Iron-Catalyzed Hydroamination and Hydroetherification of Unactivated Alkenes

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Supporting Information

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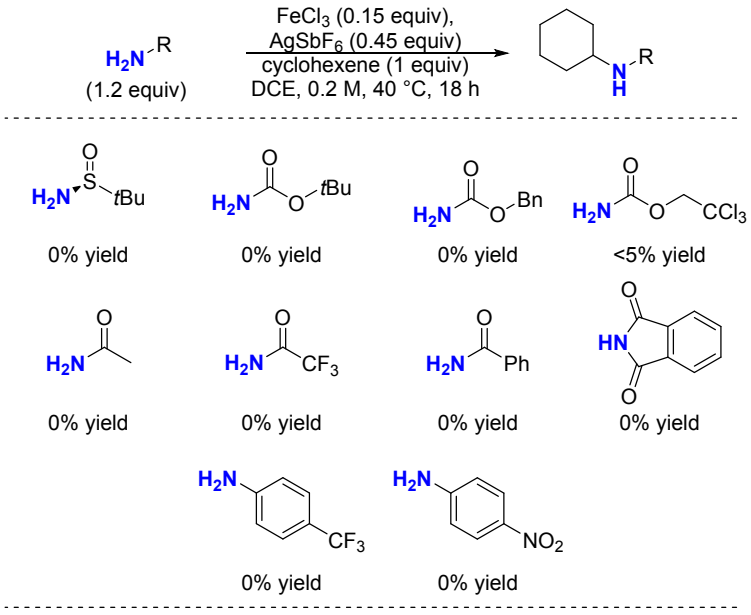
General Methods

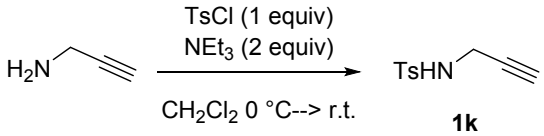
All the reactions were carried out in flame-dried glassware fitted with rubber septa under a nitrogen atmosphere unless otherwise stated. Analytical grade solvents and commercially available reagents were purchased from commercial sources and used directly without further purification unless otherwise stated. THF and MeOH were purified according to the Grubbs procedure.[1] Anhydrous CH_2Cl_2 and 1,2-dichloroethane (DCE) were dried and stored over 3Å molecular sieves. Thin-layer chromatography (TLC) was carried out on Merck 60 F254 precoated, glass silica gel plates which were visualized with either ultraviolet light or stained with vanillin or $KMnO_4$. Flash chromatography was performed using ZEOprep 60 ECO 40-63 μm silica gel. Automated column chromatography was performed on a Biotage Isolera One using Biotage Snap KPSil cartridges (25g or 50g SiO2), Biotage Snap Ultra cartridges (25g or 50g SiO2), Yamzen universal columns (16g 40μm SiO2), or RediSepRf columns (24g or 40g SiO2) collecting with full spectrum analysis between wavelengths 200-400nm and monitoring wavelengths 254nm and 280nm. ^{1}H-NMR, ^{13}C-NMR and ^{19}F-NMR spectra were recorded at 25 °C using a Varian I400 (^{1}H-NMR at 400MHz, ^{13}C-NMR at 100MHz and ^{19}F-NMR at 375MHz), Varian VXR400 (^{1}H-NMR at 400MHz, ^{13}C-NMR at 100MHz and ^{19}F-NMR at 375MHz), Varian I500 (^{1}H-NMR at 500MHz and ^{13}C-NMR at 125MHz), Varian I600 (^{1}H-NMR at 600MHz ^{13}C-NMR at 150MHz). Chemical shifts are reported in ppm with reference to solvent signals [^{1}H-NMR: CDCl3 (7.26 ppm); ^{13}C-NMR: CDCl3 (77.16 ppm)]. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; and m, multiplet. Infrared spectra (IR) were obtained on a Bruker Tensor II FT-IR Spectrometer analyzed as a thin film and recorded in wavenumbers (cm^{-1}). High Resolution Mass (HRMS) analysis was obtained using Electron Impact Ionization (EI) and reported as m/z (relative intensity) for the molecular ion [M], with Electrospray Ionization (ESI), or with Atmospheric Pressure Chemical Ionization (APCI) and reporting the molecular ion [M+H], [M+Na] or a suitable fragment ion.

Supplementary Experiments

Table SI-1: Unsuccessful Amine Substrates

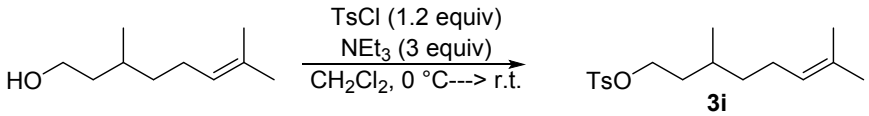


Experimental Procedures and Characterization of Starting Materials



4-Methyl-*N*-(prop-2-yn-1-yl)benzenesulfonamide (1k)
To a round-bottom flask equipped with a magnetic stir bar was added propargylamine (1.4 mL, 22 mmol), triethylamine (5.8 mL, 44 mmol) and CH_2Cl_2 (50 mL). The reaction was cooled to 0 °C in an ice bath and *p*-toluenesulfonyl chloride (4.19 g, 22 mmol) was added portion-wise over 2 minutes. After stirring for 10 minutes, the ice bath was removed, and the reaction was stirred at room temperature for 5 hours. The reaction was quenched with H_2O (50 mL), extracted with CH_2Cl_2 (3 x 50 mL), washed with brine, dried with $MgSO_4$ and concentrated under reduced pressure. The crude mixture was purified by silica chromatography (R_f = 0.19, 25% EtOAc in hexanes) yielding **1k** (3.93 g, 85% yield) as an off-white solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[1]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.12 (s, 1H), 3.80 (dd, *J* = 6.1, 2.5 Hz, 2H), 2.41 (s, 3H), 2.08 (t, *J* = 2.5 Hz, 1H).
^{13}C NMR ($CDCl_3$, 100 MHz): δ 143.87, 136.52, 129.74, 127.45, 78.09, 73.02, 32.87, 21.62.

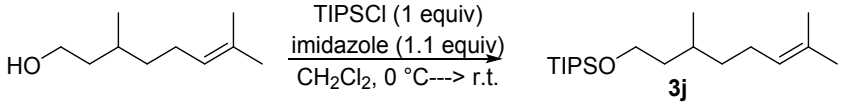


3i

3,7-Dimethyloct-6-en-1-yl 4-methylbenzenesulfonate (3i)

To a round-bottom flask equipped with a magnetic stir bar was added citronellol (1.83 mL, 10 mmol, 1 equiv), CH_2Cl_2 (40 mL, 0.25 M), and NEt_3 (4.18 mL, 30 mmol, 3 equiv). The mixture was cooled to 0 °C in an ice bath and TsCl (2.29 g, 12 mmol, 1.2 equiv) was added portion-wise. The reaction was stirred at 0 °C for 15 minutes then room temperature for 14 hours. The reaction was quenched with water (100 mL) and extracted with CH_2Cl_2 (3 x 75 mL). The organic extracts were wash with brine (100 mL), dried with $MgSO_4$ and concentrated. Purification by silica flash chromatography (R_f = 0.57, 25% EtOAc in hexanes) afforded **3i** (1.69 g, 55% yield) as a colorless liquid. ^{1}H and ^{13}C NMR spectra match those previously reported.[2]

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.77 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 5.04 – 4.95 (m, 1H), 4.10 – 3.95 (m, 2H), 2.42 (s, 3H), 2.00 – 1.78 (m, 2H), 1.73 – 1.60 (m, 4H), 1.61 – 1.45 (m, 4H), 1.45 – 1.34 (m, 1H), 1.28 – 1.16 (m, 1H), 1.15 – 1.03 (m, 1H), 0.80 (d, *J* = 6.6 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.70, 133.27, 131.42, 129.85, 127.89, 124.37, 69.10, 36.73, 35.69, 28.90, 25.73, 25.30, 21.64, 19.09, 17.66.

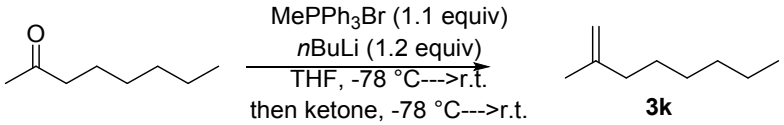


3j

((3,7-Dimethyloct-6-en-1-yl)oxy)triisopropylsilane (3j)

To a round-bottom flask equipped with a magnetic stir bar was added citronellol (1.83 mL, 10 mmol, 1 equiv), CH_2Cl_2 (30 mL, 0.33 M), and imidazole (748 mg, 11 mmol, 1.1 equvi). The mixture was cooled to 0 °C and TIPSCl (2.14 mL, 10 mmol, 1 equiv) was added dropwise over 2 minutes. The reaction was stirred at 0 °C for 15 minutes then at room temperature for 14 hours. The reaction was quenched with water (100 mL) and extracted with CH_2Cl_2 (3 x 75 mL). The organic extracts were wash with brine (100 mL), dried with $MgSO_4$ and concentrated. Purification by silica flash chromatography (R_f = 0.8 in 10% EtOAc in hexanes) afforded **3j** (2.26 g, 72% yield) as a colorless liquid. ^{1}H and ^{13}C NMR spectra match those previously reported.[3]

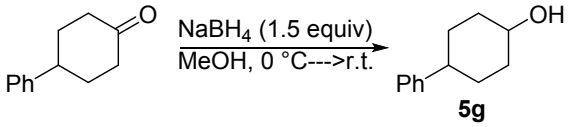
^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.18 – 5.06 (m, 1H), 3.82 – 3.62 (m, 2H), 2.07 – 1.90 (m, 2H), 1.68 (s, 3H), 1.64 – 1.51 (m, 5H), 1.41 – 1.29 (m, 2H), 1.21 – 1.13 (m, 1H), 1.13 – 1.02 (m, 21H), 0.90 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 131.14, 125.10, 61.84, 40.27, 37.44, 29.31, 25.86, 25.69, 19.86, 18.21, 17.77, 12.22.



2-Methyl-1-octene (3k)

To a flame-dried round-bottom flask equipped with a magnetic stir bar was added methyltriphenylphosphonium bromide (11.79 g, 33 mmol, 1.1. equiv). The flask was fitted with a rubber septum, evacuated, and backfilled with N_2. Anhydrous THF (100 mL) was added and the solution was cooled to –78 °C in a dry ice/ acetone bath. *n*-Butyllithium solution (14.4 mL, 36 mmol, 1.2 equiv, 2.5 M in hexanes) was added dropwise over 10 minutes, stirred at –78 °C for 15 minutes and then warmed to room temperature for 30 minutes. The reaction was re-cooled to –78 °C and 2-octanone (3.85 mL, 30 mmol, 1 equiv) was added dropwise. The reaction was stirred at –78 °C for 30 minutes then slowly warmed to room temperature and stirred for 4 hours. The reaction was quenched with saturated NH_4Cl solution (100 mL), extracted with Et_2O (3 x 100mL), washed with brine (100 mL), dried with $MgSO_4$, and concentrated under reduced pressure. To remove bulk $Ph_3P{=}O$, the resulting residue was filtered through a short silica plug, eluting with *n*-pentane. After concentrating the eluent, the resulting oil was purified by short path distillation (b.p. 145 °C) and alkene **3k** (1.45 g, 38 % yield) was isolated a colorless liquid. ^1H and ^{13}C NMR spectra match those previously reported.[4]

^1H NMR ($CDCl_3$, 500 MHz): δ 4.68 (d, J = 10.4 Hz, 2H), 2.01 (t, J = 7.7 Hz, 2H), 1.75 – 1.69 (m, 3H), 1.43 (m, 2H), 1.35 – 1.26 (m, 6H), 0.94 – 0.82 (m, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 146.33, 109.67, 38.04, 31.98, 29.21, 27.81, 22.83, 22.48, 14.22.



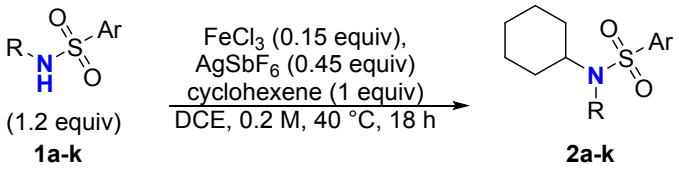
4-Phenylcyclohexanol (5g)

To a flame-dried round-bottom flask equipped with a magnetic stir bar was added $NaBH_4$ (284 mg, 7.5 mmol, 1.5 equiv). The flask was fitted with a rubber septum, evacuated, and backfilled with N_2. The flask was cooled to 0 °C in an ice bath and anhydrous MeOH (15 mL) was added. 4-Phenylcyclohexanone (870 µL, 5 mmol, 1 equiv) was added dropwise and stirred at 0 °C for 15 minutes then at room temperature for 2 hours. The reaction was quenched with saturated $NaHCO_3$ solution (50 mL), extracted with CH_2Cl_2 (3 x 50 mL) washed with brine (50 mL), dried with $MgSO_4$, and concentrated under reduced pressure. Purification by silica flash chromatography (R_f = 0.13 in 25% EtOAc in hexanes) afforded **5g** (572 mg, 65% yield) as a white solid. ^1H and ^{13}C NMR spectra match those previously reported.[5]
^1H NMR ($CDCl_3$, 500 MHz): δ 7.33 – 7.27 (m, 2H), 7.24 – 7.17 (m, 3H), 3.70 (tt, J = 10.6, 4.4 Hz, 1H), 2.51 (tt, J = 12.1, 3.6 Hz, 1H), 2.18 – 2.05 (m, 2H), 2.02 – 1.89 (m, 2H), 1.61 – 1.39 (m, 4H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 146.67, 128.49, 126.90, 126.20, 70.77, 43.55, 36.09, 32.58.

Experimental Procedures and Characterization of Products

Hydroamination

Sulfonamide Scope:



General Procedure A

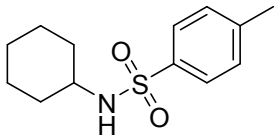
In a N_2 atmosphere glovebox, a 3-dram screw-cap vial was charged with $FeCl_3$ (12.2 mg, 0.075 mmol, 0.15 equiv). The vial was capped and removed from the glovebox. The cap of the vial was removed and a magnetic stir bar, 1,2-dichloroethane (2.5 mL, 0.2 M), alkene (0.5 mmol, 1 equiv), sulfonamide (0.6 mmol, 1.2 equiv), and $AgSbF_6$ (77 mg, 0.225 mmol, 0.45 equiv) were added. The vial was capped, sealed with PTFE tape and stirred in oil bath at 40 °C for 18 hours. Upon completion, the reaction was cooled to room temperature, filtered through a silica plug with CH_2Cl_2 (10 mL), then Et_2O (40 mL) and concentrated. The crude mixture was purified by silica chromatography using hexanes and EtOAc as the eluent.

General Procedure B

In a N_2 atmosphere glovebox, a 3-dram screw-cap vial was charged with $FeCl_3$ (12.2 mg, 0.075 mmol, 0.15 equiv). The vial was capped and removed from the glovebox. The cap of the vial was removed and a magnetic stir bar, 1,2-dichloroethane (2.5 mL, 0.2 M), alkene (0.5 mmol, 1 equiv) and sulfonamide (0.6 mmol, 1.2 equiv) were added. The vial was capped, sealed with PTFE tape and stirred in oil bath at 40 °C or at room temperature for 18 hours. Upon completion, the reaction was cooled to room temperature, filtered through a silica plug with CH_2Cl_2 (10 mL), then Et_2O (40 mL) and concentrated. The crude mixture was purified by silica chromatography using hexanes and EtOAc as the eluent.

Reaction Set-up Notes:

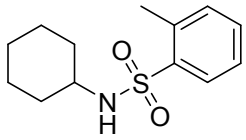
Due to its hydroscopic properties, anhydrous $FeCl_3$ was stored and weighed into the reaction vial in a N_2 atmosphere glovebox. $AgSbF_6$ was stored in the glovebox. Portions of $AgSbF_6$ were removed from the glovebox and was weighed at the bench. Reaction are not air sensitive and are reasonably tolerant to moisture.



2a

***N*-Cyclohexyl-4-methylbenzenesulfonamide** (**2a**). *p*-Toluenesulfonamide **1a** (103 mg, 0.6 mmol) and cyclohexene (51 µL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.35, 25% EtOAc in hexanes) afforded **2a** (99 mg, 78 %yield) as an off-white solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[6]

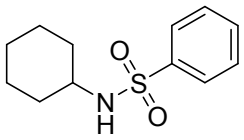
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.76 (d, 2H), 7.28 (d, 2H), 4.91 (s, 1H), 3.17 – 3.07 (m, 1H), 2.43 (s, 3H), 1.74 (dt, *J* = 12.1, 3.3 Hz, 2H), 1.63 (dq, *J* = 12.9, 3.7, 3.1 Hz, 2H), 1.50 (dt, *J* = 12.4, 4.2 Hz, 1H), 1.35 – 1.00 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.14, 138.63, 129.70, 127.03, 52.66, 33.94, 25.25, 24.72, 21.60.



2b

***N*-Cyclohexyl-2-methylbenzenesulfonamide (2b)**. *o*-Toluenesulfonamide **1b** (103 mg, 0.6 mmol) and cyclohexene (51 µL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.36, 25% EtOAc in hexanes) afforded **2b** (73.4 mg, 59% yield) as a colorless oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[7]

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.99 (d, *J* = 7.6 Hz, 1H), 7.43 (t, *J* = 7.3 Hz, 1H), 7.29 (t, *J* = 7.3 Hz, 2H), 4.77 (d, *J* = 7.8 Hz, 1H), 3.19 – 3.01 (m, 1H), 2.65 (s, 3H), 1.80 – 1.67 (m, 2H), 1.67 – 1.54 (m, 2H), 1.54 – 1.42 (m, 1H), 1.29 – 1.01 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 139.27, 136.98, 132.62, 132.53, 129.33, 126.19, 52.65, 33.99, 25.25, 24.76, 20.37.

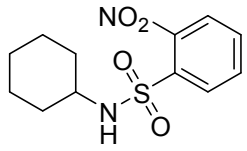


2c

***N*-Cyclohexylbenzenesulfonamide (2c)**. Benzenesulfonamide **1c** (94 mg, 0.6 mmol) and cyclohexene (51 µL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.55, 25% EtOAc in hexanes) afforded **2c** (84

mg, 70% yield) as a colorless solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[7]

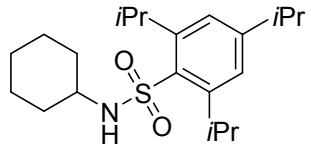
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.90 (d, *J* = 7.4 Hz, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 5.04 (d, *J* = 7.5 Hz, 1H), 3.18 – 3.02 (m, 1H), 1.79 – 1.66 (m, 2H), 1.65 – 1.55 (m, 2H), 1.51 – 1.43 (m, 1H), 1.30 – 1.02 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 141.58, 132.45, 129.09, 126.95, 52.74, 33.90, 25.20, 24.69.



2d

***N*-Cyclohexyl-2-nitrobenzenesulfonamide (2d)**. 2-Nitrobenzenesulfonamide **1d** (131 mg, 0.65 mmol) and cyclohexene (55 μL, 0.54 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.17, 25% EtOAc in hexanes) afforded **2d** (86.5 mg, 56% yield) as a brown solid.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.19 – 8.12 (m, 1H), 7.87 – 7.80 (m, 1H), 7.77 – 7.68 (m, 2H), 5.23 (d, *J* = 7.7 Hz, 1H), 3.39 – 3.21 (m, 1H), 1.86 – 1.71 (m, 2H), 1.72 – 1.59 (m, 2H), 1.52 (s, 1H), 1.34 – 1.02 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.92, 135.29, 133.50, 133.02, 130.71, 125.45, 53.68, 33.85, 25.15, 24.62.
HRMS (APCI) Calc. for $C_{12}H_{16}N_2O_4S$ [M+H] 285.0904. Found 285.0904.
FTIR (Film) cm^{-1}: 3331, 2932, 2856, 1538, 1339, 1299, 1165, 592.

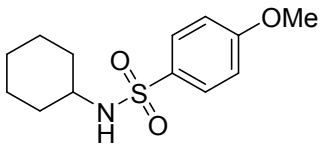


2e

***N*-Cyclohexyl-2,4,6-triisopropylbenzenesulfonamide (2e)**. 2,4,6-Triisopropylbenzenesulfonamide **1e** (170 mg, 0.6 mmol) and cyclohexene (51 μL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.55, 25% EtOAc in hexanes) afforded **2e** (126 mg, 69% yield) as a colorless solid.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.14 (s, 2H), 4.48 (d, *J* = 7.6 Hz, 1H), 4.27 – 4.12 (m, 2H), 3.28 – 3.13 (m, 1H), 2.89 (p, *J* = 6.9 Hz, 1H), 1.91 – 1.76 (m, 2H), 1.73 – 1.58 (m, 2H), 1.58 – 1.48 (m, 1H), 1.34 – 1.02 (m, 23H).

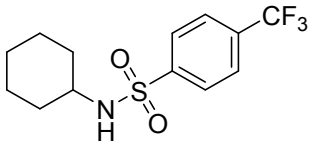
^{13}C NMR ($CDCl_3$, 125 MHz): δ 152.47, 150.01, 133.99, 123.81, 52.41, 34.28, 34.15, 29.67, 25.30, 24.97, 24.93, 23.66.
HRMS (APCI) Calc. for $C_{21}H_{35}NO_2S$ [M+H] 366.2461. Found 366.2465.
FTIR (Film) cm^{-1}: 3278, 2929, 2956, 1600, 1383, 1150, 880, 660, 568.



2f

***N*-Cyclohexyl-4-methoxybenzenesulfonamide (2f)**. 4-Methoxybenzenesulfonamide **1f** (107 mg, 0.6 mmol) and cyclohexene (51 μL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.21, 25% EtOAc in hexanes) afforded **2f** (79 mg, 58% yield) as a colorless solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[8]

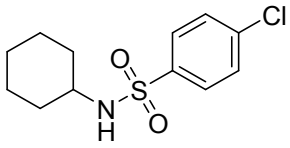
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.82 (d, *J* = 8.9 Hz, 2H), 6.95 (d, *J* = 8.9 Hz, 2H), 4.85 (d, *J* = 7.5 Hz, 1H), 3.85 (s, 3H), 3.14 – 3.01 (m, 1H), 1.78 – 1.68 (m, 2H), 1.68 – 1.57 (m, 2H), 1.55 – 1.42 (m, 1H), 1.29 – 1.02 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.73, 133.20, 129.12, 114.23, 55.67, 52.61, 33.94, 25.25, 24.73.



2g

***N*-Cyclohexyl-4-(trifluoromethyl)benzenesulfonamide (2g)**. 4-(Trifluoromethyl) benzenesulfonamide **1g** (135 mg, 0.6 mmol) and cyclohexene (51 μL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.36, 25% EtOAc in hexanes) afforded **2g** (81 mg, 53% yield) as a colorless solid.

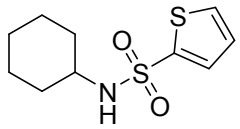
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.03 (d, *J* = 8.1 Hz, 2H), 7.77 (d, *J* = 8.2 Hz, 2H), 5.07 (d, *J* = 7.7 Hz, 1H), 3.24 – 3.11 (m, 1H), 1.80 – 1.68 (m, 2H), 1.68 – 1.57 (m, 2H), 1.55 – 1.46 (m, 1H), 1.30 – 1.04 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 145.18, 134.13 (q, *J* = 33.1 Hz), 127.37, 126.21 (q, *J* = 3.7 Hz), 123.27 (q, *J* = 272.8 Hz), 52.92, 33.88, 25.02, 24.56.
^{19}F NMR ($CDCl_3$, 375 MHz): δ -63.14.
HRMS (APCI) Calc. for $C_{13}H_{16}F_3NO_2S$ [M+H] 308.0927. Found 308.0927.
FTIR (Film) cm^{-1}: 2381, 2935, 2858, 1322, 1165, 1062, 712, 612.



2h

4-Chloro-*N*-cyclohexylbenzenesulfonamide (2h). 4-Chlorobenzenesulfonamide **1h** (115 mg, 0.6 mmol) and cyclohexene (51 μL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.52, 25% EtOAc in hexanes) afforded **2h** (99 mg, 72% yield) as a colorless solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[7]

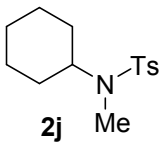
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.83 (d, *J* = 8.6 Hz, 2H), 7.46 (d, *J* = 8.6 Hz, 2H), 5.12 (d, *J* = 7.6 Hz, 1H), 3.15 – 3.06 (m, 1H), 1.75 – 1.68 (m, 2H), 1.61 (dt, *J* = 12.3, 3.8 Hz, 2H), 1.49 (dt, *J* = 13.8, 4.5 Hz, 1H), 1.27 – 1.02 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.18, 138.87, 129.39, 128.46, 52.84, 33.89, 25.16, 24.67.



2i

***N*-Cyclohexylthiophene-2-sulfonamide (2i)**. 2-Thiophenesulfonamide **1i** (121 mg, 0.74 mmol) and cyclohexene (63 μL, 0.62 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.26, 25% EtOAc in hexanes) afforded **2i** (96 mg, 63% yield) as a yellow oil.

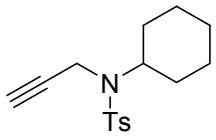
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.61 (dd, *J* = 3.8, 1.3 Hz, 1H), 7.55 (dd, *J* = 5.0, 1.3 Hz, 1H), 7.06 (dd, *J* = 5.0, 3.7 Hz, 1H), 4.97 (d, *J* = 7.6 Hz, 1H), 3.30 – 3.08 (m, 1H), 1.85 – 1.70 (m, 2H), 1.72 – 1.56 (m, 2H), 1.58 – 1.44 (m, 1H), 1.33 – 1.04 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.67, 131.85, 131.59, 127.37, 53.13, 33.85, 25.20, 24.72.
HRMS (APCI) Calc. for $C_{10}H_{15}NO_2S_2$ [M+H] 246.0617. Found 246.0619.
FTIR (Film) cm^{-1}: 3272, 2931, 2854, 1406, 1323, 1154, 1016, 715, 594.



2j

***N*-cyclohexyl-*N*,4-dimethylbenzenesulfonamide (2j)**.
N,4-Dimethylbenzenesulfonamide **1j** (111 mg, 0.6 mmol) and cyclohexene (51 μL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.43, 25% EtOAc in hexanes) afforded **2j** (41 mg, 31% yield) as an off-white solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[8]

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.1 Hz, 2H), 3.83 – 3.63 (m, 1H), 2.72 (s, 3H), 2.40 (s, 3H), 1.71 (h, J = 4.5, 4.1 Hz, 2H), 1.64 – 1.54 (m, 1H), 1.48 (d, J = 6.8 Hz, 2H), 1.35 – 1.19 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.93, 137.58, 129.69, 127.02, 56.88, 30.41, 28.70, 25.88, 25.47, 21.59.

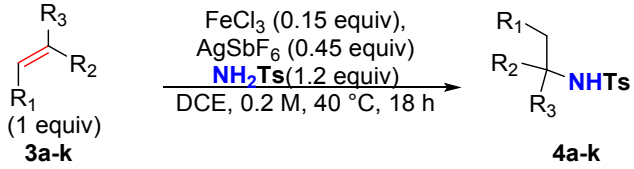


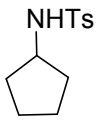
2k

***N*-cyclohexyl-4-methyl-*N*-(prop-2-yn-1-yl)benzenesulfonamide (2k)**. 4-Methyl-*N*-(prop-2-yn-1-yl)benzenesulfonamide **1k** (126 mg, 0.6 mmol) and cyclohexene (51 µL, 0.5 mmol, 1 equiv) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.43, 25% EtOAc in hexanes) afforded **2k** (43 mg, 30% yield) as a yellow oil.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.78 (d, J = 8.3 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 4.08 (d, J = 2.5 Hz, 2H), 3.66 (tt, J = 12.0, 3.7 Hz, 1H), 2.41 (s, 3H), 2.14 (t, J = 2.5 Hz, 1H), 1.82 – 1.56 (m, 5H), 1.55 – 1.42 (m, 2H), 1.32 – 1.18 (m, 2H), 1.10 – 0.98 (m, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.18, 138.29, 129.51, 127.42, 80.64, 72.23, 58.11, 32.29, 31.48, 26.13, 25.37, 21.64.
HRMS (APCI) Calc. for $C_{16}H_{21}NO_2S$ [M+H] 292.1371. Found 292.1369.
FTIR (Film) cm^{-1}: 2930, 2855, 1598, 1328, 1151, 1091, 1047, 1002, 882, 848.

Alkene Scope:



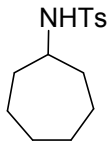


4a

***N*-Cyclopentyl-4-methylbenzenesulfonamide (4a)**. Cyclopentene **3a** (44 µL, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.35, 25% EtOAc in hexanes) yielded **4a** (81.5 mg, 68% yield) as a yellow oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[9]

^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 4.98 (d, *J* = 7.2 Hz, 1H), 3.55 (h, *J* = 6.8 Hz, 1H), 2.41 (s, 3H), 1.78 – 1.70 (m, 2H), 1.65 – 1.55 (m, 2H), 1.49 – 1.41 (m, 2H), 1.39 – 1.31 (m, 2H).

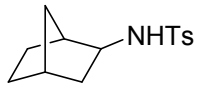
^{13}C NMR ($CDCl_3$, 100 MHz): δ 143.27, 137.97, 129.72, 127.22, 55.22, 33.46, 23.22, 21.62.



4b

***N*-Cycloheptyl-4-methylbenzenesulfonamide (4b)**. Cycloheptene **3b** (58 μL, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.38, 25% EtOAc in hexanes) yielded **4b** (57 mg, 43% yield) as a yellow oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[10]

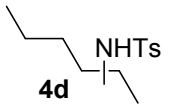
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.76 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 4.87 (d, *J* = 7.7 Hz, 1H), 3.31 (m, 1H), 2.41 (s, 3H), 1.78 – 1.70 (m, 2H), 1.56 – 1.25 (m, 8H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.14, 138.37, 129.70, 127.08, 54.88, 35.89, 28.04, 23.59, 21.60.



4c

***N*-(Bicyclo[2.2.1]heptan-2-yl)-4-methylbenzenesulfonamide (4c)**. Norbornene **3c** (53 mg, 0.56 mmol) and *p*-toluenesulfonamide (103 mg, 0.67 mmol) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.25, 25% EtOAc in hexanes) yielded **4c** (119 mg, 80% yield) as a colorless solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[11]

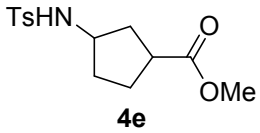
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 4.89 (d, *J* = 7.2 Hz, 1H), 3.10 (td, *J* = 7.5, 3.4 Hz, 1H), 2.42 (s, 3H), 2.19 – 2.14 (m, 1H), 2.09 (d, *J* = 4.2 Hz, 1H), 1.61 – 1.52 (m, 1H), 1.45 – 1.29 (m, 2H), 1.22 – 1.13 (m, 1H), 1.12 – 1.05 (m, 1H), 1.06 – 0.94 (m, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.25, 138.08, 129.74, 127.19, 56.77, 42.58, 40.78, 35.68, 35.25, 28.13, 26.44, 21.63.



4d

***N*-(Hexan-2-yl)-4-methylbenzenesulfonamide (4d)**. 1-Hexene **3d** (125μL, 1 mmol) and *p*-toluenesulfonamide (206 mg, 1.2 mmol) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.45, 25% EtOAc in hexanes) yielded an inseparable mixture of structural isomers **4d** (170.5 mg, 76% yield, 1:1 ratio of 2&3 substituted product) as a colorless oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[11]

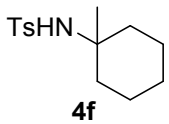
^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.79 – 7.73 (m, 4H), 7.29 – 7.21 (m, 4H), 5.27 – 5.11 (m, 1H), 3.30 – 3.17 (m, 1H), 3.18 – 3.07 (m, 1H), 2.37 (d, *J* = 4.3 Hz, 5H), 1.49 – 1.02 (m, 8H), 1.03 – 0.92 (m, 3H), 0.82 – 0.64 (m, 8H).
^{13}C NMR ($CDCl_3$, 150 MHz): δ 142.96, 142.89, 138.61, 138.43, 129.51, 129.44, 127.00, 126.95, 55.11, 49.94, 37.04, 36.53, 36.51, 27.62, 27.58, 22.28, 21.50, 21.49, 21.42, 18.44, 13.84, 13.78, 9.53.



4e

Methyl 3-((4-methylphenyl)sulfonamido)cyclopentane-1-carboxylate (4e).
Methyl 3-cyclopentenecarboxylate **3e** (61 μL, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure A. Purification by silica chromatography (R_f = 0.14, 25% EtOAc in hexanes) yielded a mixture of diastereomers **4e** (38 mg, 26% yield) as a colorless film.

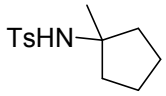
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.81 (d, *J* = 8.3 Hz, 2H, minor), 7.75 (d, *J* = 8.2 Hz, 2H, major), 7.29 (dd, *J* = 8.1, 6.3 Hz, 2H), 5.31 (d, *J* = 8.4 Hz, 1H), 4.99 (d, *J* = 6.7 Hz, 1H, major), 3.78 – 3.58 (m, 4H), 2.90 – 2.81 (m, 1H, major), 2.79 – 2.72 (m, 1H, minor), 2.42 (s, 3H), 2.09 – 1.82 (m, 3H), 1.82 – 1.67 (m, 2H), 1.67 – 1.54 (m, 1H, major), 1.50 – 1.40 (m, 1H, minor).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 177.38, 176.23, 143.57, 143.34, 138.19, 137.53, 129.87, 129.79, 127.22, 127.14, 126.52, 54.91, 54.69, 52.20, 51.95, 41.58, 41.35, 36.66, 36.58, 33.41, 33.40, 27.97, 27.66, 21.65, 21.63.
HRMS (APCI) Calc. for $C_{14}H_{19}NO_4S$ [M+H] 298.1108. Found 298.1111.
FTIR (Film) cm^{-1}: 3267, 2952, 1728, 1323, 1153, 1092, 665.



4f

4-Methyl-*N*-(1-methylcyclohexyl)benzenesulfonamide (4f). 1-Methylcyclohexene **3f** (24 µL, 0.2 mmol) and *p*-toluenesulfonamide (41 mg, 0.24 mmol) were treated using General Procedure B at 40 °C. Purification by silica chromatography (R_f = 0.43, 25% EtOAc in hexanes) yielded **4f** (28 mg, 52% yield) as a colorless solid.

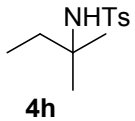
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.81 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 4.96 (s, 1H), 2.42 (s, 3H), 1.78 – 1.67 (m, 2H), 1.51 – 1.33 (m, 7H), 1.33 – 1.22 (m, 1H), 1.18 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.73, 141.08, 129.50, 126.98, 56.92, 38.49, 26.89, 25.34, 21.92, 21.58.
HRMS (APCI) Calc. for $C_{14}H_{21}NO_2S$ [M+H] 268.1366. Found 268.1366.
FTIR (Film) cm^{-1}: 3269, 2928, 1316, 1149, 1093, 814, 663, 570.



4g

4-Methyl-*N*-(1-methylcyclopentyl)benzenesulfonamide (4g). 1-Methylcyclopentene **3g** (52 µL, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure B at 40 °C. Purification by silica chromatography (R_f = 0.37, 25% EtOAc in hexanes) yielded **4g** (24 mg, 19% yield) as an off-white solid. ^{1}H and ^{13}C NMR spectra match those previously reported.

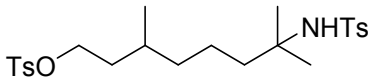
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H), 4.70 (s, 1H), 2.41 (s, 3H), 1.85 – 1.73 (m, 2H), 1.62 – 1.44 (m, 5H), 1.27 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.93, 140.53, 129.59, 127.05, 64.82, 40.25, 26.71, 23.05, 21.63.
HRMS (APCI) Calc. for $C_{13}H_{19}NO_2S$ [M+H] 254.1209. Found 254.1209
FTIR (Film) cm^{-1}: 3272, 2961, 1318, 1150, 1094, 815, 664, 549.



4h

4-Methyl-*N*-(*tert*-pentyl)benzenesulfonamide (4h). 2-Methyl-2-butene **3h** (61 µL, 0.57 mmol) and *p*-toluenesulfonamide (117 mg, 0.67 mmol) were treated using General Procedure B at room temperature. Purification by silica chromatography (R_f = 0.43, 25% EtOAc in hexanes) yielded **4h** (54.5 mg, 40% yield) as a colorless solid. ^{1}H and ^{13}C NMR spectra match those previously reported.[12]

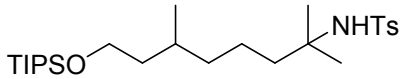
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.81 – 7.71 (m, 2H), 7.32 – 7.21 (m, 2H), 4.49 (s, 1H), 2.41 (s, 3H), 1.52 (q, *J* = 7.5 Hz, 2H), 1.15 (s, 6H), 0.83 (t, *J* = 7.5 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.90, 140.78, 129.57, 127.12, 57.49, 35.77, 27.24, 21.63, 8.36.



4i

3,7-Dimethyl-7-((4-methylphenyl)sulfonamido)octyl 4-methylbenzenesulfonate (4i). 3,7-Dimethyloct-6-en-1-yl 4-methylbenzenesulfonate **3i** (155 mg, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure B at room temperature. Purification by silica chromatography (R_f = 0.15, 25% EtOAc in hexanes) yielded **4i** (85 mg, 35% yield) as a colorless oil.

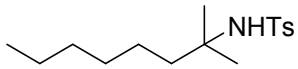
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.77 (t, *J* = 7.7 Hz, 4H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 4.91 (s, 1H), 4.06 – 3.87 (m, 2H), 2.43 (s, 3H), 2.39 (s, 3H), 1.66 – 1.53 (m, 1H), 1.49 – 1.30 (m, 4H), 1.27 – 1.01 (m, 8H), 1.00 – 0.89 (m, 1H), 0.74 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.77, 142.83, 140.75, 133.20, 129.91, 129.51, 127.92, 126.98, 69.07, 57.05, 42.96, 36.78, 35.69, 29.18, 27.81, 27.70, 21.70, 21.52, 21.08, 19.08.
HRMS (APCI) Calc. for $C_{24}H_{35}NO_5S_2$ [M+H] 482.2029. Found 482.2031.
FTIR (Film) cm^{-1}: 3277, 2930, 1355, 1323, 1174, 1147, 940, 814, 661, 553.



4j

***N*-(2,6-Dimethyl-8-((triisopropylsilyl)oxy)octan-2-yl)-4-methylbenzenesulfonamide (4j)**. ((3,7-Dimethyloct-6-en-1-yl)oxy)triisopropylsilane **3j** (156 mg, 0.5 mmol) and *p*-toluenesulfonamide (103 mg, 0.6 mmol) were treated using General Procedure B at room temperature. Purification by silica chromatography (R_f = 0.63, 25% EtOAc in hexanes) yielded **4j** (90 mg, 37% yield) as a colorless oil.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.79 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 4.85 (s, 1H), 3.76 – 3.55 (m, 2H), 2.41 (s, 3H), 1.57 – 1.34 (m, 4H), 1.34 – 0.89 (m, 32H), 0.83 (d, *J* = 6.4 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.77, 140.85, 129.52, 127.08, 61.73, 57.22, 43.30, 40.17, 37.39, 29.55, 27.81, 27.73, 21.57, 21.37, 19.75, 18.17, 12.13.
HRMS (APCI) Calc. for $C_{26}H_{49}NO_3SSi$ [M+H] 484.3281. Found 484.3277.
FTIR (Film) cm^{-1}: 3272, 2941, 2865, 1323, 1149, 1094, 814, 661, 554.



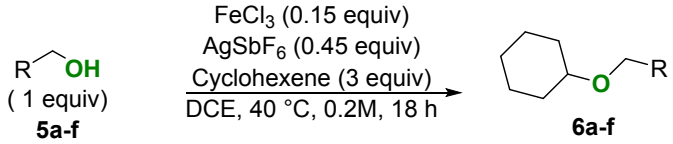
4k

4-Methyl-*N*-(2-methyloctan-2-yl)benzenesulfonamide (4k). 2-Methyl-1-octene **3k** (85 µL, 0.53 mmol) and *p*-toluenesulfonamide (103 mg, 0.64 mmol) were treated using General Procedure B at room temperature. Purification by silica chromatography (R_f = 0.55, 25% EtOAc in hexanes) yielded **4k** (52 mg, 33% yield) as a colorless oil.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 4.78 (s, 1H), 2.41 (s, 3H), 1.51 – 1.39 (m, 2H), 1.34 – 1.08 (m, 15H), 0.86 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.81, 140.84, 129.52, 127.10, 57.24, 43.11, 31.87, 29.61, 27.80, 23.93, 22.73, 21.58, 14.19.
HRMS (APCI) Calc. for $C_{16}H_{27}NO_2S$ [M+H] 298.1835. Found 298.1836.
FTIR (Film) cm^{-1}: 3273, 2928, 2957, 1322, 1147, 1095, 814, 664, 555.

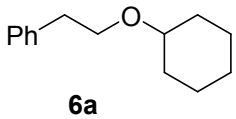
Hydroetherification and Hydrothiolation

Alcohol and Thiol Scope:



General Procedure C

In a N_2 atmosphere glovebox, a 3-dram screw-cap vial was charged with $FeCl_3$ (12.2 mg, 0.075 mmol, 0.15 equiv). The vial was capped and removed from the glovebox. The cap of the vial was removed and a magnetic stir bar, 1,2-dichloroethane (2.5 mL, 0.2 M), alcohol (0.5 mmol, 1 equiv), alkene (1.5 mmol, 3 equiv), and $AgSbF_6$ (77 mg, 0.225 mmol, 0.45 equiv) were added. The vial was capped, sealed with PTFE tape and stirred in oil bath at 40 °C for 18 hours. Upon completion, the reaction was cooled to room temperature, filtered through a silica plug with CH_2Cl_2 (10 mL), then Et_2O (40 mL) and concentrated. The crude mixture was purified by silica chromatography using hexanes and EtOAc as the eluent.

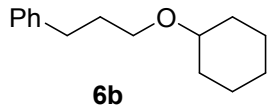


6a

(2-(Cyclohexyloxy)ethyl)benzene (6a). 2-Phenylethanol **5a** (60 µL, 0.5 mmol) and cyclohexene (151 µL, 1.5 mmol) were treated using General Procedure C. Purification

by silica chromatography (R_f = 0.63, 25% EtOAc in hexanes) yielded **6a** (53 mg, 52% yield) as a colorless oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[13]

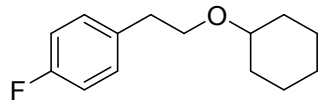
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.32 – 7.27 (m, 2H), 7.26 – 7.18 (m, 3H), 3.66 (t, *J* = 7.4 Hz, 2H), 3.29 – 3.19 (m, 1H), 2.88 (t, *J* = 7.5 Hz, 2H), 1.96 – 1.85 (m, 2H), 1.78 – 1.68 (m, 2H), 1.60 – 1.48 (m, 1H), 1.35 – 1.13 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 139.39, 129.07, 128.40, 126.21, 77.77, 69.09, 37.05, 32.45, 26.00, 24.33.



6b

(3-(Cyclohexyloxy)propyl)benzene (6b). 3-Phenyl-1-propanol **5b** (68 µL, 0.5 mmol) and cyclohexene (151 µL, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.5, 10% EtOAc in hexanes) yielded **6b** (59 mg, 54% yield) as a colorless oil. ^{1}H and ^{13}C NMR spectra match those previously reported.[14]

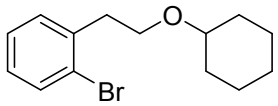
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.32 – 7.25 (m, 2H), 7.23 – 7.16 (m, 3H), 3.46 (t, *J* = 6.4 Hz, 2H), 3.26 – 3.11 (m, 1H), 2.70 (d, *J* = 7.5 Hz, 2H), 1.95 – 1.84 (m, 4H), 1.82 – 1.69 (m, 2H), 1.58 – 1.51 (m, 1H), 1.35 – 1.15 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.33, 128.63, 128.40, 125.81, 77.66, 67.09, 32.58, 32.55, 31.88, 26.03, 24.41.



6c

1-(2-(Cyclohexyloxy)ethyl)-4-fluorobenzene (6c). 4-Fluorophenylethanol **5c** (62 µL, 0.5 mmol) and cyclohexene (151 µL, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.5, 10% EtOAc in hexanes) yielded **6c** (68 mg, 61% yield) as a colorless oil.

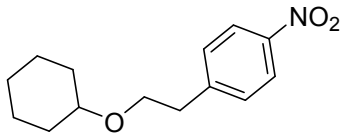
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.22 – 7.14 (m, 2H), 7.01 – 6.88 (m, 2H), 3.62 (t, *J* = 7.1 Hz, 2H), 3.28 – 3.16 (m, 1H), 2.84 (t, *J* = 7.2 Hz, 2H), 1.90 – 1.82 (m, 2H), 1.79 – 1.70 (m, 2H), 1.60 – 1.47 (m, 1H), 1.33 – 1.08 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.55, 160.61, 135.15 (d, *J* = 3.3 Hz), 130.40 (d, *J* = 7.9 Hz), 115.07 (d, *J* = 21.1 Hz), 77.76, 68.93, 36.16, 32.38, 25.96, 24.24.
^{19}F NMR ($CDCl_3$, 375 MHz): δ -117.65 – -117.80 (m).
HRMS (EI) Calc. for $C_{14}H_{19}FO$ [M+]. 222.1420. Found 222.1417.
FTIR (Film) cm^{-1}: 2930, 2955, 1601, 1509, 1221, 1097.



6d

1-Bromo-2-(2-(cyclohexyloxy)ethyl)benzene (6d). 2-Bromophenylethanol **5d** (68 µL, 0.5 mmol) and cyclohexene (151 µL, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.44, 10% EtOAc in hexanes) yielded **6d** (81.5 mg, 58% yield) as colorless oil.

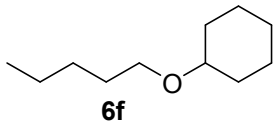
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.52 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.29 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.23 (td, *J* = 7.4, 1.2 Hz, 1H), 7.06 (td, *J* = 7.7, 1.8 Hz, 1H), 3.67 (t, *J* = 7.3 Hz, 2H), 3.31 – 3.18 (m, 1H), 3.02 (t, *J* = 7.3 Hz, 2H), 1.95 – 1.82 (m, 2H), 1.80 – 1.64 (m, 2H), 1.58 – 1.48 (m, 1H), 1.33 – 1.16 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 138.61, 132.79, 131.26, 127.96, 127.39, 124.77, 77.66, 67.02, 37.16, 32.40, 25.97, 24.24.
HRMS (APCI) Calc. for $C_{14}H_{19}BrO$ [M+H] 283.0692. Found 283.0692.
FTIR (Film) cm^{-1}: 2928, 2854, 1472, 1442, 1099, 1038, 749.



6e

1-(2-(Cyclohexyloxy)ethyl)-4-nitrobenzene (6e). 4-Nitrophenylethanol **5e** (95 mg, 0.57 mmol) and cyclohexene (173 µL, 1.7 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.57, 25% EtOAc in hexanes) yielded **6e** (103 mg, 73% yield) as a yellow oil.

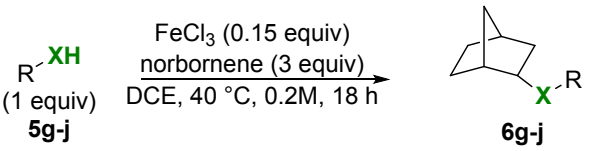
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.11 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 3.67 (t, *J* = 6.6 Hz, 2H), 3.27 – 3.13 (m, 1H), 2.94 (t, *J* = 6.6 Hz, 2H), 2.00 – 1.75 (m, 2H), 1.73 – 1.61 (m, 2H), 1.55 – 1.40 (m, 1H), 1.33 – 1.04 (m, 5H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.78, 146.57, 129.84, 123.44, 77.76, 67.72, 36.72, 32.19, 25.83, 24.05.
HRMS (ESI) Calc. for $C_{14}H_{19}NO_3$ [M+Na] 272.1257. Found 272.1258.
FTIR (Film) cm^{-1}: 2930, 2854, 1600, 1515, 1341, 1099, 854, 697.



6f

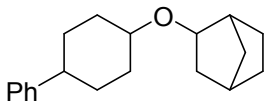
(Pentyloxy)cyclohexane (6f). 1-Pentanol **5f** (78 µL, 0.72 mmol) and cyclohexene (218 µL, 2.16 mmol) were treated using General Procedure C. This yielded **6f** in 49% GC yield using dodecane as internal standard.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.40 (t, *J* = 6.8 Hz, 2H), 3.22 – 3.07 (m, 1H), 1.88 (dt, *J* = 7.3, 4.2 Hz, 2H), 1.79 – 1.64 (m, 2H), 1.52 (tdd, *J* = 9.7, 8.0, 5.5 Hz, 3H), 1.36 – 1.07 (m, 9H), 0.95 – 0.80 (m, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 77.50, 68.04, 32.50, 30.08, 28.57, 26.01, 24.38, 22.69, 14.15.
HRMS (APCI) Calc. for $C_{11}H_{22}O$ [M+H] 171.1743. Found 171.1744.
FTIR (Film) cm^{-1}: 2929, 2855, 1450, 1363, 1104.



General Procedure D

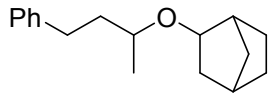
In a N_2 atmosphere glovebox, a 3-dram screw-cap vial was charged with $FeCl_3$ (12.2 mg, 0.075 mmol, 0.15 equiv). The vial was capped and removed from the glovebox. The cap of the vial was removed and a magnetic stir bar, 1,2-dichloroethane (2.5 mL, 0.2 M), alcohol or thiol (0.5 mmol, 1 equiv), and alkene (1.5 mmol, 3 equiv) were added. The vial was capped, sealed with PTFE tape and stirred in oil bath at 40 °C for 18 hours. Upon completion, the reaction was cooled to room temperature, filtered through a silica plug with CH_2Cl_2 (10 mL), then Et_2O (40 mL) and concentrated. The crude mixture was purified by silica chromatography using hexanes and EtOAc as the eluent.



6g

2-((4-Phenylcyclohexyl)oxy)bicyclo[2.2.1]heptane (6g). 4-Phenylcyclohexanol **5g** (88 mg, 0.5 mmol) and norbornene (141 mg, 1.5 mmol) were treated using General Procedure D. Purification by silica chromatography (R_f = 0.47, 10% EtOAc in hexanes) yielded **6g** (113 mg, 84% yield) as a colorless solid.

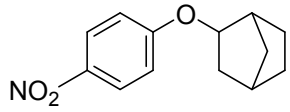
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.23 – 7.15 (m, 3H), 3.54 (dt, *J* = 6.9, 1.7 Hz, 1H), 3.34 (tt, *J* = 10.8, 4.2 Hz, 1H), 2.50 (tt, *J* = 12.1, 3.5 Hz, 1H), 2.32 – 2.20 (m, 2H), 2.14 – 2.08 (m, 2H), 1.94 (dq, *J* = 13.2, 3.1 Hz, 2H), 1.65 – 1.33 (m, 9H), 1.13 – 0.94 (m, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.01, 128.45, 126.90, 126.11, 79.81, 75.48, 43.91, 41.43, 40.49, 35.39, 34.97, 33.68, 33.39, 32.94, 32.88, 28.76, 24.94.
HRMS (APCI) Calc. for $C_{19}H_{26}O$ [M+H] 271.2056. Found 271.2057.
FTIR (Film) cm^{-1}: 2931, 2868, 1450, 1093, 699.



6h

2-((4-Phenylbutan-2-yl)oxy)bicyclo[2.2.1]heptane (6h). 4-Phenyl-2-butanol **5h** (35 μL, 0.22 mmol) and norbornene (62 mg, 66 mmol) were treated using General Procedure D. Purification by silica chromatography (R_f = 0.47, 10% EtOAc in hexanes) yielded a mixture of diastereomers **6h** (13 mg, 24% yield) as a colorless oil.

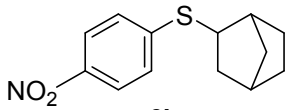
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.31 – 7.25 (m, 2H), 7.23 – 7.15 (m, 3H), 3.52 – 3.39 (m, 2H), 2.83 – 2.68 (m, 1H), 2.67 – 2.55 (m, 1H), 2.28 – 2.19 (m, 2H), 1.85 – 1.75 (m, 1H), 1.74 – 1.64 (m, 1H), 1.63 – 1.34 (m, 4H), 1.15 (dd, *J* = 11.8, 6.1 Hz, 3H), 1.13 – 0.83 (m, 4H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.79, 142.72, 128.53, 128.51, 128.42, 125.76, 80.21, 79.73, 72.32, 71.98, 42.00, 40.69, 40.50, 40.08, 39.07, 39.02, 35.41, 35.27, 35.10, 34.98, 32.35, 32.25, 28.79, 24.96, 24.84, 20.75, 20.43.
HRMS (APCI) Calc. for $C_{17}H_{24}O$ [M+H] 245.1900. Found 245.1900.
FTIR (Film) cm^{-1}: 2953, 2869, 1453, 1340, 1088, 698.



6i

(4-Nitrophenoxy)bicyclo[2.2.1]heptane (6i). 4-Nitrophenol **5i** (83 mg, 0.6 mmol) and norbornene (169 mg, 1.8 mmol) were treated using General Procedure D. Purification by silica chromatography (R_f = 0.46, 25% EtOAc in hexanes) yielded **6i** (103 mg, 74% yield) as an off-white solid.

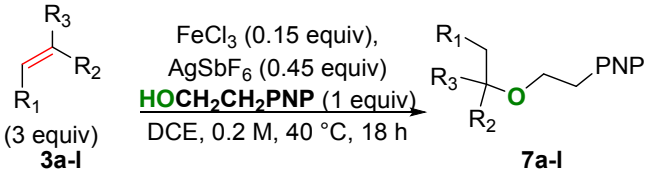
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.15 (d, *J* = 9.2 Hz, 2H), 6.87 (d, *J* = 9.3 Hz, 2H), 4.32 – 4.03 (m, 1H), 2.46 (d, *J* = 4.9 Hz, 1H), 2.37 – 2.30 (m, 1H), 1.86 – 1.74 (m, 1H), 1.73 – 1.44 (m, 5H), 1.27 – 1.11 (m, 4H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 163.19, 141.13, 125.94, 115.32, 81.34, 41.30, 40.01, 35.58, 35.35, 28.40, 24.25.
HRMS (ESI) Calc. for $C_{13}H_{15}NO_3$ [M+H] 234.1125. Found 234.1125.
FTIR (Film) cm^{-1}: 2959, 2873, 1592, 1511, 1340, 1259, 1111, 993, 844.

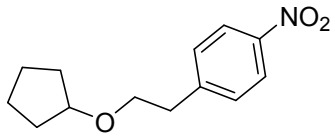


6j

Bicyclo[2.2.1]heptan-2-yl(4-nitrophenyl)sulfane (6j). 4-Nitrothiophenol **5j** (77.5 mg, 0.5 mmol) and norbornene (141 mg, 1.5 mmol) were treated using General Procedure D. Purification by silica chromatography (R_f = 0.5, 25% EtOAc in hexanes) yielded **6j** (39 mg, 31% yield) as an off-white solid.
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.11 (d, *J* = 8.9 Hz, 2H), 7.28 (d, *J* = 8.9 Hz, 2H), 3.34 – 3.23 (m, 1H), 2.40 – 2.29 (m, 2H), 1.95 – 1.86 (m, 1H), 1.77 – 1.54 (m, 3H), 1.53 – 1.42 (m, 1H), 1.40 – 1.31 (m, 1H), 1.31 – 1.19 (m, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 149.02, 144.80, 126.24, 124.03, 46.83, 42.41, 38.58, 36.53, 36.12, 29.02, 28.66.
HRMS (EI) Calc. for $C_{13}H_{15}NO_2S$ [M+] 249.0823. Found 249.0819.
FTIR (Film) cm^{-1}: 2955, 2870, 1576, 1508, 1336, 1093, 852, 742.

Alkene Scope:

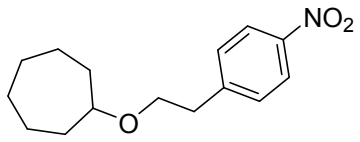




7a

1-(2-(Cyclopentyloxy)ethyl)-4-nitrobenzene (7a). 4-Nitrophenylethanol **5e** (83 mg, 0.5 mmol) and cyclopentene **3a** (133 µL, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.54, 25% EtOAc in hexanes) yielded **7a** (73 mg, 62% yield) as a yellow oil.

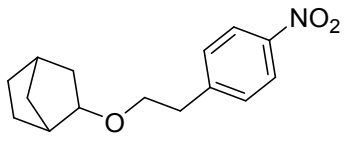
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.12 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 3.86 (dd, *J* = 6.2, 3.1 Hz, 1H), 3.61 (t, *J* = 6.6 Hz, 2H), 2.93 (t, *J* = 6.6 Hz, 2H), 1.82 – 1.36 (m, 6H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.76, 146.62, 129.84, 123.49, 81.66, 68.59, 36.57, 32.29, 23.56.
HRMS (ESI) Calc. for $C_{13}H_{17}NO_3$ [M+Na] 258.1101. Found 258.1101
FTIR (Film) cm^{-1}: 2953, 2868, 1600, 1515, 1342, 1342, 1093, 852, 697.



7b

(4-Nitrophenethoxy)cycloheptane (7b). 4-Nitrophenylethanol **5e** (83 mg, 0.5 mmol) and cycloheptene **3b** (175 μL, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.57, 25% EtOAc in hexanes) yielded **7b** (50 mg, 38% yield) as a yellow oil.

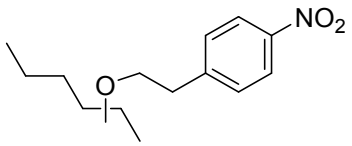
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.13 (d, *J* = 8.3 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 3.64 (t, *J* = 6.6 Hz, 2H), 3.49 – 3.31 (m, 1H), 2.94 (t, *J* = 6.6 Hz, 2H), 1.90 – 1.72 (m, 2H), 1.71 – 1.42 (m, 8H), 1.43 – 1.28 (m, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.86, 146.64, 129.90, 123.51, 80.47, 68.22, 36.79, 33.90, 28.45, 22.99.
HRMS (ESI) Calc. for $C_{15}H_{21}NO_3$ [M+Na] 286.1414. Found 286.1414.
FTIR (Film) cm^{-1}: 2923, 2854, 1600, 1516, 1341, 1090, 854, 698, 617.



7c

2-(4-Nitrophenethoxy)bicyclo[2.2.1]heptane (7c). 4-Nitrophenylethanol **5e** (83 mg, 0.5 mmol) and norbornene **3c** (147 mg, 1.5 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.52, 25% EtOAc in hexanes) yielded **7c** (106 mg, 81% yield) as a yellow oil.

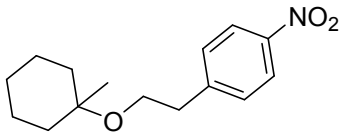
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.12 (d, *J* = 8.6 Hz, 2H), 7.38 (d, *J* = 8.5 Hz, 2H), 3.73 – 3.51 (m, 2H), 3.34 – 3.25 (m, 1H), 2.93 (t, *J* = 6.6 Hz, 2H), 2.31 – 2.10 (m, 2H), 1.59 – 1.33 (m, 4H), 1.33 – 1.20 (m, 1H), 1.01 (m, 2H), 0.97 – 0.88 (m, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.80, 146.62, 129.88, 123.49, 82.85, 68.10, 40.38, 39.58, 36.58, 35.18, 34.87, 28.62, 24.61.
HRMS (ESI) Calc. for $C_{15}H_{19}NO_3$ [M+Na] 284.1257. Found 284.1258.
FTIR (Film) cm^{-1}: 2952, 2869, 1600, 1516, 1342, 1096, 852, 697, 618.



7d

1-(2-(Hexan-2-yloxy)ethyl)-4-nitrobenzene (7d). 4-Nitrophenylethanol **5e** (96 mg, 0.57 mmol) and 1-hexene **3d** (212 µL, 1.71 mmol) were treated using General Procedure C. Purification by silica chromatography (R_f = 0.57, 25% EtOAc in hexanes) yielded a mixture of structural isomers **7d** (105 mg, 72% yield, 2.7:1 ratio or 2&3 substituted product) as a yellow oil.

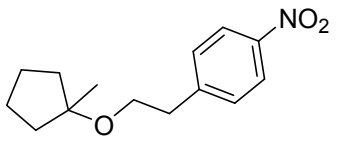
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.13 (d, *J* = 8.6 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 3.79 – 3.70 (m, 1H, major), 3.70 – 3.62 (m, 2H, minor), 3.62 – 3.53 (m, 1H, major), 3.39 – 3.28 (m, 1H), 3.20 – 3.12 (m, 1H), 2.95 (t, *J* = 6.6 Hz, 2H), 1.54 – 1.12 (m, 12H), 1.09 (d, *J* = 6.1 Hz, 3H, major), 0.90 – 0.79 (m, 9H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.78, 146.62, 129.87, 123.48, 123.46, 80.98 (minor), 75.96 (major), 68.73 (minor), 68.29 (major), 36.81 (minor), 36.73 (major), 36.38 (major), 35.73 (minor), 27.78 (major), 26.41 (minor), 22.81 (major), 19.66 (major), 18.68 (minor), 14.31 (minor), 14.15 (major) , 9.55 (minor).
HRMS (ESI) Calc. for $C_{14}H_{21}NO_3$ [M+Na] 274.1414. Found 274.1415.
FTIR (Film) cm^{-1}: 2930, 2959, 1601, 1517, 1342, 1089, 854, 697, 617.



7f

1-(2-((1-Methylcyclohexyl)oxy)ethyl)-4-nitrobenzene (7f). 4-Nitrophenylethanol **5e** (125 mg, 0.75 mmol) and 1-methylcyclohexene **3f** (269 µL, 2.25 mmol) were treated using General Procedure D. Purification by silica chromatography (R_f = 0.57, 25% EtOAc in hexanes) yielded **7f** (66 mg, 33% yield) as a yellow oil.

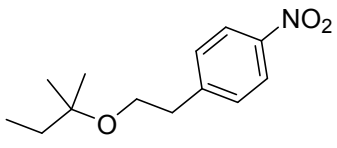
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.12 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 3.53 (t, *J* = 6.5 Hz, 2H), 2.92 (t, *J* = 6.5 Hz, 2H), 1.77 – 1.57 (m, 2H), 1.48 – 1.13 (m, 6H), 1.04 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.31, 146.59, 130.08, 123.39, 73.65, 60.73, 37.26, 36.47, 25.76, 24.78, 22.12.
HRMS (ESI) Calc. for $C_{15}H_{21}NO_3$ [M+Na] 286.1414. Found 286.1415.
FTIR (Film) cm^{-1}: 2927, 2857, 1601, 1516, 1342, 1081, 845, 696, 617.



7g

1-(2-((1-Methylcyclopentyl)oxy)ethyl)-4-nitrobenzene (7g). 4-Nitrophenylethanol **5e** (84 mg, 0.5 mmol) and 1-methylcyclopentene **3g** (128 μL, 1.5 mmol) were treated using General Procedure D at room temperature. Purification by silica chromatography (R_f = 0.53, 25% EtOAc in hexanes) yielded **7g** (22.6 mg, 18% yield) as a yellow oil.

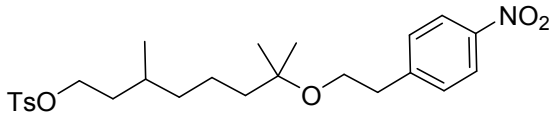
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.13 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 3.55 (t, *J* = 6.5 Hz, 2H), 2.90 (t, *J* = 6.5 Hz, 2H), 1.79 – 1.62 (m, 2H), 1.65 – 1.49 (m, 4H), 1.44 – 1.32 (m, 2H), 1.20 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.24, 146.74, 130.11, 123.56, 84.64, 62.66, 38.16, 37.36, 24.02, 23.51.
HRMS (ESI) Calc. for $C_{14}H_{19}NO_3$ [M+Na] 272.1257. Found 272.1258.
FTIR (Film) cm^{-1}: 2960, 2868, 1600, 1516, 1343, 1081, 854, 749, 697.



7g

1-Nitro-4-(2-(*tert*-pentyloxy)ethyl)benzene (7g). 4-Nitrophenylethanol **5e** (84 mg, 0.5 mmol) and 2-methyl-2-butene **3g** (159 μL, 1.5 mmol) were treated using General Procedure D at room temperature. Purification by silica chromatography (R_f = 0.53, 25% EtOAc in hexanes) yielded **7g** (47.4 mg, 40 % yield) as a yellow oil.

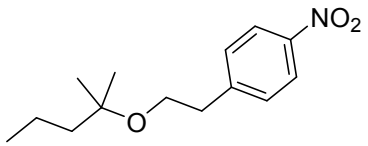
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.13 (d, *J* = 8.7 Hz, 2H), 7.39 (d, *J* = 8.6 Hz, 2H), 3.54 (t, *J* = 6.6 Hz, 2H), 2.90 (t, *J* = 6.6 Hz, 2H), 1.44 (q, *J* = 7.5 Hz, 2H), 1.06 (s, 6H), 0.78 (t, *J* = 7.5 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.11, 146.63, 130.03, 123.44, 75.13, 61.47, 37.24, 32.88, 25.00, 8.28.
HRMS (ESI) Calc. for $C_{13}H_{19}NO_3$ [M+Na] 260.1257. Found 260.1258.
FTIR (Neat) cm^{-1}: 2970, 2928, 2875, 1601, 1517, 1342, 1085, 849, 749, 697.



7i

3,7-Dimethyl-7-(4-nitrophenethoxy)octyl 4-methylbenzenesulfonate (7i). 4-Nitrophenylethanol **5e** (95 mg, 0.57 mmol) and 3,7-dimethyloct-6-en-1-yl 4-methylbenzenesulfonate **3i** (177 mg, 0.57 mmol) were treated using General Procedure D at room temperature. Purification by silica chromatography (R_f = 0.31, 25% EtOAc in hexanes) yielded **7i** (84.2 mg, 31% yield) as a yellow oil.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.10 (d, *J* = 8.3 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 4.12 – 3.90 (m, 2H), 3.52 (t, *J* = 6.5 Hz, 2H), 2.88 (t, *J* = 6.5 Hz, 2H), 2.42 (s, 3H), 1.78 – 1.55 (m, 1H), 1.56 – 0.91 (m, 14H), 0.76 (d, *J* = 6.6 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.08, 146.53, 144.73, 133.25, 129.99, 129.88, 127.90, 123.35, 74.78, 69.08, 61.44, 40.61, 37.13, 37.11, 35.77, 29.19, 25.41, 25.37, 21.68, 20.96, 19.08.
HRMS (ESI) Calc. for $C_{25}H_{35}NO_6S$ [M+Na] 500.2077. Found 500.2079.
FTIR (Film) cm^{-1}: 2966, 2933, 2867, 1599, 1517, 1344, 1175, 1095, 841, 749, 698.



7l

1-(2-((2-Methylpentan-2-yl)oxy)ethyl)-4-nitrobenzene (7l). 4-Nitrophenylethanol **5e** (87 mg, 0.52 mmol) and 2-methyl-2-pentene **3l** (190 µL, 1.56 mmol) were treated using General Procedure D at room temperature. Purification by silica chromatography (R_f = 0.56, 25% EtOAc in hexanes) yielded **7l** (79.6 mg, 63% yield) as a yellow oil.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.13 (d, *J* = 8.6 Hz, 2H), 7.38 (d, *J* = 8.6 Hz, 2H), 3.55 (t, *J* = 6.6 Hz, 2H), 2.90 (t, *J* = 6.6 Hz, 2H), 1.43 – 1.33 (m, 2H), 1.31 – 1.17 (m, 2H), 1.07 (s, 6H), 0.86 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.12, 146.65, 130.03, 123.47, 75.05, 61.52, 42.97, 37.26, 25.48, 17.23, 14.78.
HRMS (ESI) Calc. for $C_{14}H_{21}NO_3$ [M+H] 274.1414. Found 274.1415.
FTIR (Film) cm^{-1}: 2959, 2932, 2871, 1601, 1517, 1343, 1081, 850, 749, 697.

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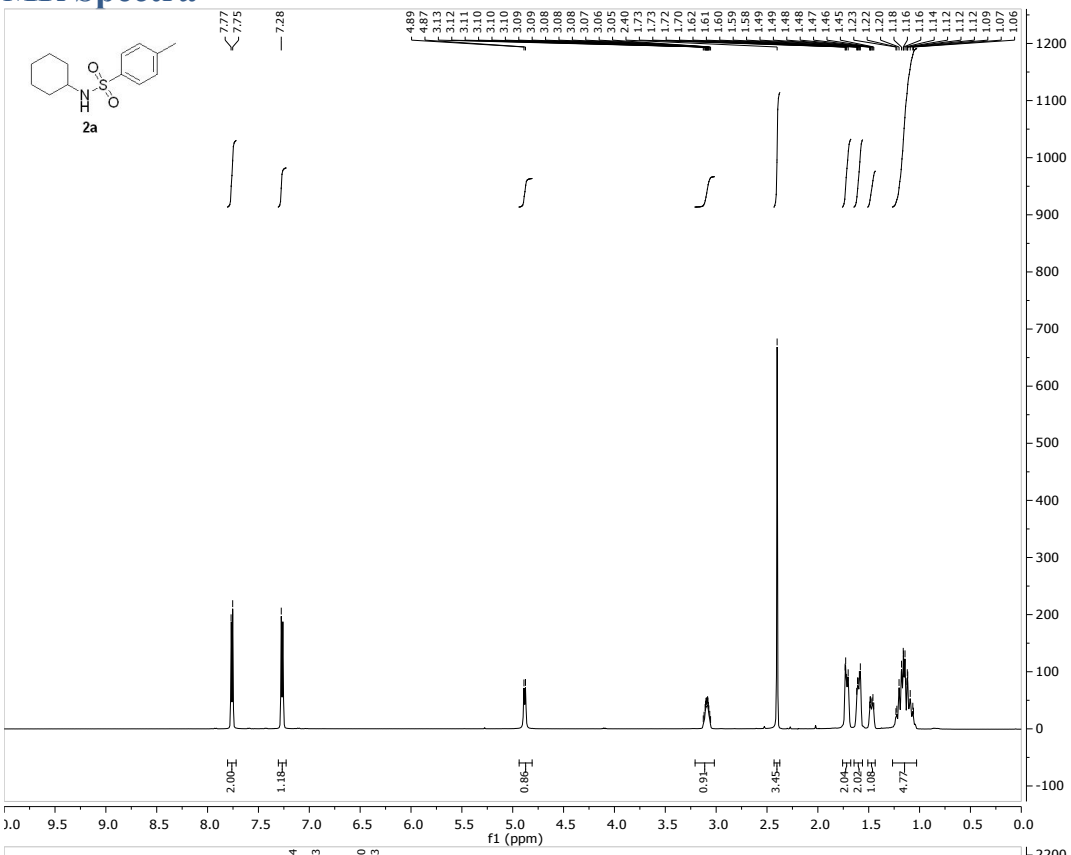
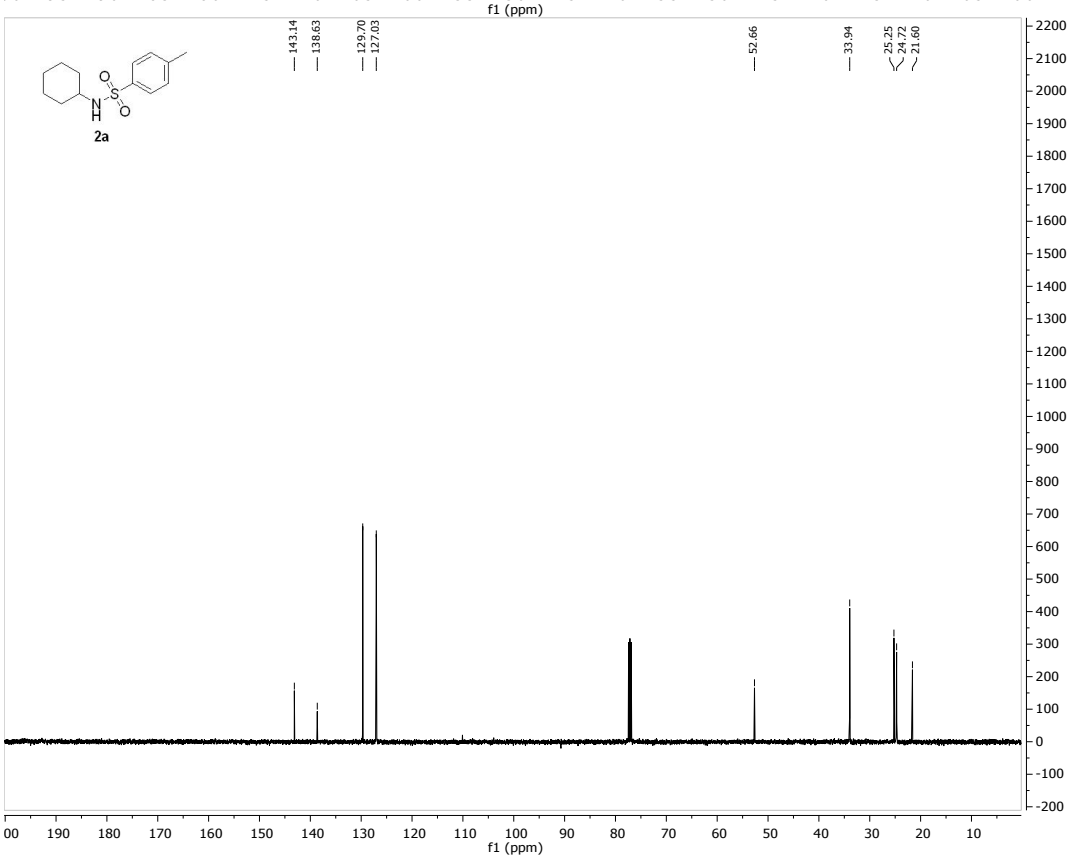
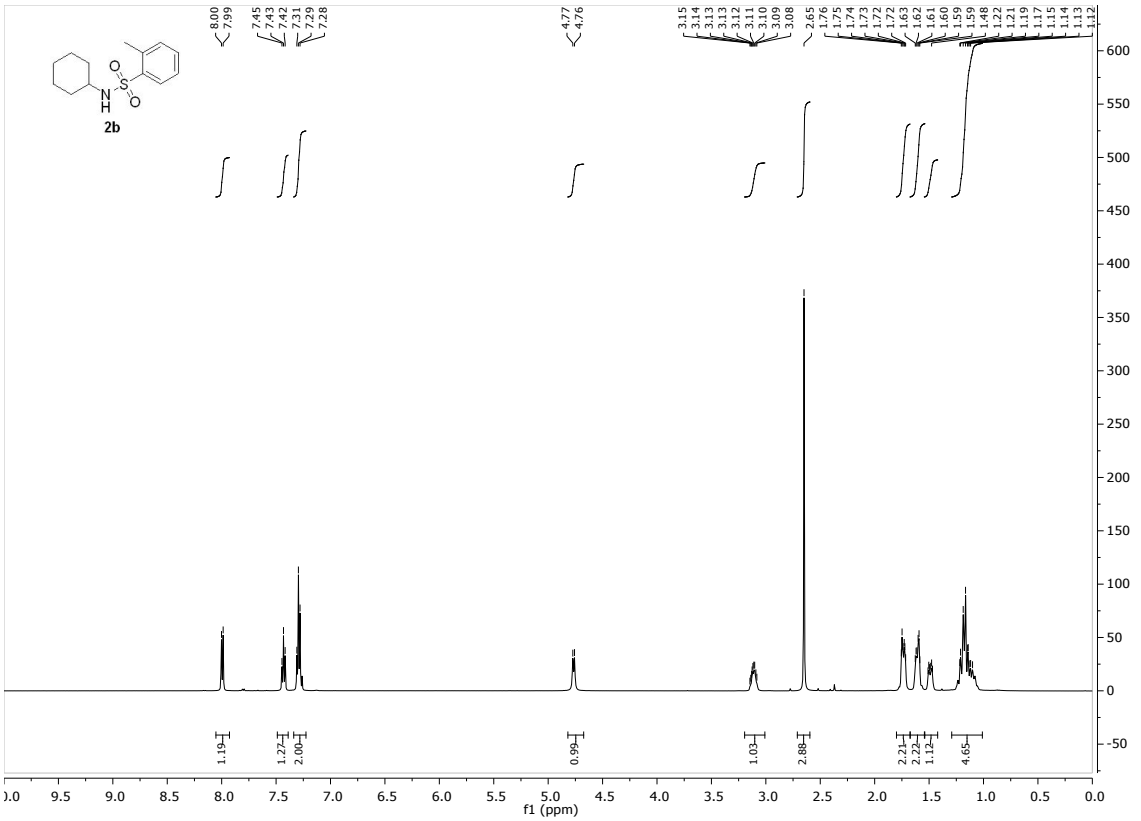
NMR Spectra



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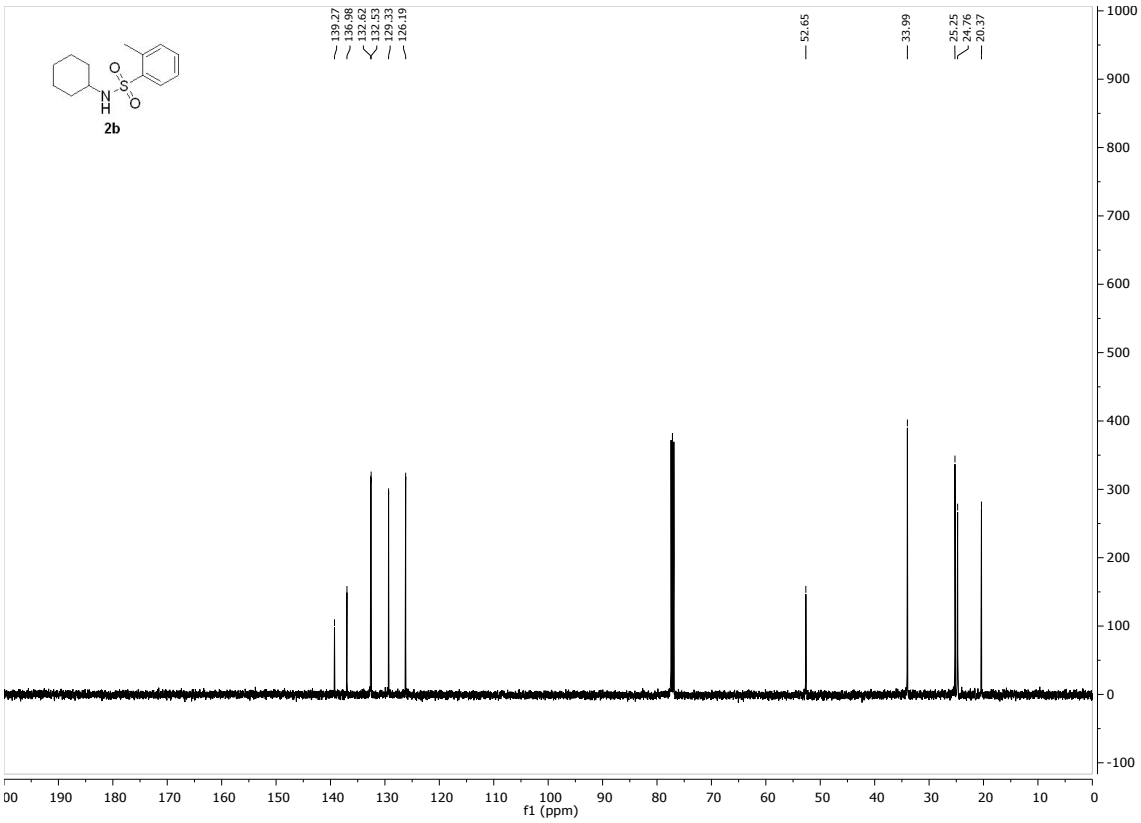
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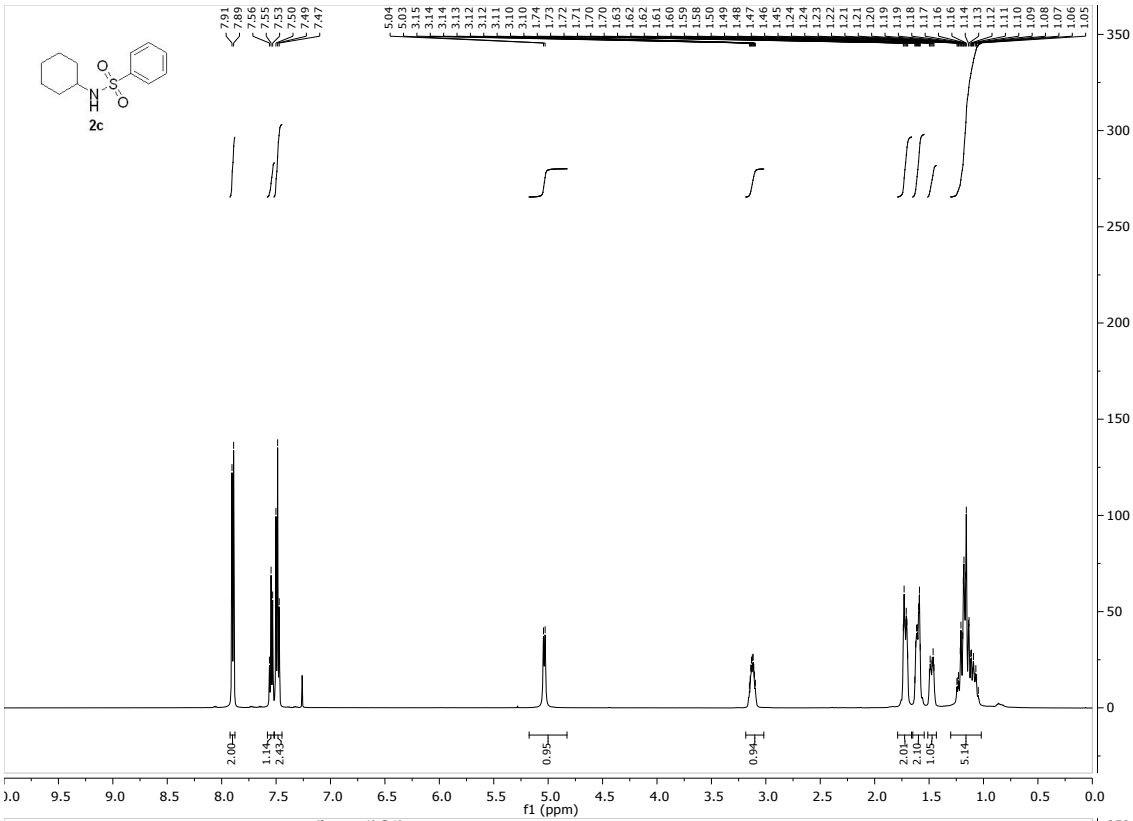
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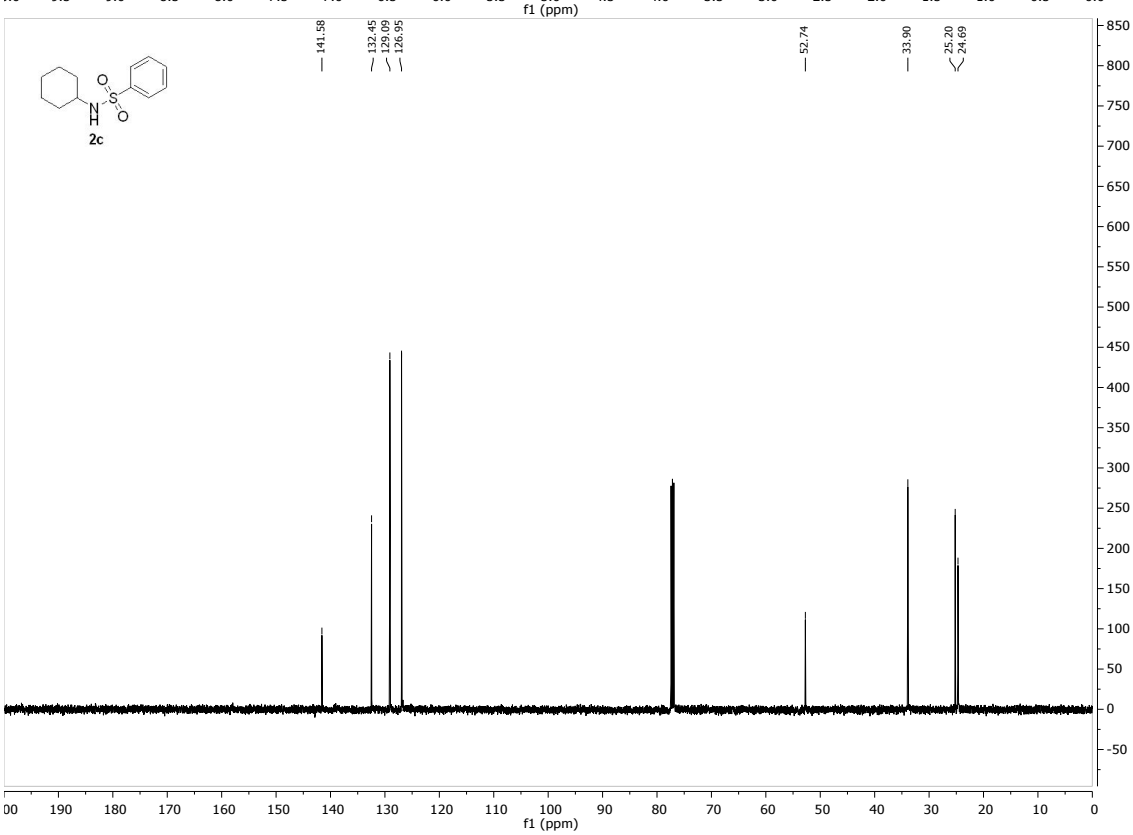
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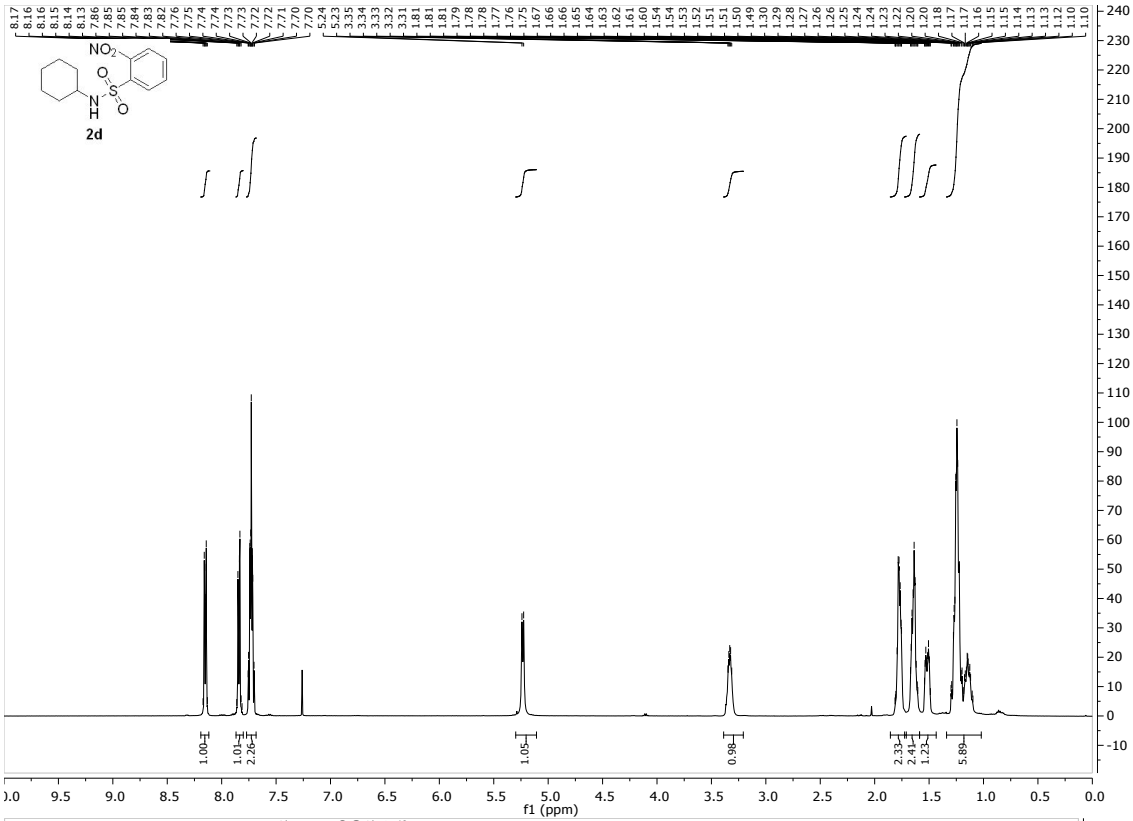
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S
O
N
H
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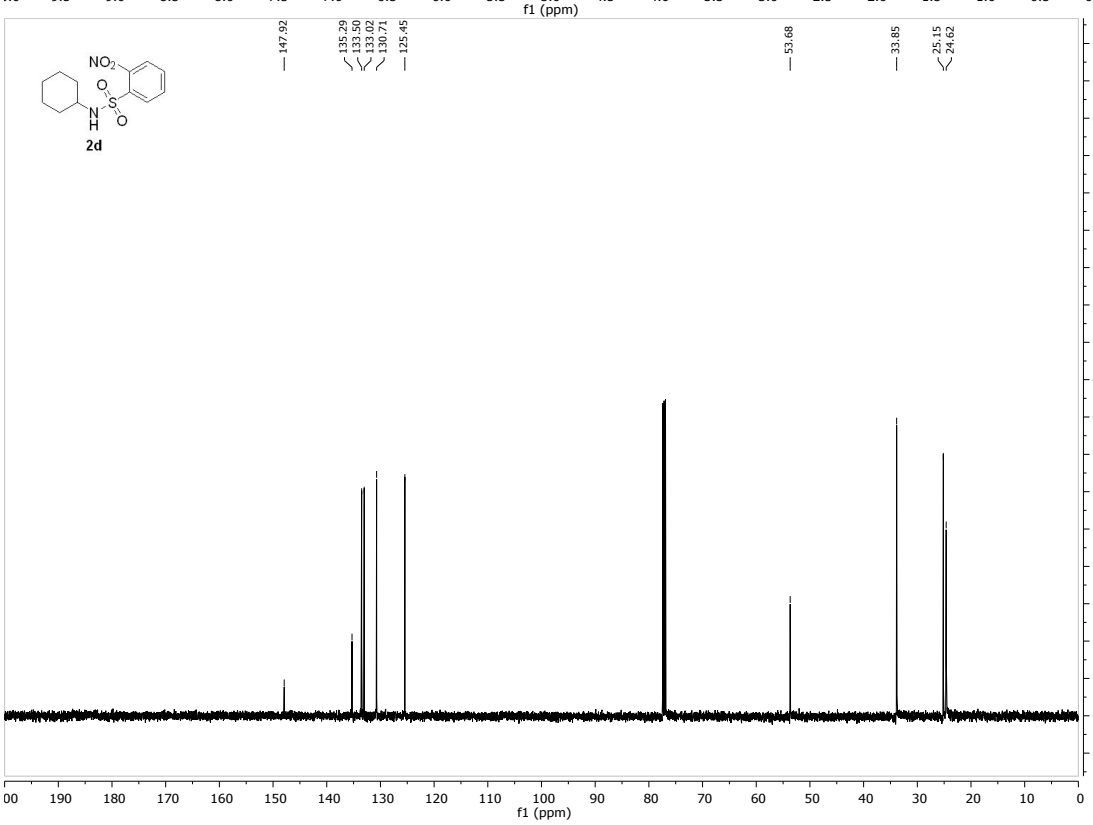
f1 (ppm)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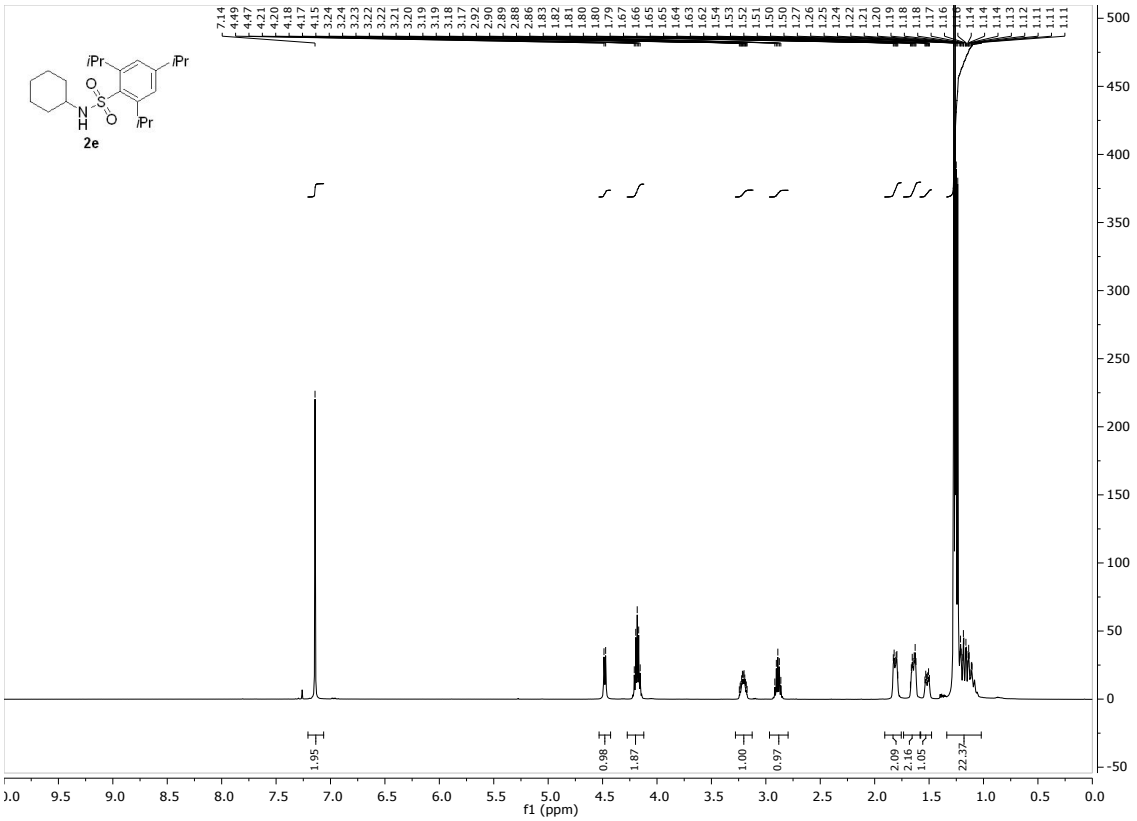
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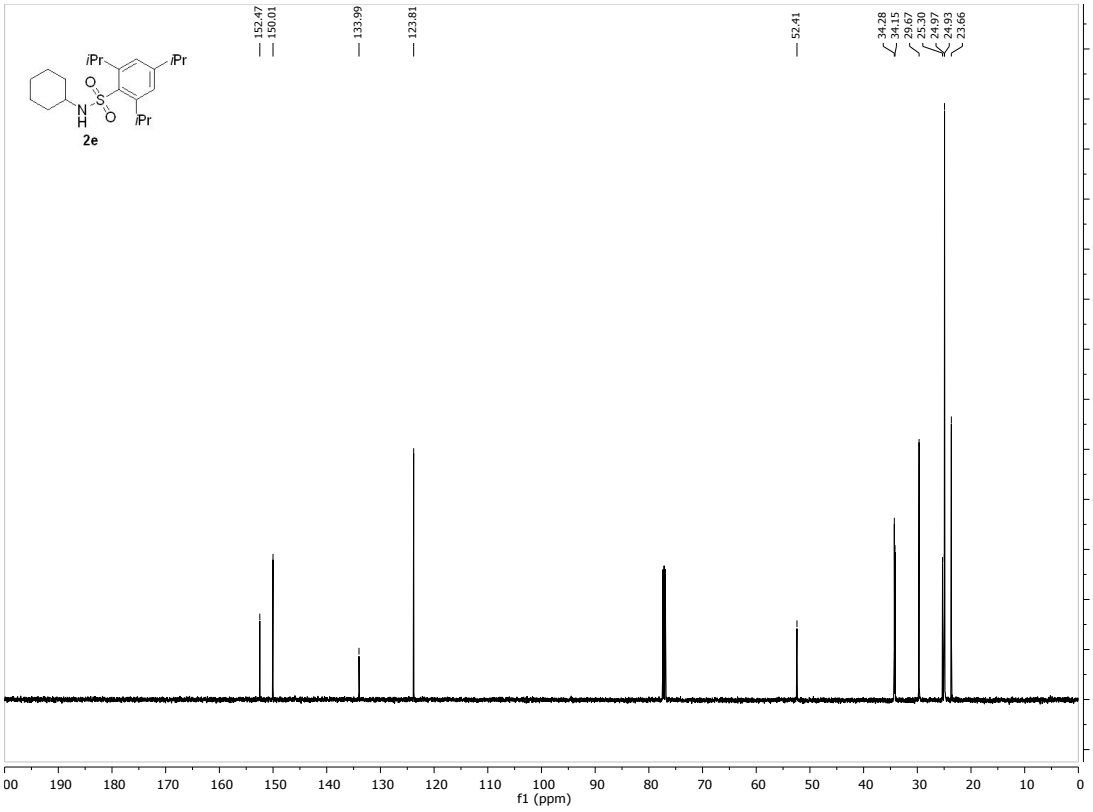
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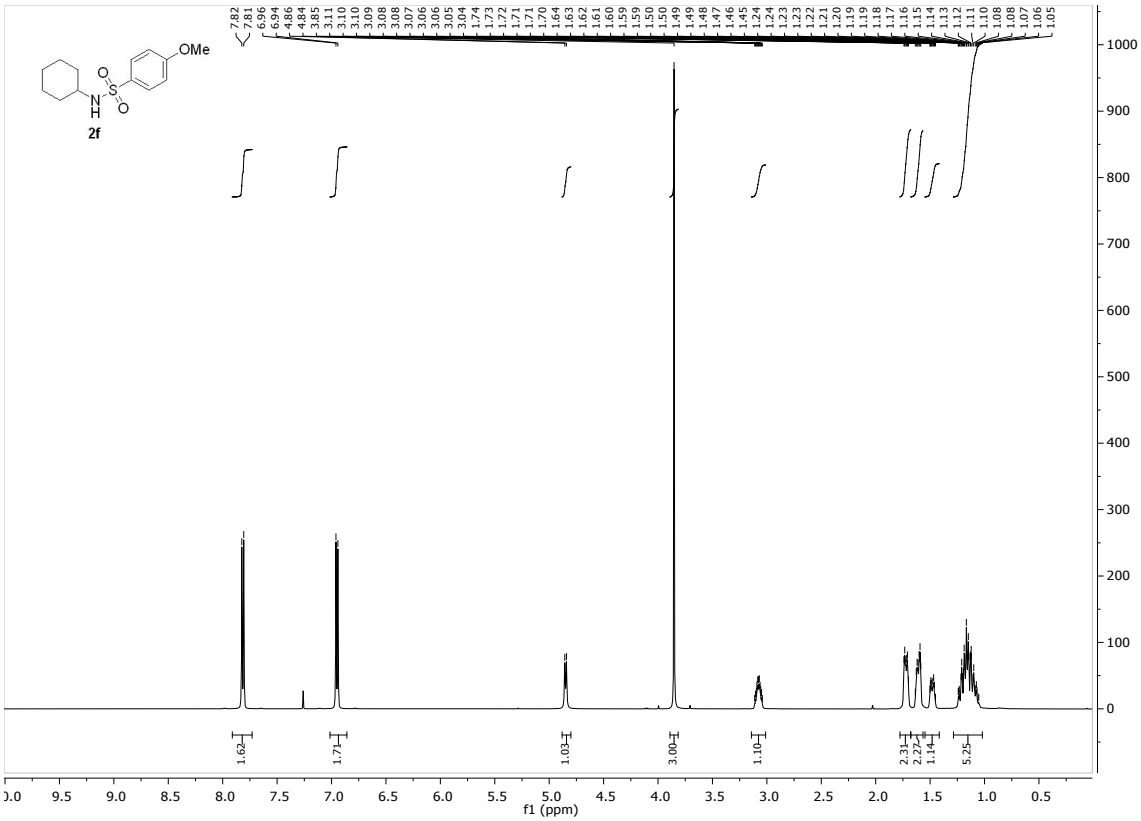
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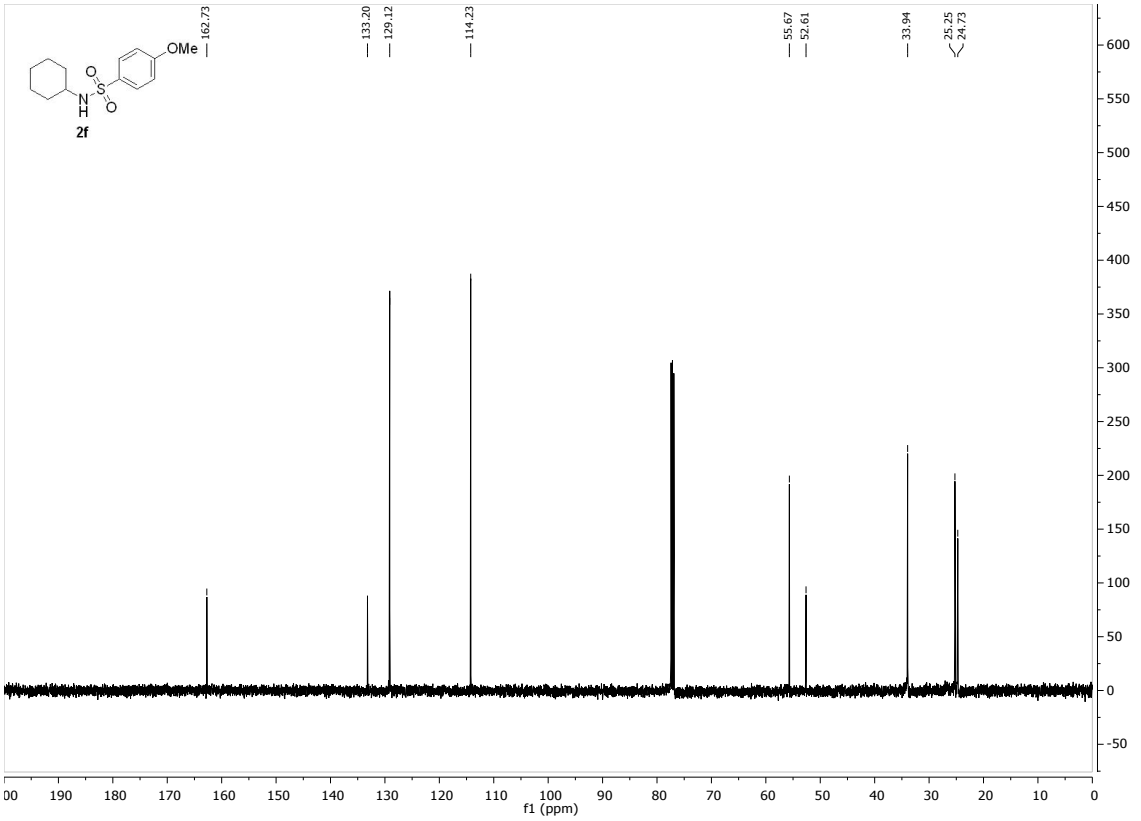
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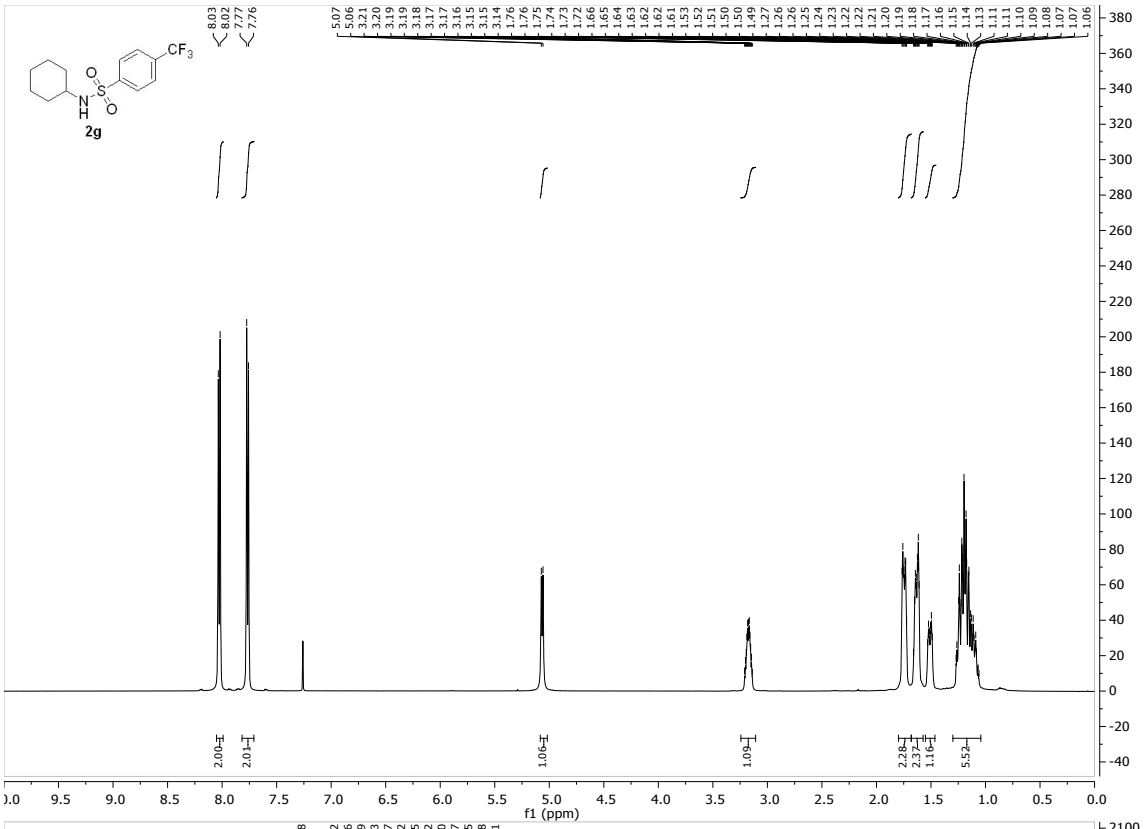
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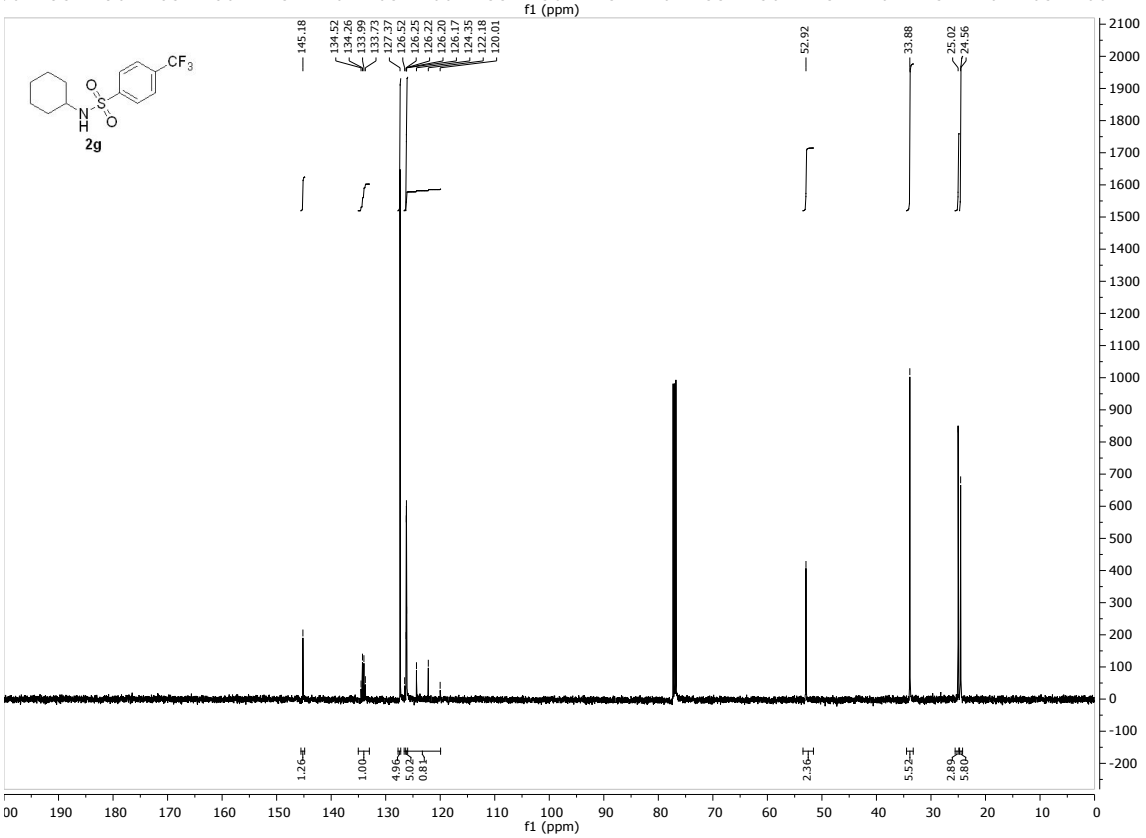
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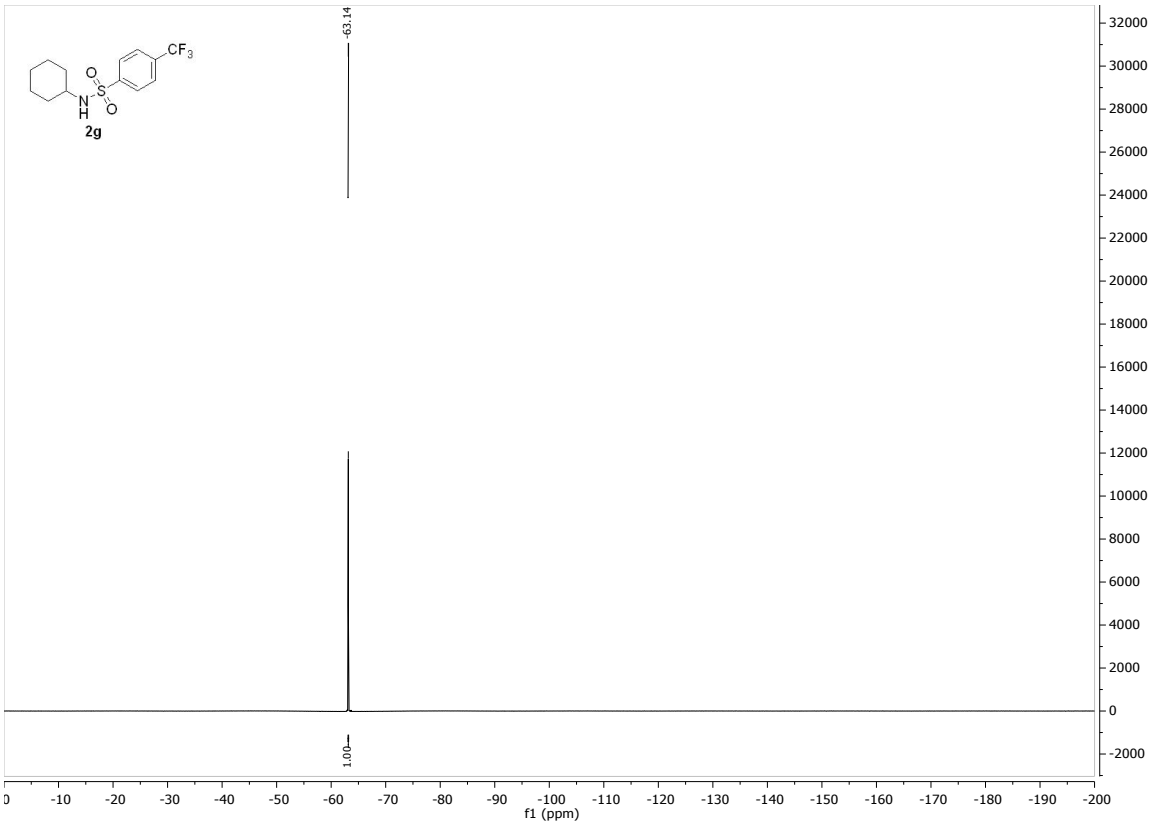
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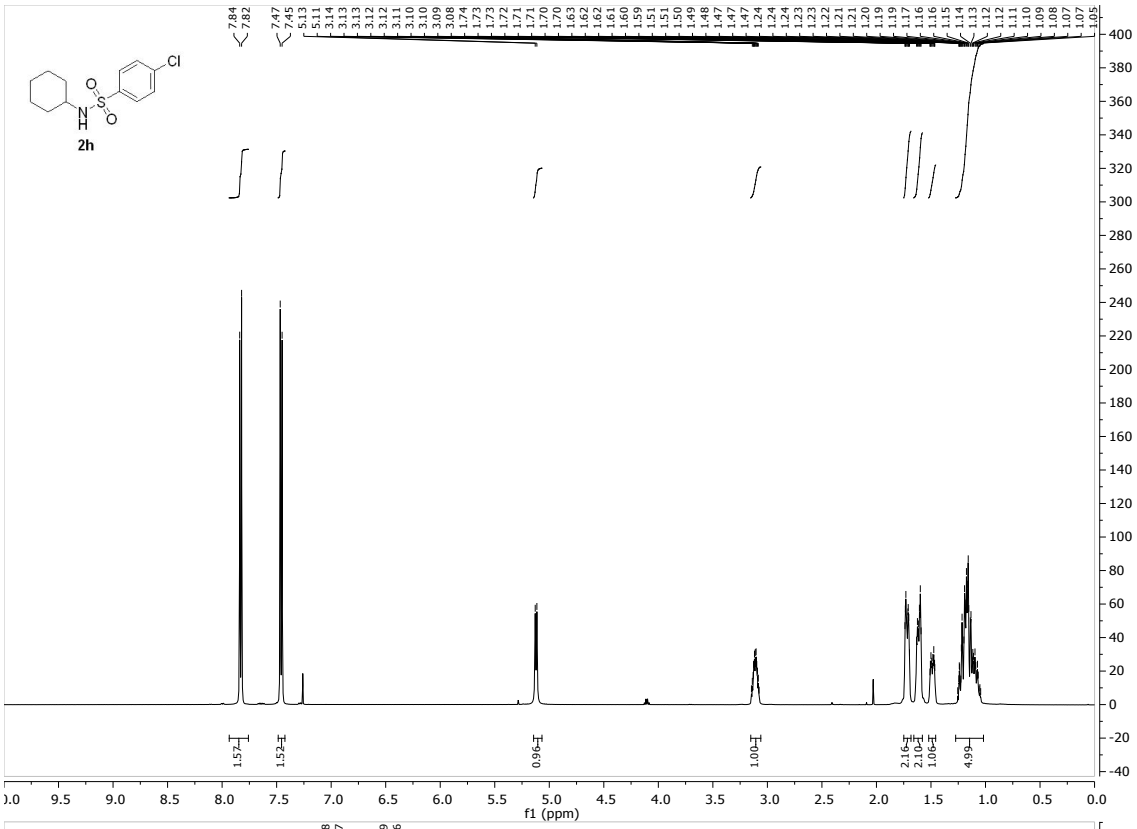
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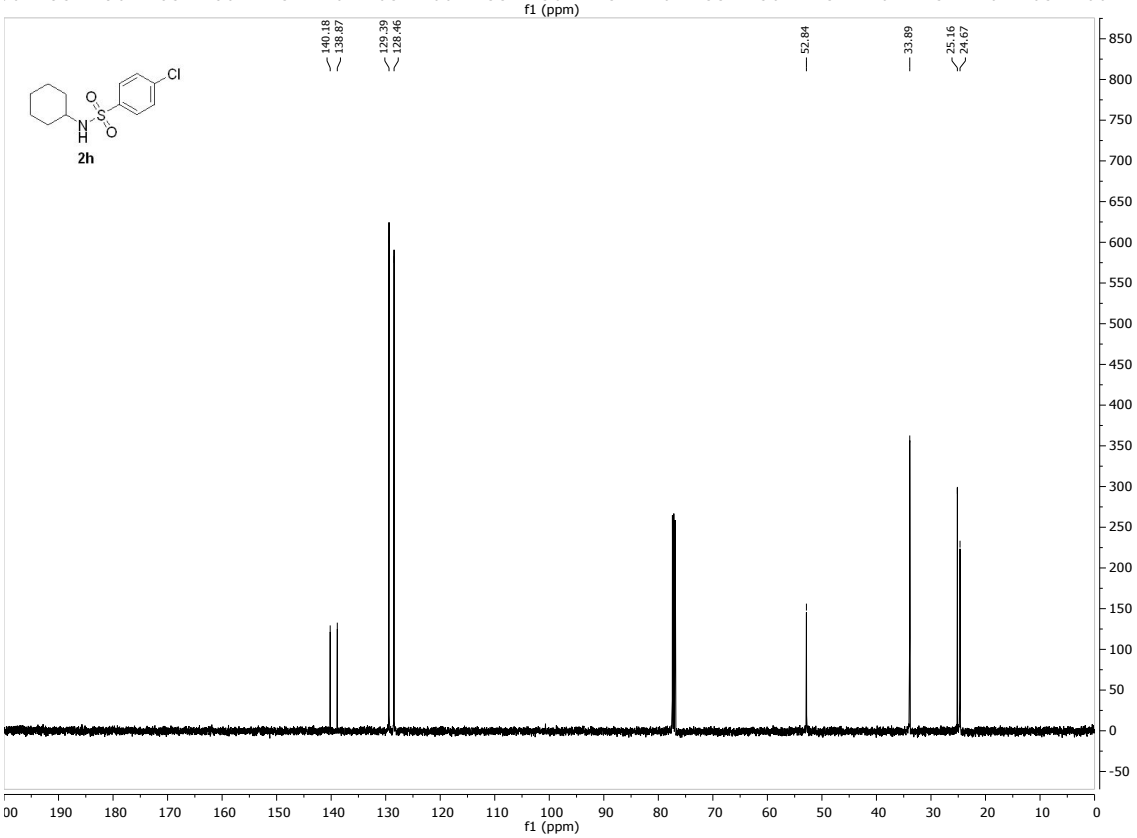
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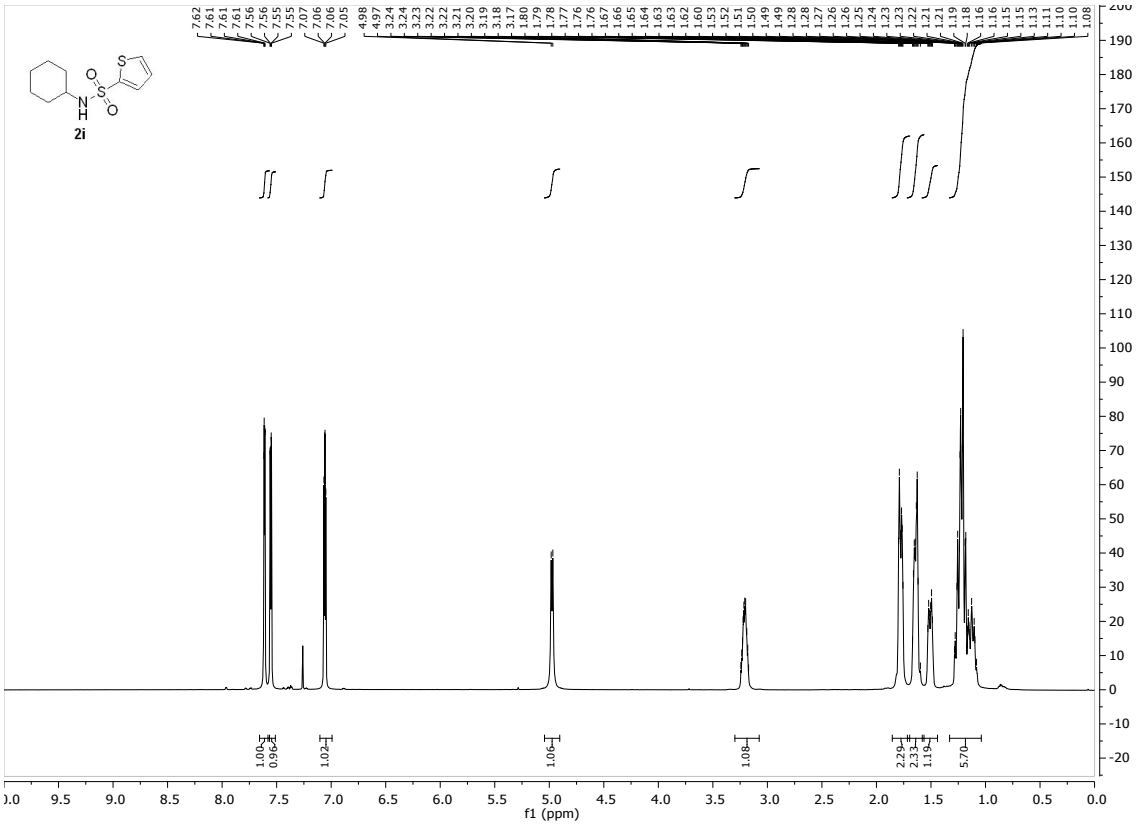
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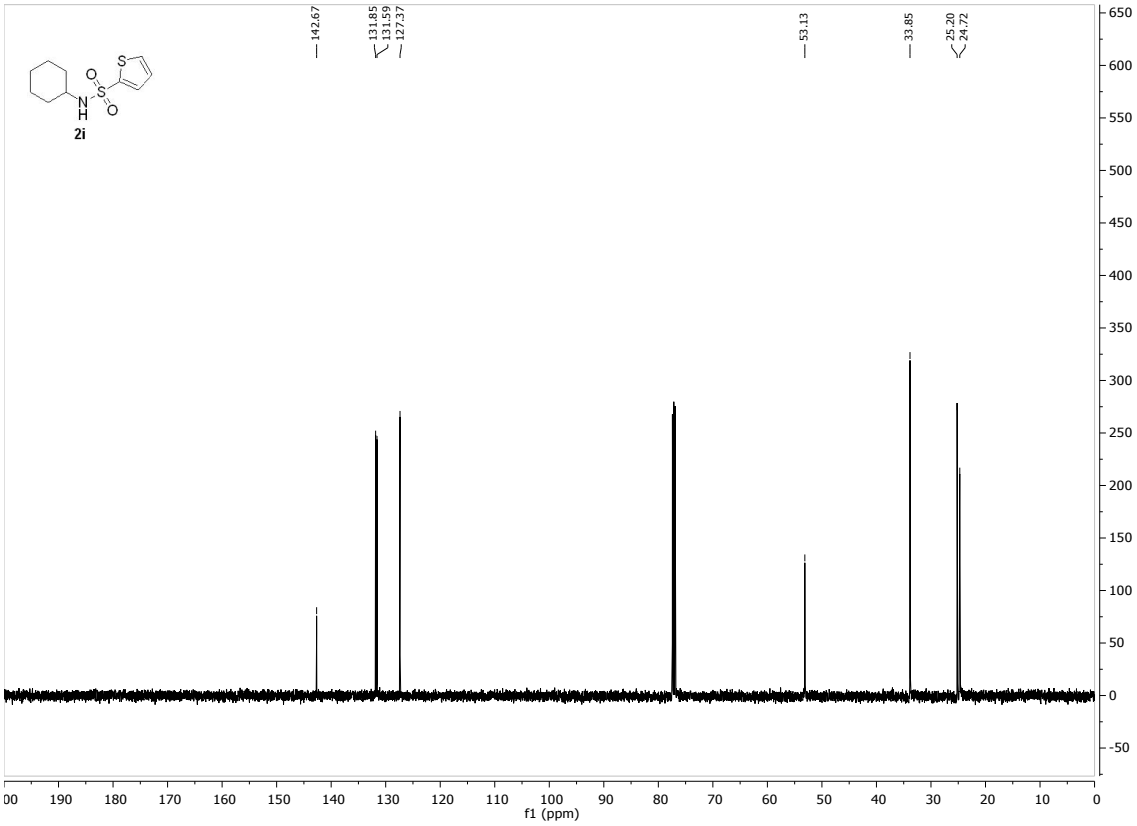
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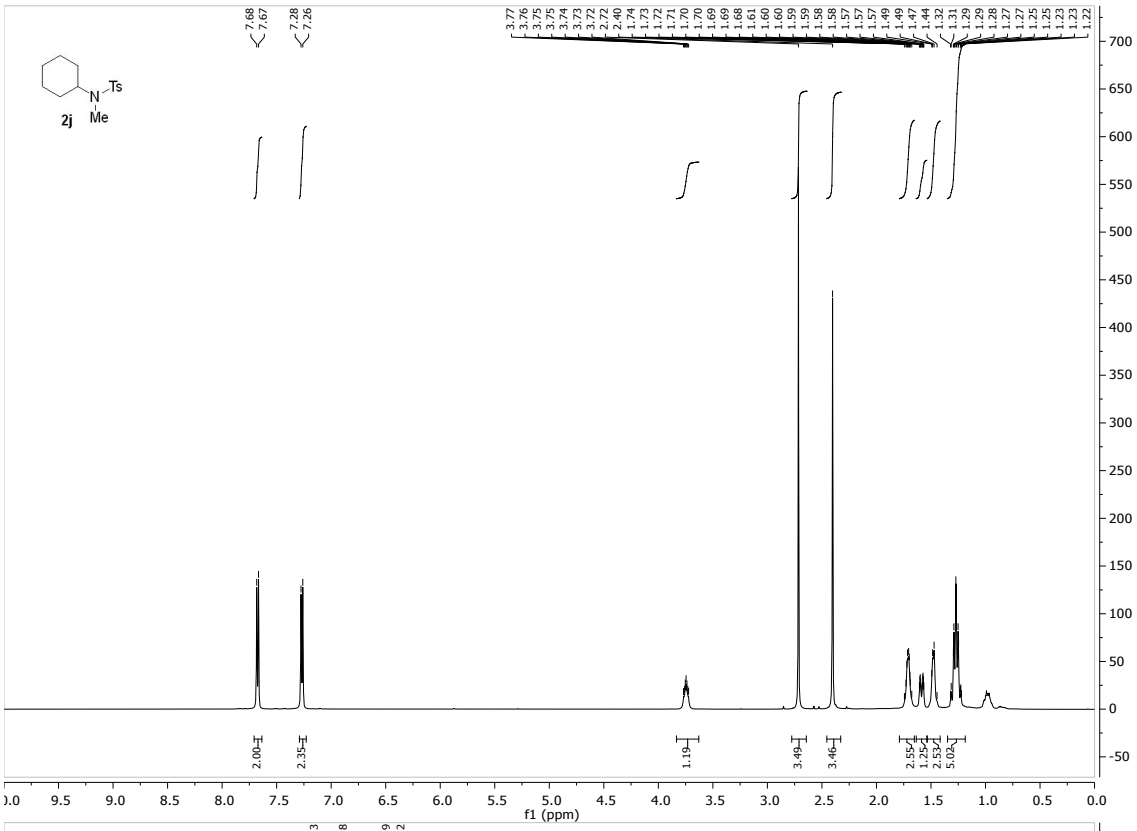
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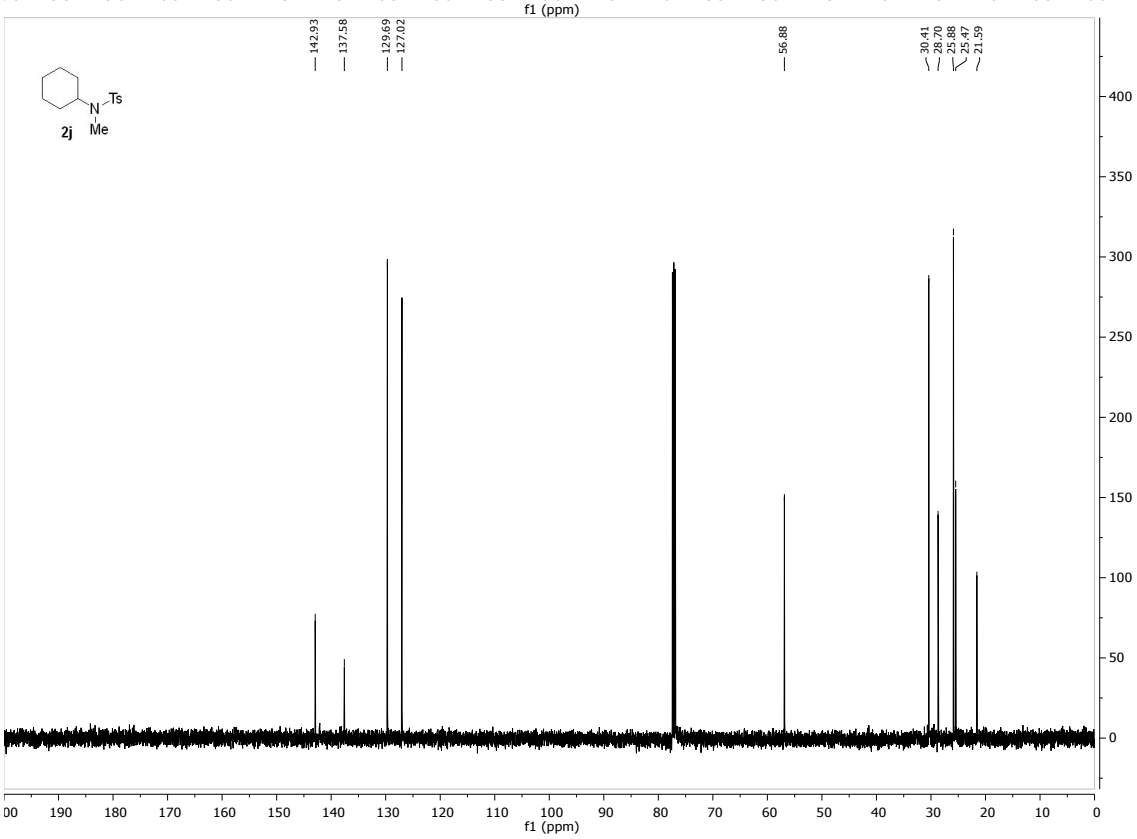
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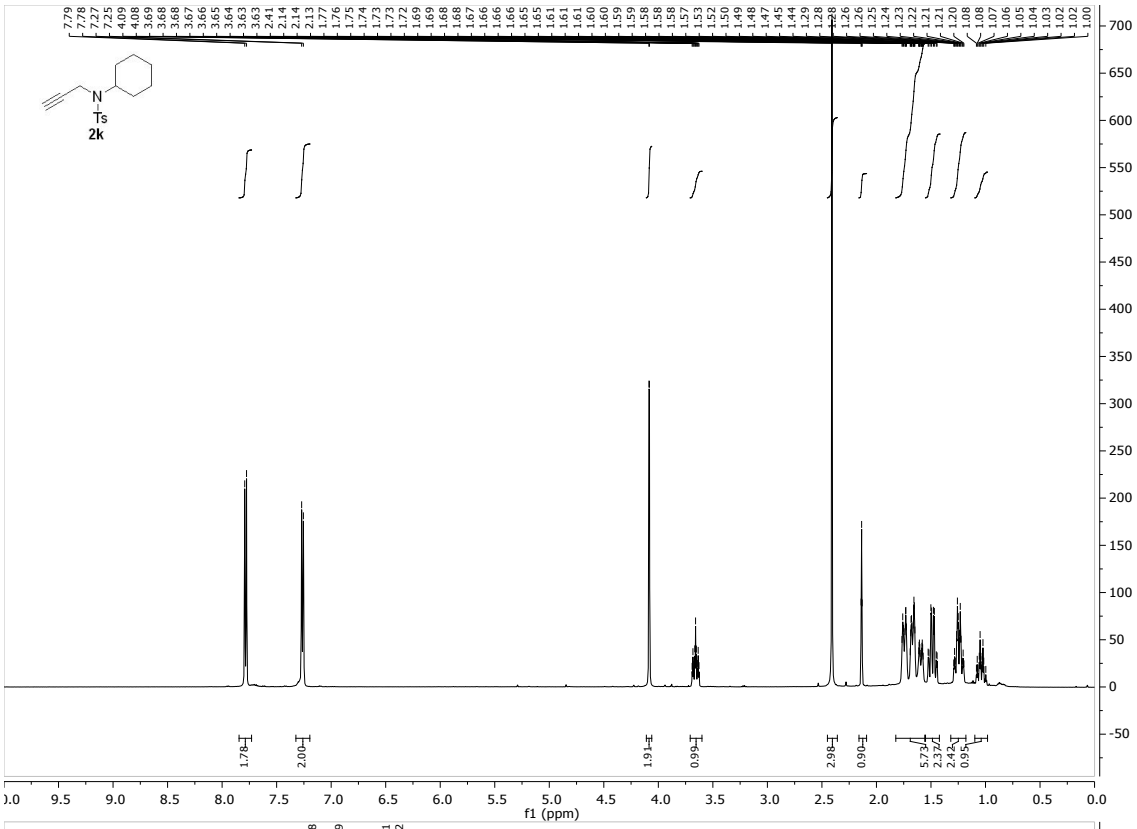
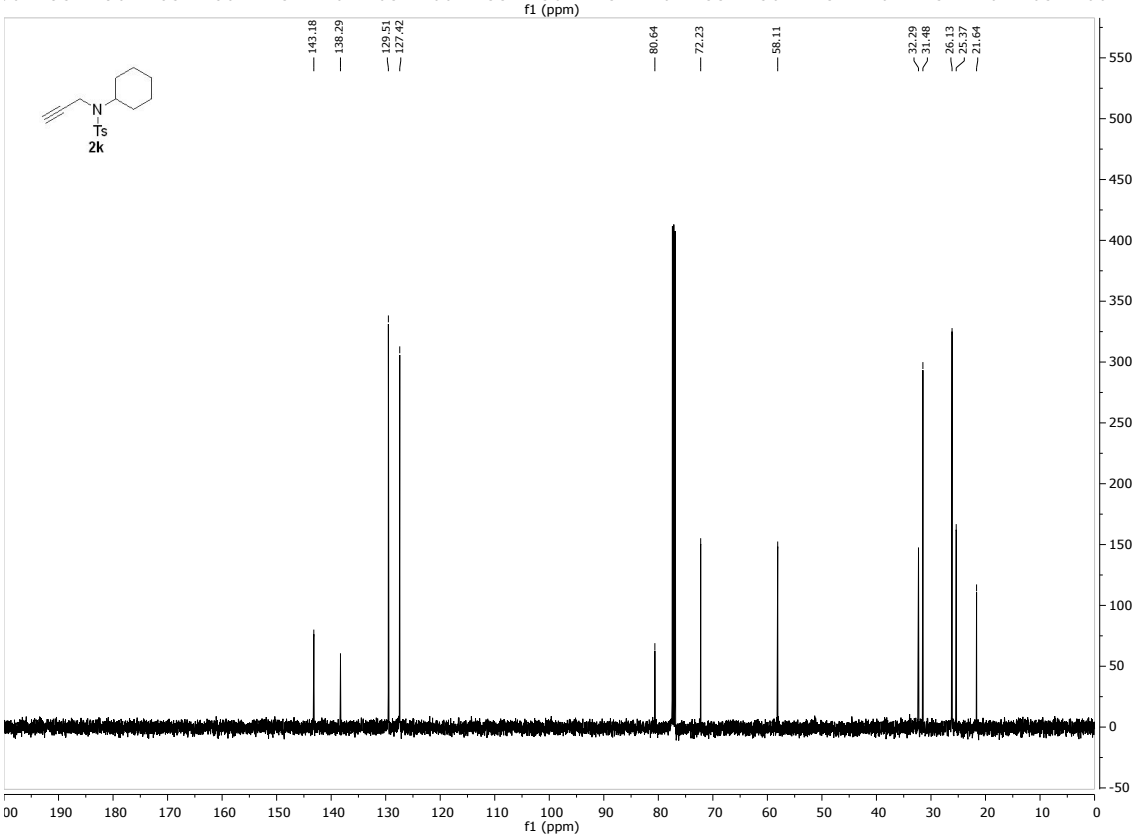
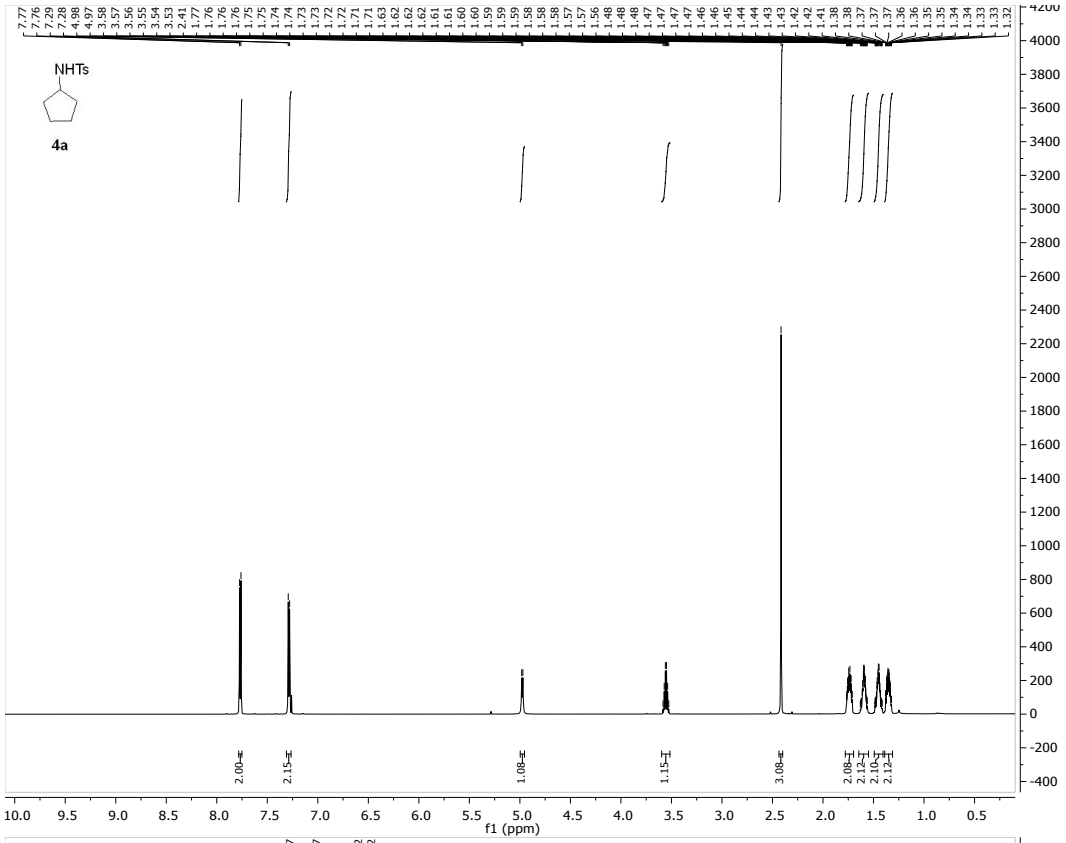
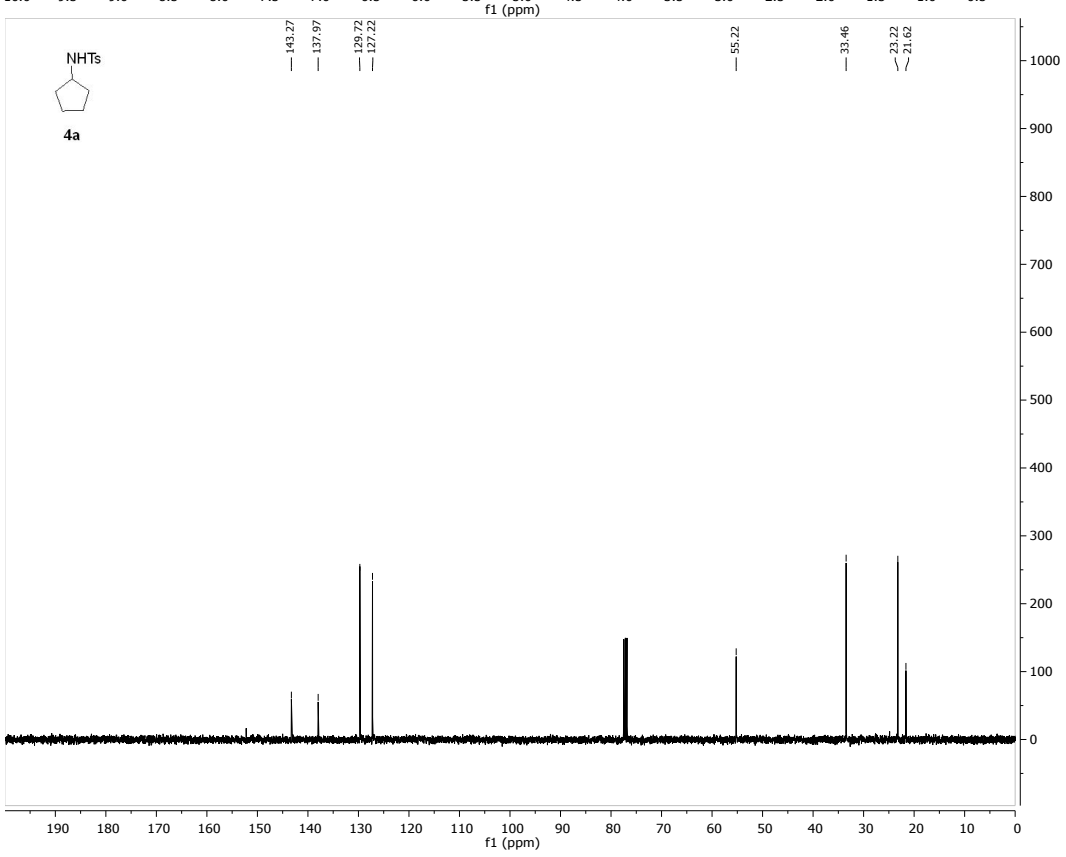
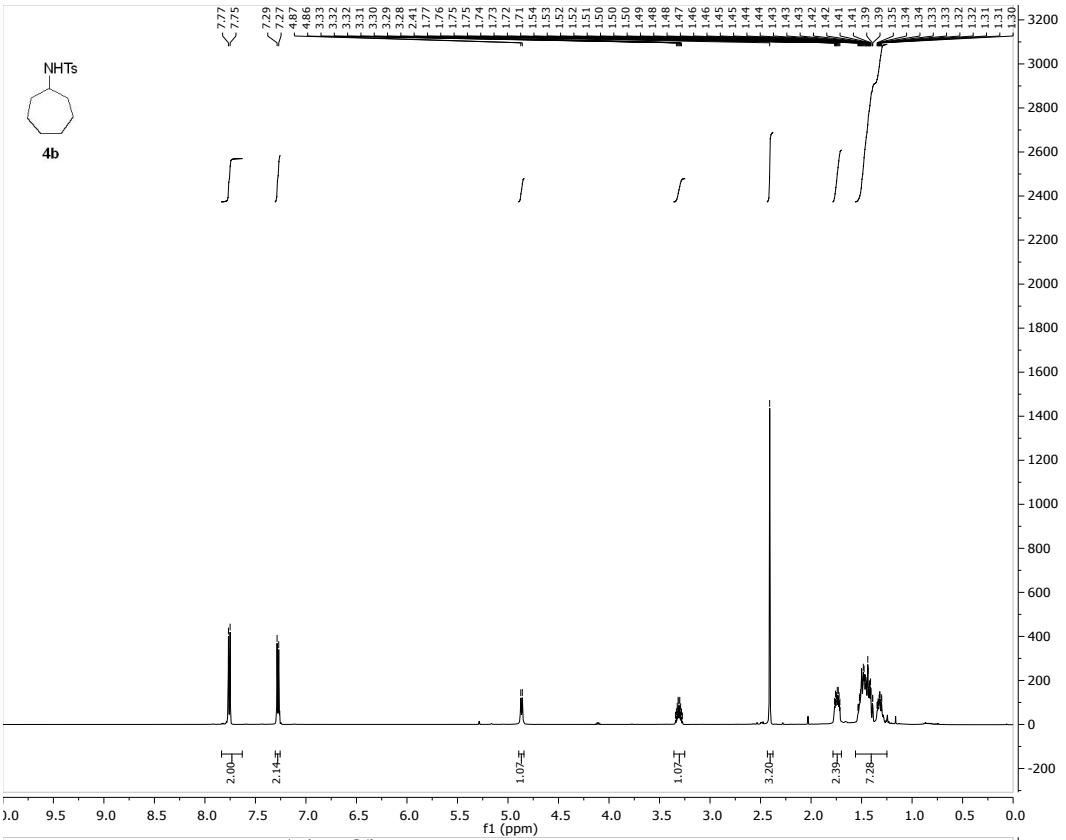
2j
f1 (ppm)

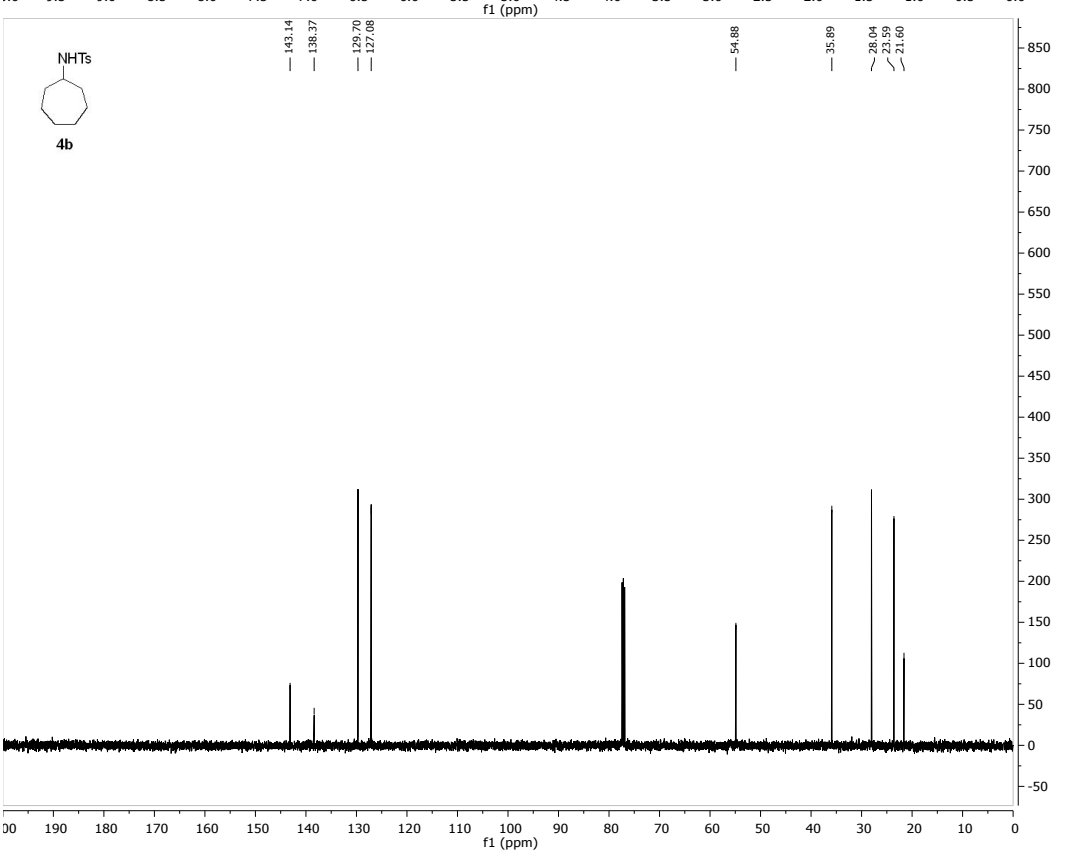

N
Ts
2k
f1 (ppm)

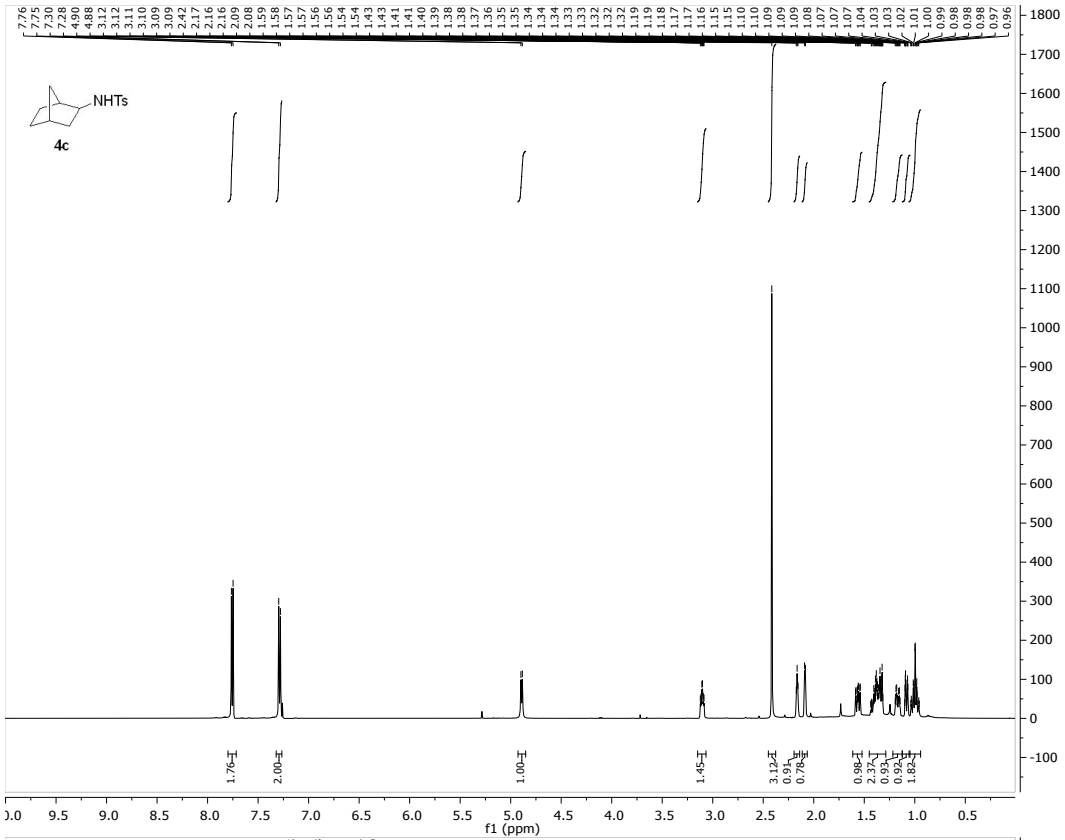

143.18
138.29
129.51
127.42
80.64
72.23
58.11
32.29
31.48
26.13
25.37
21.64
N
Ts
2k
f1 (ppm)

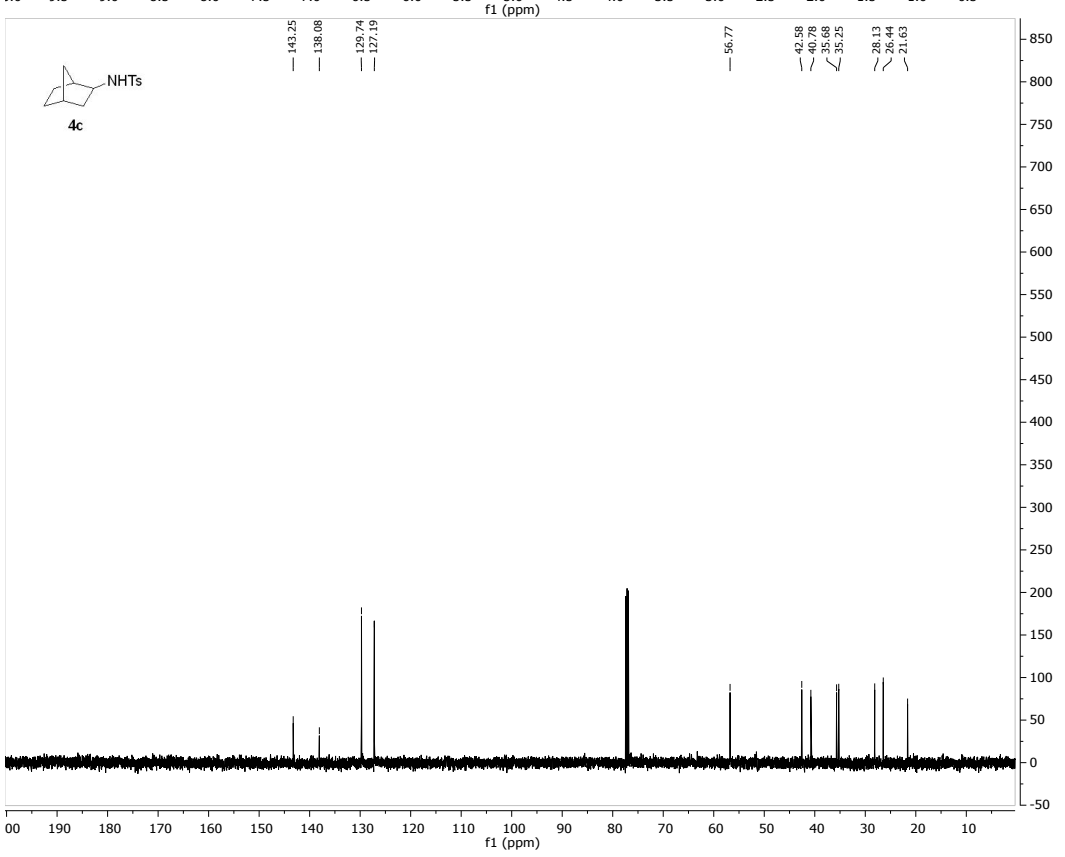

NHTs
4a
f1 (ppm)

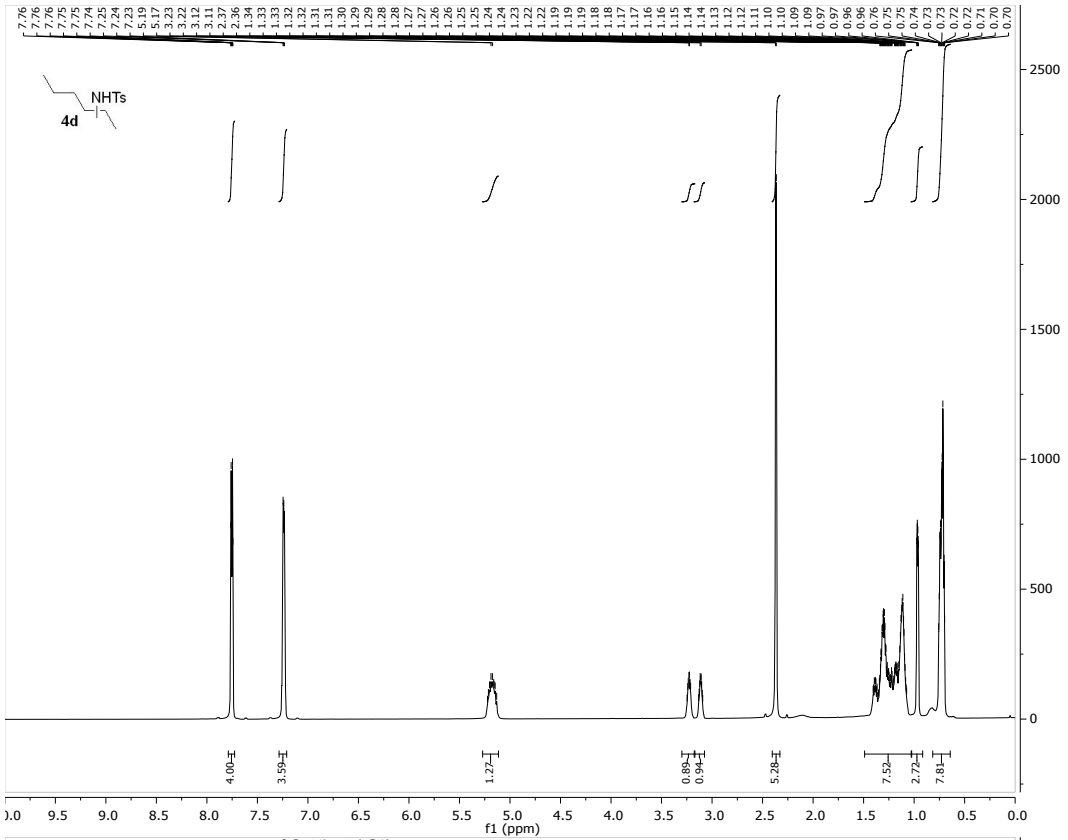

NHTs
4a
143.27
137.97
129.72
127.22
55.22
33.46
23.22
21.62
f1 (ppm)

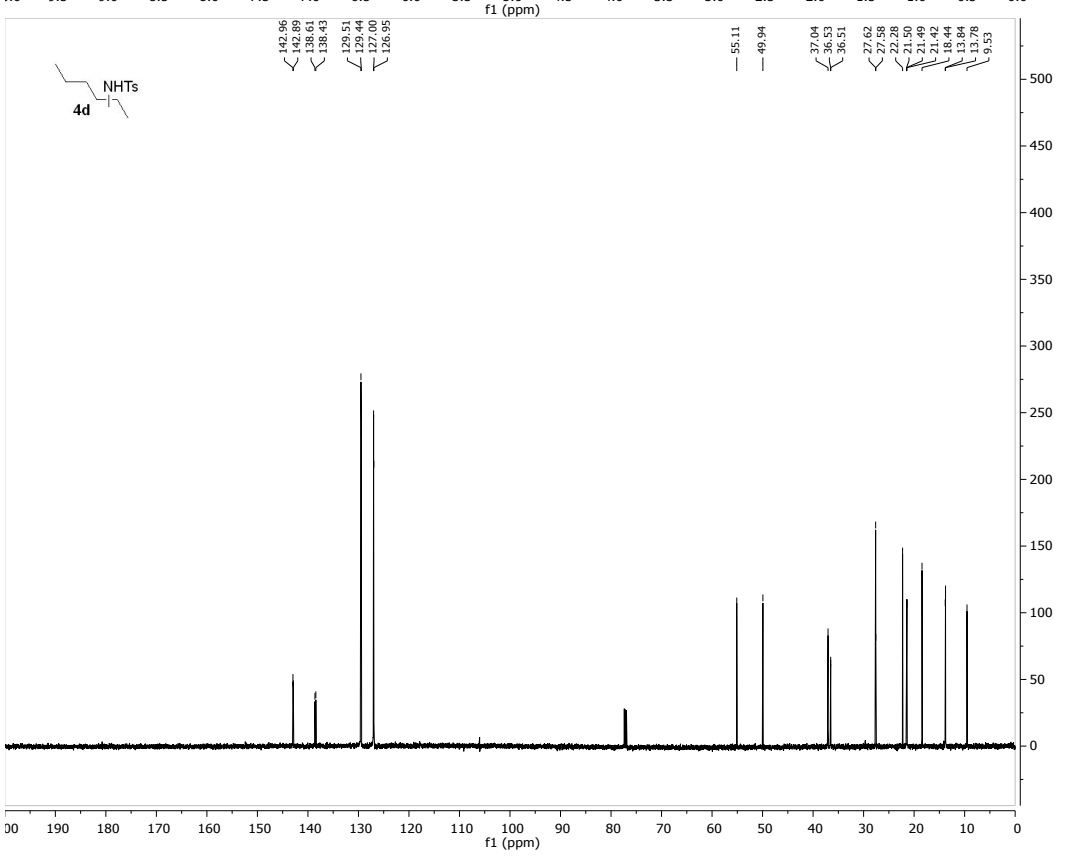

NHTs
4b
7.77
7.75
7.29
7.27
4.87
4.86
3.33
3.32
3.31
3.30
3.29
3.28
2.41
1.77
1.76
1.75
1.75
1.74
1.73
1.72
1.71
1.54
1.53
1.52
1.52
1.51
1.50
1.50
1.50
1.49
1.48
1.48
1.47
1.46
1.46
1.45
1.45
1.44
1.44
1.43
1.43
1.43
1.42
1.42
1.41
1.41
1.39
1.39
1.35
1.34
1.34
1.33
1.33
1.32
1.32
1.31
1.31
1.30
2.00
2.14
1.07
1.07
3.20
2.39
7.28
f1 (ppm)

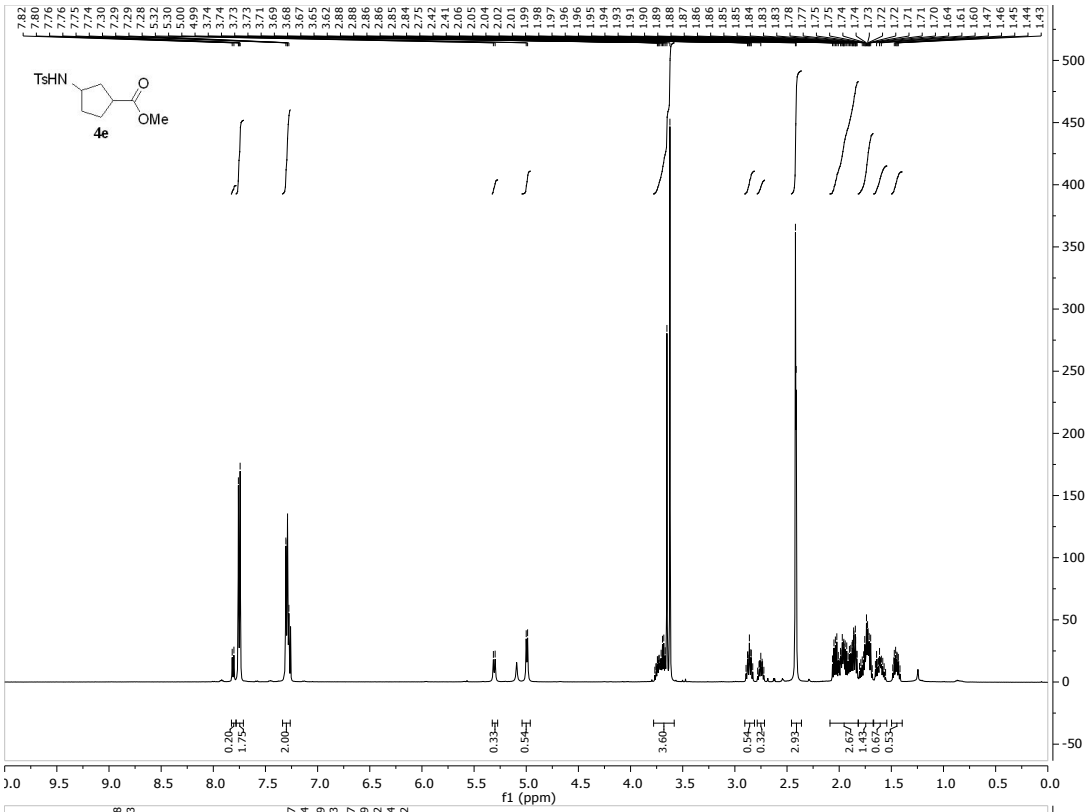

NHTs
4b
143.14
138.37
129.70
127.08
54.88
35.89
28.04
23.59
21.60
f1 (ppm)

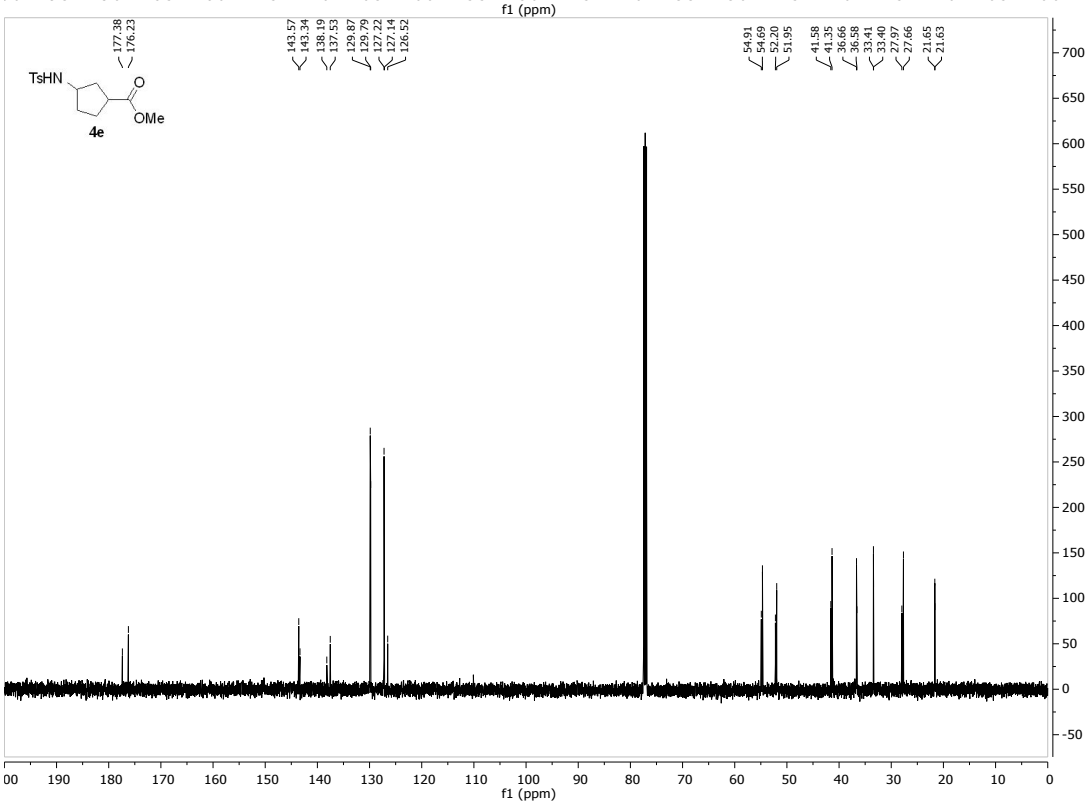

NHTs
4c
f1 (ppm)

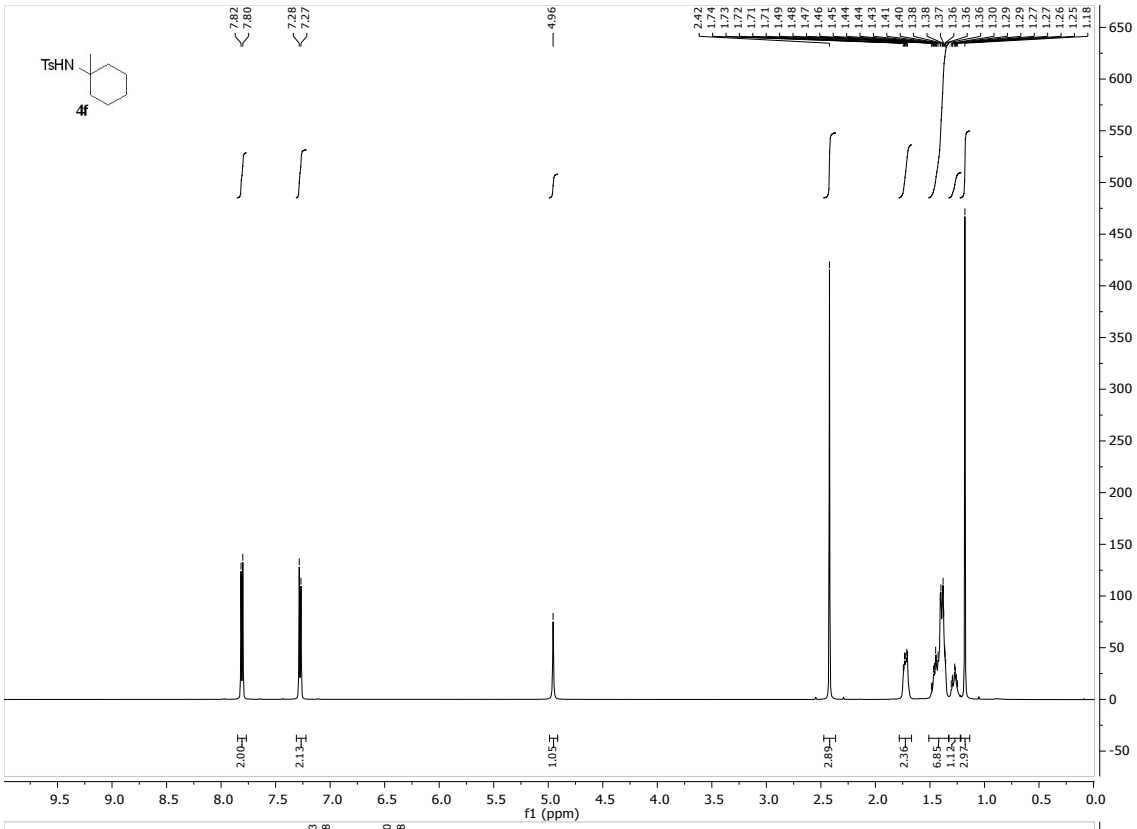

NHTs
4c
143.25
138.08
129.74
127.19
56.77
42.58
40.78
35.68
35.25
28.13
26.44
21.63
f1 (ppm)

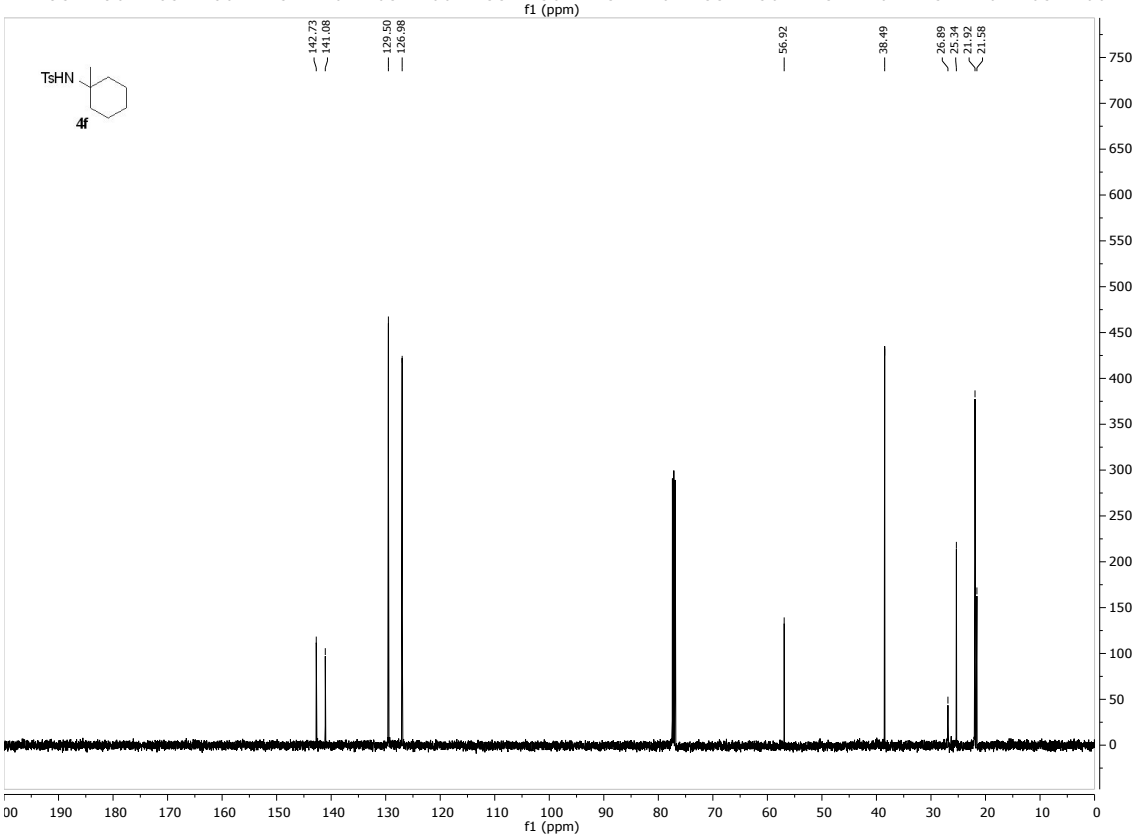

NHTs
4d
f1 (ppm)

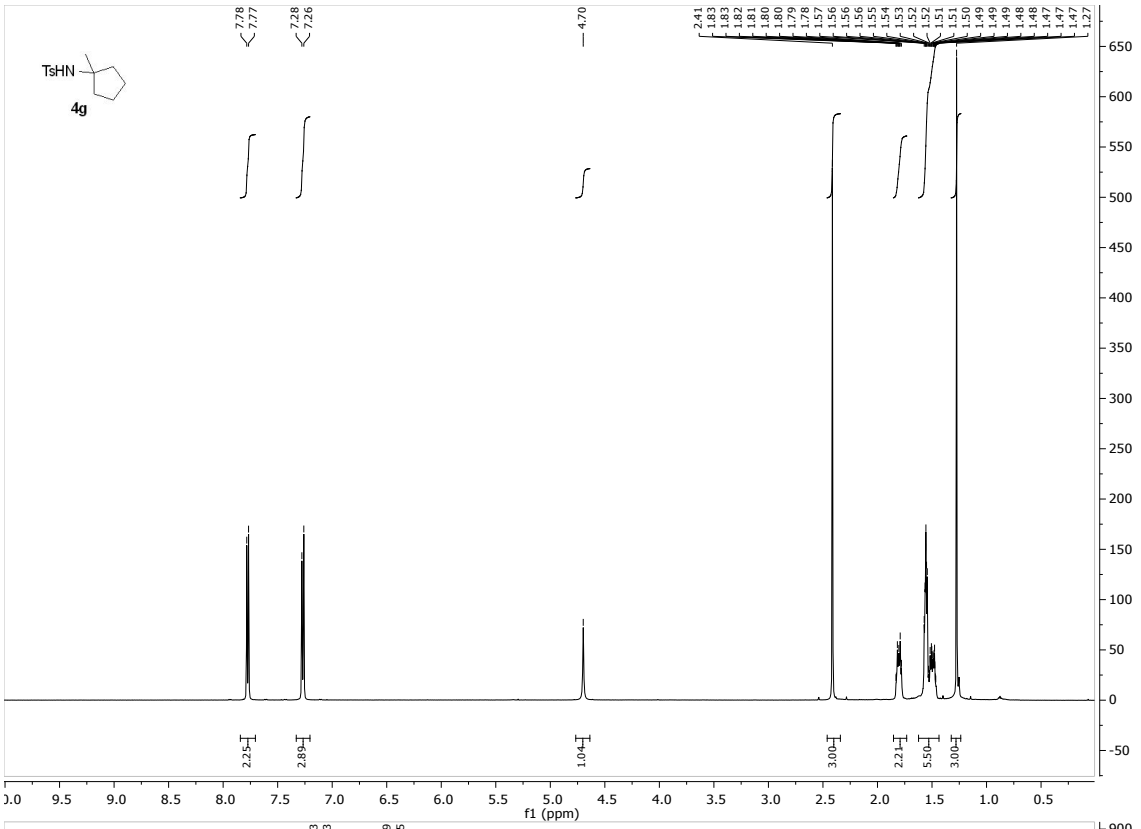

NHTs
4d
f1 (ppm)

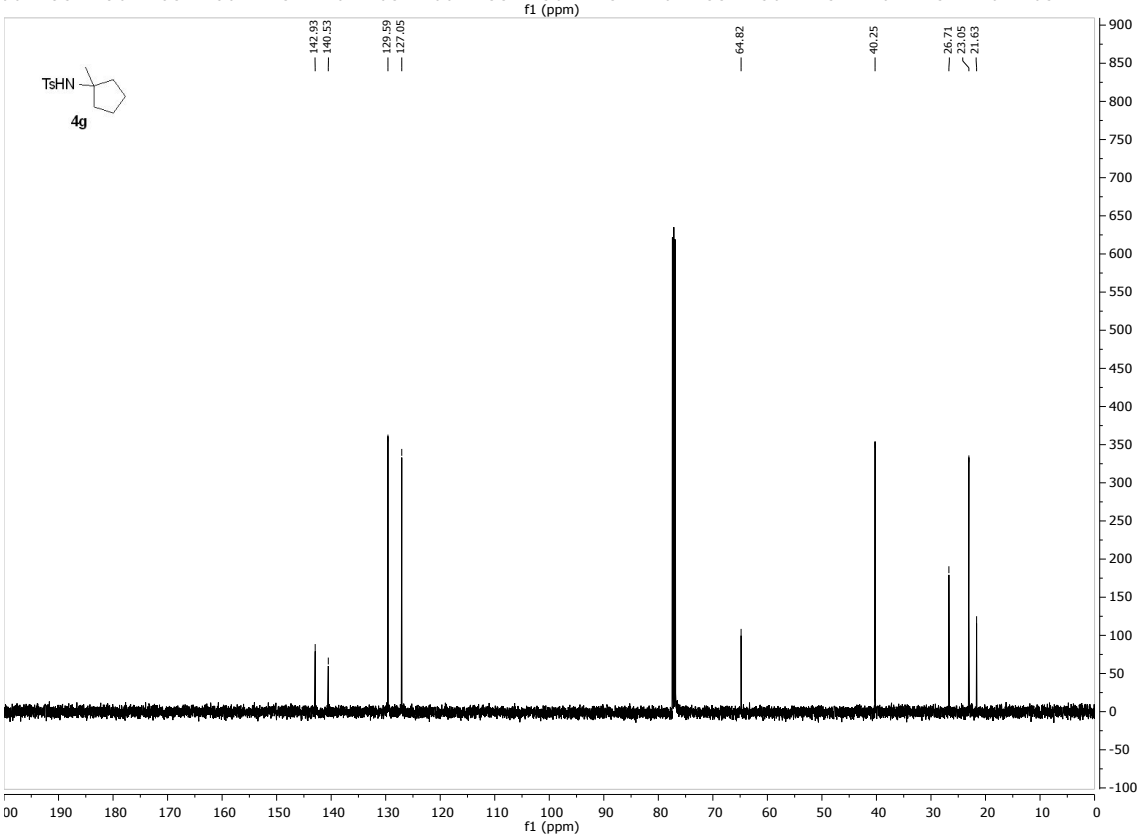

TsHN
O
OMe
4e
f1 (ppm)

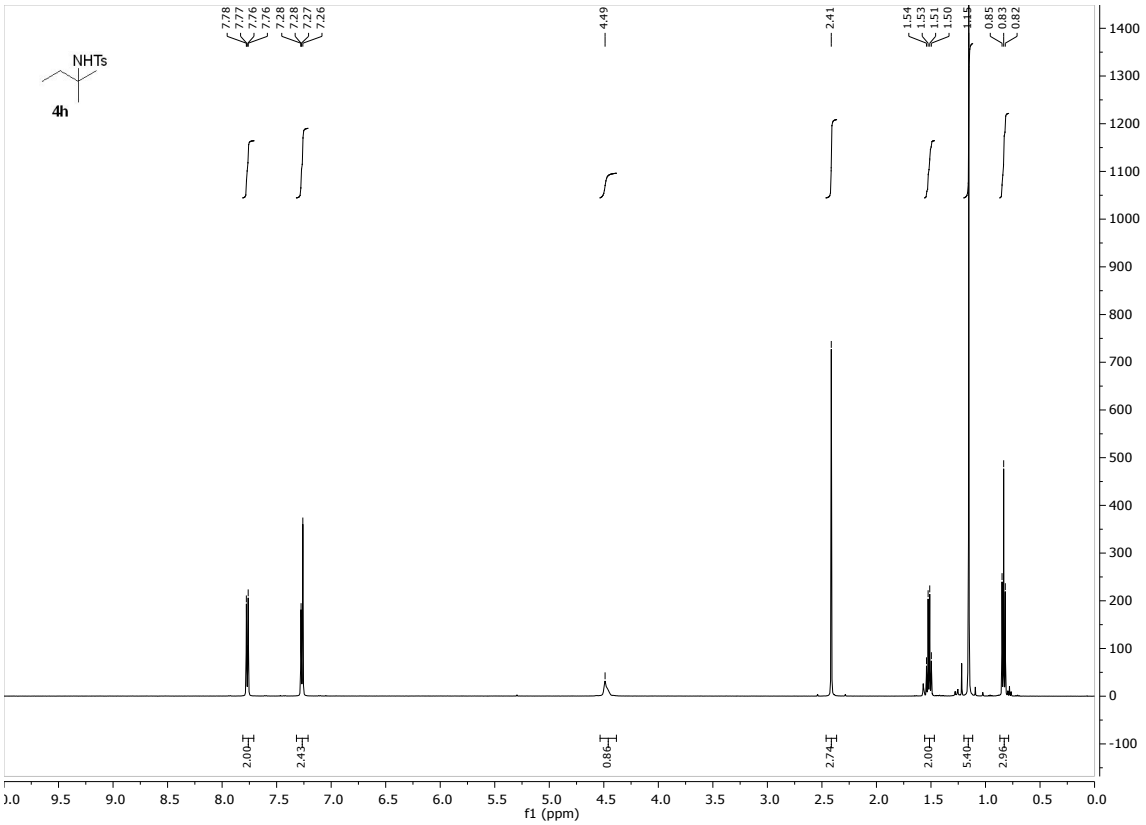

TsHN
O
OMe
4e
f1 (ppm)

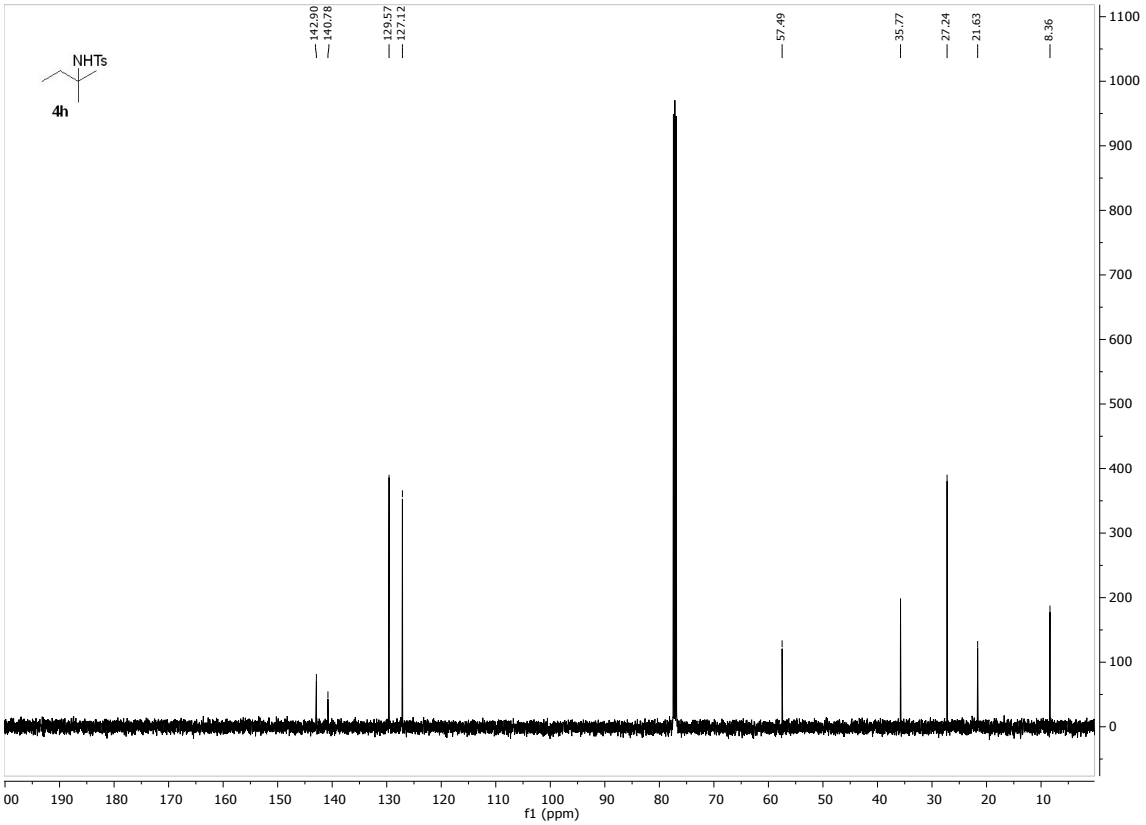

TsHN
4f
7.82
7.80
7.28
7.27
4.96
2.42
1.74
1.73
1.72
1.71
1.71
1.49
1.48
1.47
1.46
1.45
1.44
1.44
1.43
1.41
1.40
1.38
1.38
1.37
1.36
1.36
1.30
1.29
1.29
1.27
1.27
1.26
1.25
1.18
2.00
2.13
1.05
2.89
2.36
6.85
1.12
2.97
f1 (ppm)

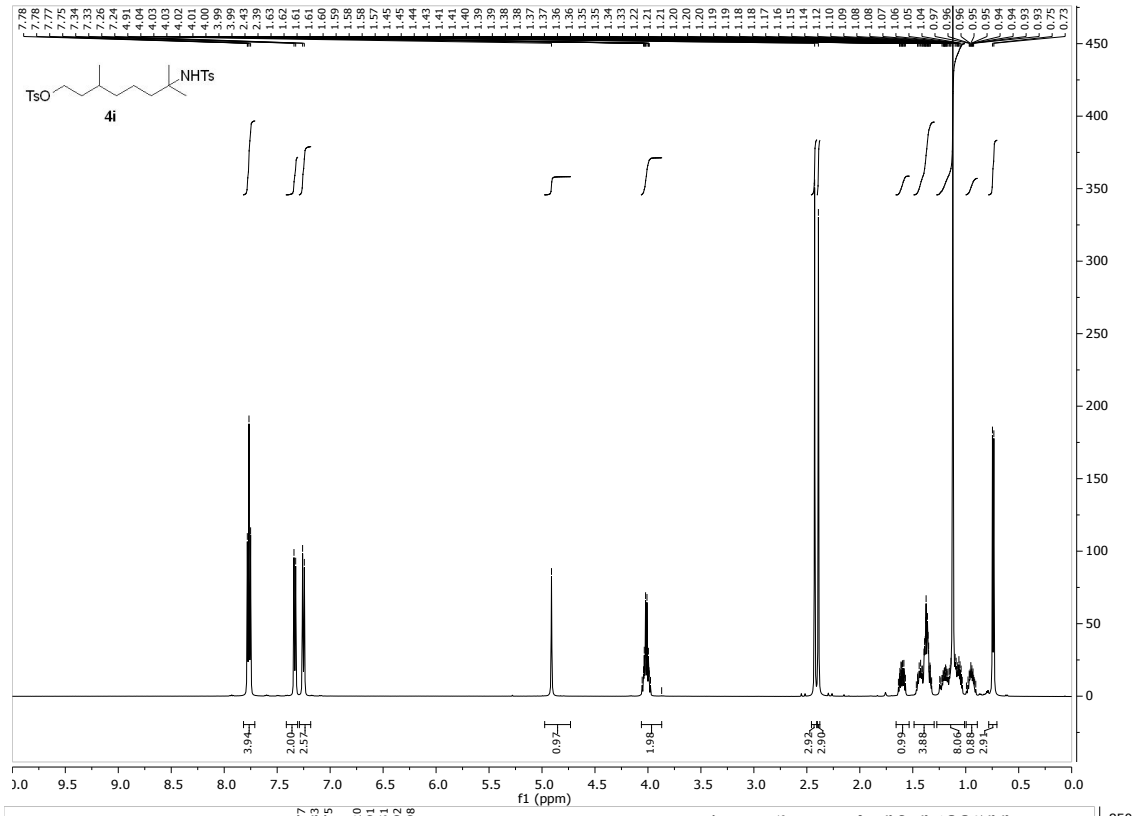

TsHN
4f
142.73
141.08
129.50
126.98
56.92
38.49
26.89
25.34
21.92
21.58
f1 (ppm)

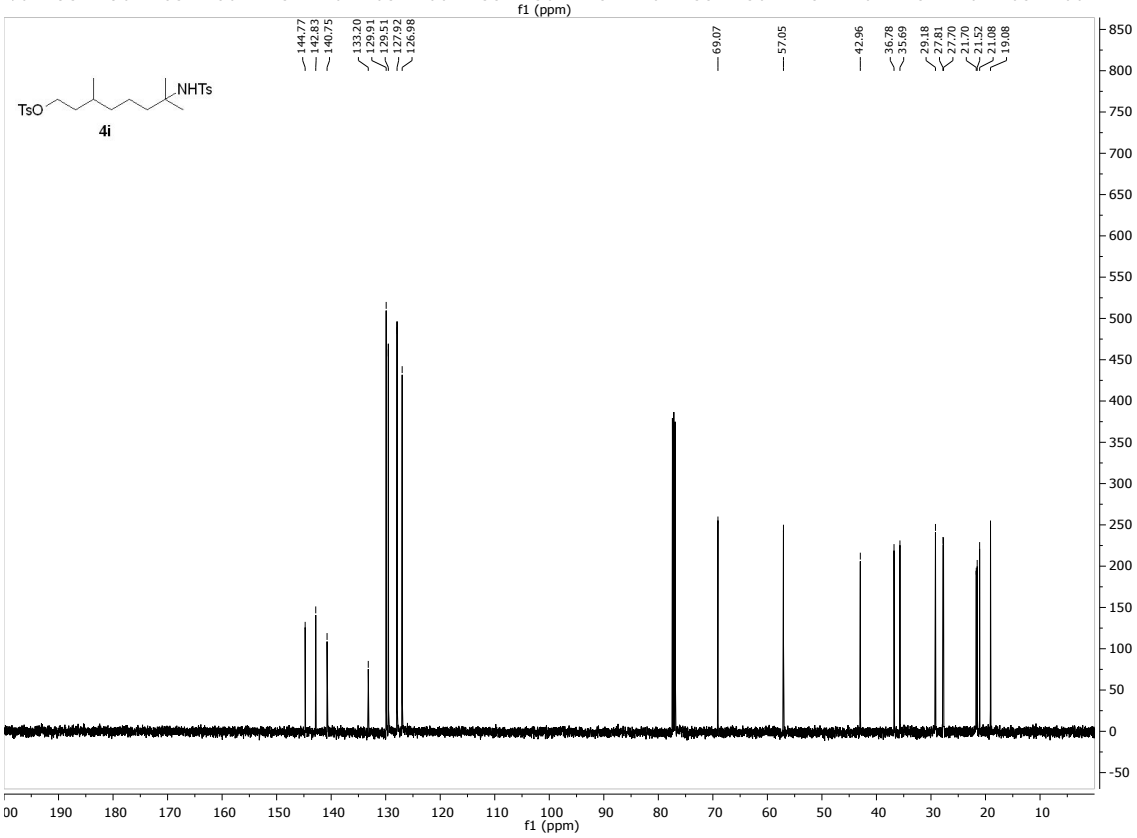

TsHN
4g
7.78
7.77
7.28
7.26
4.70
2.41
1.83
1.82
1.81
1.80
1.79
1.78
1.57
1.56
1.55
1.54
1.53
1.52
1.51
1.50
1.49
1.48
1.47
1.27
2.25
2.89
1.04
3.00
2.21
5.50
3.06
f1 (ppm)

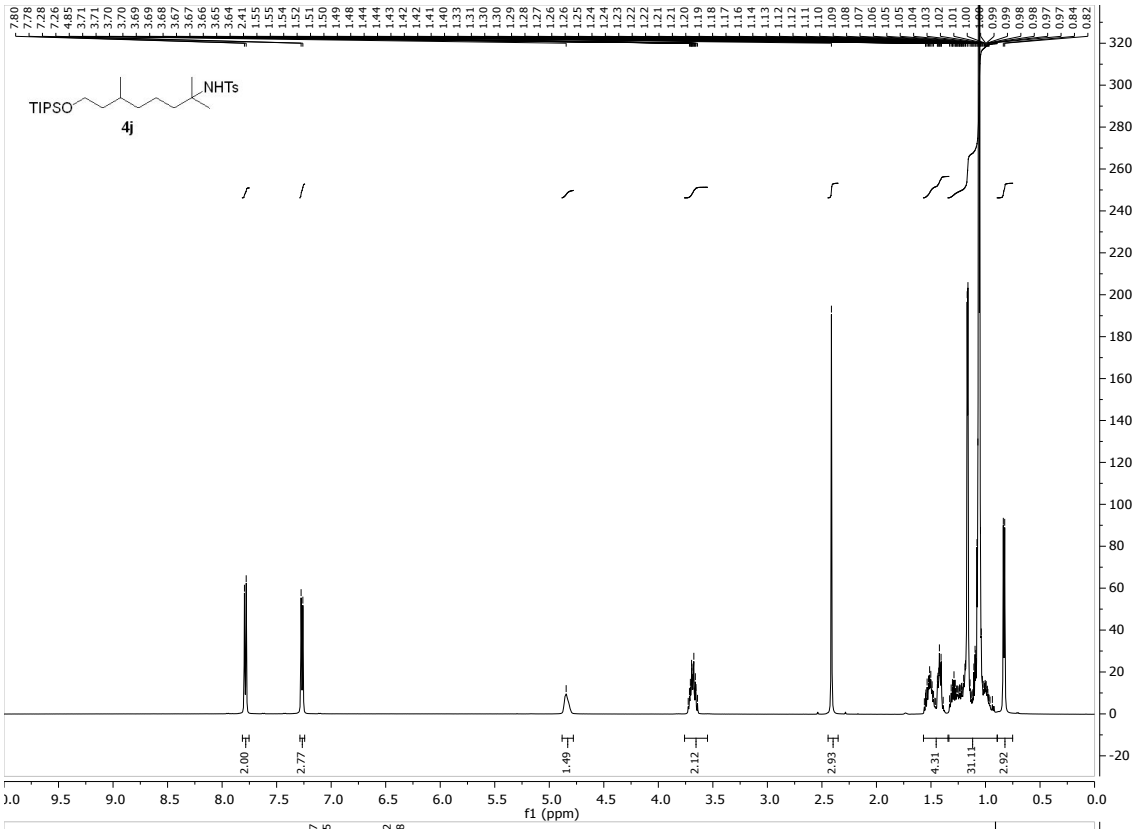

TsHN
4g
142.93
140.53
129.59
127.05
64.82
40.25
26.71
23.05
21.63
f1 (ppm)

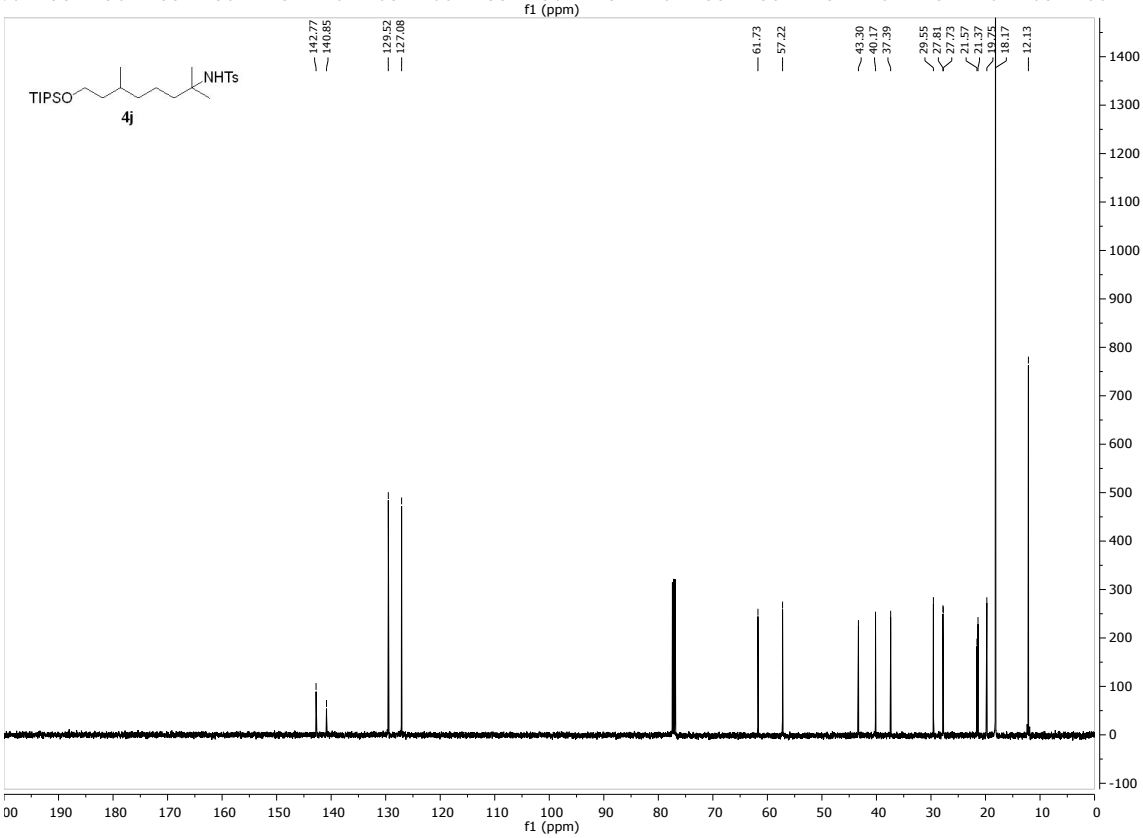

NHTs
4h
7.78
7.77
7.76
7.76
7.28
7.27
7.26
4.49
2.41
1.54
1.53
1.51
1.50
1.15
0.85
0.83
0.82
2.00
2.43
0.86
2.74
2.00
5.40
2.96
f1 (ppm)

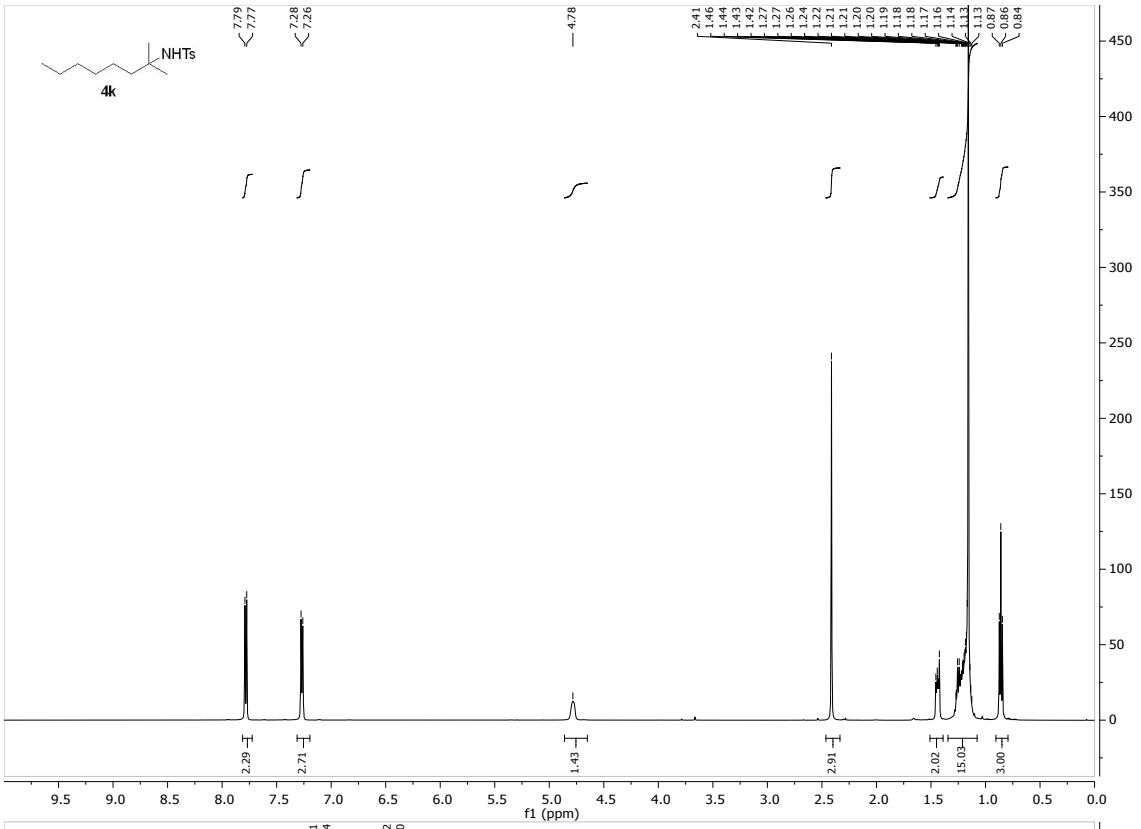

NHTs
4h
142.90
140.78
129.57
127.12
57.49
35.77
27.24
21.63
8.36
f1 (ppm)

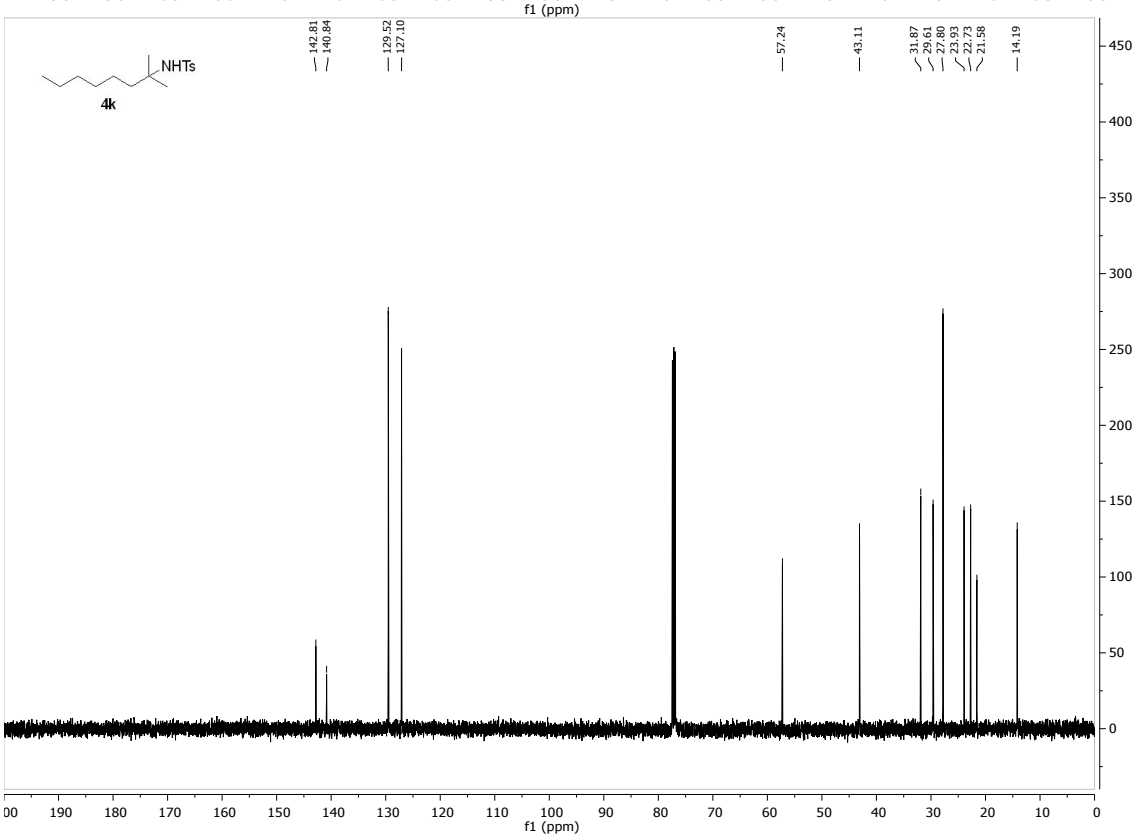

TsO
NHTs
4i
f1 (ppm)

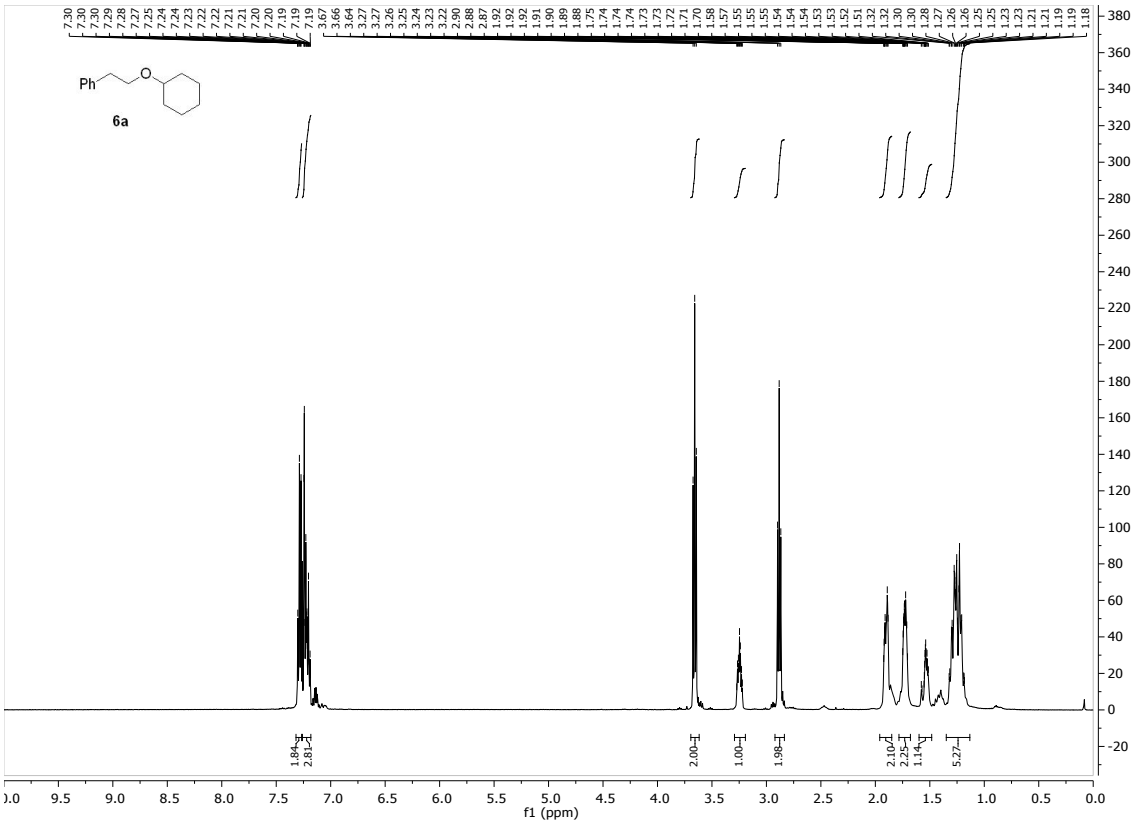

TsO
NHTs
4i
f1 (ppm)

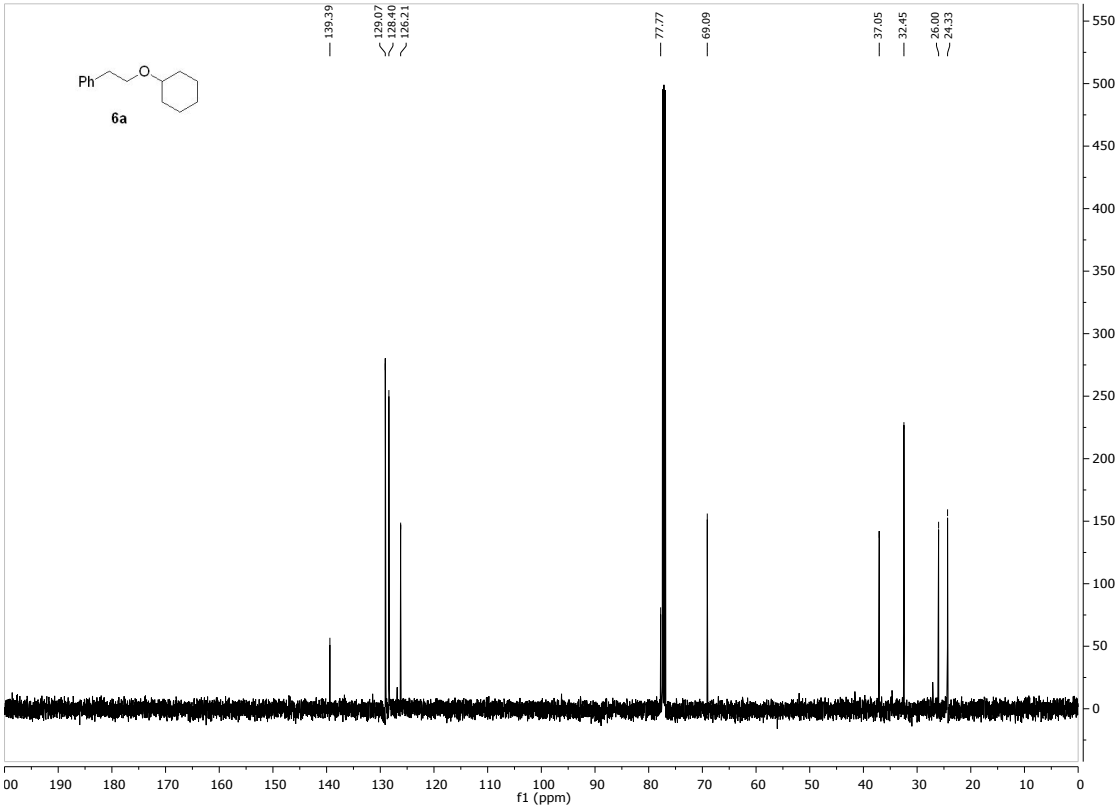

TIPSO
NHTs
4j
f1 (ppm)

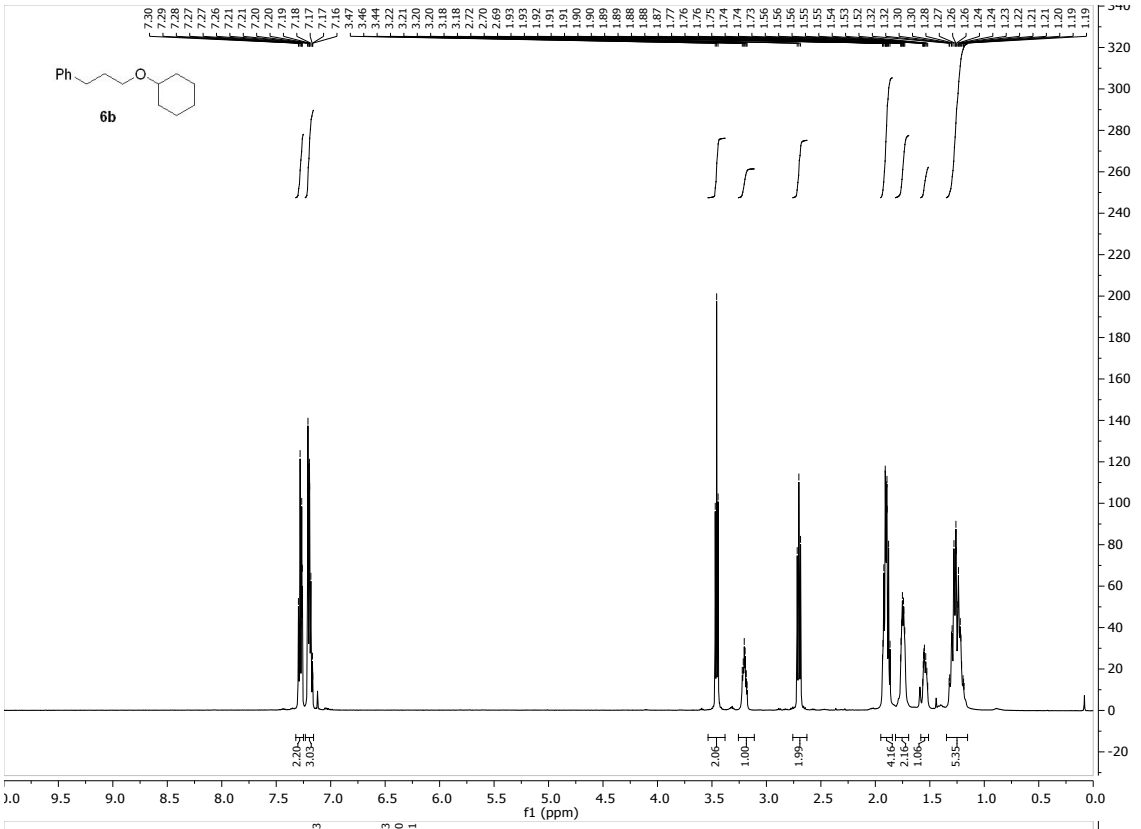

TIPSO
NHTs
4j
f1 (ppm)

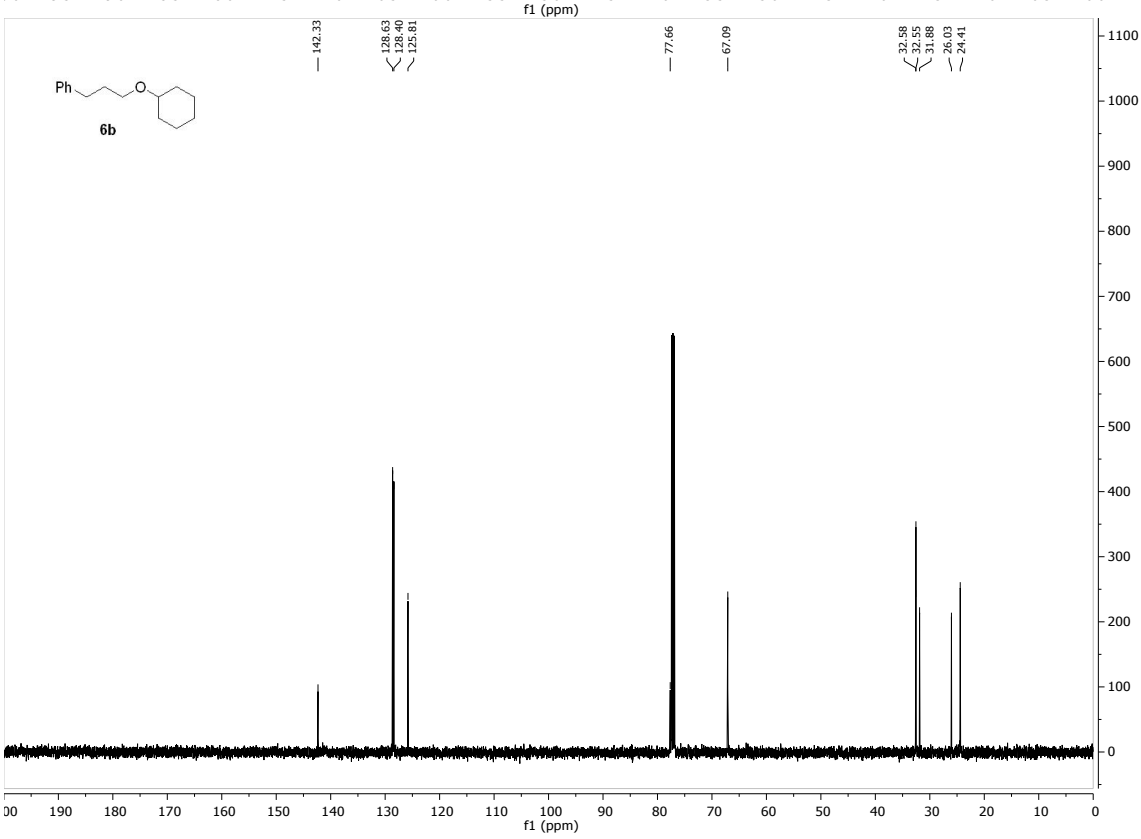

NHTs
4k
7.79
7.77
7.28
7.26
4.78
2.41
1.46
1.44
1.43
1.42
1.27
1.27
1.26
1.24
1.22
1.21
1.21
1.20
1.20
1.19
1.19
1.18
1.18
1.17
1.16
1.14
1.13
1.13
0.87
0.86
0.84
2.29
2.71
1.43
2.91
2.02
15.03
3.00
f1 (ppm)

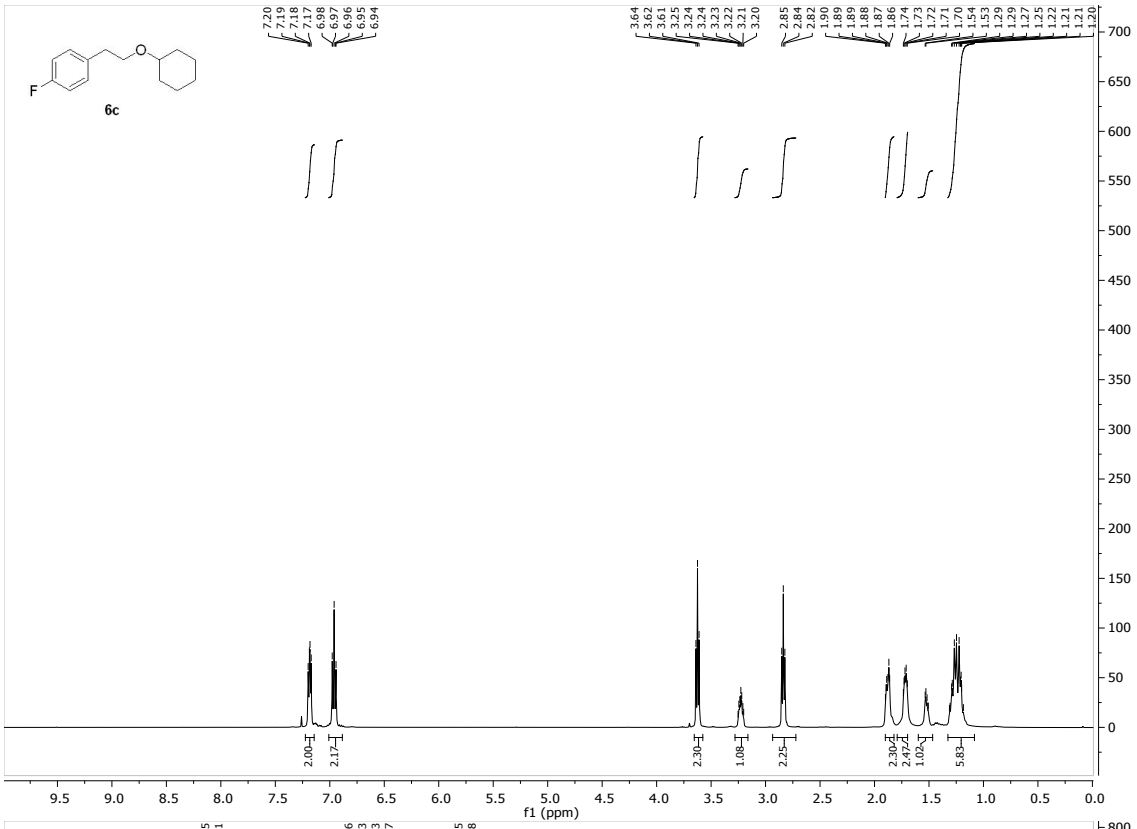

NHTs
4k
142.81
140.84
129.52
127.10
57.24
43.11
31.87
29.61
27.80
23.93
22.73
21.58
14.19
f1 (ppm)

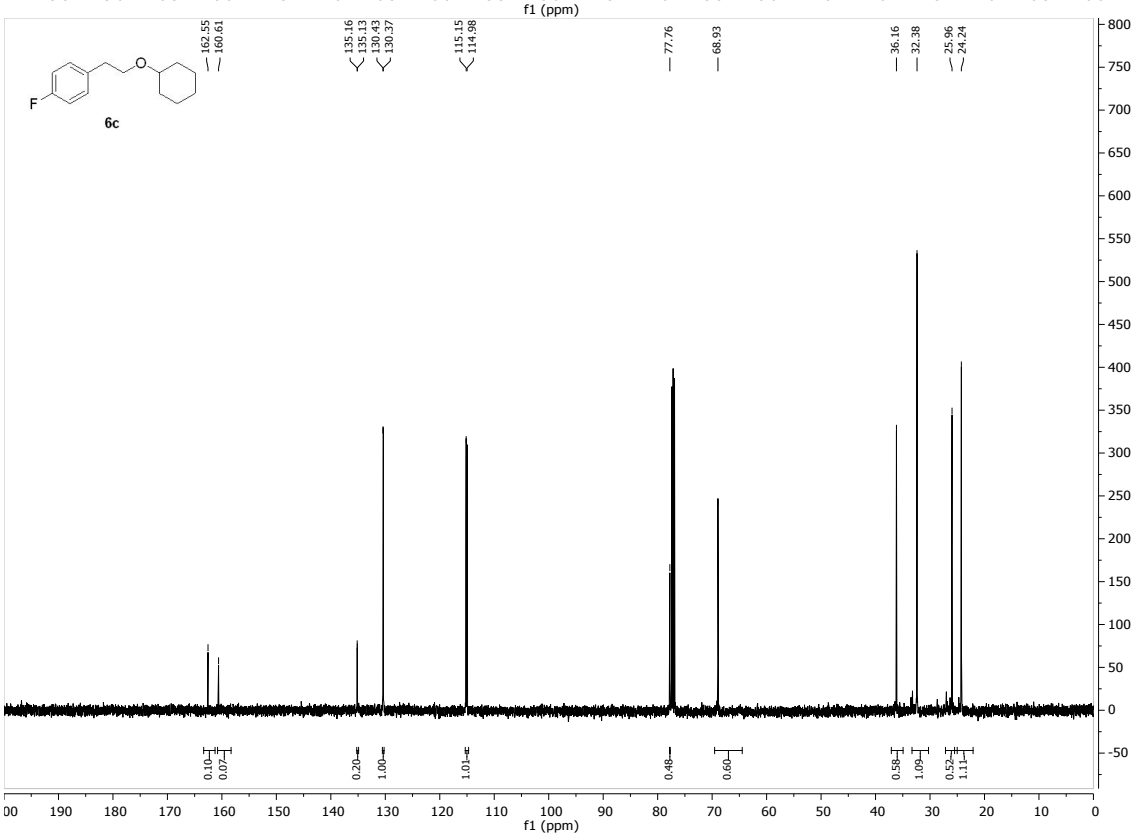

Ph
O
6a
f1 (ppm)

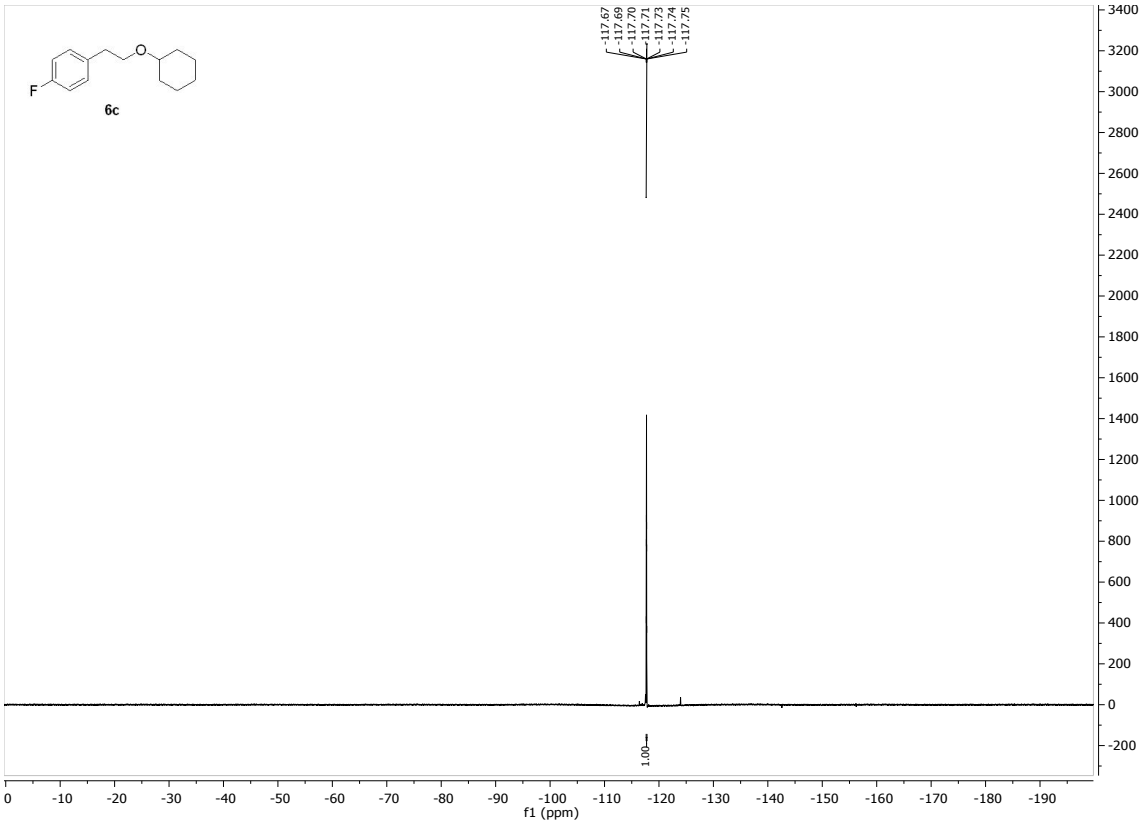

Ph
O
6a
139.39
129.07
128.40
126.21
77.77
69.09
37.05
32.45
26.00
24.33
f1 (ppm)

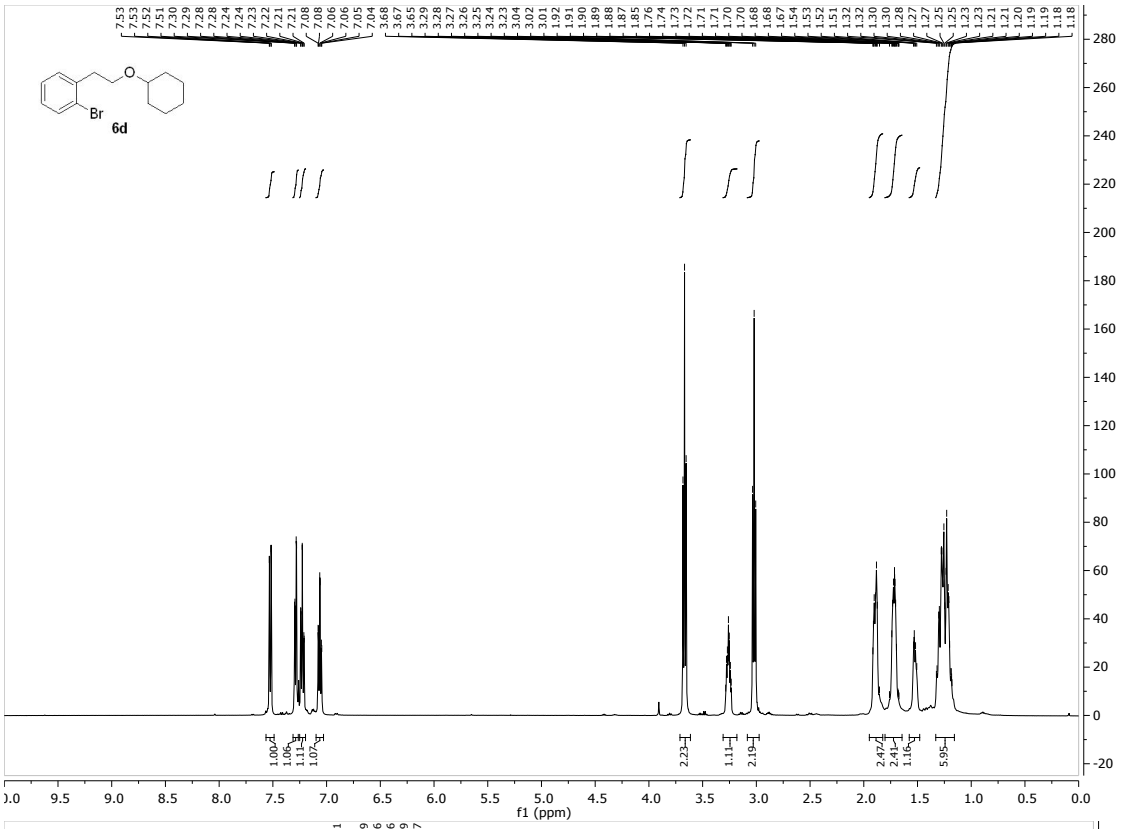

Ph
O
6b
f1 (ppm)

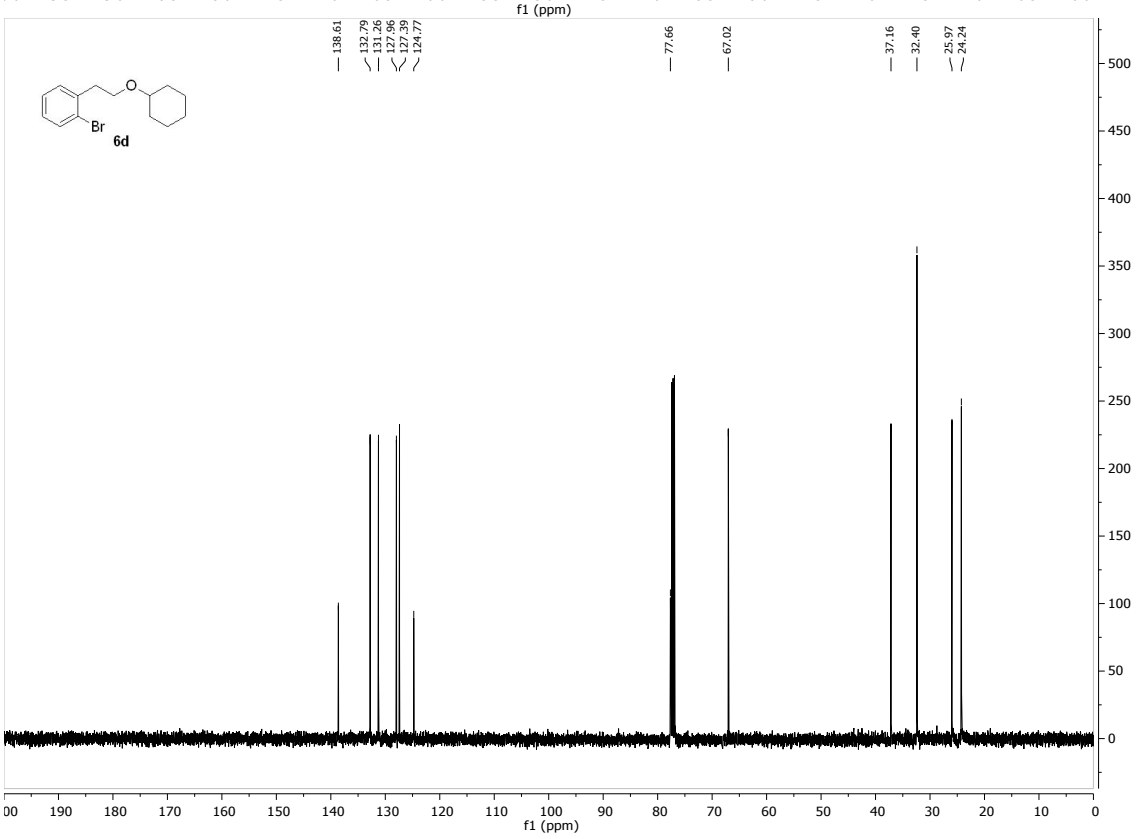

Ph
O
6b
142.33
128.63
128.40
125.81
77.66
67.09
32.58
32.55
31.88
26.03
24.41
f1 (ppm)

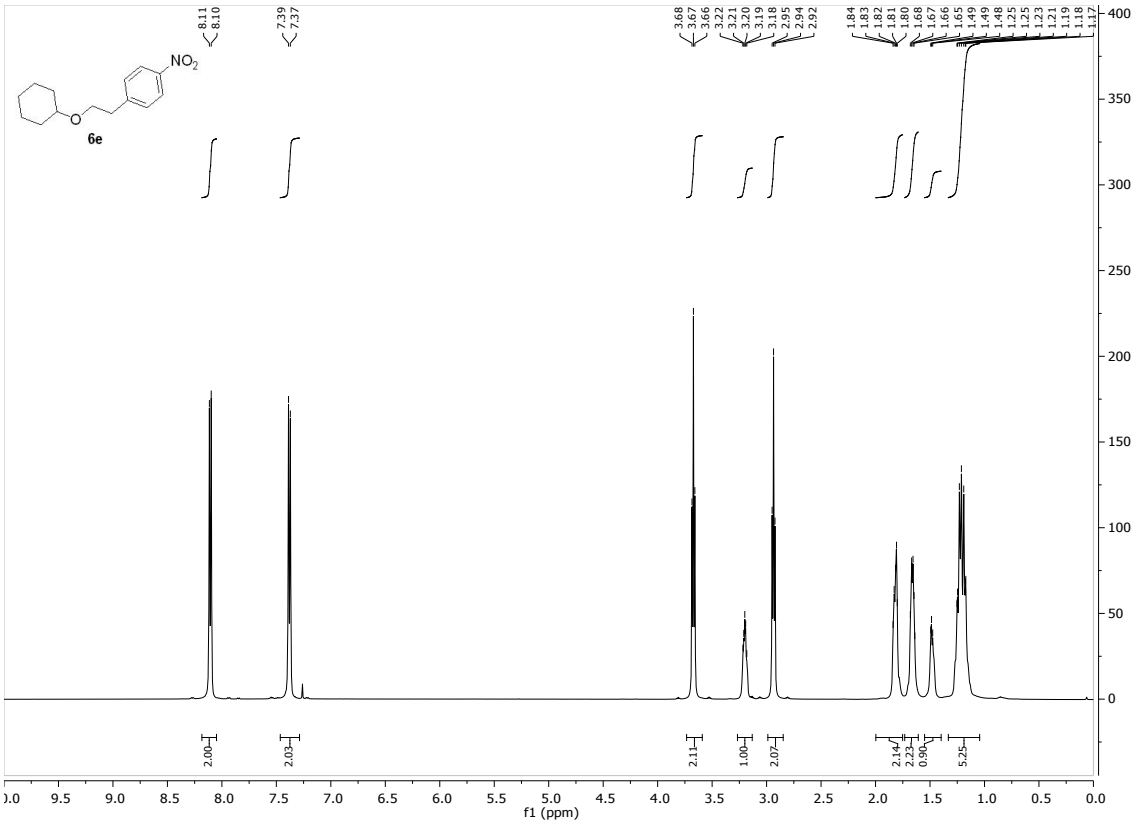

6c
F
O
7.20
7.19
7.18
7.17
6.98
6.97
6.96
6.95
6.94
3.64
3.62
3.61
3.25
3.24
3.23
3.22
3.21
3.20
2.85
2.84
2.82
1.90
1.89
1.89
1.88
1.87
1.86
1.74
1.73
1.72
1.71
1.70
1.54
1.53
1.29
1.29
1.27
1.25
1.22
1.21
1.21
1.20
2.00
2.17
2.30
1.08
2.25
2.30
2.47
1.02
5.83
f1 (ppm)

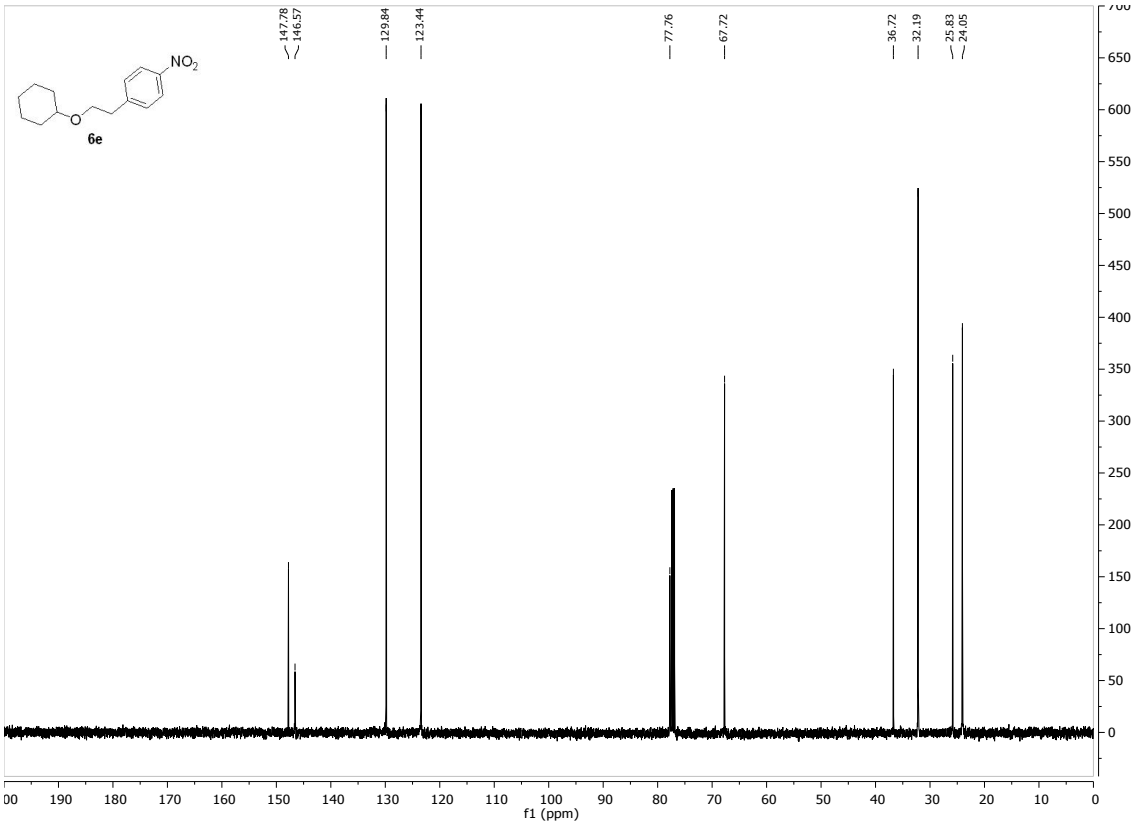

6c
F
O
162.55
160.61
135.16
135.13
130.43
130.37
115.15
114.98
77.76
68.93
36.16
32.38
25.96
24.24
0.10
0.07
0.20
1.00
1.01
0.48
0.60
0.58
1.09
0.52
1.11
f1 (ppm)

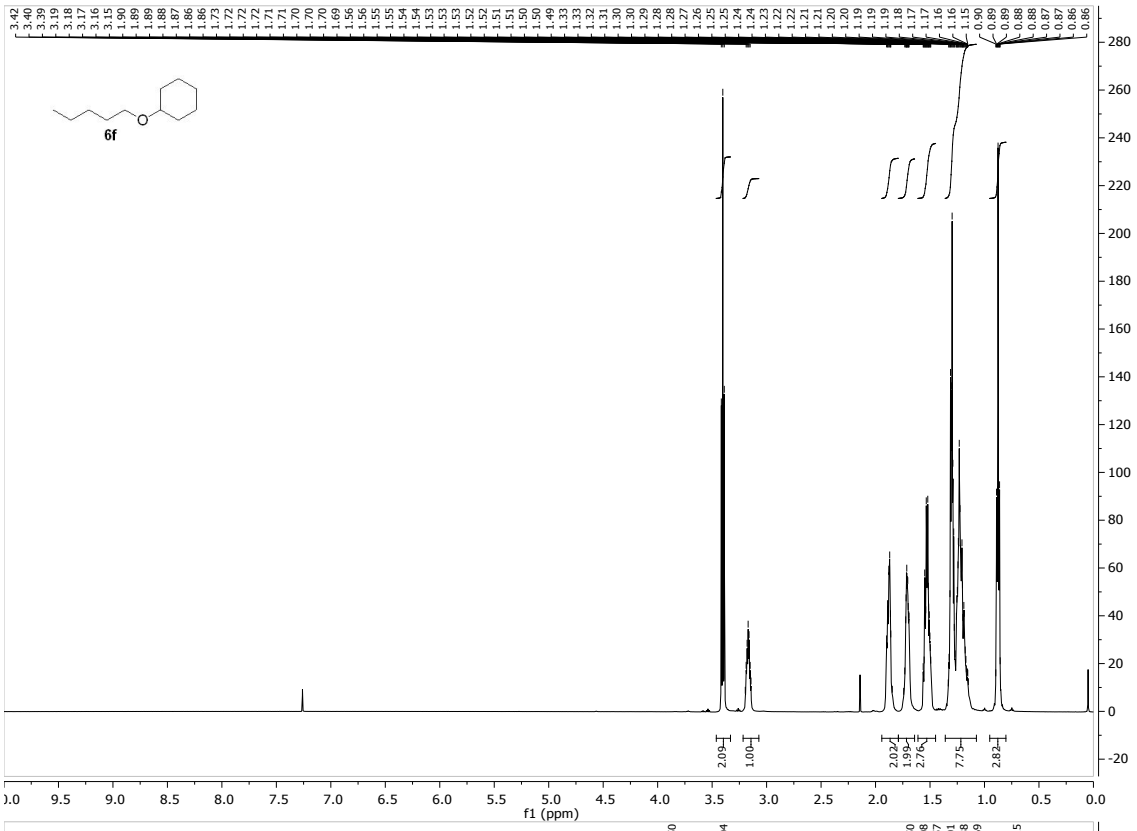

F
O
6c
-117.67
-117.69
-117.70
-117.71
-117.73
-117.74
-117.75
1.00
f1 (ppm)

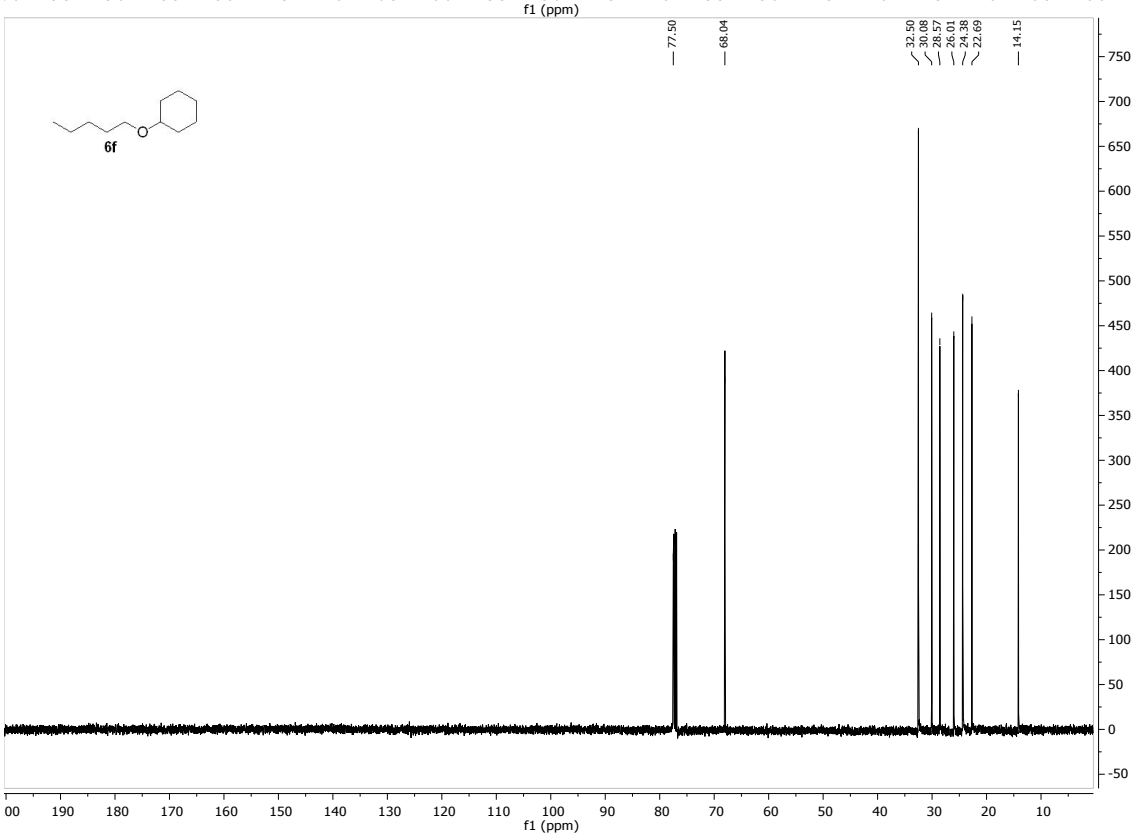

6d
f1 (ppm)

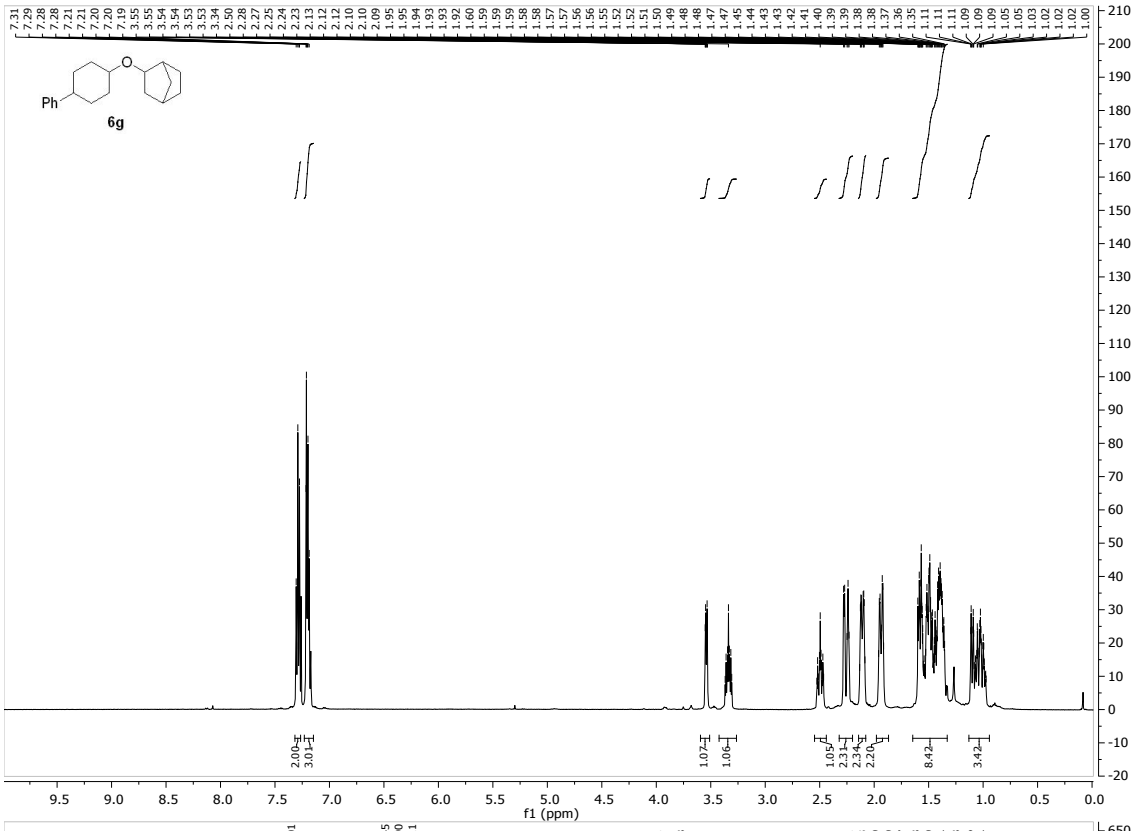

6d
f1 (ppm)

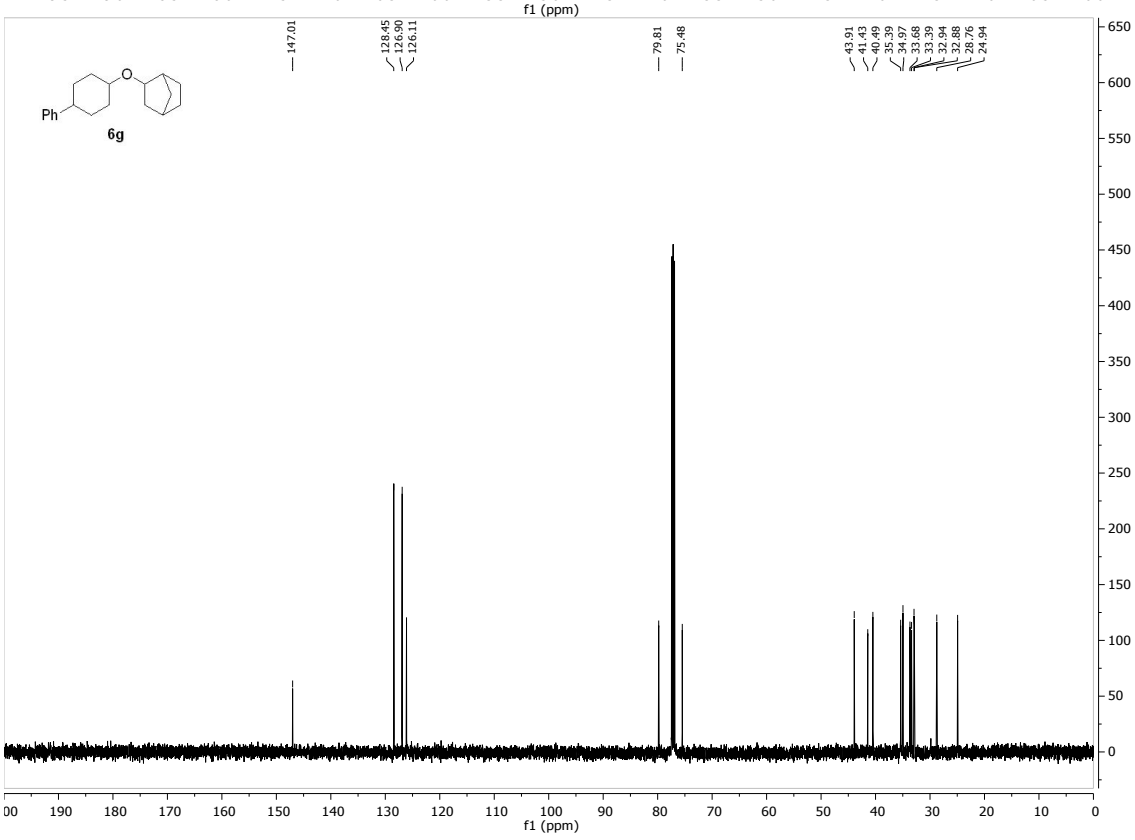

8.11
8.10
7.39
7.37
3.68
3.67
3.66
3.22
3.21
3.20
3.19
3.18
2.95
2.94
2.92
1.84
1.83
1.82
1.81
1.80
1.68
1.67
1.66
1.65
1.49
1.49
1.48
1.25
1.25
1.23
1.21
1.19
1.18
1.17
NO2
O
6e
2.00
2.03
2.11
1.00
2.07
2.14
2.23
0.90
5.25
f1 (ppm)

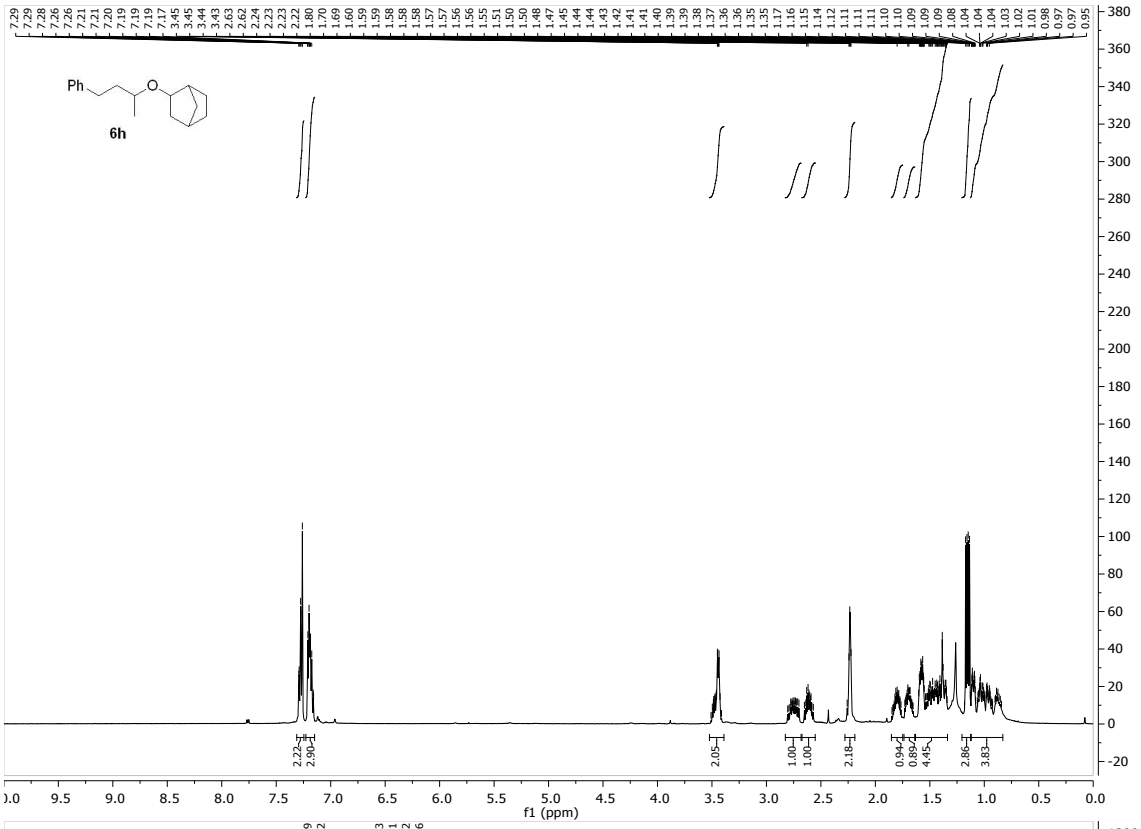

147.78
146.57
129.84
123.44
77.76
67.72
36.72
32.19
25.83
24.05
NO2
O
6e
f1 (ppm)

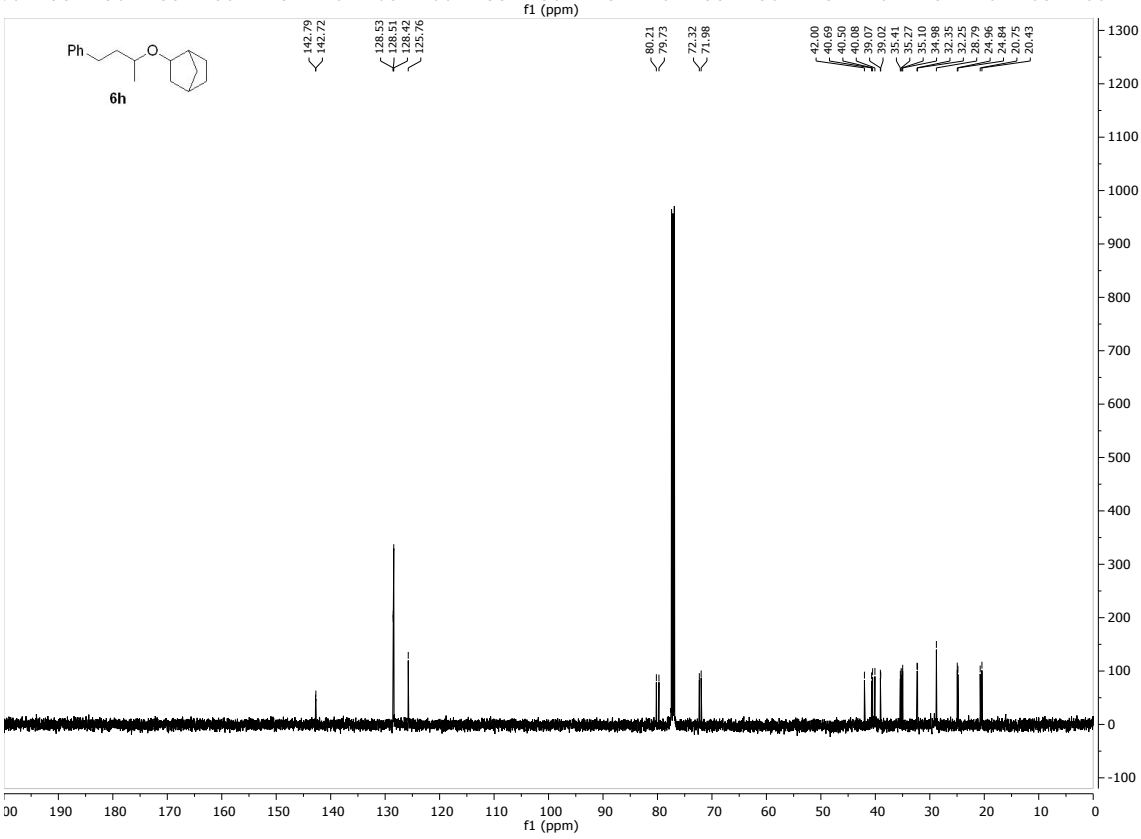

6f
2.09
1.00
2.02
1.99
2.76
7.75
2.82
f1 (ppm)

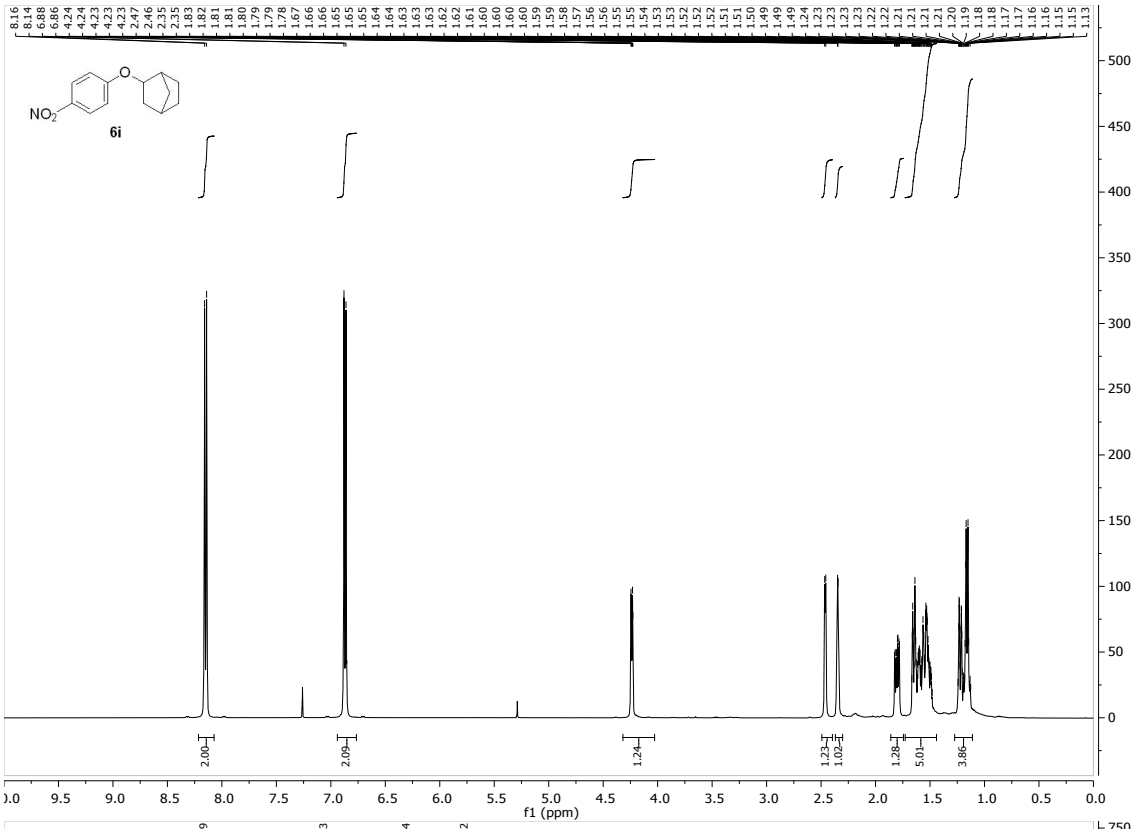

6f
77.50
68.04
32.50
30.08
28.57
26.01
24.38
22.69
14.15
f1 (ppm)

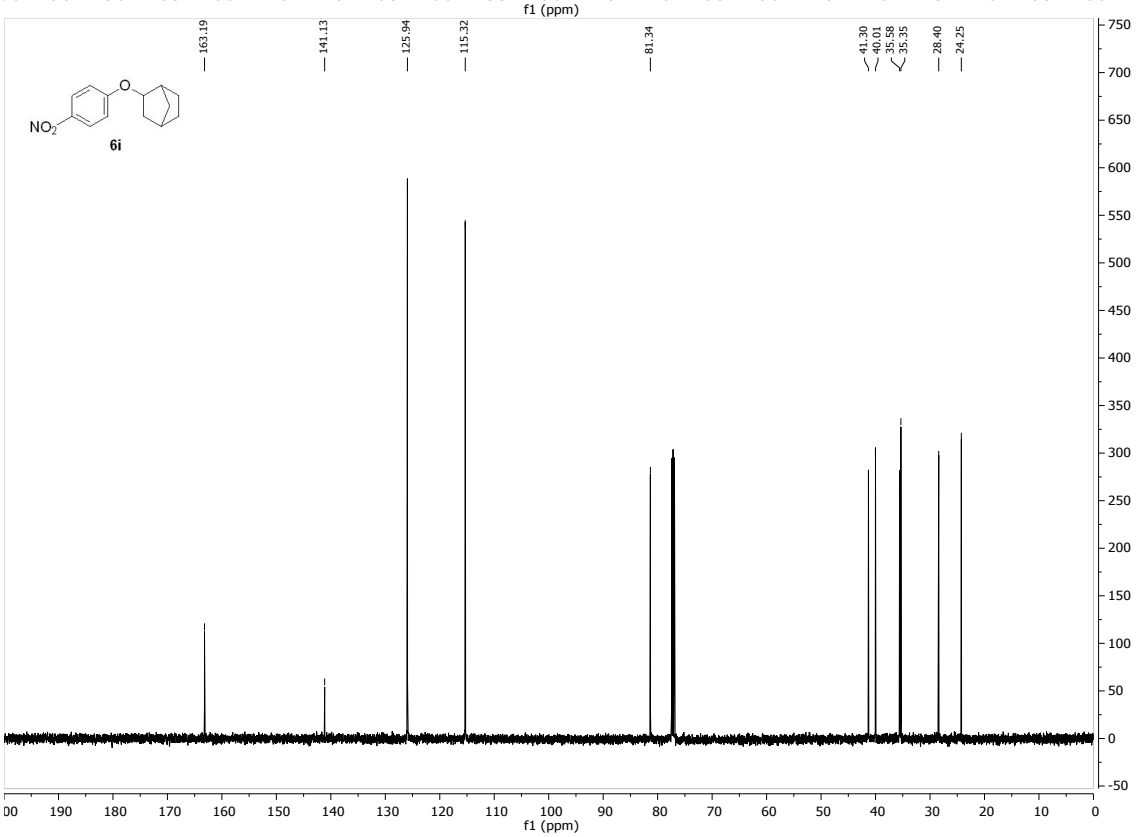

Ph
6g
f1 (ppm)

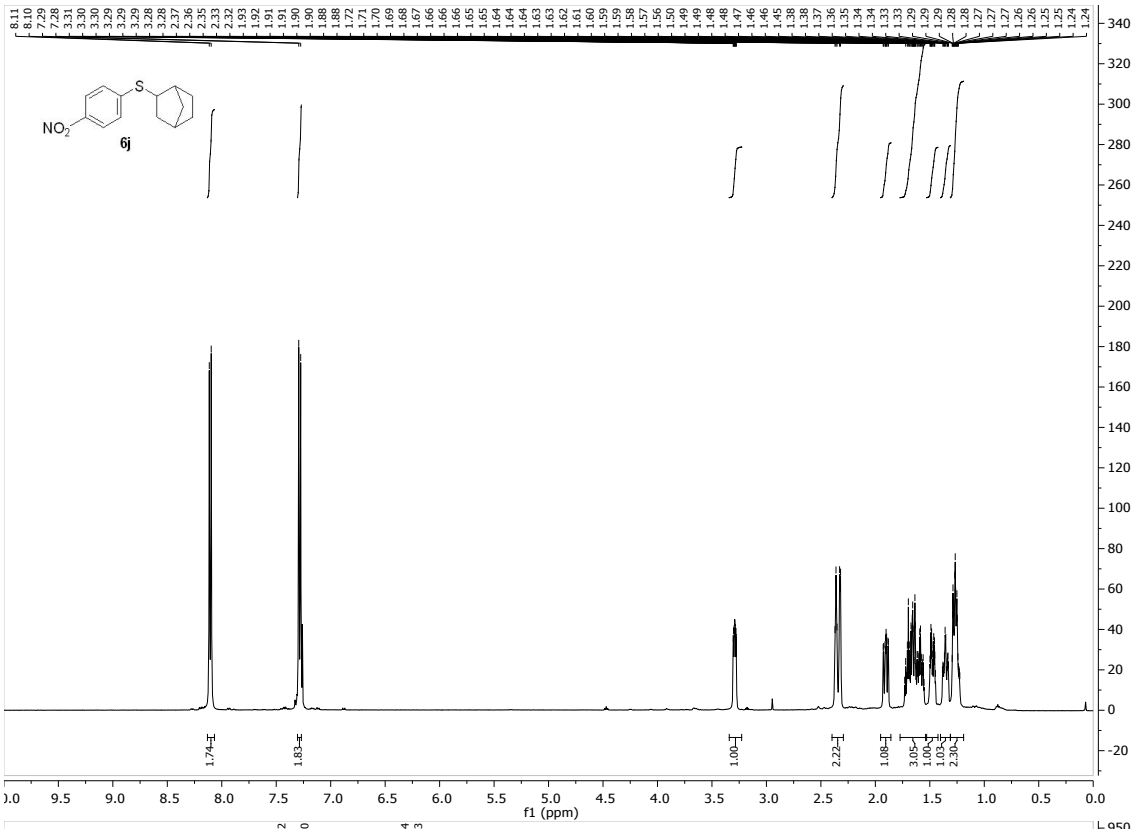

Ph
6g
147.01
128.45
126.90
126.11
79.81
75.48
43.91
41.43
40.49
35.39
34.97
33.68
33.39
32.94
32.88
28.76
24.94
f1 (ppm)

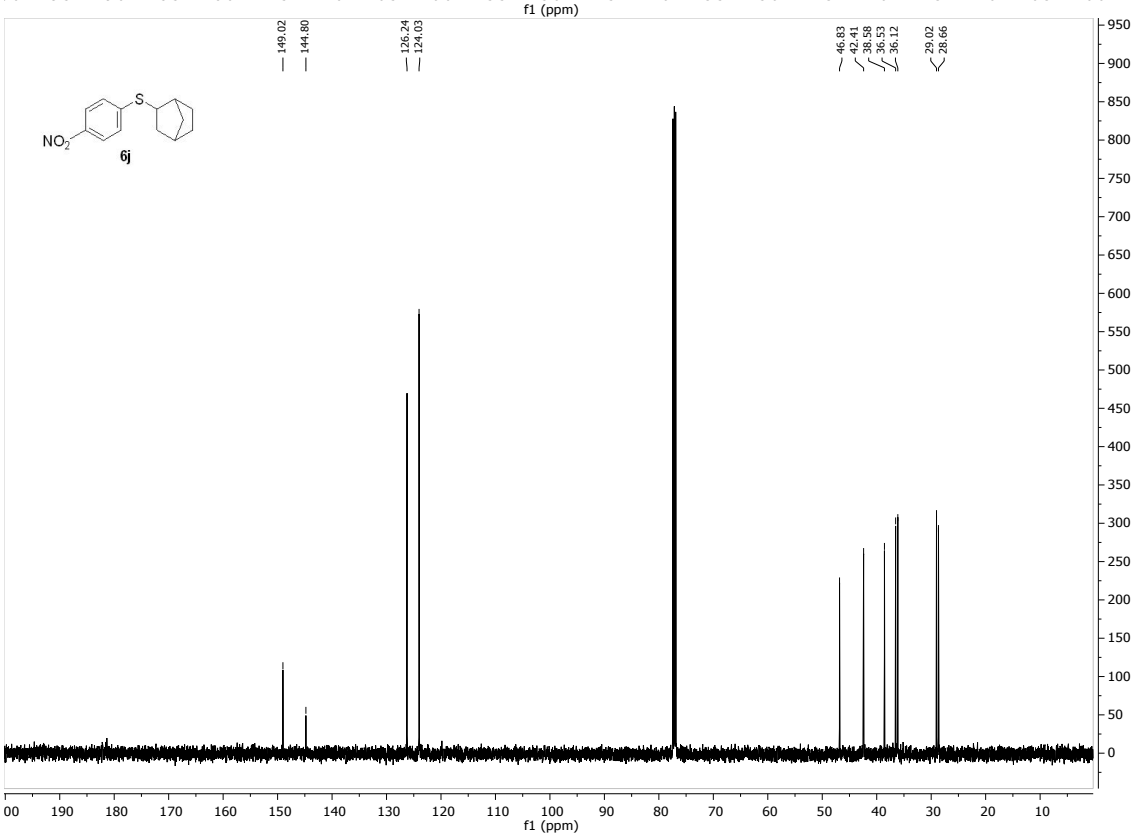

Ph
O
6h
f1 (ppm)

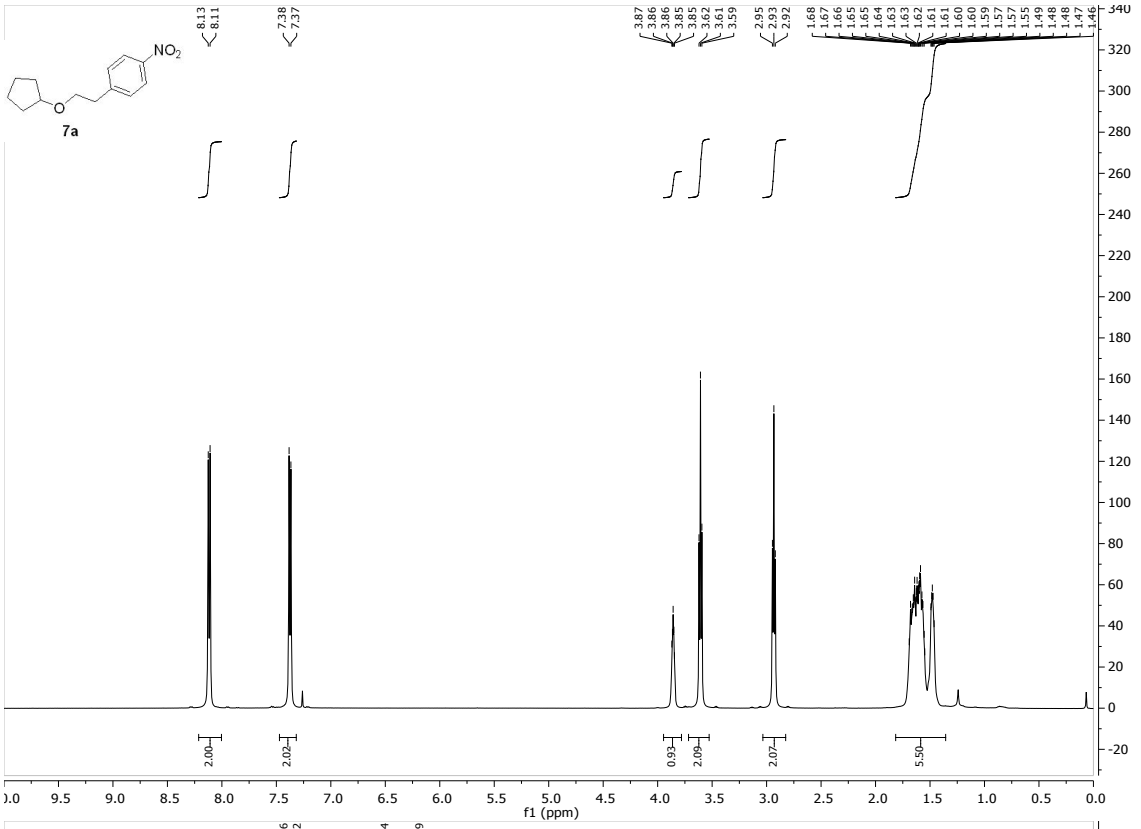

Ph
O
6h
f1 (ppm)

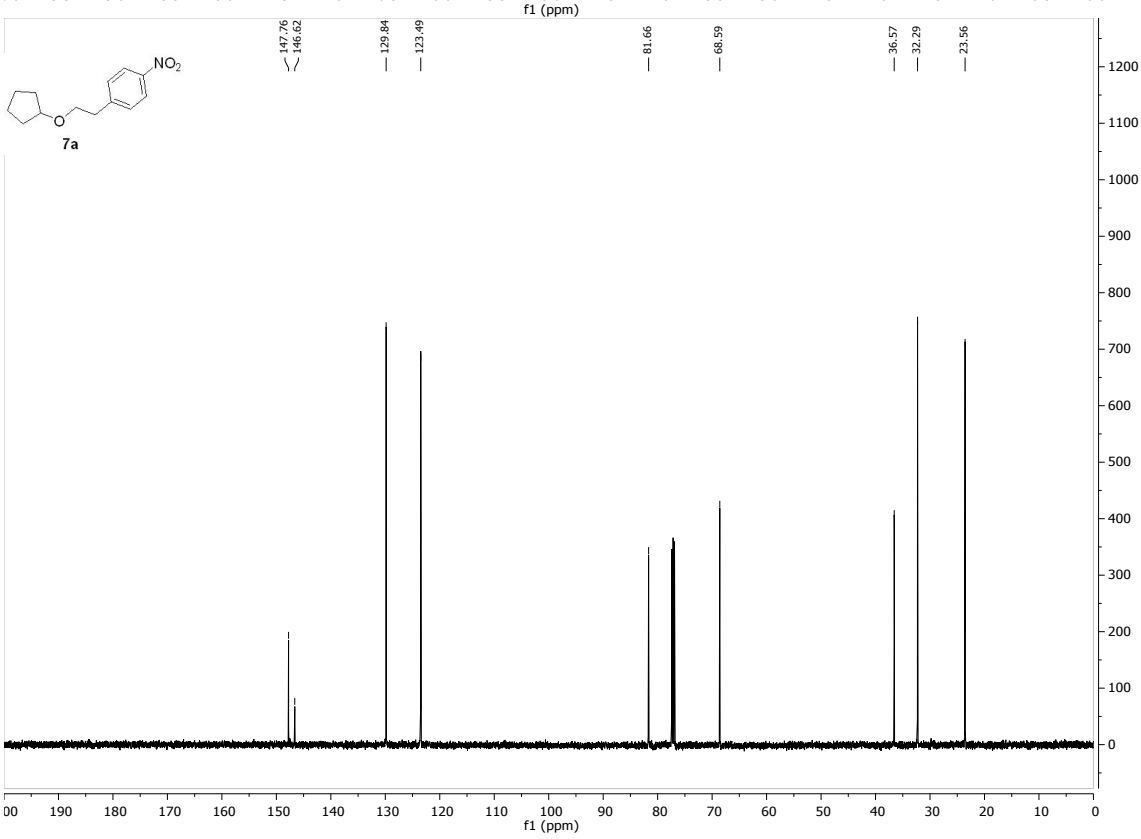

NO2
O
6i
f1 (ppm)


NO2
O
6i
163.19
141.13
125.94
115.32
81.34
41.30
40.01
35.58
35.35
28.40
24.25
f1 (ppm)


NO2
S
6j
1.74
1.83
1.00
2.22
1.08
3.05
1.06
1.03
2.30
f1 (ppm)


149.02
144.80
126.24
124.03
46.83
42.41
38.58
36.53
36.12
29.02
28.66
NO2
S
6j
f1 (ppm)


8.13
8.11
7.38
7.37
3.87
3.86
3.86
3.85
3.85
3.62
3.61
3.59
2.95
2.93
2.92
1.68
1.67
1.66
1.65
1.65
1.64
1.63
1.63
1.62
1.61
1.61
1.60
1.60
1.59
1.57
1.57
1.55
1.49
1.48
1.48
1.47
1.46
NO2
O
7a
2.00
2.02
0.93
2.09
2.07
5.50
f1 (ppm)


147.76
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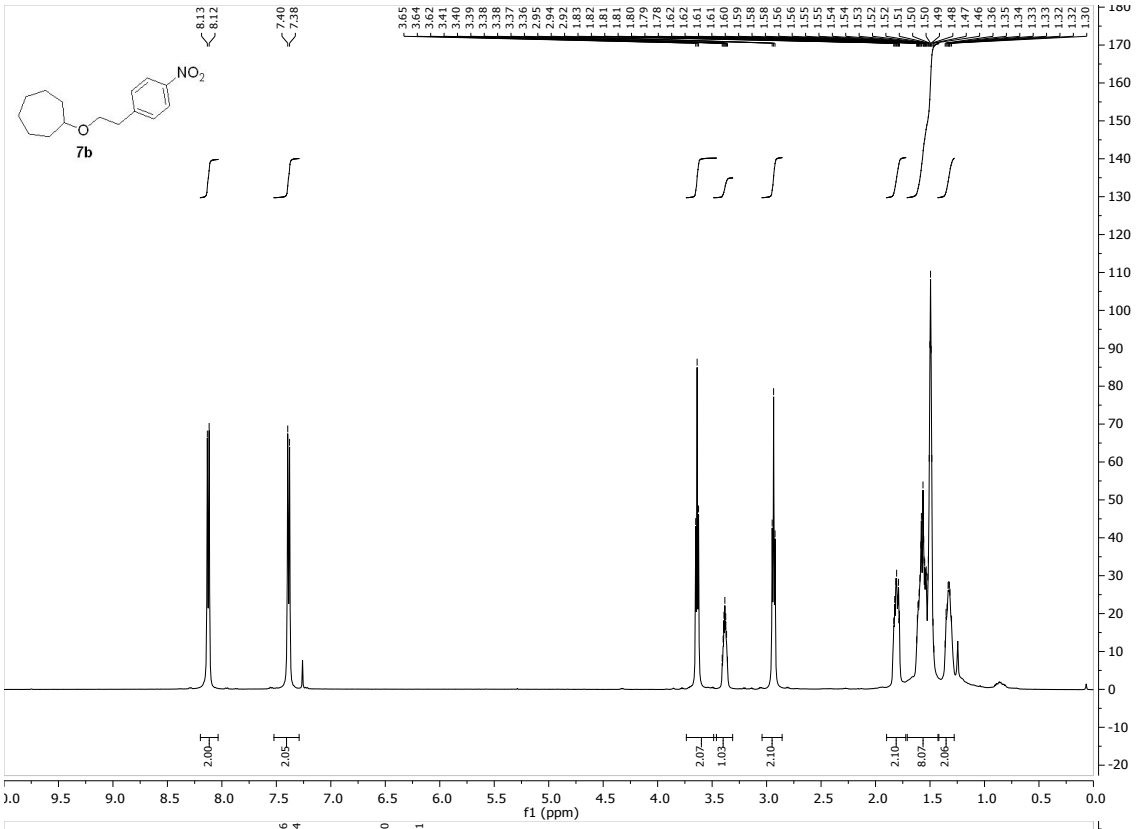
123.49
81.66
68.59
36.57
32.29
23.56
NO2
O
7a
f1 (ppm)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7.40
7.38
3.65
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3.62
3.41
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3.39
3.38
3.37
3.36
2.95
2.94
2.92
1.83
1.82
1.81
1.81
1.80
1.79
1.78
1.62
1.62
1.61
1.61
1.60
1.59
1.58
1.58
1.56
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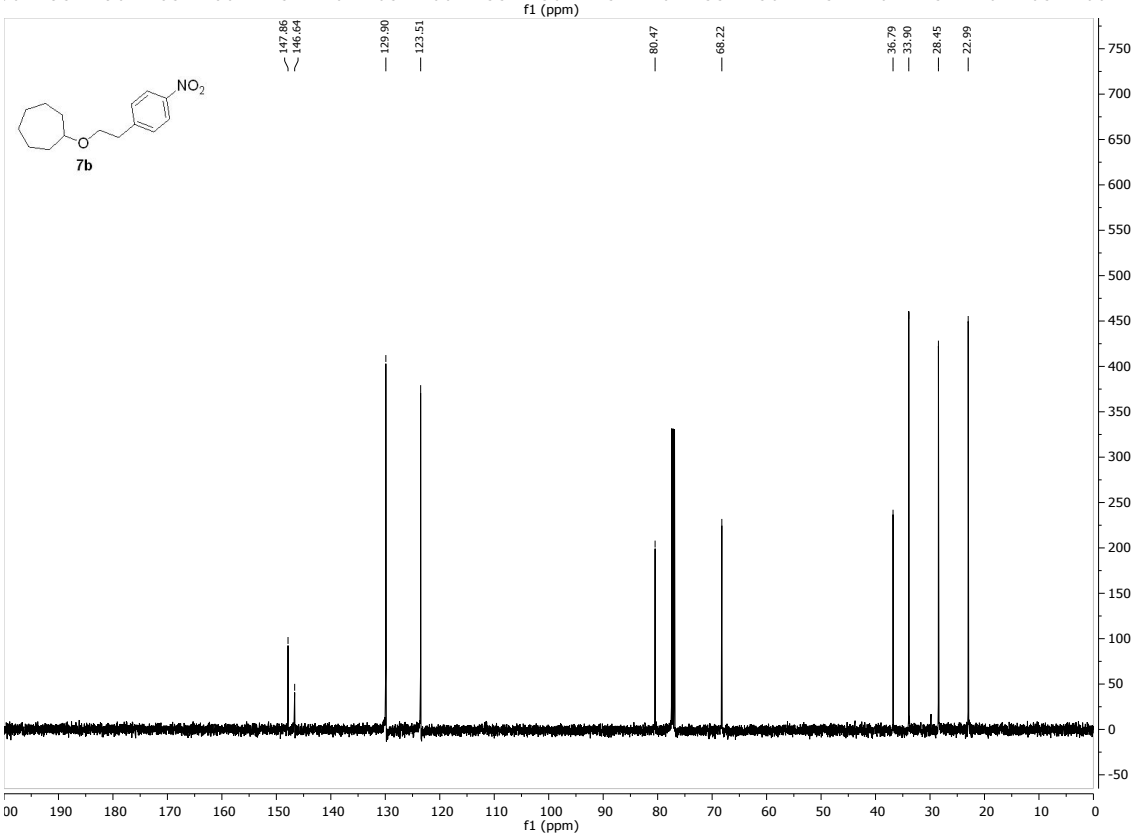
1.51
1.50
1.50
1.49
1.48
1.47
1.46
1.36
1.35
1.34
1.33
1.33
1.32
1.32
1.30
NO2
O
7b
2.00
2.05
2.07
1.03
2.10
2.10
8.07
2.06
f1 (ppm)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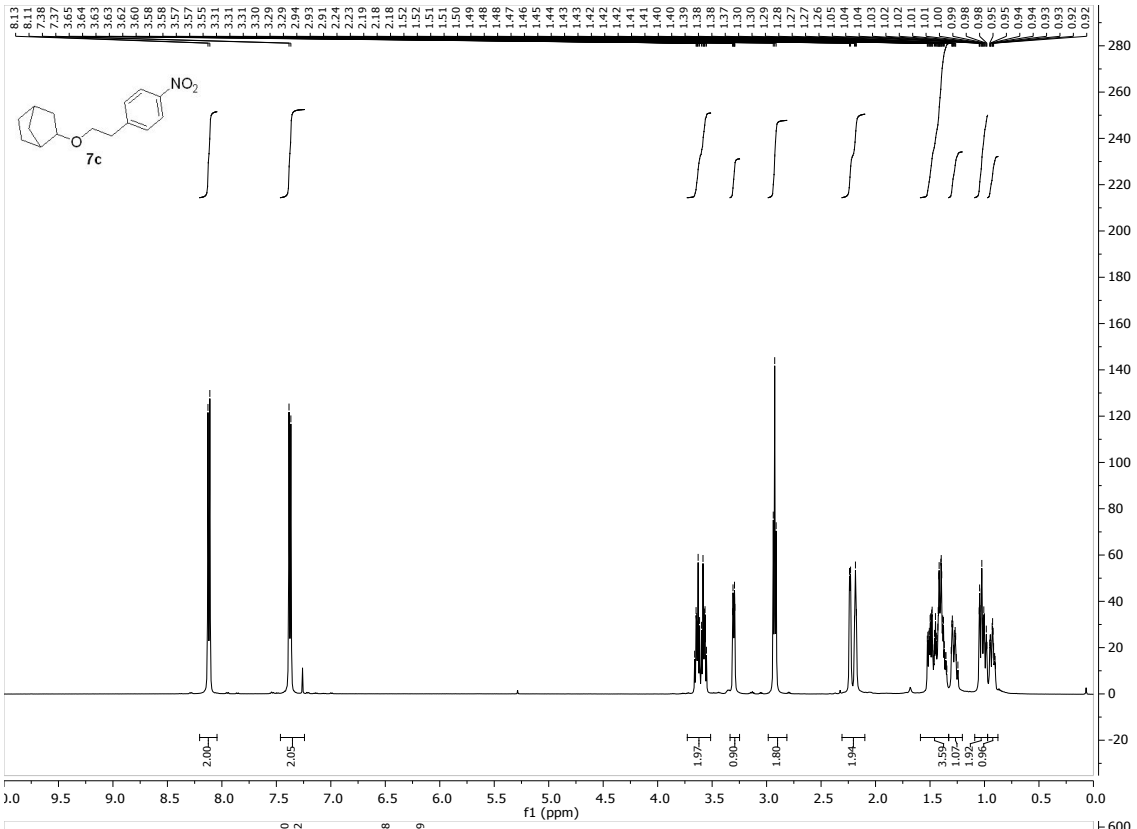
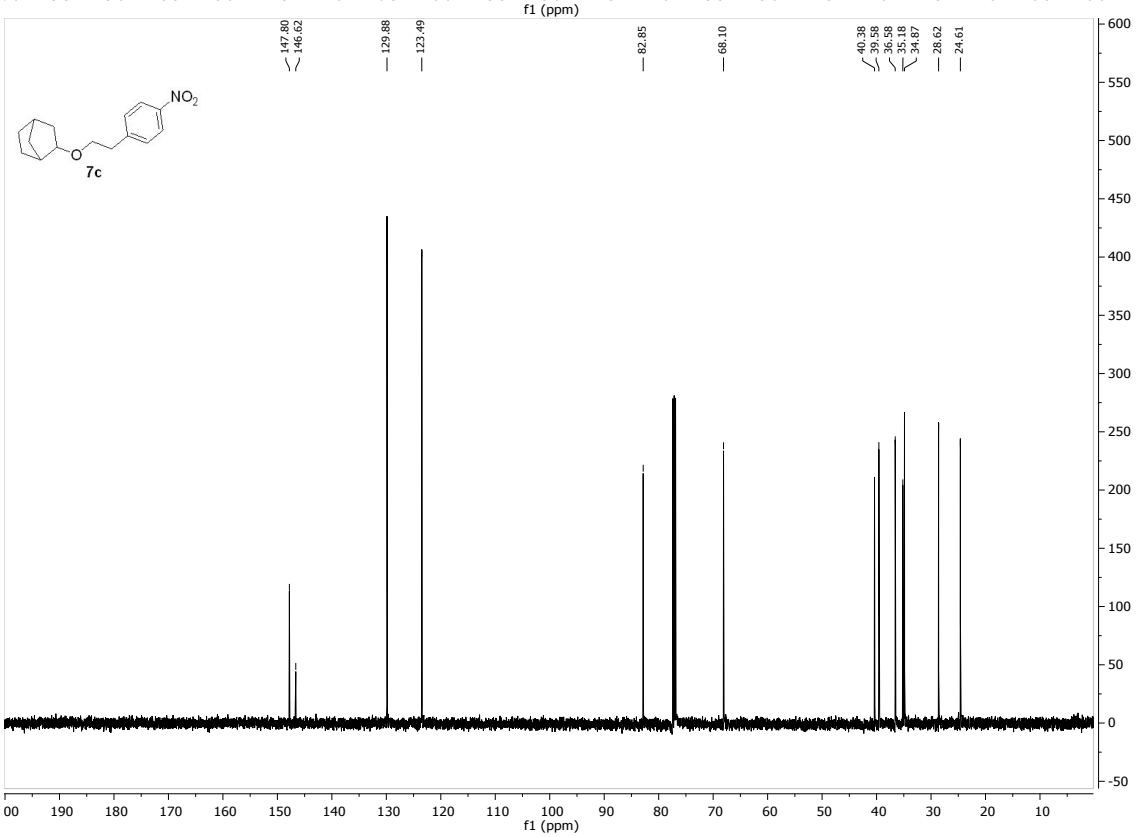
146.64
129.90
123.51
80.47
68.22
36.79
33.90
28.45
22.99
NO2
O
7b
f1 (ppm)


8.13 8.11 7.38 7.37 3.65 3.64 3.63 3.63 3.62 3.60 3.58 3.58 3.57 3.57 3.55 3.31 3.31 3.31 3.30 3.29 3.29 2.94 2.93 2.91 2.24 2.23 2.19 2.18 2.18 1.52 1.52 1.51 1.51 1.50 1.49 1.48 1.48 1.47 1.46 1.45 1.44 1.43 1.43 1.42 1.42 1.42 1.41 1.41 1.40 1.40 1.39 1.38 1.38 1.37 1.30 1.30 1.29 1.28 1.27 1.27 1.26 1.05 1.04 1.04 1.03 1.02 1.02 1.01 1.01 1.00 0.99 0.98 0.98 0.95 0.95 0.94 0.94 0.93 0.93 0.92 0.92
NO2
O
7c
2.00
2.05
1.97
0.90
1.80
1.94
3.59
1.07
1.92
0.96
f1 (ppm)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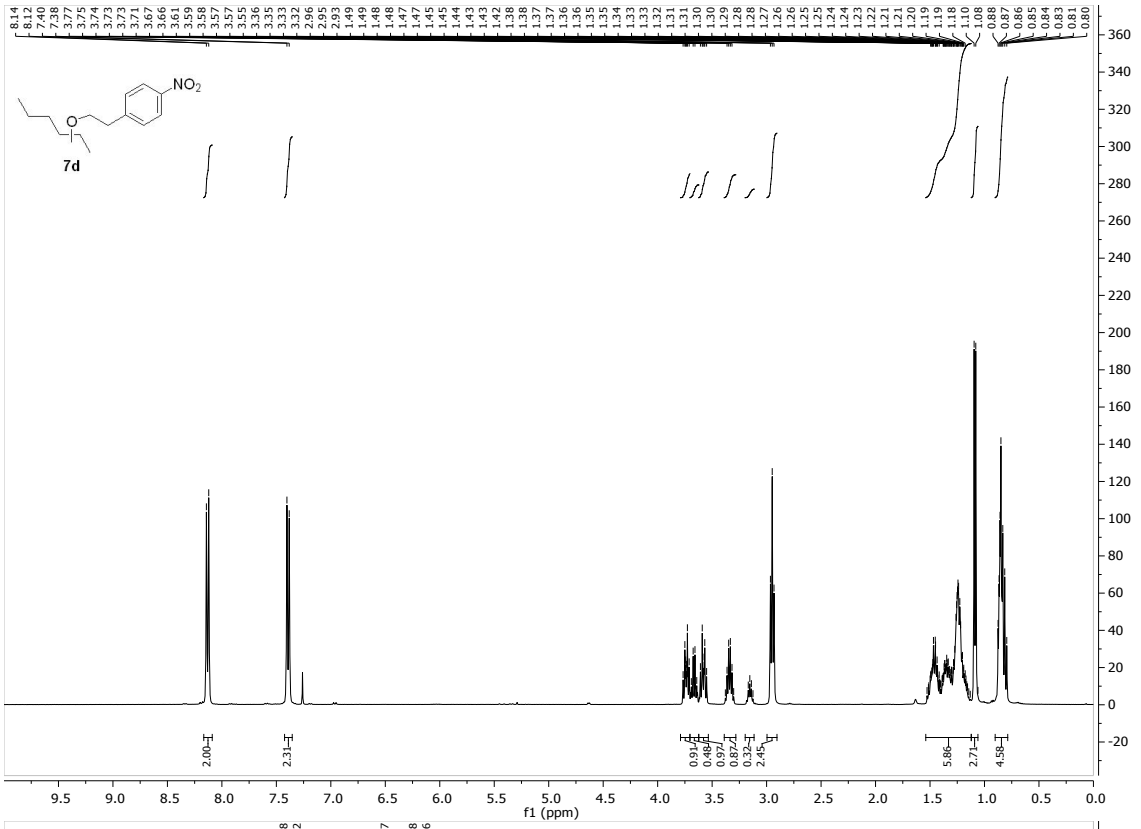
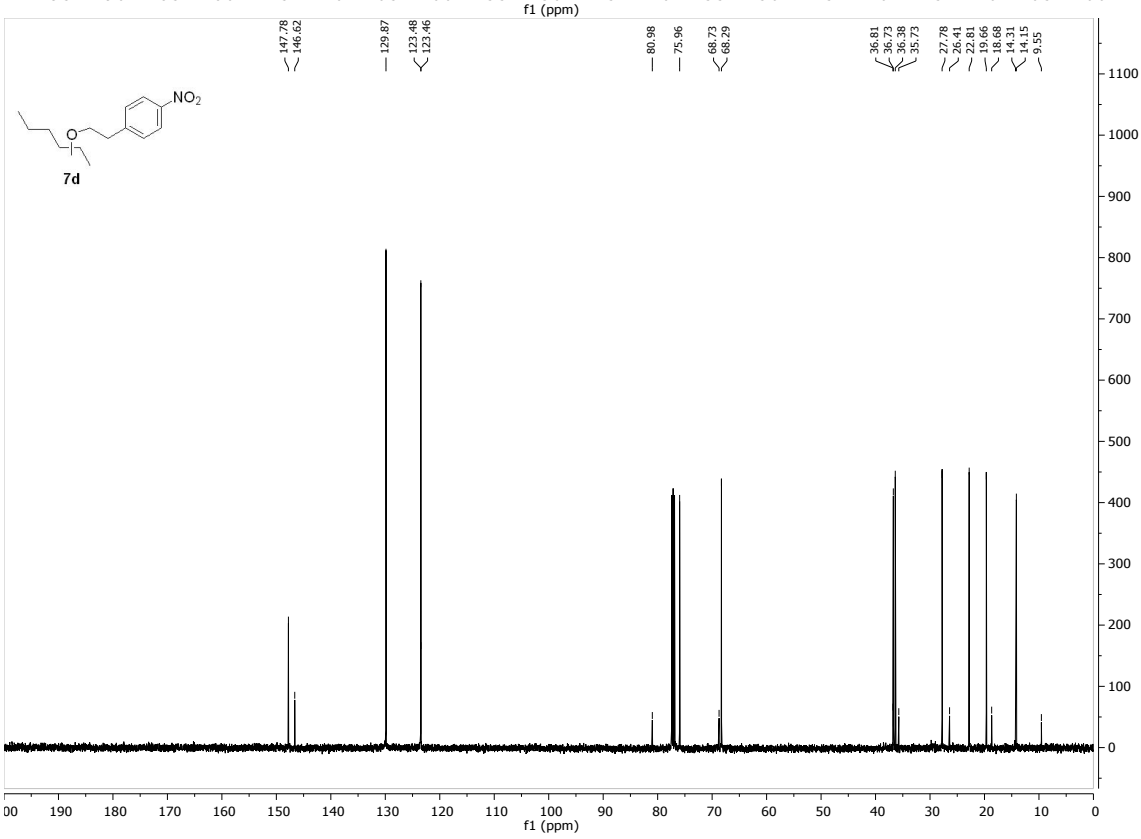
146.62
129.88
123.49
82.85
68.10
40.38
39.58
36.58
35.18
34.87
28.62
24.61
NO2
O
7c
f1 (ppm)


NO2
O
7d
f1 (ppm)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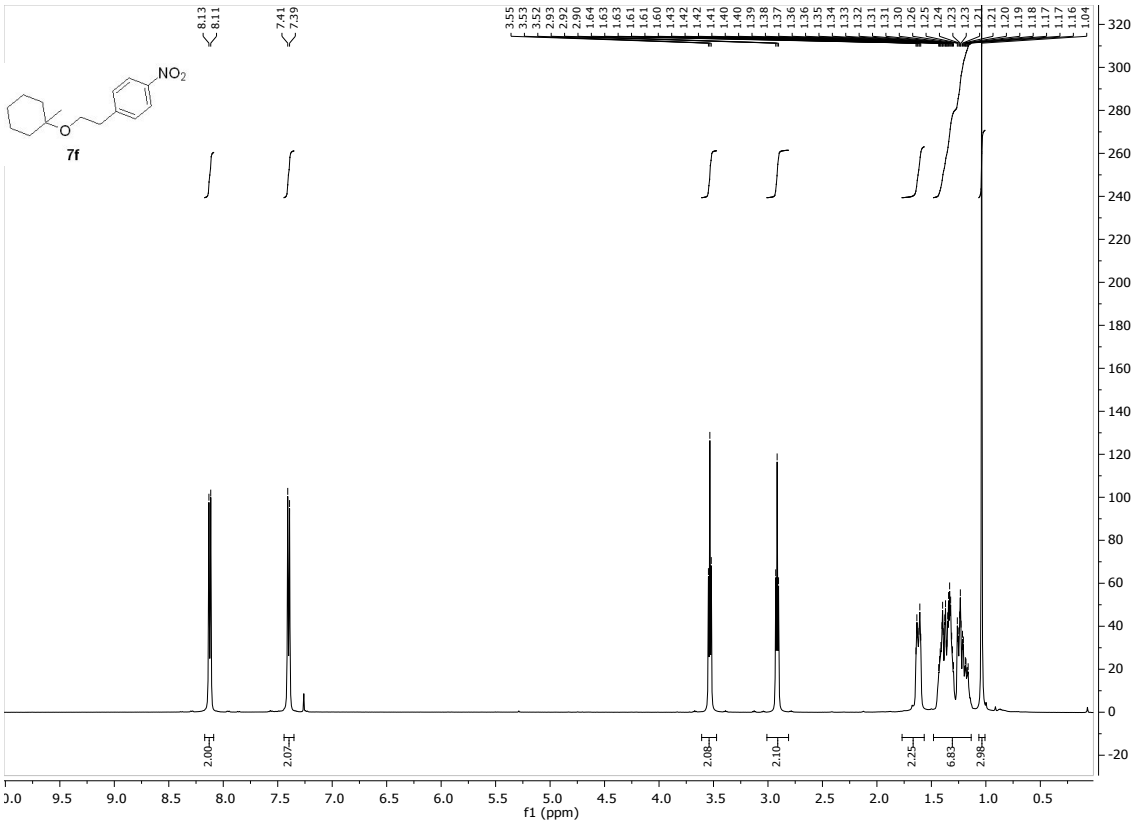
O
7d
f1 (ppm)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7.41
7.39
3.55
3.53
3.52
2.93
2.92
2.90
1.64
1.63
1.63
1.61
1.61
1.60
1.43
1.42
1.42
1.41
1.40
1.40
1.39
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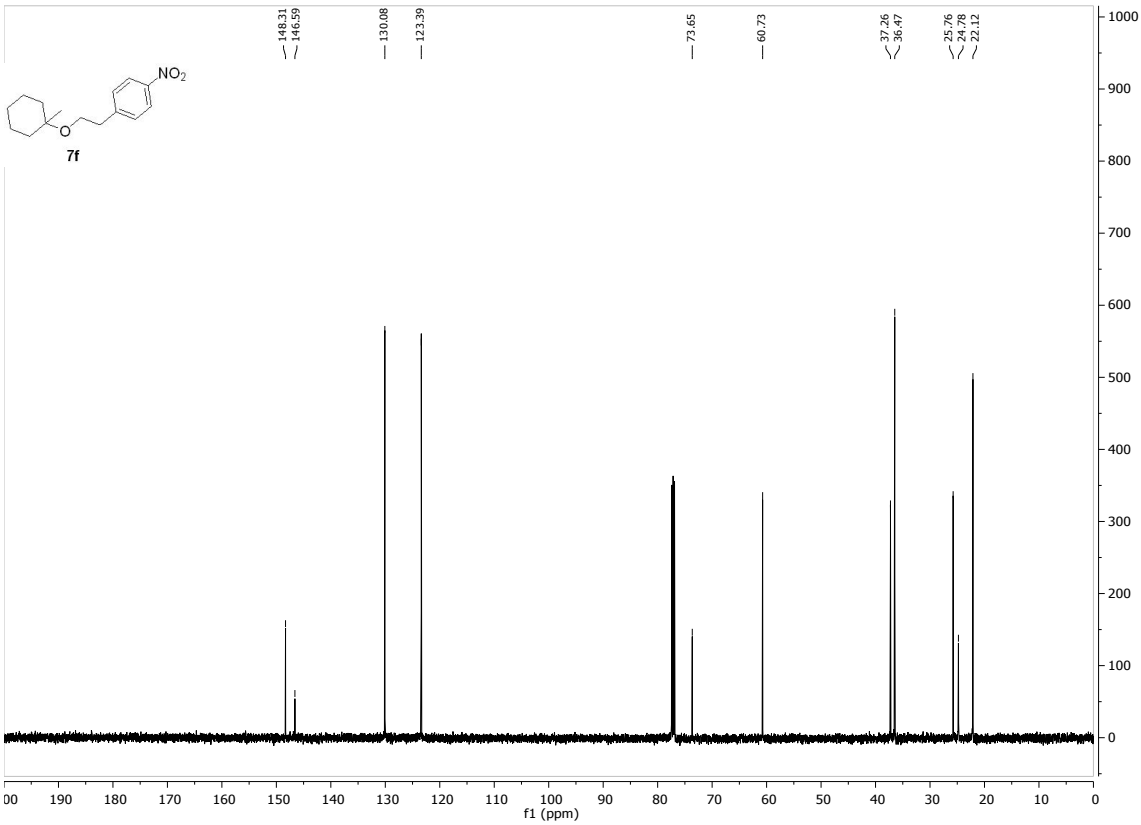
1.33
1.32
1.31
1.31
1.30
1.26
1.25
1.24
1.23
1.23
1.21
1.21
1.20
1.19
1.18
1.17
1.17
1.16
1.04
NO2
O
7f
2.00
2.07
2.08
2.10
2.25
6.83
2.98
f1 (ppm)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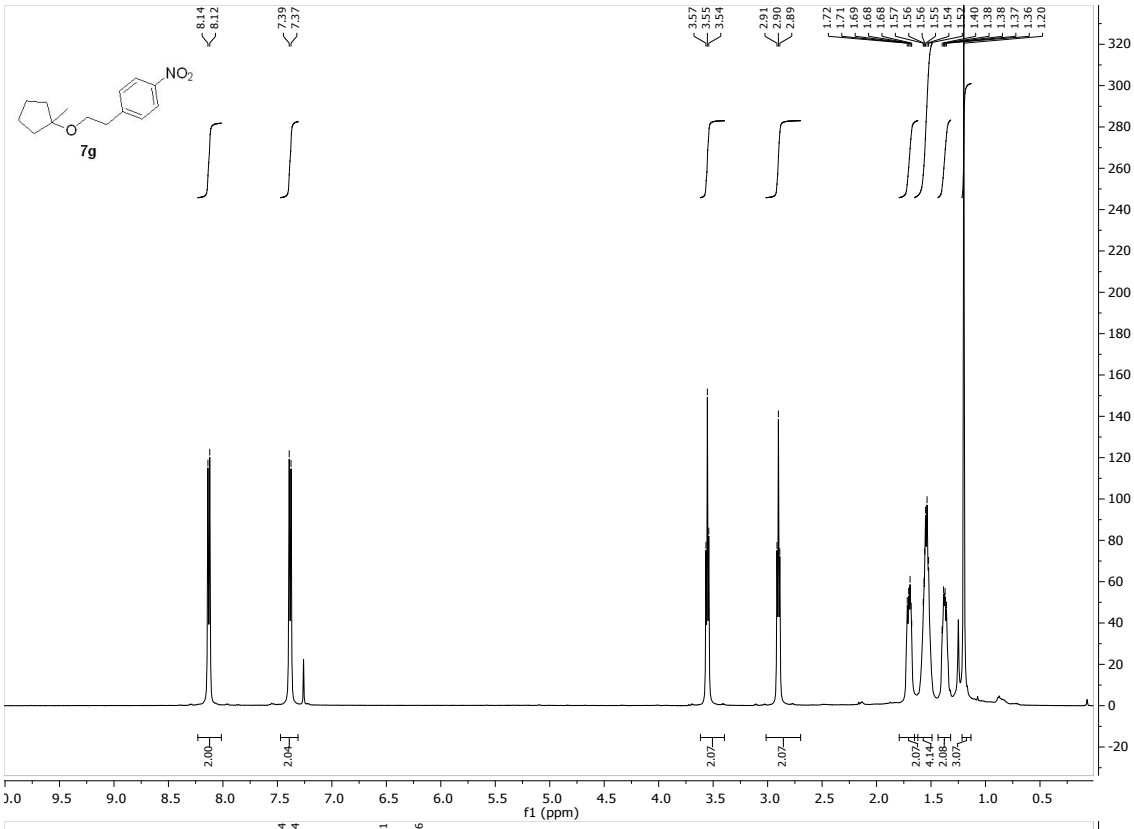
123.39
73.65
60.73
37.26
36.47
25.76
24.78
22.12
NO2
O
7f
f1 (ppm)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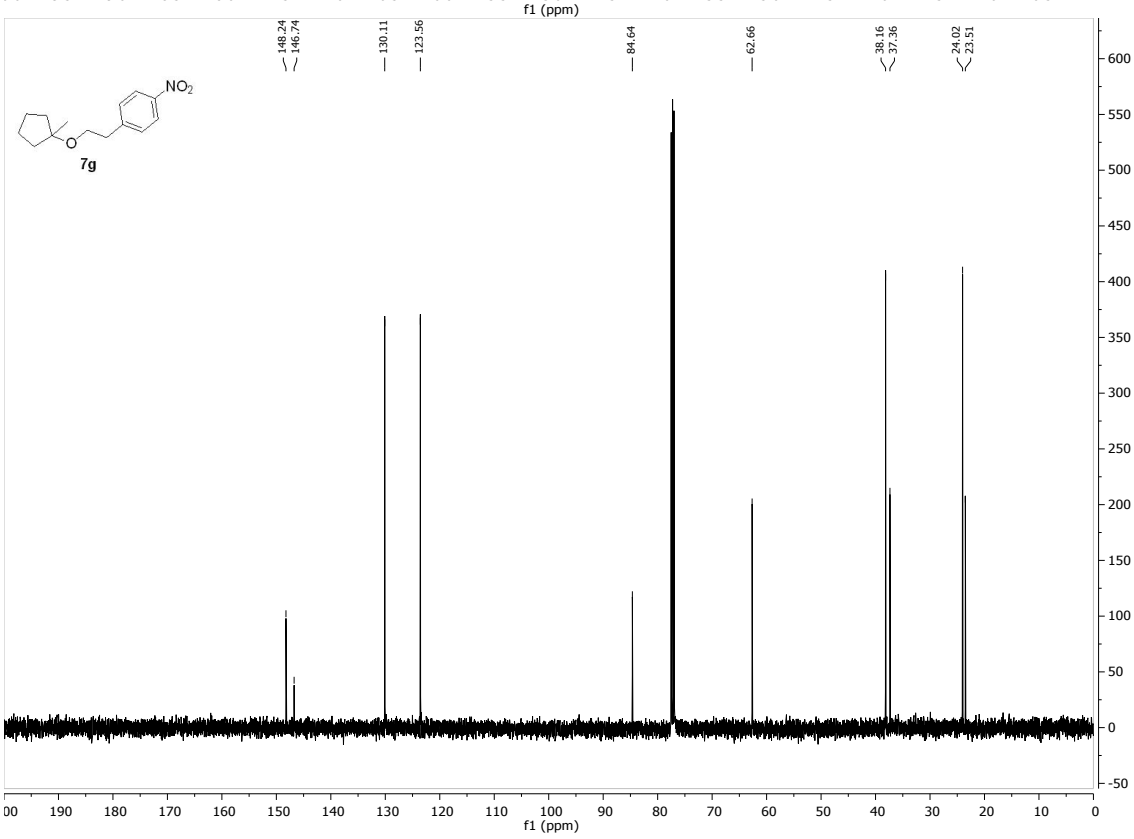
8.12
7.39
7.37
3.57
3.55
3.54
2.91
2.90
2.89
1.72
1.71
1.69
1.68
1.68
1.57
1.56
1.56
1.55
1.54
1.52
1.40
1.38
1.38
1.37
1.36
1.20
NO2
O
7g
2.00
2.04
2.07
2.07
2.07
4.14
2.08
3.07
f1 (ppm)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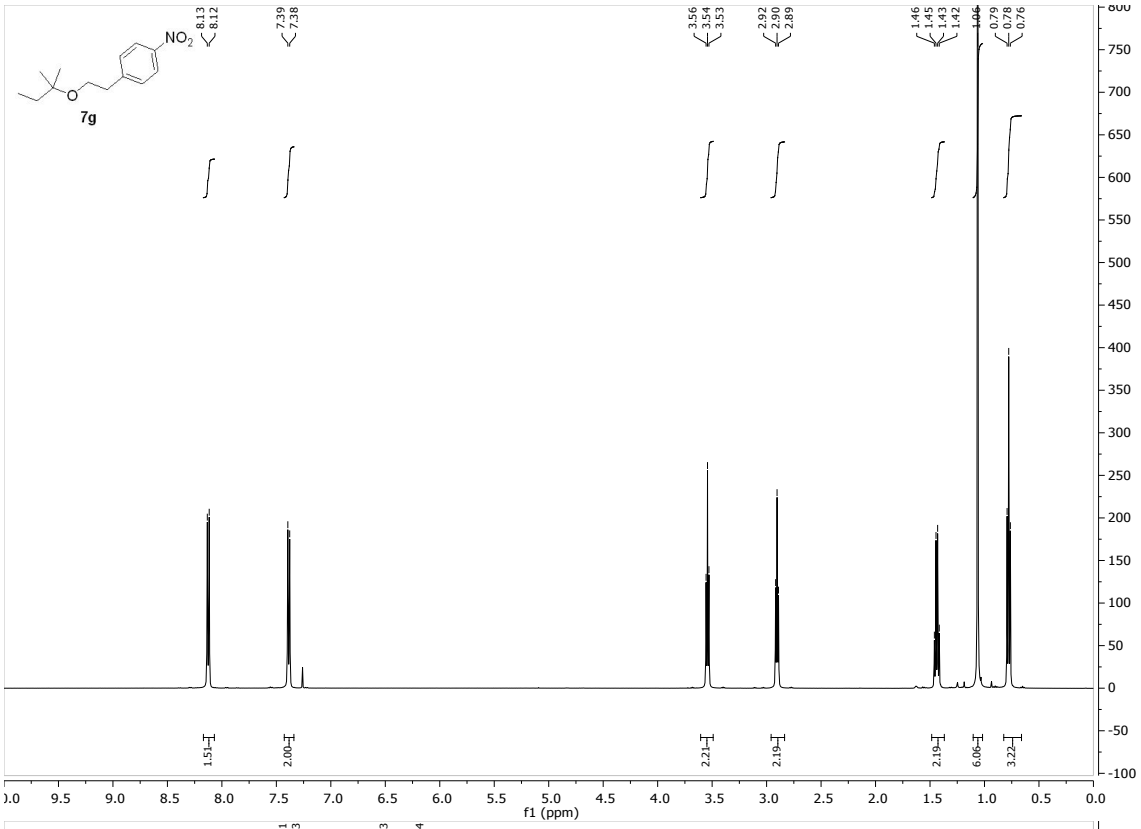
62.66
38.16
37.36
24.02
23.51
NO2
O
7g
f1 (ppm)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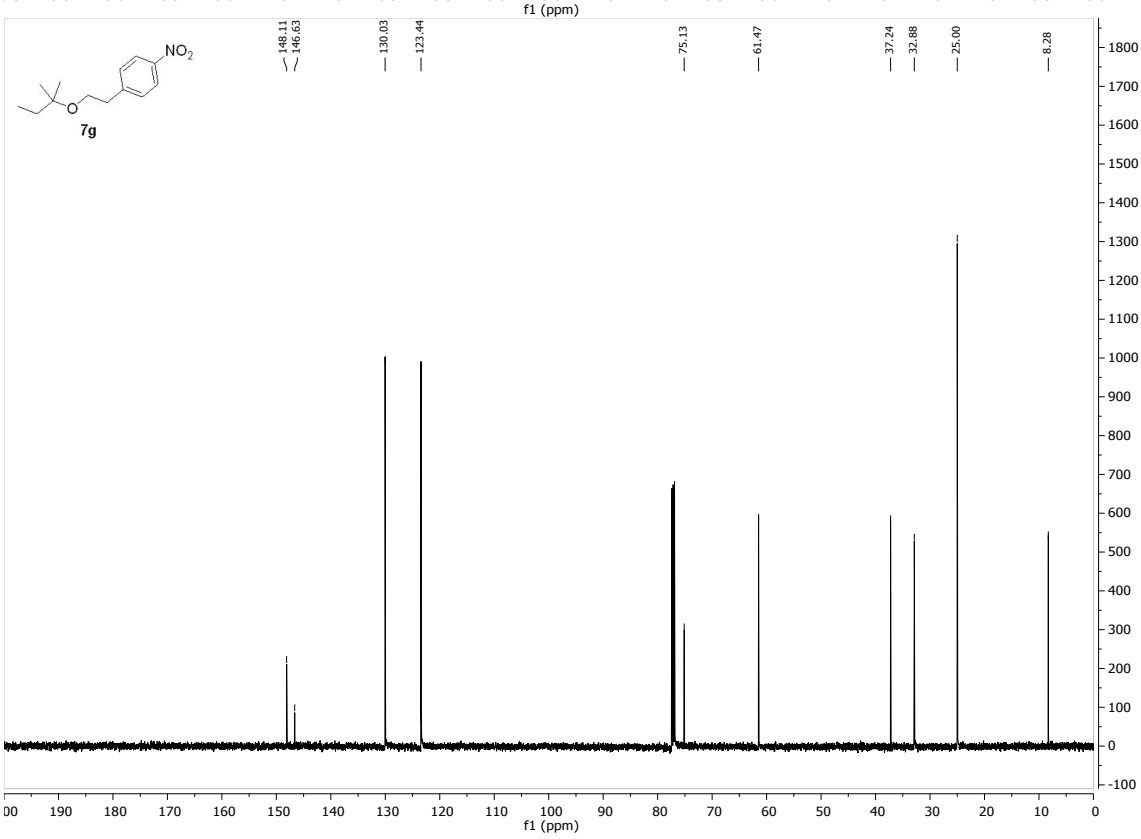
8.12
7.39
7.38
3.56
3.54
3.53
2.92
2.90
2.89
1.46
1.45
1.43
1.42
1.06
0.79
0.78
0.76
NO2
O
7g
1.51
2.00
2.21
2.19
2.19
6.06
3.22
f1 (ppm)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61.47
37.24
32.88
25.00
8.28
NO2
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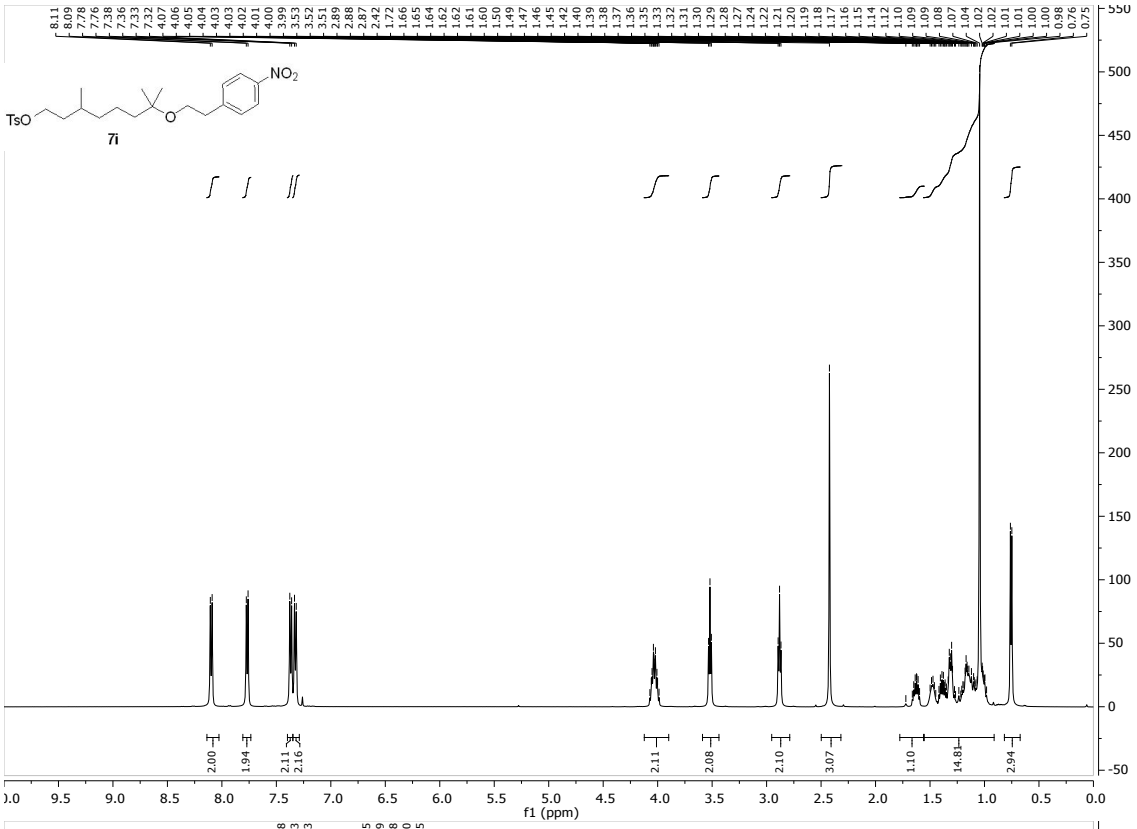
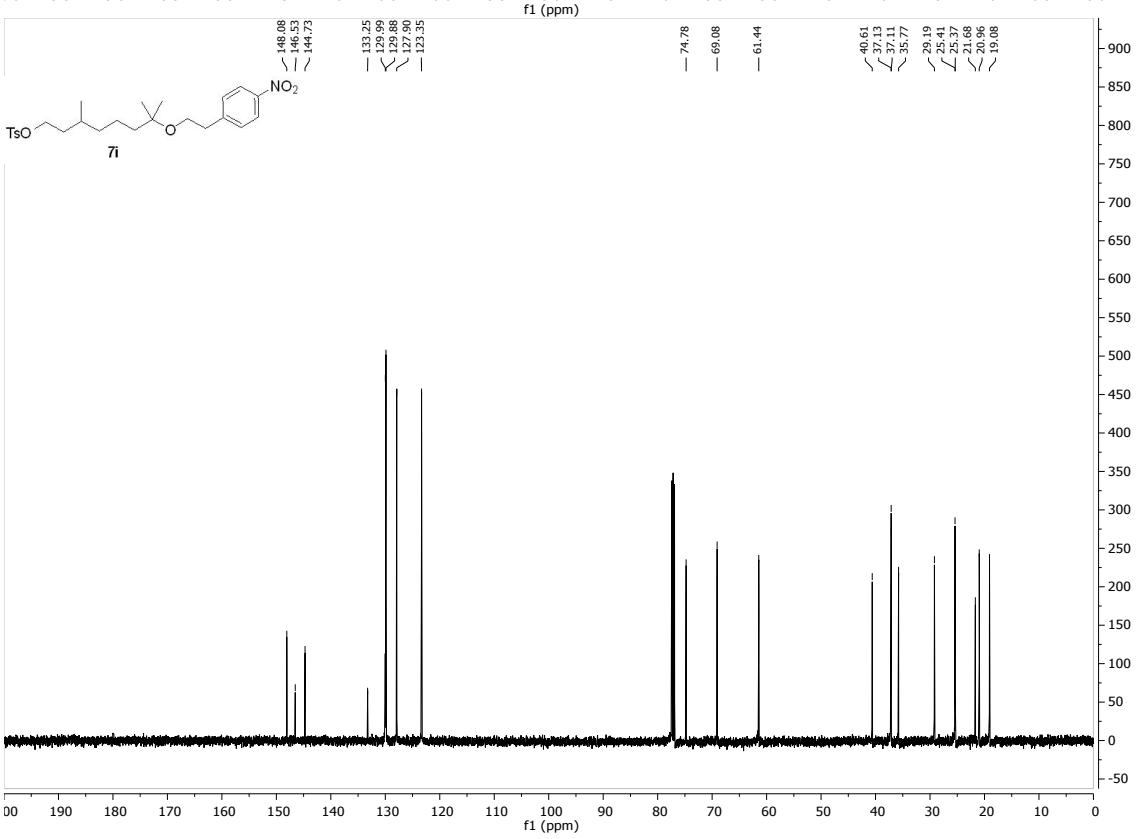
7g
f1 (ppm)


NO2
TsO
O
7i
f1 (ppm)


NO2
TsO
O
7i
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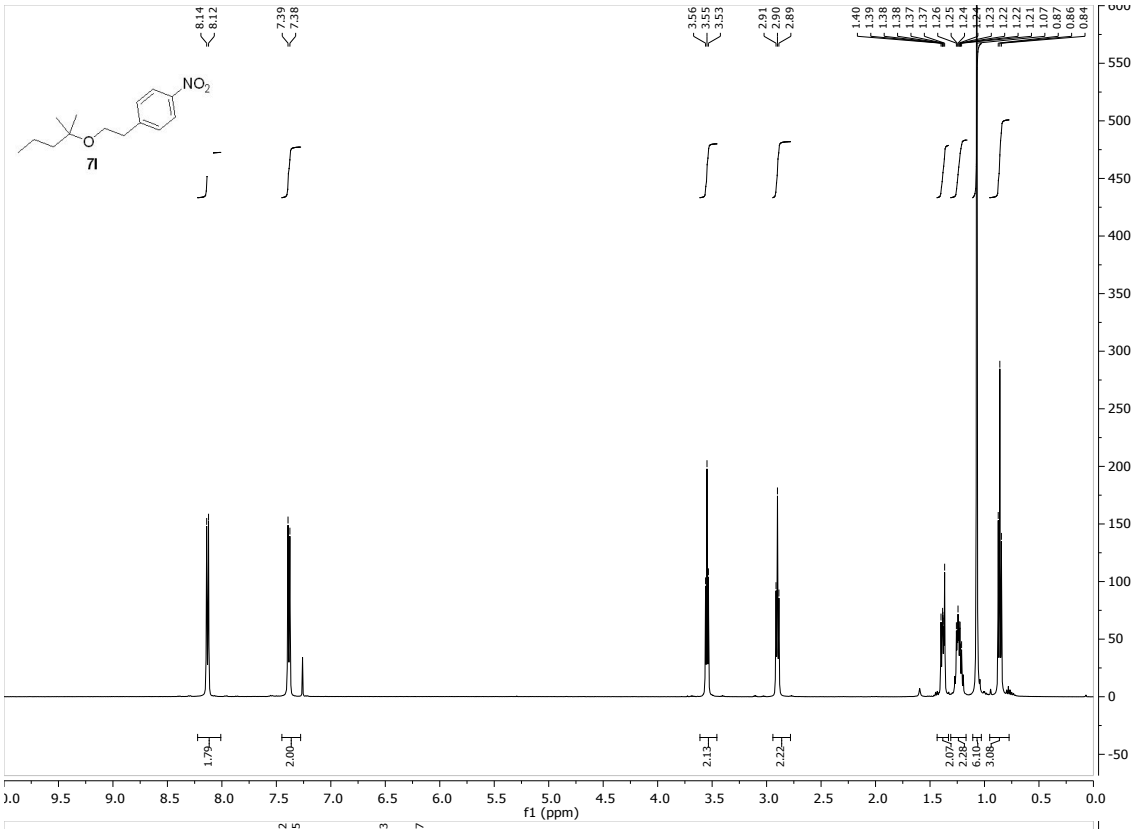
144.73
133.25
129.99
129.88
127.90
123.35
74.78
69.08
61.44
40.61
37.13
37.11
35.77
29.19
25.41
25.37
21.68
20.96
19.08
f1 (ppm)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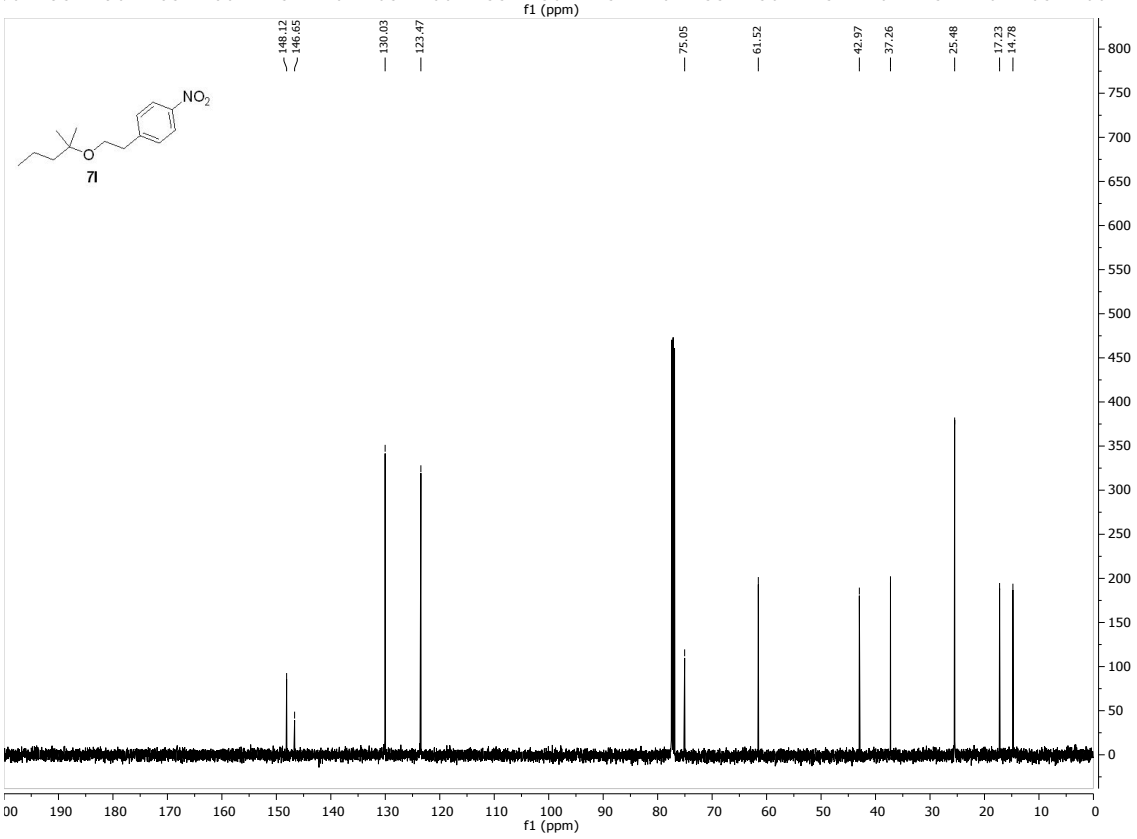
1.38
1.38
1.37
1.37
1.26
1.25
1.24
1.24
1.23
1.22
1.22
1.21
1.07
0.87
0.86
0.84
NO2
O
7l
1.79
2.00
2.13
2.22
2.07
2.28
6.10
3.08
f1 (ppm)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123.47
75.05
61.52
42.97
37.26
25.48
17.23
14.78
NO2
O
7l
f1 (ppm)