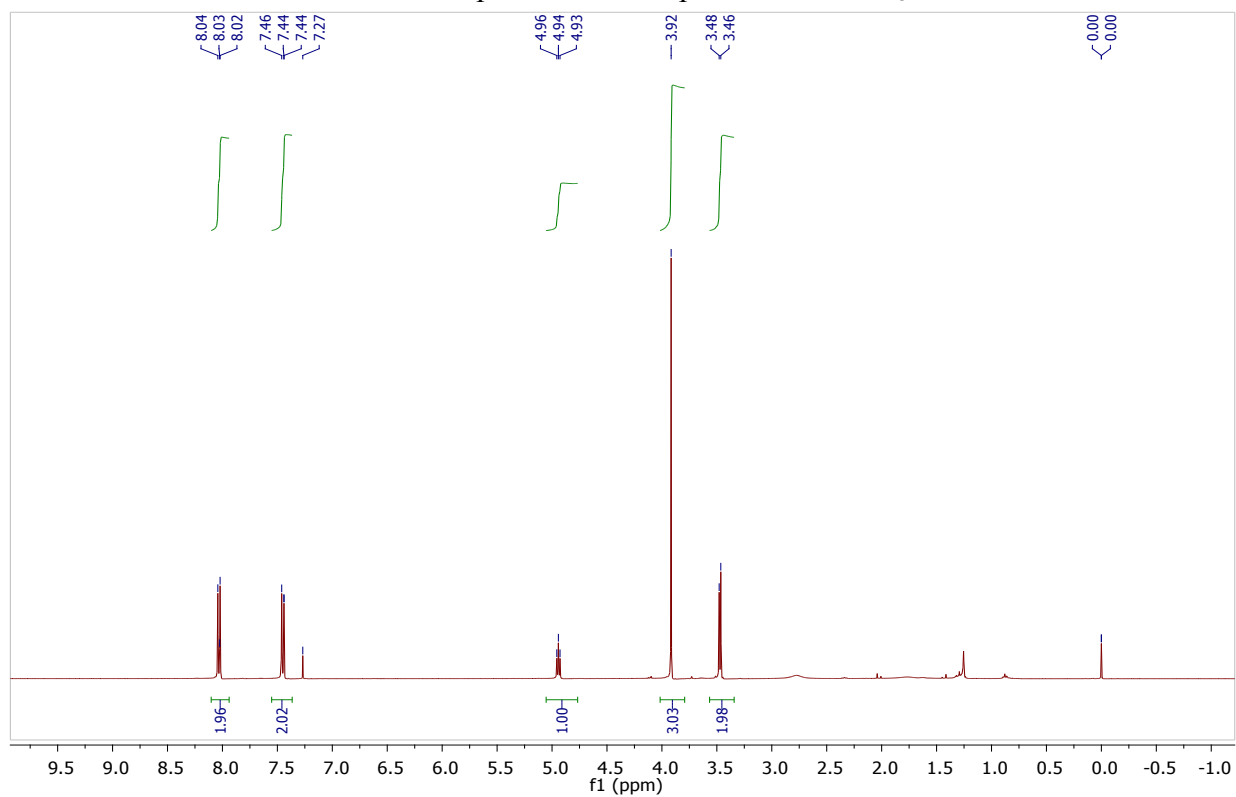
¹H NMR Spectrum of compound 7 in CDCl₃



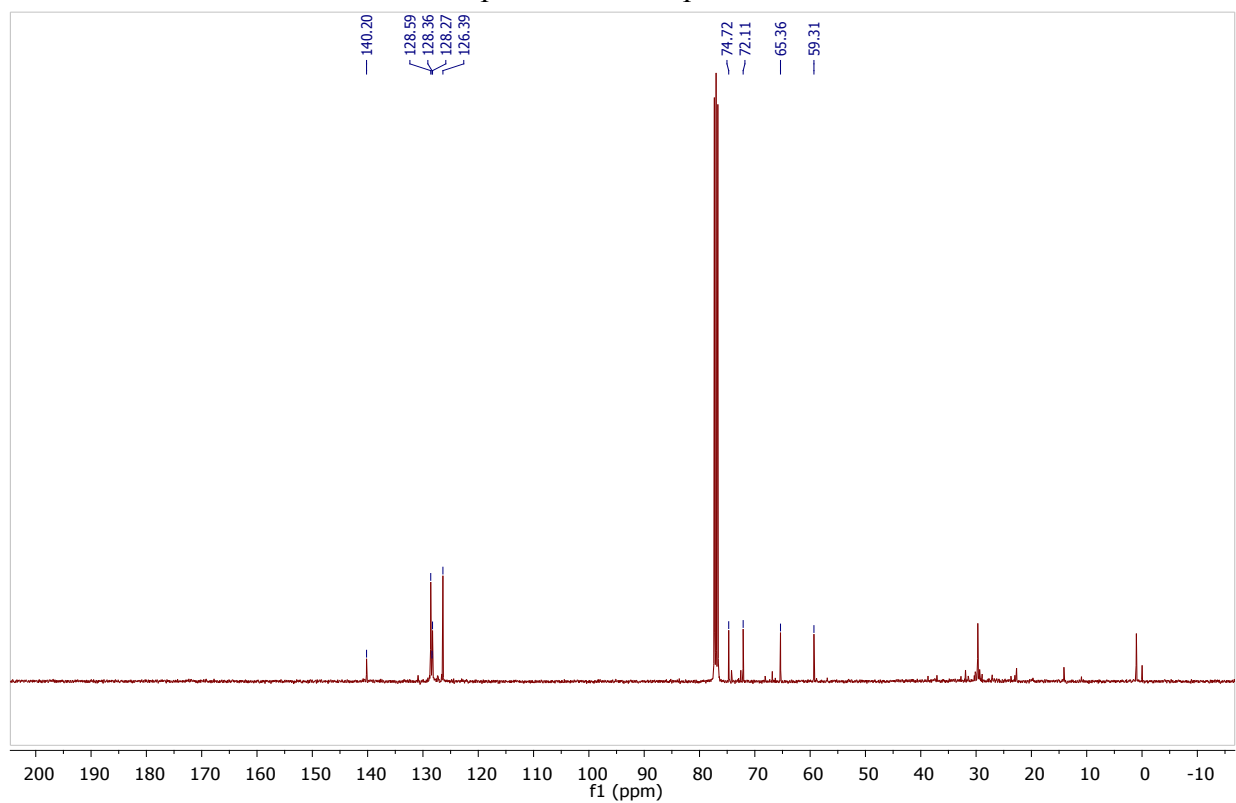
¹³C NMR Spectrum of compound 7 in CDCl₃



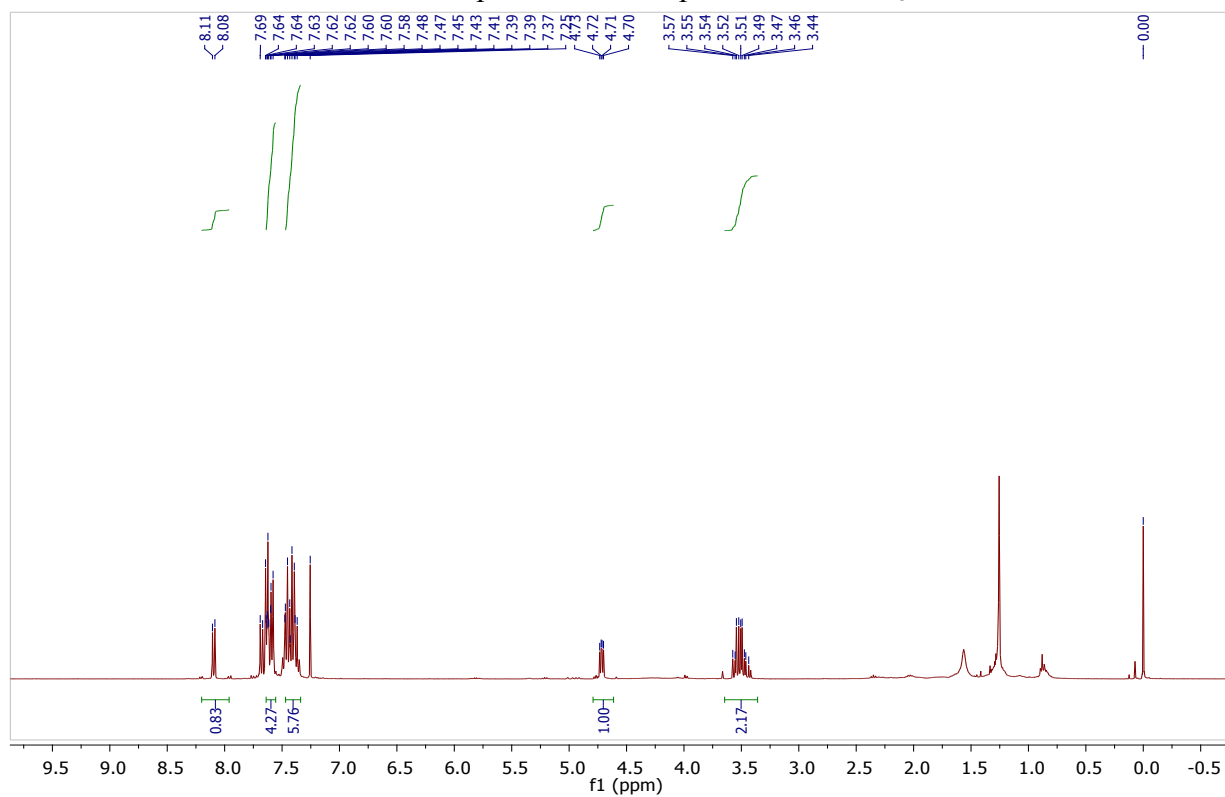
^1H NMR Spectrum of compound **8** in CDCl_3



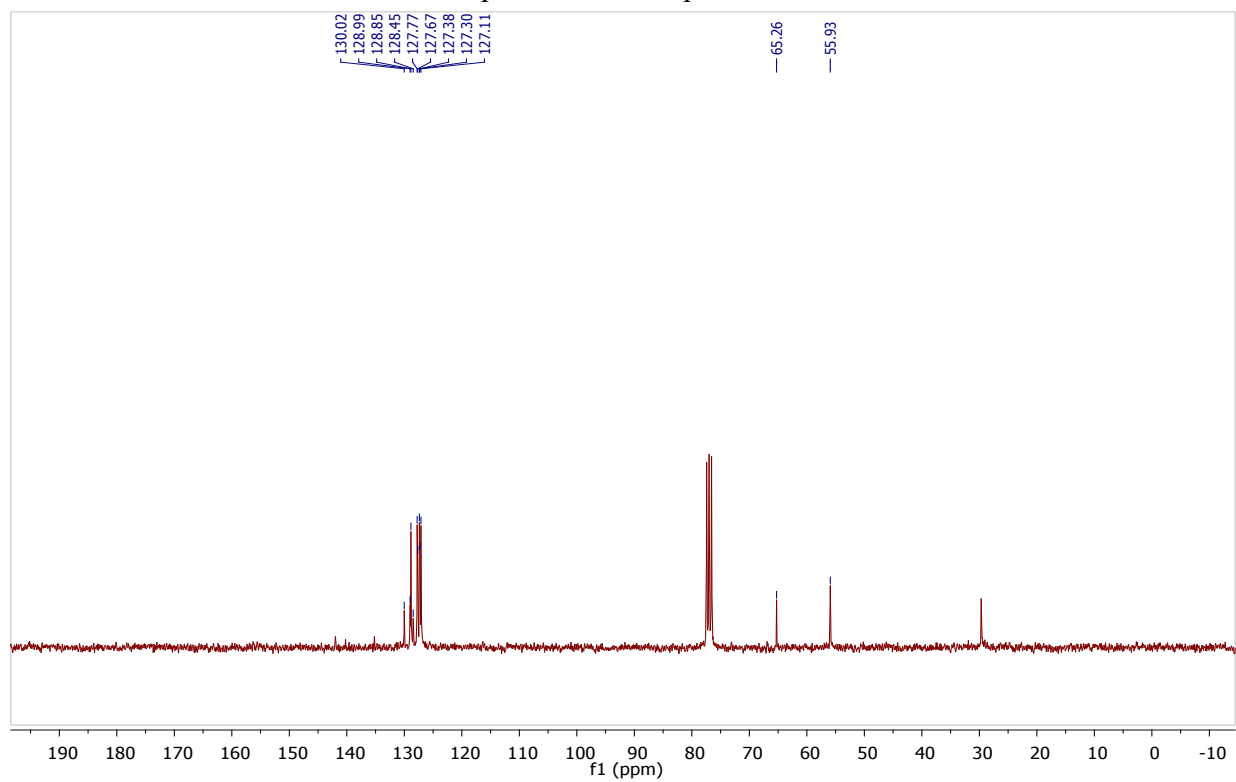
^{13}C NMR Spectrum of compound **8** in CDCl_3



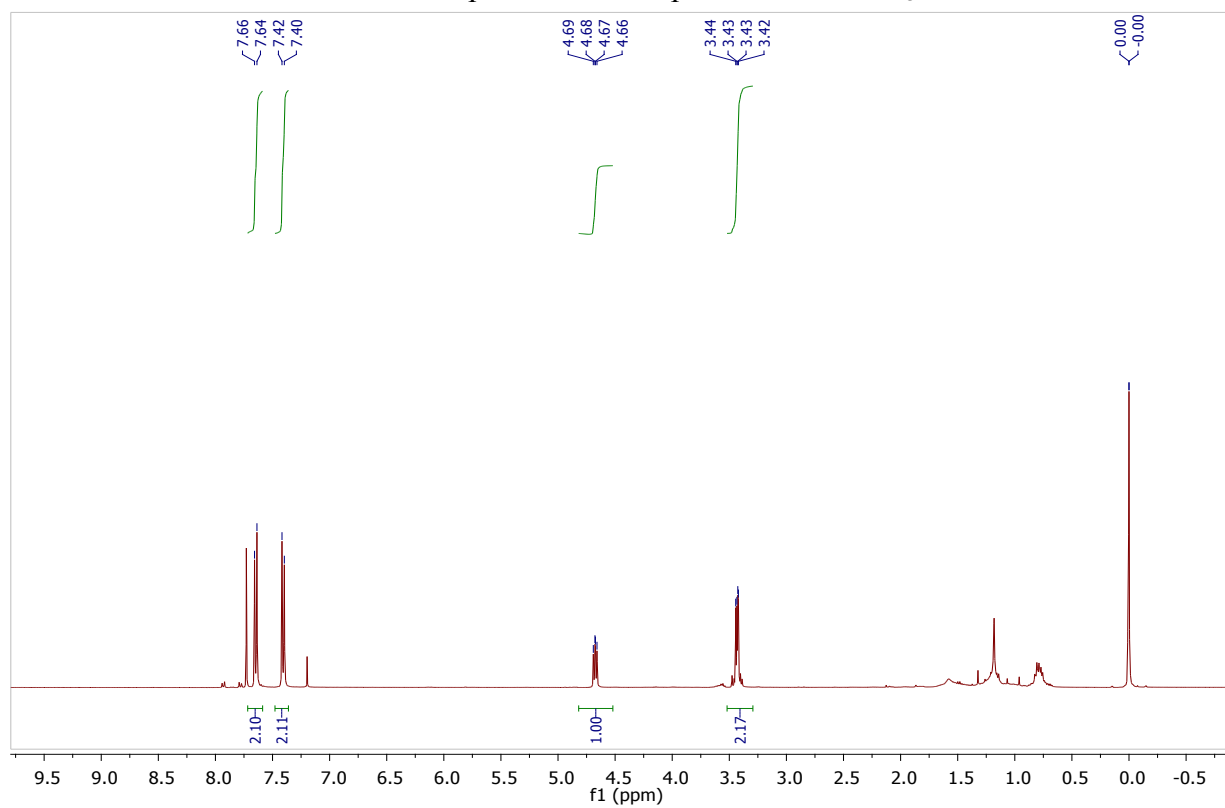
¹H NMR Spectrum of compound **9** in CDCl₃



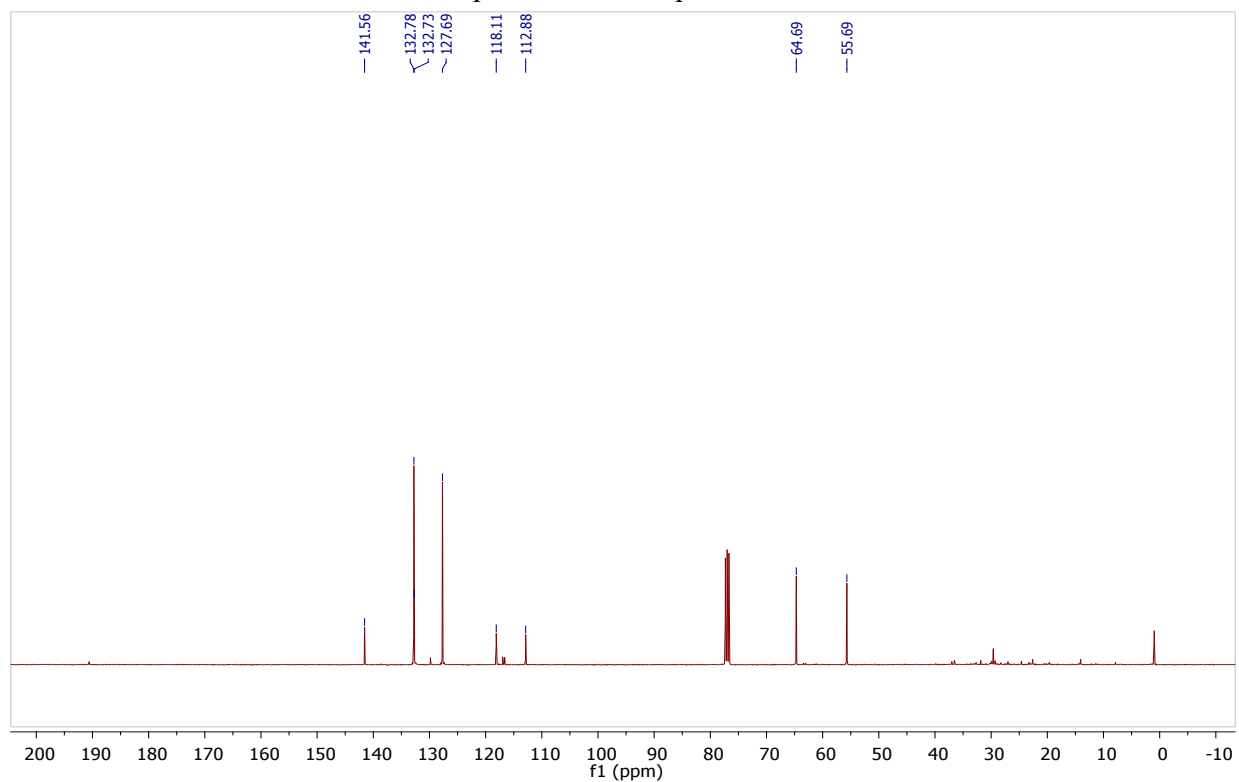
¹³C NMR Spectrum of compound **9** in CDCl₃



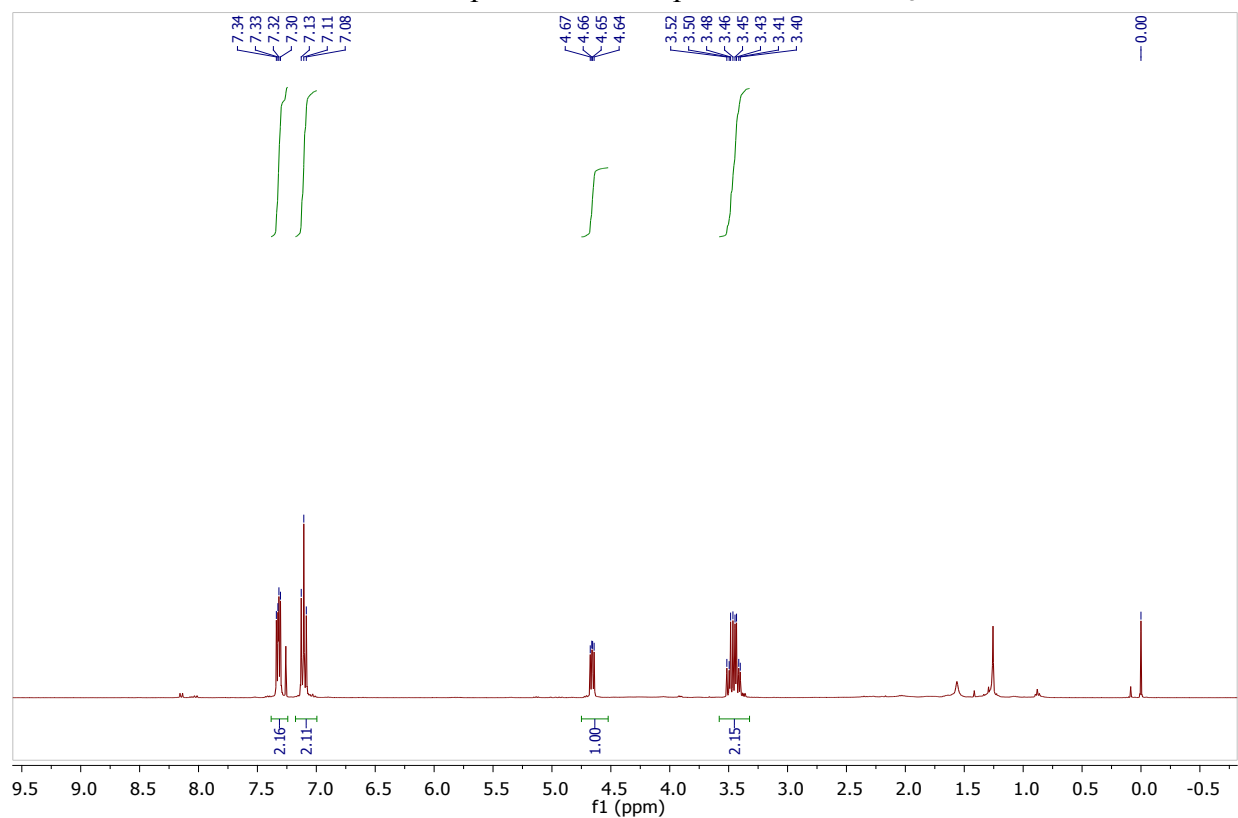
^1H NMR Spectrum of compound **10** in CDCl_3



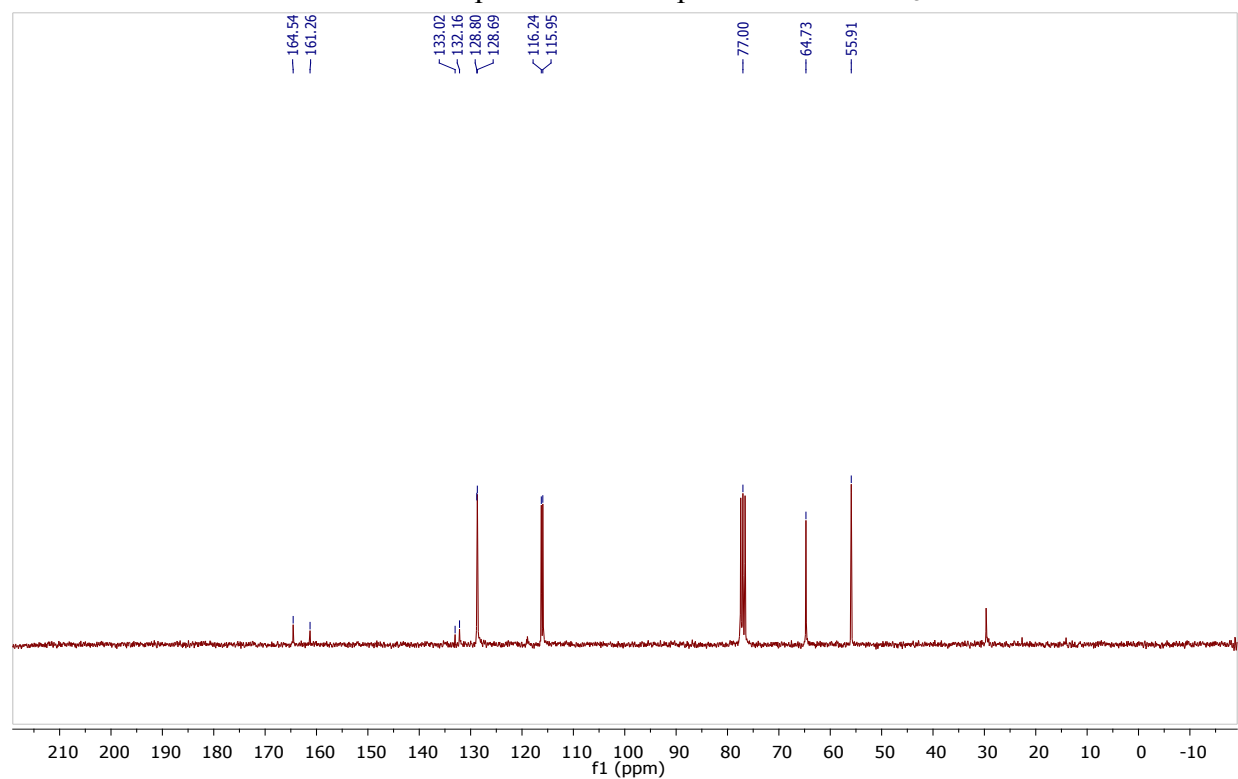
^{13}C NMR Spectrum of compound **10** in CDCl_3



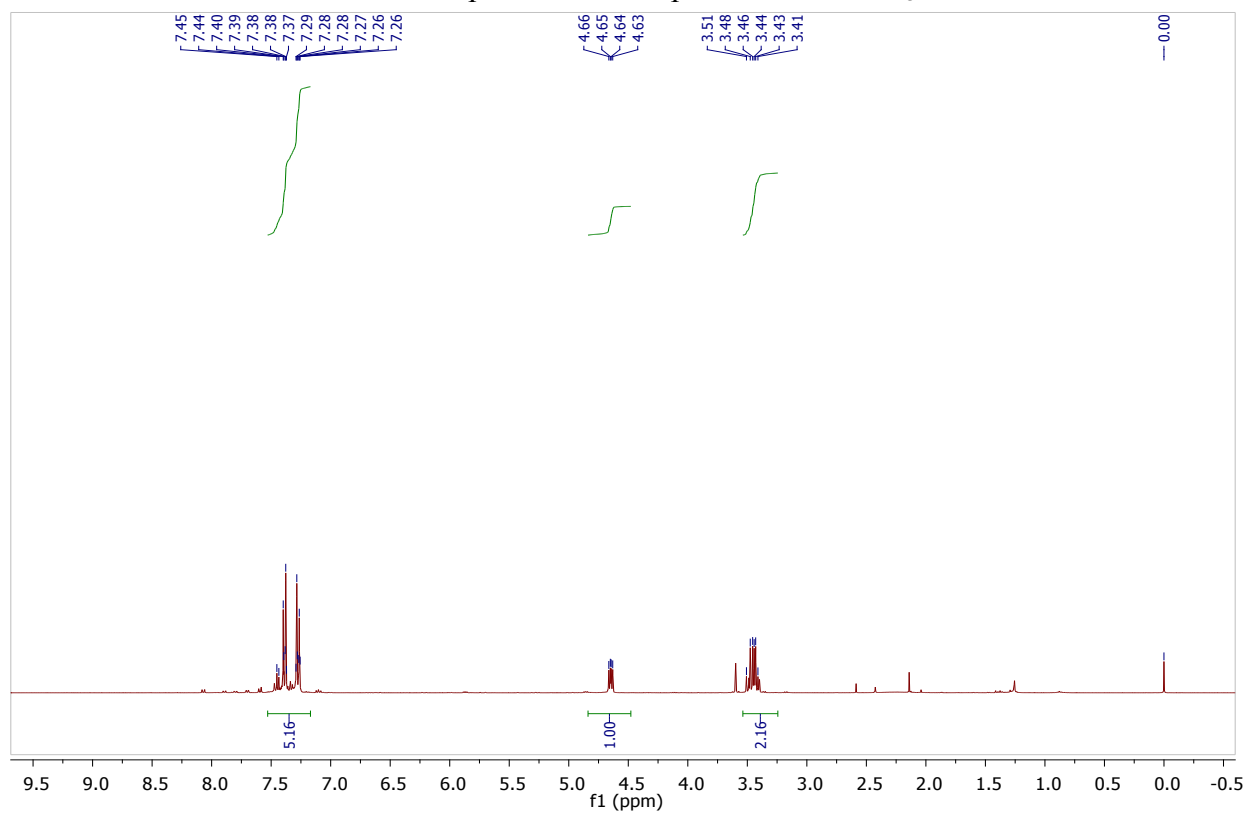
¹H NMR Spectrum of compound **11** in CDCl₃



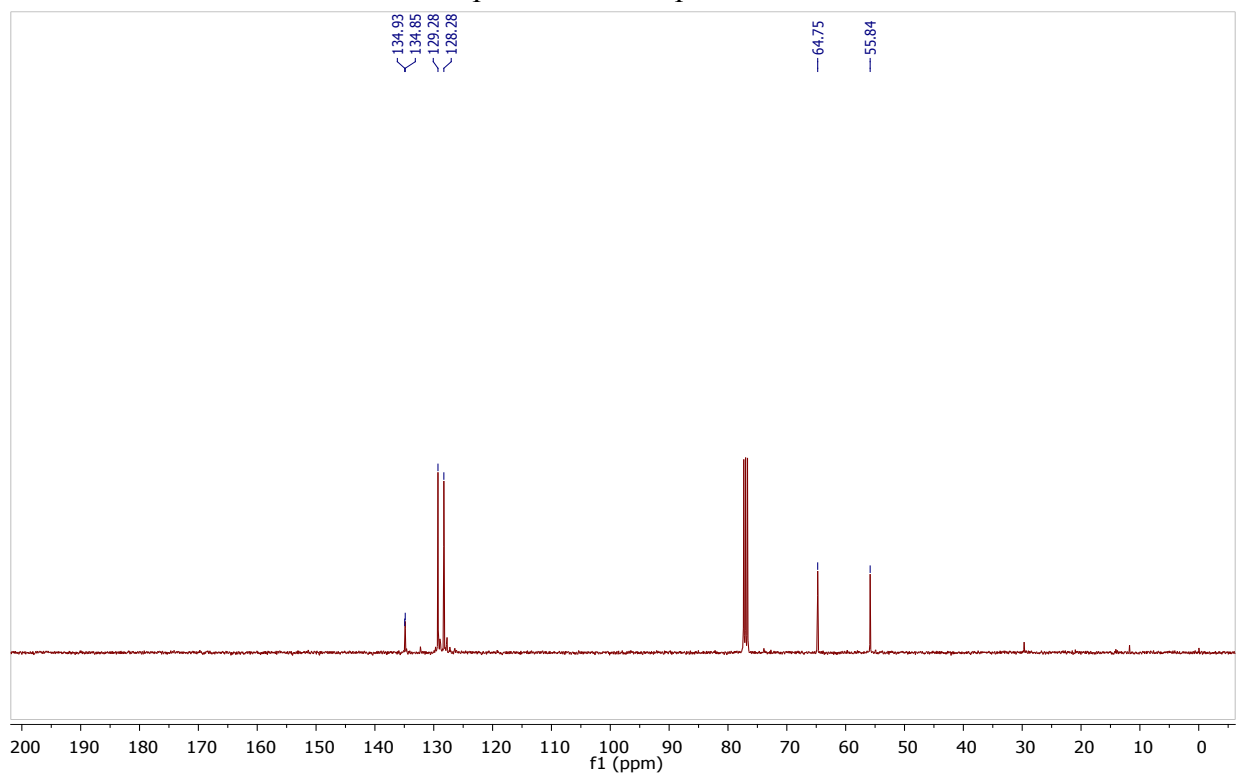
¹³C NMR Spectrum of compound **11** in CDCl₃



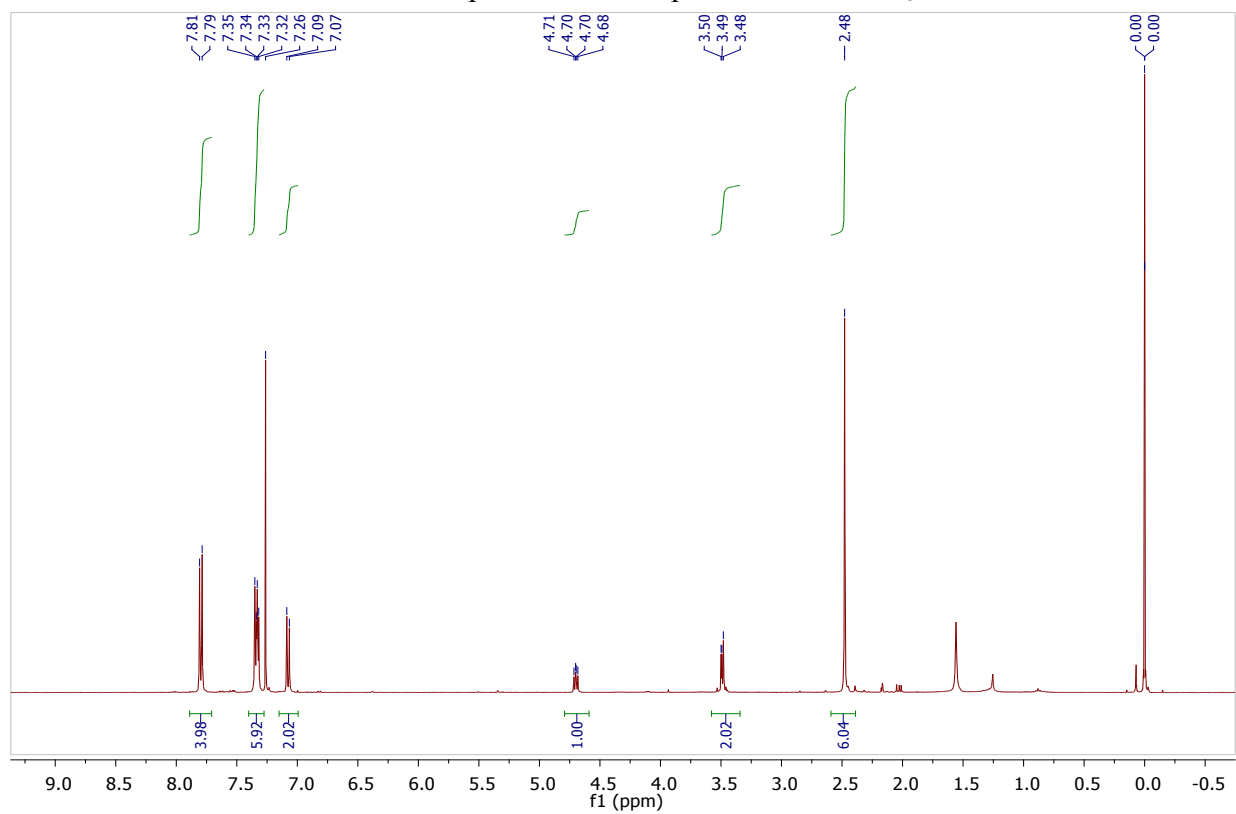
¹H NMR Spectrum of compound **12** in CDCl₃



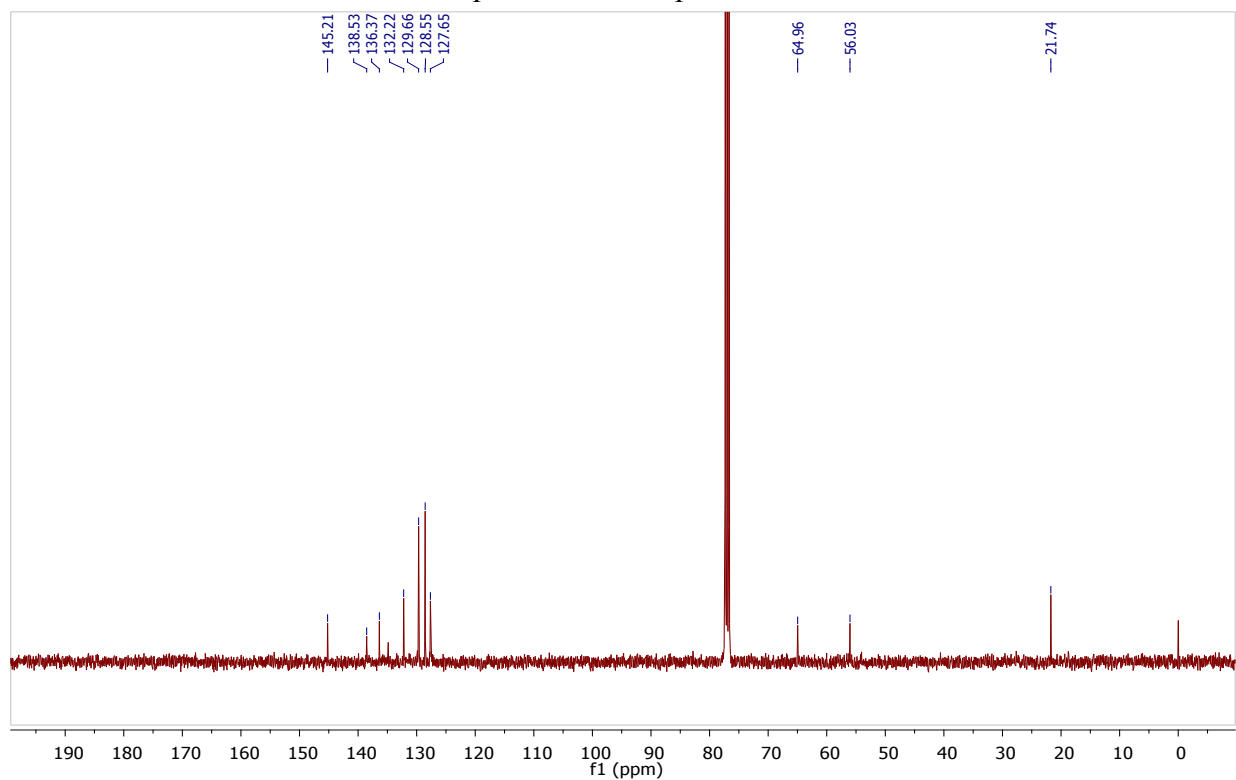
¹³C NMR Spectrum of compound **12** in CDCl₃



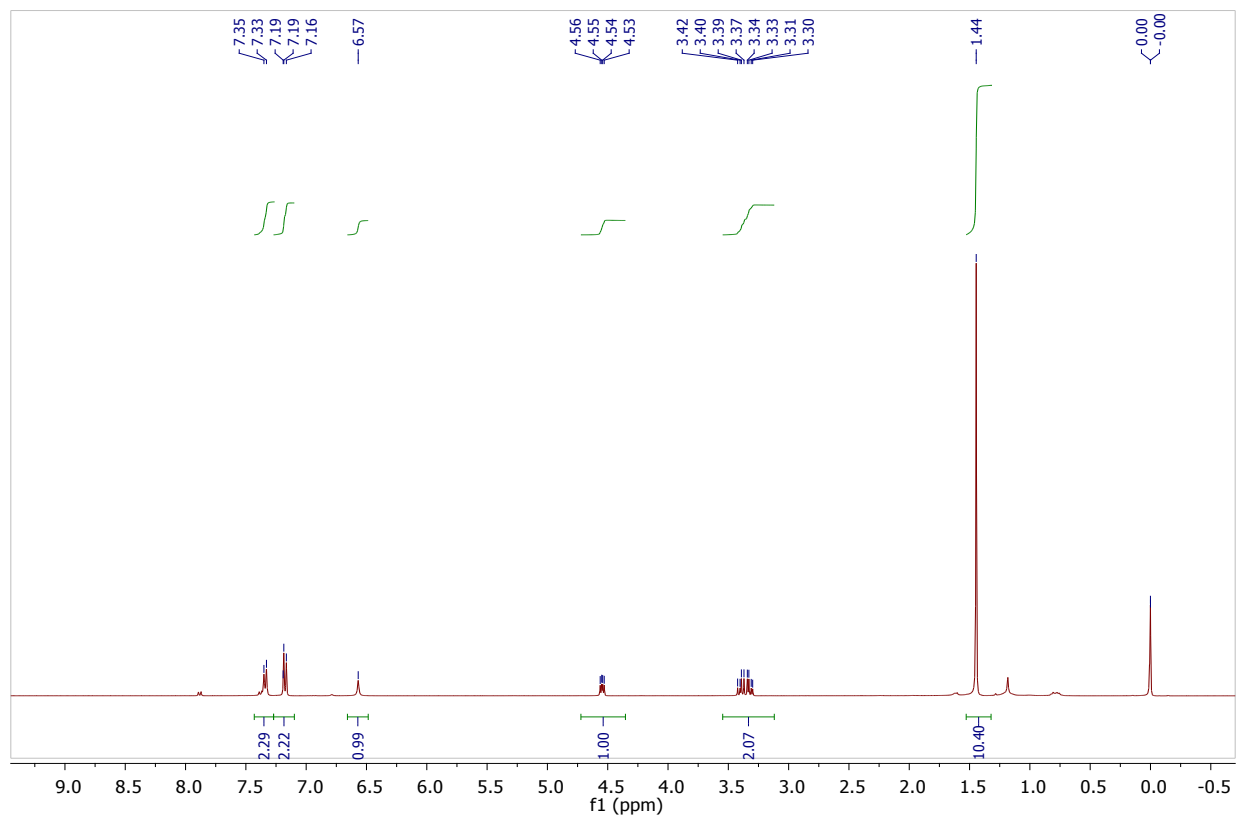
^1H NMR Spectrum of compound **13** in CDCl_3



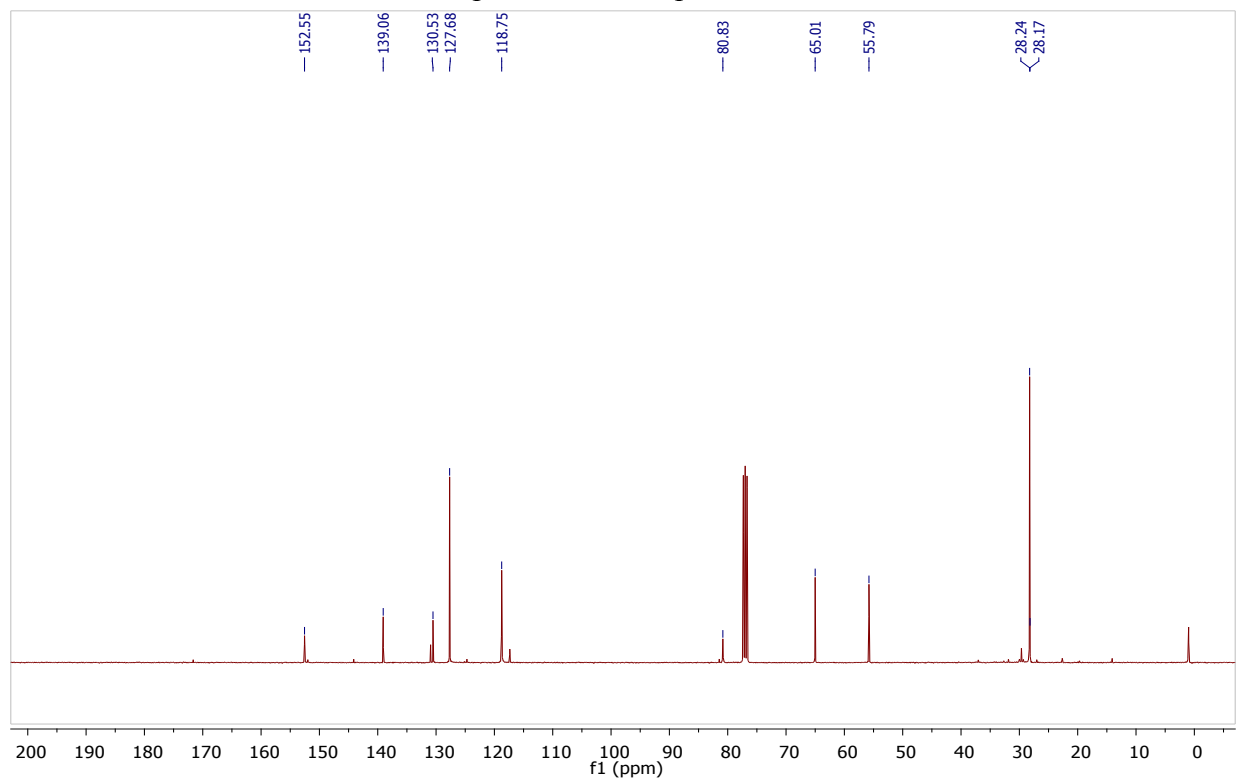
^{13}C NMR Spectrum of compound **13** in CDCl_3



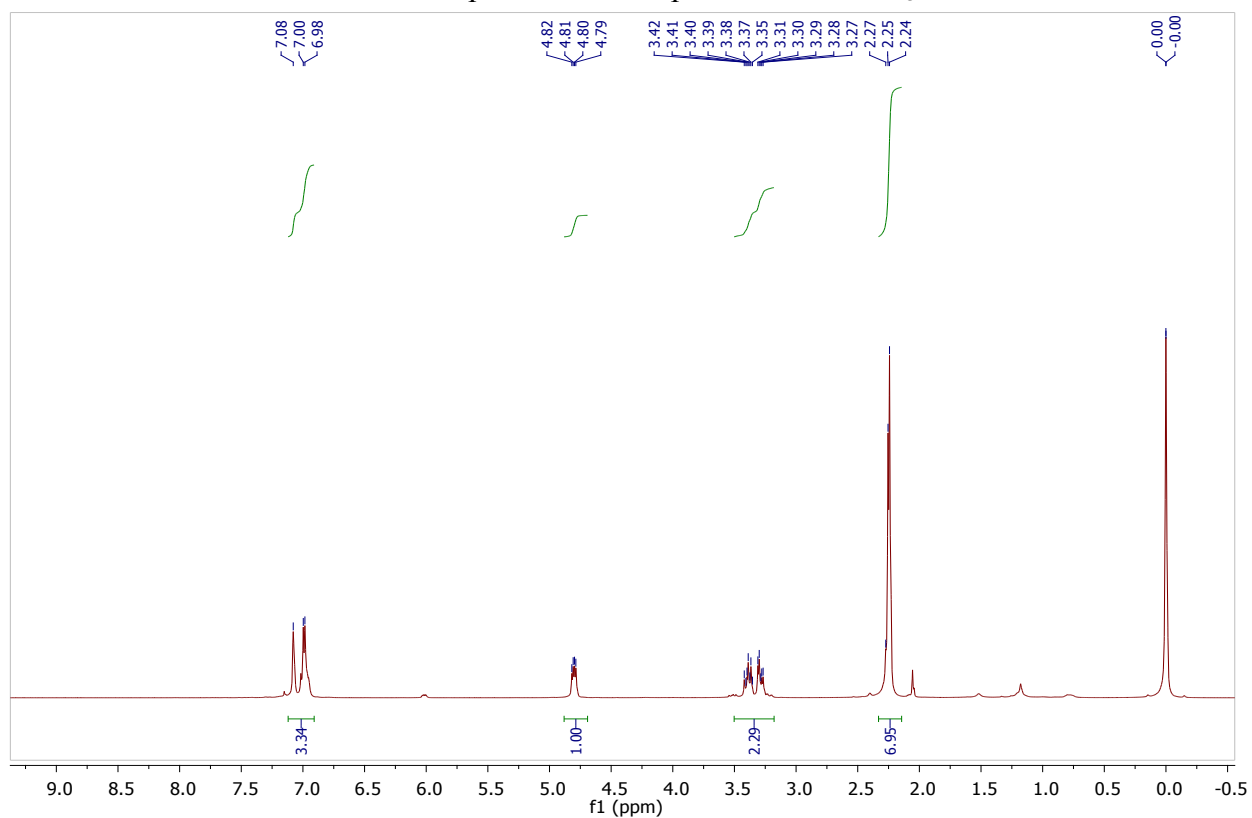
¹H NMR Spectrum of compound **14** in CDCl₃



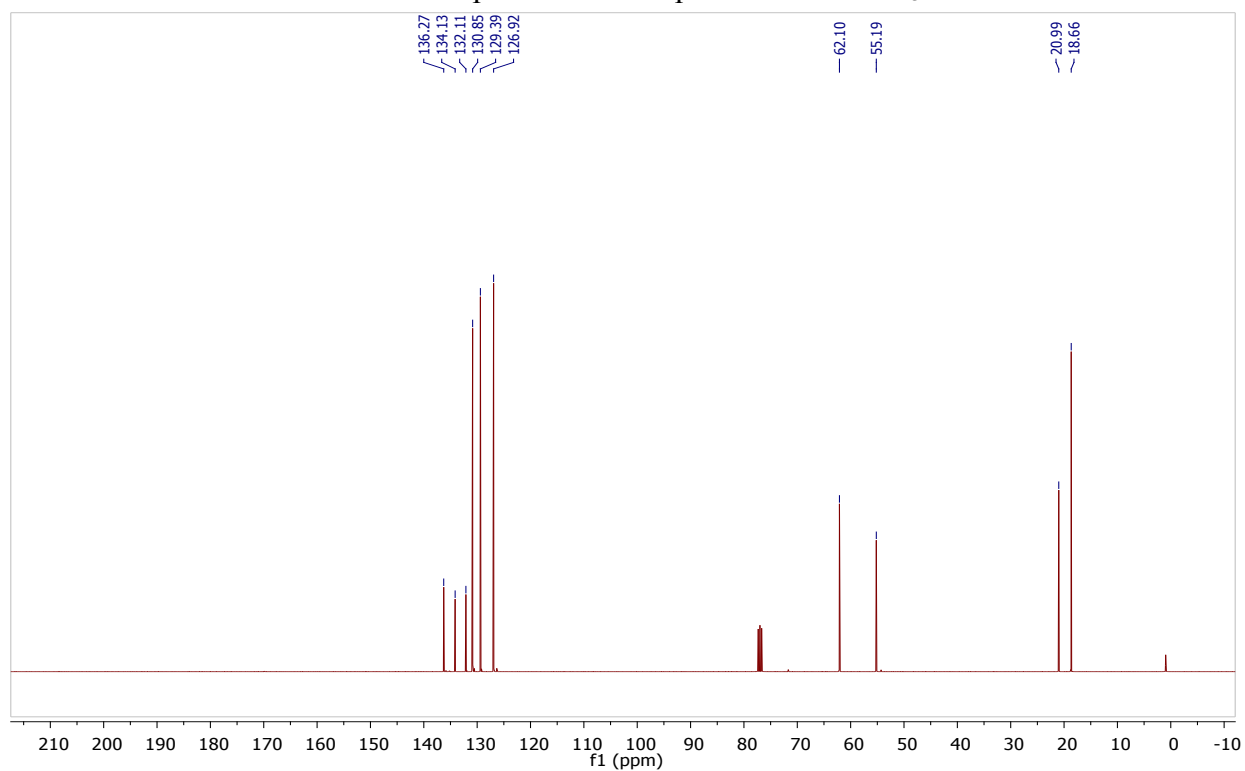
¹³C NMR Spectrum of compound **14** in CDCl₃



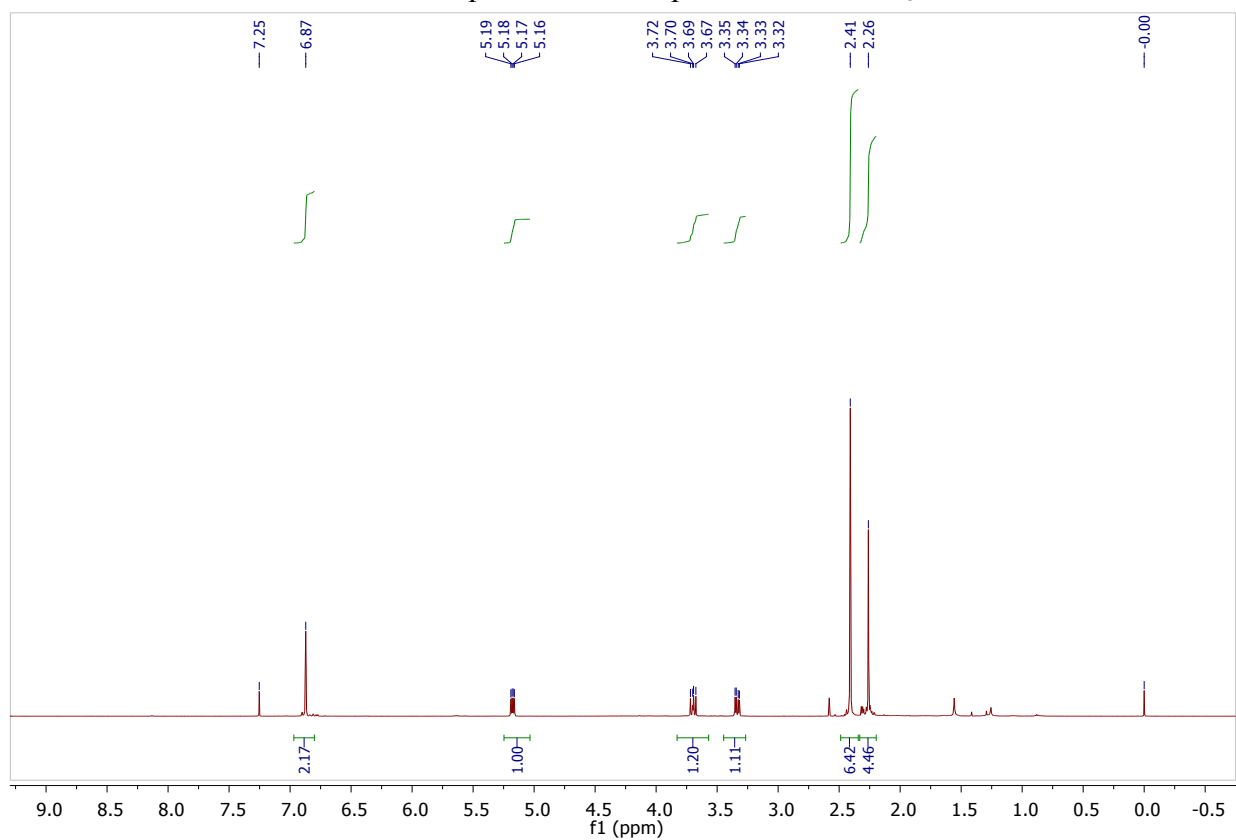
¹H NMR Spectrum of compound **15** in CDCl₃



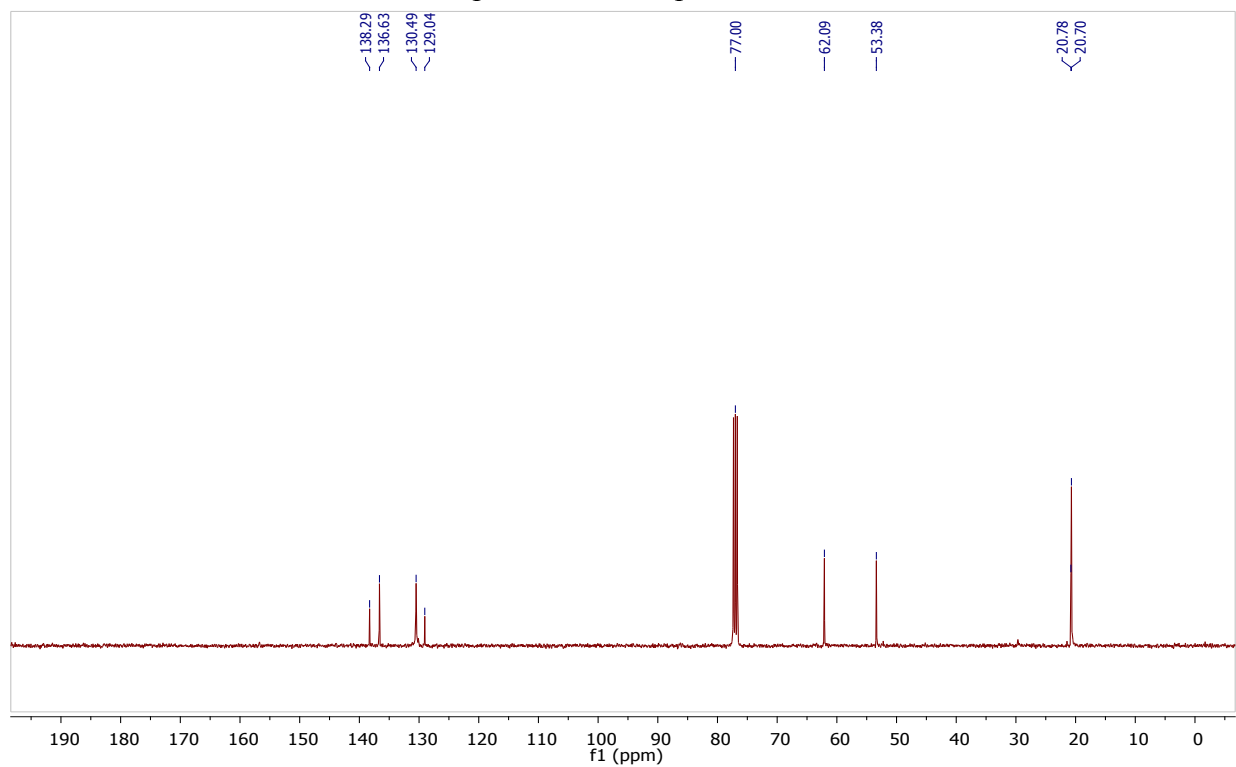
¹³C NMR Spectrum of compound **15** in CDCl₃



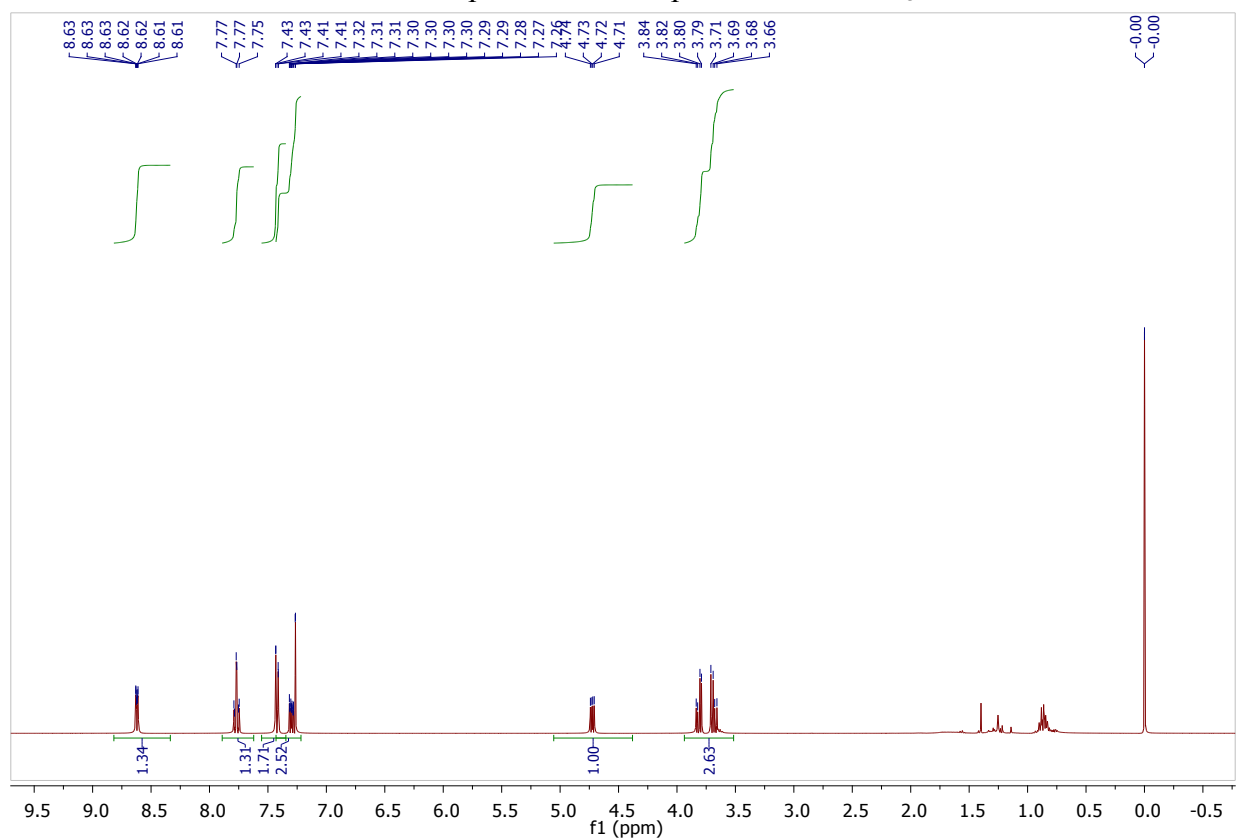
¹H NMR Spectrum of compound **16** in CDCl₃



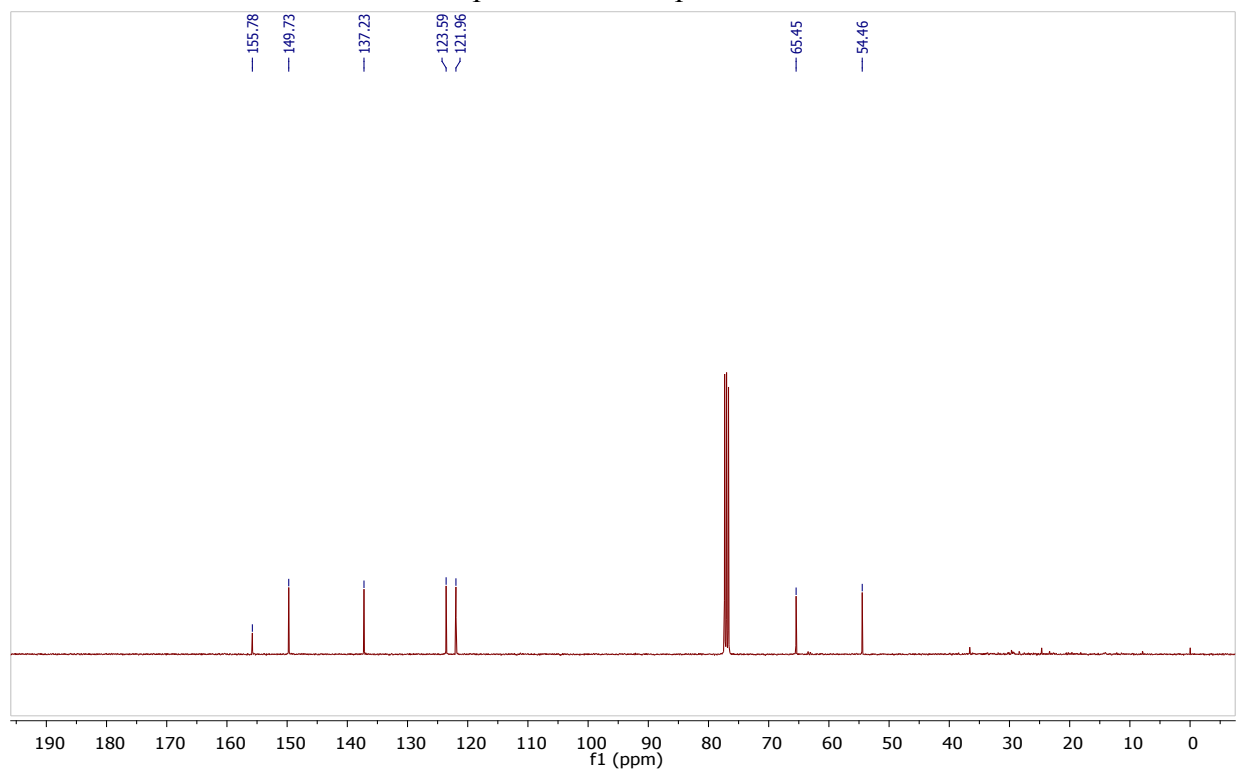
¹³C NMR Spectrum of compound **16** in CDCl₃



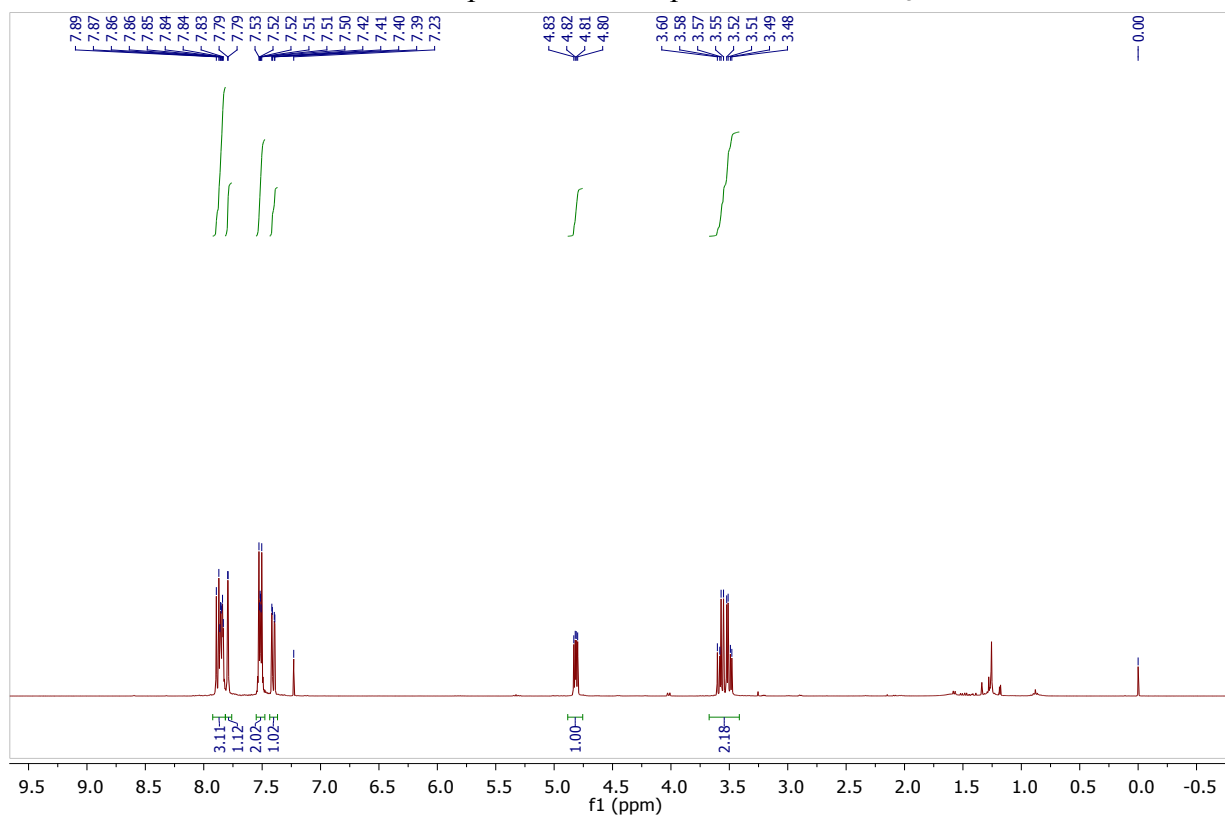
^1H NMR Spectrum of compound **17** in CDCl_3



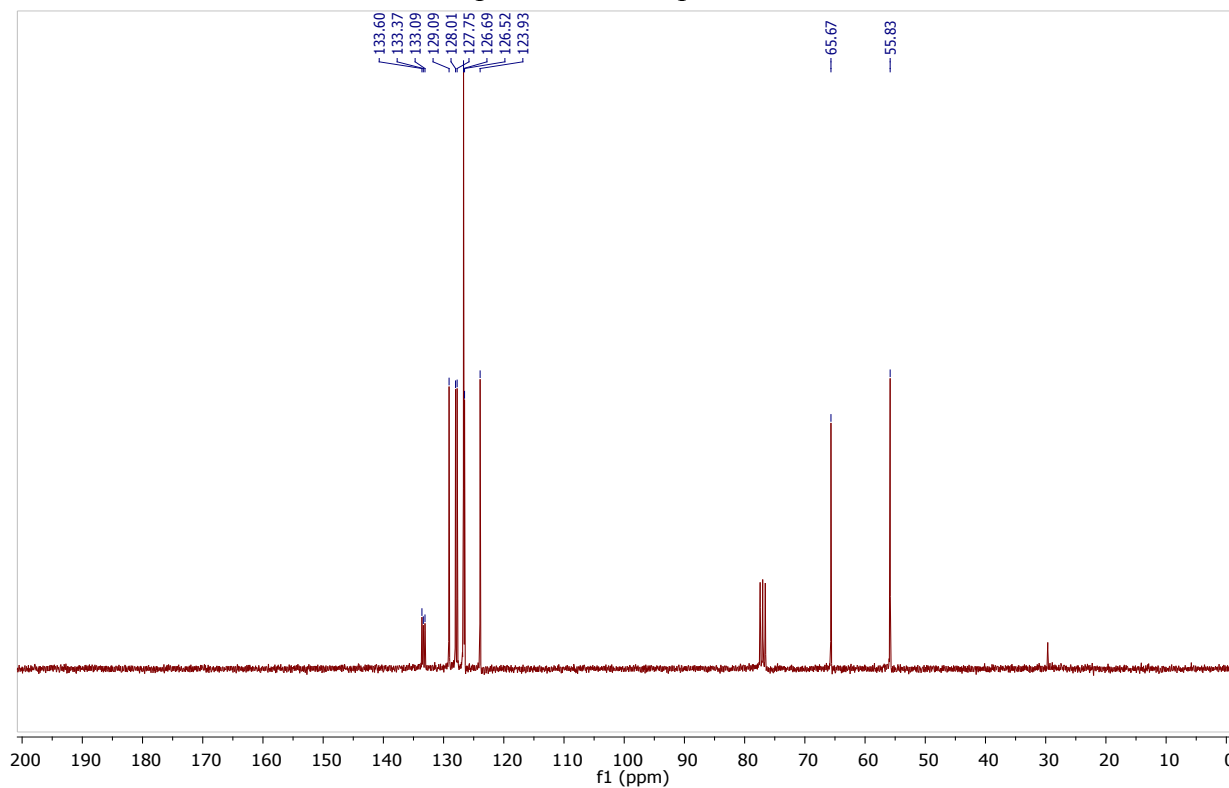
^{13}C NMR Spectrum of compound **17** in CDCl_3



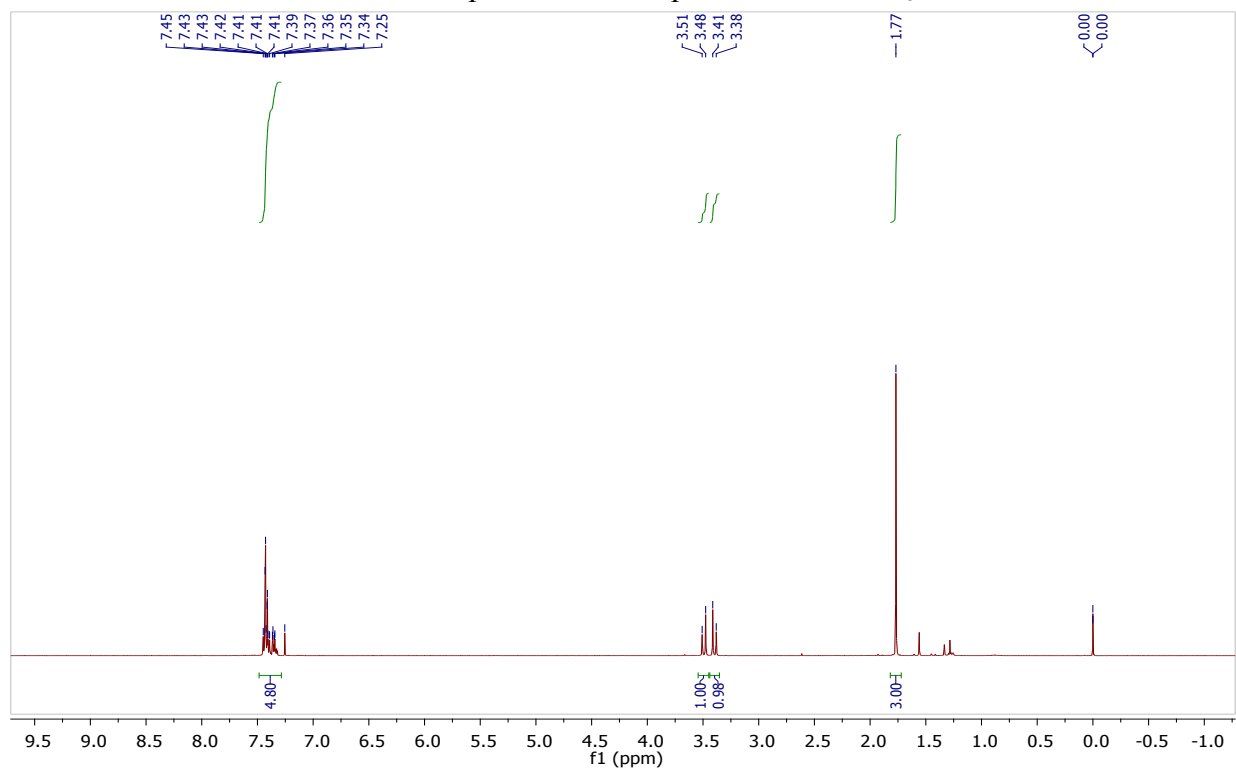
¹H NMR Spectrum of compound **18** in CDCl₃



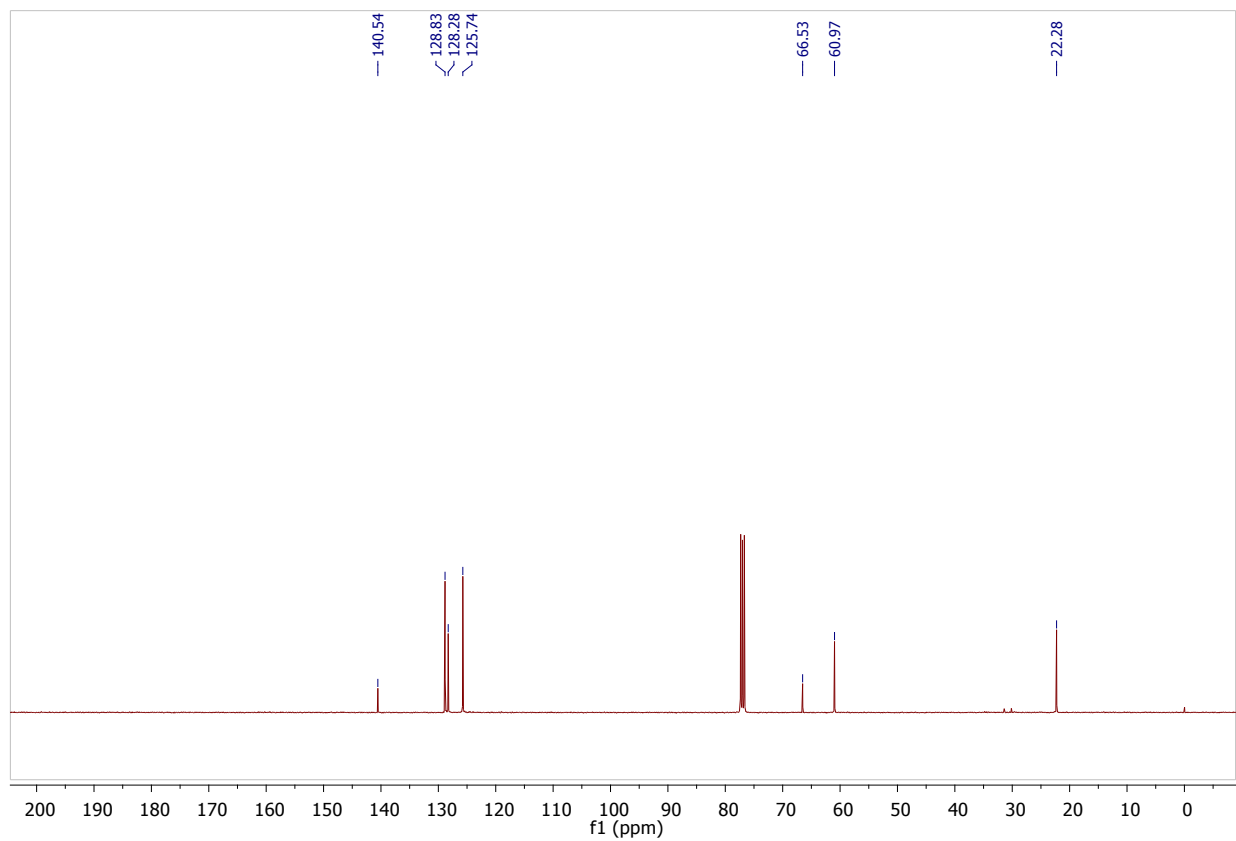
¹³C NMR Spectrum of compound **18** in CDCl₃



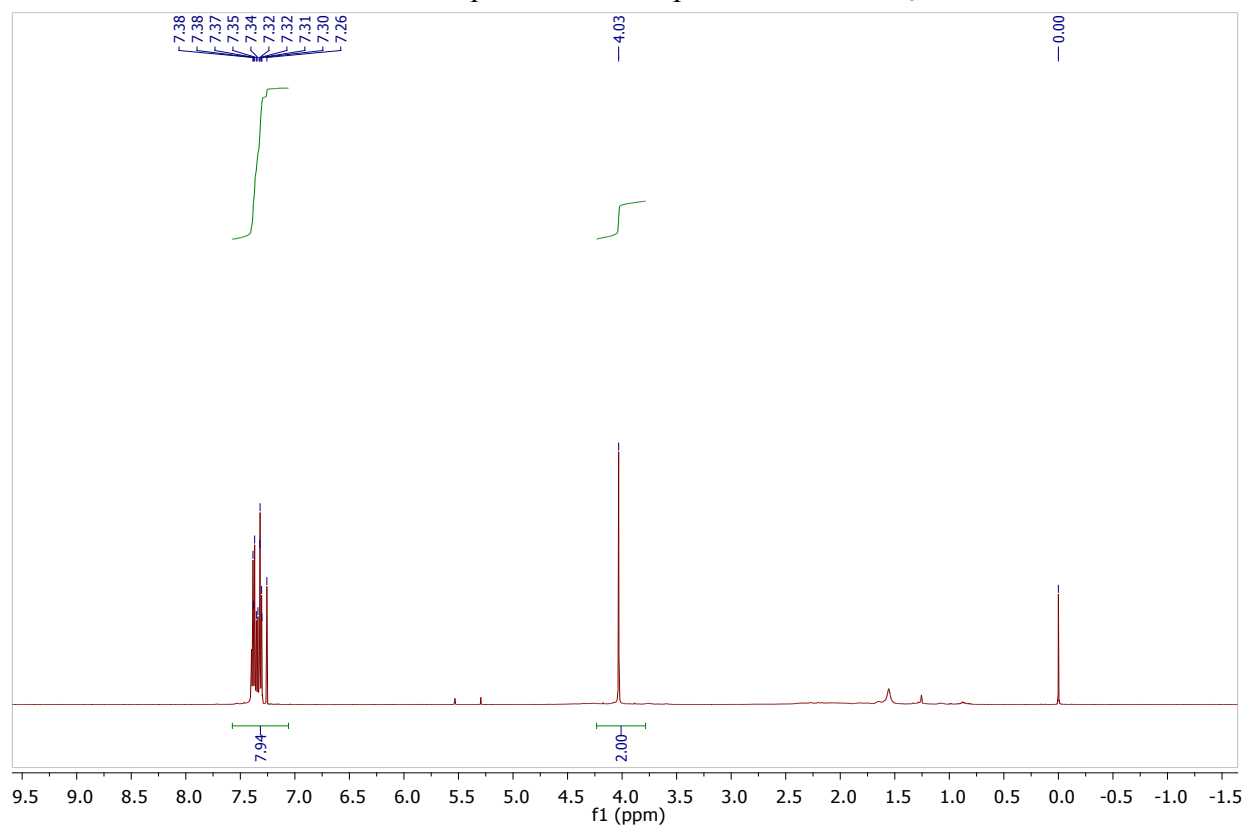
¹H NMR Spectrum of compound **19** in CDCl₃



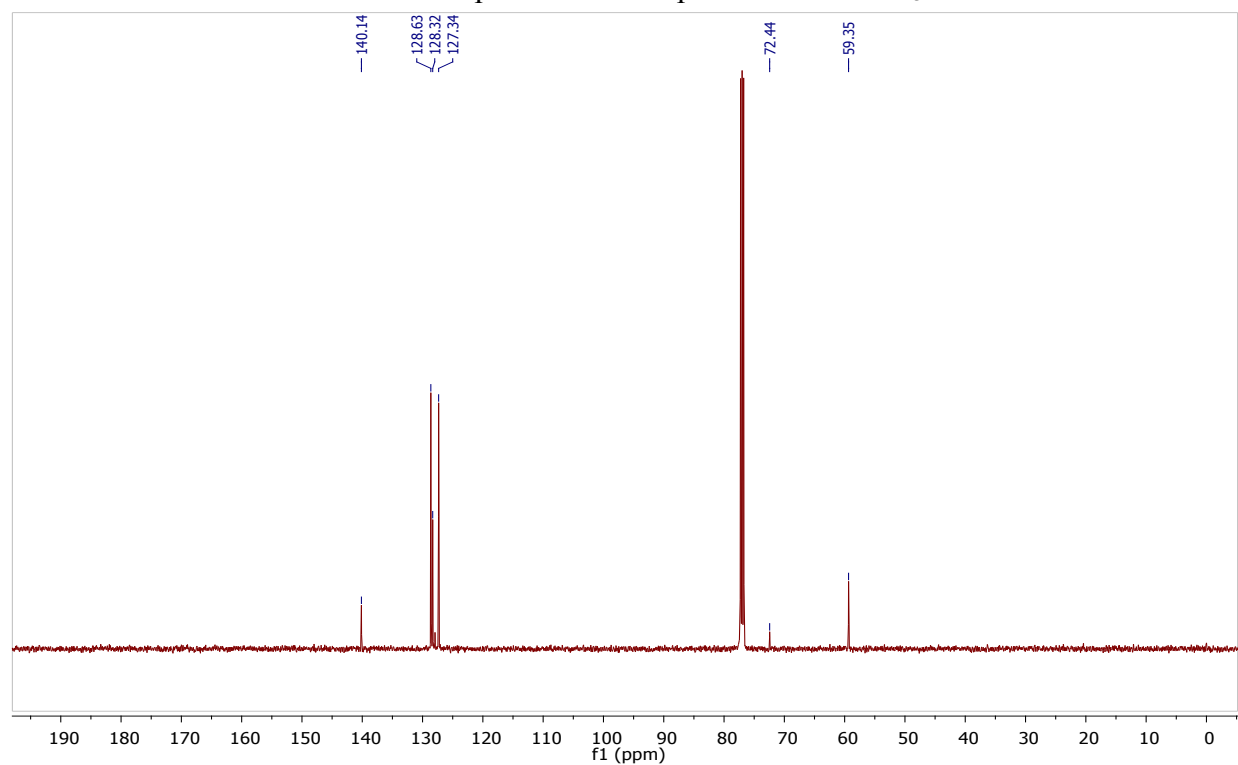
¹³C NMR Spectrum of compound **19** in CDCl₃



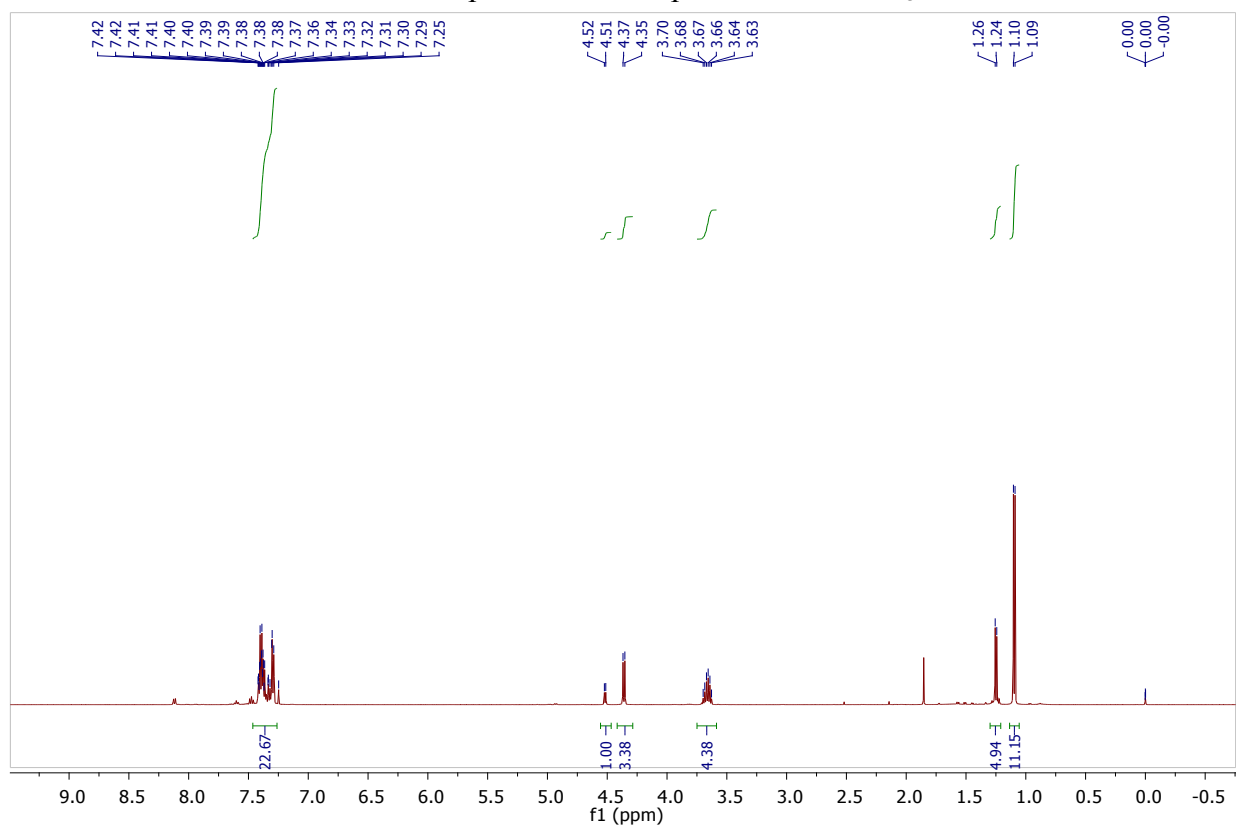
^1H NMR Spectrum of compound **20** in CDCl_3



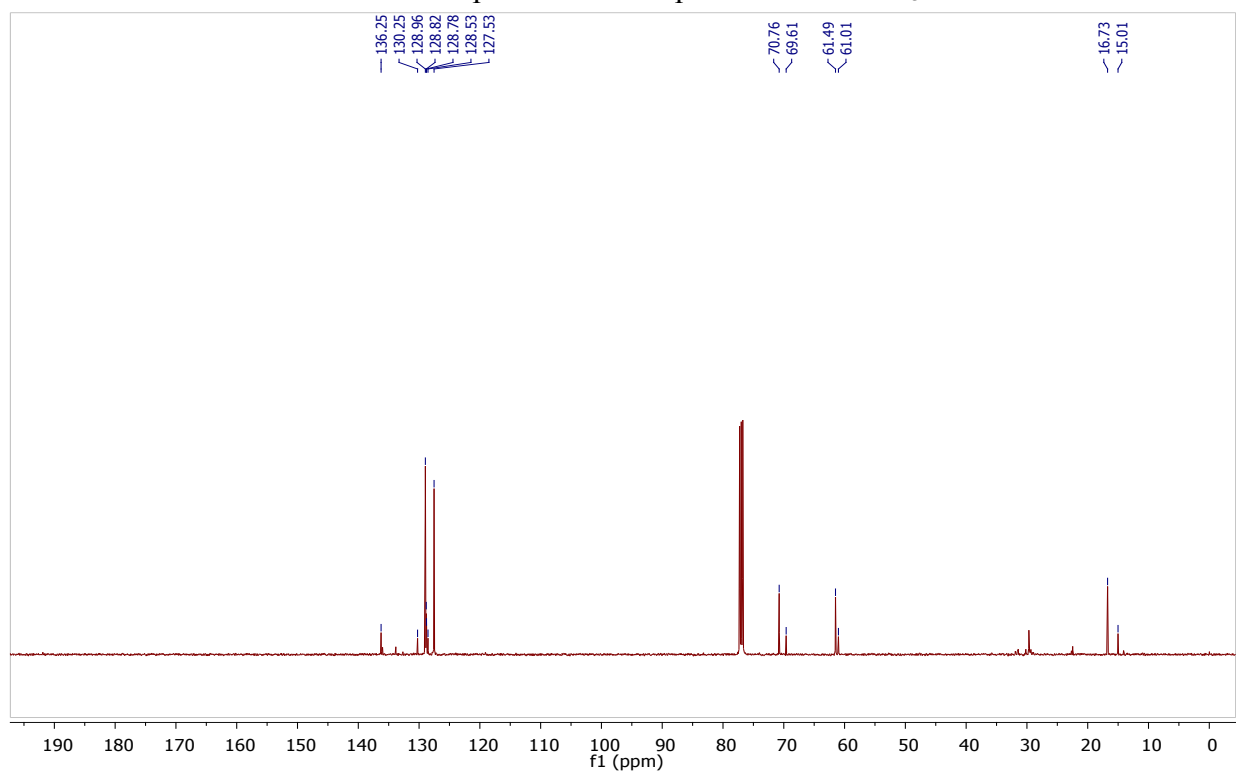
^{13}C NMR Spectrum of compound **20** in CDCl_3



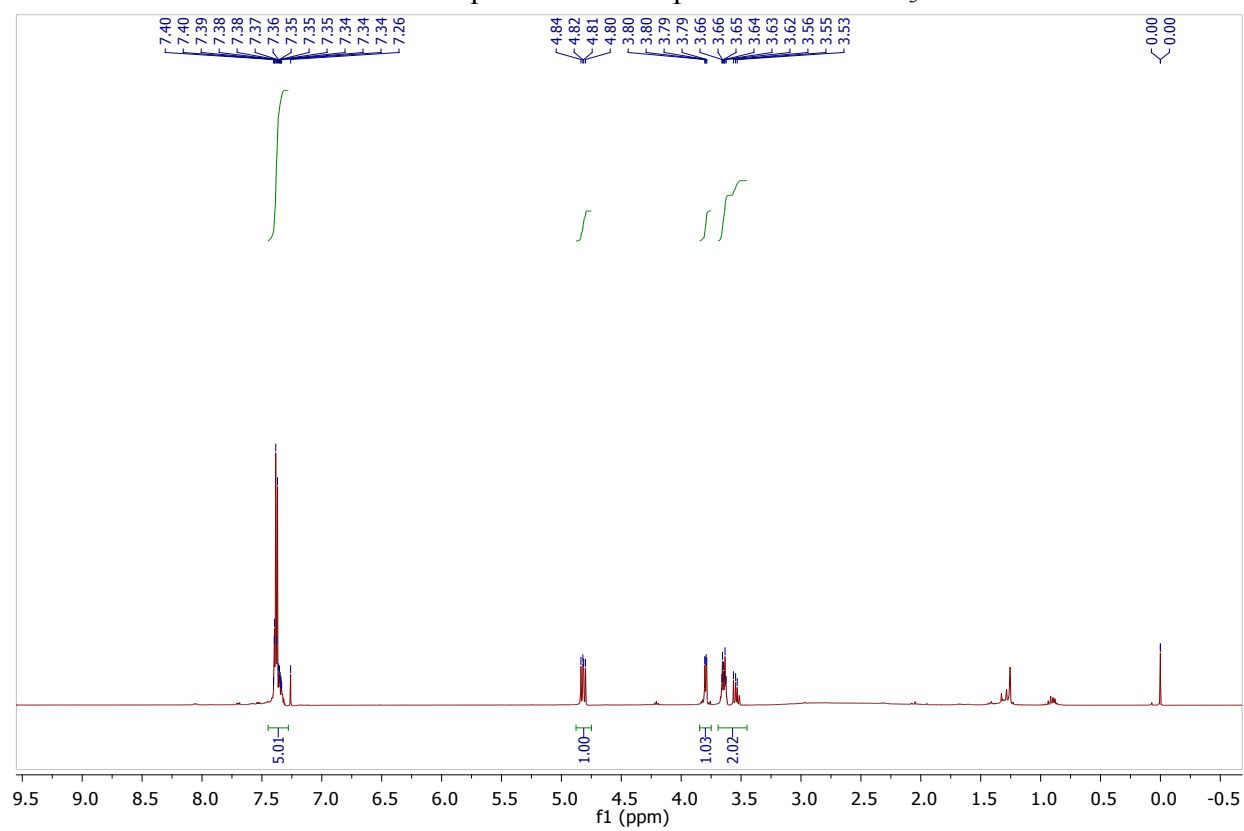
¹H NMR Spectrum of compound **21** in CDCl₃



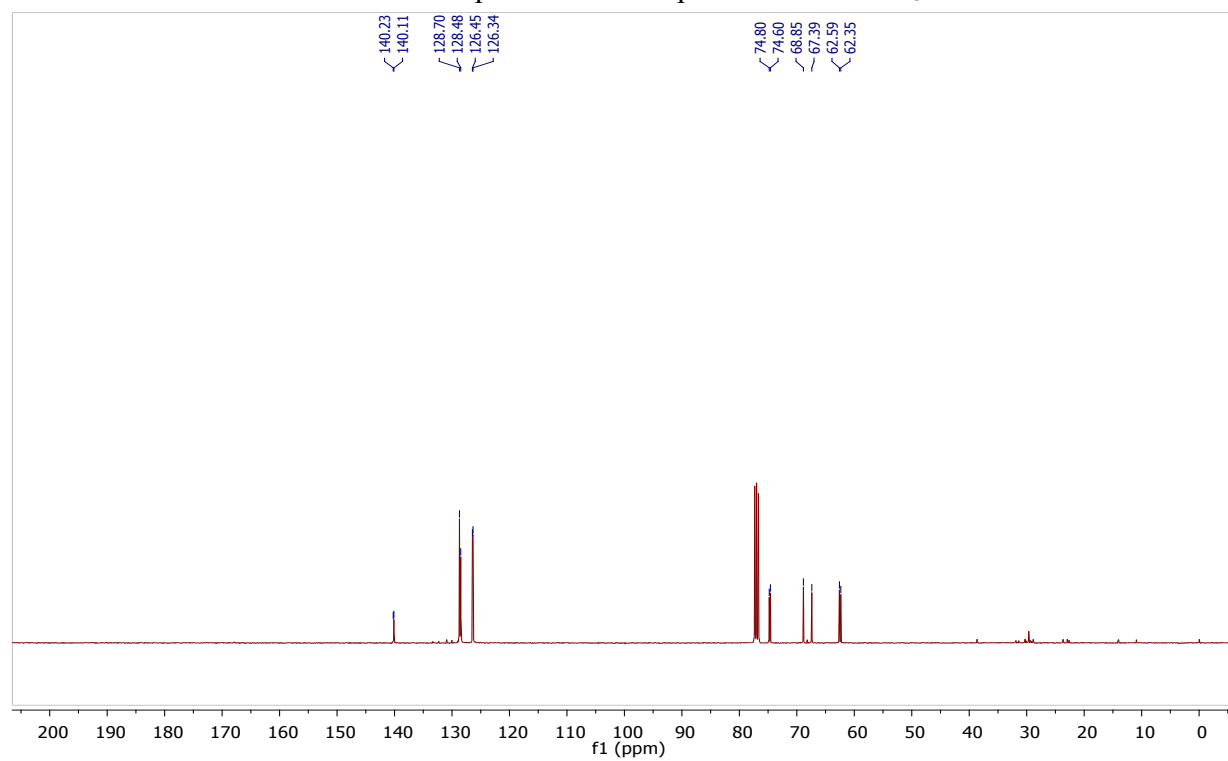
¹³C NMR Spectrum of compound **21** in CDCl₃



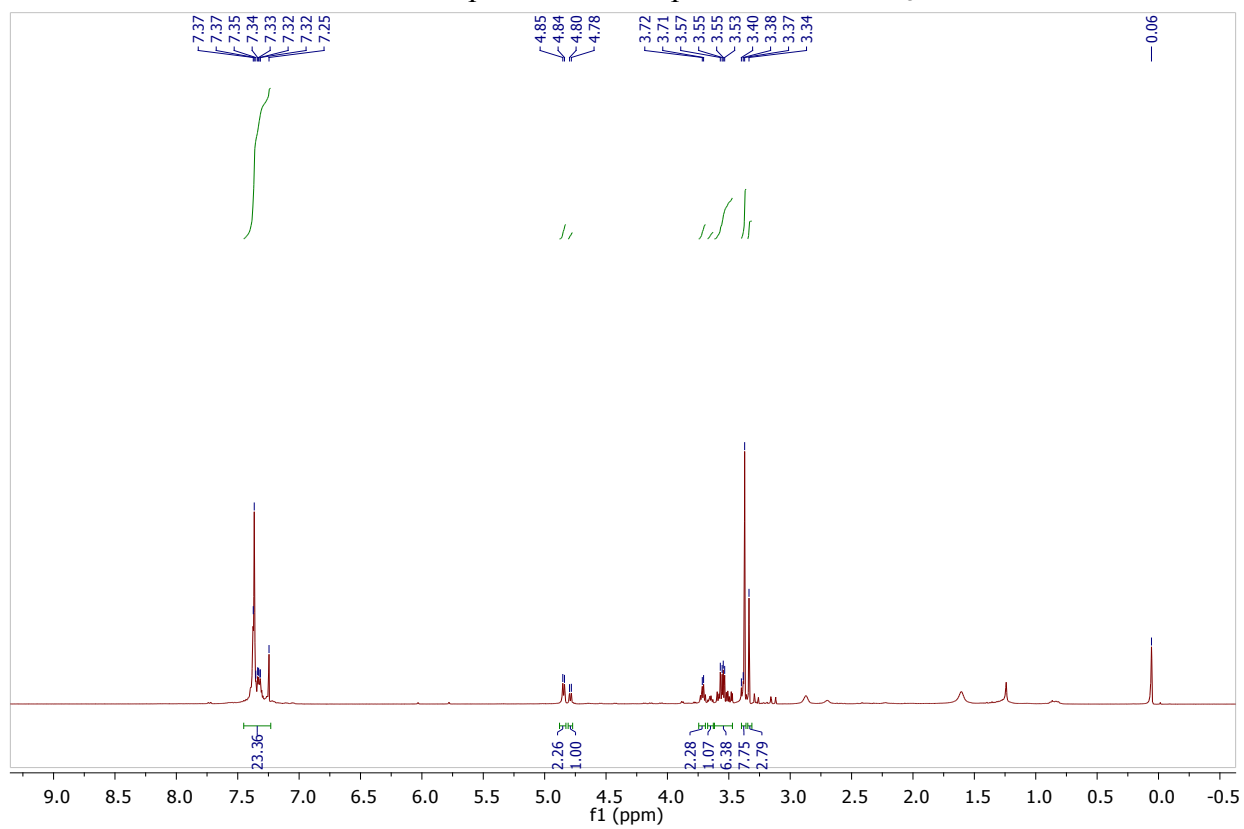
^1H NMR Spectrum of compound **22** in CDCl_3



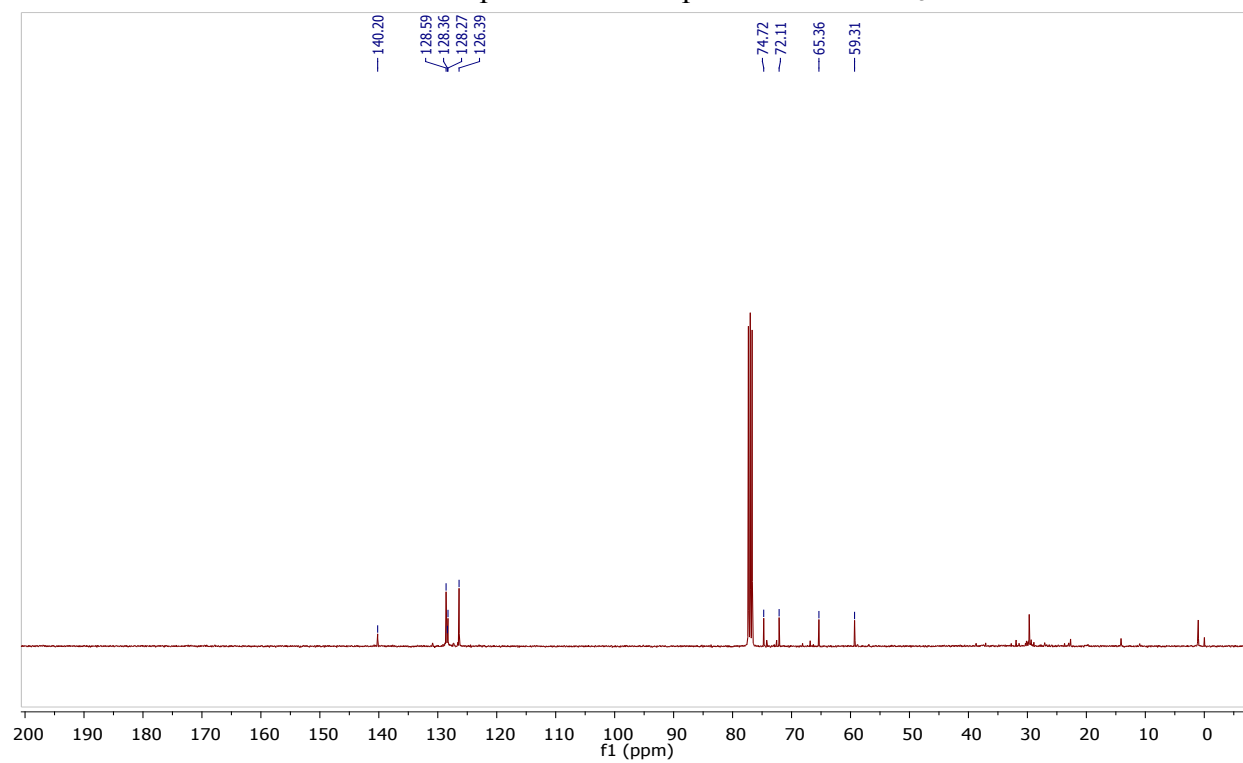
^{13}C NMR Spectrum of compound **22** in CDCl_3



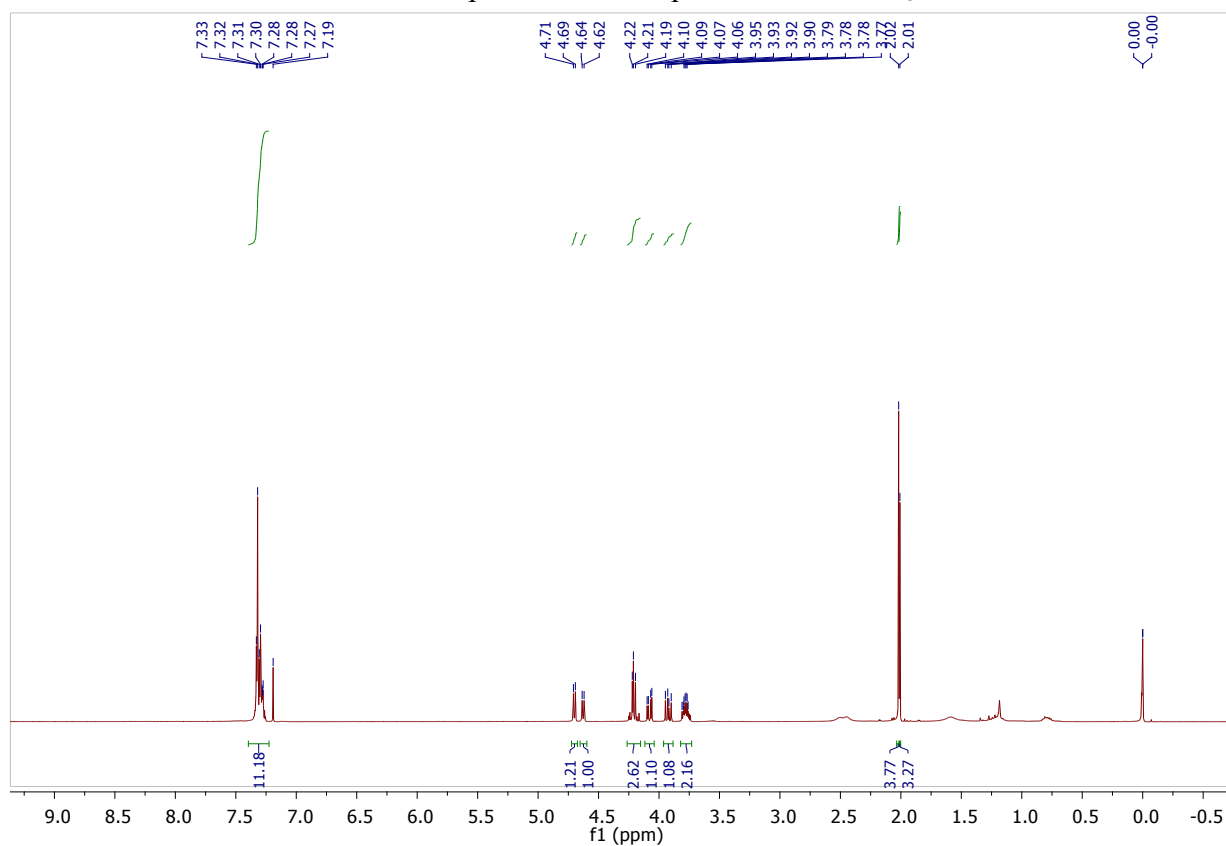
^1H NMR Spectrum of compound **23** in CDCl_3



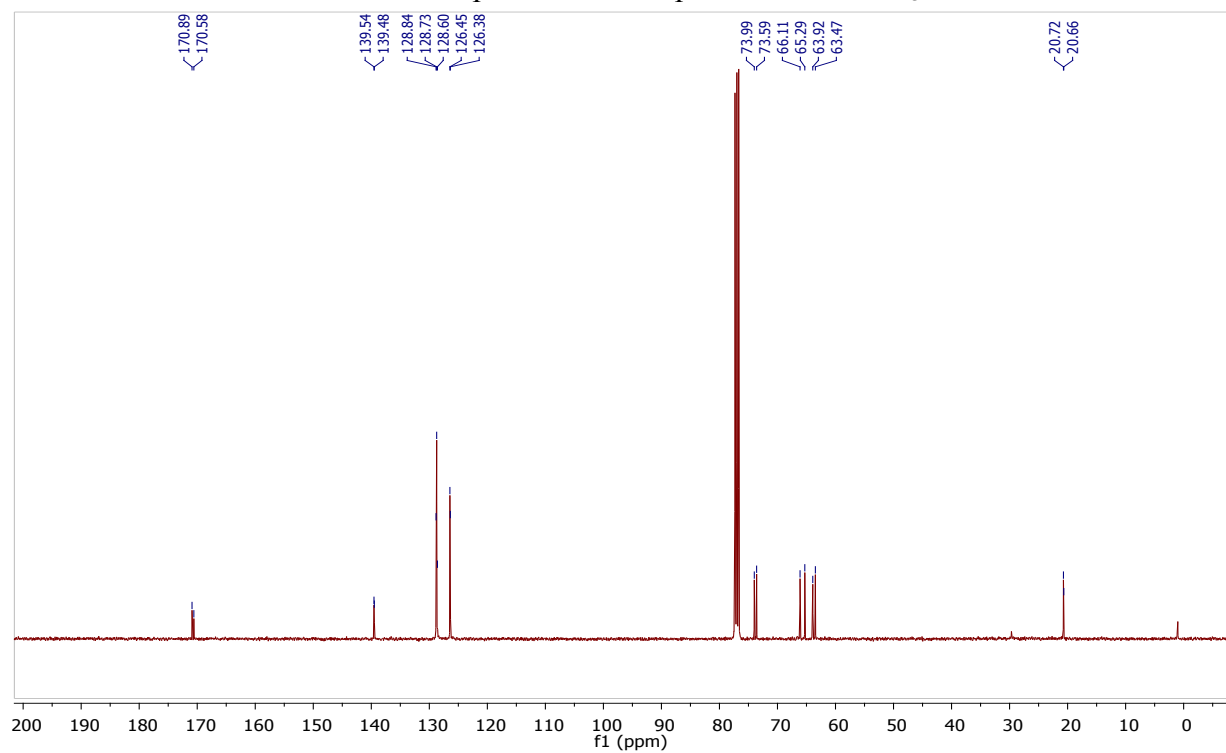
^{13}C NMR Spectrum of compound **23** in CDCl_3



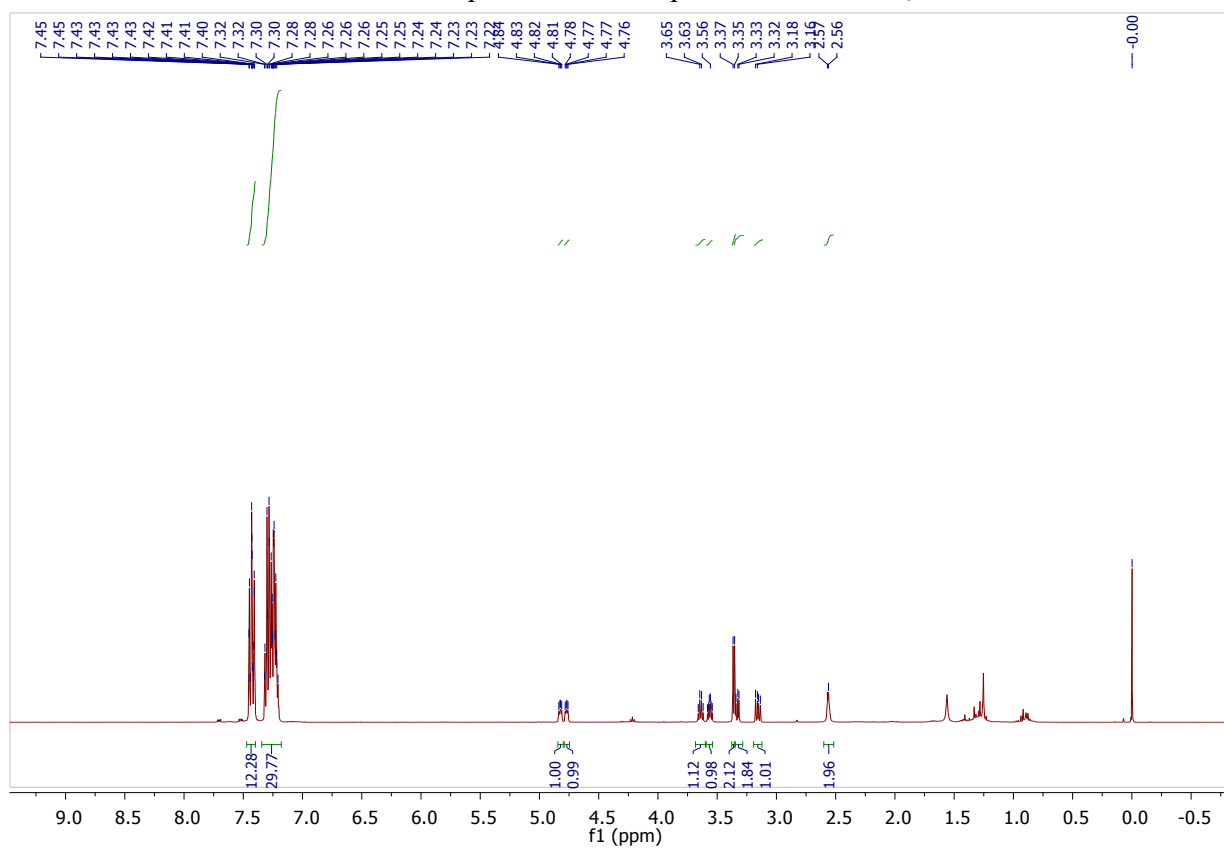
¹H NMR Spectrum of compound **24** in CDCl₃



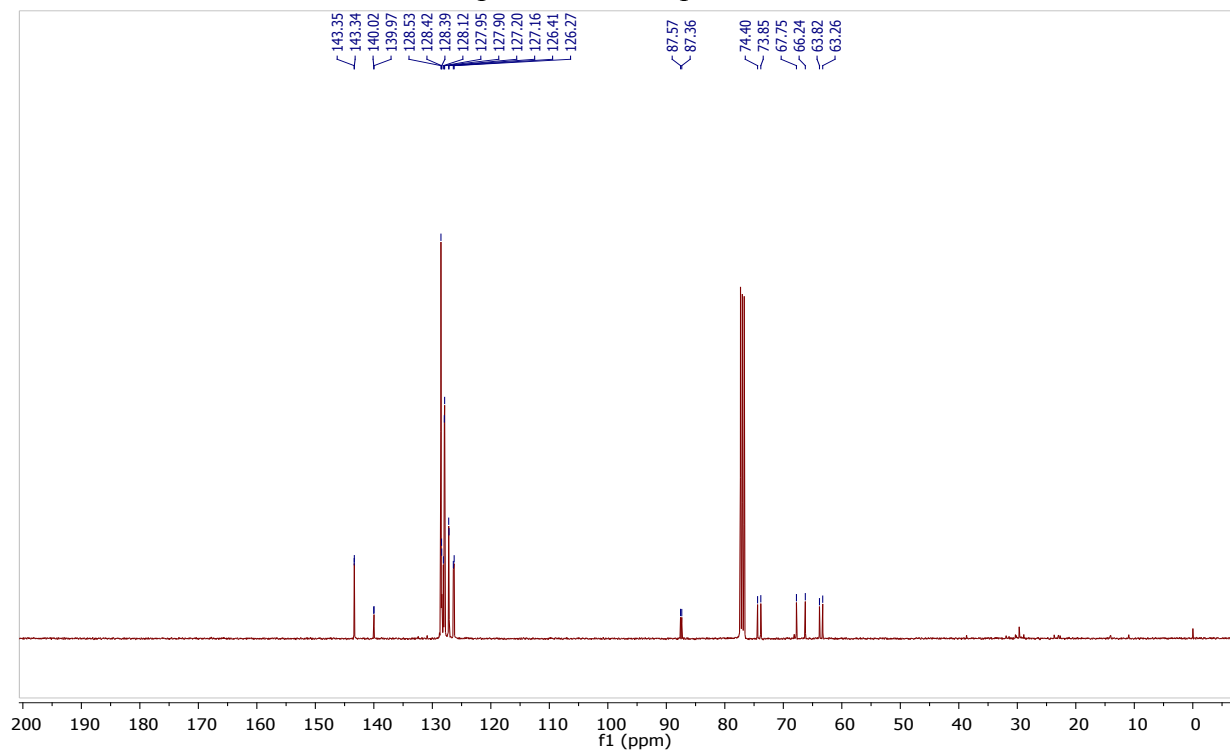
¹³C NMR Spectrum of compound **24** in CDCl₃



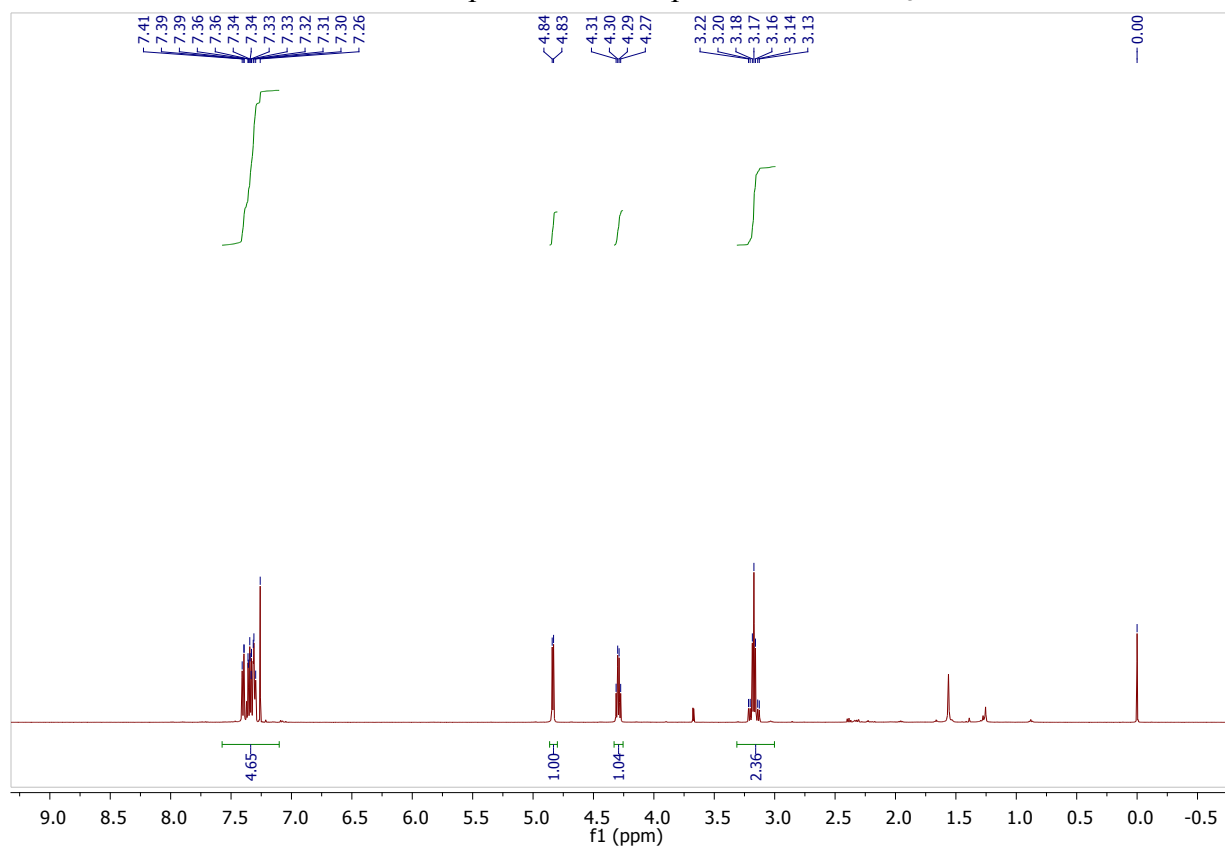
¹H NMR Spectrum of compound **25** in CDCl₃



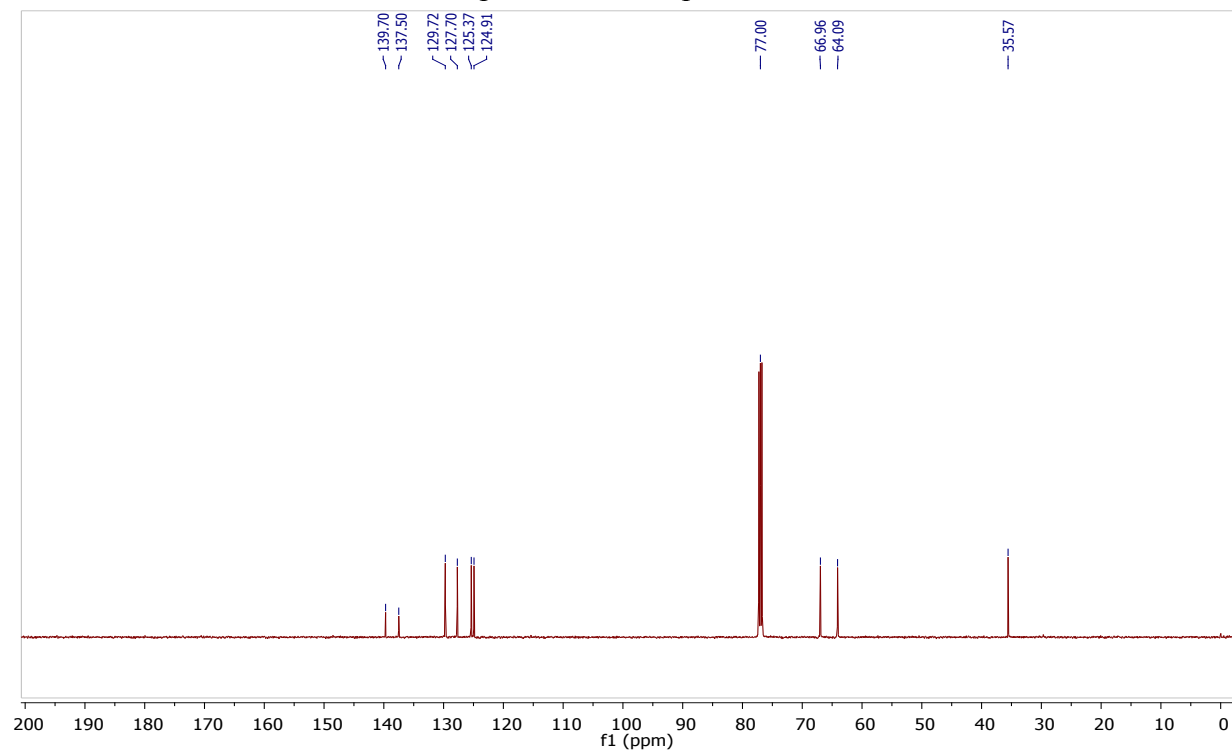
¹³C NMR Spectrum of compound **25** in CDCl₃



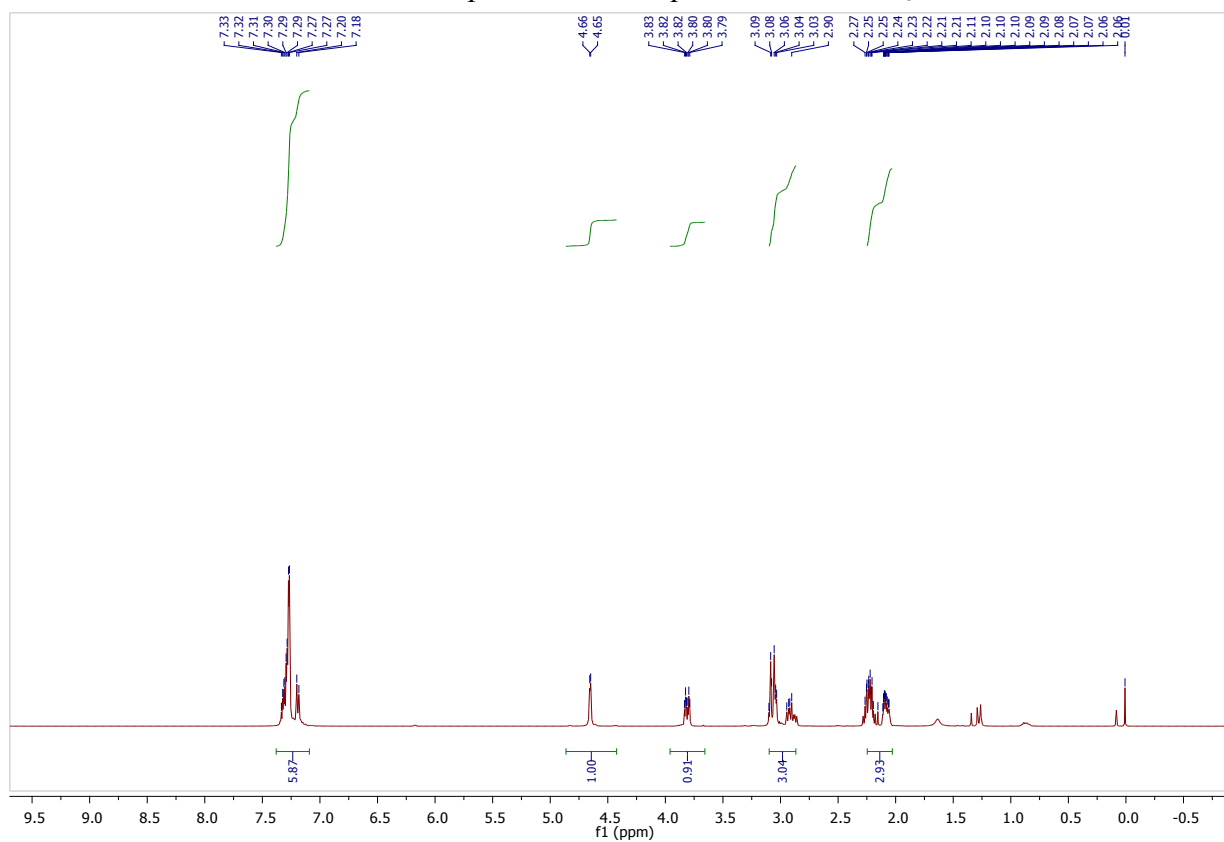
^1H NMR Spectrum of compound **26** in CDCl_3



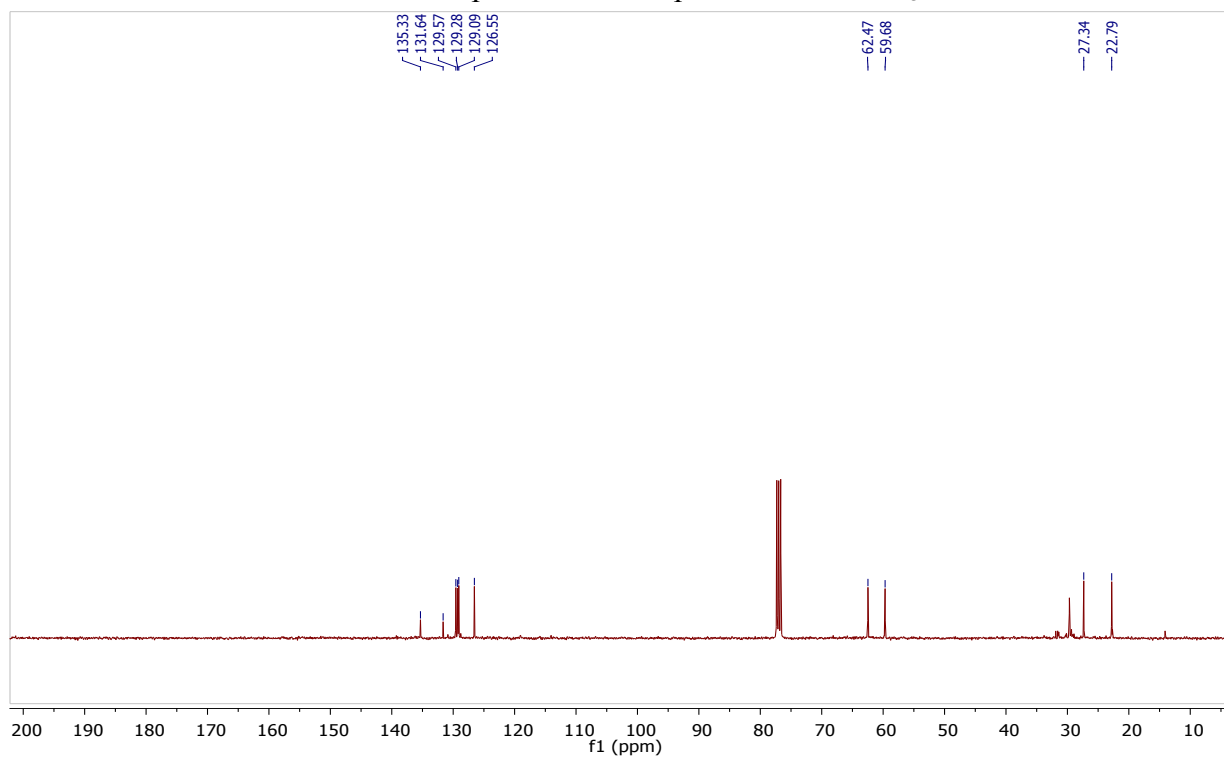
^{13}C NMR Spectrum of compound **26** in CDCl_3



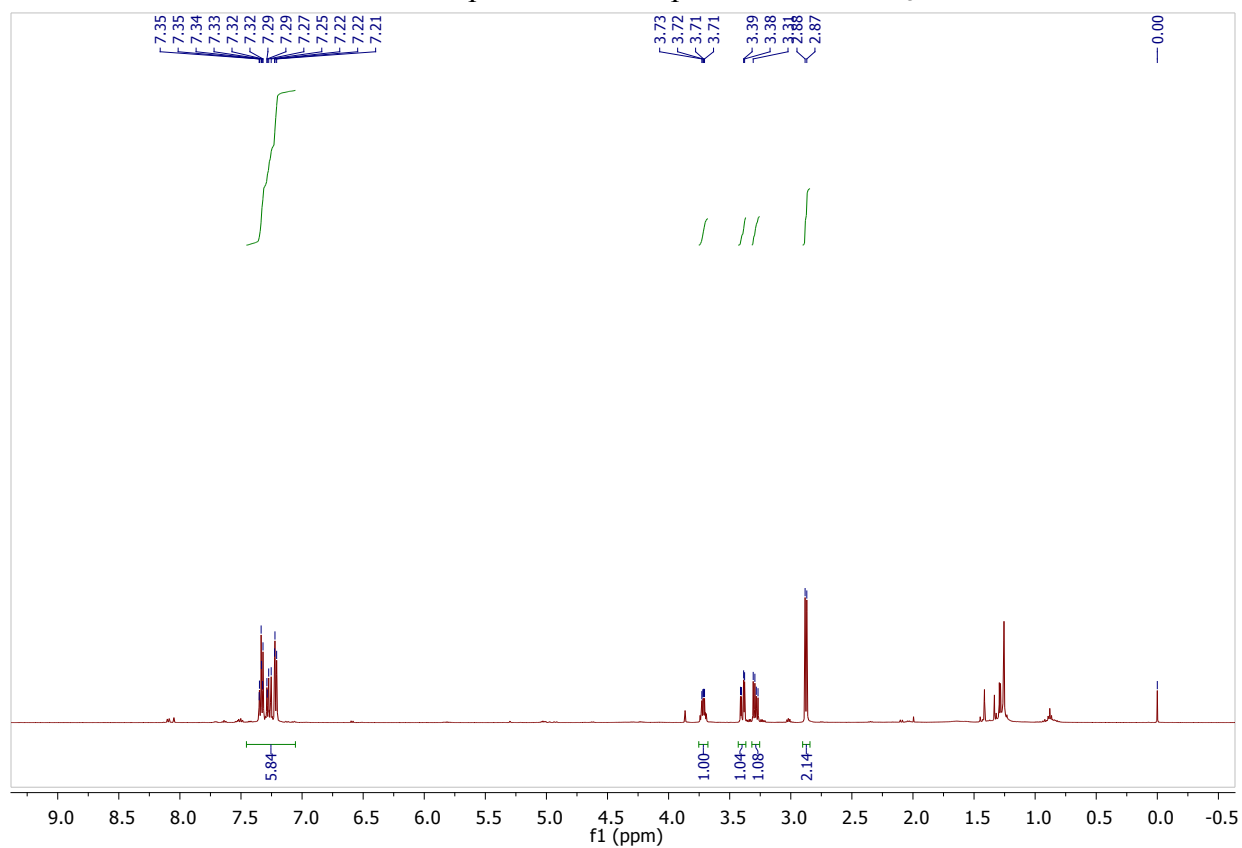
¹H NMR Spectrum of compound **27** in CDCl₃



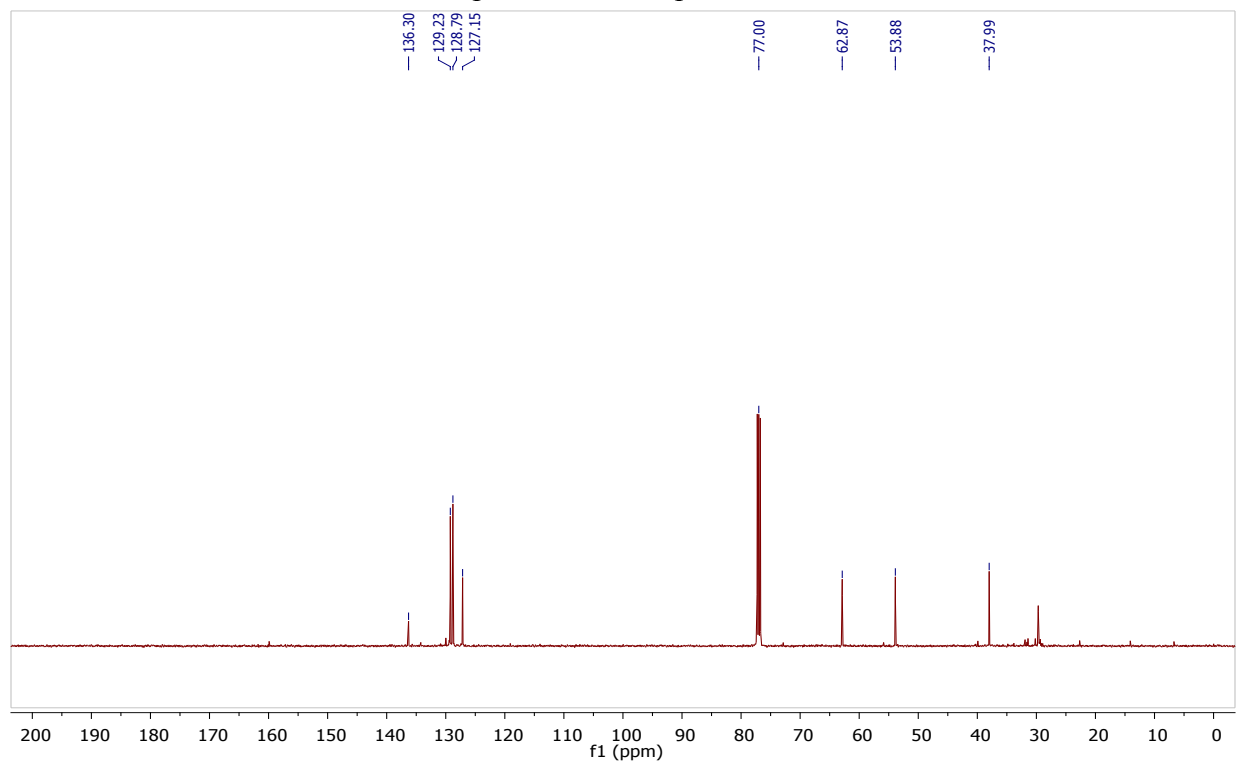
¹³C NMR Spectrum of compound **27** in CDCl₃



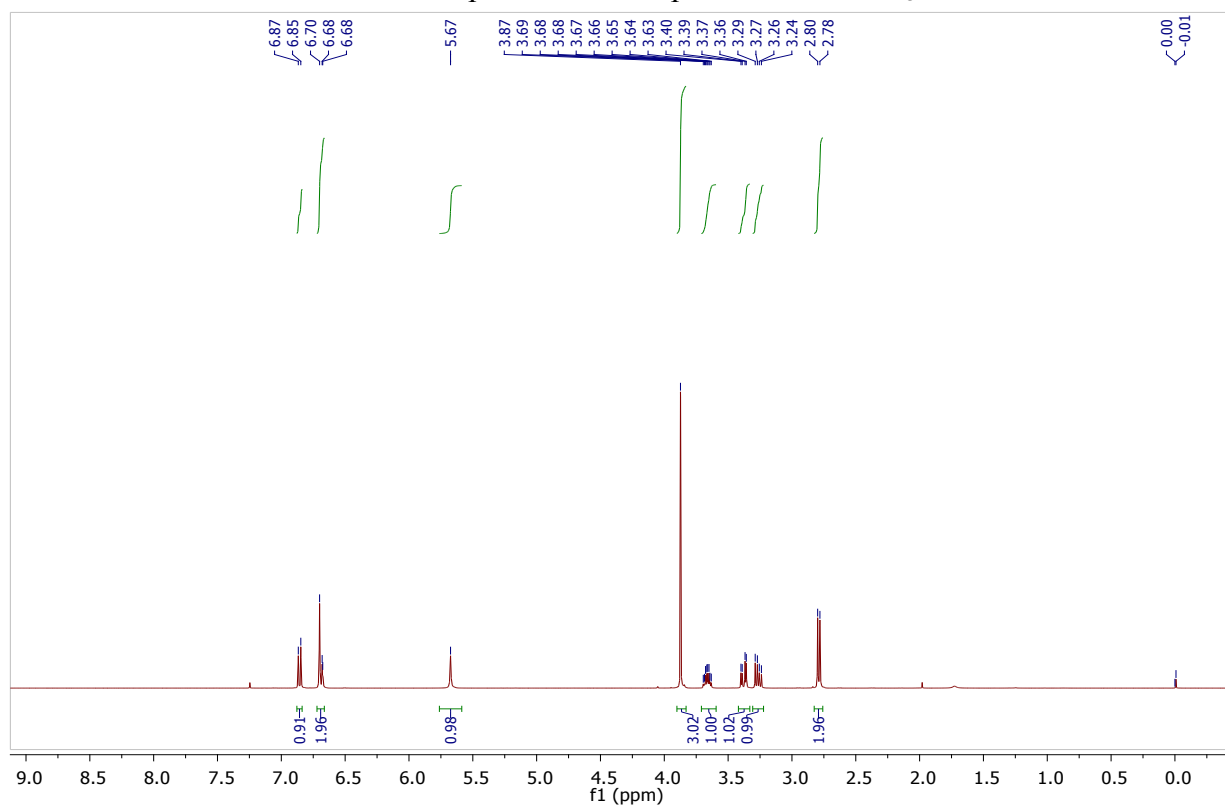
^1H NMR Spectrum of compound **28** in CDCl_3



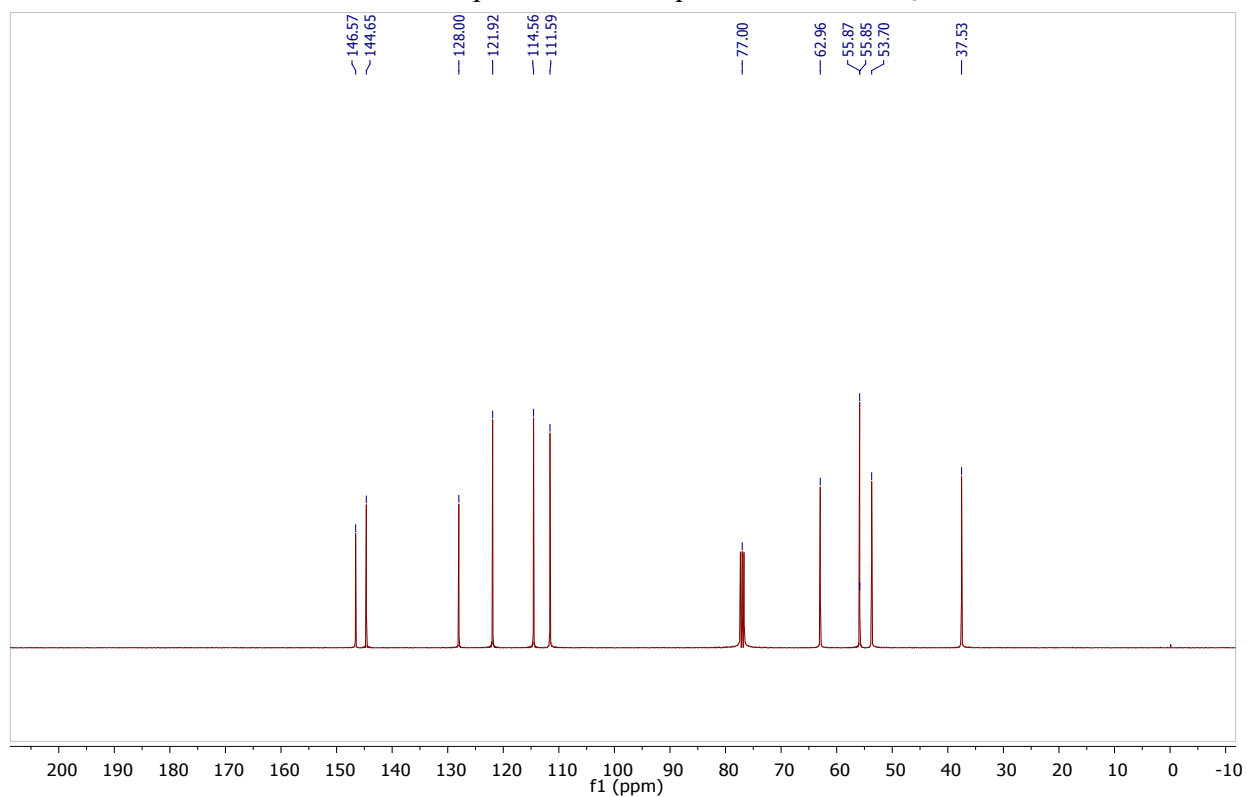
^{13}C NMR Spectrum of compound **28** in CDCl_3



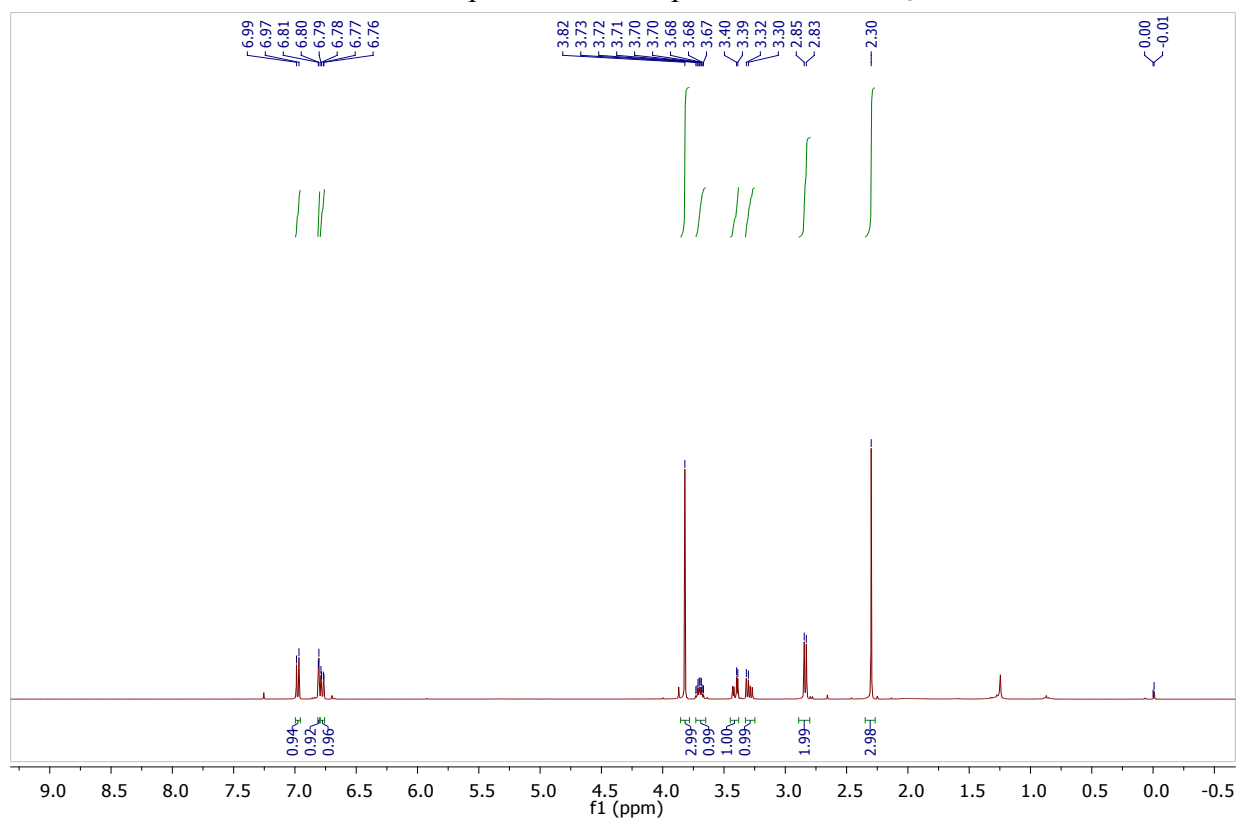
¹H NMR Spectrum of compound **29** in CDCl₃



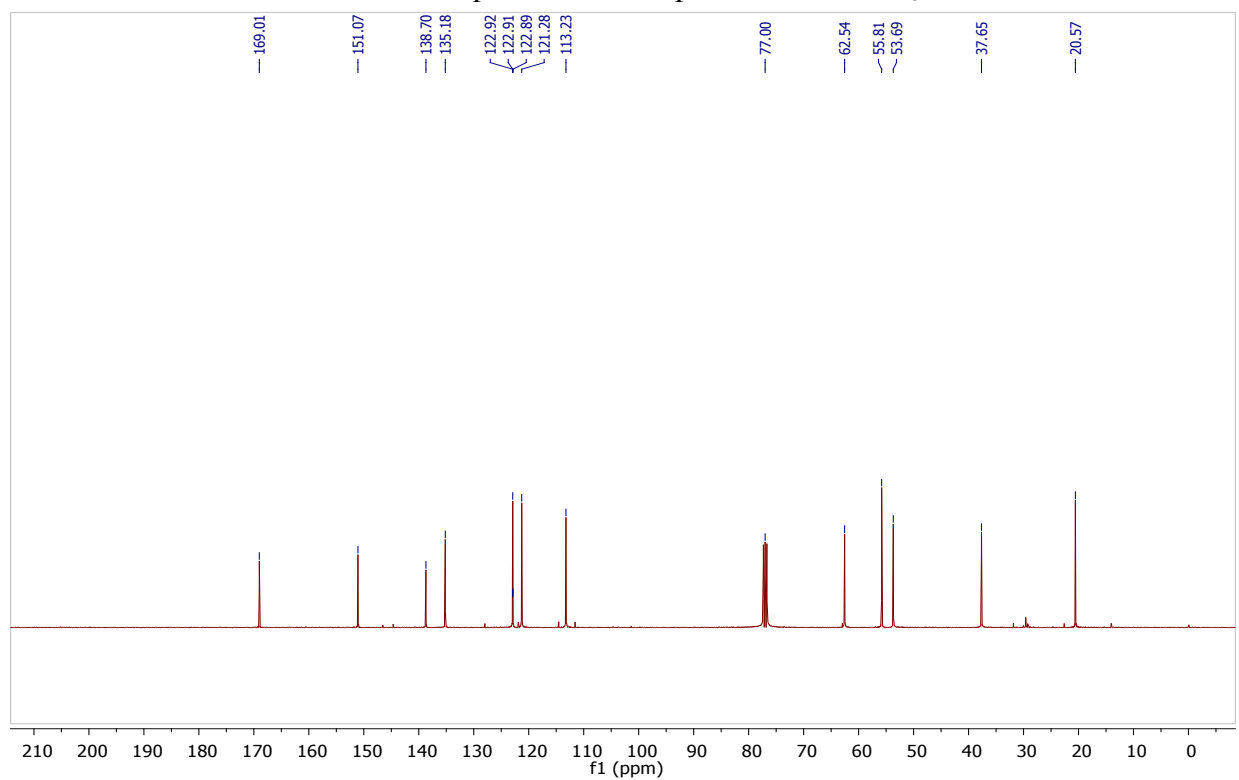
¹³C NMR Spectrum of compound **29** in CDCl₃



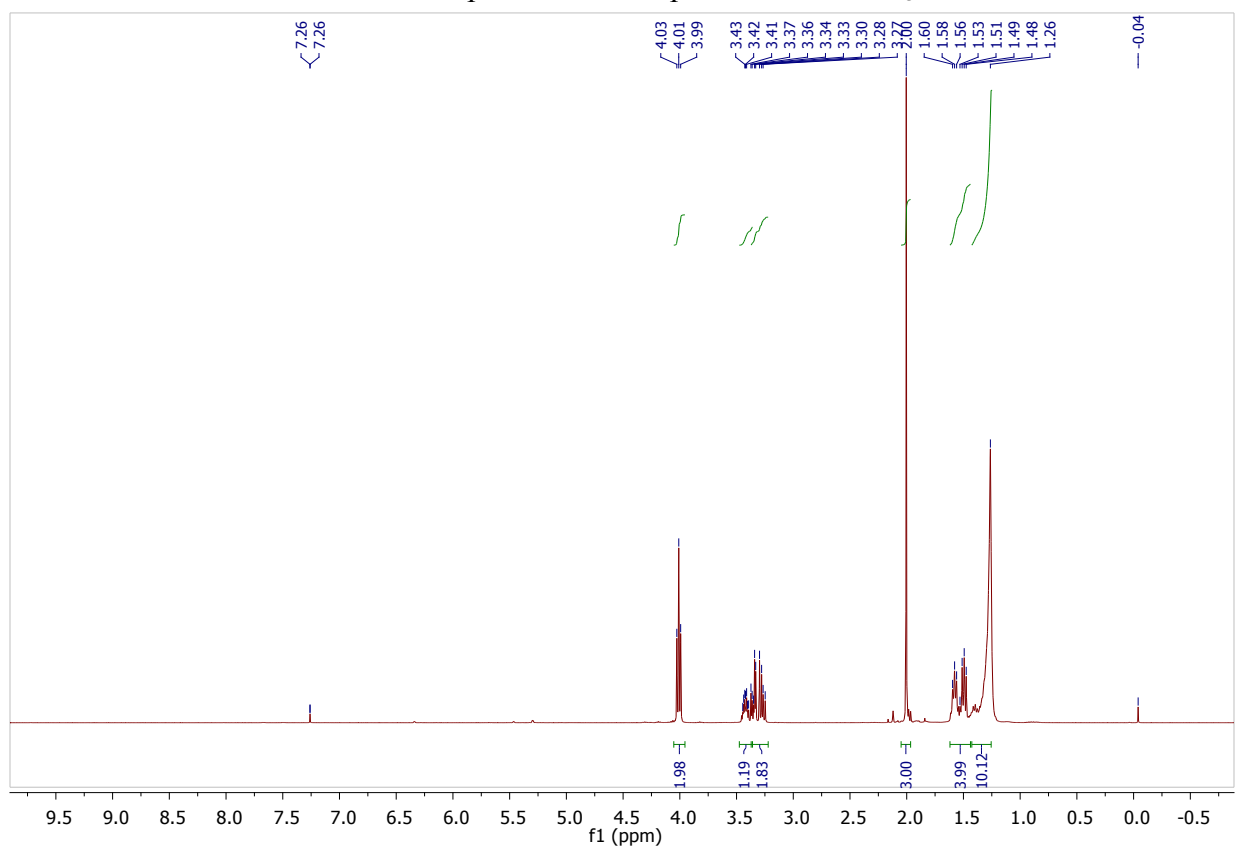
¹H NMR Spectrum of compound **30** in CDCl₃



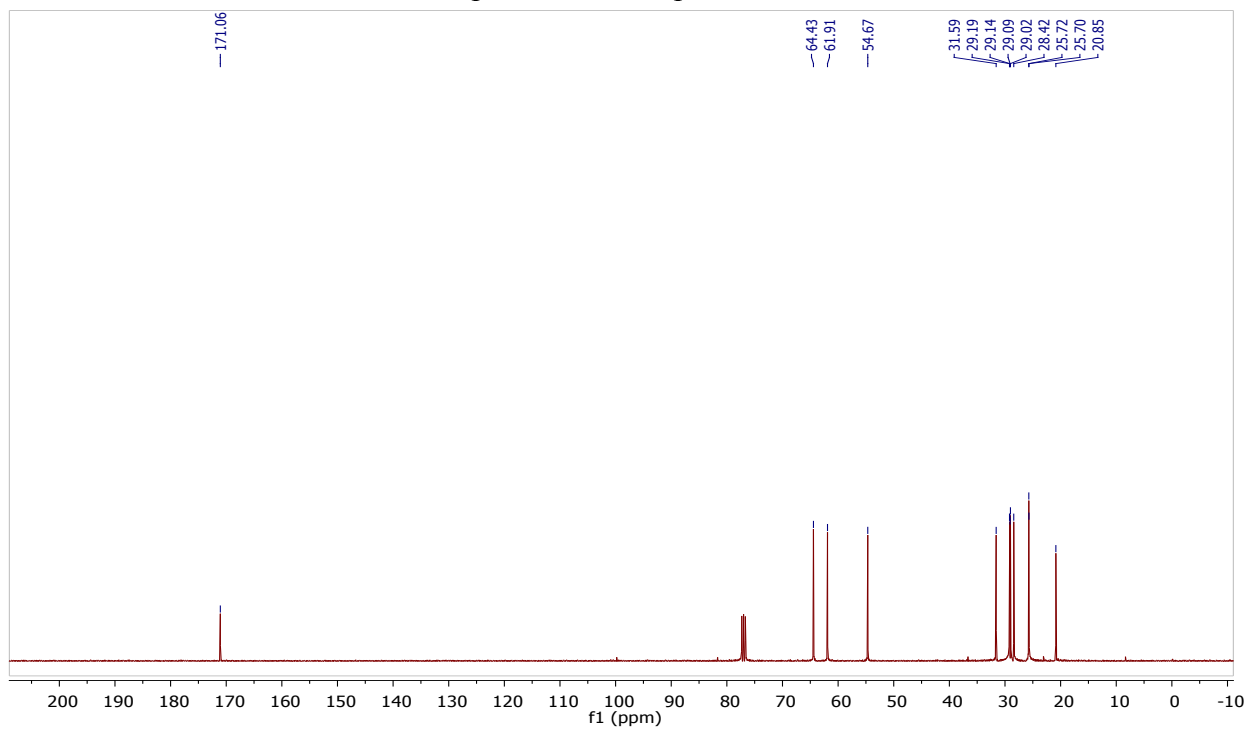
¹³C NMR Spectrum of compound **30** in CDCl₃



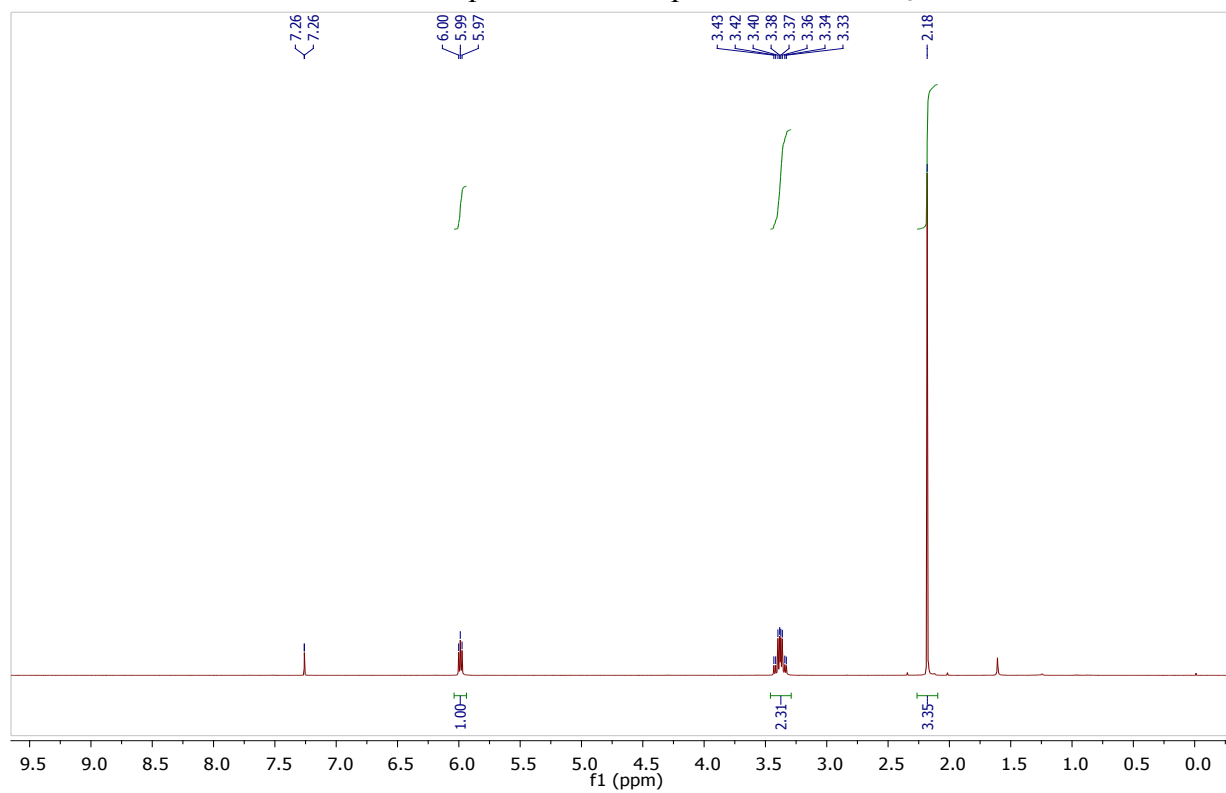
¹H NMR Spectrum of compound **31** in CDCl₃



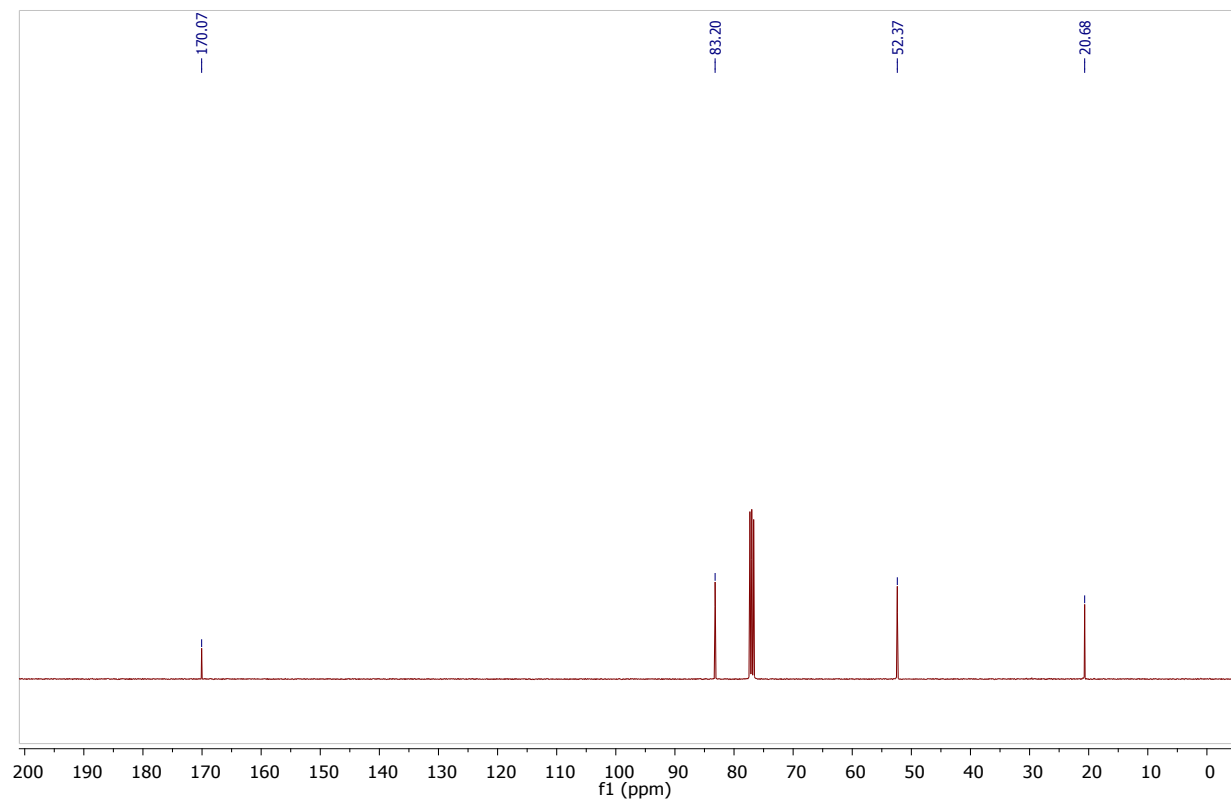
¹³C NMR Spectrum of compound **31** in CDCl₃



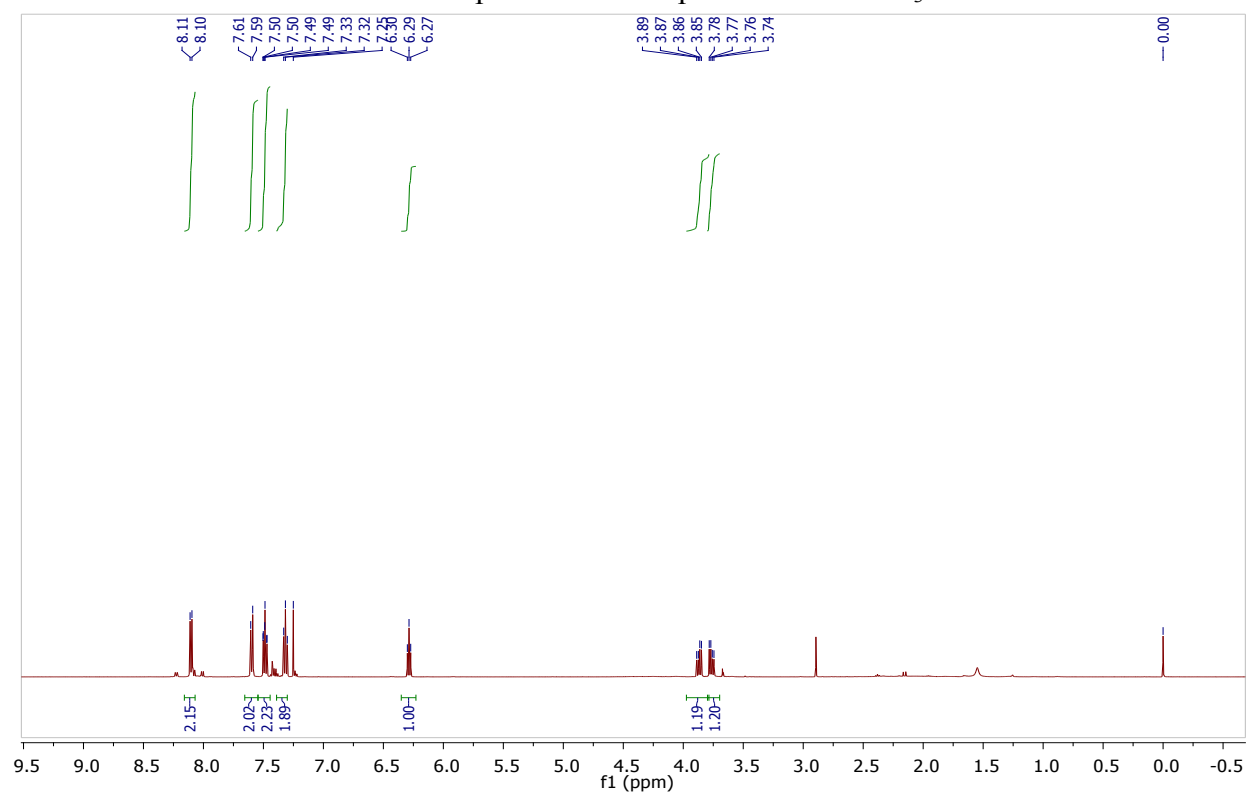
¹H NMR Spectrum of compound **32** in CDCl₃



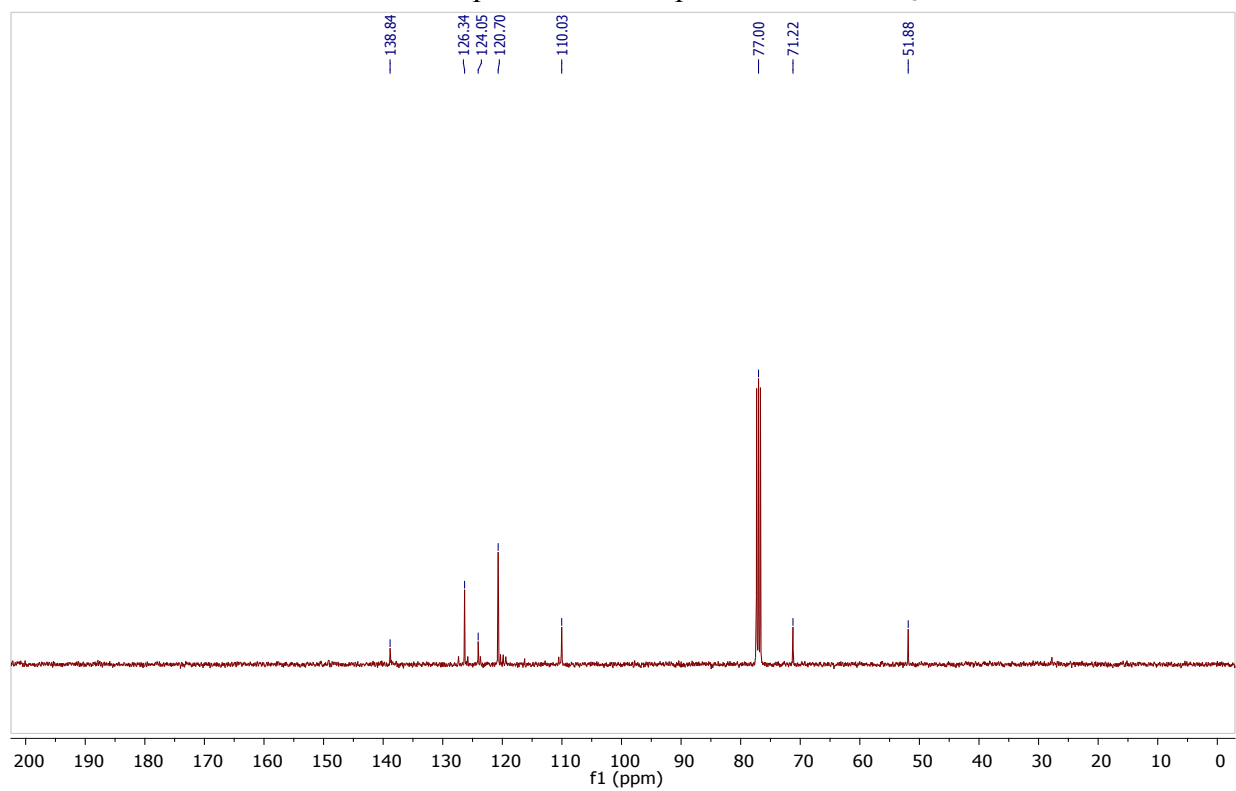
¹³C NMR Spectrum of compound **32** in CDCl₃



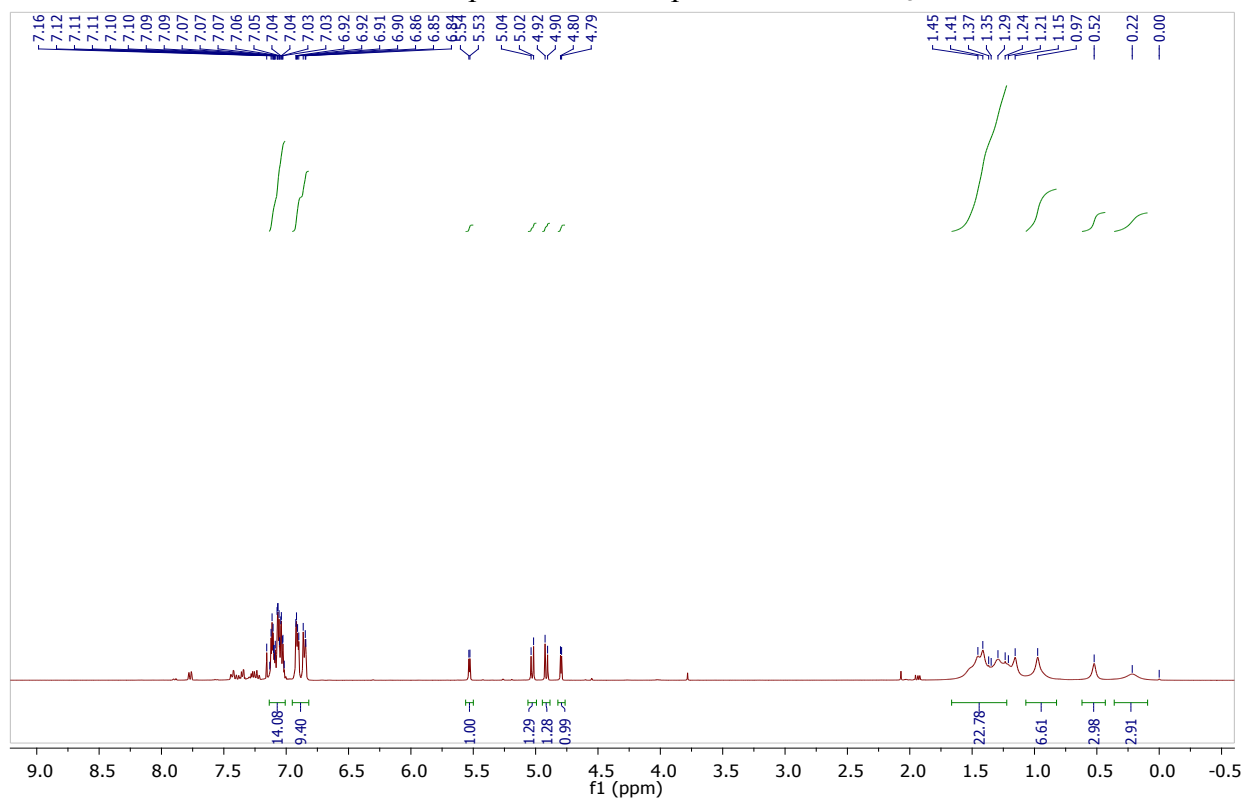
¹H NMR Spectrum of compound **33** in CDCl₃



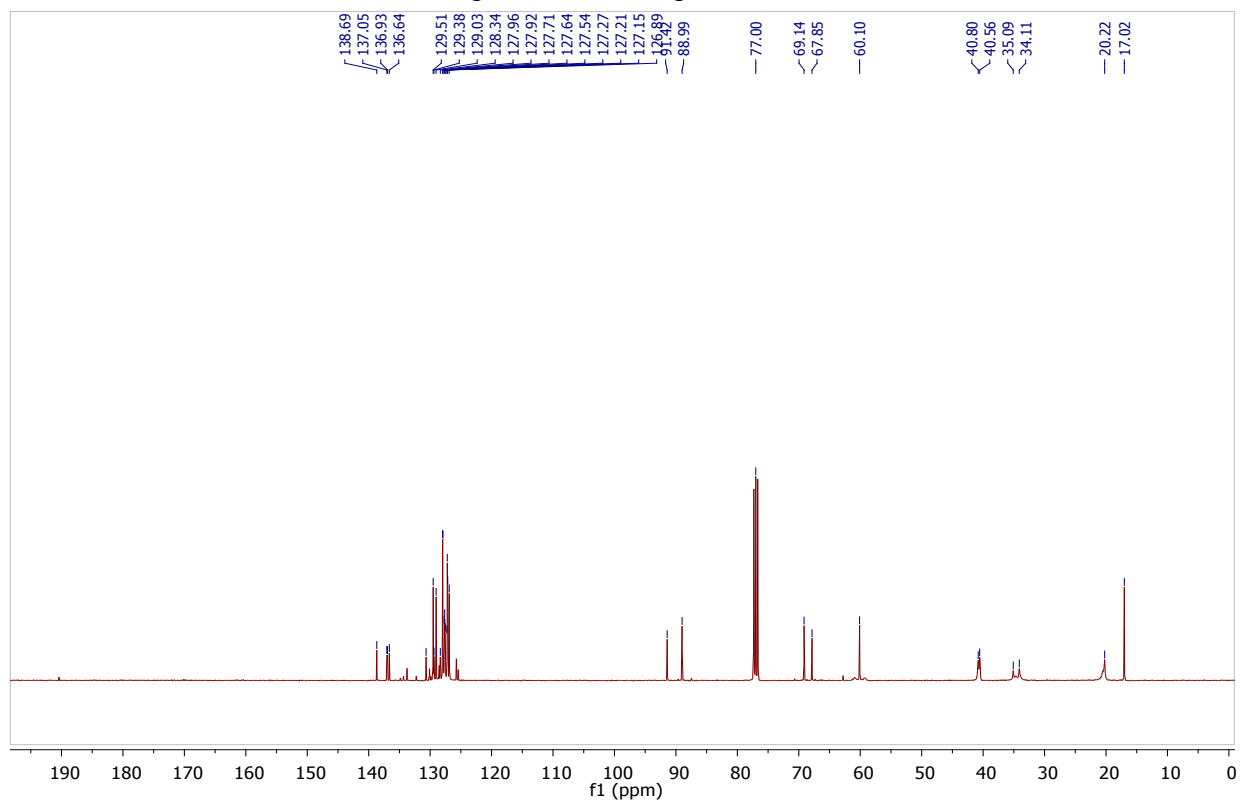
¹³C NMR Spectrum of compound **33** in CDCl₃



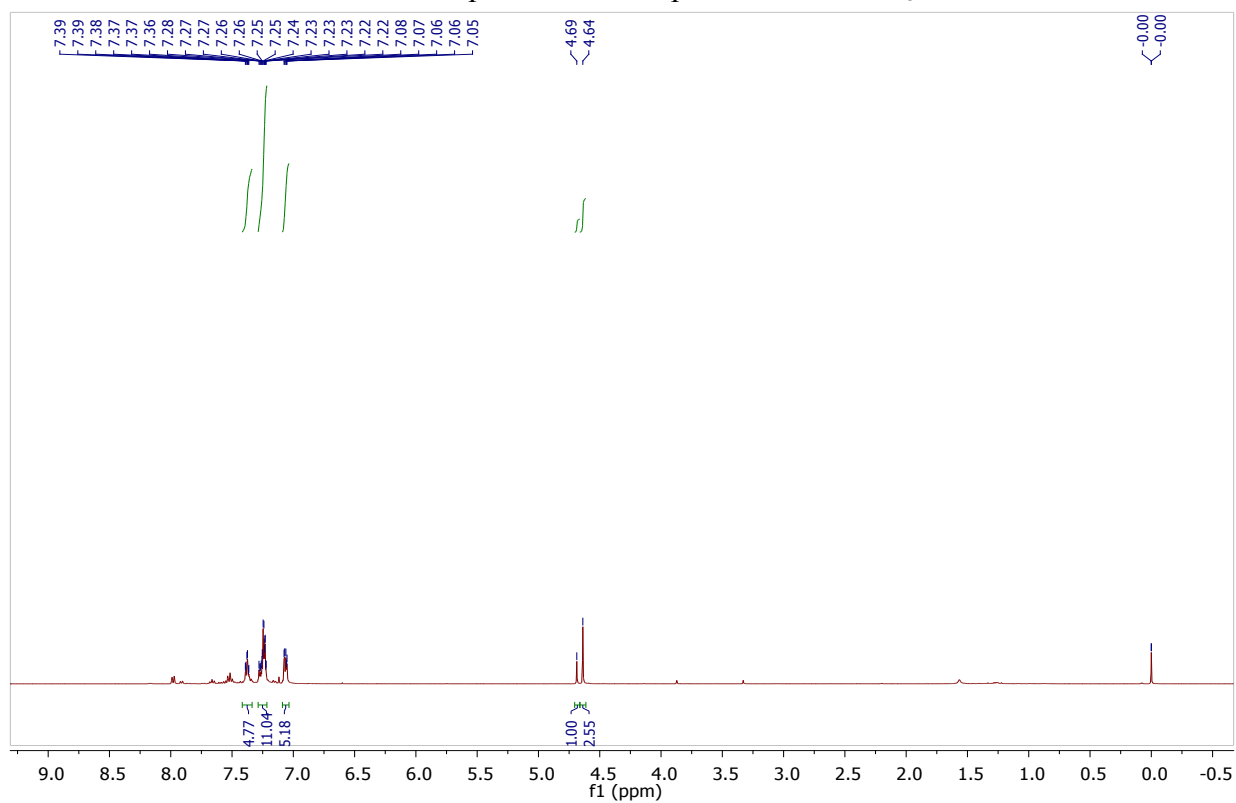
¹H NMR Spectrum of compound **34** in CDCl₃



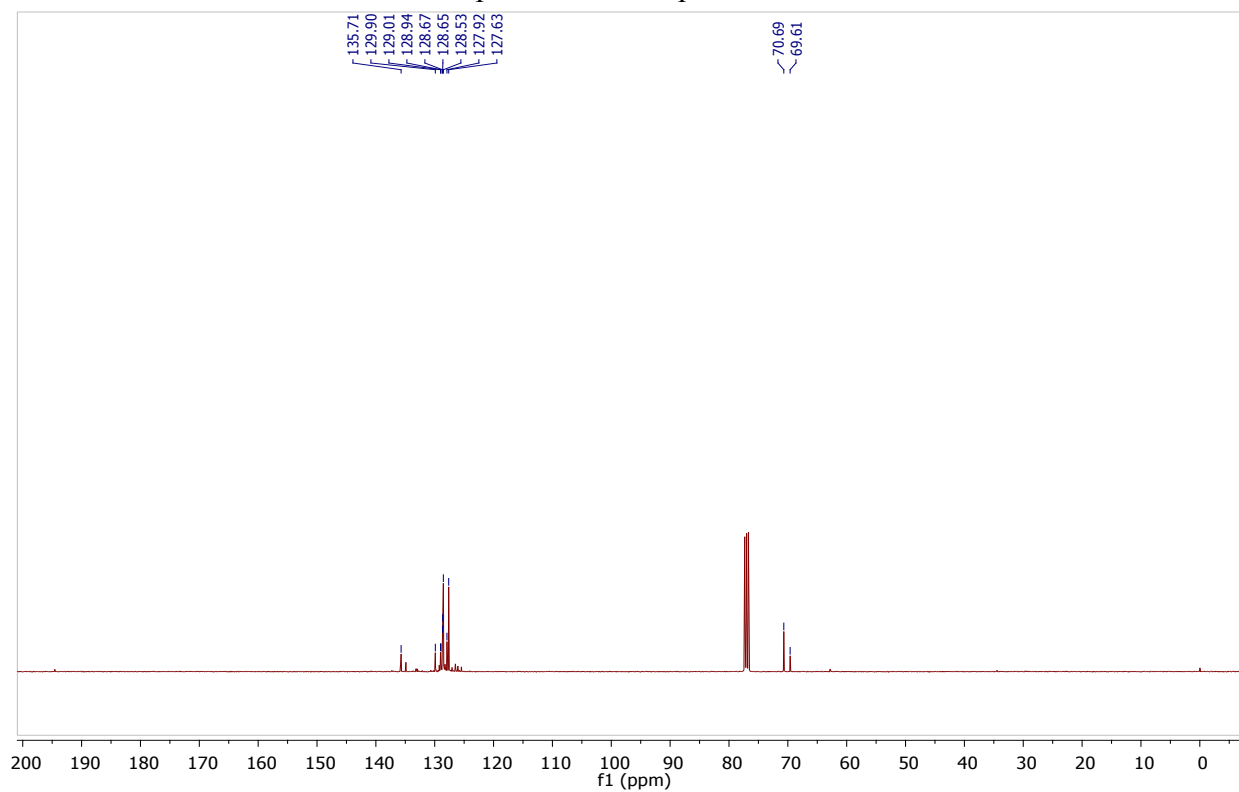
¹³C NMR Spectrum of compound **34** in CDCl₃



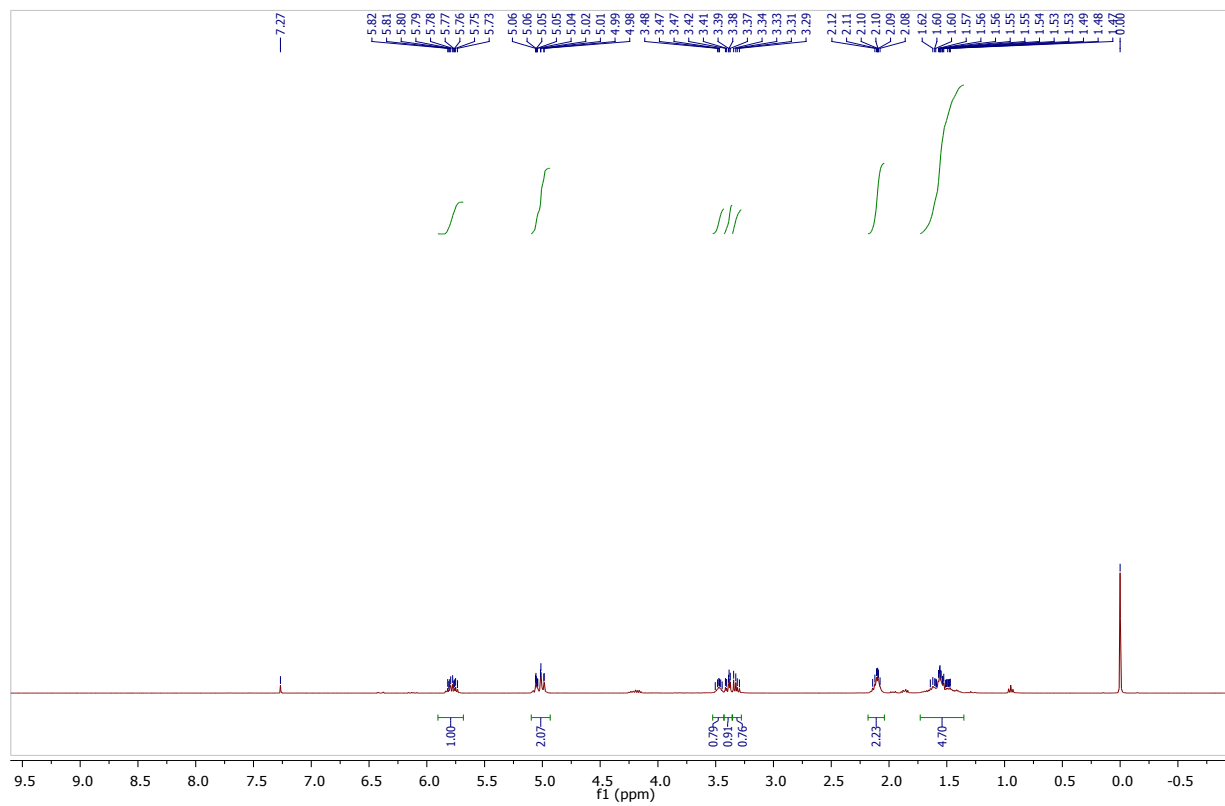
¹H NMR Spectrum of compound **35** in CDCl₃



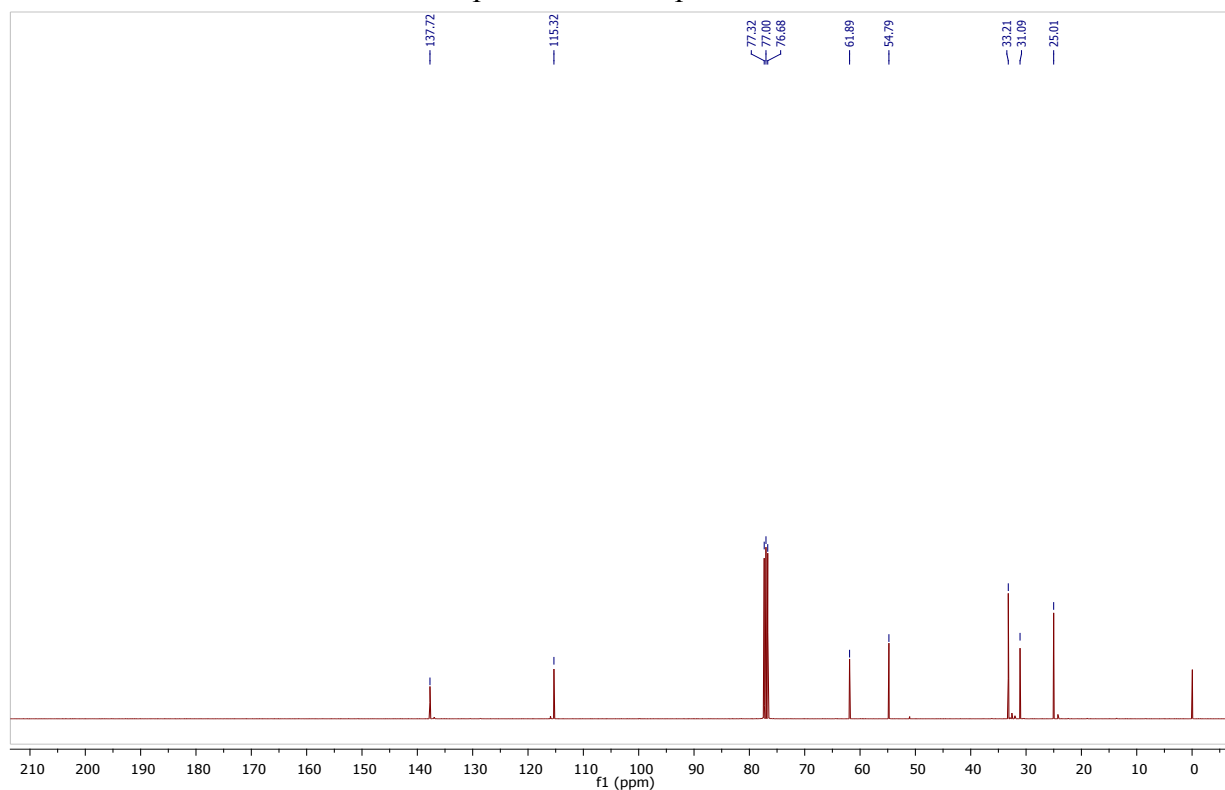
¹³C NMR Spectrum of compound **35** in CDCl₃



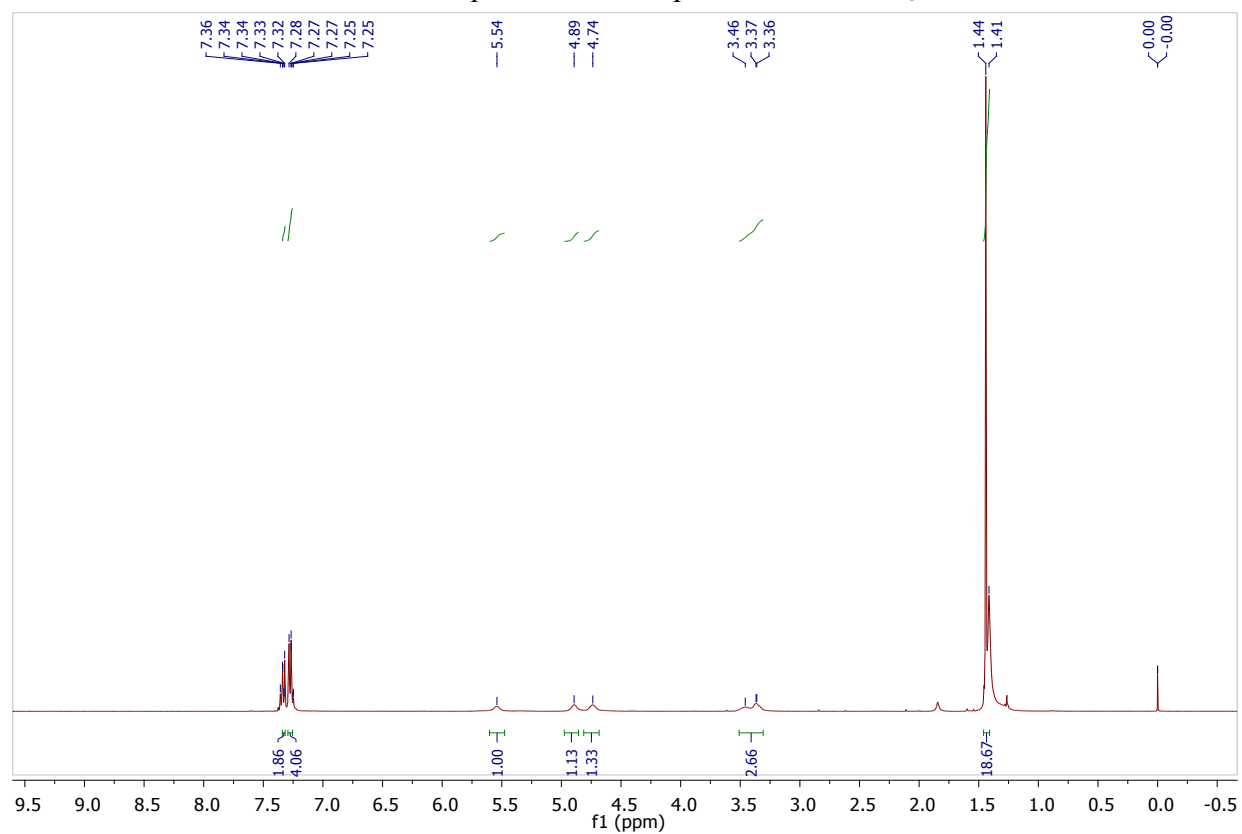
¹H NMR Spectrum of compound **36** in CDCl₃



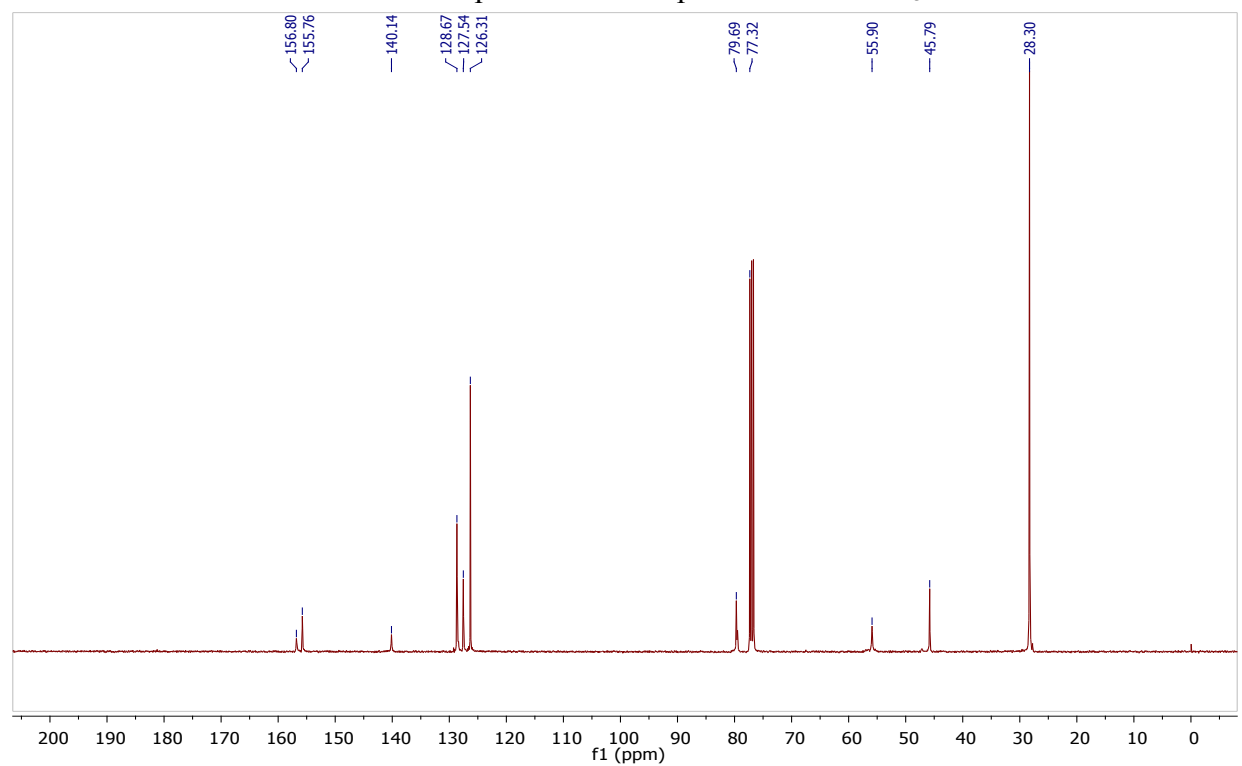
¹³C NMR Spectrum of compound **36** in CDCl₃



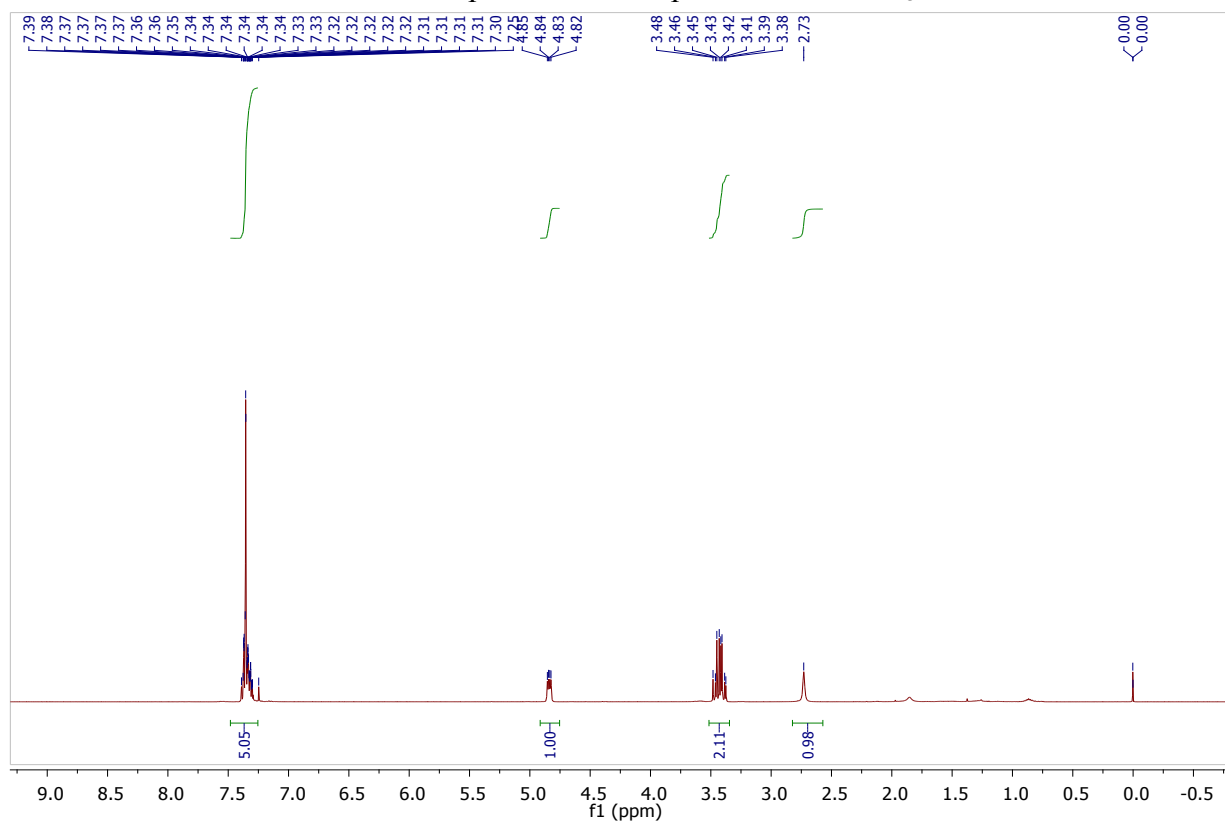
¹H NMR Spectrum of compound **37** in CDCl₃



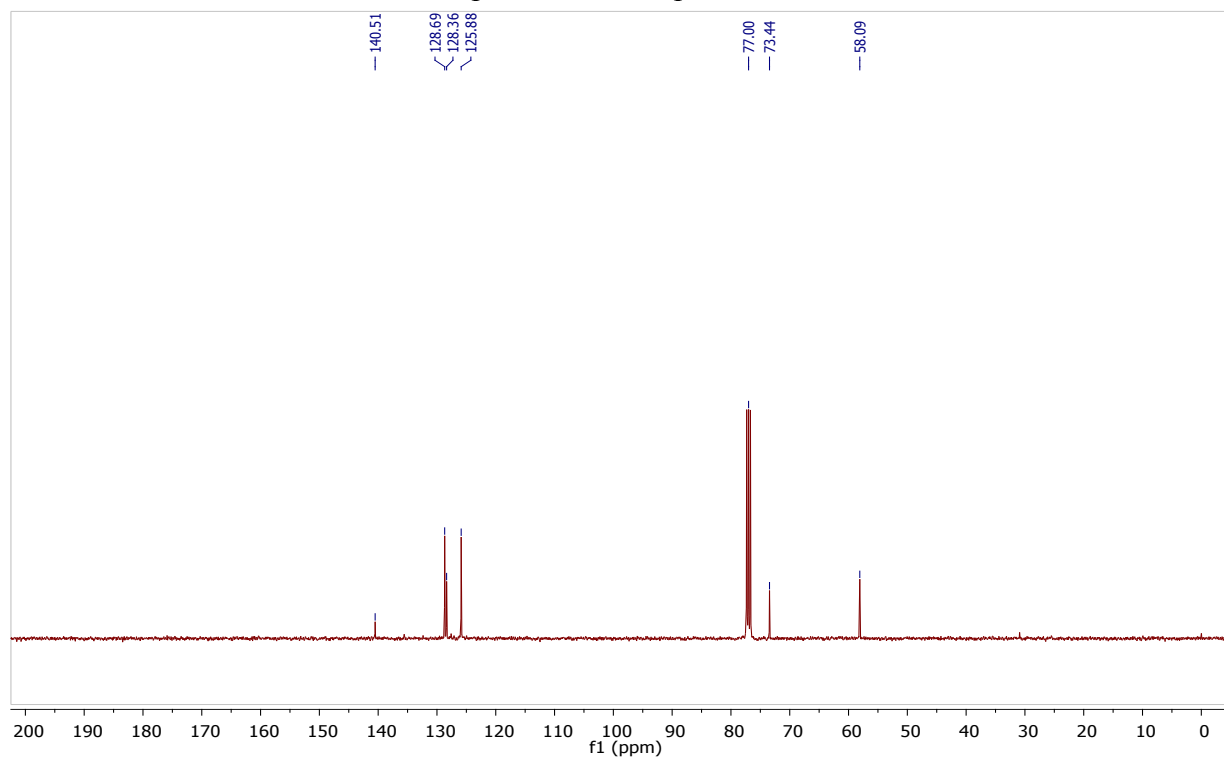
¹³C NMR Spectrum of compound **37** in CDCl₃



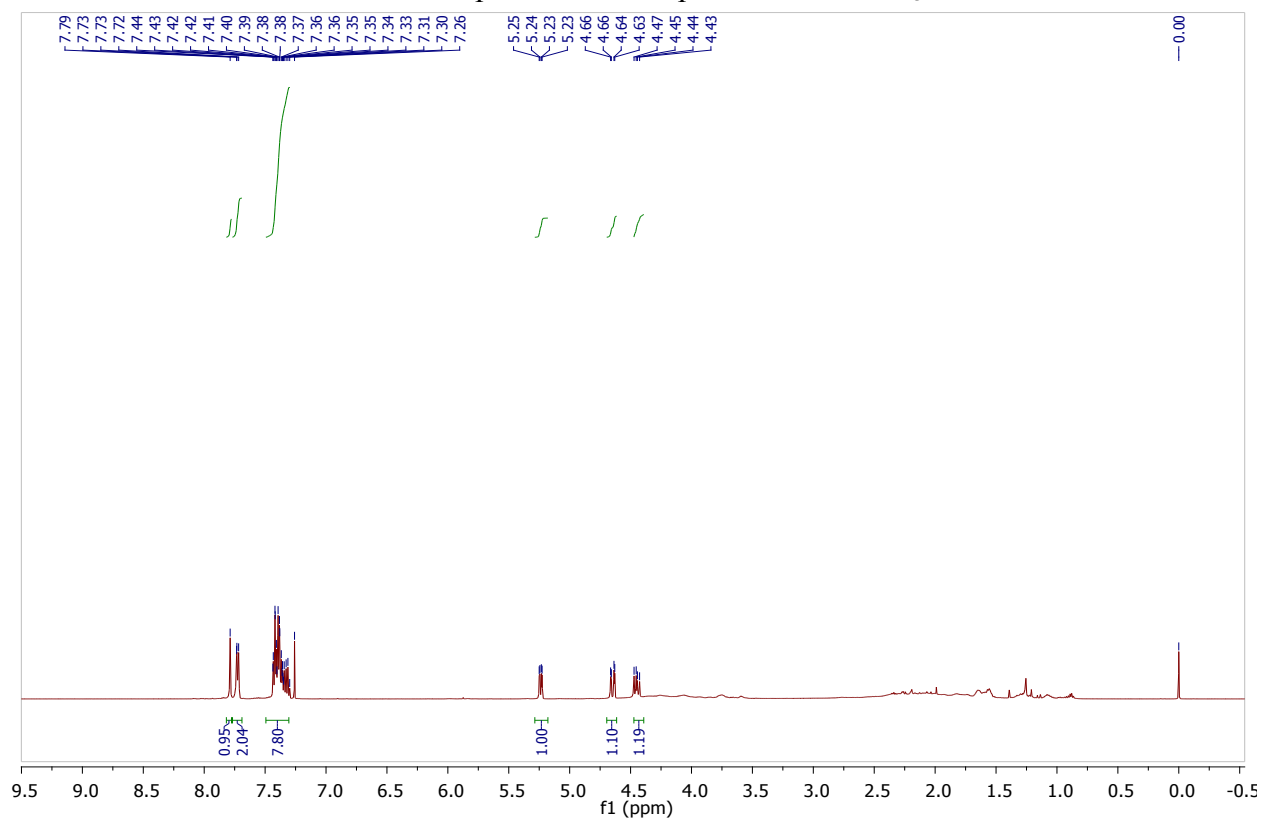
^1H NMR Spectrum of compound **38** in CDCl_3



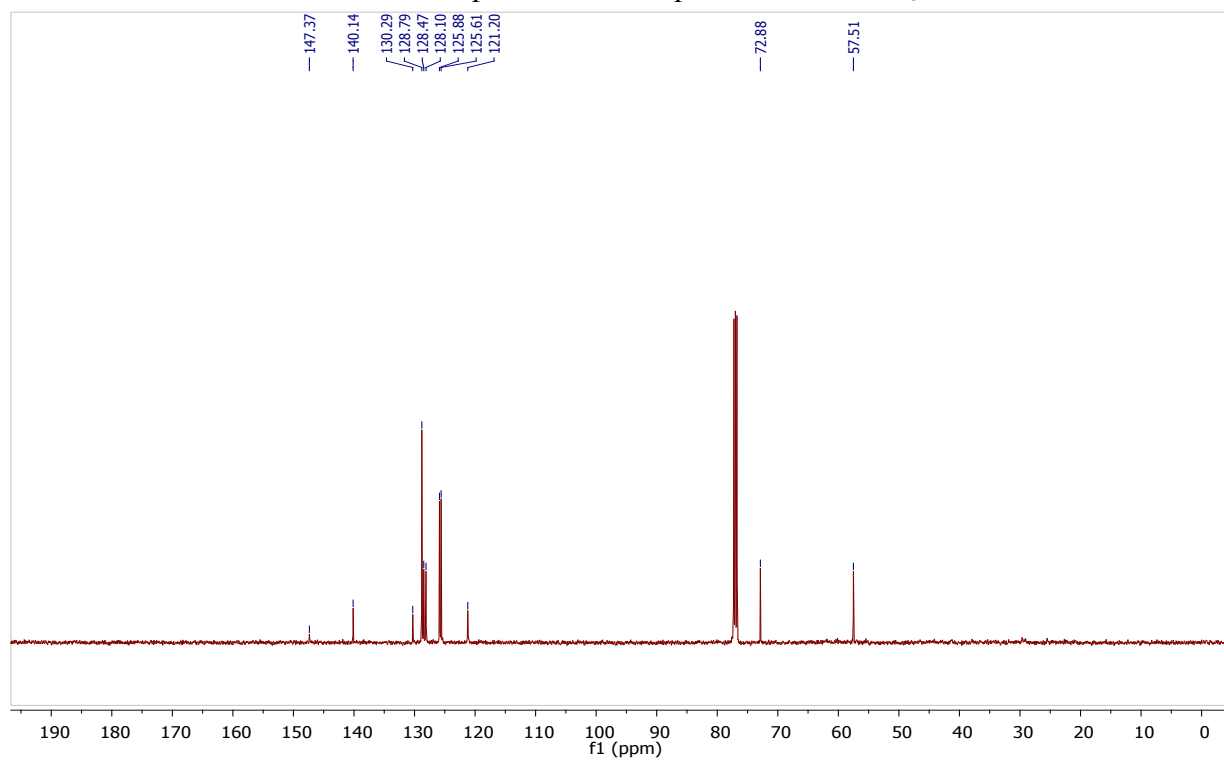
^{13}C NMR Spectrum of compound **38** in CDCl_3



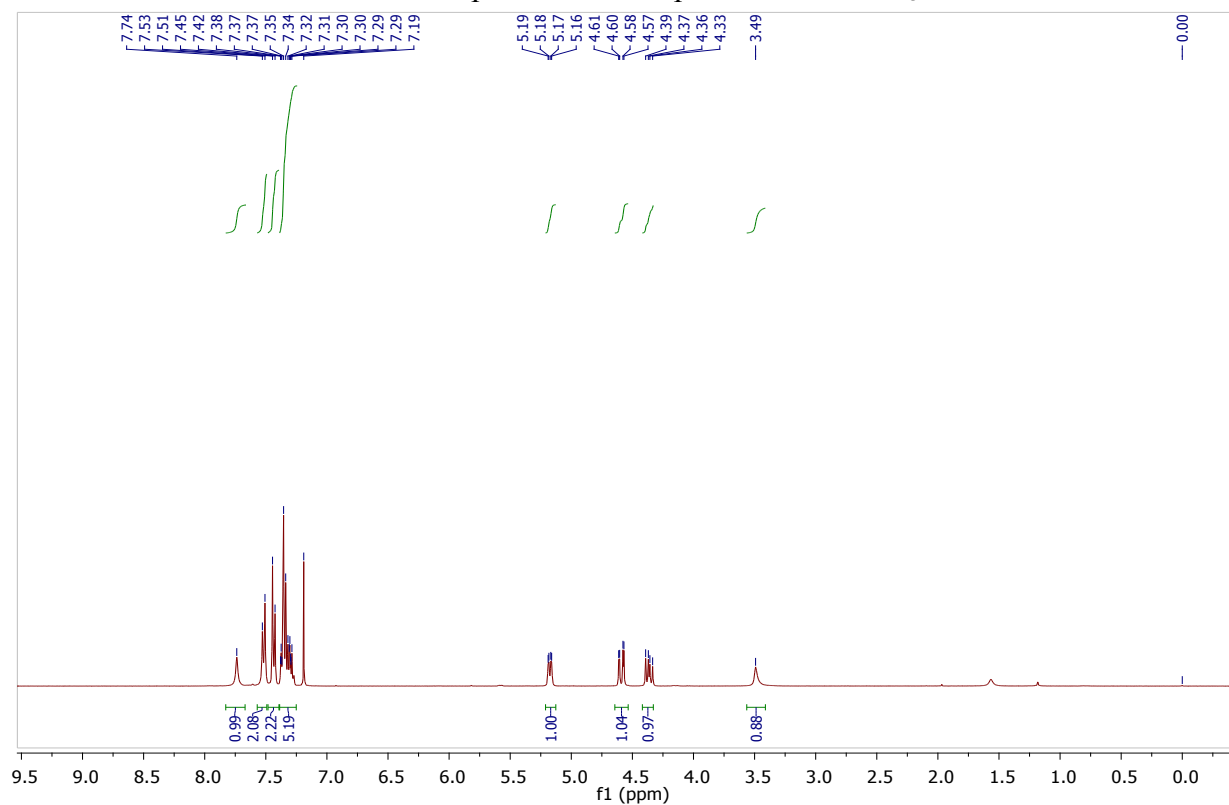
¹H NMR Spectrum of compound **39** in CDCl₃



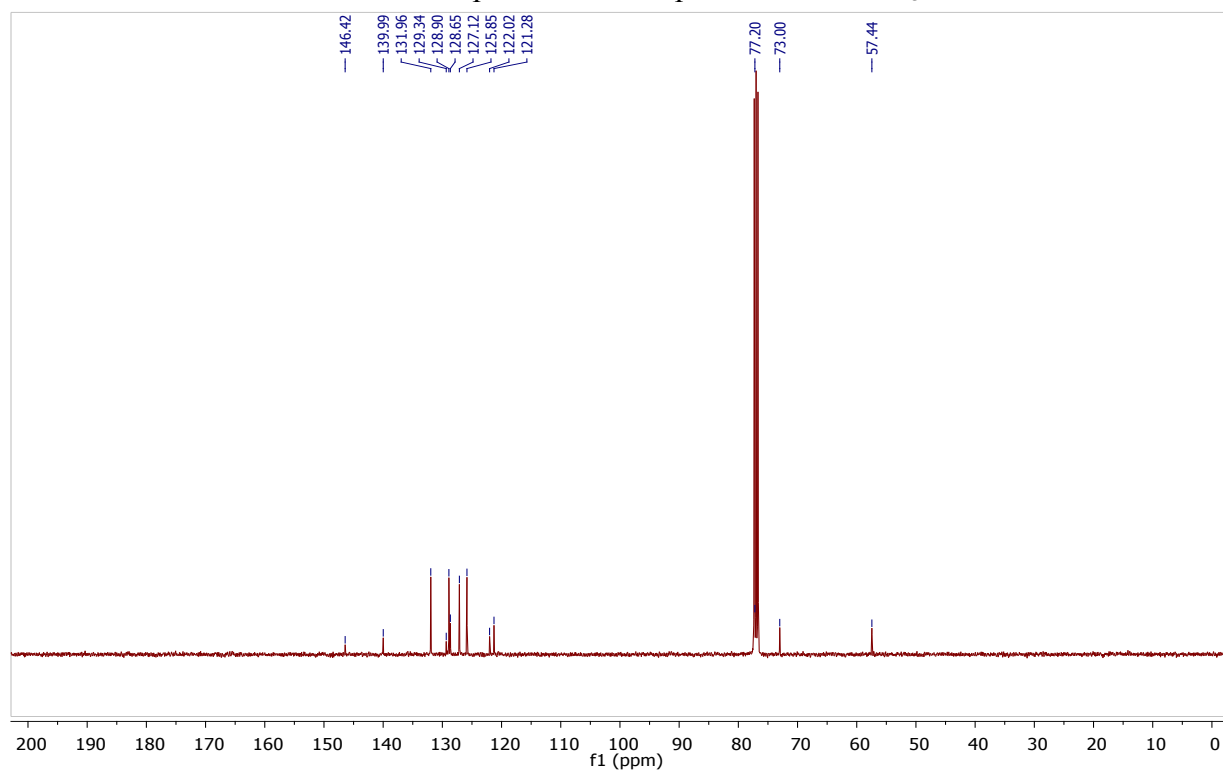
¹³C NMR Spectrum of compound **39** in CDCl₃



^1H NMR Spectrum of compound **40** in CDCl_3



^{13}C NMR Spectrum of compound **40** in CDCl_3



References and Notes:

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