Supporting Information

For

Copper-Catalyzed, N-Directed Remote C(sp^3)–H Azidation and Thiocyanation

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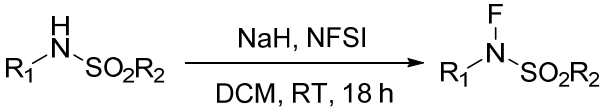
1. General remarks

^{1}H NMR spectra were recorded on 400 or 600 MHz (100 or 150 MHz for ^{13}C NMR, and 564 or 377 MHz for ^{19}F NMR) agilent NMR spectrometer with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to the $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) or 77.16 ppm (for ^{13}C NMR); ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ at δ 0.00 ppm. HRMS was recorded on an Agilent 6540 Q-TOF (ESI) Mass Spectrometer. Infrared (FT-IR) spectra were recorded on a Varian 1000FT-IR, ν_{max} in cm^{-1}. Melting points were measured using SGW, X-4B and values are uncorrected. All commercially available reagents and solvents were used as received unless otherwise specified.

2. General procedure for synthesis of substrates

All the *N*-fluoro-*N*-alkylsulfonamides and *N*-fluoro-*N*-alkylcarboxamides were prepared by N-fluorination of their parent sulfonamides or carboxamides according to conventional methods.

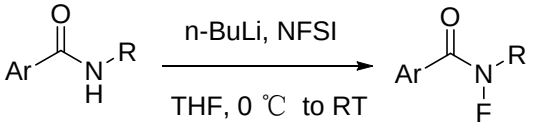
General procedure for the synthesis of *N*-fluoro-*N*-alkylsulfonamides



N-Alkylsulfonamide (10 mmol) was dissolved in dry dichloromethane (120 mL) in an oven-dried round-bottom flask. NaH (1.20 g, 30 mmol, 60% dispersion in mineral oil) was added into the solution in portions. The mixture was stirred for 6 h at room temperature under nitrogen atmosphere. NFSI (18.9 g, 60 mmol) was added in portions to the mixture and the resulting slurry was stirred at room temperature for another 18 h. The reaction was then quenched by water. The resulting mixture was extracted with dichloromethane (3 × 100 mL). The organic layers were combined, washed with brine, and dried over anhydrous Na_2SO_4. After the removal of solvent under reduced pressure, the crude product was purified by column chromatography on

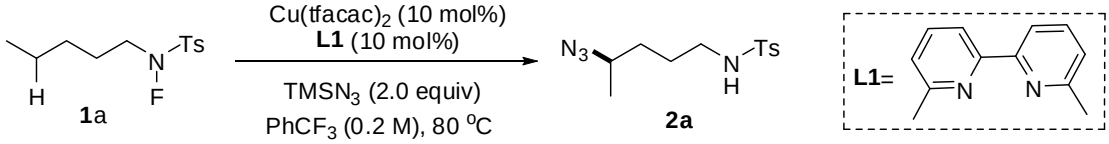
silica gel with petroleum ether (PE)–dichloromethane (DCM) (~ 10:1, v:v) as the eluent to give the pure product *N*-fluoro-*N*-alkylsulfonamide. (*Angew. Chem. Int. Ed.* **2019**, *58*, 2510.)

General procedure for the synthesis of *N*-fluoro-*N*-alkylcarboxamides



To a flame-dried round-bottom flask equipped with a stirring bar was added the amide (10 mmol) under nitrogen atmosphere. Anhydrous THF (80 mL) was then added and the solution was cooled down to 0 ℃ with the aid of an ice bath. *n*-Butyllithium (4.6 mL, 11mmol, 2.5 M solution in hexanes) was added dropwise. The mixture was maintained at 0 ℃ for 1.5 h. NFSI (25 mL, 15 mmol, 0.6 M in THF) was added dropwise (~ 1 drop/sec). The resulting mixture was stirred at 0 ℃ overnight and then allowed to warm up to room temperature. After 15 h, the reaction was quenched with 1 M aqueous HCl. The resulting mixture was diluted with DCM (100 mL) and water (100 mL). The two layers were separated and the aqueous layer was extracted with DCM (3 × 50 mL). The combined organic layers were washed with saturated aqueous $NaHCO_3$ and then brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The expected product *N*-fluoroamide was purified by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v) as the eluent. (*Angew. Chem. Int. Ed.* **2019**, *58*, 2510.)

3. Typical experimental procedure



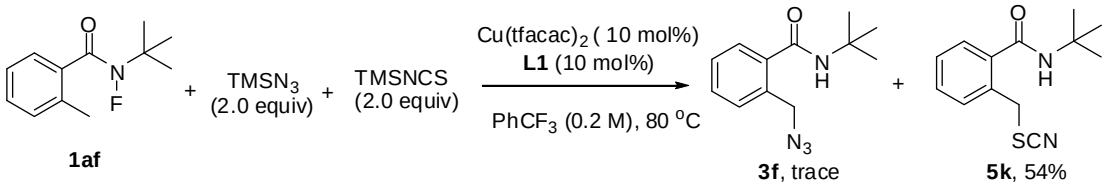
A mixture of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide (**1a**, 52 mg, 0.2 mmol), $TMSN_3$ (53 μL, 0.4 mmol), $Cu(tfacac)_2$ (7.4 mg, 10 mol%), **L1** (3.7 mg, 10

mol%) and degassed $PhCF_3$ (1.0 mL) in a 10 mL of Schlenk tube was stirred at 80 ℃ (oil bath) for 3 h. The solvent was removed under vaccum and the residue was purified by flash column chromatography on silica gel (PE : EA = 10:1) to afford 53 mg (94% yield) of **2a** as a yellow oil.

Gram-scale transformation:

A mixture of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide (**1a**, 1.04 g, 4.0 mmol), $TMSN_3$ (1.06 mL, 8.0 mmol), $Cu(tfacac)_2$ (148 mg, 10 mol%), **L1** (74 mg, 10 mol%) and degassed $PhCF_3$ (20.0 mL) in a 100 mL of Schlenk tube was stirred at 80 ℃ (oil bath) for 4 h. The solvent was removed under vaccum and the residue was purified by flash column chromatography on silica gel (PE : EA = 10:1) to afford 924 mg (82% yield) of **2a** as a yellow oil.

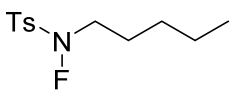
Competitive experiment:



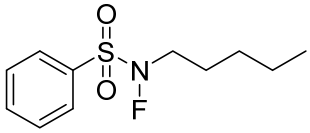
5k:**3f** > 20:1 (determined by crude ^1H NMR)

A mixture of *N*-*tert*-butyl-*N*-fluoro-2-methylbenzamide (**1af**, 42 mg, 0.2 mmol), $TMSN_3$ (53 μL, 0.4 mmol), TMSNCS (56 μL, 0.4 mmol), $Cu(tfacac)_2$ (7.4 mg, 10 mol%), **L1** (3.7 mg, 10 mol%) and degassed $PhCF_3$ (1.0 mL) in a 10 mL of Schlenk tube was stirred at 80 ℃ (oil bath) for 3 h. The solvent was removed under vaccum and the residue was purified by flash column chromatography on silica gel (PE : EA = 8:1) to afford 27 mg (54% yield) of **5k** as a white solid.

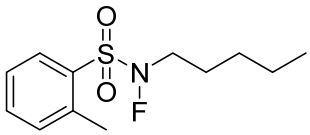
4. Characterization of the substrates and products



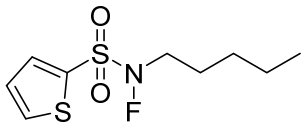
***N*-Fluoro-4-methyl-*N*-pentylbenzenesulfonamide (1a):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 3.19 (dt, *J* = 40.8, 7.0 Hz, 2H), 2.48 (s, 3H), 1.74 – 1.67 (m, 2H), 1.39 – 1.30 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.1, 130.0, 129.1, 53.8 (d, $J_{C\text{-}F}$ = 12.4 Hz), 28.9, 26.1, 22.3, 21.9, 14.0; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -49.30 (t, *J* = 39.5 Hz, 1F).



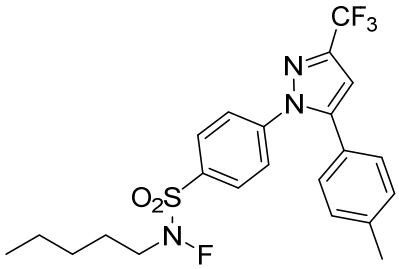
***N*-Fluoro-*N*-pentylbenzenesulfonamide (1b):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.74 (t, *J* = 7.5 Hz, 1H), 7.61 (t, *J* = 7.8 Hz, 2H), 3.21 (dt, *J* = 40.6, 7.0 Hz, 2H), 1.74 – 1.69 (m, 2H), 1.41 – 1.28 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 135.0, 132.2, 130.0, 129.4, 53.8 (d, $J_{C\text{-}F}$ = 12.5 Hz), 28.8, 26.1, 22.3, 14.0; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -50.02 (t, *J* = 40.6 Hz, 1F).



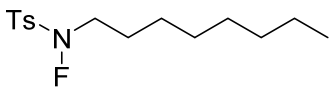
***N*-Fluoro-2-methyl-*N*-pentylbenzenesulfonamide (1c):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.03 (d, *J* = 7.9 Hz, 1H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.43 – 7.35 (m, 2H), 3.45 (dt, *J* = 41.2, 6.9 Hz, 2H), 2.69 (s, 3H), 1.81 – 1.72 (m, 2H), 1.44 – 1.32 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 140.7, 134.9, 133.1, 132.3, 131.9, 126.5, 51.6 (d, $J_{C\text{-}F}$ = 12.7 Hz), 28.9, 26.0, 22.3, 21.0, 14.0; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -50.04 (t, *J* = 41.3 Hz, 1F).



***N*-Fluoro-*N*-pentylthiophene-2-sulfonamide (1d):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 4.7 Hz, 1H), 7.81 (d, *J* = 3.0 Hz, 1H), 7.27 (s, 1H), 3.23 (dt, *J* = 40.2, 6.9 Hz, 2H), 1.79 – 1.71 (m, 2H), 1.41 – 1.31 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 136.9, 136.1, 131.0, 128.3, 54.5 (d, $J_{C\text{-}F}$ = 12.3 Hz), 28.8, 26.1, 22.3, 14.0; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -47.04 (t, *J* = 40.2 Hz, 1F).

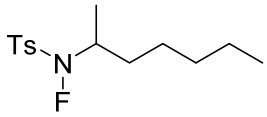


***N*-Fluoro-*N*-pentyl-4-(5-(*p*-tolyl)-3-(trifluoromethyl)-*1H*-pyrazol-1-yl)benzenesulfonamide (1e):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 7.7 Hz, 2H), 7.12 (d, *J* = 7.7 Hz, 2H), 6.76 (s, 1H), 3.20 (dt, *J* = 40.3, 6.8 Hz, 2H), 2.39 (s, 3H), 1.77 – 1.68 (m, 2H), 1.39 – 1.30 (m, 4H), 0.90 (t, *J* = 6.6 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.6, 144.8, 144.4, 140.2, 131.3, 131.1, 130.0, 128.9, 125.7, 125.5, 121.1 (q, $J_{C\text{-}F}$ = 269.2 Hz), 106.8, 53.8 (d, $J_{C\text{-}F}$ = 12.6 Hz), 28.8, 26.0, 22.3, 21.5, 14.0; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -49.60 (t, *J* = 40.3 Hz, 1F), -62.55 (s, 3F).

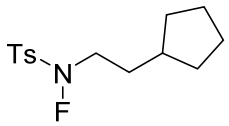


***N*-Fluoro-4-methyl-*N*-octylbenzenesulfonamide (1f):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 3.19 (dt, *J* = 40.7, 7.0 Hz, 2H), 2.48 (s, 3H), 1.73 – 1.67 (m, 2H), 1.40 – 1.36 (m, 2H), 1.30

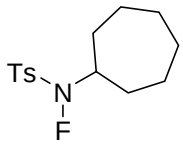
– 1.24 (m, 8H), 0.87 (t, J = 6.8 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.1, 130.0, 129.1, 53.9 (d, J_{C-F} = 12.3 Hz), 31.8, 29.2, 26.7, 26.4, 22.7, 21.9, 14.2; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.97 (t, J = 40.7 Hz, 1F).



***N*-Fluoro-*N*-(heptan-2-yl)-4-methylbenzenesulfonamide (1g)**: Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, J = 7.8 Hz, 2H), 7.37 (d, J = 7.8 Hz, 2H), 4.10 – 3.93 (m, 1H), 2.46 (s, 3H), 1.79 – 1.67 (m, 1H), 1.56 – 1.47 (m, 1H), 1.44 – 1.36 (m, 2H), 1.33 – 1.26 (m, 4H), 1.23 (d, J = 6.3 Hz, 3H), 0.88 (t, J = 6.2 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 145.8, 132.9, 130.0, 129.4, 59.3 (d, J_{C-F} = 13.1 Hz), 34.4, 31.6, 25.8, 22.7, 21.9, 16.0 (d, J_{C-F} = 11.2 Hz), 14.2; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -80.43 (d, J = 37.4 Hz, 1F).

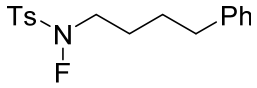


***N*-(2-Cyclopentylethyl)-*N*-fluoro-4-methylbenzenesulfonamide (1h):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H), 3.22 (dt, J = 40.6, 7.1 Hz, 2H), 2.48 (s, 3H), 1.91 – 1.83 (m, 1H), 1.80 – 1.71 (m, 4H), 1.63 – 1.48 (m, 4H), 1.13 – 1.02 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.0, 129.1, 53.3 (d, J_{C-F} = 12.4 Hz), 37.5, 32.6, 32.5, 25.1, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.78 (t, J = 40.6 Hz, 1F).

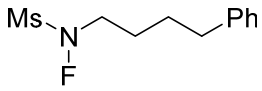


***N*-Cycloheptyl-*N*-fluoro-4-methylbenzenesulfonamide (1i):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.83 (d, J = 8.3 Hz, 2H), 7.36 (d, J = 8.1 Hz, 2H), 4.09 – 3.97 (m, 1H), 2.45 (s, 3H), 2.04 – 1.99 (m, 2H), 1.90 – 1.83 (m, 2H), 1.71 – 1.66 (m,

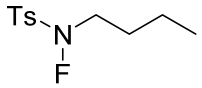
2H), 1.56 – 1.51 (m, 3H), 1.51 – 1.42 (m, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 145.8, 132.9, 129.9, 129.4, 64.5 (d, J_{C-F} = 12.6 Hz), 31.2 (d, J_{C-F} = 5.3 Hz), 28.6, 24.9, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -74.63 (d, *J* = 38.4 Hz, 1F).



***N*-Fluoro-4-methyl-*N*-(4-phenylbutyl)benzenesulfonamide (1j):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 (d, *J* = 7.6 Hz, 2H), 7.37 (d, *J* = 7.7 Hz, 2H), 7.23 (d, *J* = 6.4 Hz, 2H), 7.18 – 7.09 (m, 3H), 3.19 (d, *J* = 40.7 Hz, 2H), 2.64 – 2.57 (m, 2H), 2.45 (s, 3H), 1.74 – 1.69 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.4, 141.8, 130.1, 129.0, 128.5, 126.0, 53.6 (d, *J* = 12.4 Hz), 35.4, 28.4, 26.0, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.76 (t, *J* = 40.7 Hz, s, 1F).

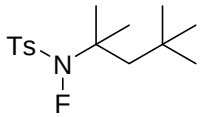


***N*-Fluoro-*N*-(4-phenylbutyl)methanesulfonamide (1k):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.22 – 7.16 (m, 3H), 3.48 (dt, *J* = 41.7, 6.7 Hz, 2H), 3.13 (s, 3H), 2.67 (t, *J* = 7.4 Hz, 2H), 1.85 – 1.77 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 141.8, 128.6, 128.5, 126.1, 50.8 (d, J_{C-F} = 12.5 Hz), 36.3, 35.4, 28.5, 25.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.36 (t, *J* = 41.7 Hz, 1F).

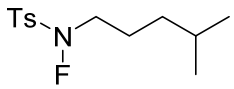


***N*-Butyl-*N*-fluoro-4-methylbenzenesulfonamide (1l):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 3.20 (dt, *J* = 40.8, 6.9 Hz, 2H), 2.47 (s, 3H), 1.72 – 1.64 (m, 2H), 1.47 – 1.37 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.0, 129.0, 53.6 (d, J_{C-F}

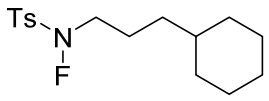
= 12.4 Hz), 28.4, 21.9, 19.9, 13.7; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -49.97 (t, *J* = 40.8 Hz, 1F).



***N*-Fluoro-4-methyl-*N*-(2,4,4-trimethylpentan-2-yl)benzenesulfonamide (1m):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 2.44 (s, 3H), 1.73 (d, *J* = 1.3 Hz, 2H), 1.53 (d, *J* = 1.7 Hz, 6H), 1.02 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.3, 135.1, 129.7, 129.1, 70.5 (d, J = 11.6 Hz), 52.7 (d, J = 2.9 Hz), 31.8, 31.7, 26.8 (d, J = 7.2 Hz), 21.8; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -62.40 (s, 1F).

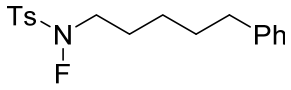


***N*-Fluoro-4-methyl-*N*-(4-methylpentyl)benzenesulfonamide (1n):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 3.18 (dt, *J* = 40.7, 7.0 Hz, 2H), 2.48 (s, 3H), 1.73 – 1.67 (m, 2H), 1.57 – 1.51 (m, 1H), 1.29 – 1.24 (m, 2H), 0.87 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.1, 130.0, 129.1, 54.2 (d, $J_{C\text{-}F}$ = 12.3 Hz), 35.8, 27.8, 24.4, 22.5, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.91 (t, *J* = 40.7 Hz, 1F).

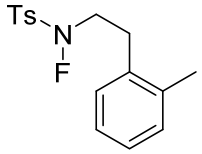


***N*-(3-Cyclohexylpropyl)-*N*-fluoro-4-methylbenzenesulfonamide (1o):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 3.18 (dt, *J* = 40.7, 7.1 Hz, 2H), 2.48 (s, 3H), 1.74 – 1.66 (m, 7H), 1.28 – 1.12 (m, 6H), 0.90 – 0.83 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 130.1, 130.0,

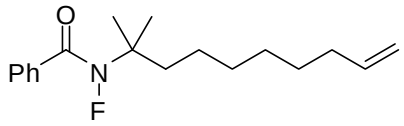
129.2, 54.2 (d, $J_{C\text{-}F}$ = 12.3 Hz), 37.4, 34.4, 33.3, 26.7, 26.4, 23.9, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.89 (t, *J* = 40.7 Hz, 1F).



***N*-Fluoro-4-methyl-*N*-(5-phenylpentyl)benzenesulfonamide (1p):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.2 Hz, 2H), 7.41 (d, *J* = 8.2 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.20 – 7.16 (m, 3H), 3.21 (dt, *J* = 40.6, 7.0 Hz, 2H), 2.62 (t, *J* = 7.7 Hz, 2H), 2.49 (s, 3H), 1.77 – 1.72 (m, 2H), 1.68 – 1.62 (m, 2H), 1.48 – 1.42 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.3, 142.3, 130.0, 129.0, 128.5, 128.4, 125.9, 53.7 (d, $J_{C\text{-}F}$ = 12.3 Hz), 35.8, 31.0, 26.3, 21.9; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -49.85 (t, *J* = 40.7 Hz, 1F).

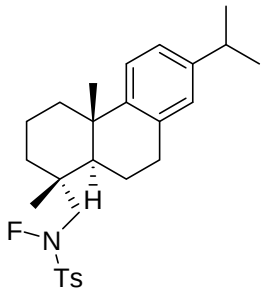


***N*-Fluoro-4-methyl-*N*-(2-methylphenethyl)benzenesulfonamide (1q):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.17 – 7.13 (m, 4H), 3.48 – 3.38 (m, 2H), 3.09 – 3.05 (m, 2H), 2.49 (s, 3H), 2.30 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 146.4, 136.4, 135.7, 130.6, 130.1, 130.0, 129.5, 129.0, 127.1, 126.4, 54.1 (d, $J_{C\text{-}F}$ = 12.1 Hz), 30.3, 21.9, 19.4; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -49.99 (t, *J* = 40.8 Hz, 1F).

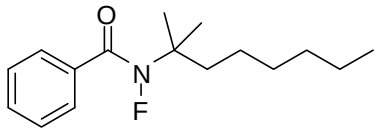


***N*-Fluoro-*N*-(2-methyldec-9-en-2-yl)benzamide (1r):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 – 7.64 (m, 2H), 7.54 – 7.46 (m, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 5.87 – 5.77 (m, 1H), 5.11 – 4.86 (m, 2H), 2.10 – 2.00 (m, 2H), 1.90 – 1.80 (m, 2H), 1.51 (d, *J* = 1.9 Hz, 6H), 1.48 – 1.23 (m, 8H); **^{13}C NMR (150 MHz, $CDCl_3$)**

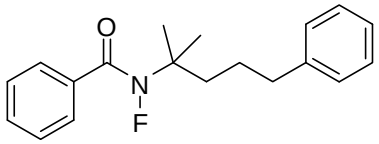
δ 174.3 (d, *J* = 7.5 Hz), 139.2, 134.4, 131.8, 128.9 (d, *J* = 5.9 Hz), 128.1, 114.3, 67.0 (d, *J* = 9.6 Hz), 40.1 (d, *J* = 4.3 Hz), 33.9, 30.0, 29.2, 29.0, 25.3 (d, *J* = 5.9 Hz), 24.2; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -62.60 (s, 1F).



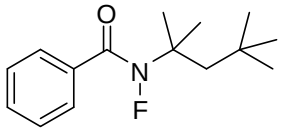
***N*-Fluoro-*N*-(((1R,4aS,10aR)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)-4-methylbenzenesulfonamide (1s):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 10:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.2 Hz, 1H), 7.00 – 6.95 (m, 1H), 6.89 – 6.85 (m, 1H), 3.30 – 2.95 (m, 2H), 2.88 – 2.77 (m, 3H), 2.48 (s, 3H), 2.30 – 2.25 (m, 1H), 1.80 – 1.65 (m, 6H), 1.47 – 1.40 (m, 2H), 1.23 – 1.22 (m, 6H), 1.21 (s, 3H), 0.97 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.2, 146.2, 145.7, 134.9, 130.1, 130.0, 129.9, 127.0, 124.3, 123.9, 63.3(d, $J_{C\text{-}F}$ = 10.0 Hz), 44.7, 38.3, 37.7, 37.7, 36.5, 33.6, 30.1, 25.6, 24.1 (d, *J* = 2.8 Hz), 21.9, 19.1, 19.1, 18.7; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -32.57 (t, *J* = 44.1 Hz, 1F).



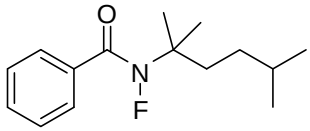
***N*-Fluoro-*N*-(2-methyloctan-2-yl)benzamide (1aa):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.69 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 1.94 – 1.77 (m, 2H), 1.51 (s, 6H), 1.47 – 1.38 (m, 2H), 1.30 (s, 6H), 0.89 (t, *J* = 6.0 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 174.3 (d, *J* = 7.5 Hz), 134.5, 131.8, 128.9 (d, *J* = 5.9 Hz), 128.1, 67.0 (d, *J* = 9.6 Hz), 40.1 (d, *J* = 4.4 Hz), 31.9, 29.8, 25.3 (d, *J* = 6.0 Hz), 24.2, 22.8, 14.2; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -66.19 (s, 1F).



***N*-Fluoro-*N*-(2-methyl-5-phenylpentan-2-yl)benzamide (1ab):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.70 (d, *J* = 7.3 Hz, 2H), 7.50 (t, *J* = 7.1 Hz, 1H), 7.42 (t, *J* = 7.4 Hz, 2H), 7.30 (t, *J* = 7.3 Hz, 2H), 7.24 – 7.14 (m, 3H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.00 – 1.88 (m, 2H), 1.86 – 1.72 (m, 2H), 1.51 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 174.4 (d, *J* = 7.5 Hz), 142.3, 134.3, 131.8, 129.0 (d, *J* = 5.9 Hz), 128.50, 128.46, 128.1, 125.9, 66.8 (d, *J* = 9.6 Hz), 39.9 (d, *J* = 4.4 Hz), 36.3, 26.2, 25.2 (d, *J* = 5.9 Hz); **^{19}F NMR (564 MHz, $CDCl_3$)** δ -66.16 (s, 1F).

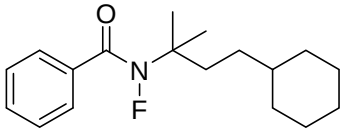


***N*-Fluoro-*N*-(2,4,4-trimethylpentan-2-yl)benzamide (1ac):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.67 (d, *J* = 6.9 Hz, 2H), 7.53 – 7.45 (m, 1H), 7.41 (t, *J* = 6.9 Hz, 2H), 1.90 (s, 2H), 1.60 (s, 6H), 1.07 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.6 (d, *J* = 8.0 Hz), 134.6, 131.7, 128.9 (d, *J* = 6.0 Hz), 128.1, 67.8 (d, *J* = 9.7 Hz), 51.0 (d, *J* = 3.5 Hz), 31.8, 31.5, 27.6 (d, *J* = 6.5 Hz); **^{19}F NMR (564 MHz, $CDCl_3$)** δ -62.31 (s, 1F);

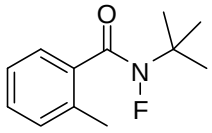


***N*-(2,5-Dimethylhexan-2-yl)-*N*-fluorobenzamide (1ad):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 1.88 – 1.83 (m, 2H), 1.56 – 1.52 (m, 1H), 1.51 (d, *J* = 1.4 Hz, 6H), 1.34 – 1.30 (m, 2H), 0.91 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 174.3 (d, $J_{C\text{-}F}$ = 7.6 Hz), 134.5, 131.8, 128.9 (d, *J* = 6.0 Hz), 128.1, 67.0 (d, *J* = 9.6 Hz), 38.0

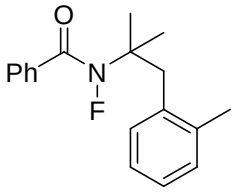
(d, J = 4.5 Hz), 33.2, 28.5, 25.3 (d, J = 6.0 Hz), 22.8; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -66.23 (s, 1F).



***N*-(4-Cyclohexyl-2-methylbutan-2-yl)-*N*-fluorobenzamide (1ae):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.69 (d, J = 7.3 Hz, 1H), 7.49 (t, J = 7.1 Hz, 1H), 7.41 (t, J = 7.4 Hz, 1H), 1.89 – 1.84 (m, 1H), 1.75 – 1.60 (m, 3H), 1.50 (s, 4H), 1.36 – 1.28 (m, 1H), 1.26 – 1.12 (m, 2H), 0.96 – 0.86 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 174.2 (d, $J_{C\text{-}F}$ = 7.5 Hz), 134.5, 131.7, 128.9 (d, J = 5.9 Hz), 128.1, 67.0 (d, $J_{C\text{-}F}$ = 9.5 Hz), 38.2, 37.5 (d, J = 4.4 Hz), 33.5, 31.7, 26.8, 26.5, 25.2 (d, J = 5.9 Hz); **^{19}F NMR (377 MHz, $CDCl_3$)** δ -61.90 (s, 1F).

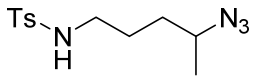


***N*-(*tert*-Butyl)-*N*-fluoro-2-methylbenzamide (1af):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.35 – 7.28 (m, 2H), 7.24 – 7.17 (m, 2H), 2.40 (s, 3H), 1.55 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 175.2 (d, $J_{C\text{-}F}$ = 10.7 Hz), 135.5 (d, J = 2.3 Hz), 135.2, 130.6, 130.1, 127.3 (d, J = 4.4 Hz), 125.5, 64.5 (d, $J_{C\text{-}F}$ = 10.7 Hz), 27.3 (d, J = 5.7 Hz), 19.5; **^{19}F NMR (564 MHz, $CDCl_3$)** δ -64.70 (s, 1F).

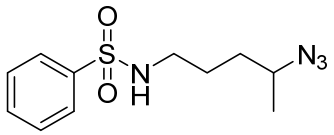


***N*-Fluoro-*N*-(2-methyl-1-(o-tolyl)propan-2-yl)benzamide (1ag):** Purification by column chromatography on silica gel with petroleum ether–dichloromethane (~ 20:1, v:v); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.67 (m, 2H), 7.54 – 7.48 (m, 1H), 7.45 – 7.40 (m, 2H), 7.25 – 7.22 (m, 1H), 7.20 – 7.11 (m, 3H), 3.27 (s, 2H), 2.40 (s, 3H),

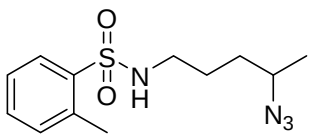
1.53 (d, J = 2.3 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 174.3 (d, $J_{C\text{-}F}$ = 8.0 Hz), 137.5, 135.6, 134.4, 131.9, 131.7, 129.1, 129.0, 128.2, 126.8, 125.7, 68.4 (d, J = 9.4 Hz), 39.8 (d, J = 3.6 Hz), 25.3 (d, J = 6.6 Hz), 20.6; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -63.81 (s, 1F).



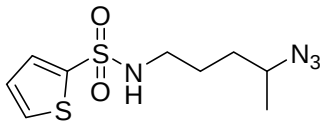
***N*-(4-Azidopentyl)-4-methylbenzenesulfonamide (2a):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 94% yield (53 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76 – 7.73 (m, 2H), 7.31 (d, J = 8.0 Hz, 2H), 4.86 (t, J = 6.2 Hz, 1H), 3.42 – 3.33 (m, 1H), 2.93 (q, J = 6.7 Hz, 2H), 2.42 (s, 3H), 1.60 – 1.39 (m, 4H), 1.20 (d, J = 6.5 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.0, 129.9, 127.2, 57.5, 42.9, 33.2, 26.3, 21.6, 19.5; **HRMS (ESI)** calcd $C_{12}H_{18}N_4O_2SNa$ $[M + Na]^+$: 305.1043, found: 305.1037; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3287, 2967, 2096, 1640, 1157, 660.



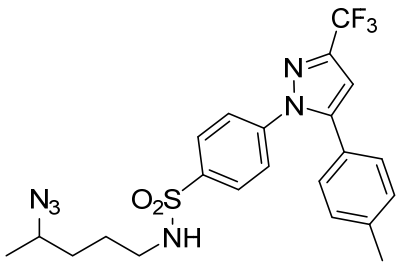
***N*-(4-Azidopentyl)benzenesulfonamide (2b):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 87% yield (46.6 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 – 7.85 (m, 2H), 7.62 – 7.56 (m, 1H), 7.55 – 7.49 (m, 2H), 4.84 (t, J = 6.0 Hz, 1H), 3.43 – 3.34 (m, 1H), 2.96 (q, J = 6.6 Hz, 2H), 1.62 – 1.39 (m, 4H), 1.20 (d, J = 6.5 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 140.0, 132.8, 129.3, 127.1, 57.5, 43.0, 33.2, 26.3, 19.5; **HRMS (ESI)** calcd $C_{11}H_{16}N_4O_2SNa$ $[M + Na]^+$: 291.0886, found: 291.0878; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3282, 2930, 2097, 1446, 1156, 688.



***N*-(4-Azidopentyl)-2-methylbenzenesulfonamide (2c):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 69% yield (39 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.34 – 7.28 (m, 2H), 4.87 – 4.82 (m, 1H), 3.38 – 3.33 (m, 1H), 2.95 (q, *J* = 6.6 Hz, 2H), 2.64 (s, 3H), 1.60 – 1.48 (m, 2H), 1.46 – 1.39 (m, 2H), 1.19 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 138.0, 137.1, 132.9, 132.7, 129.5, 126.3, 57.5, 42.8, 33.1, 26.4, 20.4, 19.5; **HRMS (ESI)** calcd $C_{12}H_{18}N_4O_2SNa$ $[M + Na]^+$: 305.1043, found: 305.1034; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3295, 2932, 2098, 1455, 1156, 732.

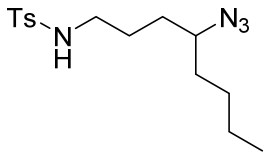


***N*-(4-Azidopentyl)thiophene-2-sulfonamide (2d):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 55% yield (30 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.63 – 7.58 (m, 2H), 7.11 – 7.08 (m, 1H), 4.75 (t, *J* = 5.4 Hz, 1H), 3.47 – 3.38 (m, 1H), 3.06 (q, *J* = 6.4 Hz, 2H), 1.68 – 1.42 (m, 4H), 1.24 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 141.0, 132.3, 132.0, 127.6, 57.5, 43.2, 33.2, 26.2, 19.5; **HRMS (ESI)** calcd $C_9H_{14}N_4O_2S_2Na$ $[M + Na]^+$: 297.0450, found: 297.0444; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3282, 2929, 2098, 1405, 1153, 720.

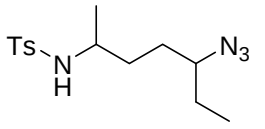


***N*-(4-Azidopentyl)-4-(5-(*p*-tolyl)-3-(trifluoromethyl)-*1H*-pyrazol-1-yl)benzenesulfonamide (2e):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 70% yield (56 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.17 (d,

J = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 6.74 (s, 1H), 4.63 (t, *J* = 6.2 Hz, 1H), 3.45 – 3.39 (m, 1H), 2.96 (q, *J* = 6.7 Hz, 2H), 2.38 (s, 3H), 1.61 – 1.41 (m, 4H), 1.24 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 145.4, 144.3 (q, *J* = 38.7 Hz), 142.7, 140.0, 139.5, 129.9, 128.9, 128.2, 125.8, 125.7, 121.2 (q, *J* = 269.2 Hz), 106.5, 57.5, 43.1, 33.2, 26.4, 21.5, 19.5; **HRMS (ESI)** calcd $C_{22}H_{24}F_3N_6O_2S$ $[M + H]^+$: 493.1628, found: 493.1633; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3281, 2927, 2102, 1471, 1159, 806.

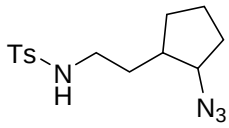


***N*-(4-Azidooctyl)-4-methylbenzenesulfonamide (2f):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 88% yield (57 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.57 (t, *J* = 6.0 Hz, 1H), 3.20 – 3.15 (m, 1H), 2.99 – 2.93 (m, 2H), 2.43 (s, 3H), 1.63 – 1.57 (m, 2H), 1.55 – 1.40 (m, 6H), 1.34 – 1.29 (m, 2H), 0.90 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.1, 129.9, 127.2, 62.6, 43.1, 34.2, 31.4, 28.3, 26.4, 22.6, 21.7, 14.1; **HRMS (ESI)** calcd $C_{15}H_{24}N_4O_2SNa$ $[M + Na]^+$: 347.1512, found: 347.1504; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3282, 2926, 2096, 1457, 1157, 732.

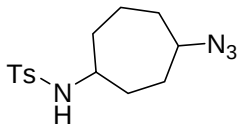


***N*-(5-Azidoheptan-2-yl)-4-methylbenzenesulfonamide (2g):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 71% yield (44 mg); dr= 1.2:1; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 2H), 4.42 – 4.30 (m, 1H), 3.37 – 3.27 (m, 1H), 3.10 – 3.05 (m, 1H), 2.42 (s, 3H), 1.52 – 1.33 (m, 6H), 1.02 (d, *J* = 6.5 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.5, 138.3, 129.8, 127.1, 64.4, 64.0, 50.2, 49.7, 34.2, 33.8, 30.3, 29.8, 27.5, 27.4, 22.0, 21.9, 21.6, 10.5, 10.4; **HRMS**

(ESI) calcd $C_{14}H_{22}N_4O_2SNa$ $[M + Na]^+$: 333.1356, found: 333.1349; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3276, 2926, 2093, 1459, 1159, 663.

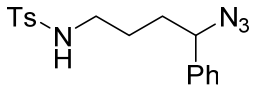


***N*-(2-(2-Azidocyclopentyl)ethyl)-4-methylbenzenesulfonamide (2h):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 68% yield (42 mg); dr = 8:1; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 – 7.71 (m, 2H), 7.34 – 7.29 (m, 2H), 4.68 – 4.61 (m, 0.89H)/3.86 – 3.80 (m, 0.11H), 3.34 – 3.28 (m, 0.89H)/3.86 – 3.80 (m, 0.11H), 3.01 – 2.94 (m, 2H), 2.43 (s, 3H), 2.00 – 1.93 (m, 1H), 1.90 – 1.81 (m, 1H), 1.75 – 1.60 (m, 5H), 1.51 – 1.41 (m, 1H), 1.20 – 1.09 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.0, 129.9, 127.2, 67.6, 65.6, 43.2, 42.3, 42.2, 42.1, 34.0, 31.2, 31.1, 30.4, 30.0, 29.1, 22.3, 21.8, 21.7; **HRMS (ESI)** calcd $C_{14}H_{20}N_4O_2SNa$ $[M + Na]^+$: 331.1199, found: 331.1190; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3314, 2927, 2093, 1643, 1157, 712.

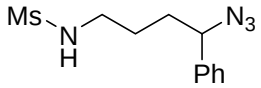


***N*-(4-Azidocycloheptyl)-4-methylbenzenesulfonamide (2i):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; 75% yield (46 mg); dr = 1.5:1. **Isomer 2i-a:** yellow oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 4.80 – 4.76 (m, 1H), 3.60 – 3.54 (m, 1H), 3.38 – 3.33 (m, 1H), 2.42 (s, 3H), 1.93 – 1.88 (m, 1H), 1.84 – 1.78 (m, 1H), 1.74 – 1.60 (m, 5H), 1.57 – 1.50 (m, 1H), 1.43 – 1.37 (m, 1H), 1.29 – 1.22 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.5, 138.2, 129.8, 127.1, 61.4, 53.8, 36.1, 33.9, 30.0, 28.6, 21.7, 20.0; **HRMS (ESI)** calcd $C_{14}H_{20}N_4O_2SNa$ $[M + Na]^+$: 331.1199, found: 331.1189; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2930, 2091, 1445, 1155, 662. **Isomer 2i-b:** yellow oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 4.65 (d, *J* = 7.2 Hz, 1H), 3.55 – 3.50 (m, 1H), 3.38

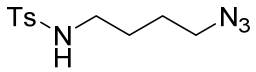
– 3.31 (m, 1H), 2.43 (s, 3H), 1.93 – 1.79 (m, 4H), 1.64 – 1.55 (m, 2H), 1.52 – 1.37 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.5, 138.0, 129.9, 127.1, 61.8, 54.2, 35.8, 33.7, 30.8, 29.1, 21.7, 19.4; **HRMS (ESI)** calcd $C_{14}H_{20}N_4O_2SNa$ $[M + Na]^+$: 331.1199, found: 331.1190; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2931, 2090, 1460, 1154, 663.



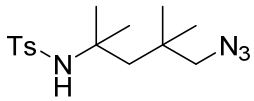
***N*-(4-Azido-4-phenylbutyl)-4-methylbenzenesulfonamide (2j):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 84% yield (58 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (d, *J* = 7.9 Hz, 2H), 7.38 – 7.32 (m, 3H), 7.31 – 7.27 (m, 2H), 7.23 (d, *J* = 6.9 Hz, 2H), 4.69 (t, *J* = 5.6 Hz, 1H), 4.36 (t, *J* = 6.9 Hz, 1H), 2.97 – 2.90 (m, 2H), 2.42 (s, 3H), 1.83 – 1.64 (m, 2H), 1.61 – 1.51 (m, 1H), 1.49 – 1.39 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 139.3, 137.0, 129.9, 129.0, 128.5, 127.2, 126.9, 65.8, 42.8, 33.2, 26.4, 21.6; **HRMS (ESI)** calcd $C_{17}H_{20}N_4O_2SNa$ $[M + Na]^+$: 367.1199, found: 367.1190; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3279, 2930, 2094, 1453, 1155, 661.



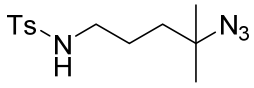
***N*-(4-Azido-4-phenylbutyl)methanesulfonamide (2k):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 62% yield (33 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41 – 7.36 (m, 2H), 7.36 – 7.32 (m, 1H), 7.32 – 7.28 (m, 2H), 4.52 – 4.45 (m, 2H), 3.13 (q, *J* = 6.7 Hz, 2H), 2.92 (s, 3H), 1.91 – 1.77 (m, 2H), 1.73 – 1.64 (m, 1H), 1.61 – 1.52 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 139.3, 129.0, 128.6, 126.9, 65.9, 42.9, 40.5, 33.3, 27.0; **HRMS (ESI)** calcd $C_{11}H_{16}N_4O_2SNa$ $[M + Na]^+$: 291.0886, found: 291.0877; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3287, 2932, 2094, 1454, 1146, 700.



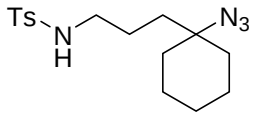
***N*-(4-Azidobutyl)-4-methylbenzenesulfonamide (2l):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 40% yield (21.5 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 4.51 (t, *J* = 5.9 Hz, 1H), 3.25 (t, *J* = 5.8 Hz, 2H), 2.97 (q, *J* = 6.1 Hz, 2H), 2.43 (s, 3H), 1.63 – 1.52 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.7, 137.0, 129.9, 127.2, 51.0, 42.8, 27.0, 26.0, 21.7; **HRMS (ESI)** calcd $C_{11}H_{16}N_4O_2SNa$ $[M + Na]^+$: 291.0886, found: 291.0878; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3277, 2922, 2093, 1458, 1154, 660.



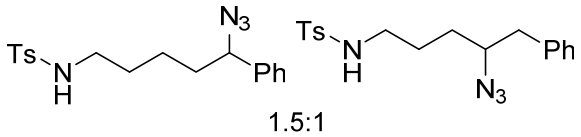
***N*-(5-Azido-2,4,4-trimethylpentan-2-yl)-4-methylbenzenesulfonamide (2m):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 45% yield (29 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 4.97 (s, 1H), 3.19 (s, 2H), 2.42 (s, 3H), 1.55 (s, 2H), 1.24 (s, 6H), 1.02 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.0, 140.8, 129.6, 127.1, 62.9, 57.9, 51.6, 36.5, 29.7, 27.6, 21.6; **HRMS (ESI)** calcd $C_{15}H_{25}N_4O_2S$ $[M + H]^+$: 325.1693, found: 325.1694; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3279, 2965, 2097, 1467, 1151, 662.



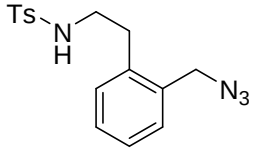
***N*-(4-Azido-4-methylpentyl)-4-methylbenzenesulfonamide (2n):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 85% yield (50 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 4.71 (t, *J* = 6.1 Hz, 1H), 2.94 (q, *J* = 6.7 Hz, 2H), 2.42 (s, 3H), 1.55 – 1.49 (m, 2H), 1.44 – 1.40 (m, 2H), 1.20 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.1, 129.9, 127.2, 61.2, 43.4, 38.4, 26.0, 24.7, 21.6; **HRMS (ESI)** calcd $C_{13}H_{20}N_4O_2SNa$ $[M + Na]^+$: 319.1199, found: 319.1191; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3279, 2926, 2094, 1599, 1155, 661.



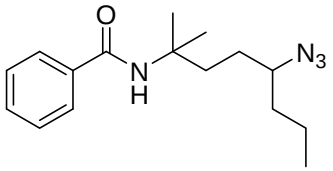
***N*-(3-(1-Azidocyclohexyl)propyl)-4-methylbenzenesulfonamide (2o):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 90% yield (60.5 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 – 7.73 (m, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 4.81 (t, *J* = 6.2 Hz, 1H), 2.94 (q, *J* = 6.4 Hz, 2H), 2.42 (s, 3H), 1.62 – 1.41 (m, 11H), 1.31 – 1.18 (m, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.0, 129.8, 127.2, 63.7, 43.5, 37.0, 34.5, 25.4, 23.7, 22.2, 21.6; **HRMS (ESI)** calcd $C_{16}H_{24}N_4O_2SNa$ $[M + Na]^+$: 359.1512, found: 359.1505; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3280, 2931, 2097, 1448, 1155, 662.



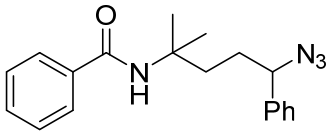
N-(5-Azido-5-phenylpentyl)-4-methylbenzenesulfonamide (2pa) and N-(4-Azido-5-phenylpentyl)-4-methylbenzenesulfonamide (2pb): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 71% yield (51 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 – 7.73 (m, 2H), 7.38 – 7.35 (m, 1H), 7.33 – 7.29 (m, 3H), 7.26 – 7.23 (m, 2H), 7.17 (d, *J* = 7.2 Hz, 1H), 4.91 – 4.76 (m, 1H), 4.36 – 4.31 (m, 0.75H), 3.48 – 3.42 (m, 0.50H), 2.95 – 2.87 (m, 2H), 2.76 (d, *J* = 6.3 Hz, 1H), 2.42 (s, 3H), 1.77 – 1.71 (m, 1H), 1.68 – 1.61 (m, 1H), 1.58 – 1.50 (m, 1H), 1.49 – 1.45 (m, 1H), 1.44 – 1.33 (m, 1H), 1.30 – 1.19 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 143.5, 139.6, 137.5, 137.0, 129.9, 129.8, 129.3, 128.9, 128.7, 128.4, 127.2, 126.9, 66.2, 63.7, 43.0, 42.9, 41.0, 35.7, 31.0, 29.3, 26.4, 23.3, 21.6; **HRMS (ESI)** calcd $C_{18}H_{23}N_4O_2S$ $[M + H]^+$: 359.1536, found: 359.1528; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3280, 2940, 2094, 1454, 1155, 661.



***N*-(2-(Azidomethyl)phenethyl)-4-methylbenzenesulfonamide (2q):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 58% yield (38.4 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.23 (m, 5H), 7.14 (d, *J* = 7.1 Hz, 1H), 4.76 (t, *J* = 5.5 Hz, 1H), 4.29 (s, 2H), 3.22 – 3.18 (m, 2H), 2.85 (t, *J* = 7.3 Hz, 2H), 2.42 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.6, 137.0, 136.8, 133.7, 130.4, 130.3, 129.9, 129.2, 127.4, 127.2, 52.7, 43.9, 32.8, 21.6; **HRMS (ESI)** calcd $C_{16}H_{19}N_4O_2S$ $[M + H]^+$: 331.1223, found: 331.1217; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3276, 2928, 2093, 1494, 1154, 661.

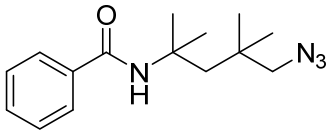


***N*-(5-Azido-2-methyloctan-2-yl)benzamide (3a):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; white solid; m.p. 67 – 70 ℃; 71% yield (41 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 – 7.70 (m, 2H), 7.50 – 7.46 (m, 1H), 7.44 – 7.39 (m, 2H), 5.89 (s, 1H), 3.31 – 3.25 (m, 1H), 2.05 – 1.87 (m, 2H), 1.57 – 1.49 (m, 5H), 1.45 (s, 3H), 1.43 (s, 3H), 1.40 – 1.34 (m, 1H), 0.93 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.1, 135.9, 131.3, 128.7, 126.8, 63.1, 54.0, 36.6, 36.4, 29.2, 27.4, 27.3, 19.5, 14.0; **HRMS (ESI)** calcd $C_{16}H_{25}N_4O$ $[M + H]^+$: 289.2023, found: 289.2016; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3361, 2965, 2097, 1638, 1253, 713.

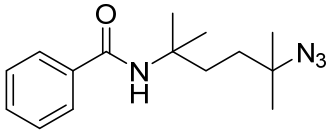


***N*-(5-Azido-2-methyl-5-phenylpentan-2-yl)benzamide (3b):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 85% yield (55 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.41 – 7.35 (m, 4H), 7.34 – 7.28 (m, 3H), 5.90 (s, 1H), 4.42 (t, *J* = 6.3 Hz, 1H), 2.02 – 1.97 (m, 1H), 1.89 – 1.84 (m, 2H), 1.83 – 1.76 (m,

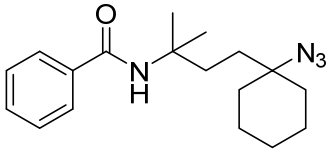
1H), 1.42 (s, 3H), 1.41 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.0, 139.6, 135.7, 131.3, 128.9, 128.6, 128.4, 126.9, 126.8, 66.7, 53.8, 36.6, 31.2, 27.3, 27.2; **HRMS (ESI)** calcd $C_{19}H_{23}N_4O$ $[M + H]^+$: 323.1866, found: 323.1856; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3317, 2967, 2093, 1642, 1245, 698.



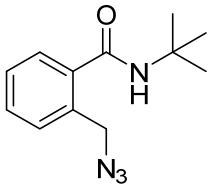
***N*-(5-Azido-2,4,4-trimethylpentan-2-yl)benzamide (3c):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 91% yield (50 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.71 (m, 2H), 7.49 – 7.44 (m, 1H), 7.43 – 7.38 (m, 2H), 6.36 (s, 1H), 3.23 (s, 2H), 1.85 (s, 2H), 1.53 (s, 6H), 1.08 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.0, 136.0, 131.2, 128.6, 126.8, 63.0, 55.2, 48.4, 36.7, 29.2, 27.4; **HRMS (ESI)** calcd $C_{15}H_{23}N_4O$ $[M + H]^+$: 275.1866, found: 275.1860; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3315, 2964, 2097, 1642, 1158, 662.



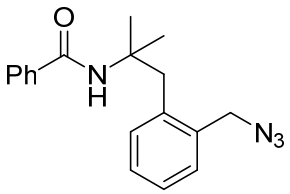
***N*-(5-Azido-2,5-dimethylhexan-2-yl)benzamide (3d):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 55% yield (30 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.71 (d, J = 7.3 Hz, 2H), 7.47 (t, J = 7.4 Hz, 1H), 7.41 (t, J = 7.5 Hz, 2H), 5.90 (s, 1H), 1.95 – 1.89 (m, 2H), 1.53 – 1.47 (m, 2H), 1.43 (s, 6H), 1.27 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.0, 135.9, 131.3, 128.7, 126.8, 61.7, 53.9, 35.9, 34.5, 27.3, 26.1; **HRMS (ESI)** calcd $C_{15}H_{22}N_4ONa$ $[M + Na]^+$: 297.1686, found: 297.1677; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3317, 2970, 2093, 1641, 1261, 712.



***N*-(4-(1-Azidocyclohexyl)-2-methylbutan-2-yl)benzamide (3e):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; white solid; m.p. 106 – 109 ℃; 70% yield (44 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.4 Hz, 2H), 7.46 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 5.86 (s, 1H), 1.97 – 1.93 (m, 2H), 1.70 – 1.65 (m, 2H), 1.58 – 1.49 (m, 7H), 1.44 (s, 6H), 1.43 – 1.37 (m, 2H), 1.27 – 1.23 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.0, 135.9, 131.2, 128.6, 126.8, 64.2, 53.9, 34.6, 34.3, 33.5, 27.3, 25.5, 22.3; **HRMS (ESI)** calcd $C_{18}H_{27}N_4O$ $[M + H]^+$: 315.2179, found: 315.2171; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3358, 2929, 2098, 1639, 1258, 693.

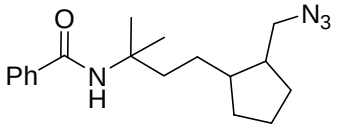


2-(Azidomethyl)-N-(*tert*-butyl)benzamide (3f): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 52% yield (24 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48 – 7.43 (m, 1H), 7.42 – 7.39 (m, 1H), 7.37 – 7.31 (m, 2H), 5.92 (s, 1H), 4.56 (s, 2H), 1.47 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 168.5, 137.5, 133.4, 130.3, 130.1, 128.6, 127.7, 52.8, 52.2, 28.9; **HRMS (ESI)** calcd $C_{12}H_{16}N_4ONa$ $[M + Na]^+$: 255.1216, found: 255.1208; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3300, 2967, 2095, 1640, 1220, 744.

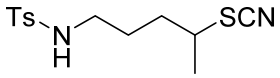


***N*-(1-(2-(Azidomethyl)phenyl)-2-methylpropan-2-yl)benzamide (3g):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 47% yield (29 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.71 – 7.67 (m, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 6.9 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.25 – 7.22 (m, 2H), 5.87 (s, 1H), 4.48 (s, 2H), 3.30 (s, 2H), 1.46 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.6, 136.7, 135.9, 134.8, 132.2, 131.4,

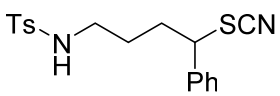
130.0, 128.7, 128.3, 127.2, 126.8, 55.1, 52.7, 40.3, 27.7; **HRMS (ESI)** calcd $C_{18}H_{20}N_4ONa$ $[M + Na]^+$: 331.1529, found: 331.1519; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3319, 2970, 2094, 1645, 1248, 728.



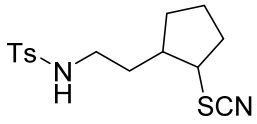
***N*-(4-(2-(Azidomethyl)cyclopentyl)-2-methylbutan-2-yl)benzamide (4):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 10:1, v:v) as the eluent; yellow oil; 61% (38 mg); dr =2:1**; ^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.3 Hz, 2H), 7.46 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 2H), 5.89 (s, 0.66H)/5.85 (s, 0.34H), 3.35 – 3.27 (m, 1H), 3.14 (dd, *J* = 11.7, 8.1 Hz, 0.34H)/3.10 – 3.03 (m, 0.66H), 2.20 – 2.11 (m, 1H), 1.92 – 1.46 (m, 8H), 1.43 (s, 6H), 1.38 – 1.12 (m, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.02/166.98, 136.0, 131.2, 128.6, 126.8, 56.1, 54.19, 54.17, 52.4, 45.5, 43.7, 42.3, 41.7, 39.2, 38.7, 32.6, 30.8, 30.6, 29.7, 29.3, 27.4, 27.32, 27.25, 27.19, 24.3, 24.2, 22.7; **HRMS (ESI)** calcd $C_{18}H_{27}N_4O$ $[M + H]^+$: 315.2179, found: 315.2170. **FT-IR** (thin film, KBr): 3321, 2942, 2091, 1640, 1277, 712.



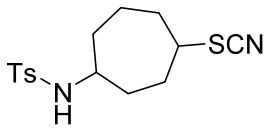
4-Methyl-*N*-(4-thiocyanatopentyl)benzenesulfonamide (5a): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 70% yield (42 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 4.79 (t, *J* = 6.2 Hz, 1H), 3.25 – 3.14 (m, 1H), 2.96 (q, *J* = 6.6 Hz, 2H), 2.43 (s, 3H), 1.76 – 1.70 (m, 2H), 1.65 – 1.58 (m, 2H), 1.47 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.8, 136.9, 130.0, 127.2, 111.1, 45.2, 42.6, 34.0, 27.2, 22.1, 21.7; **HRMS (ESI)** calcd $C_{13}H_{18}N_2O_2S_2Na$ $[M + Na]^+$: 321.0702, found: 321.0695; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2926, 2152, 1450, 1155, 661.



4-Methyl-*N*-(4-phenyl-4-thiocyanatobutyl)benzenesulfonamide (5b): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 65% yield (46.5 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.6 Hz, 2H), 7.38 – 7.33 (m, 3H), 7.32 – 7.23 (m, 4H), 4.80 (br, 1H), 4.25 (t, *J* = 7.4 Hz, 1H), 2.99 – 2.88 (m, 2H), 2.42 (s, 3H), 2.20 – 2.10 (m, 2H), 1.60 – 1.40 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.8, 137.9, 136.8, 129.9, 129.3, 129.2, 127.5, 127.2, 111.6, 53.0, 42.5, 32.8, 27.6, 21.7; **HRMS (ESI)** calcd $C_{18}H_{20}N_2O_2S_2Na$ $[M + Na]^+$: 383.0858, found: 383.0849; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2926, 2151, 1454, 1154, 661.

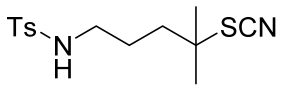


4-Methyl-*N*-(2-(2-thiocyanatocyclopentyl)ethyl)benzenesulfonamide (5c): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 57% yield (37 mg); dr = 2:1; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 – 7.73 (m, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 4.71 – 4.66 (m, 1H), 3.04 – 2.93 (m, 3H), 2.43 (s, 3H), 2.25 – 2.15 (m, 1H), 2.03 – 1.95 (m, 1H), 1.93 – 1.87 (m, 1H), 1.86 – 1.79 (m, 2H), 1.78 – 1.62 (m, 3H), 1.47 – 1.36 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.8, 143.7, 136.9, 130.0, 127.23, 127.20, 111.9, 54.8, 52.5, 44.5, 42.0, 41.9, 34.0, 33.9, 33.8, 31.3, 31.2, 29.0, 23.3, 21.8, 21.7; **HRMS (ESI)** calcd $C_{15}H_{20}N_2O_2S_2Na$ $[M + Na]^+$: 347.0858, found: 347.0850; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2924, 2152, 1450, 1156, 662.

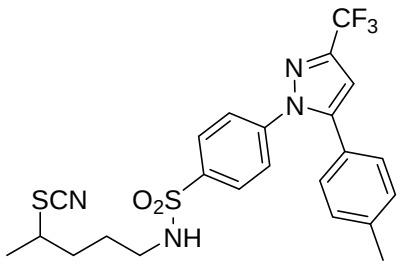


4-Methyl-*N*-(4-thiocyanatocycloheptyl)benzenesulfonamide (5d): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; 51% yield (33 mg); dr = 1.4:1. **Isomer 5d-a:** yellow oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 4.60 (d, *J* = 7.1 Hz, 1H), 3.46 – 3.42 (m, 1H), 3.40 – 3.36 (m, 1H), 2.43 (s, 3H), 2.24 – 2.18 (m, 1H), 2.00

– 1.87 (m, 3H), 1.81 – 1.76 (m, 1H), 1.76 – 1.69 (m, 2H), 1.67 – 1.61 (m, 1H), 1.40 – 1.30 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.7, 138.0, 130.0, 127.1, 111.9, 53.6, 49.1, 36.2, 35.7, 31.5, 29.8, 22.1, 21.7; **HRMS (ESI)** calcd $C_{15}H_{20}N_2O_2S_2Na$ [M + Na]$^+$: 347.0858, found: 347.0852; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3273, 2921, 2150, 1461, 1155, 664. **Isomer 5d-b:** yellow oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 4.63 (d, *J* = 7.2 Hz, 1H), 3.43 – 3.38 (m, 1H), 3.36 – 3.30 (m, 1H), 2.44 (s, 3H), 2.17 – 2.10 (m, 2H), 1.99 – 1.94 (m, 1H), 1.86 – 1.81 (m, 1H), 1.78 – 1.70 (m, 1H), 1.64 – 1.55 (m, 3H), 1.51 – 1.41 (m, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.7, 137.8, 130.0, 127.1, 111.8, 54.1, 49.2, 35.6, 35.0, 33.0, 31.1, 21.7, 20.9; **HRMS (ESI)** calcd $C_{15}H_{20}N_2O_2S_2Na$ [M + Na]$^+$: 347.0858, found: 347.0851; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3274, 2925, 2151, 1460, 1154, 663.

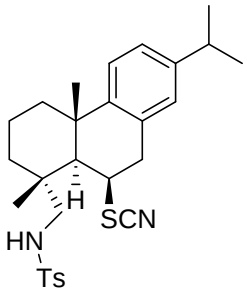


4-Methyl-*N*-(4-methyl-4-thiocyanatopentyl)benzenesulfonamide (5e): Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 51% yield (31.5 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 4.84 (t, *J* = 6.2 Hz, 1H), 2.96 (q, *J* = 6.6 Hz, 2H), 2.43 (s, 3H), 1.72 – 1.69 (m, 2H), 1.61 – 1.57 (m, 2H), 1.45 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 143.8, 136.9, 129.9, 127.2, 111.7, 55.4, 43.1, 39.8, 28.9, 25.5, 21.7; **HRMS (ESI)** calcd $C_{14}H_{21}N_2O_2S_2$ [M + H]$^+$: 313.1039, found: 313.1038; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3275, 2924, 2150, 1457, 1155, 661.

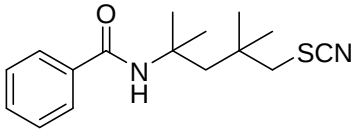


***N*-(4-Thiocyanatopentyl)-4-(5-(*p*-tolyl)-3-(trifluoromethyl)-*1H*-pyrazol-1-yl)benzenesulfonamide (5f):** Purification by column chromatography on silica gel with

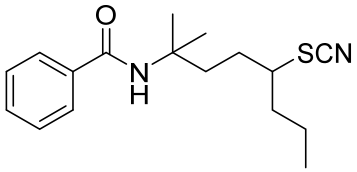
petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 44% yield (37 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.6 Hz, 2H), 7.48 (d, *J* = 8.6 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 6.74 (s, 1H), 4.74 (t, *J* = 6.2 Hz, 1H), 3.25 – 3.19 (m, 1H), 2.98 (q, *J* = 6.6 Hz, 2H), 2.38 (s, 3H), 1.81 – 1.73 (m, 2H), 1.72 – 1.66 (m, 2H), 1.50 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 145.4, 144.3 (q, J_{C-F} = 38.4 Hz), 142.8, 140.0, 139.4, 129.9, 128.9, 128.2, 125.8, 121.17 (q, J_{C-F} = 268.9 Hz), 111.1, 106.5, 45.1, 42.7, 34.0, 27.4, 22.2, 21.5; **HRMS (ESI)** calcd $C_{23}H_{24}F_3N_4O_2S_2$ $[M + H]^+$: 509.1287, found: 509.1292; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3274, 2925, 2152, 1471, 1158, 807.



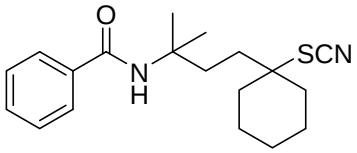
***N*-(((*1R,4aS,10R,10aR*)-7-Isopropyl-1,4a-dimethyl-10-thiocyanato-1,2,3,4,4a,9,10, 10a-octahydrophenanthren-1-yl)methyl)-4-methylbenzenesulfonamide (5g):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 46% yield (46 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.04 (s, 1H), 4.63 – 4.57 (m, 1H), 3.87 – 3.83 (m, 1H), 3.63 (dd, *J* = 16.6, 5.8 Hz, 1H), 3.11 (dd, *J* = 16.6, 1.1 Hz, 1H), 2.95 (dd, *J* = 12.9, 9.1 Hz, 1H), 2.91 – 2.85 (m, 1H), 2.70 (dd, *J* = 12.9, 5.5 Hz, 1H), 2.43 (s, 3H), 2.17 – 2.14 (m, 1H), 1.82 – 1.75 (m, 2H), 1.62 – 1.55 (m, 2H), 1.54 – 1.49 (m, 1H), 1.39 – 1.34 (m, 1H), 1.26 (s, 3H), 1.25 (d, *J* = 6.9 Hz, 6H), 1.14 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 147.1, 145.7, 143.8, 136.8, 132.1, 130.0, 127.6, 127.1, 125.6, 121.8, 112.0, 55.0, 52.4, 47.8, 39.1, 39.0, 38.3, 37.5, 36.3, 33.7, 24.2, 24.1, 22.6, 21.7, 18.3, 17.9; **HRMS (ESI)** calcd $C_{28}H_{37}N_2O_2S_2$ $[M + H]^+$: 497.2291, found: 497.2289; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3278, 2929, 2150, 1445, 1159, 728.



***N*-(2,4,4-Trimethyl-5-thiocyanatopentan-2-yl)benzamide (5h):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 60% yield (35 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.70 – 7.67 (m, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 5.89 (s, 1H), 3.06 (s, 2H), 2.08 (s, 2H), 1.52 (s, 6H), 1.17 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.3, 135.7, 131.5, 128.8, 126.7, 114.0, 55.0, 49.1, 47.3, 36.6, 30.0, 27.4; **HRMS (ESI)** calcd $C_{16}H_{22}N_2OSNa$ $[M + Na]^+$: 313.1345, found: 313.1338; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3316, 2964, 2153, 1641, 1221, 715.

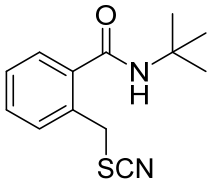


***N*-(2-Methyl-5-thiocyanatooctan-2-yl)benzamide (5i):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; yellow oil; 51% yield (31 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 5.86 (s, 1H), 3.08 – 3.02 (m, 1H), 2.18 – 2.12 (m, 1H), 2.06 – 2.00 (m, 1H), 1.82 – 1.69 (m, 4H), 1.57 – 1.52 (m, 1H), 1.46 (s, 3H), 1.43 (s, 3H), 1.42 – 1.37 (m, 1H), 0.93 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.2, 135.7, 131.5, 128.7, 126.8, 111.4, 53.9, 51.7, 37.4, 36.6, 30.5, 27.6, 20.3, 13.7; **HRMS (ESI)** calcd $C_{17}H_{24}N_2OSNa$ $[M + Na]^+$: 327.1502, found: 327.1494; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3321, 2927, 2151, 1643, 1292, 713.



***N*-(2-Methyl-4-(1-thiocyanatocyclohexyl)butan-2-yl)benzamide (5j):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1,

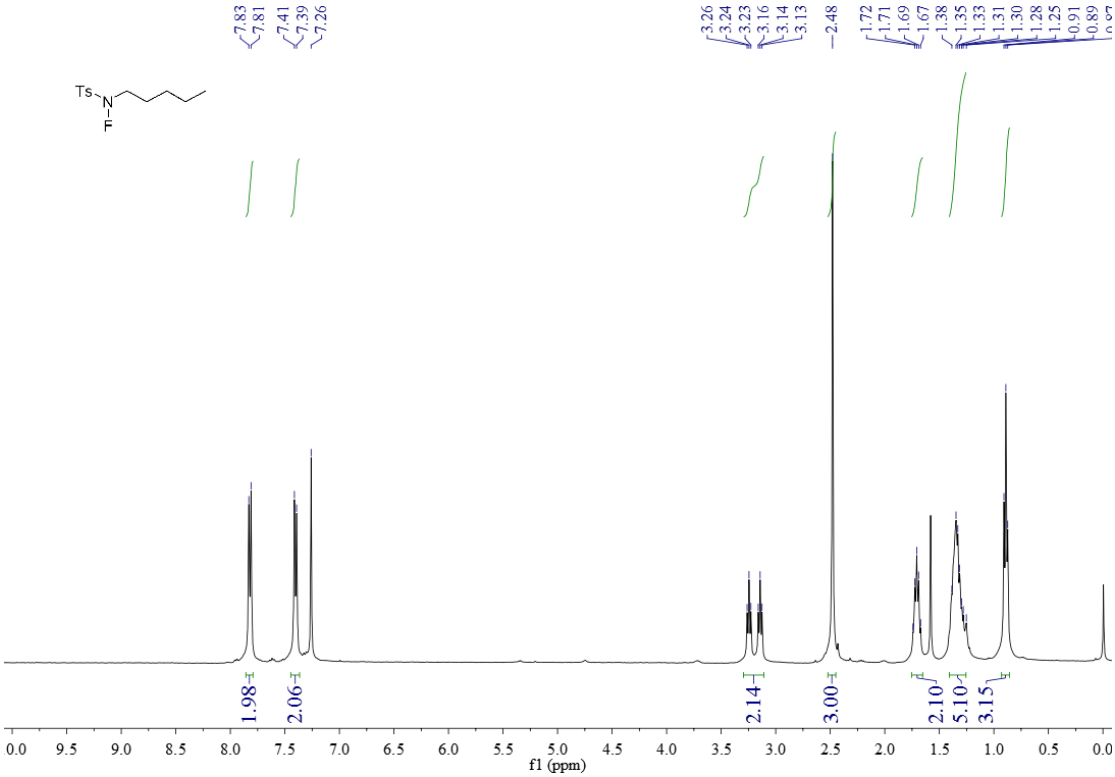
v:v) as the eluent; yellow oil; yellow oil; 43% yield (28.3 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.3 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 5.87 (s, 1H), 2.11 – 2.06 (m, 2H), 1.92 – 1.87 (m, 2H), 1.82 – 1.78 (m, 2H), 1.68 – 1.57 (m, 8H), 1.46 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.2, 135.8, 131.4, 128.7, 126.8, 111.9, 61.6, 53.8, 36.4, 36.3, 33.6, 27.6, 25.4, 22.4; **HRMS (ESI)** calcd $C_{19}H_{26}N_2OSNa$ $[M + Na]^+$: 353.1658, found: 353.1650; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3357, 2931, 2148, 1645, 1278, 729.



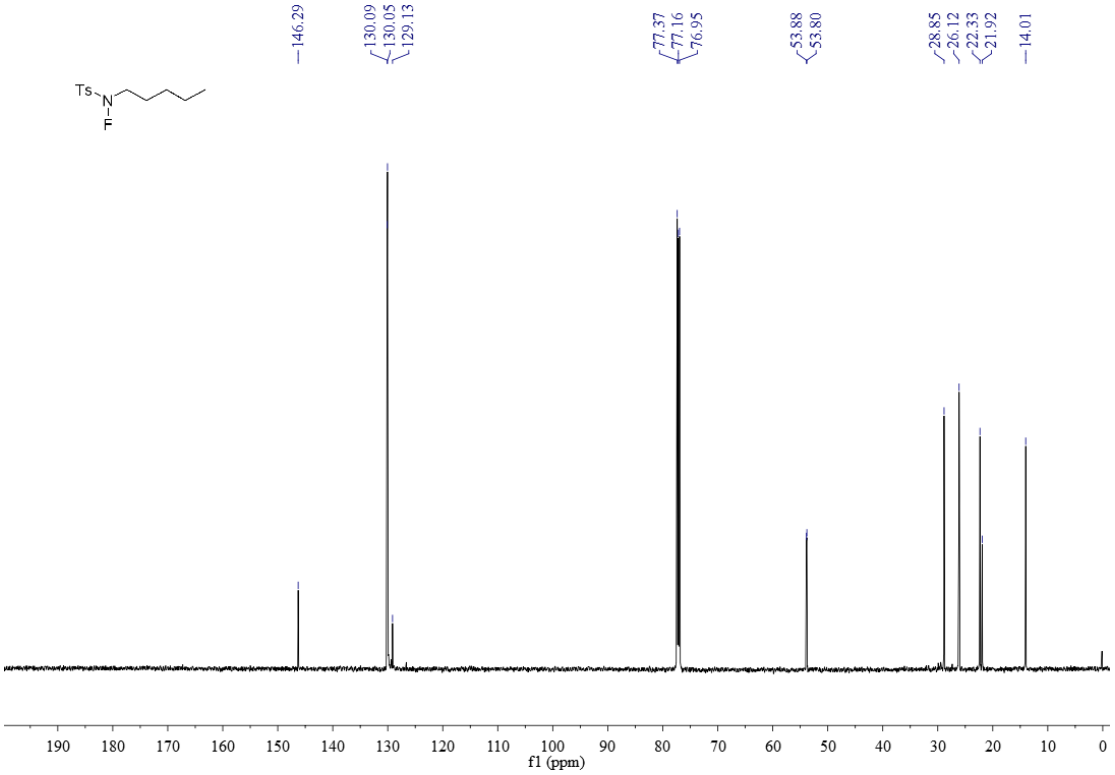
***N*-(*tert*-Butyl)-2-(thiocyanatomethyl)benzamide (5k):** Purification by column chromatography on silica gel with petroleum ether–ethyl acetate (~ 8:1, v:v) as the eluent; white solid; m.p. 86 – 89 ℃; 58% yield (29 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.47 – 7.40 (m, 2H), 7.40 – 7.34 (m, 2H), 5.87 (s, 1H), 4.38 (s, 2H), 1.46 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 168.4, 136.2, 134.6, 131.3, 130.8, 129.1, 127.4, 113.2, 52.3, 36.6, 28.8; **HRMS (ESI)** calcd $C_{13}H_{16}N_2OSNa$ $[M + Na]^+$: 271.0876, found: 271.0868; **FT-IR** (thin film, KBr): ν (cm^{-1}) 3310, 2924, 2152, 1577, 1220, 659.

5. NMR Spectra for the substrates and products

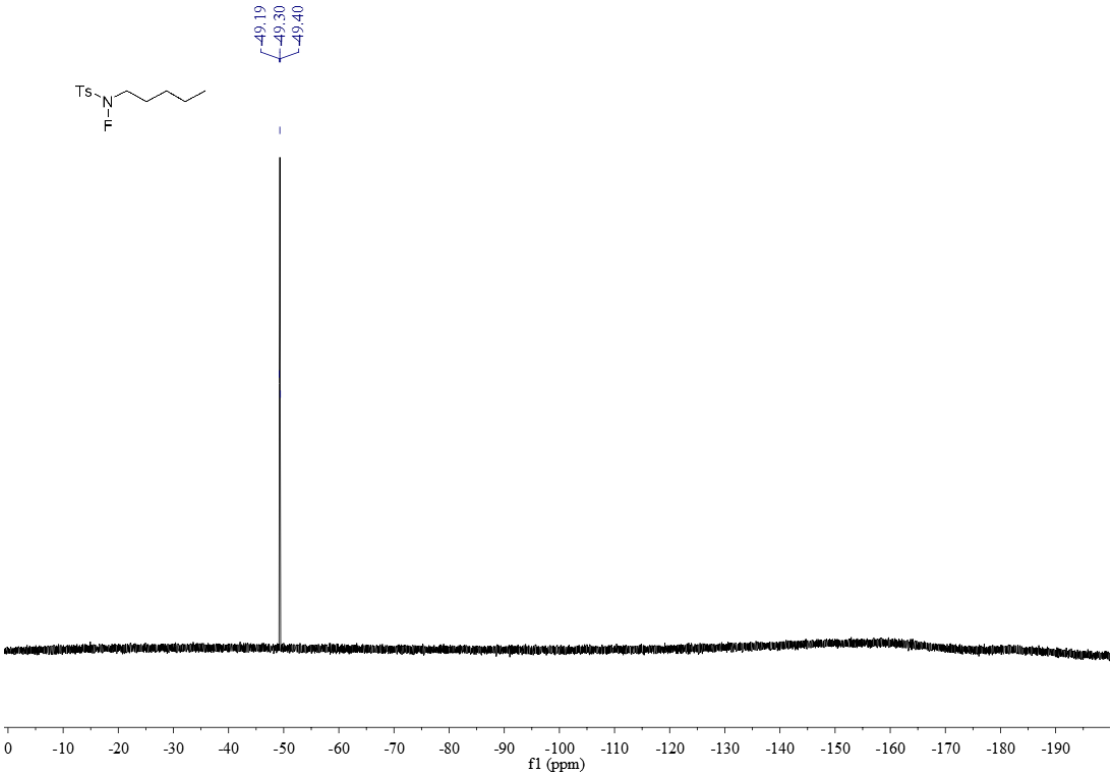
^{1}H NMR of 1a: 400 MHz, $CDCl_3$



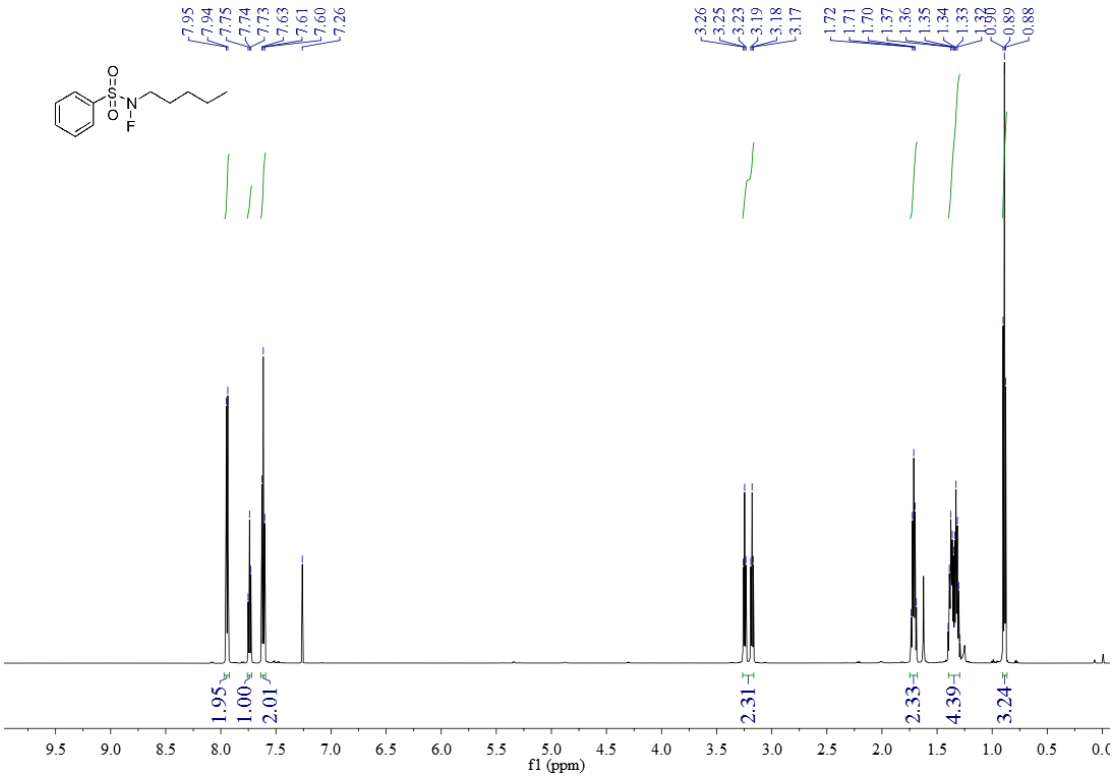
^{13}C NMR of 1a: 150 MHz, $CDCl_3$



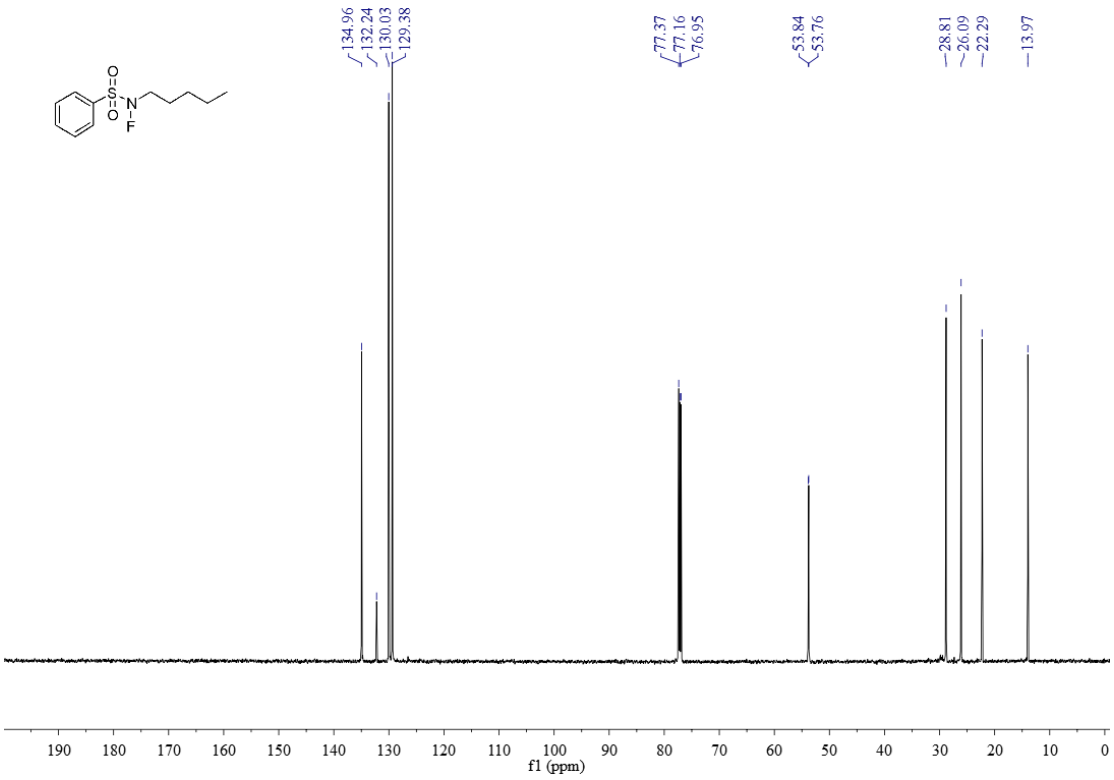
^{19}F NMR of 1a: 377 MHz, $CDCl_3$



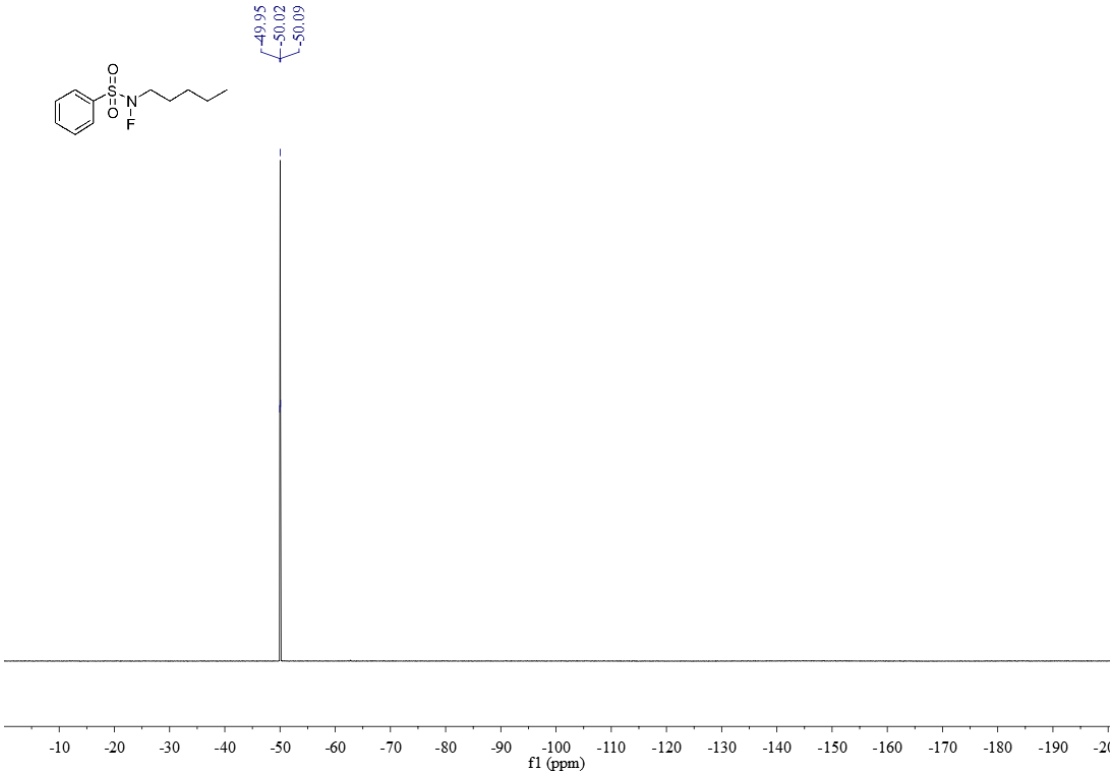
^{1}H NMR of 1b: 600 MHz, $CDCl_3$



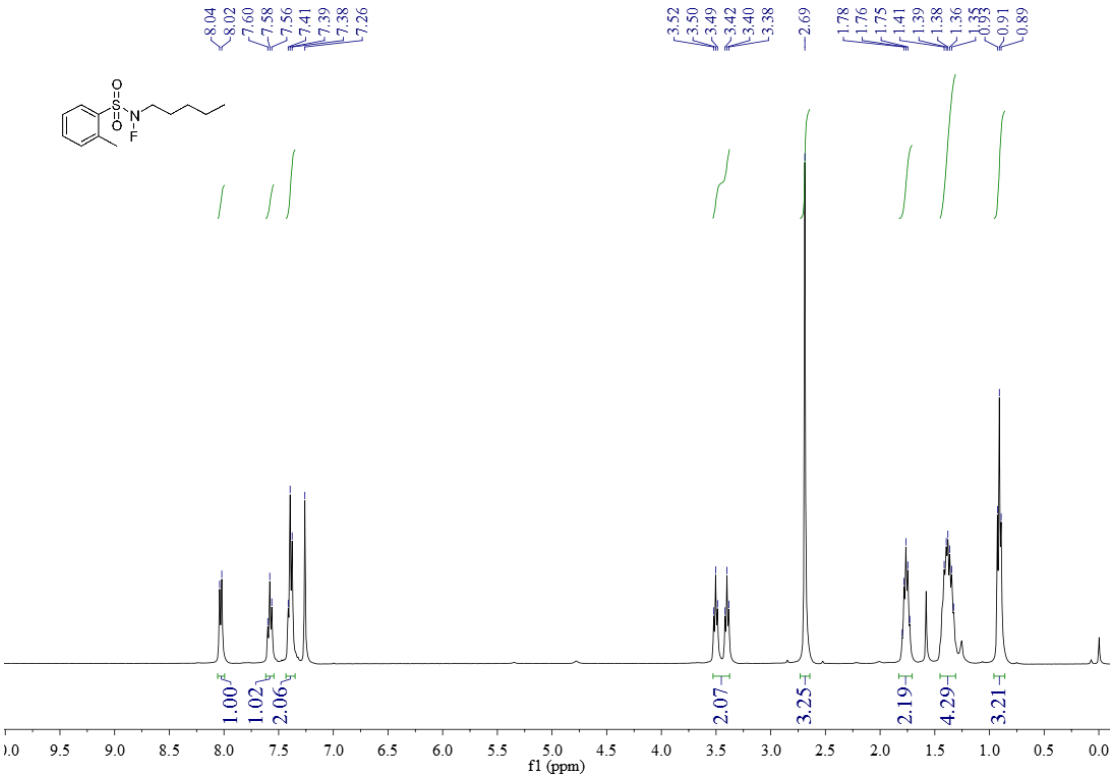
^{13}C NMR of 1b: 150 MHz, $CDCl_3$



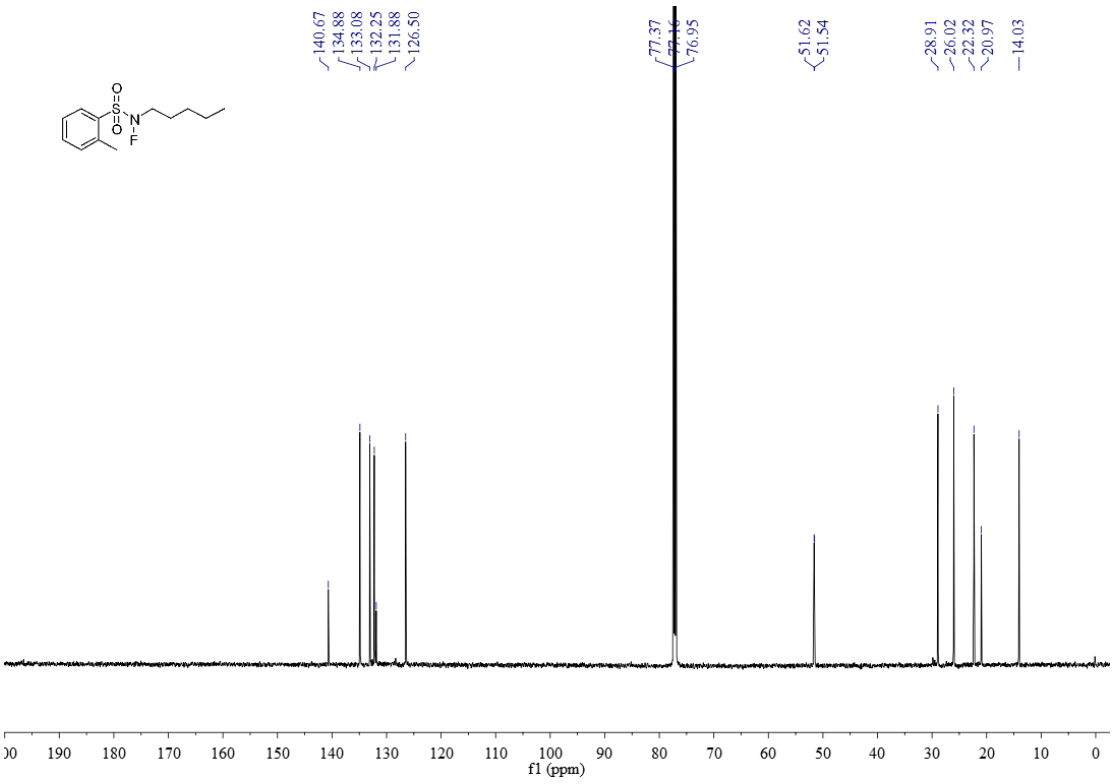
^{19}F NMR of 1b: 564 MHz, $CDCl_3$



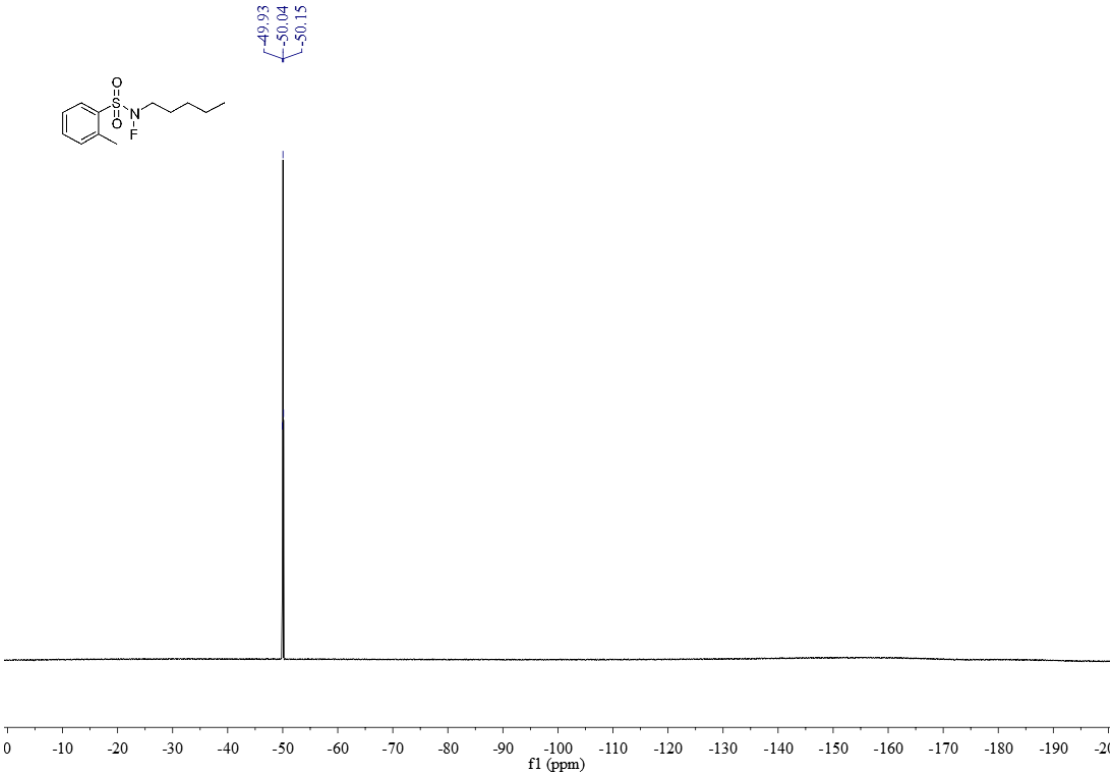
^{1}H NMR of 1c: 400 MHz, $CDCl_3$



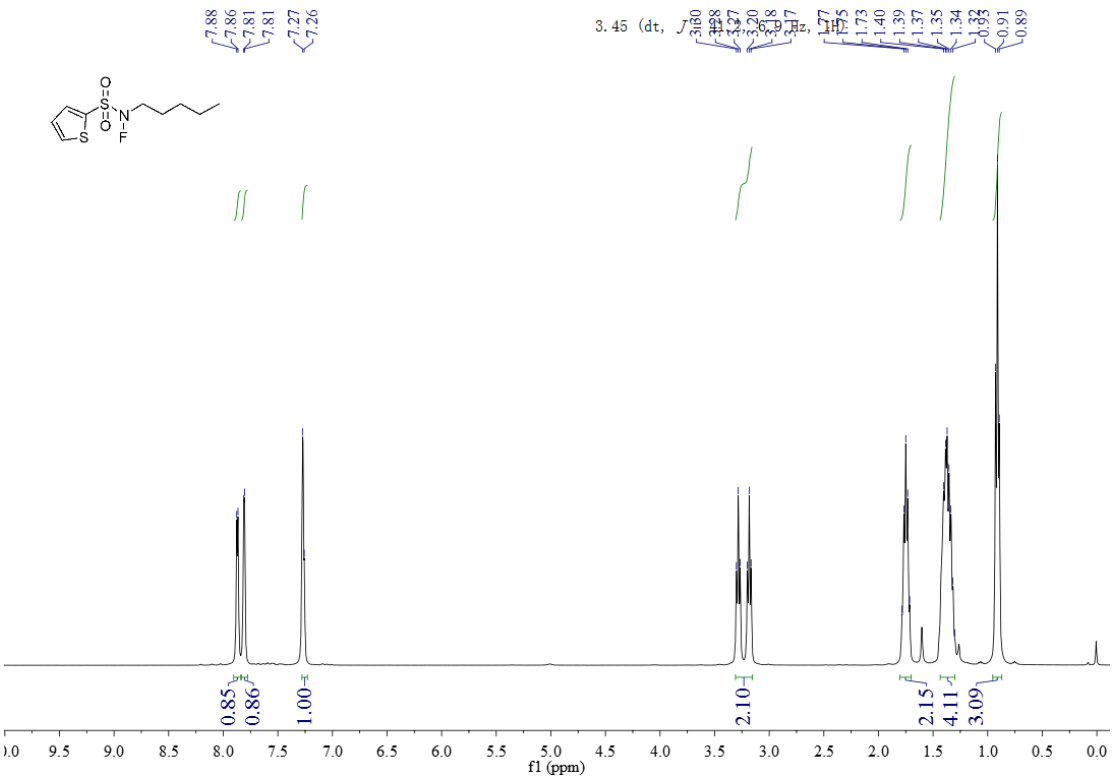
^{13}C NMR of 1c: 150 MHz, $CDCl_3$



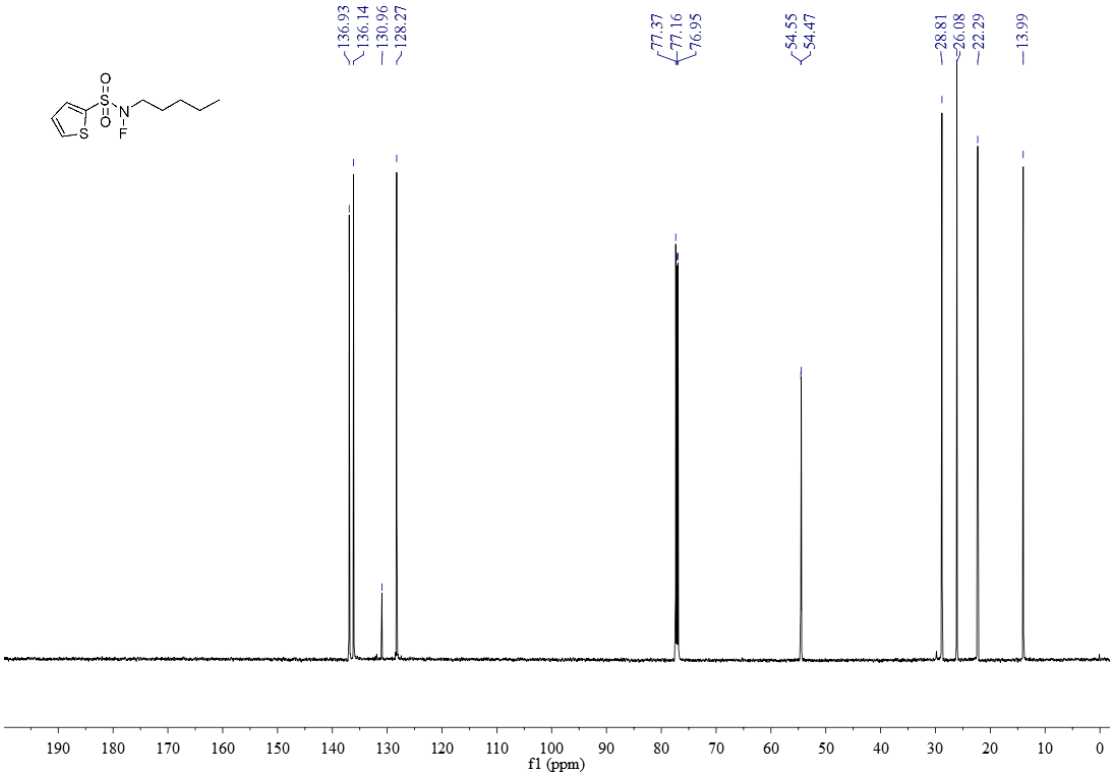
^{19}F NMR of 1c: 377 MHz, $CDCl_3$



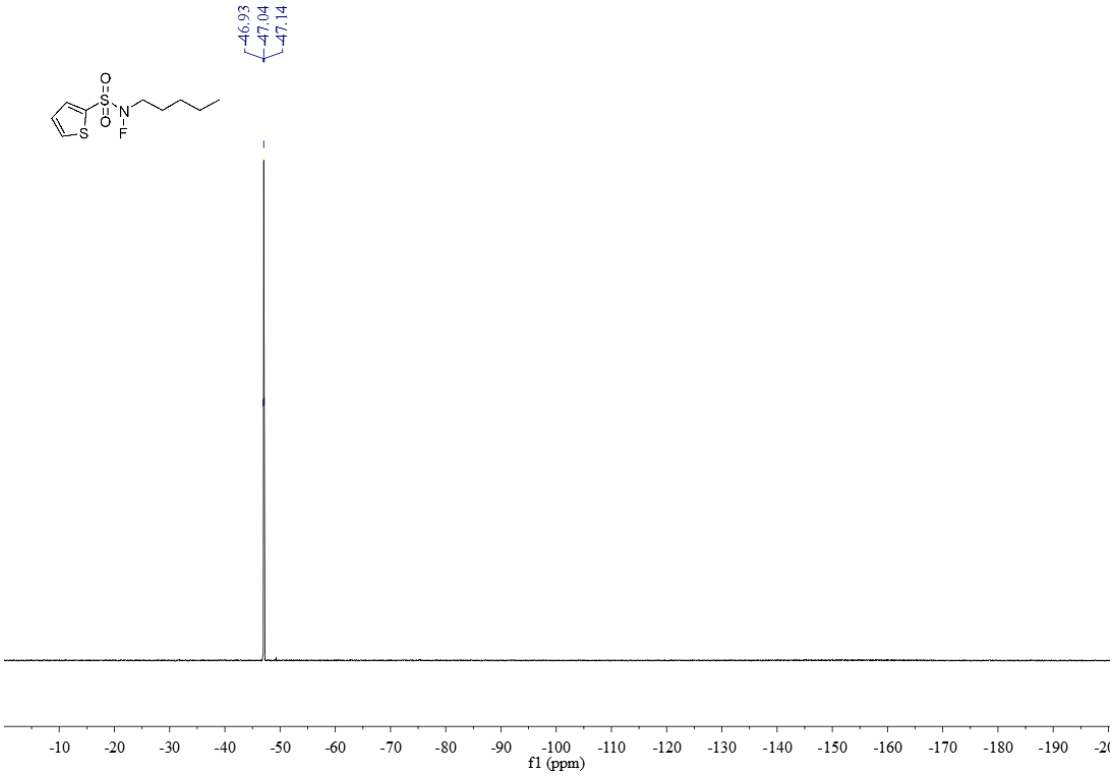
^{1}H NMR of 1d: 400 MHz, $CDCl_3$



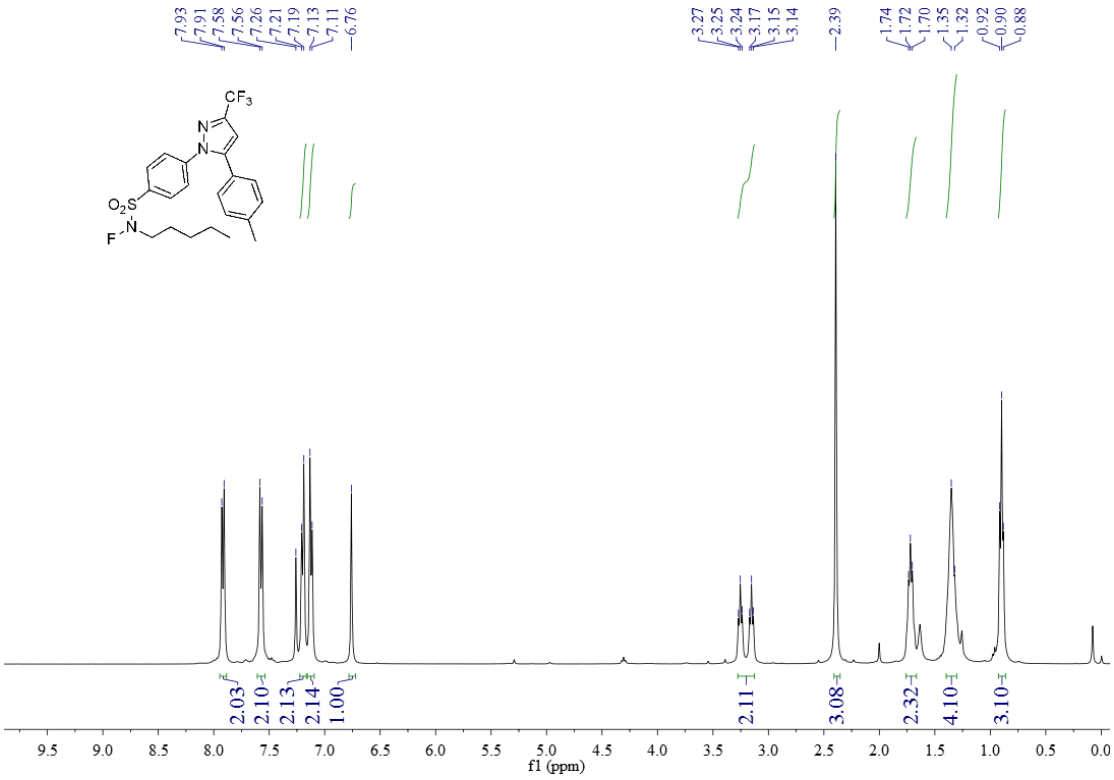
^{13}C NMR of 1d: 150 MHz, $CDCl_3$



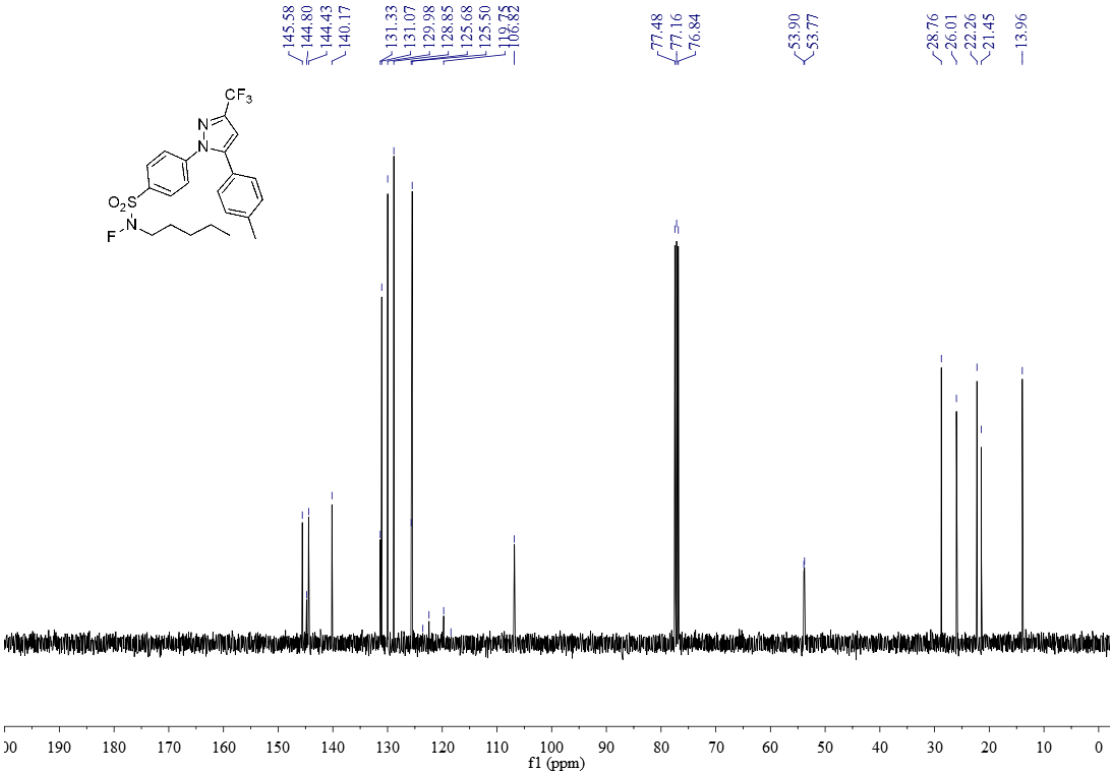
^{19}F NMR of 1d: 377 MHz, $CDCl_3$



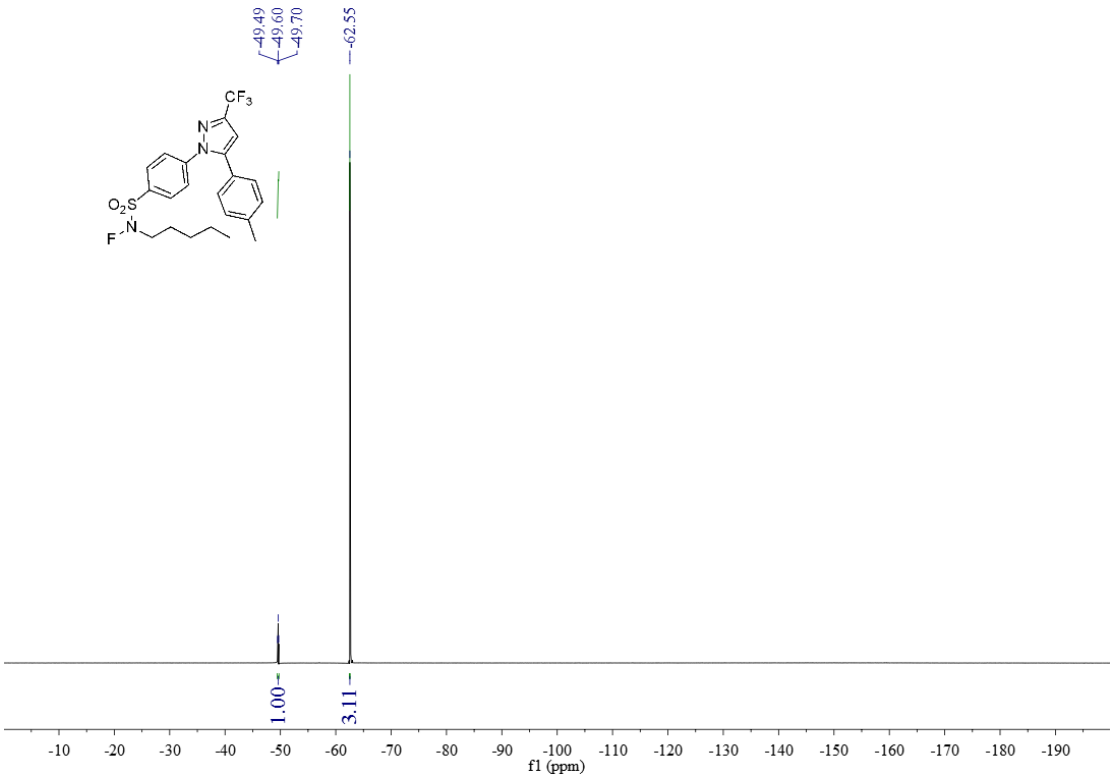
^{1}H NMR of 1e: 400 MHz, $CDCl_3$



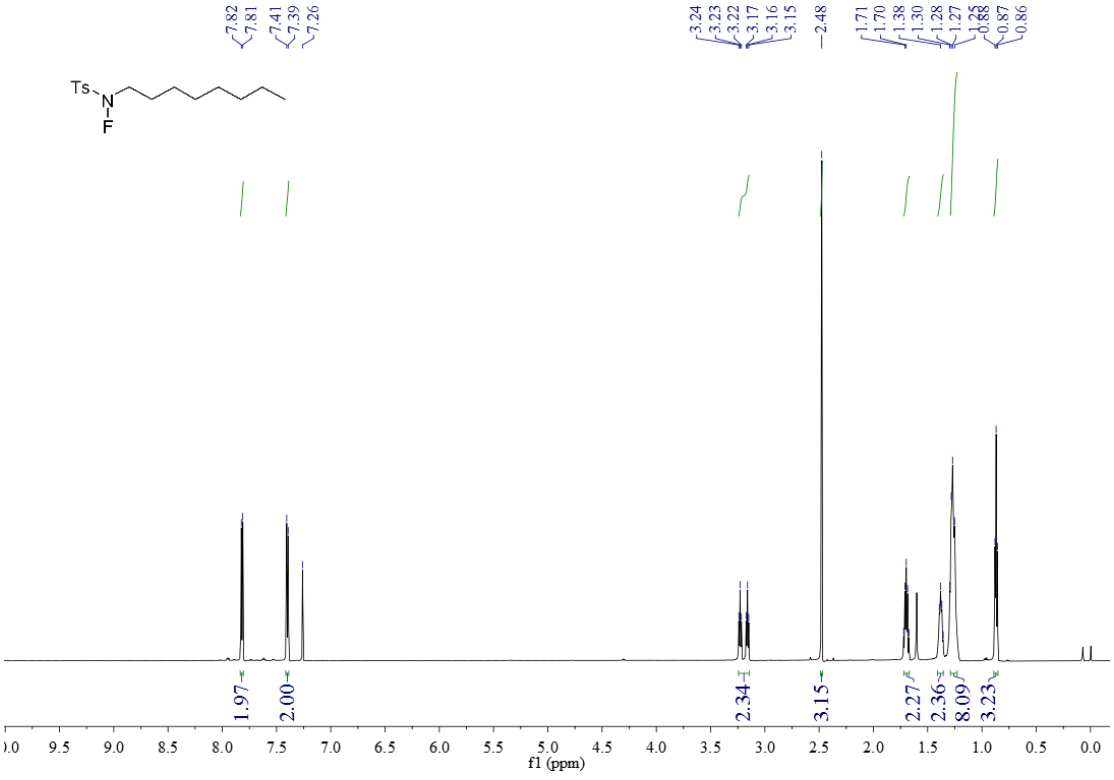
^{13}C NMR of 1e: 100 MHz, $CDCl_3$



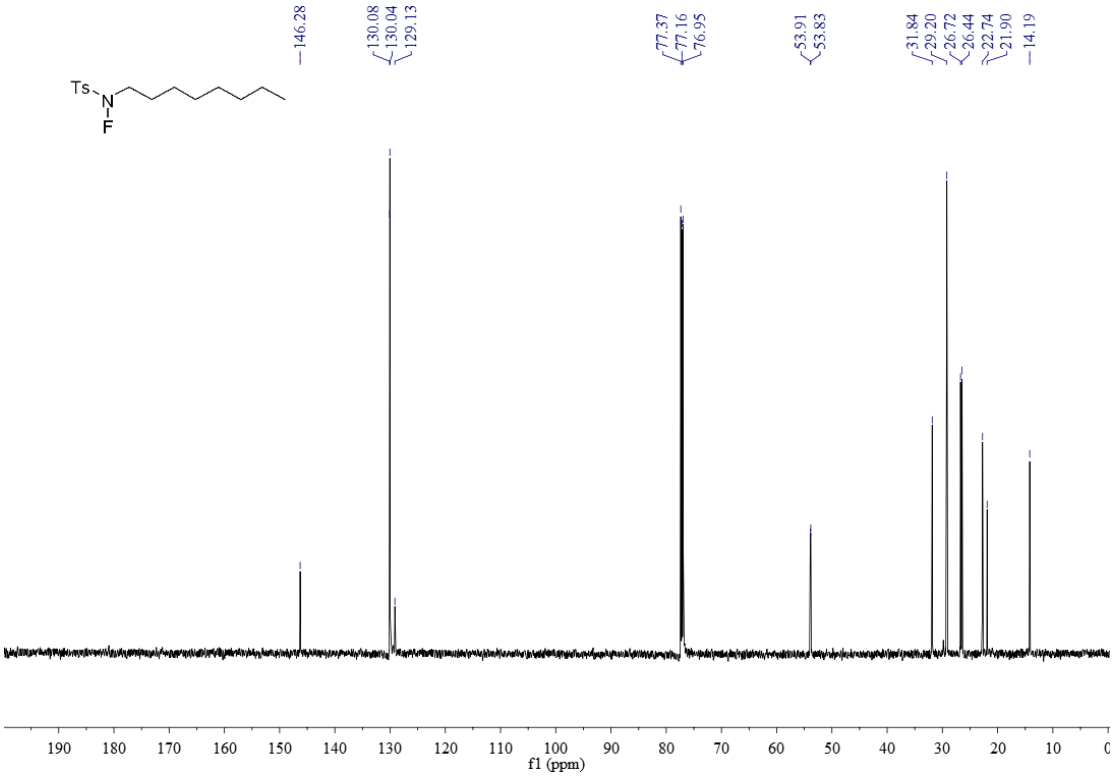
^{19}F NMR of 1e: 377 MHz, $CDCl_3$



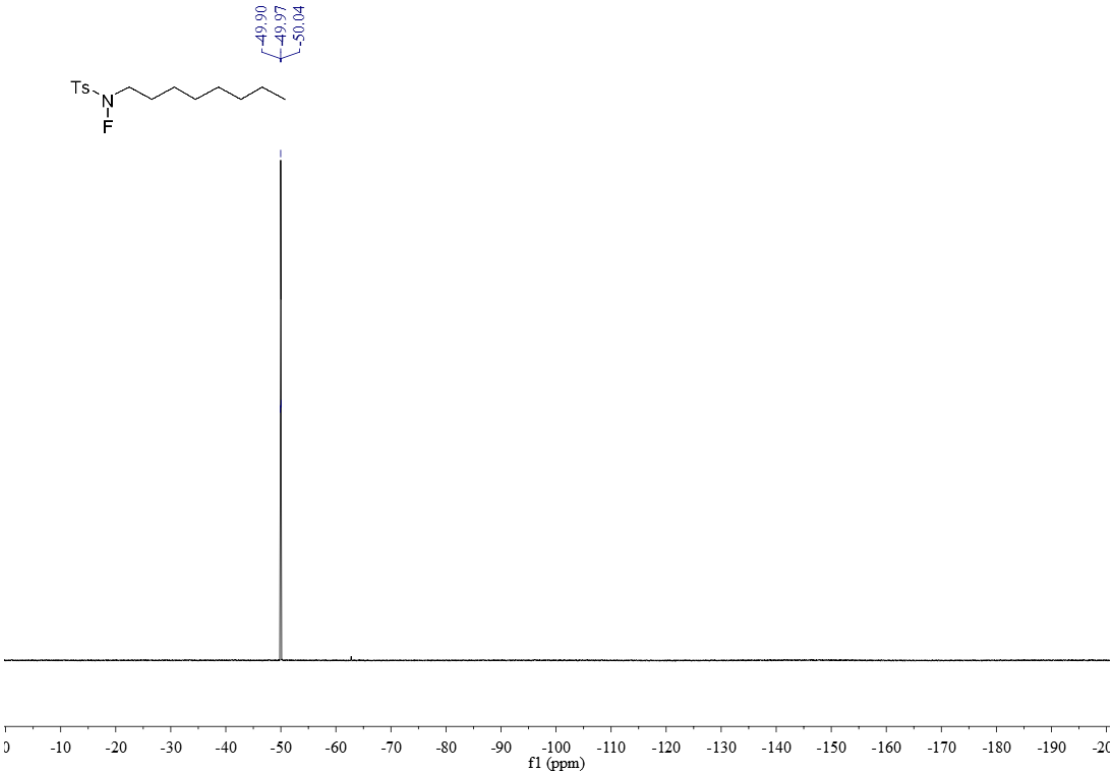
^{1}H NMR of 1f: 600 MHz, $CDCl_3$



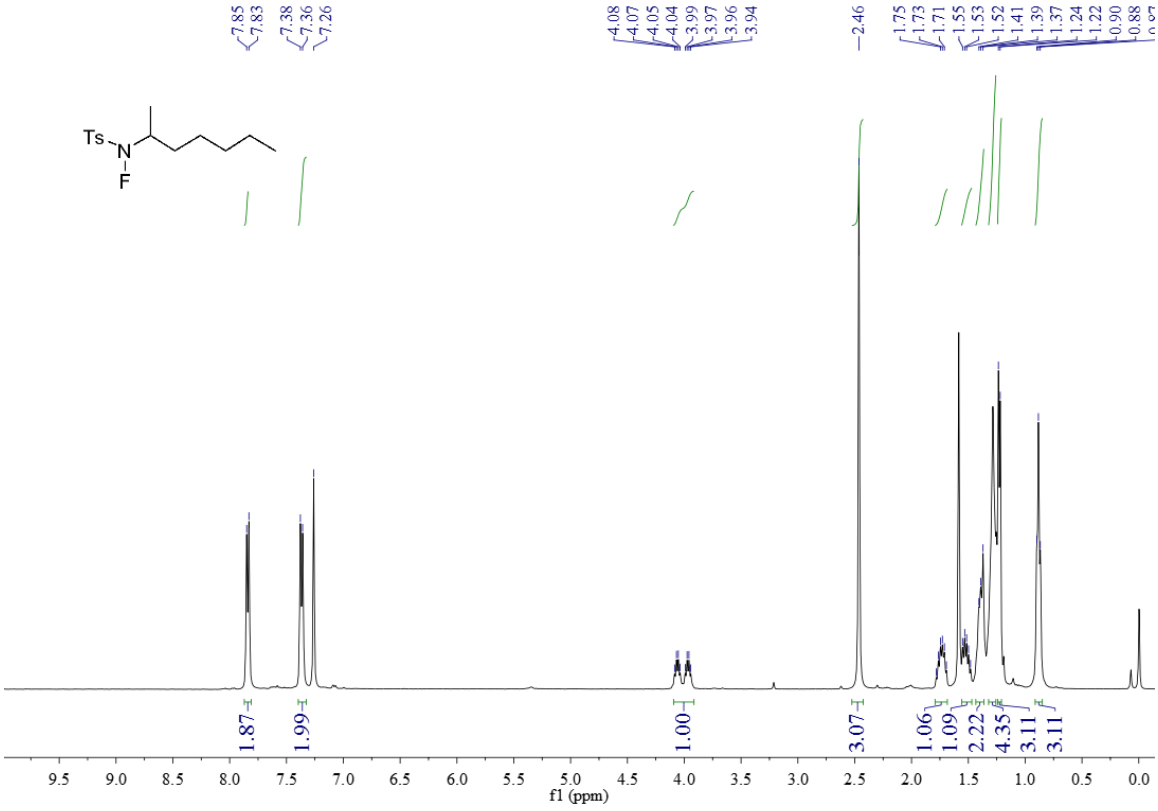
^{13}C NMR of 1f: 150 MHz, $CDCl_3$



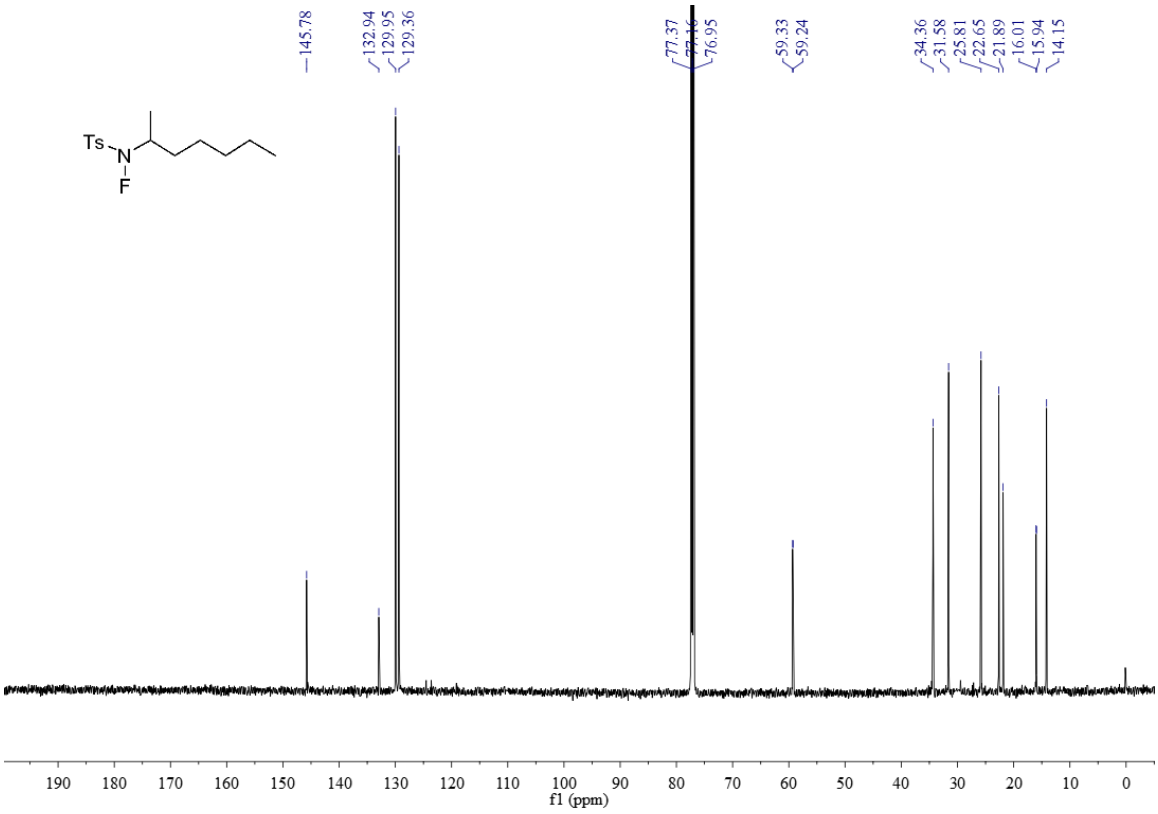
^{19}F NMR of 1f: 564 MHz, $CDCl_3$



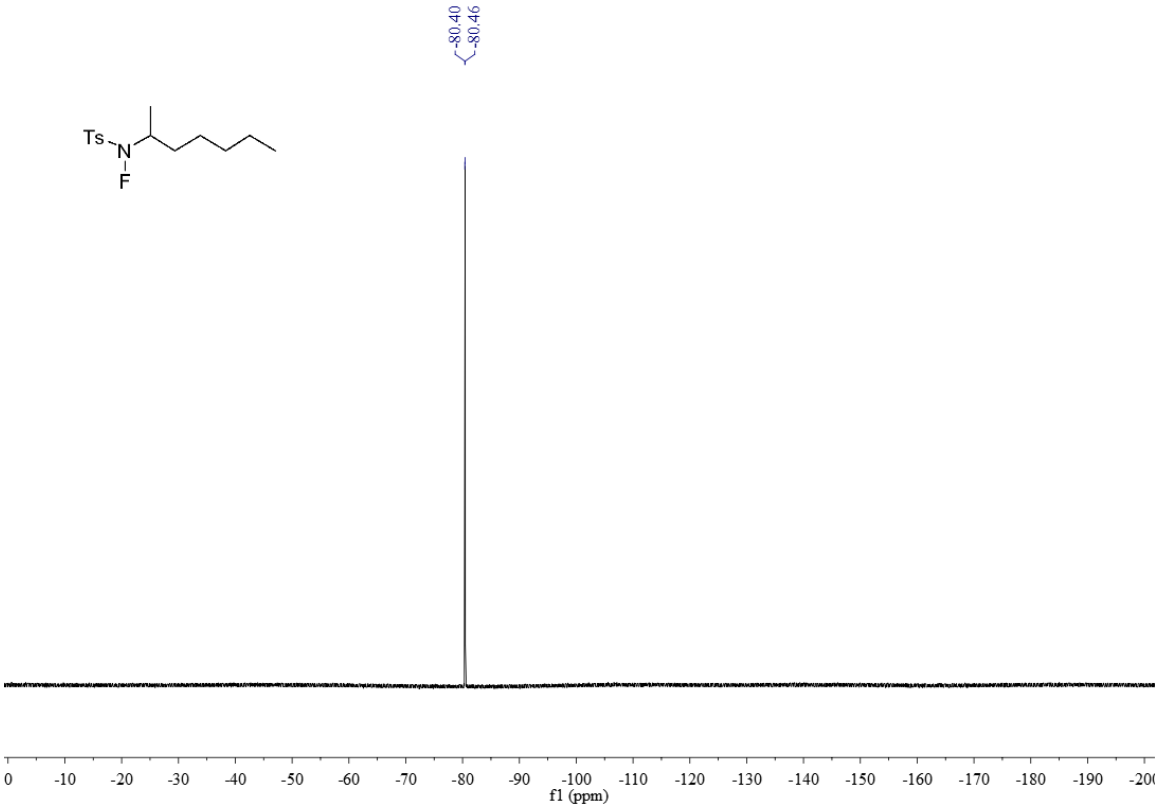
^{1}H NMR of 1g: 400 MHz, $CDCl_3$



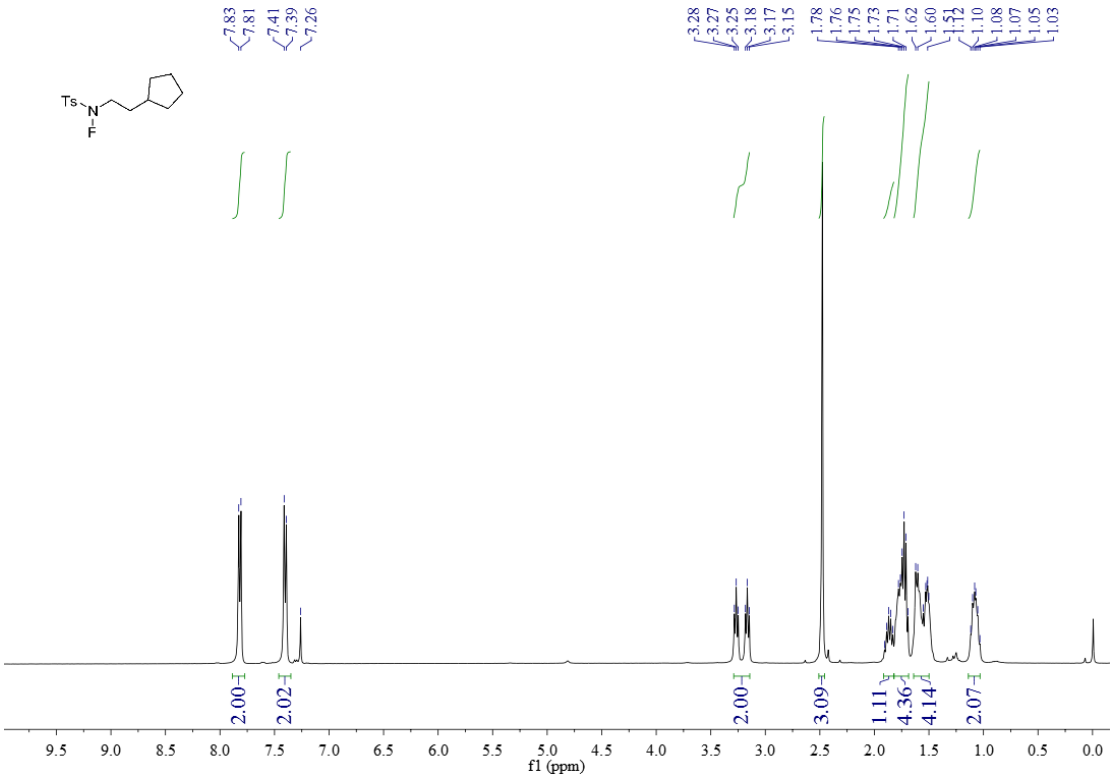
^{13}C NMR of 1g: 150 MHz, $CDCl_3$



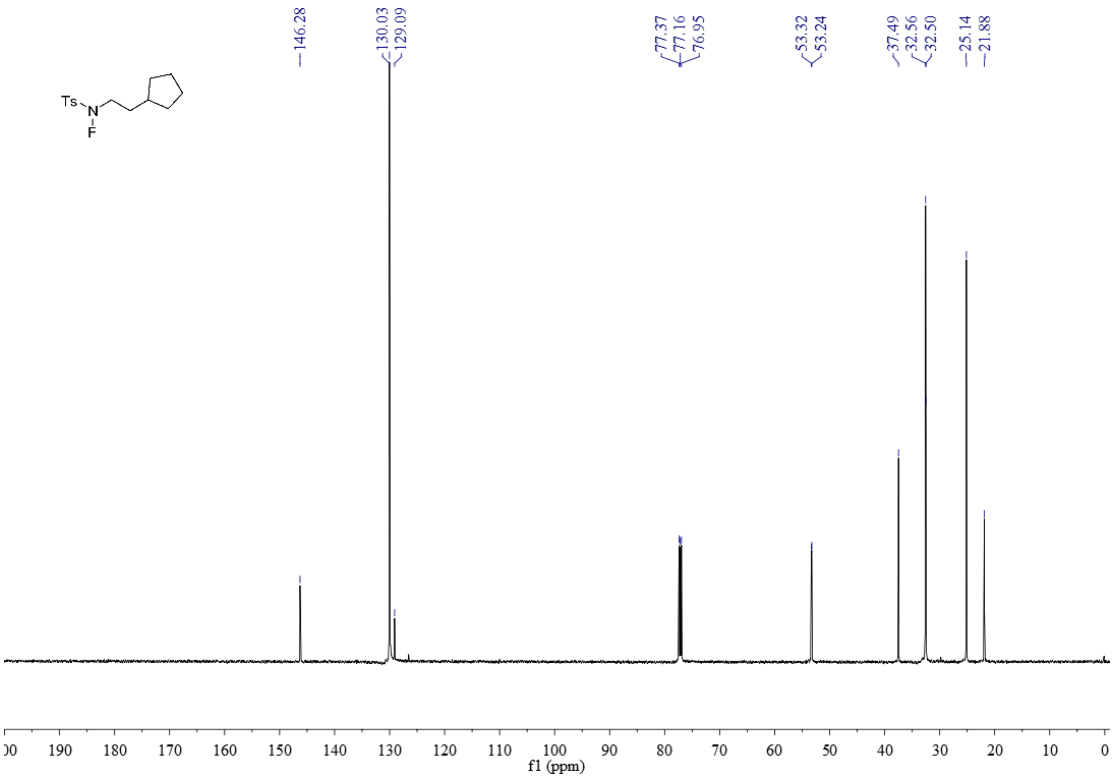
^{19}F NMR of 1g: 564 MHz, $CDCl_3$



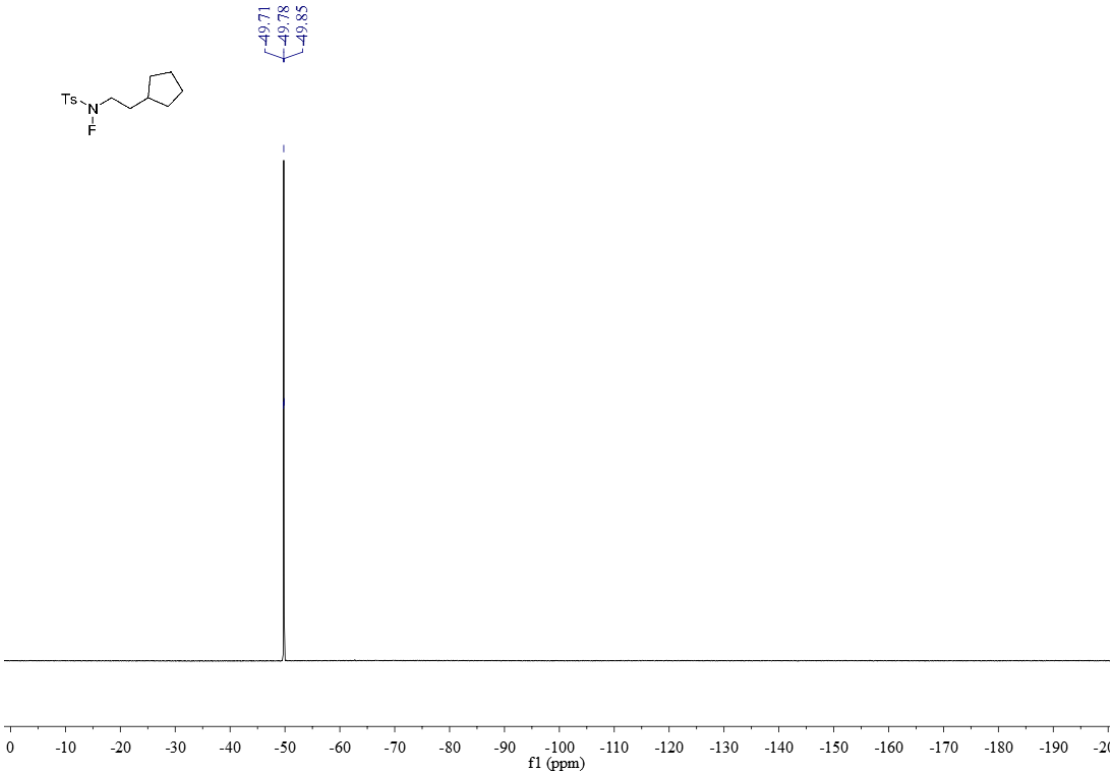
^{1}H NMR of 1h: 400 MHz, $CDCl_3$



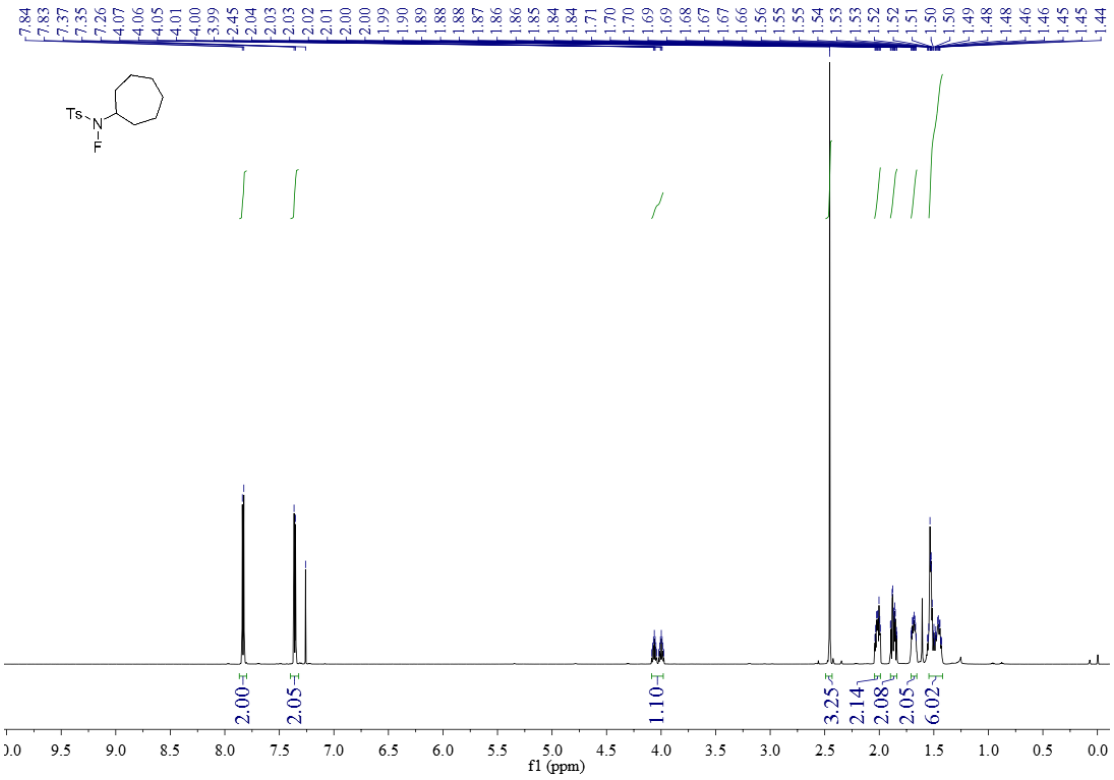
^{13}C NMR of 1h: 150 MHz, $CDCl_3$



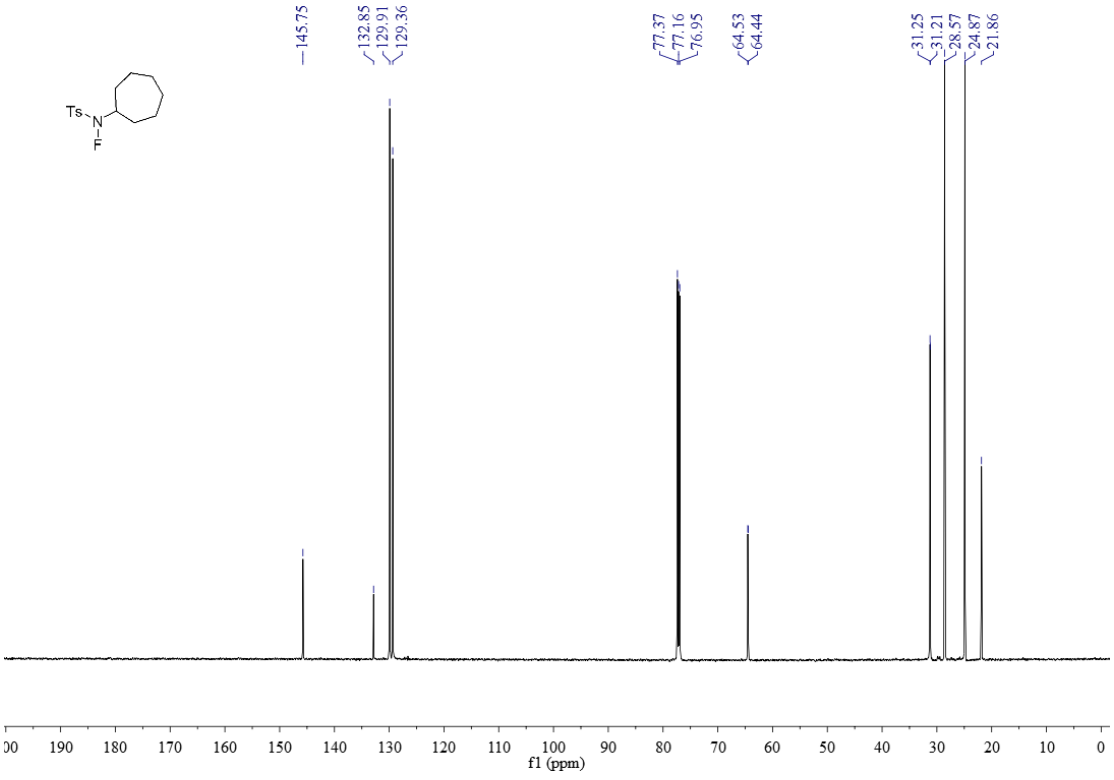
^{19}F NMR of 1h: 564 MHz, $CDCl_3$



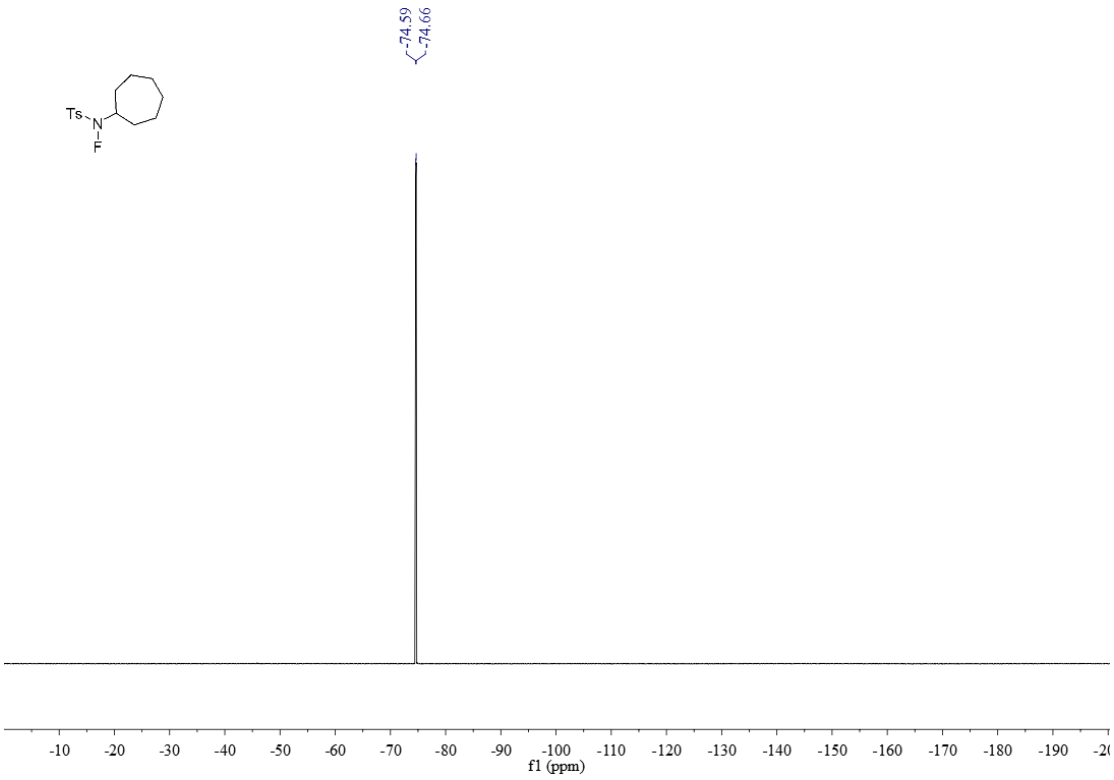
^{1}H NMR of 1i: 600 MHz, $CDCl_3$



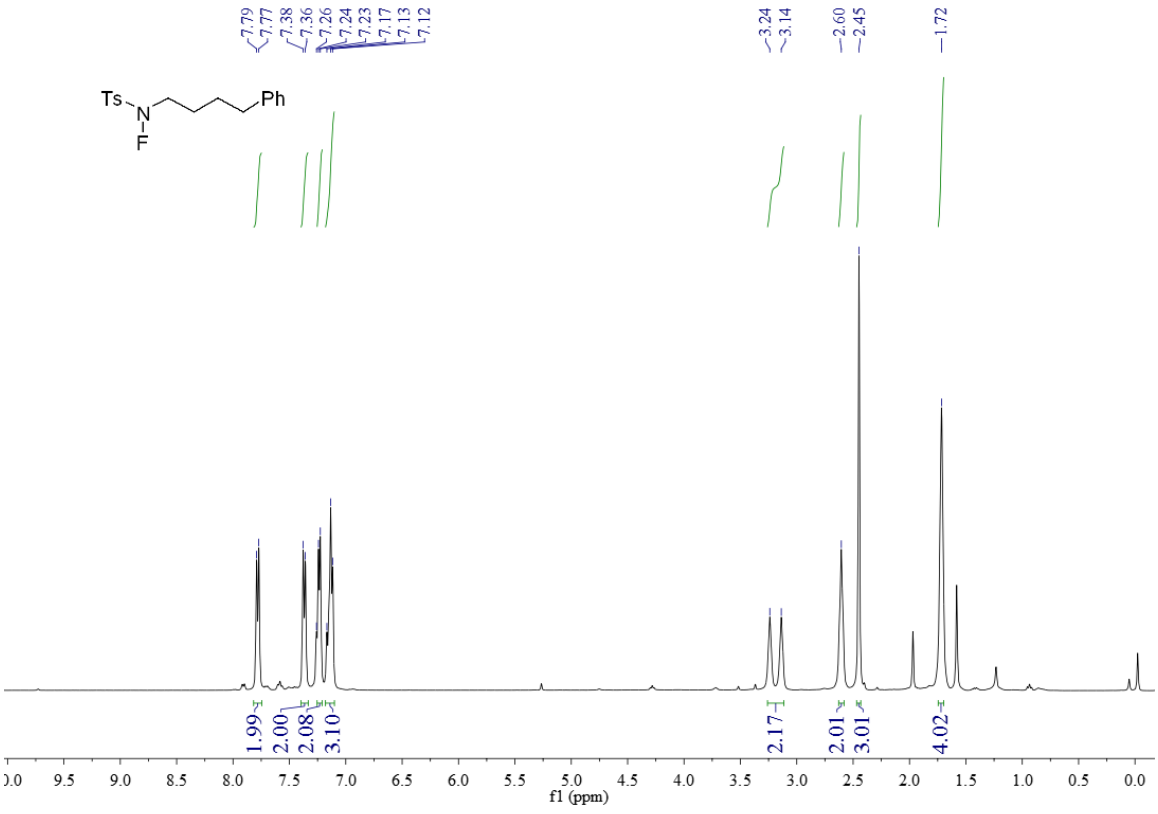
^{13}C NMR of 1i: 150 MHz, $CDCl_3$



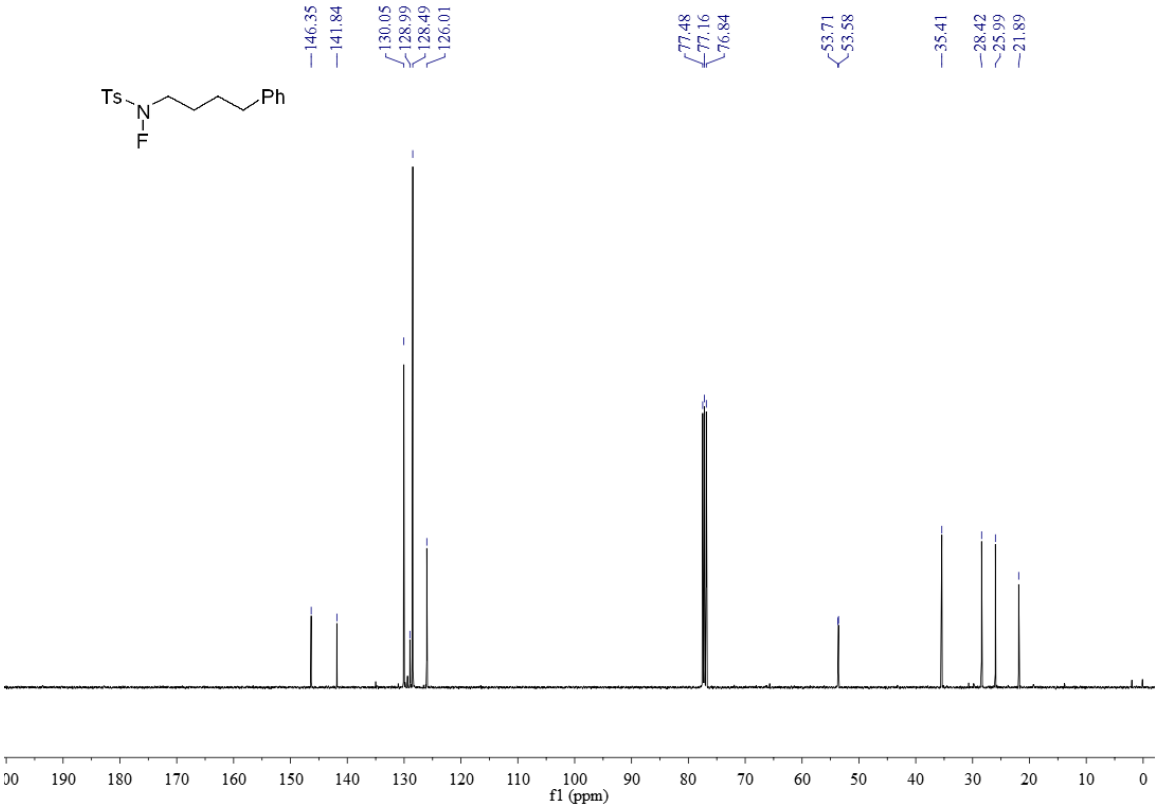
^{19}F NMR of 1i: 564 MHz, $CDCl_3$



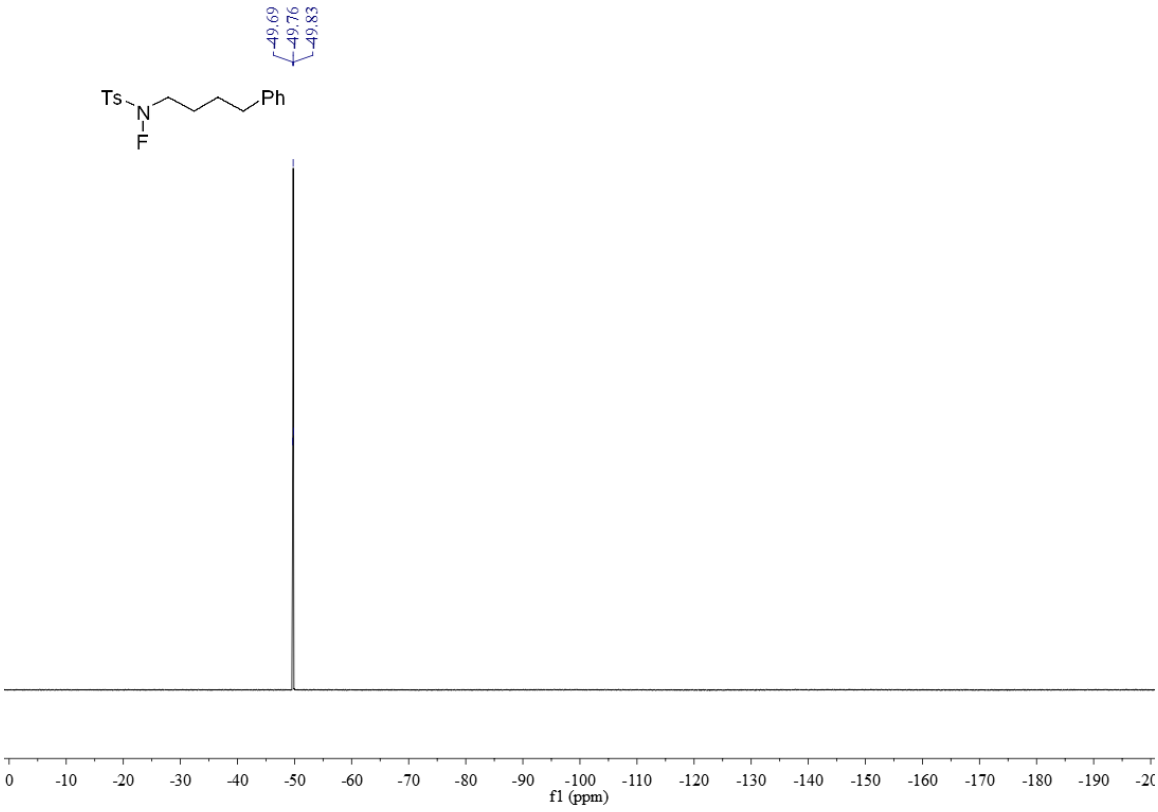
^{1}H NMR of 1j: 400 MHz, $CDCl_3$



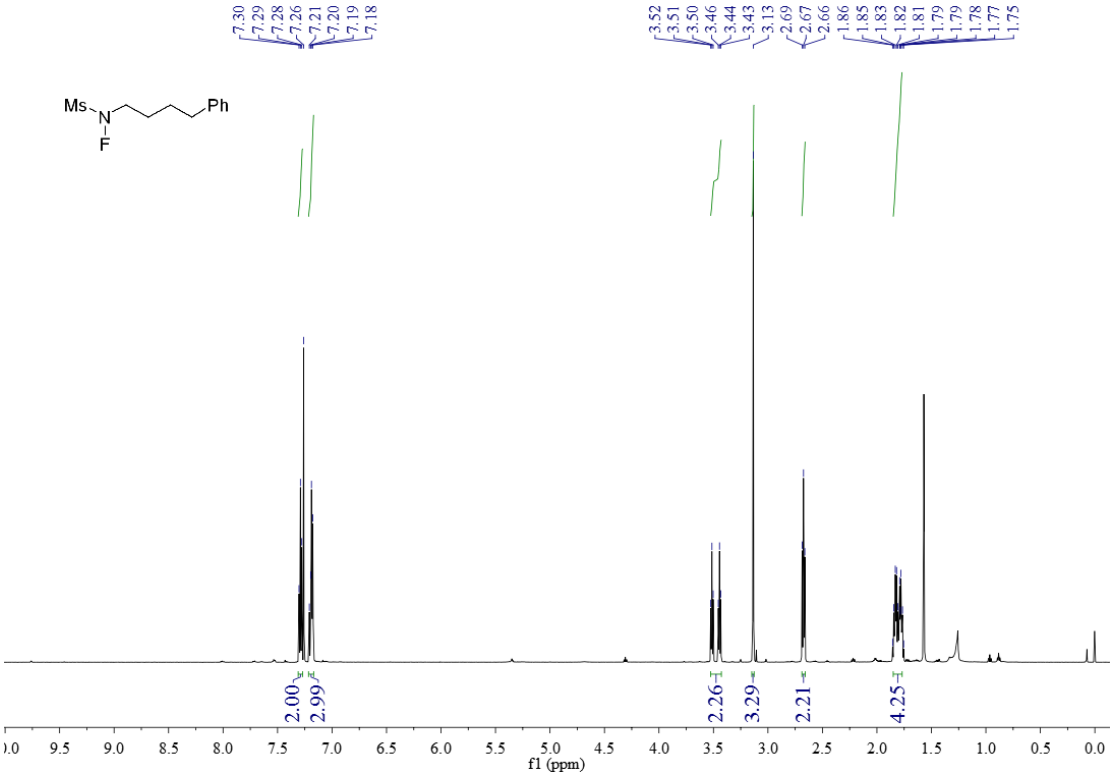
^{13}C NMR of 1j: 100 MHz, $CDCl_3$



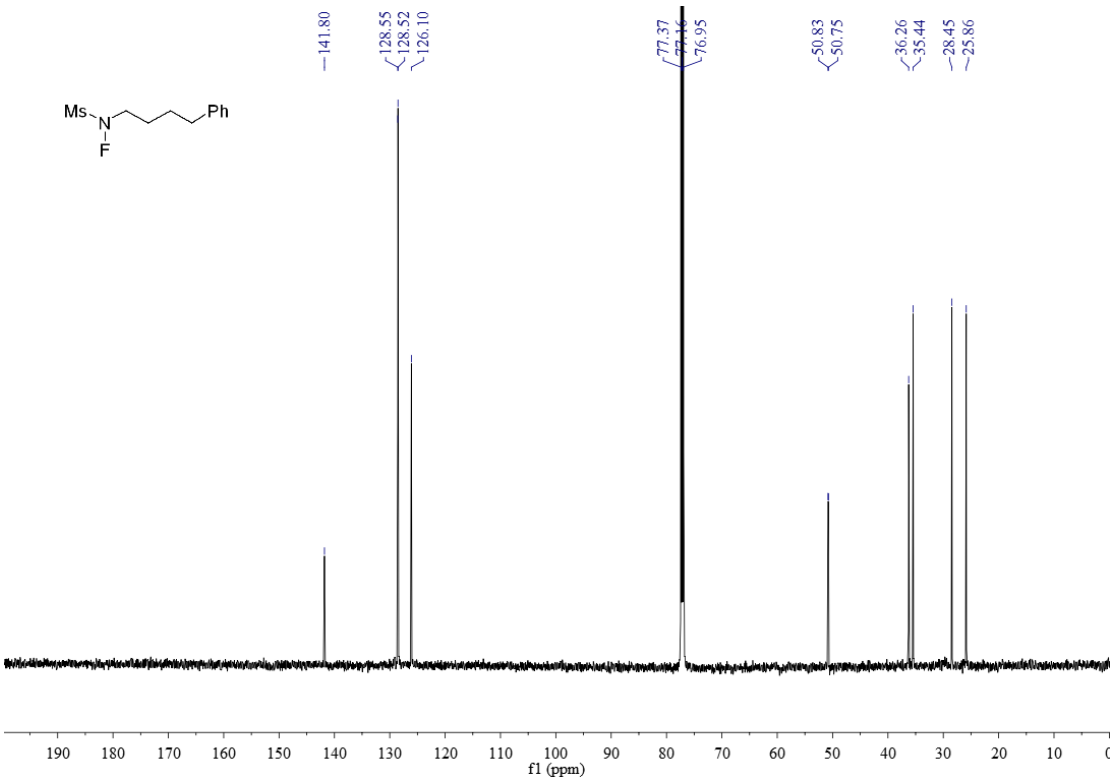
^{19}F NMR of 1j: 564 MHz, $CDCl_3$



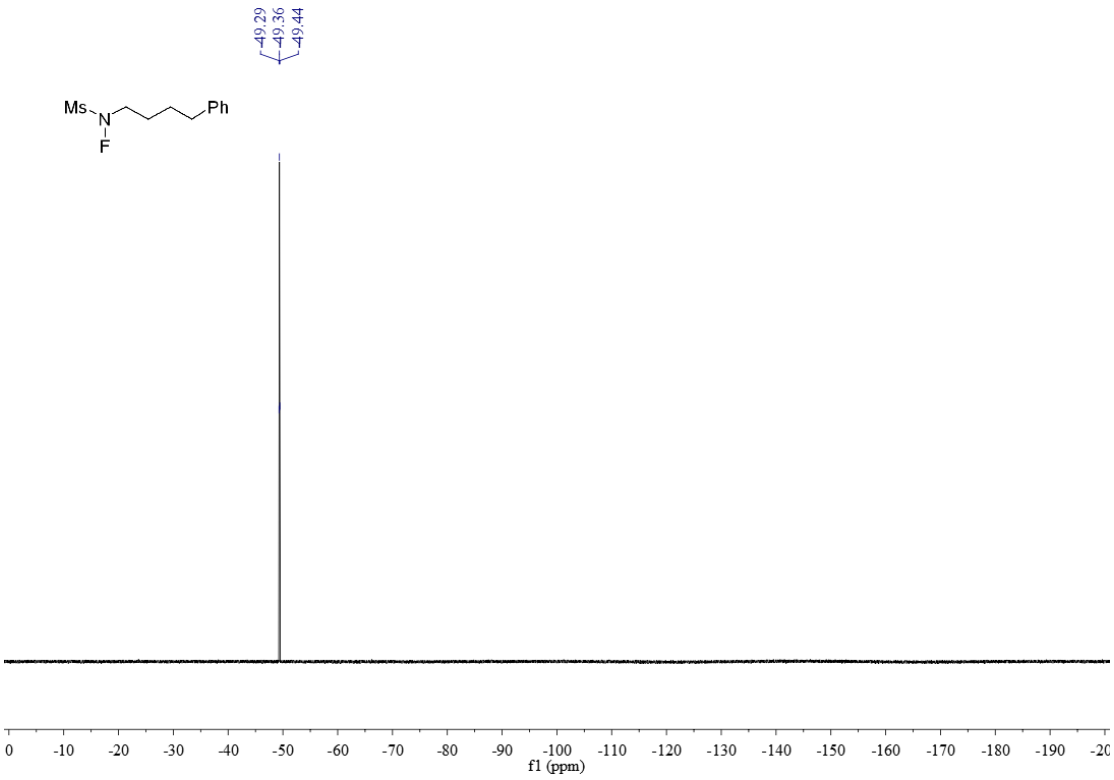
^{1}H NMR of 1k: 600 MHz, $CDCl_3$



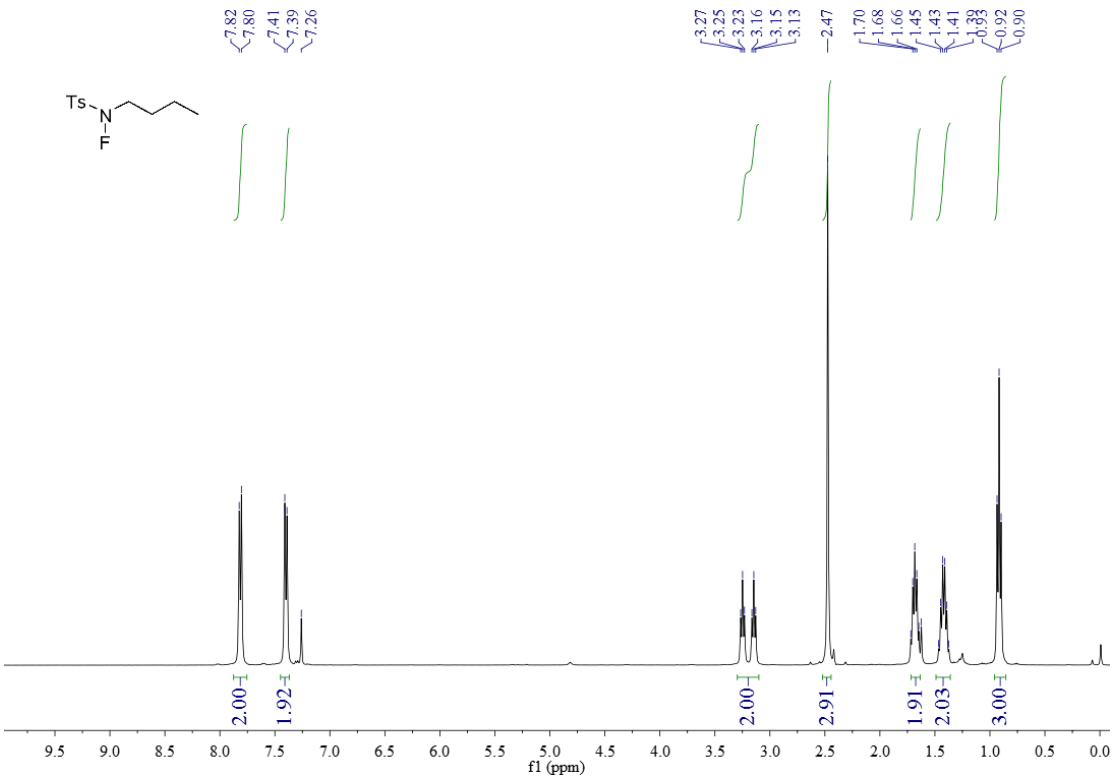
^{13}C NMR of 1k: 150 MHz, $CDCl_3$



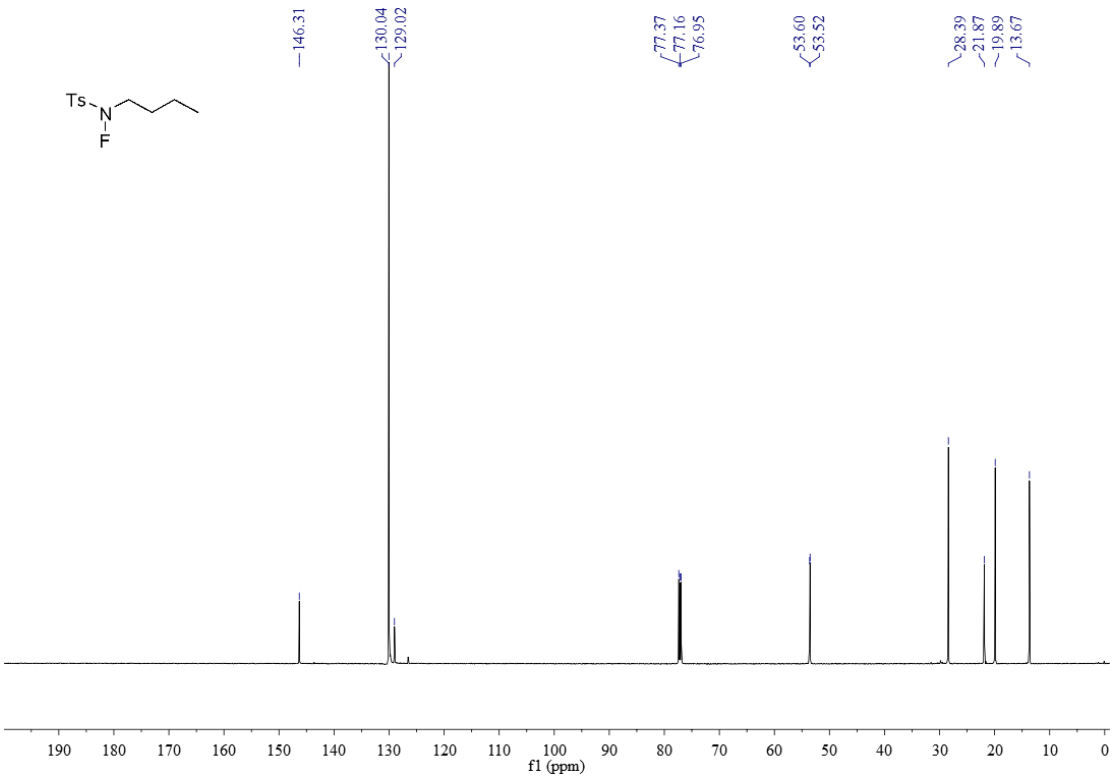
^{19}F NMR of 1k: 564 MHz, $CDCl_3$



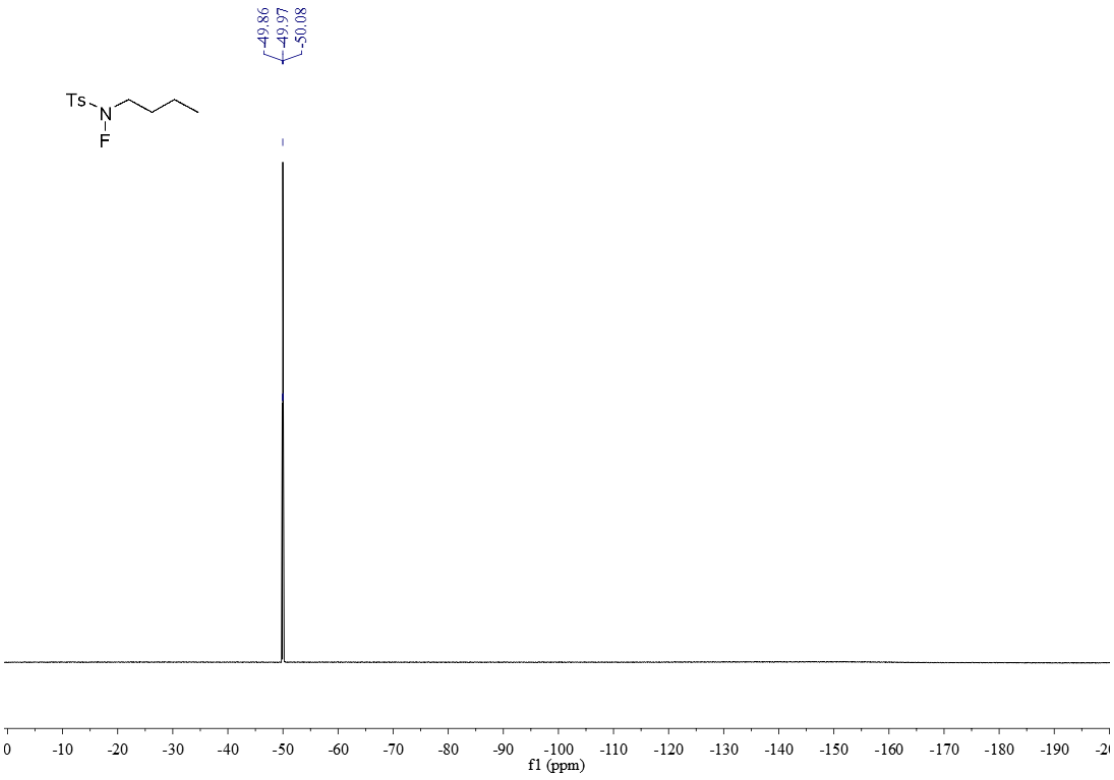
^{1}H NMR of 1l: 400 MHz, $CDCl_3$



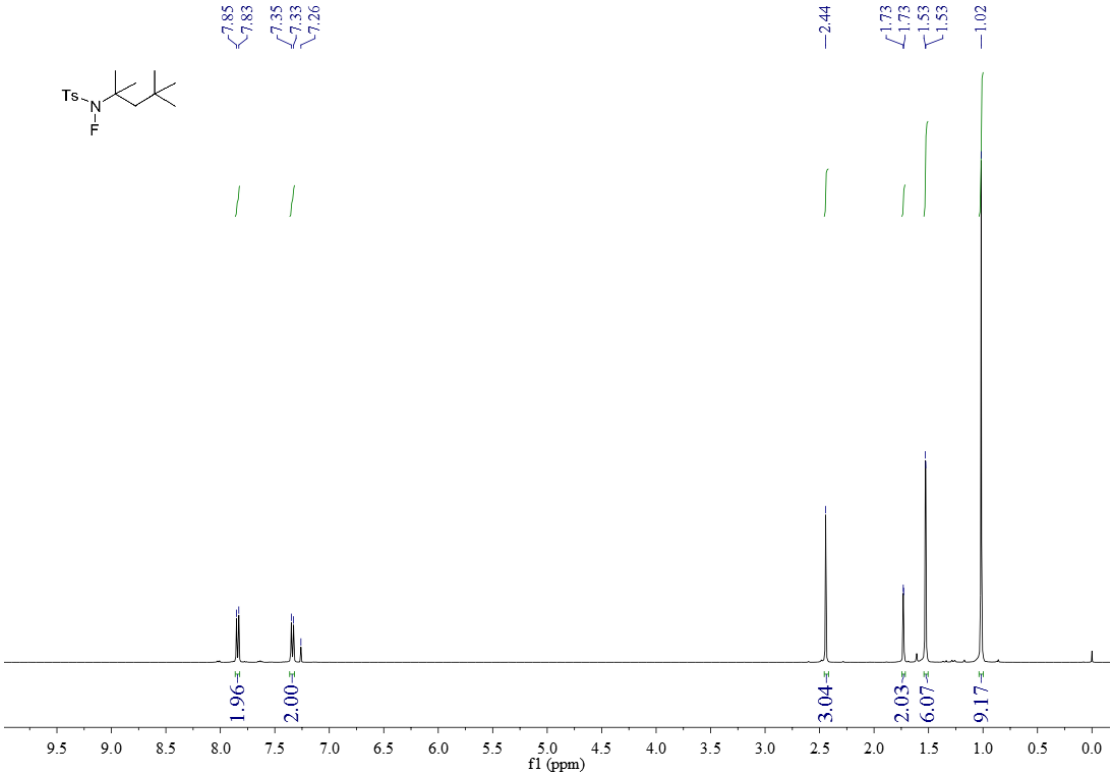
^{13}C NMR of 1l: 150 MHz, $CDCl_3$



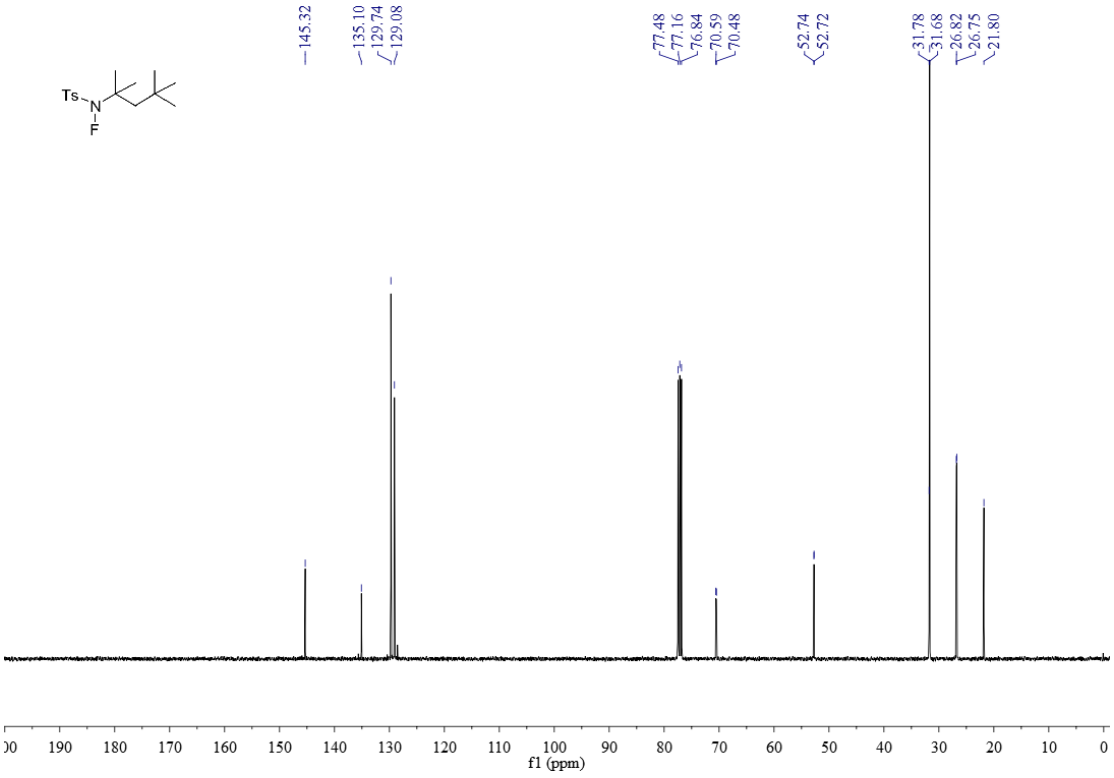
^{19}F NMR of 1l: 377 MHz, $CDCl_3$



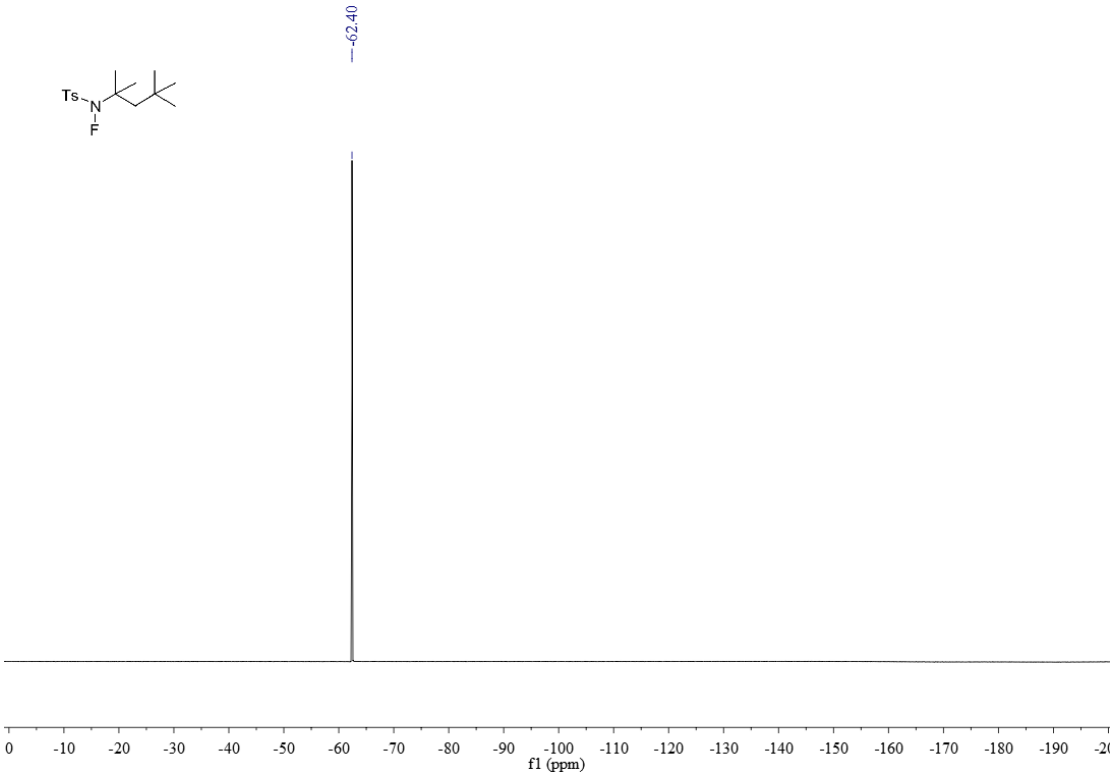
^{1}H NMR of 1m: 400 MHz, $CDCl_3$



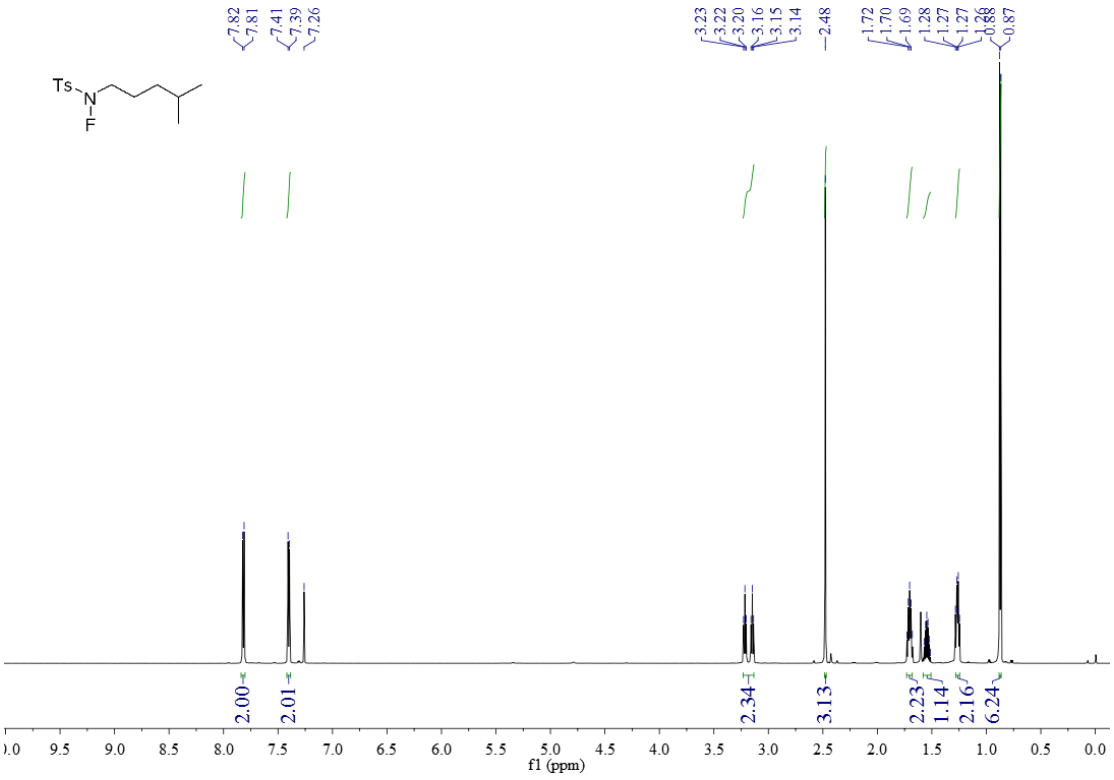
^{13}C NMR of 1m: 100 MHz, $CDCl_3$



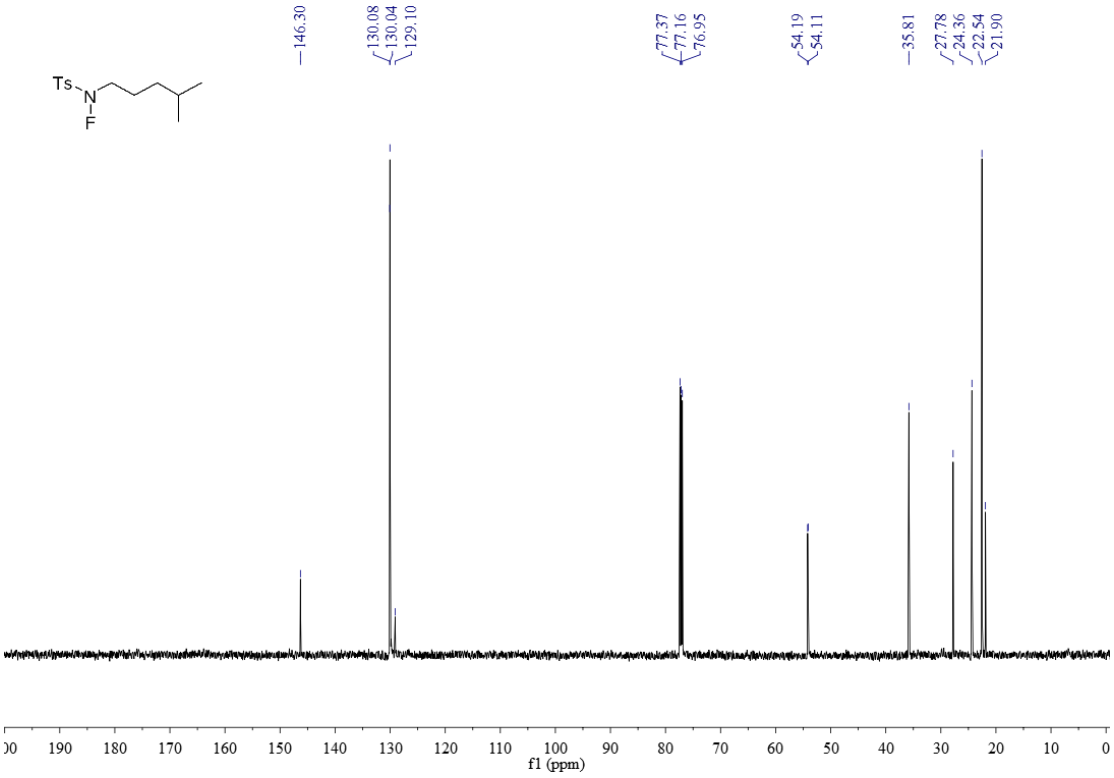
^{19}F NMR of 1m: 377 MHz, $CDCl_3$



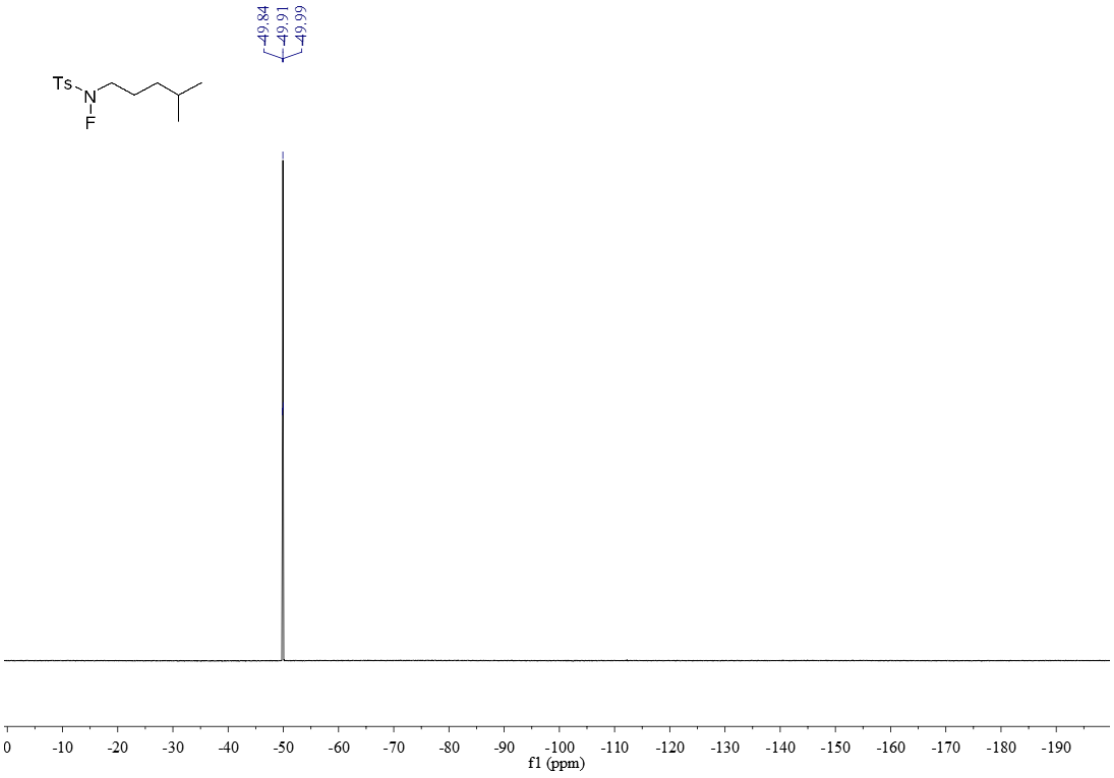
^{1}H NMR of 1n: 600 MHz, $CDCl_3$



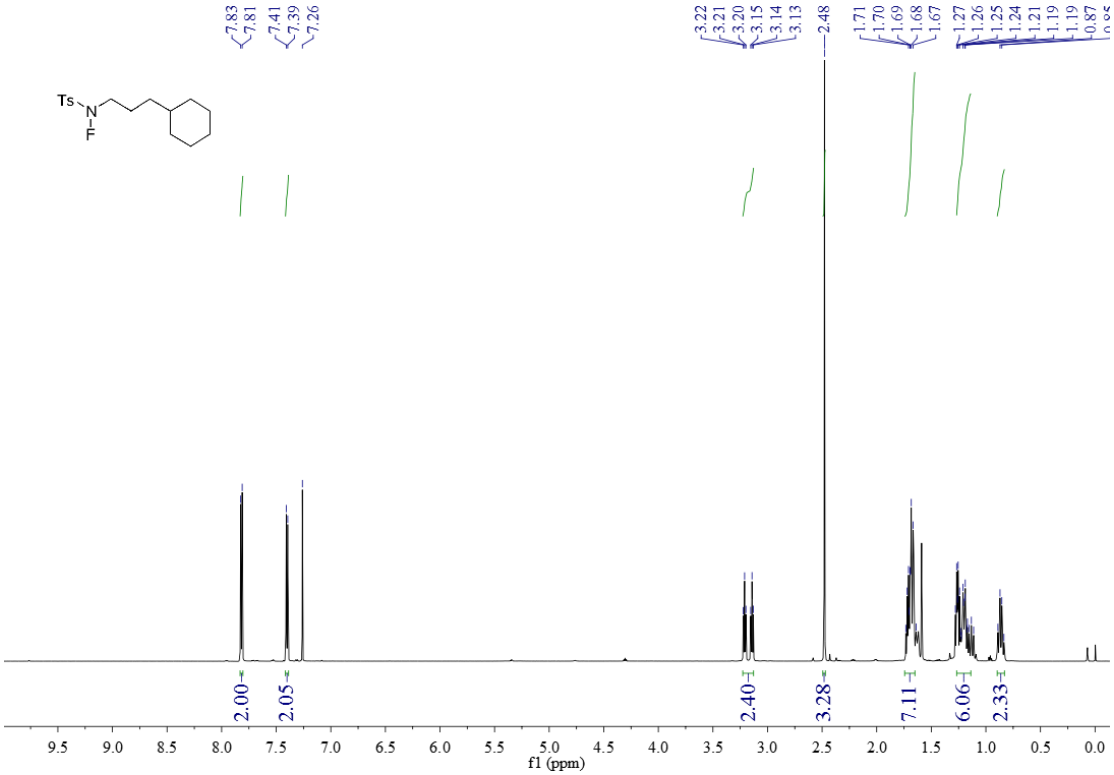
^{13}C NMR of 1n: 150 MHz, $CDCl_3$



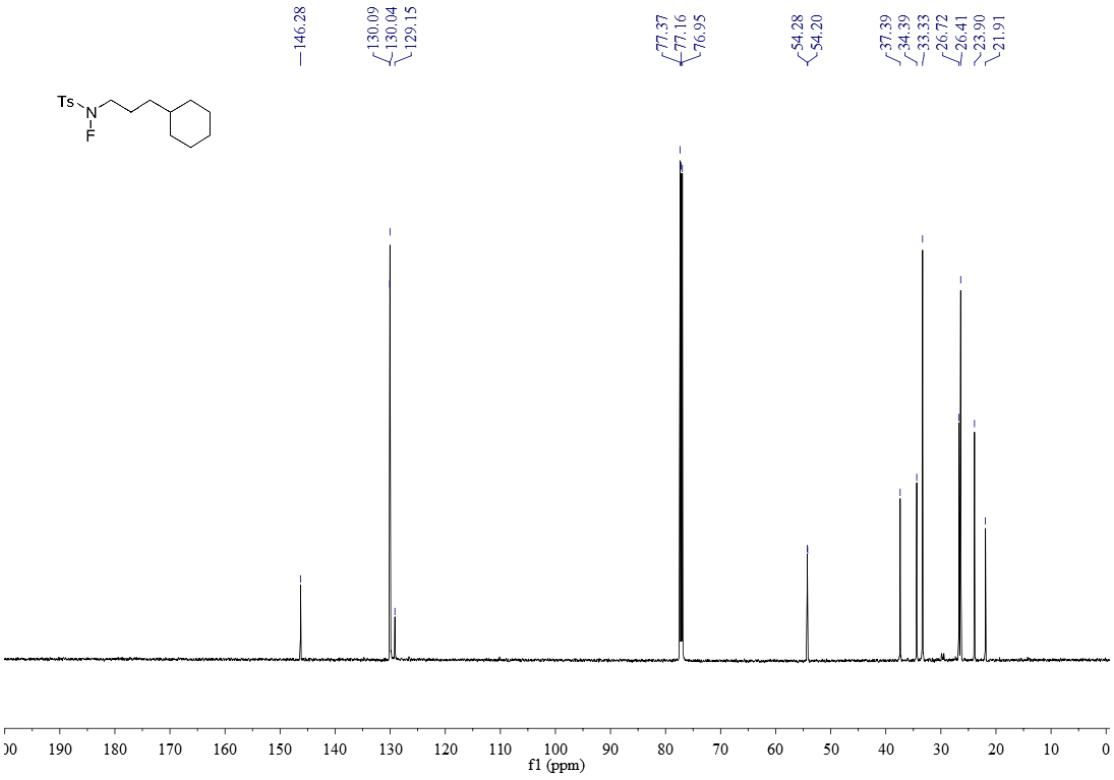
^{19}F NMR of 1n: 564 MHz, $CDCl_3$



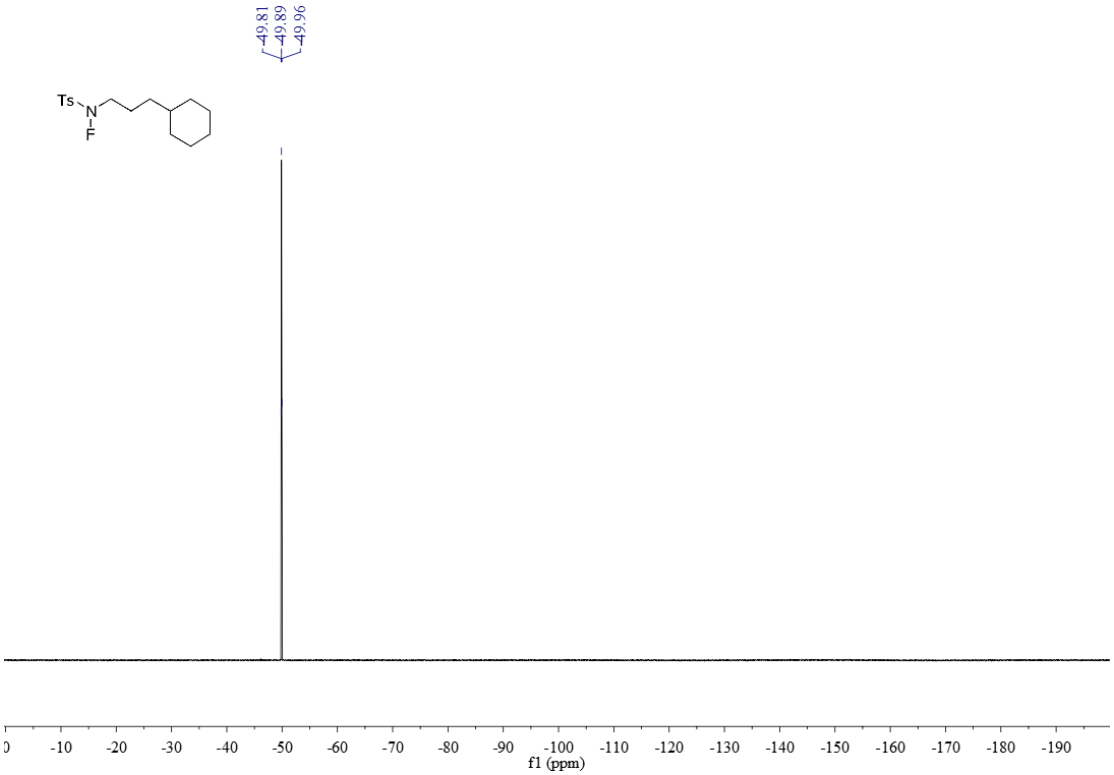
^{1}H NMR of 1o: 600 MHz, $CDCl_3$



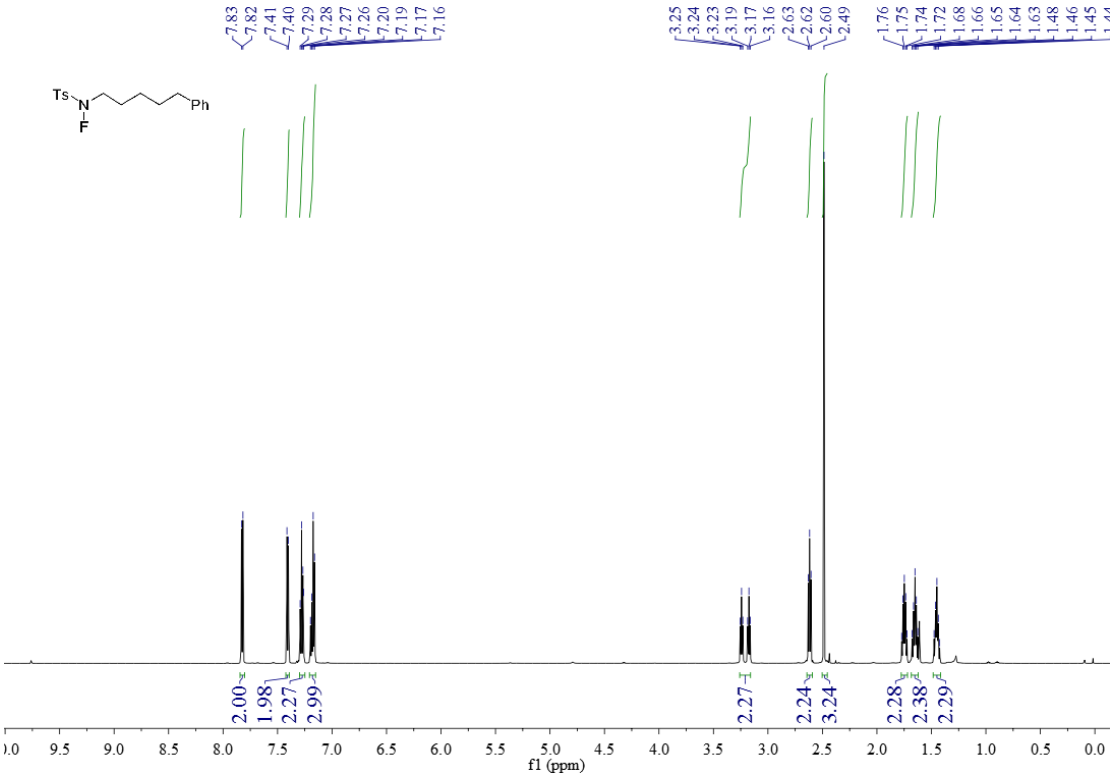
^{13}C NMR of 1o: 150 MHz, $CDCl_3$



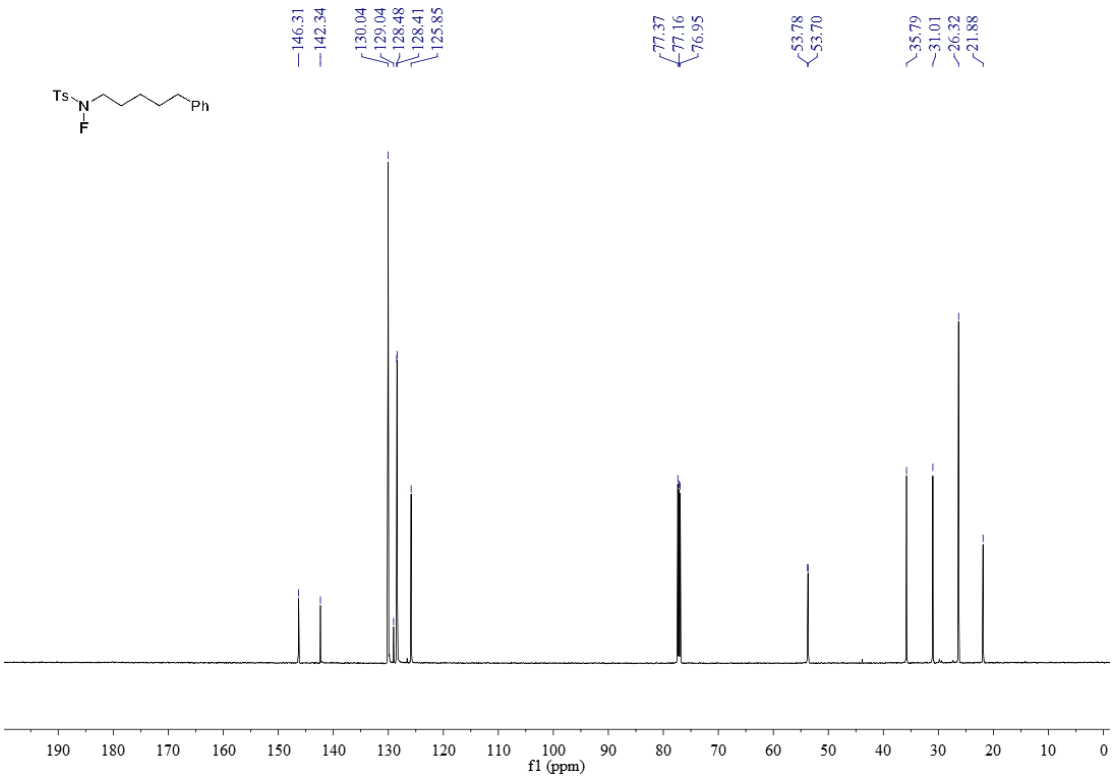
^{19}F NMR of 1o: 564 MHz, $CDCl_3$



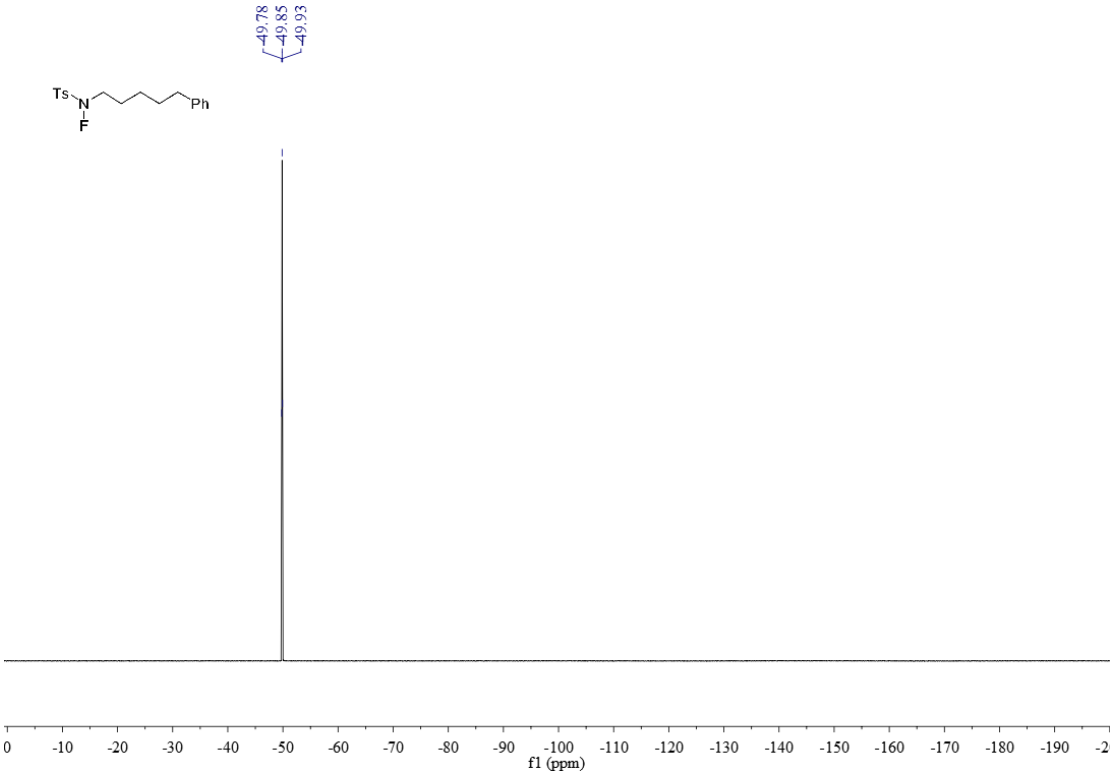
^{1}H NMR of 1p: 600 MHz, $CDCl_3$



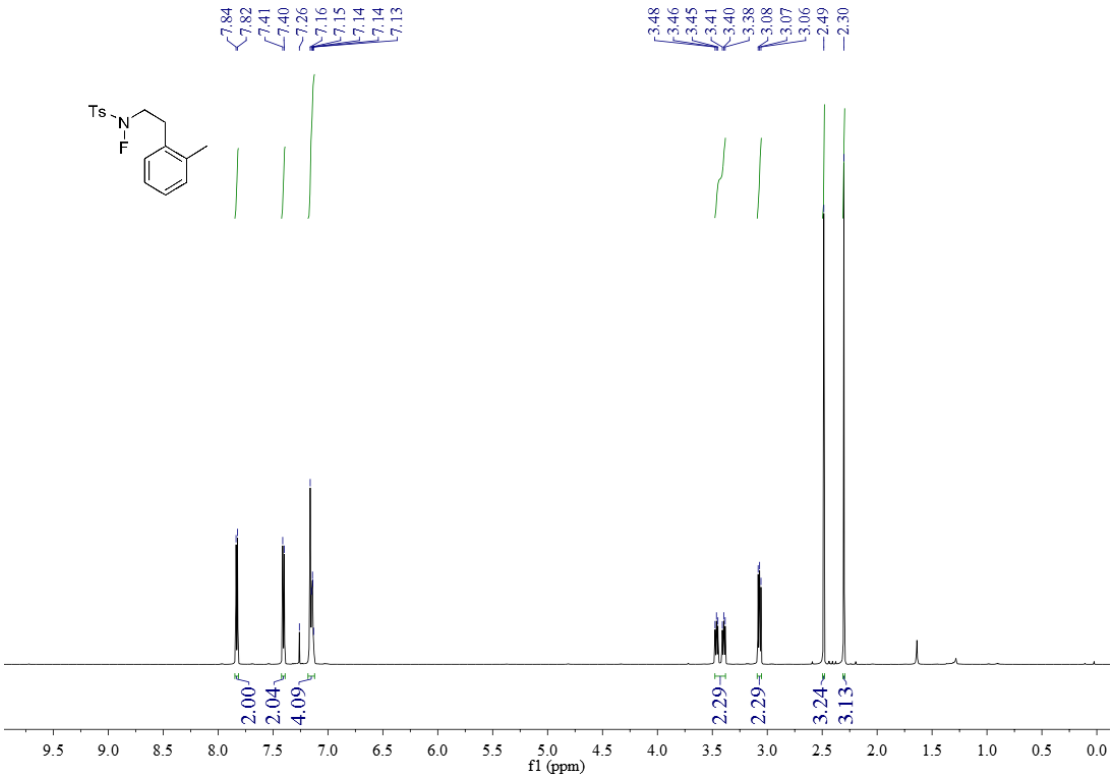
^{13}C NMR of 1p: 150 MHz, $CDCl_3$



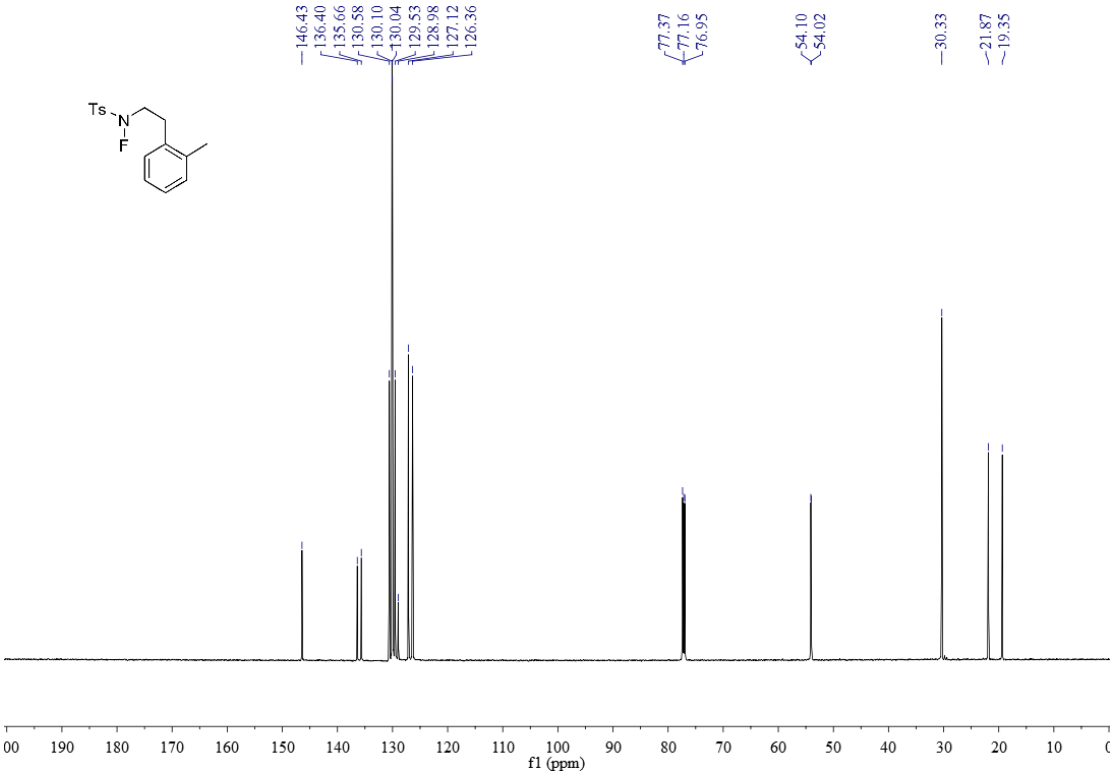
^{19}F NMR of 1p: 564 MHz, $CDCl_3$



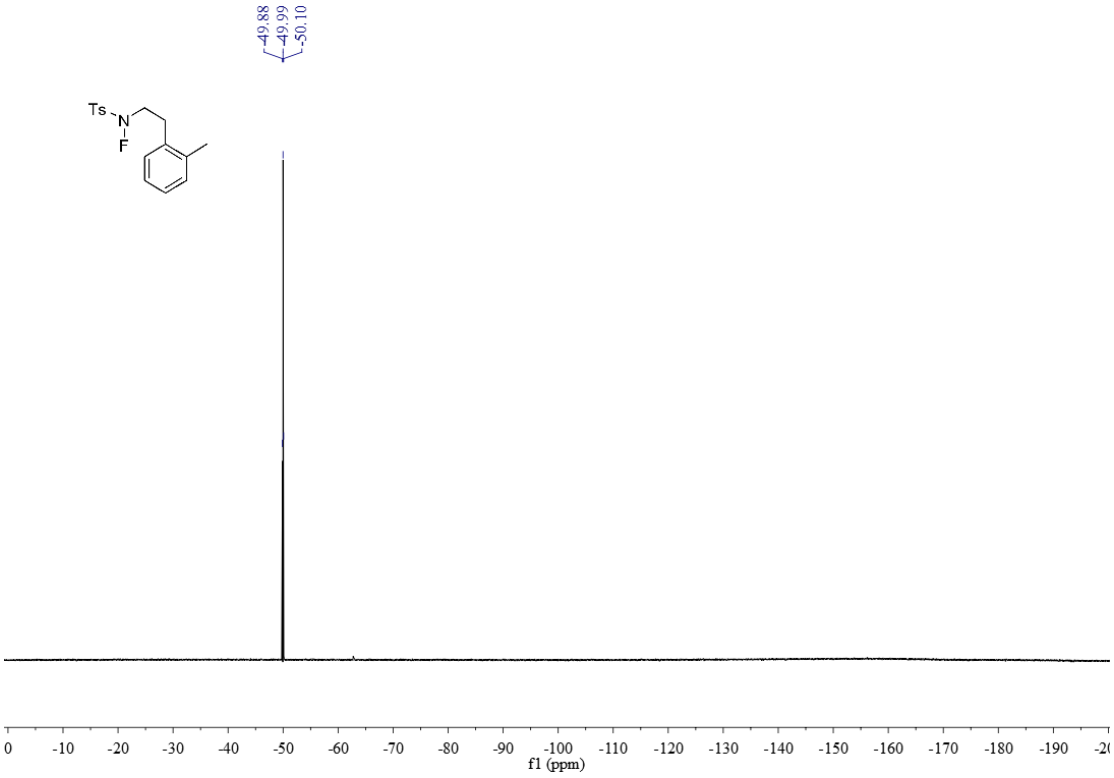
^{1}H NMR of 1q: 600 MHz, $CDCl_3$



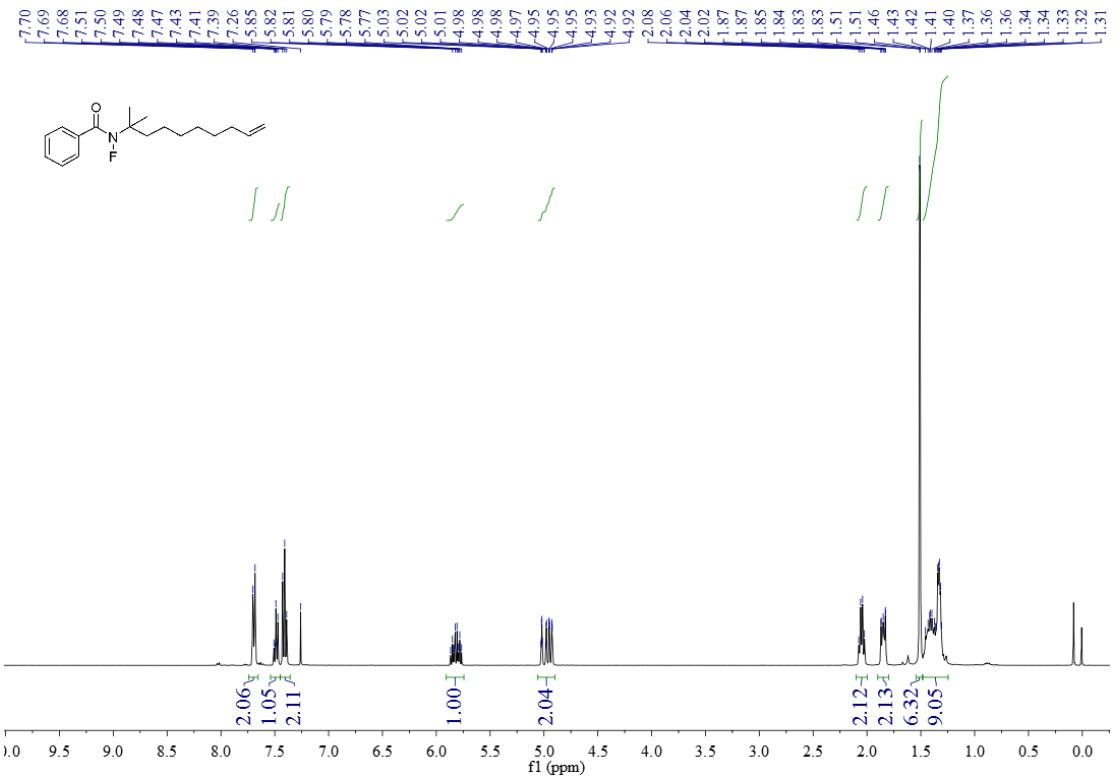
^{13}C NMR of 1q: 150 MHz, $CDCl_3$



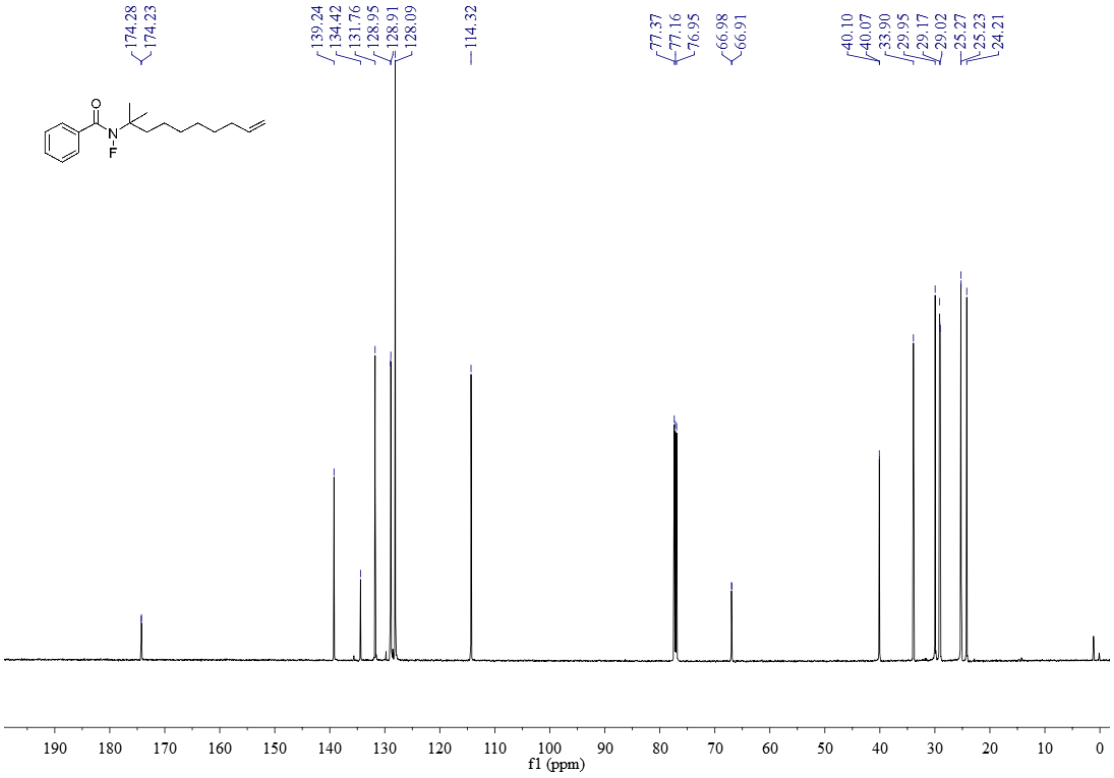
^{19}F NMR of 1q: 377 MHz, $CDCl_3$



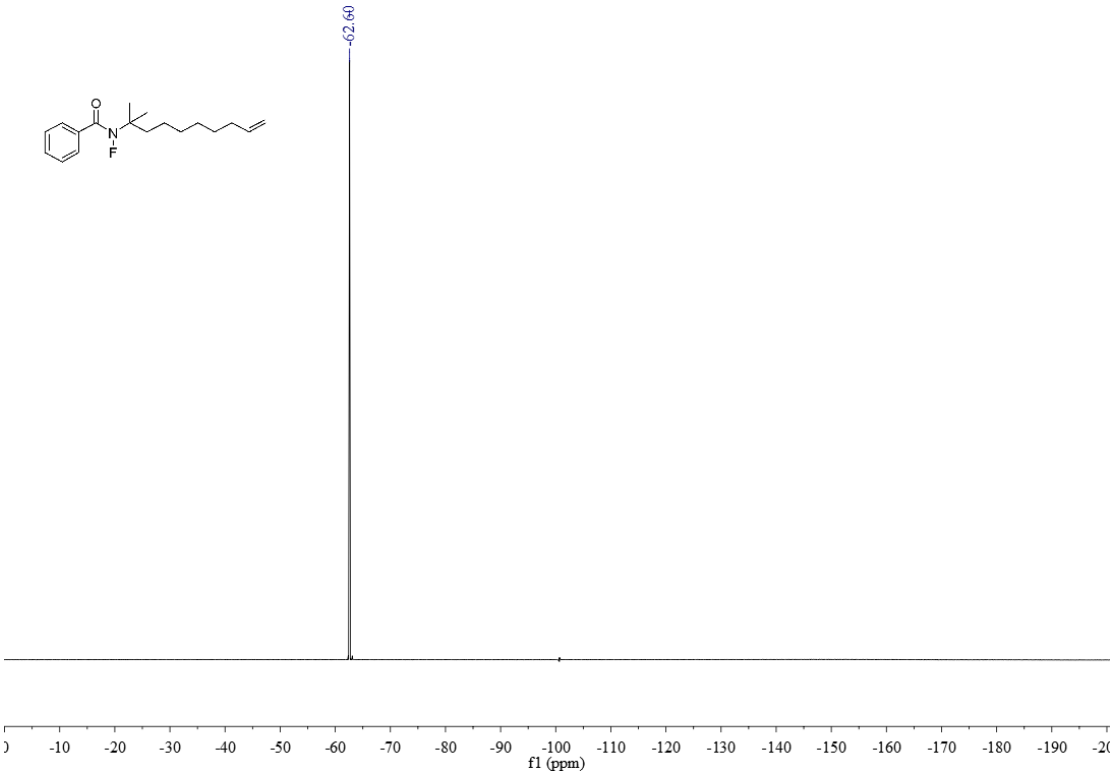
^{1}H NMR of 1r: 400 MHz, $CDCl_3$



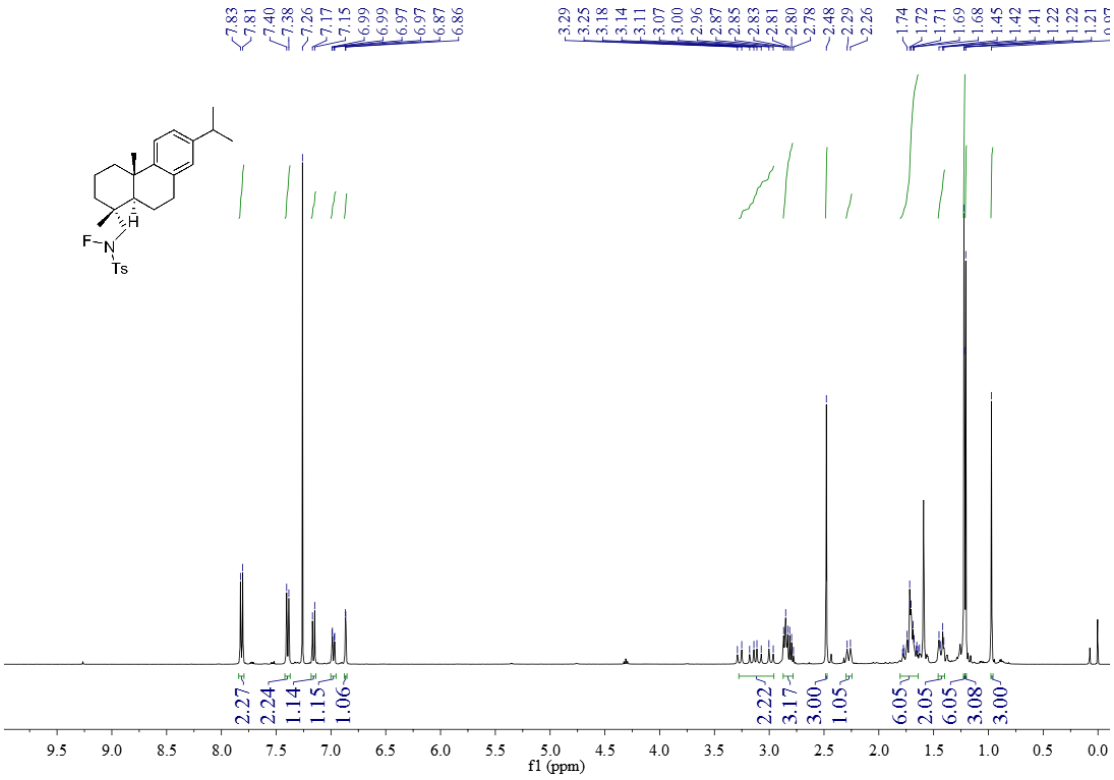
^{13}C NMR of 1r: 150 MHz, $CDCl_3$



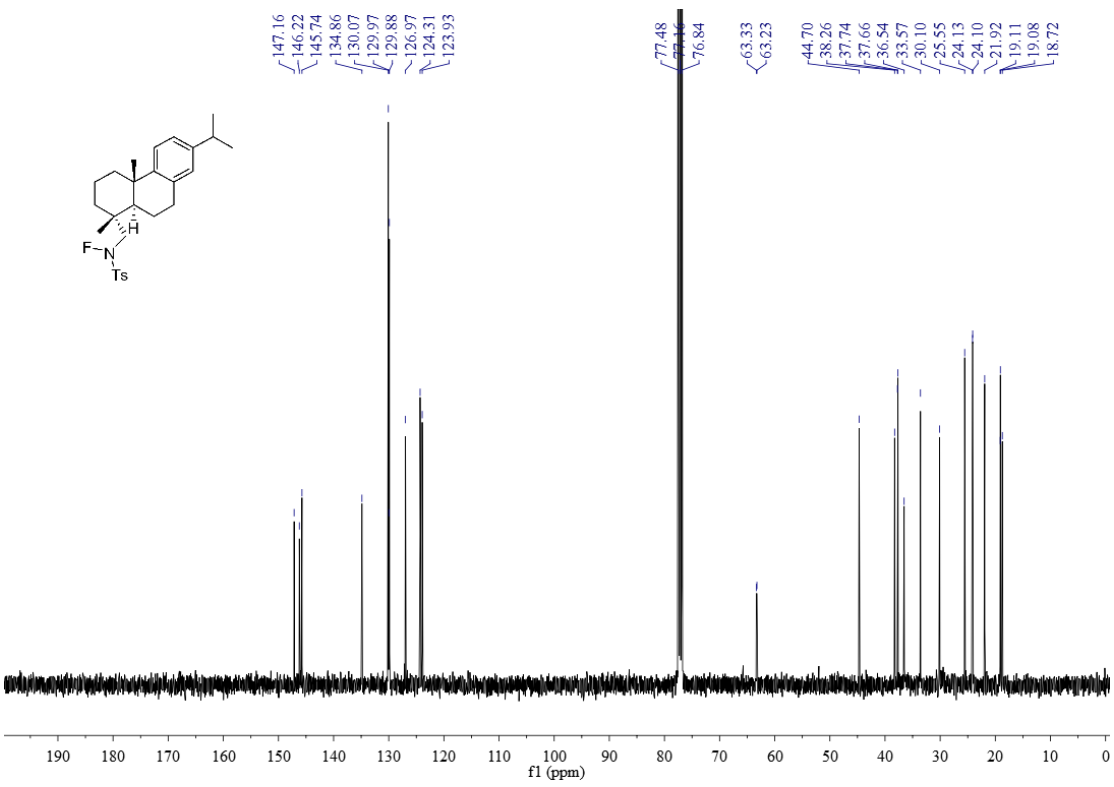
^{19}F NMR of 1r: 377 MHz, $CDCl_3$



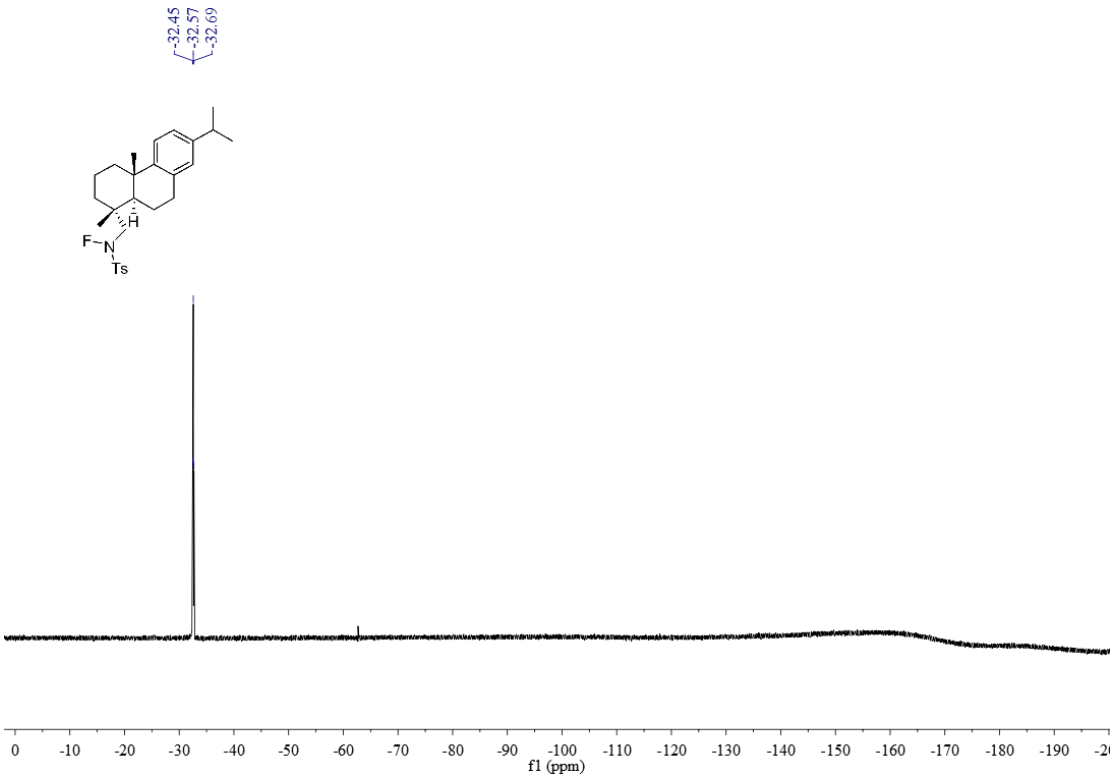
^{1}H NMR of 1s: 400 MHz, $CDCl_3$



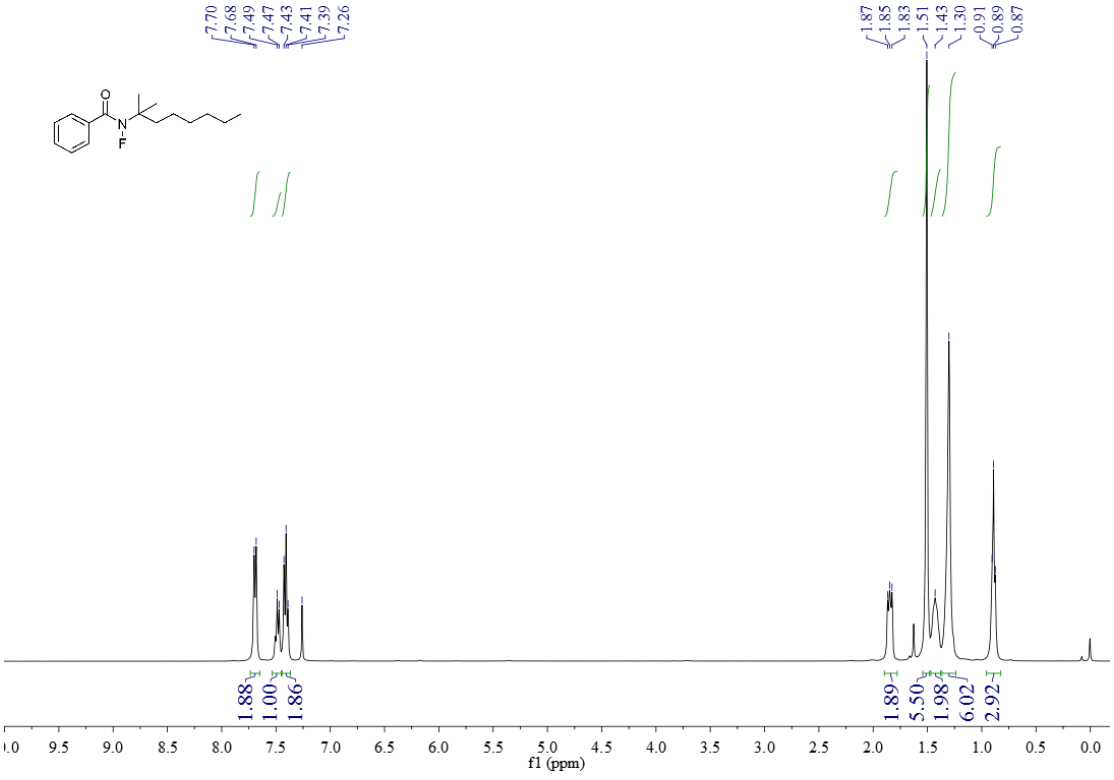
^{13}C NMR of 1s: 100 MHz, $CDCl_3$



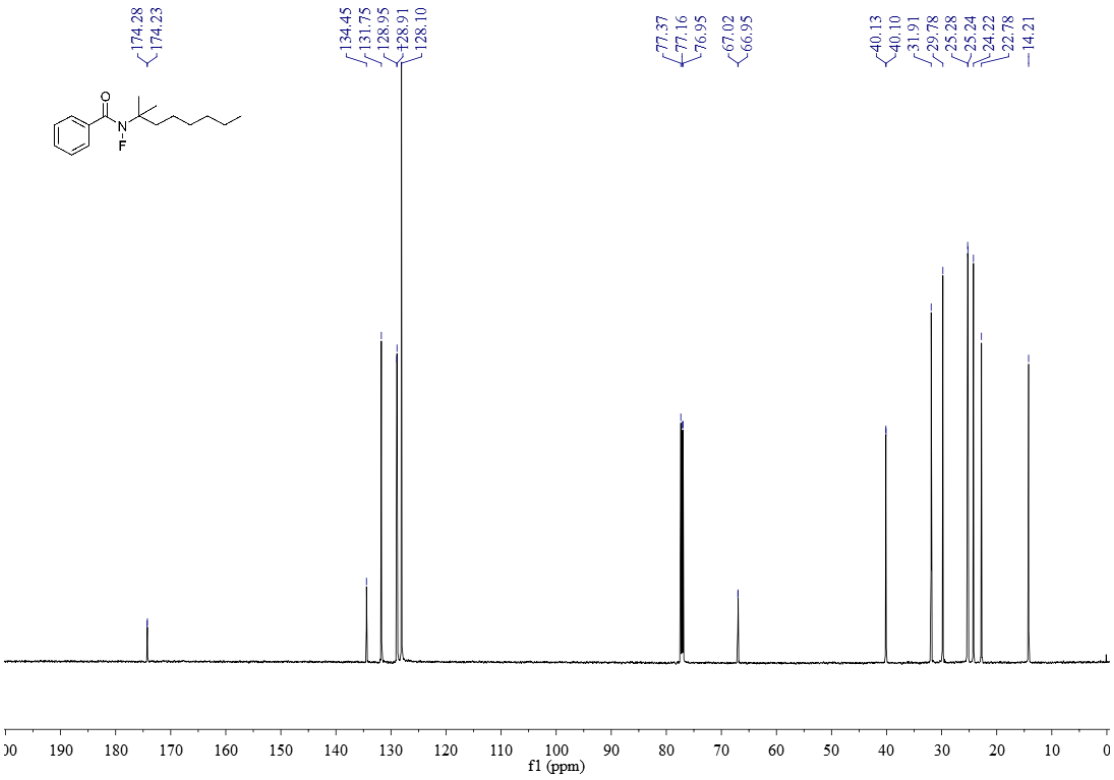
^{19}F NMR of 1s: 377 MHz, $CDCl_3$



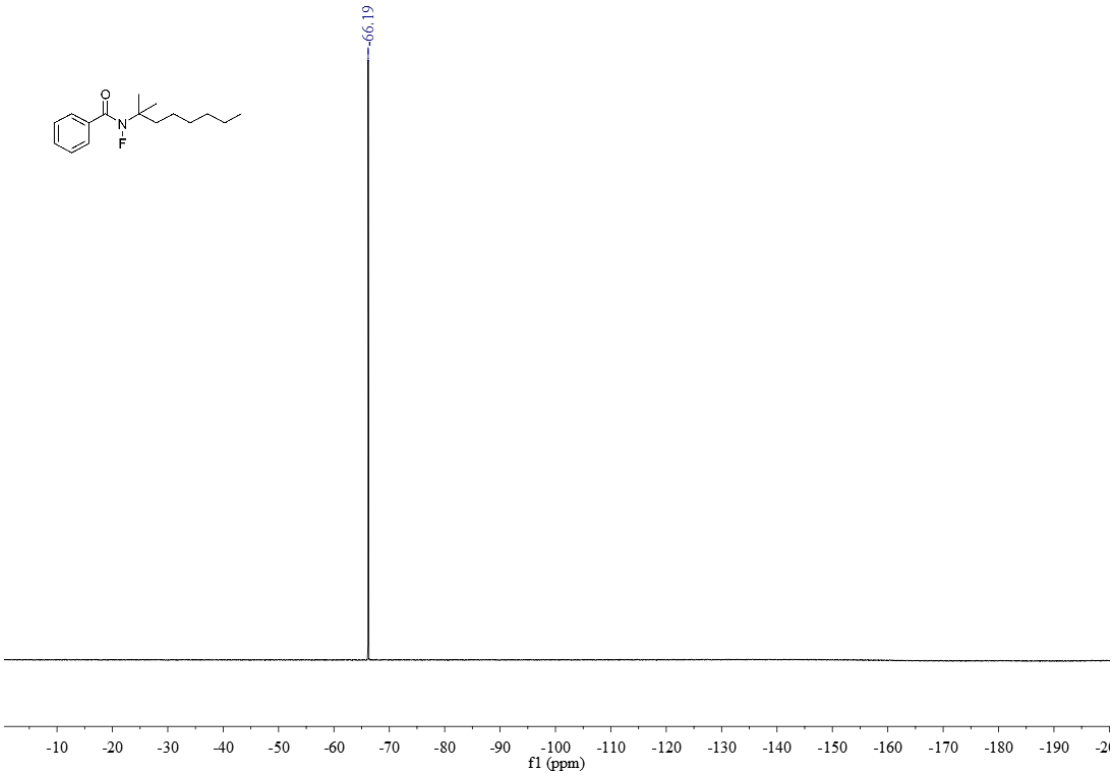
^{1}H NMR of 1aa: 400 MHz, $CDCl_3$



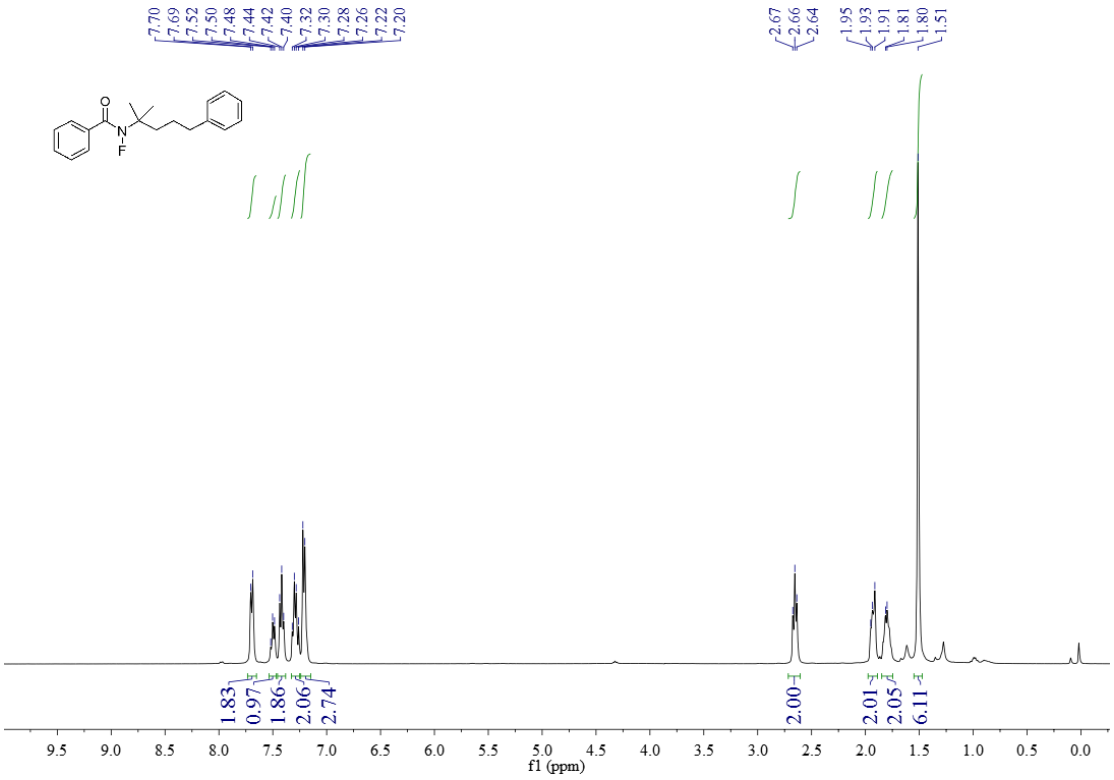
^{13}C NMR of 1aa: 150 MHz, $CDCl_3$



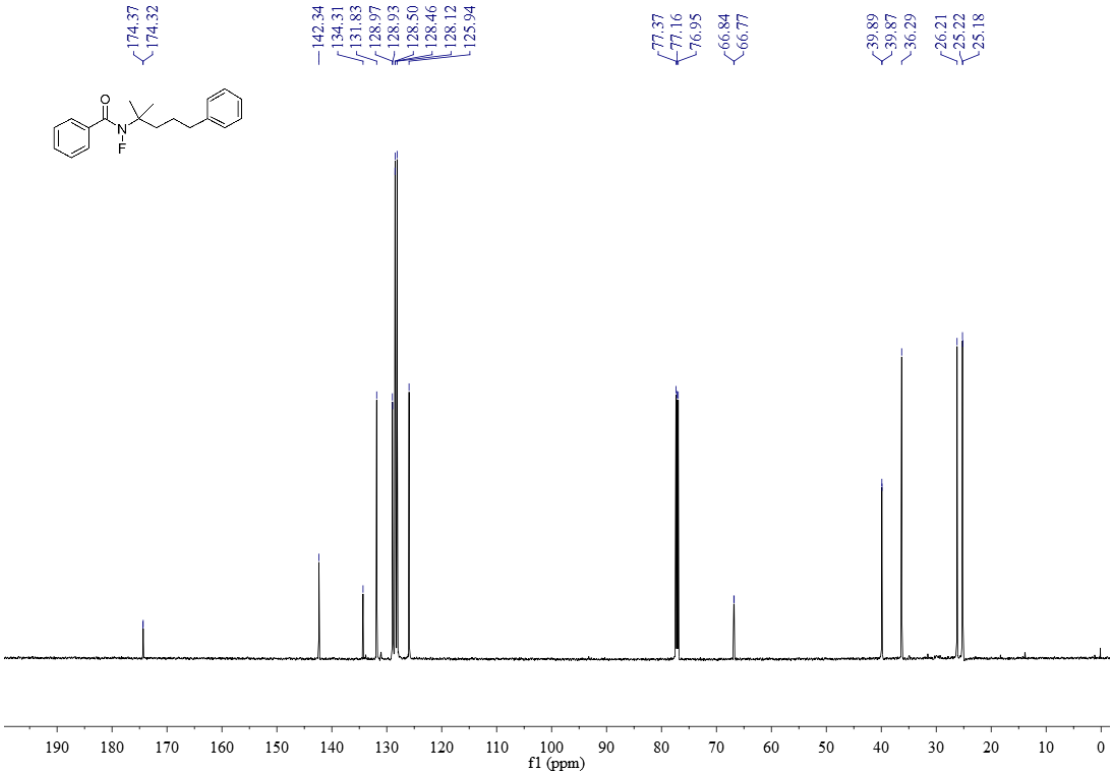
^{19}F NMR of 1aa: 377 MHz, $CDCl_3$



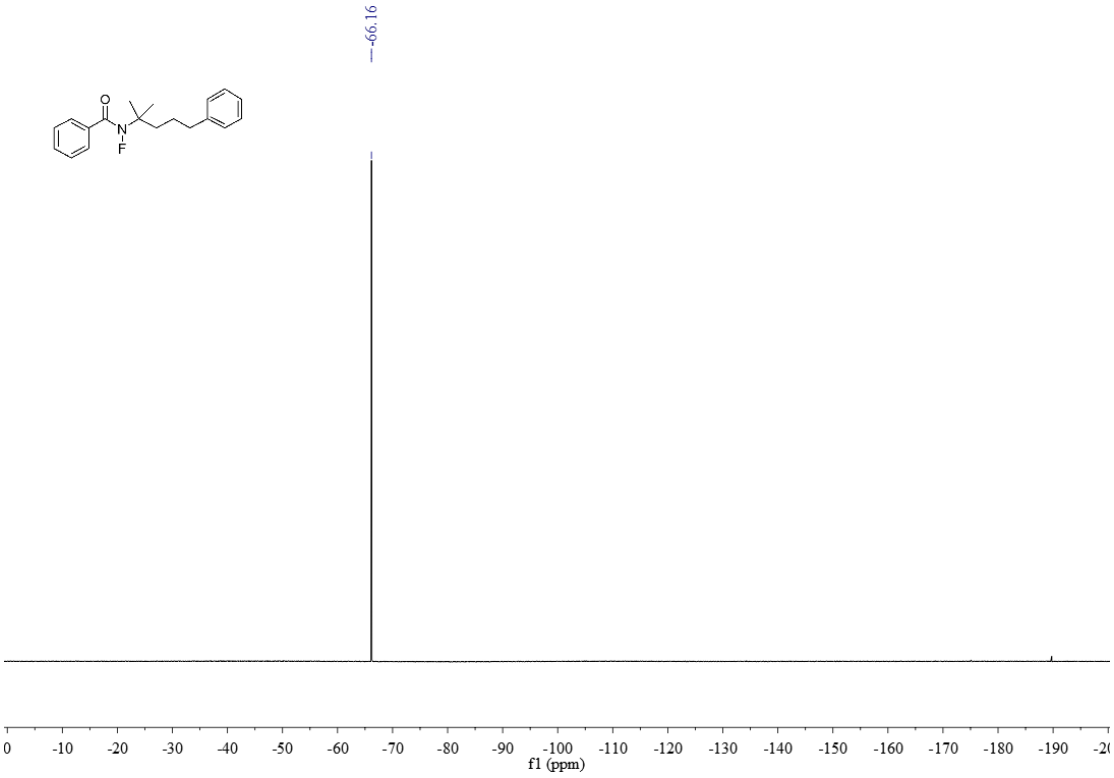
^{1}H NMR of 1ab: 400 MHz, $CDCl_3$



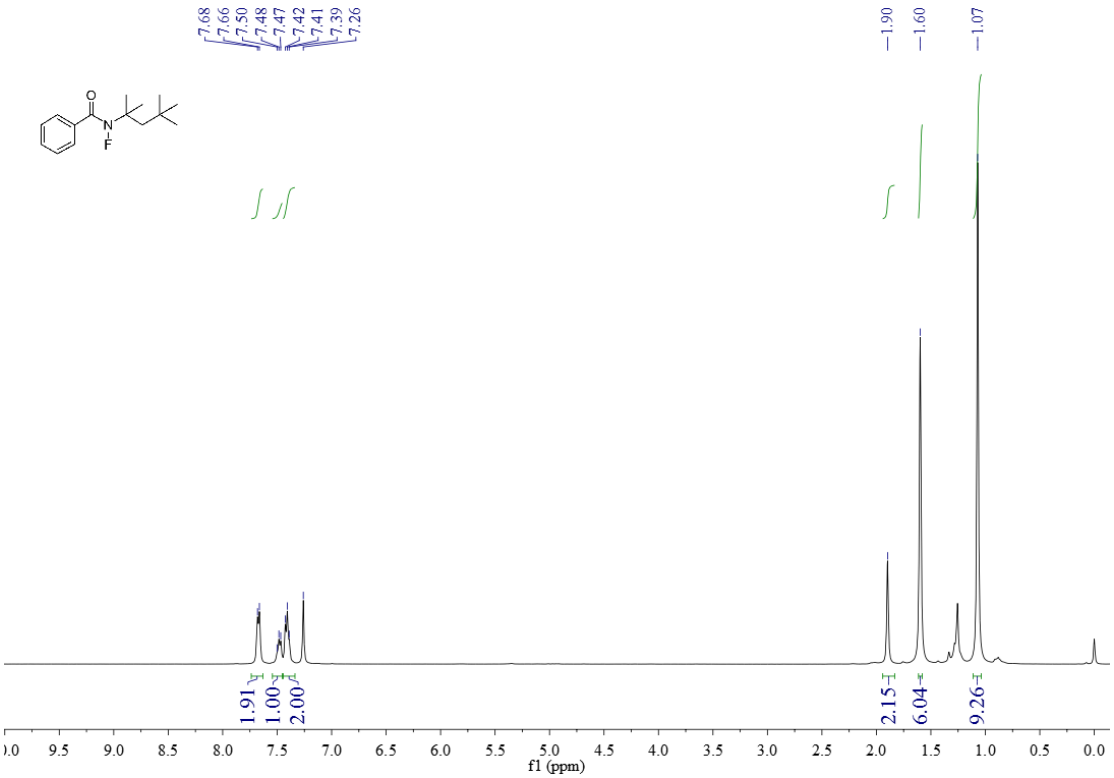
^{13}C NMR of 1ab: 150 MHz, $CDCl_3$



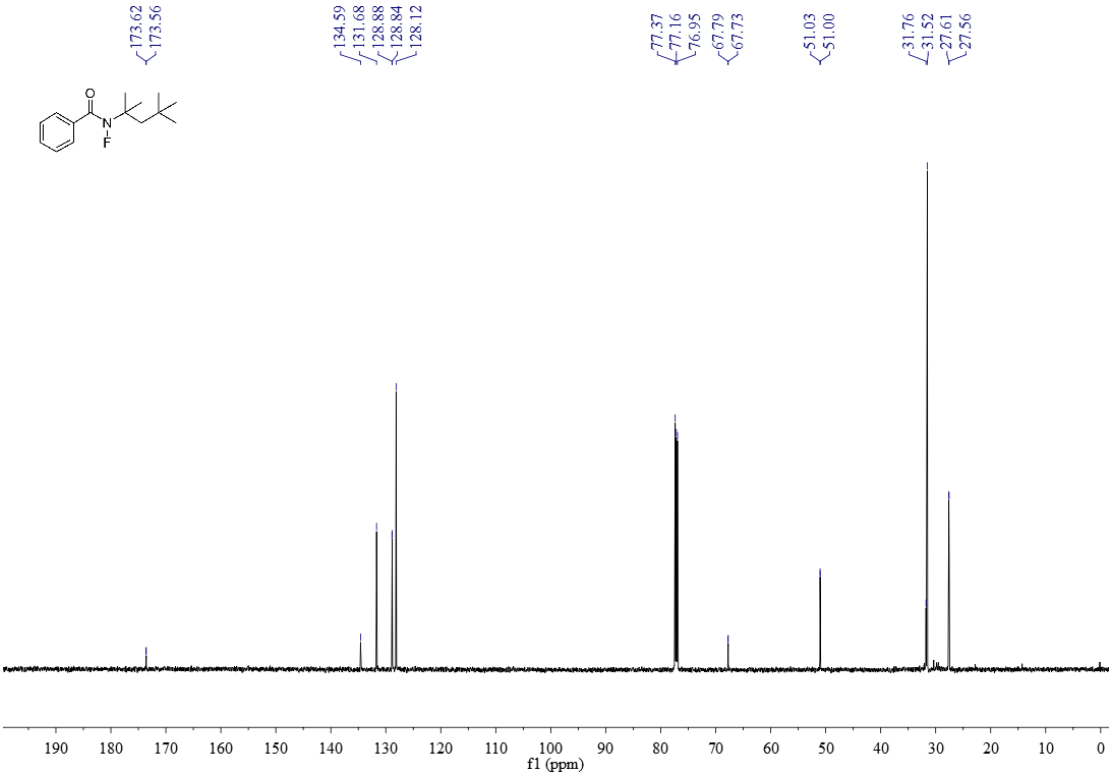
^{19}F NMR of 1ab: 564 MHz, $CDCl_3$



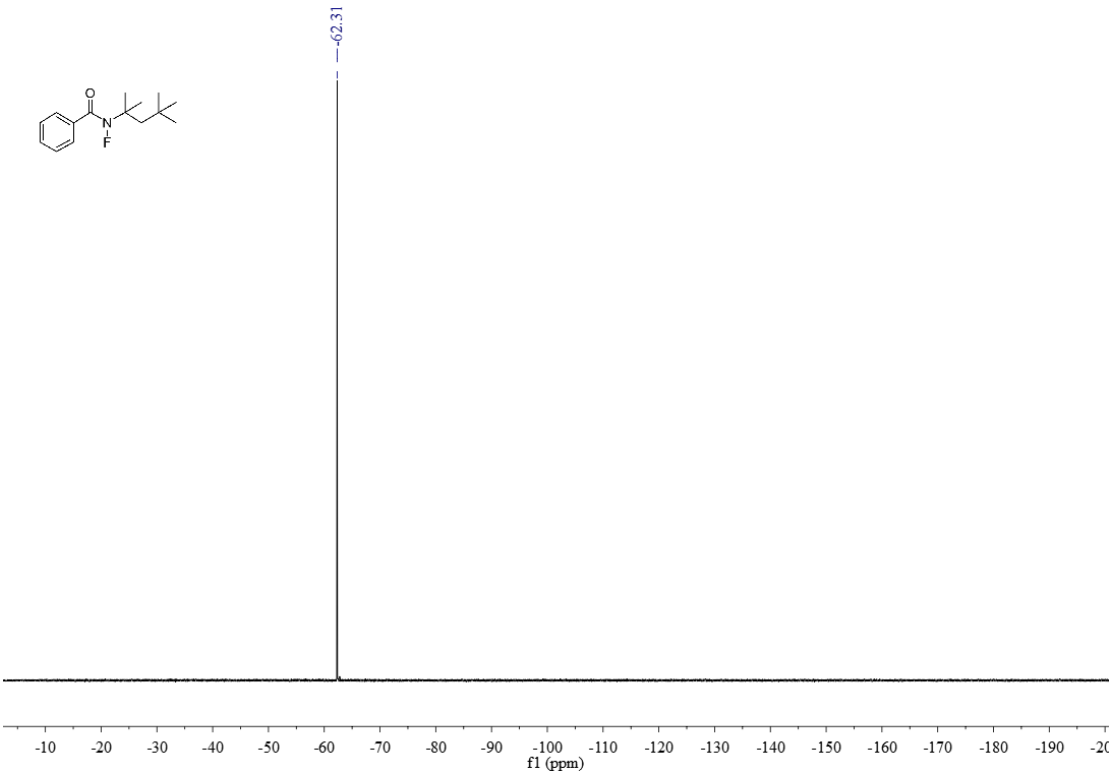
^{1}H NMR of 1ac: 400 MHz, $CDCl_3$



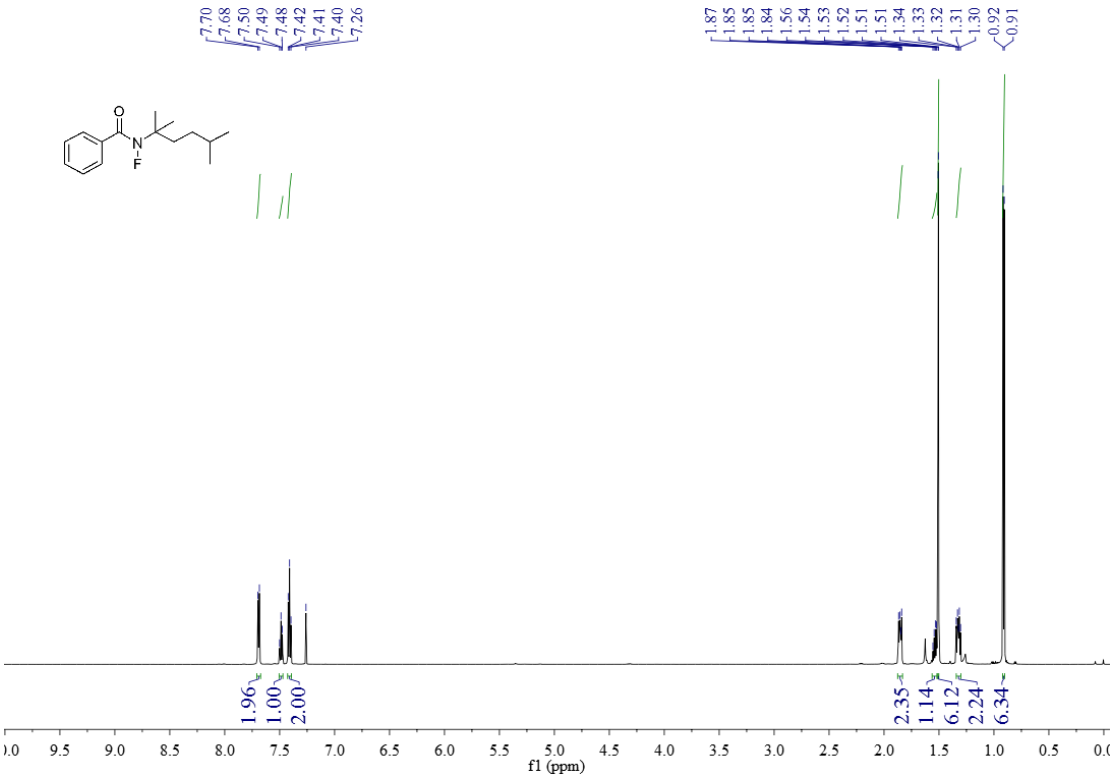
^{13}C NMR of 1ac: 150 MHz, $CDCl_3$



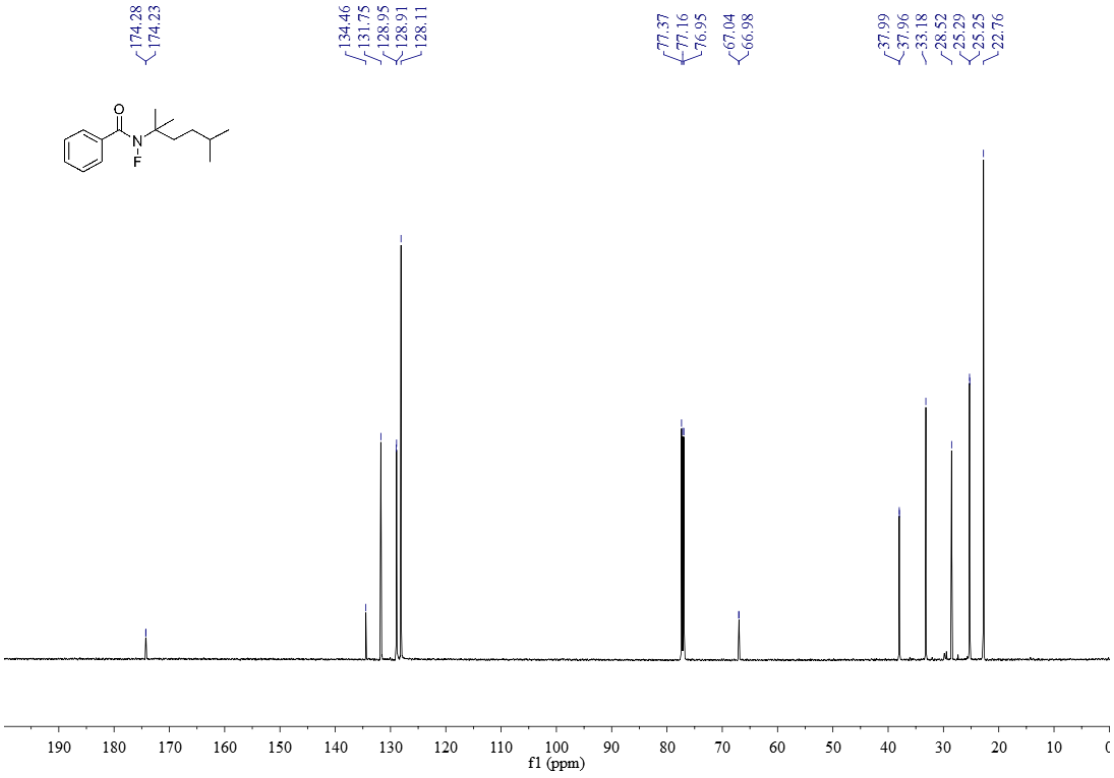
^{19}F NMR of 1ac: 564 MHz, $CDCl_3$



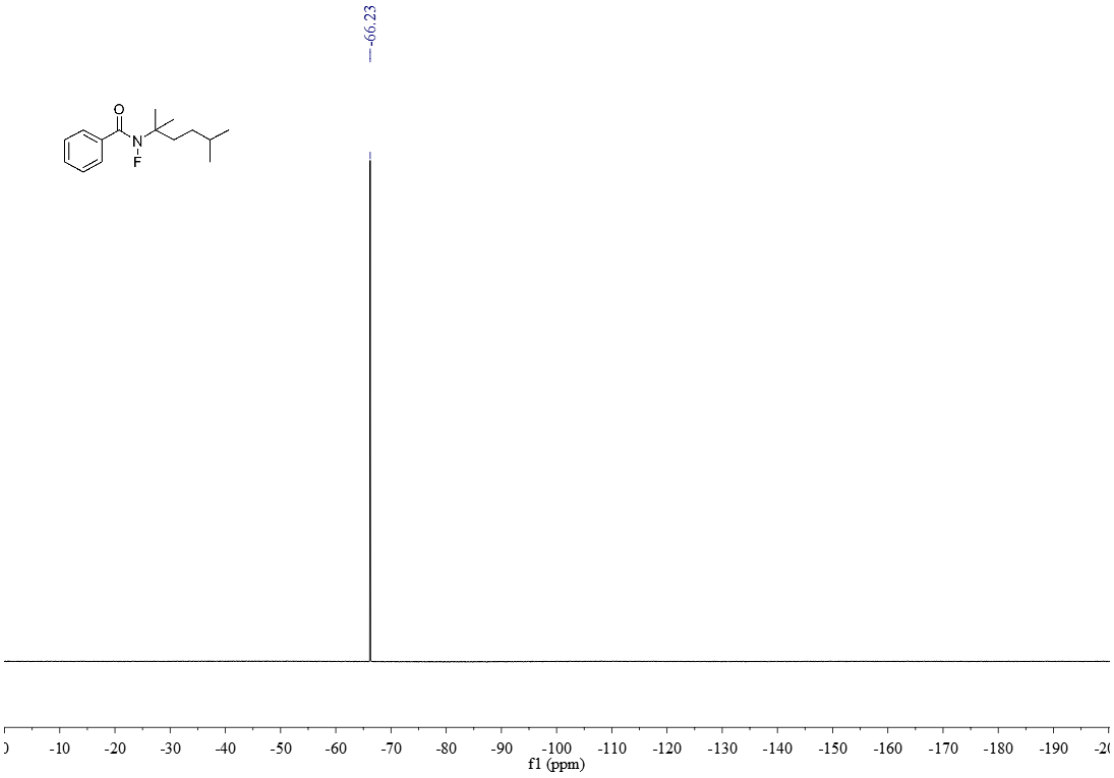
^{1}H NMR of 1ad: 600 MHz, $CDCl_3$



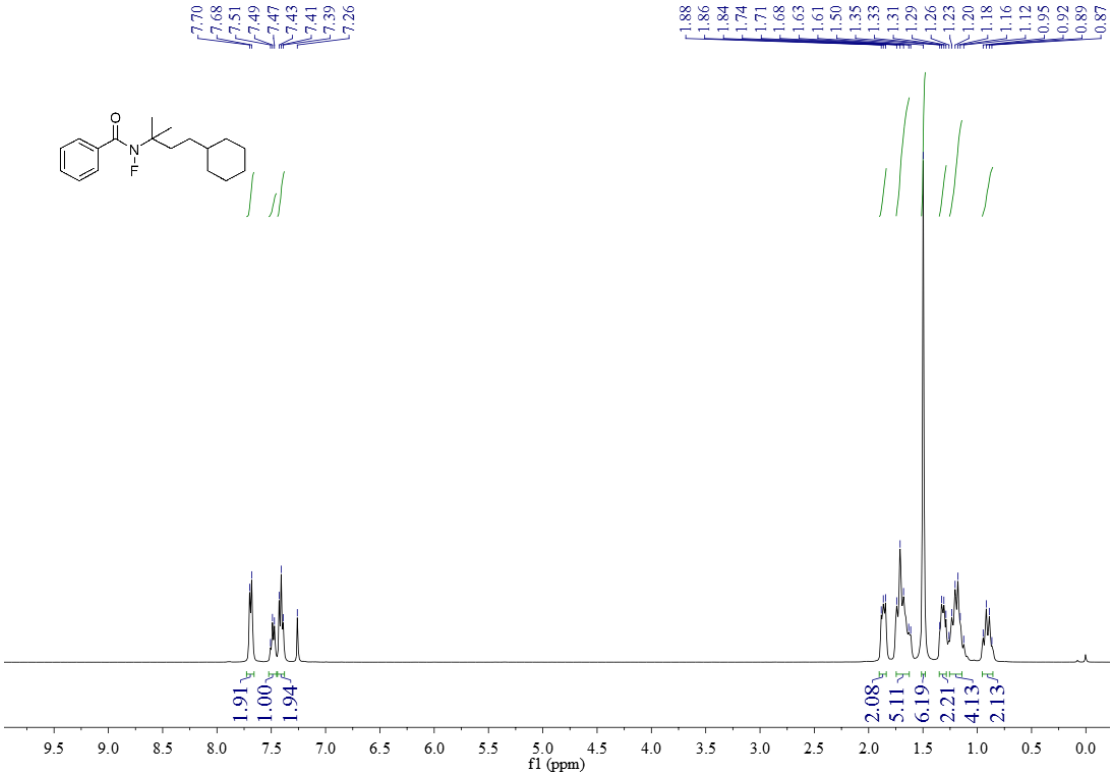
^{13}C NMR of 1ad: 150 MHz, $CDCl_3$



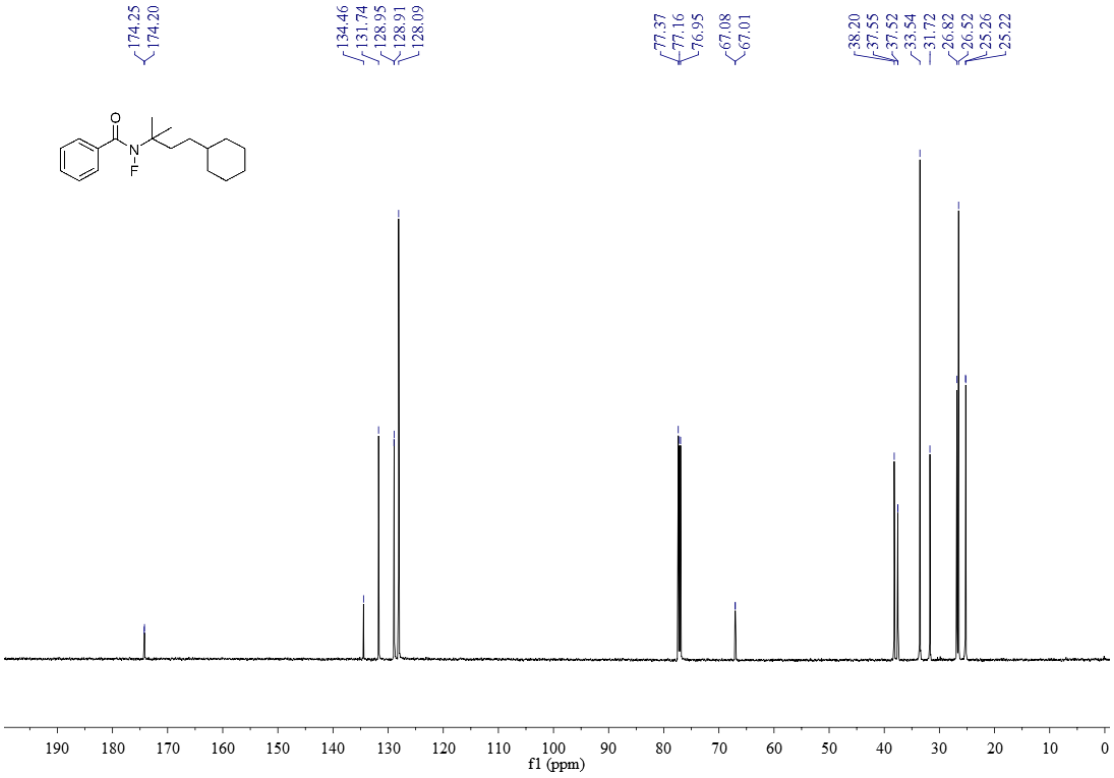
^{19}F NMR of 1ad: 564 MHz, $CDCl_3$



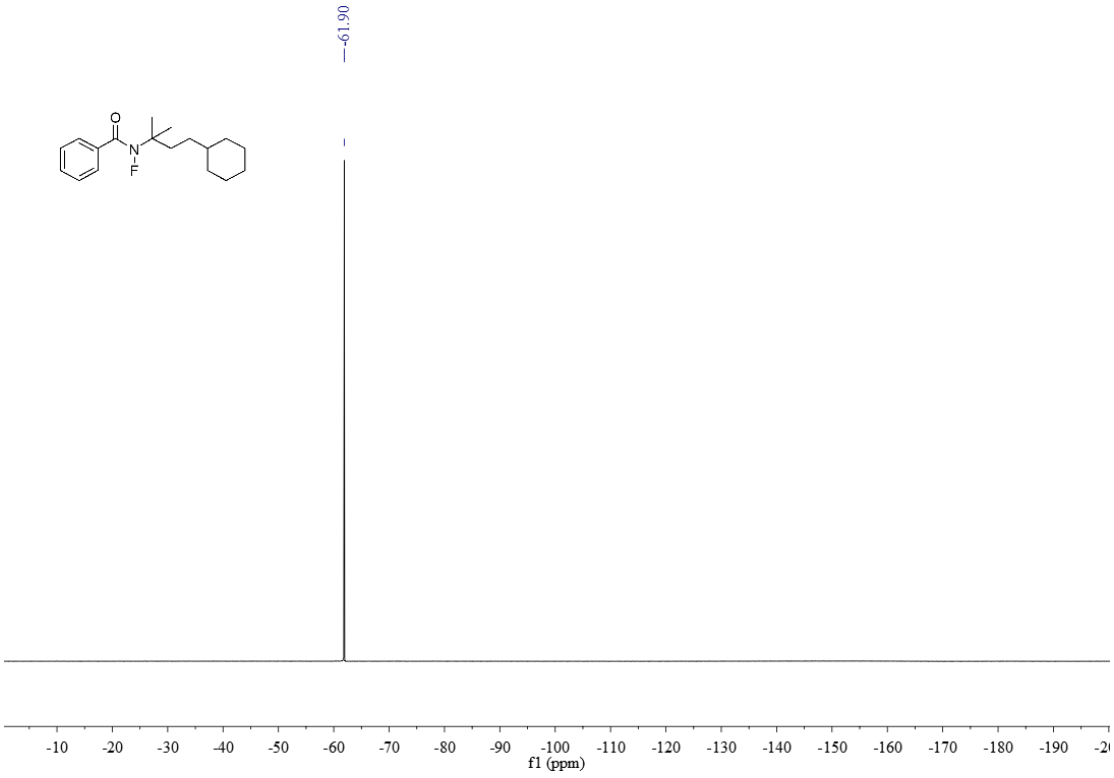
^{1}H NMR of 1ae: 400 MHz, $CDCl_3$



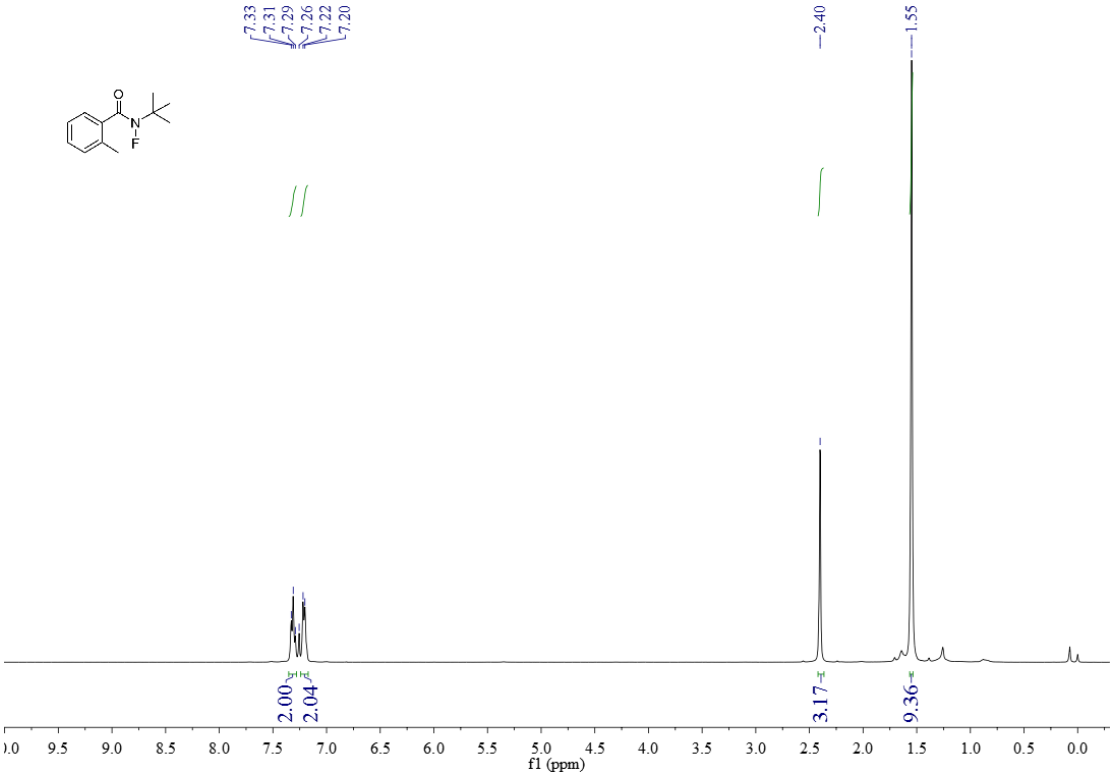
^{13}C NMR of 1ae: 150 MHz, $CDCl_3$



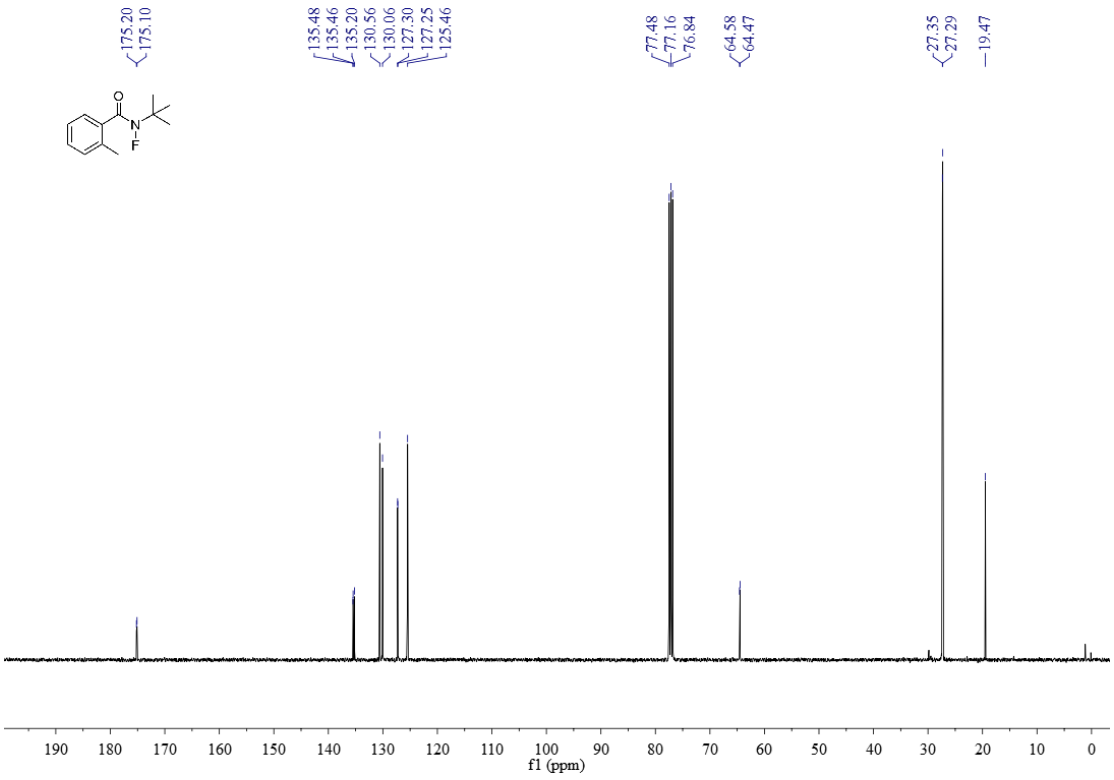
^{19}F NMR of 1ae: 377 MHz, $CDCl_3$



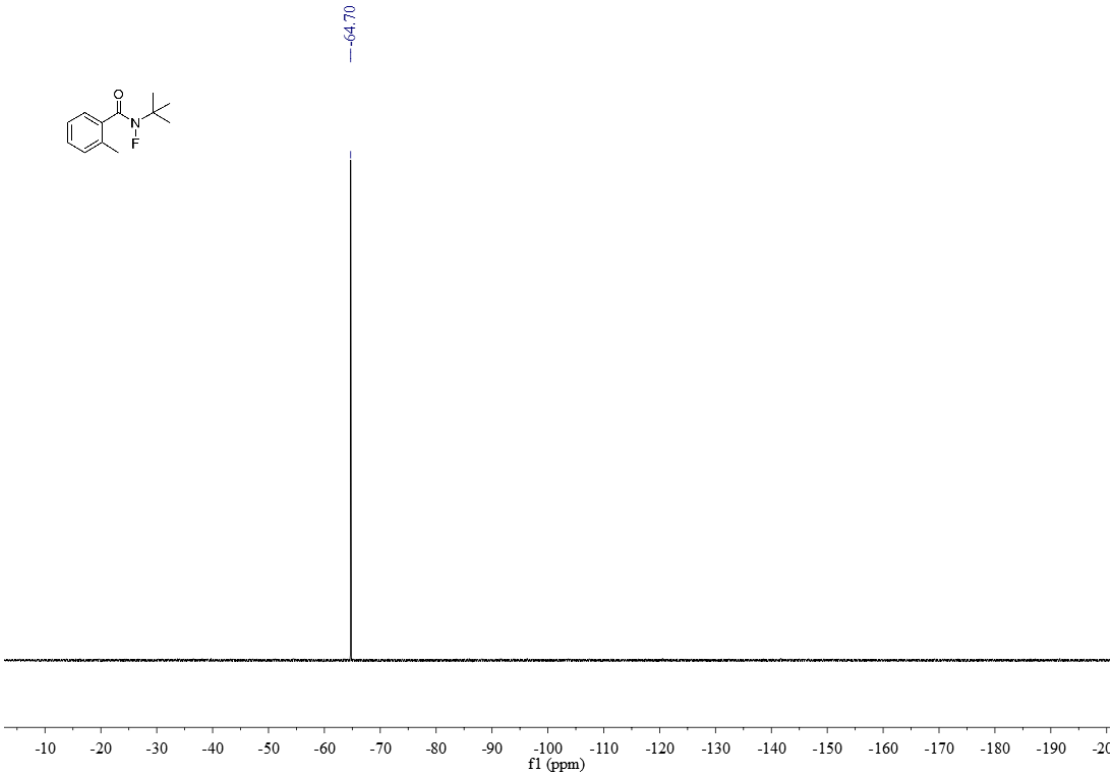
^{1}H NMR of 1af: 400 MHz, $CDCl_3$



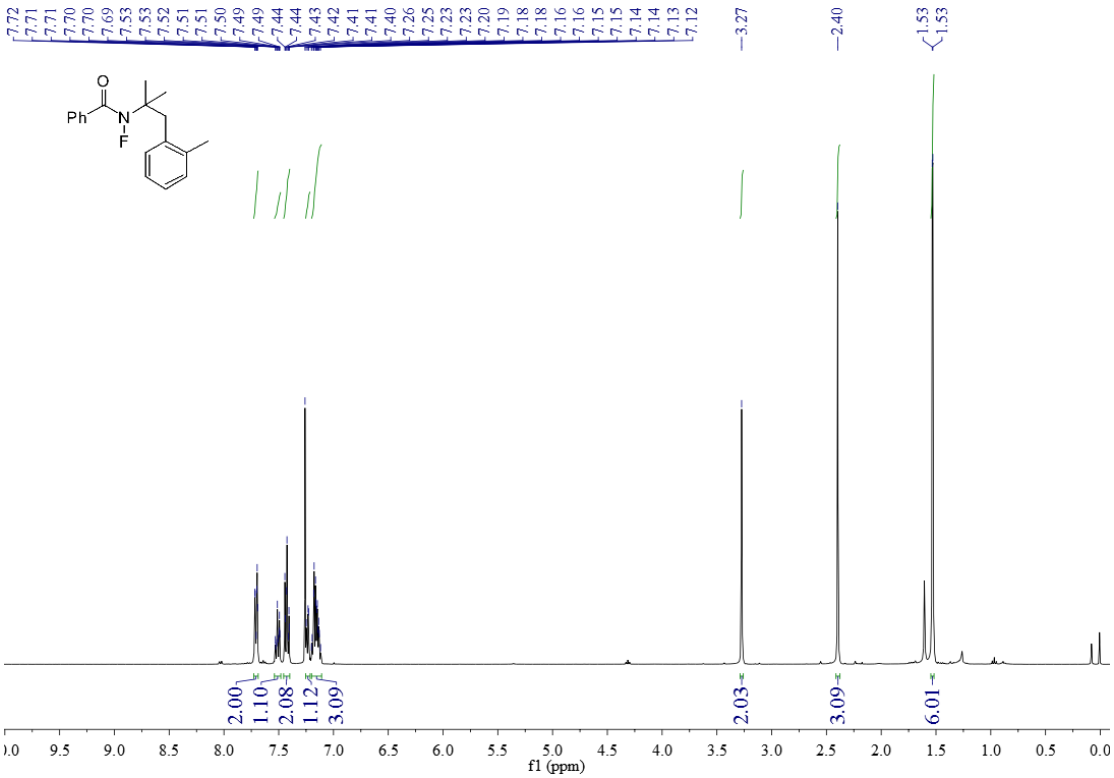
^{13}C NMR of 1af: 100 MHz, $CDCl_3$



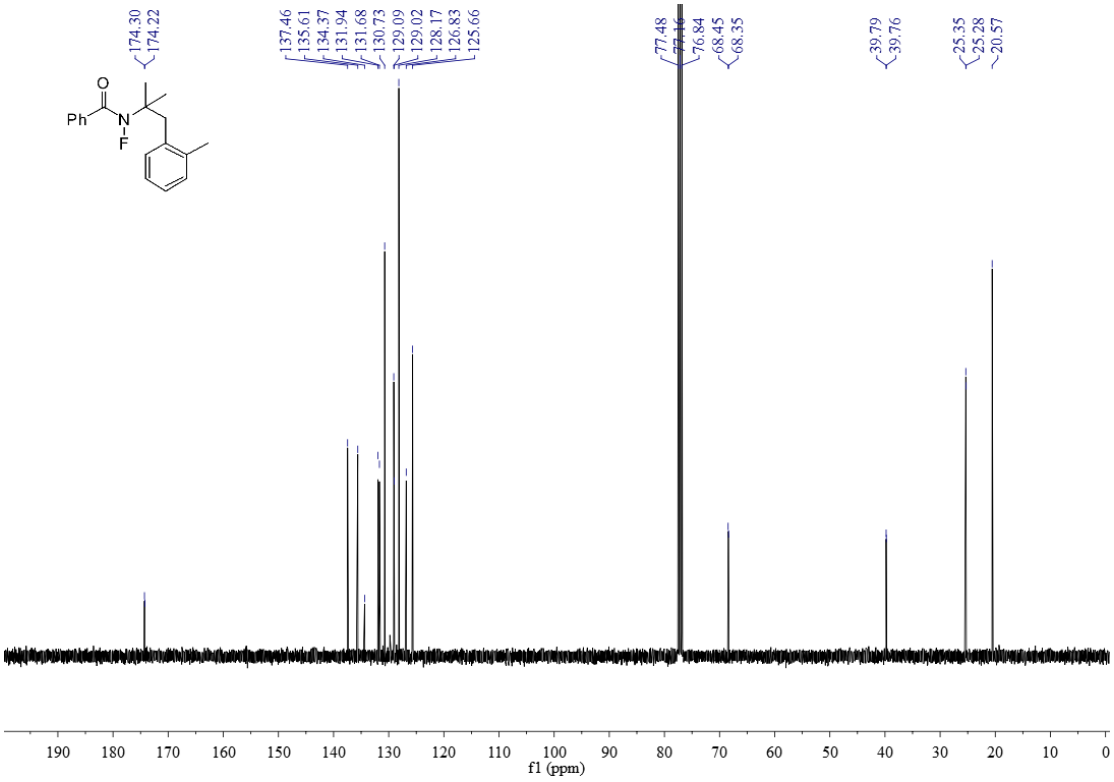
^{19}F NMR of 1af: 564 MHz, $CDCl_3$



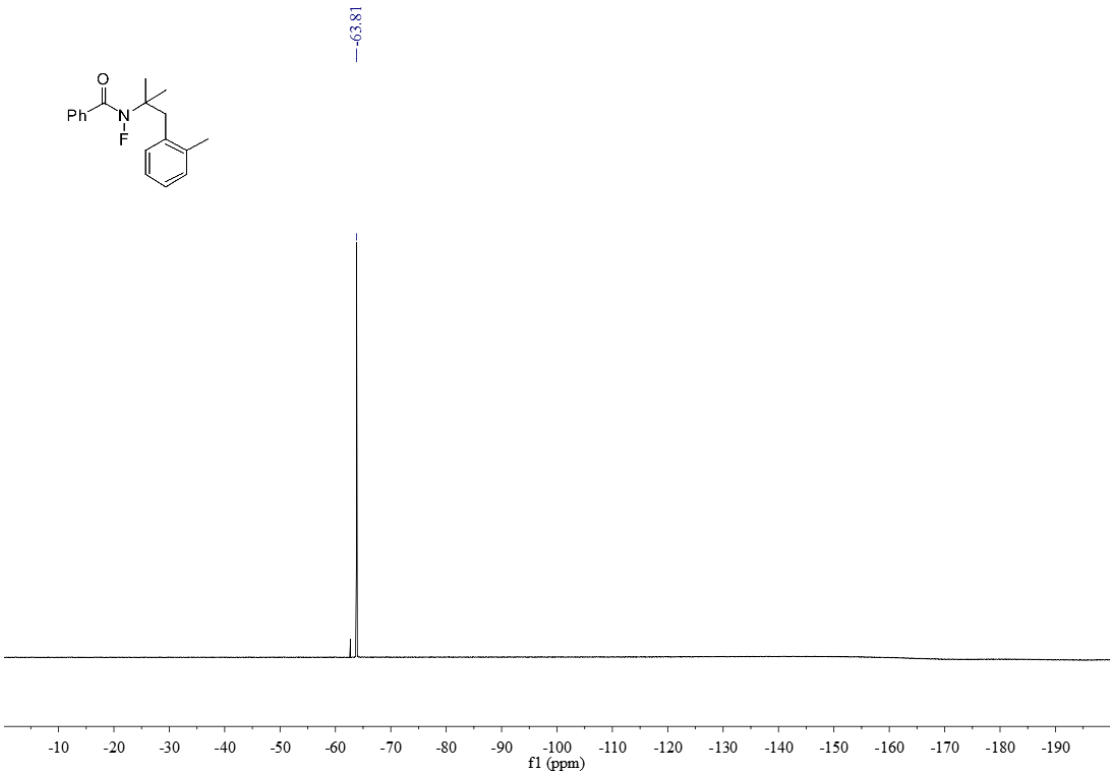
^{1}H NMR of 1ag: 400 MHz, $CDCl_3$



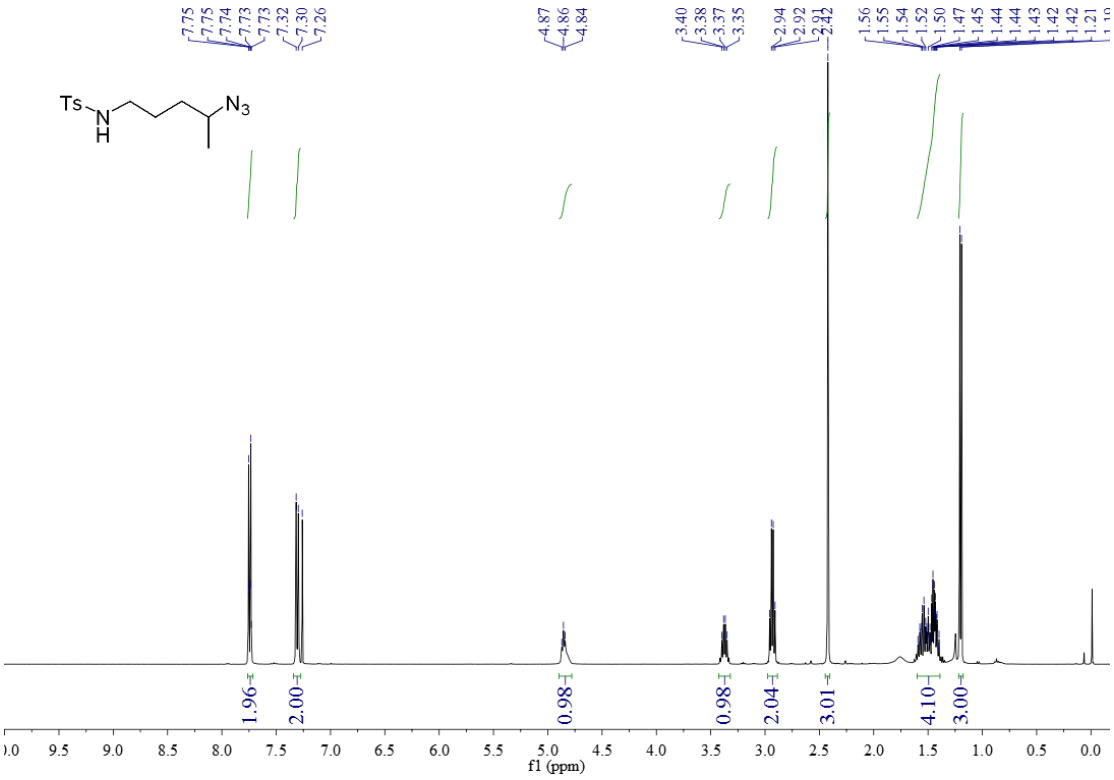
^{13}C NMR of 1ag: 100 MHz, $CDCl_3$



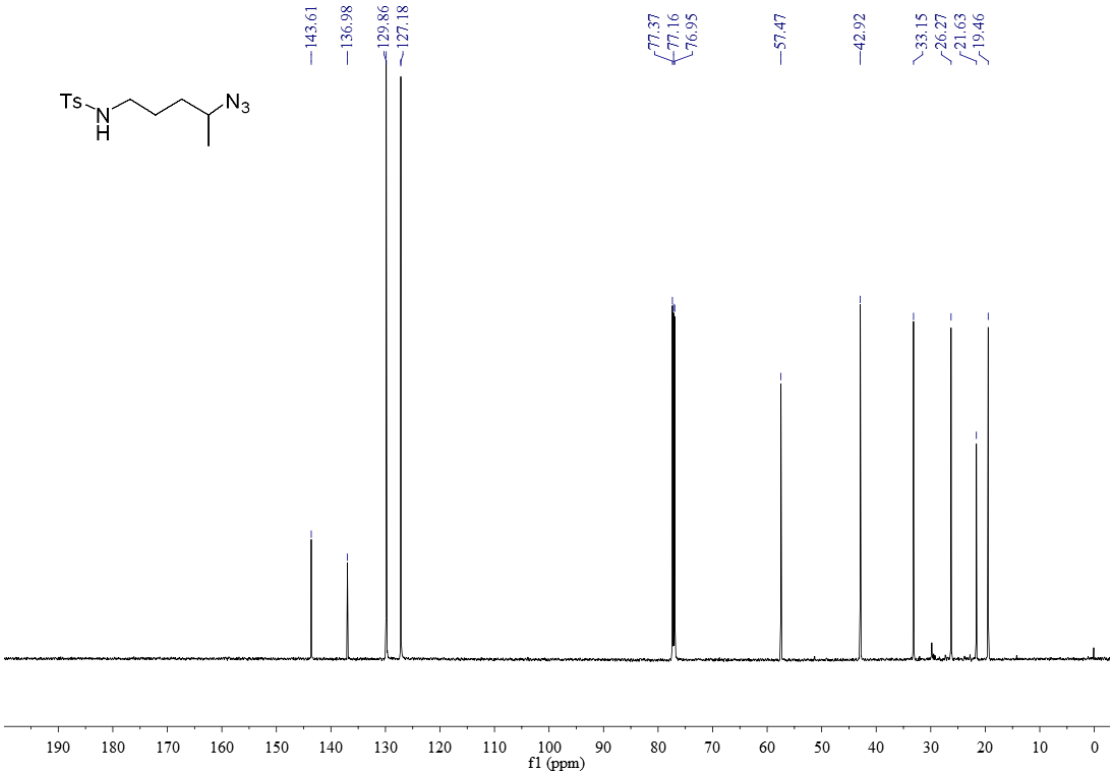
^{19}F NMR of 1ag: 377 MHz, $CDCl_3$



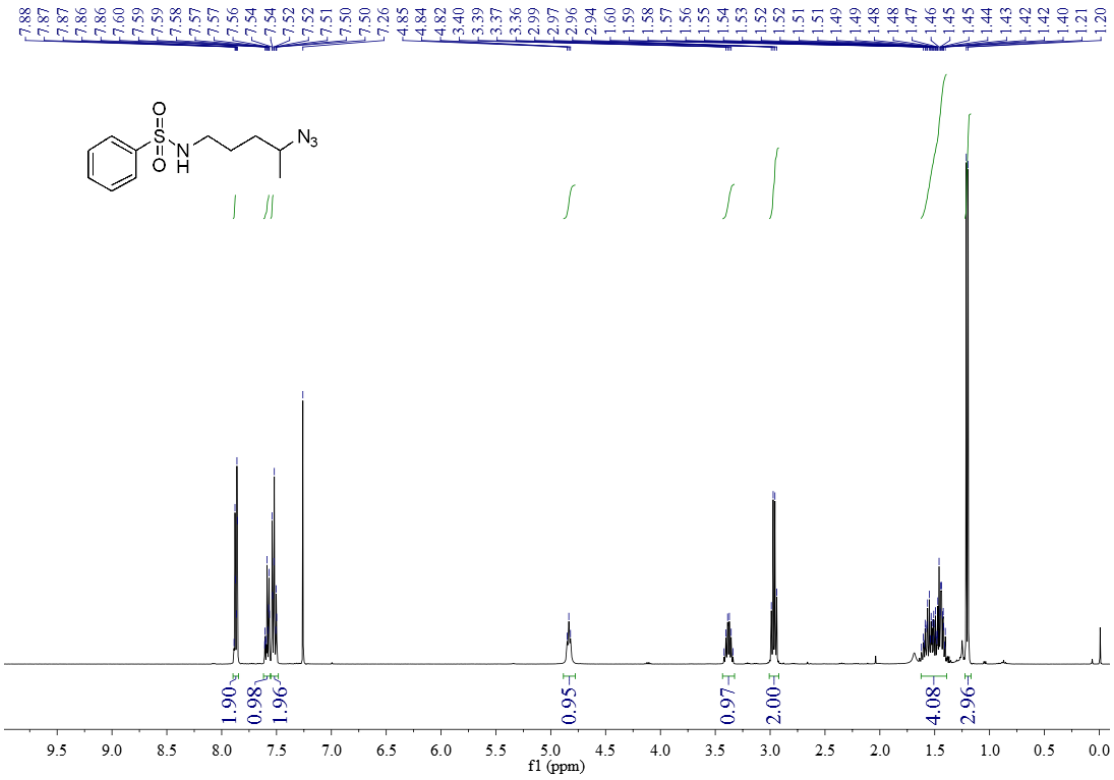
^{1}H NMR of 2a: 400 MHz, $CDCl_3$



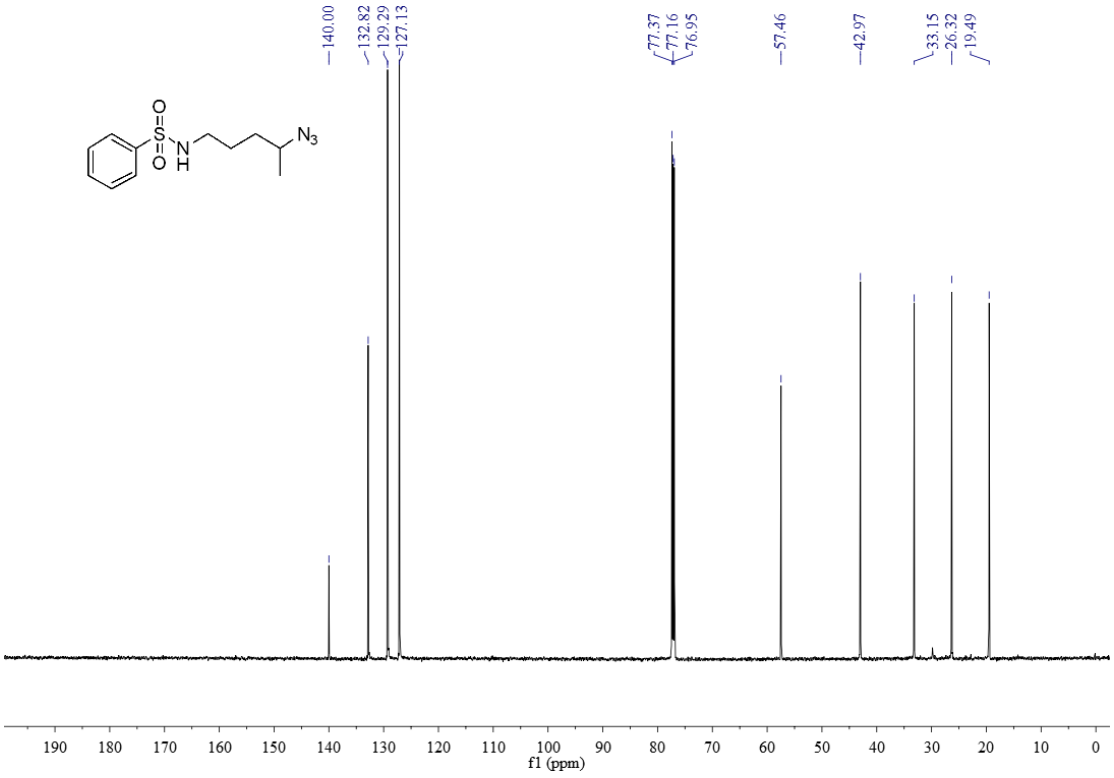
^{13}C NMR of 2a: 150 MHz, $CDCl_3$



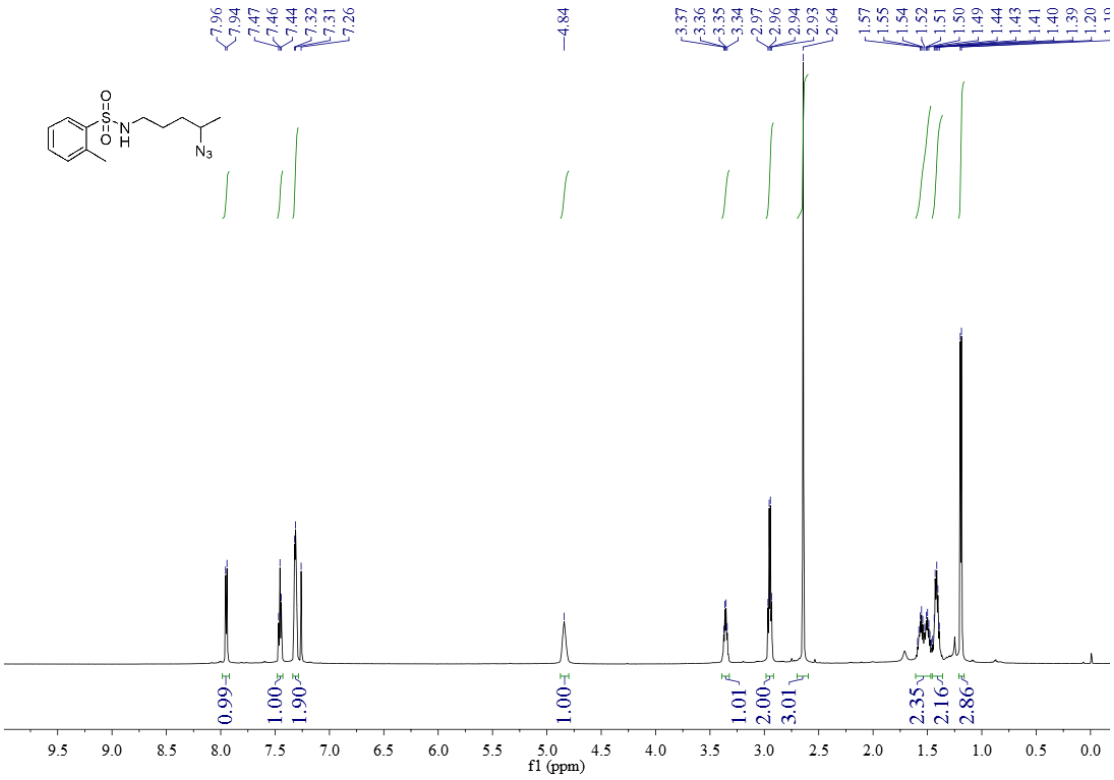
^{1}H NMR of 2b: 400 MHz, $CDCl_3$



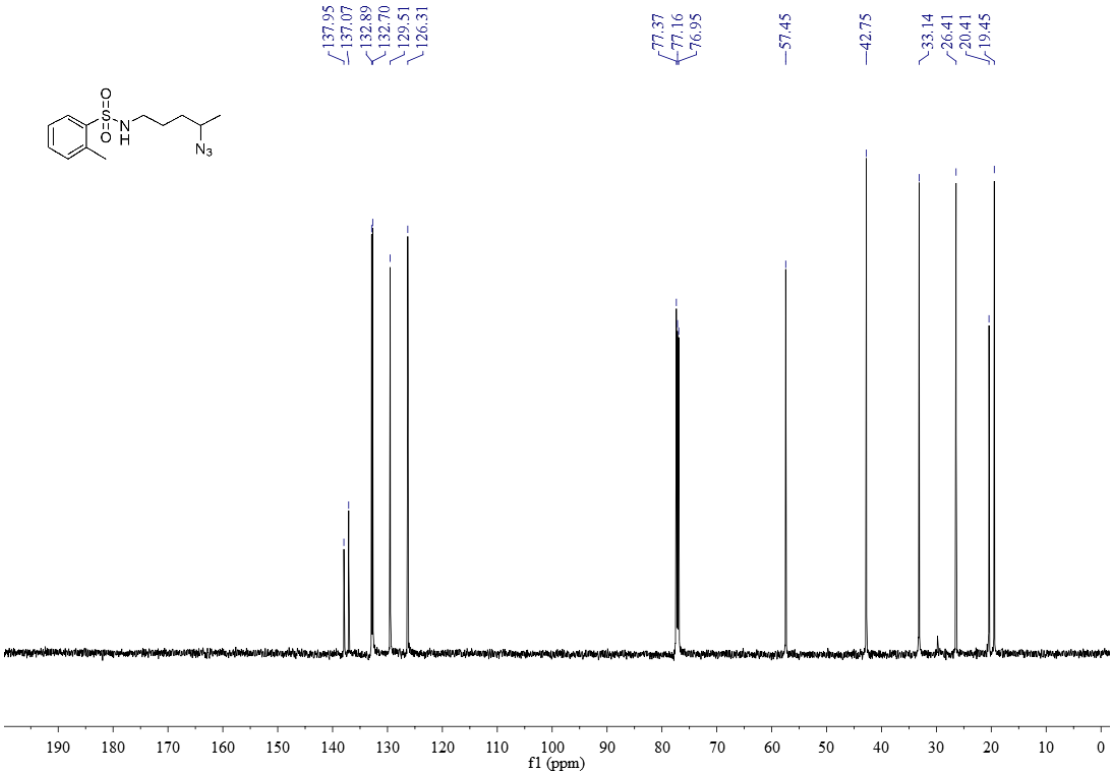
^{13}C NMR of 2b: 150 MHz, $CDCl_3$



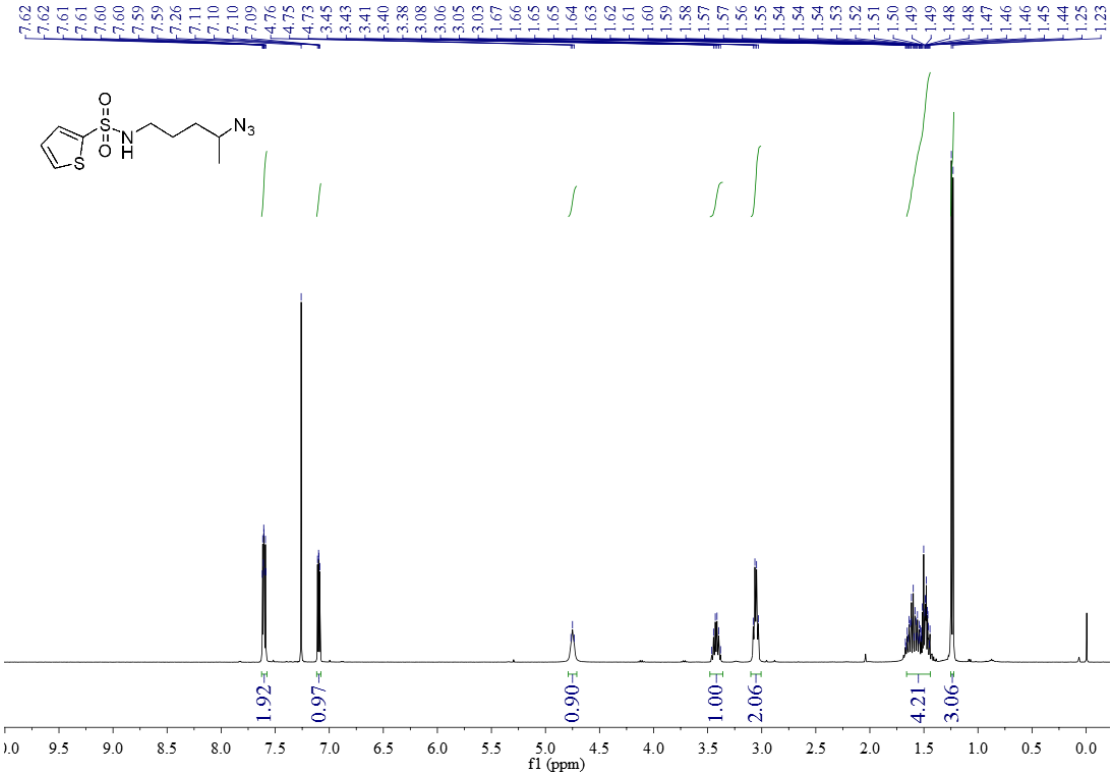
^{1}H NMR of 2c: 600 MHz, $CDCl_3$



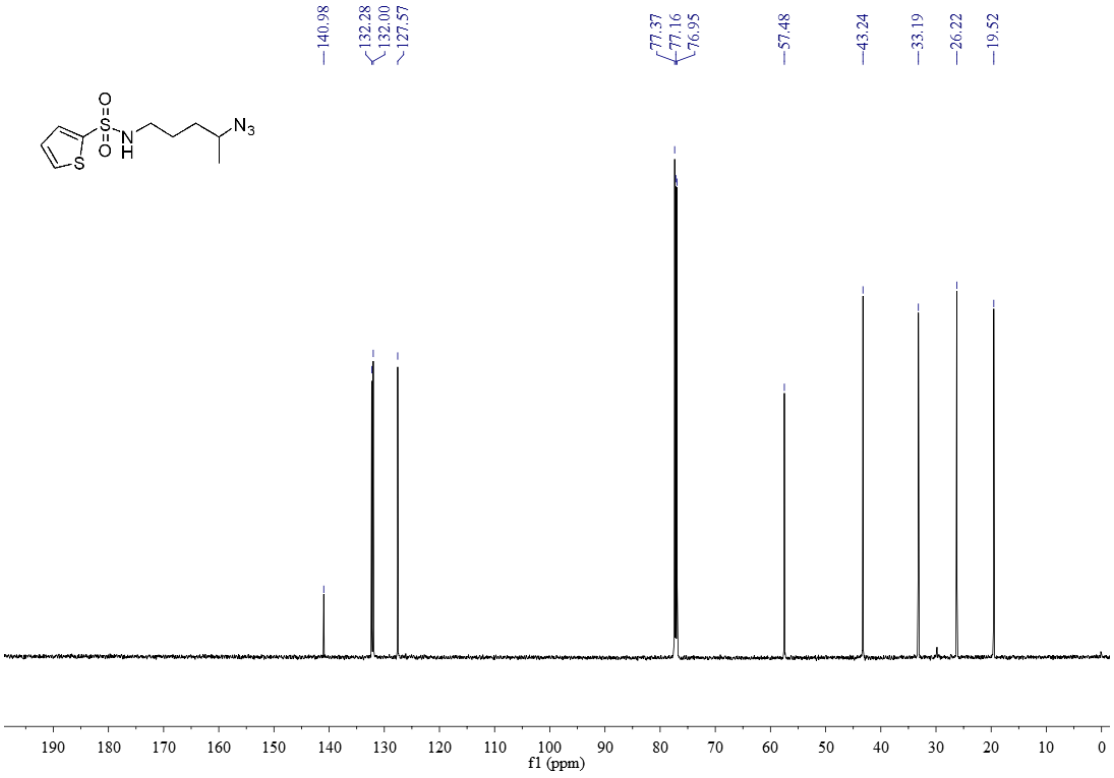
^{13}C NMR of 2c: 150 MHz, $CDCl_3$



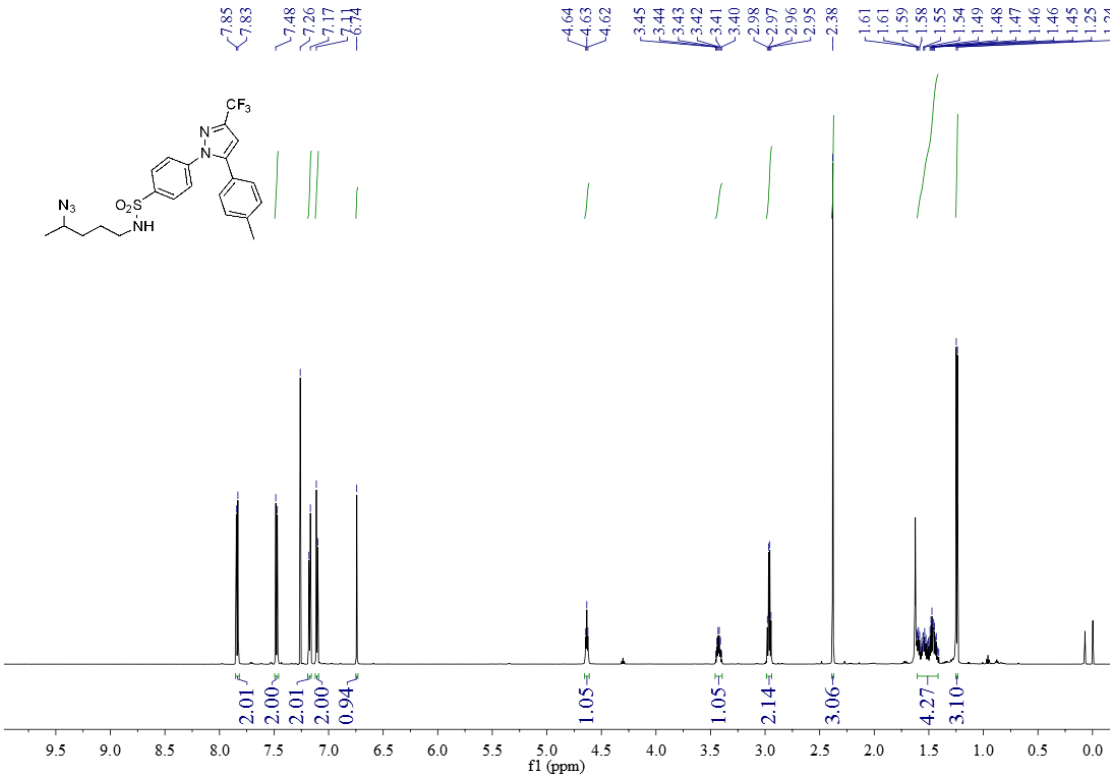
^{1}H NMR of 2d: 400 MHz, $CDCl_3$



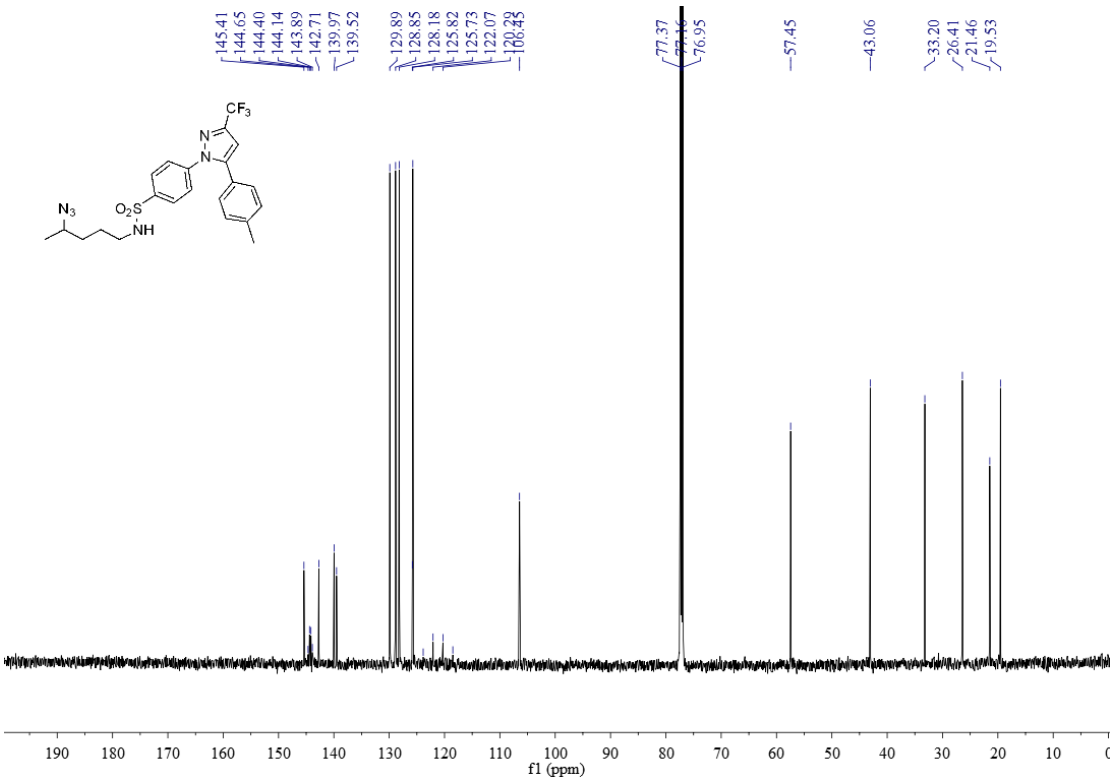
^{13}C NMR of 2d: 150 MHz, $CDCl_3$



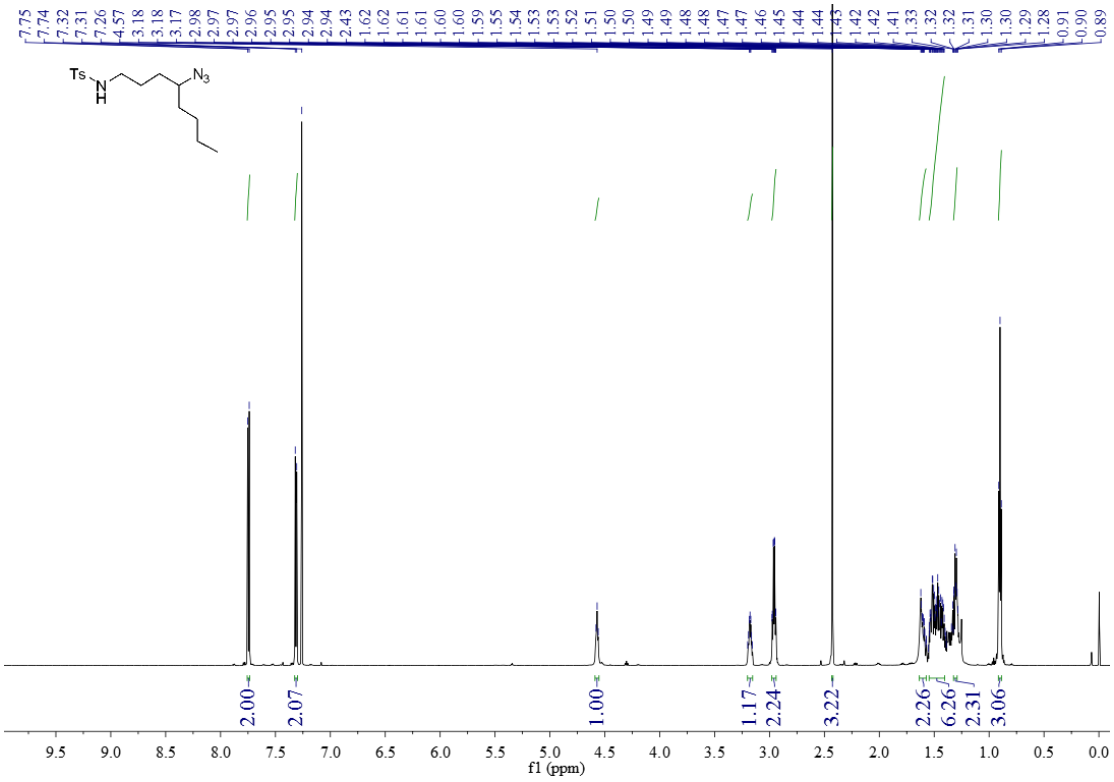
^{1}H NMR of 2e: 600 MHz, $CDCl_3$



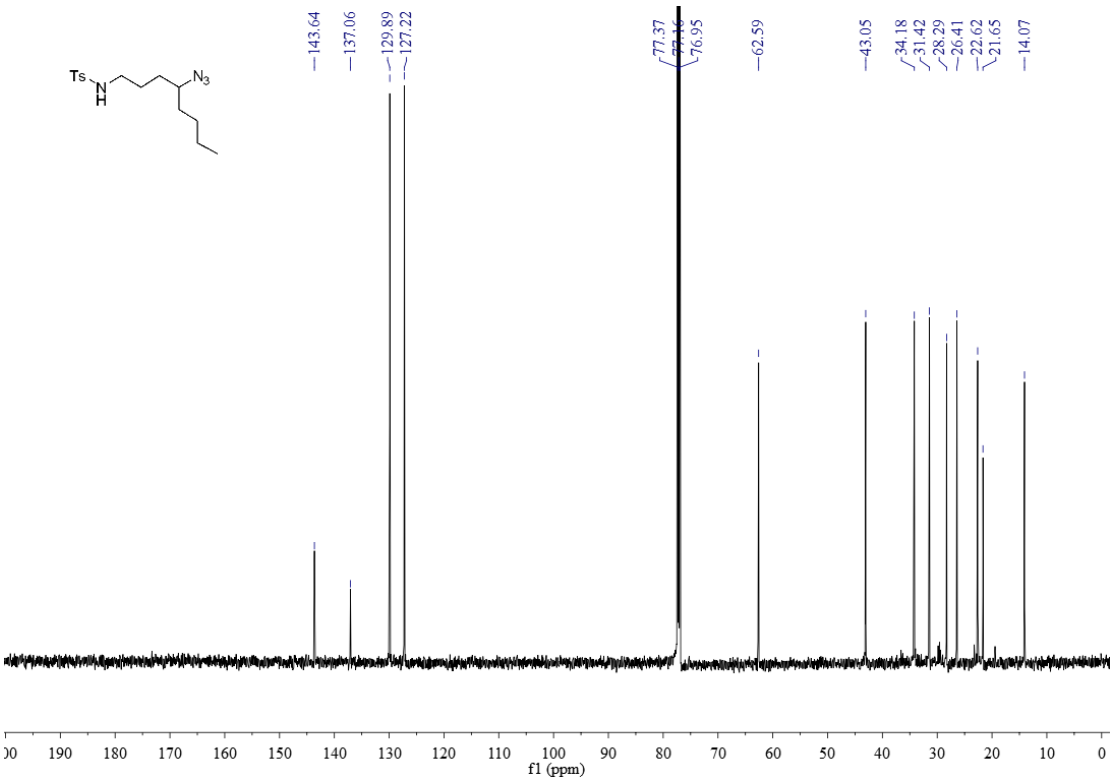
^{13}C NMR of 2e: 150 MHz, $CDCl_3$



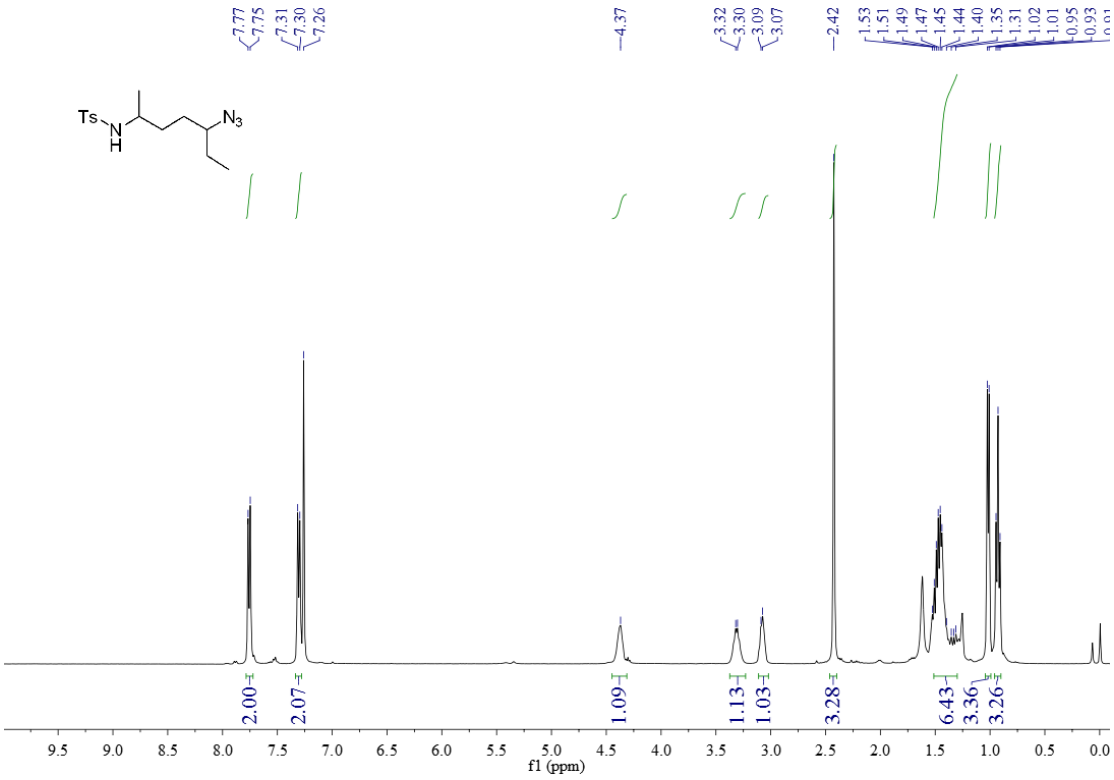
^{1}H NMR of 2f: 600 MHz, $CDCl_3$



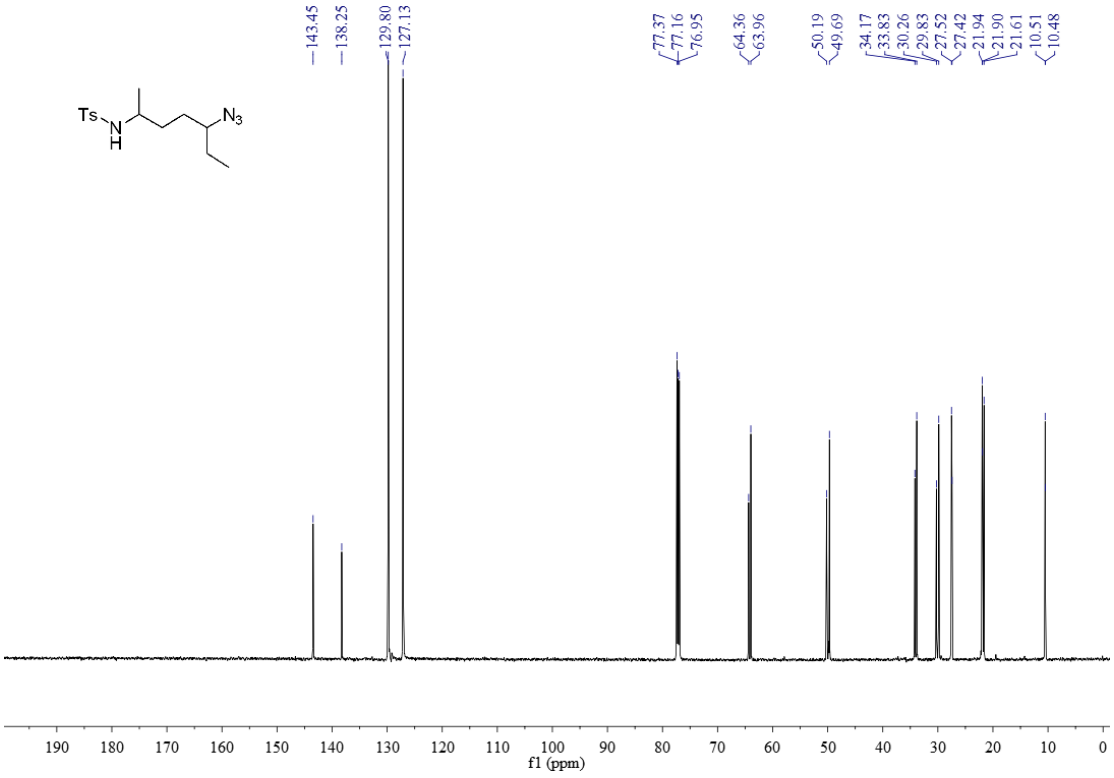
^{13}C NMR of 2f: 150 MHz, $CDCl_3$



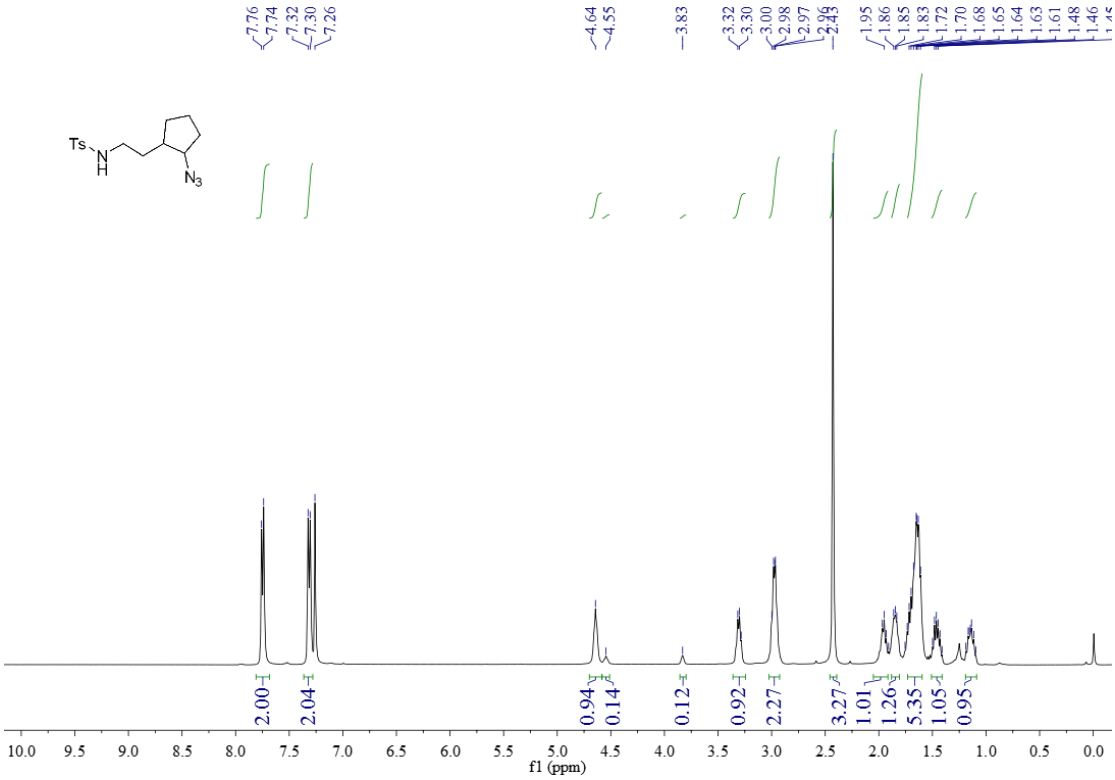
^{1}H NMR of 2g: 400 MHz, $CDCl_3$



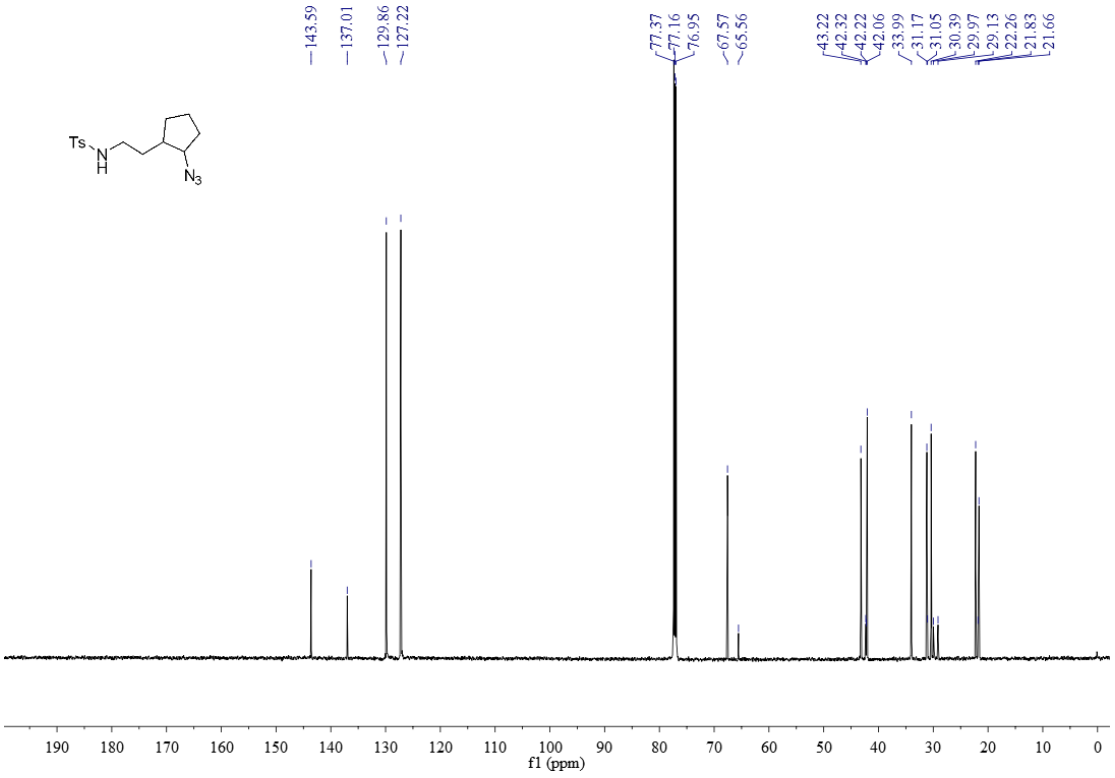
^{13}C NMR of 2g: 150 MHz, $CDCl_3$



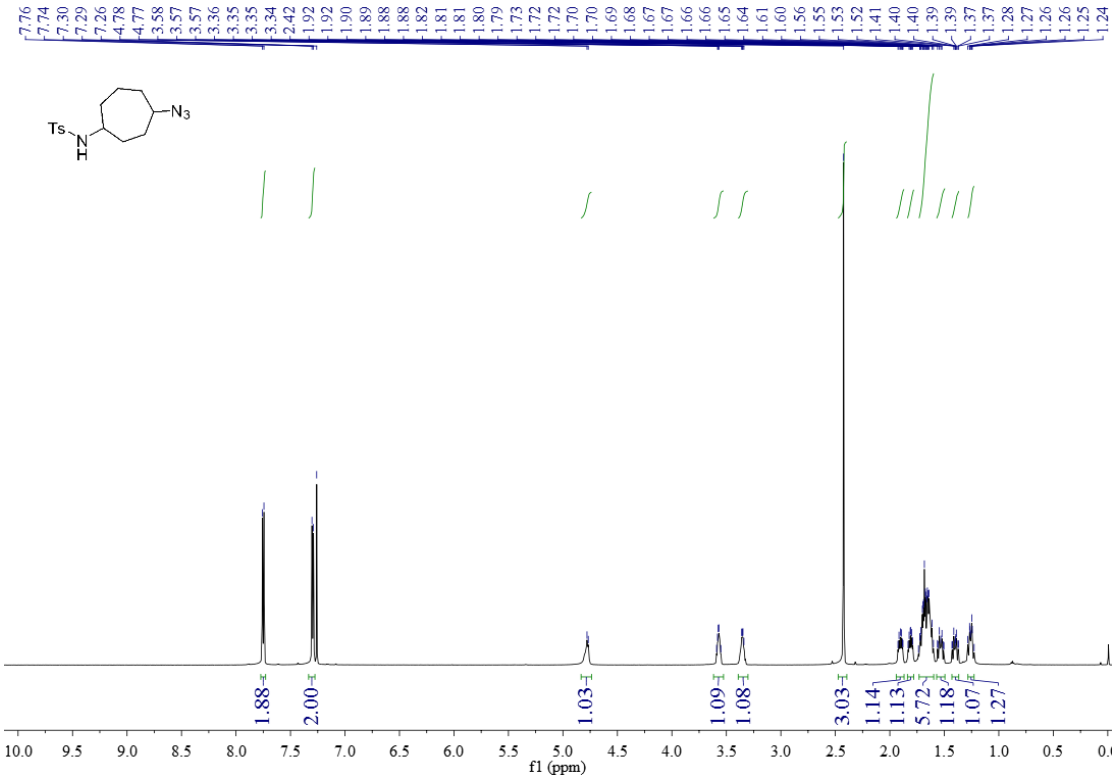
^{1}H NMR of 2h: 400 MHz, $CDCl_3$



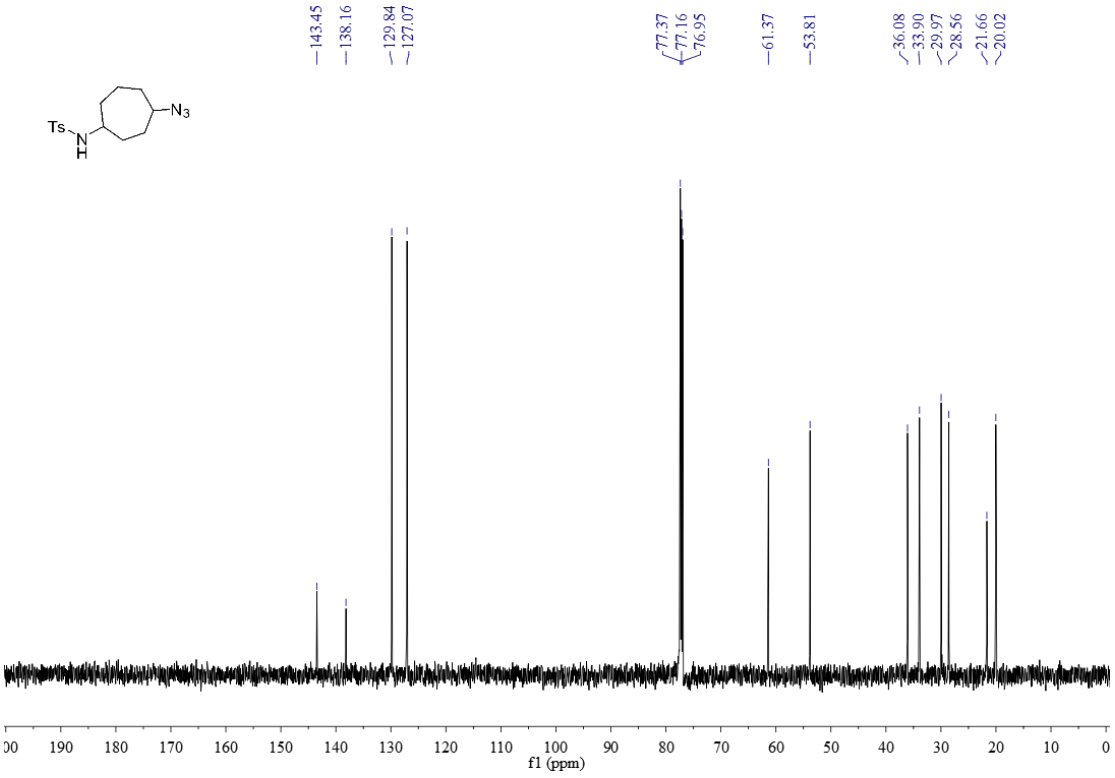
^{13}C NMR of 2h: 150 MHz, $CDCl_3$



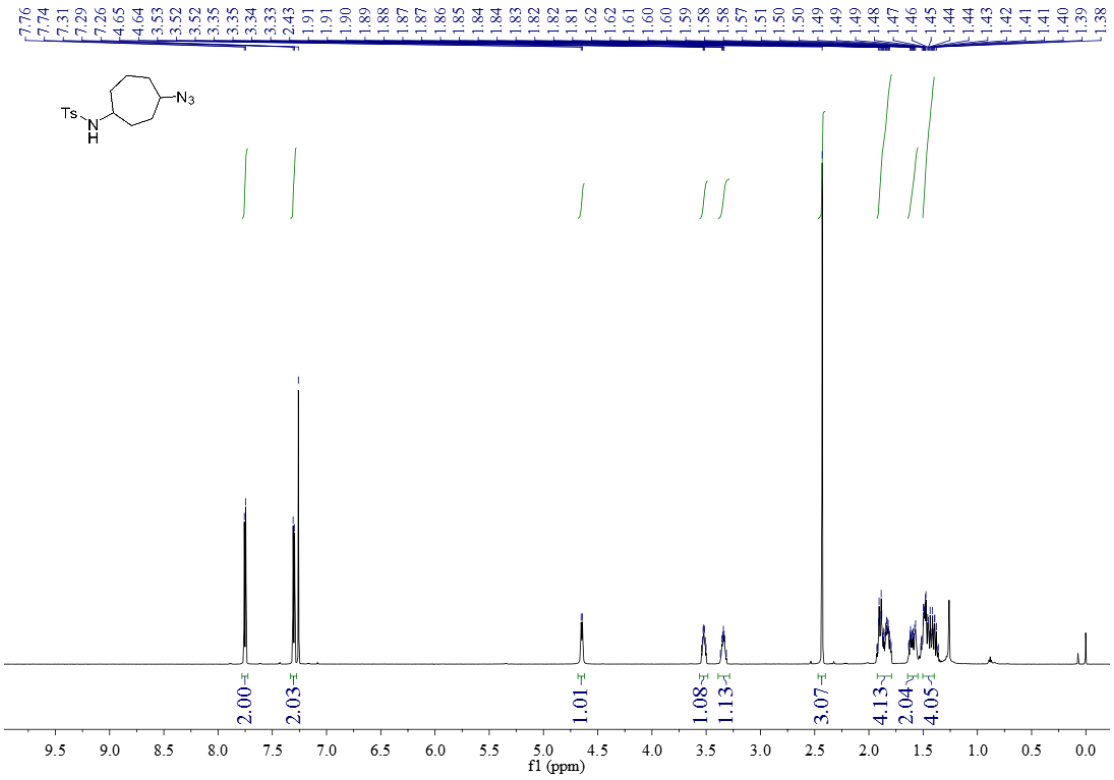
^{1}H NMR of 2i-a: 600 MHz, $CDCl_3$



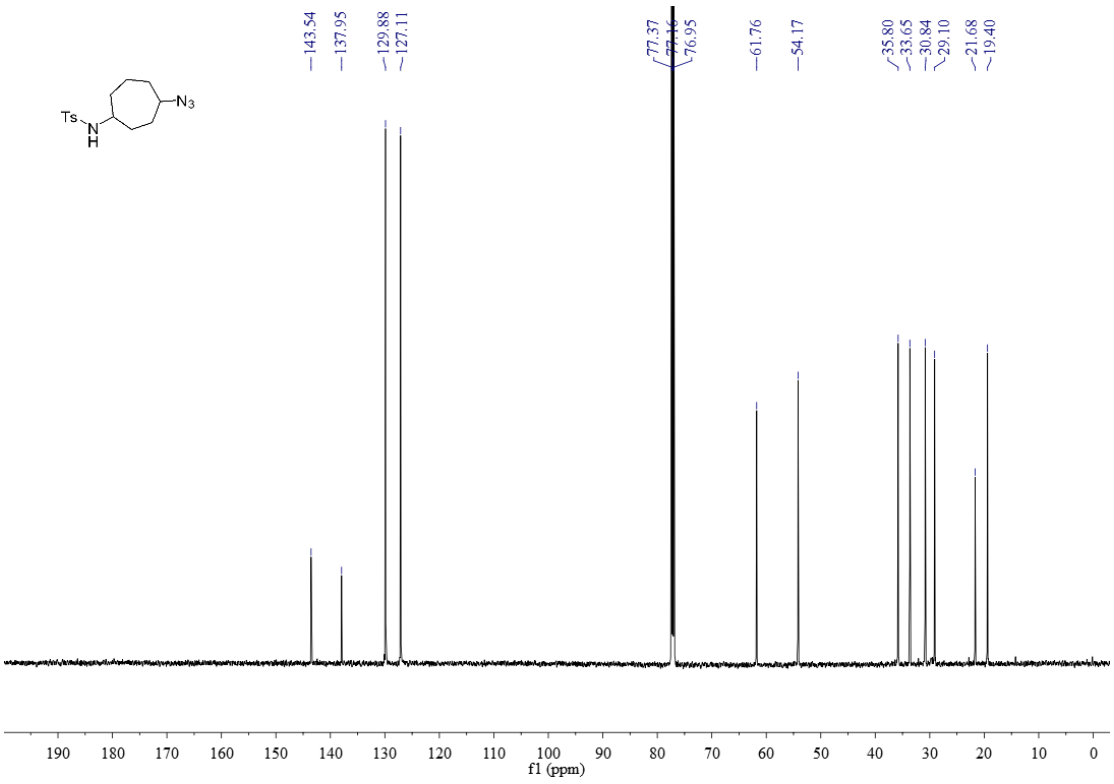
^{13}C NMR of 2i-a: 150 MHz, $CDCl_3$



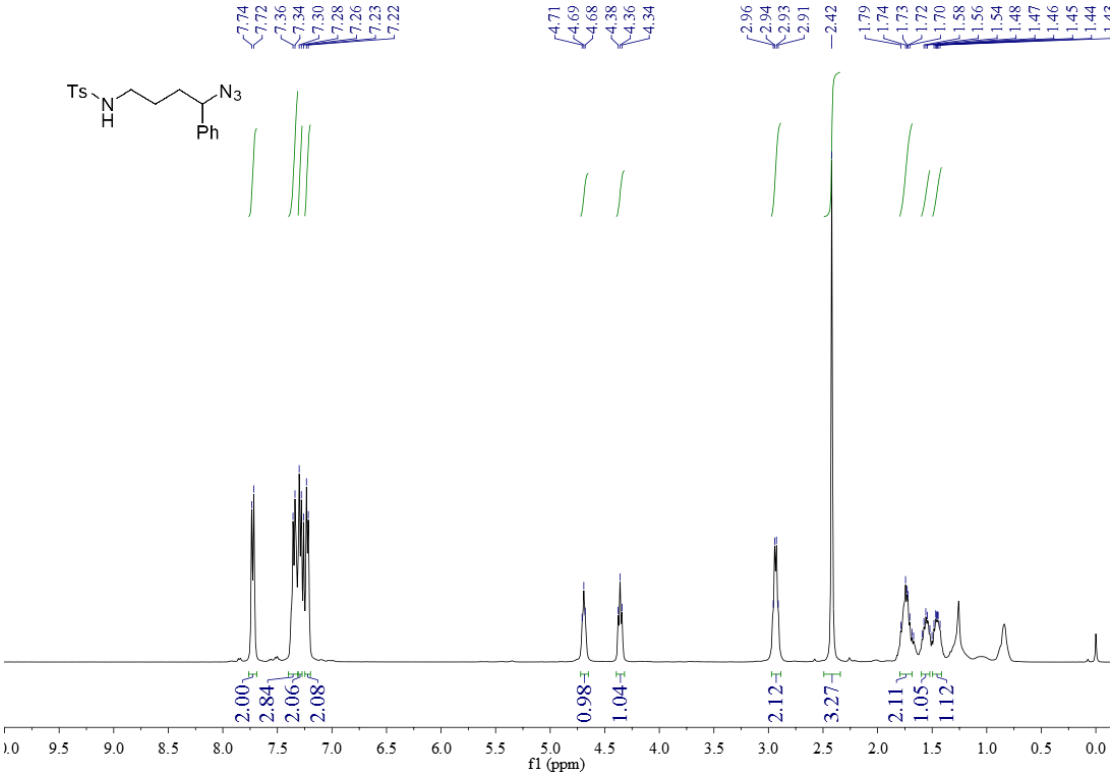
^{1}H NMR of 2i-b: 600 MHz, $CDCl_3$



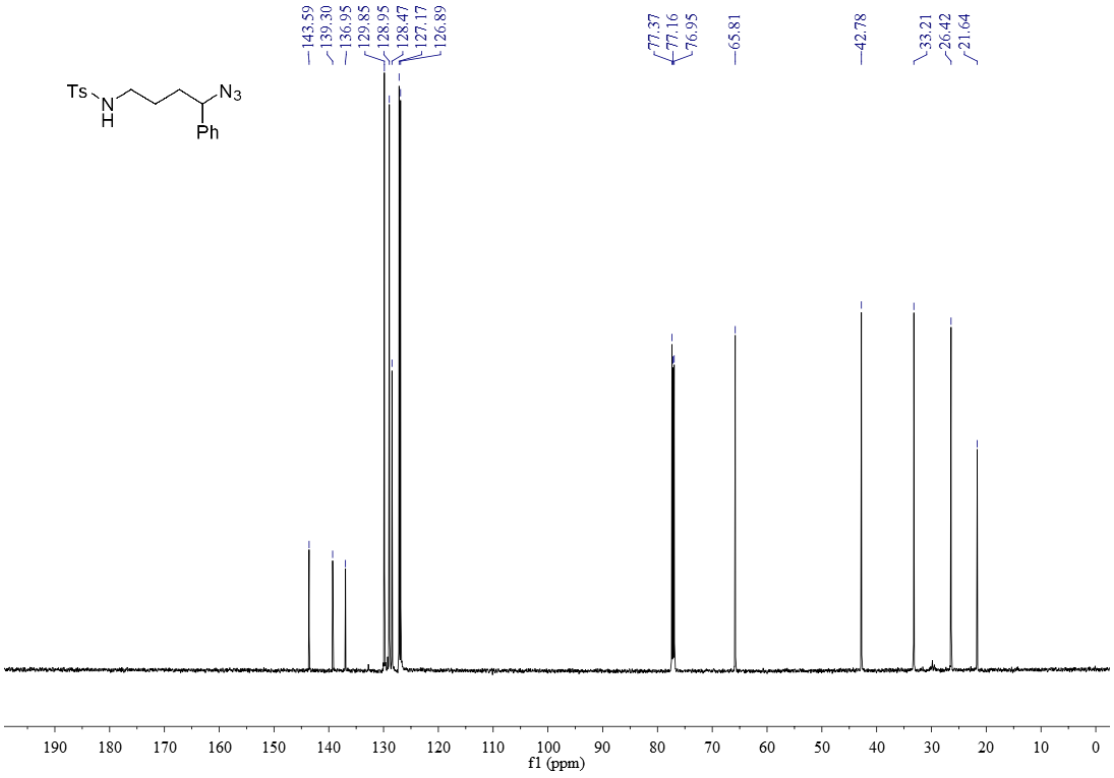
^{13}C NMR of 2i-b: 150 MHz, $CDCl_3$



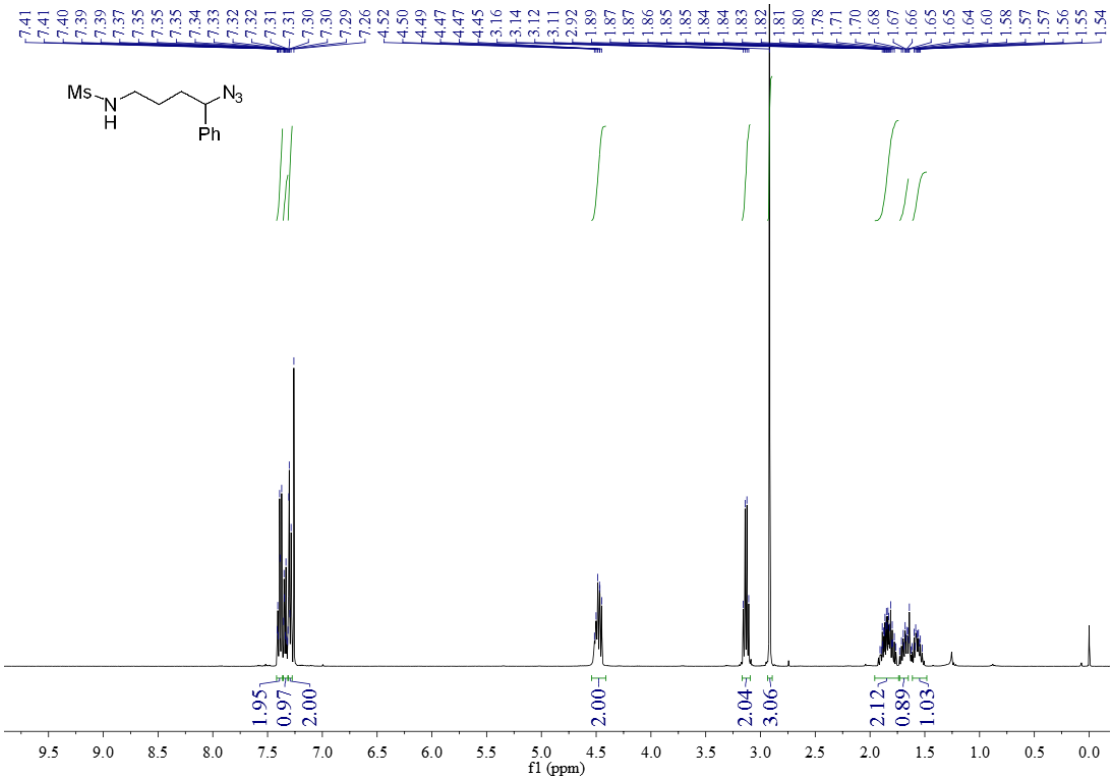
^{1}H NMR of 2j: 400 MHz, $CDCl_3$



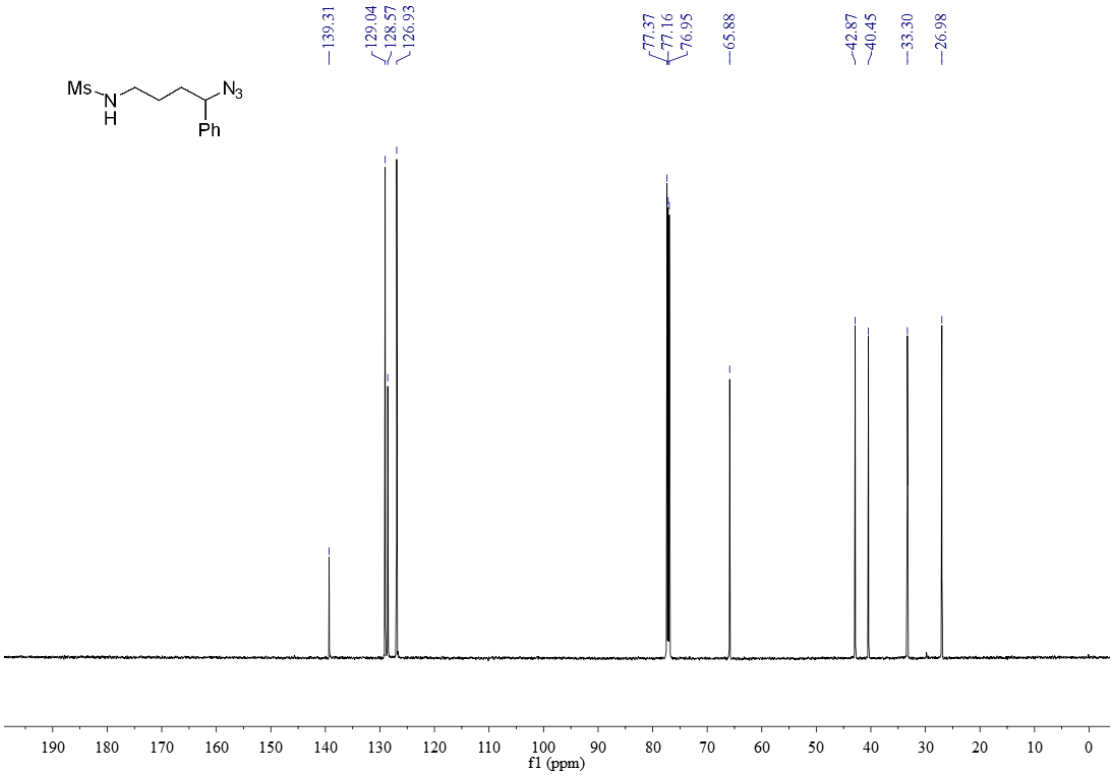
^{13}C NMR of 2j: 150 MHz, $CDCl_3$



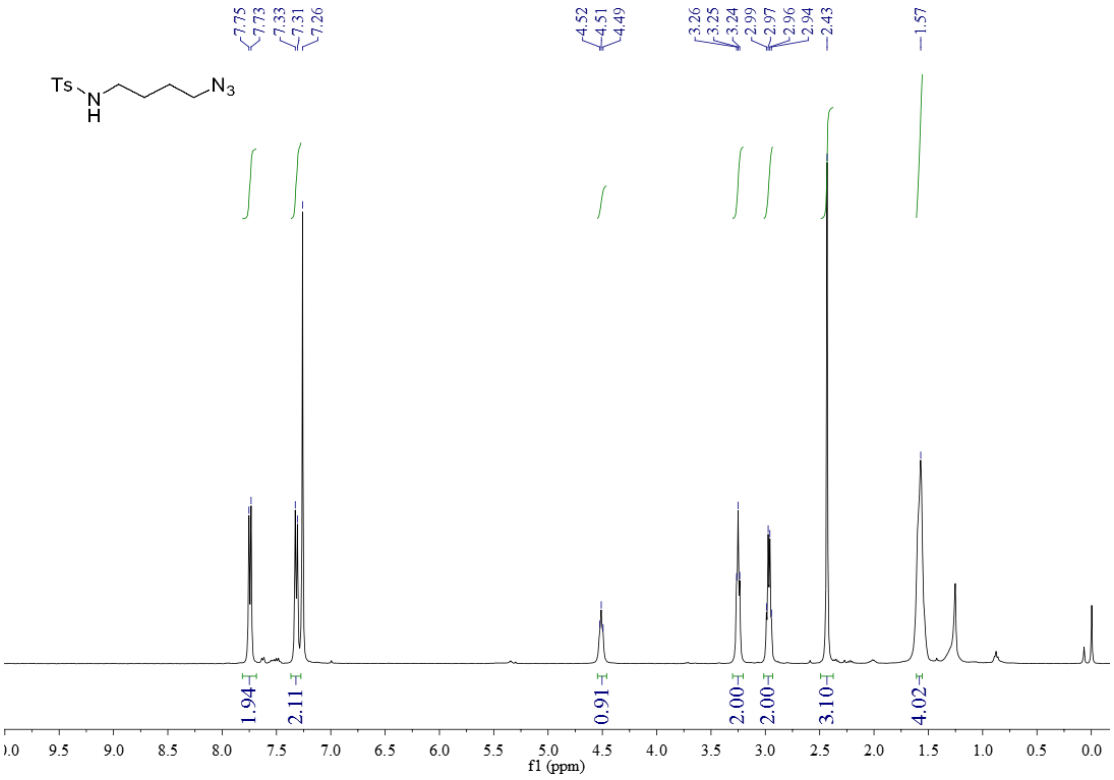
^{1}H NMR of 2k: 400 MHz, $CDCl_3$



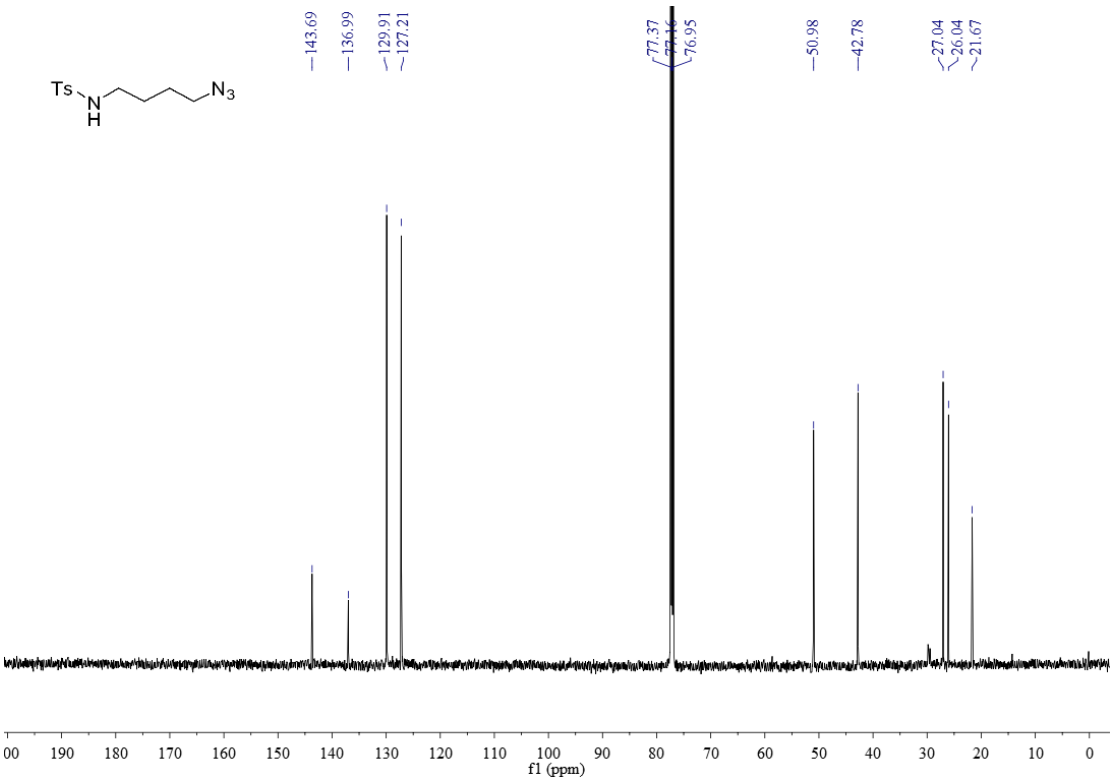
^{13}C NMR of 2k: 150 MHz, $CDCl_3$



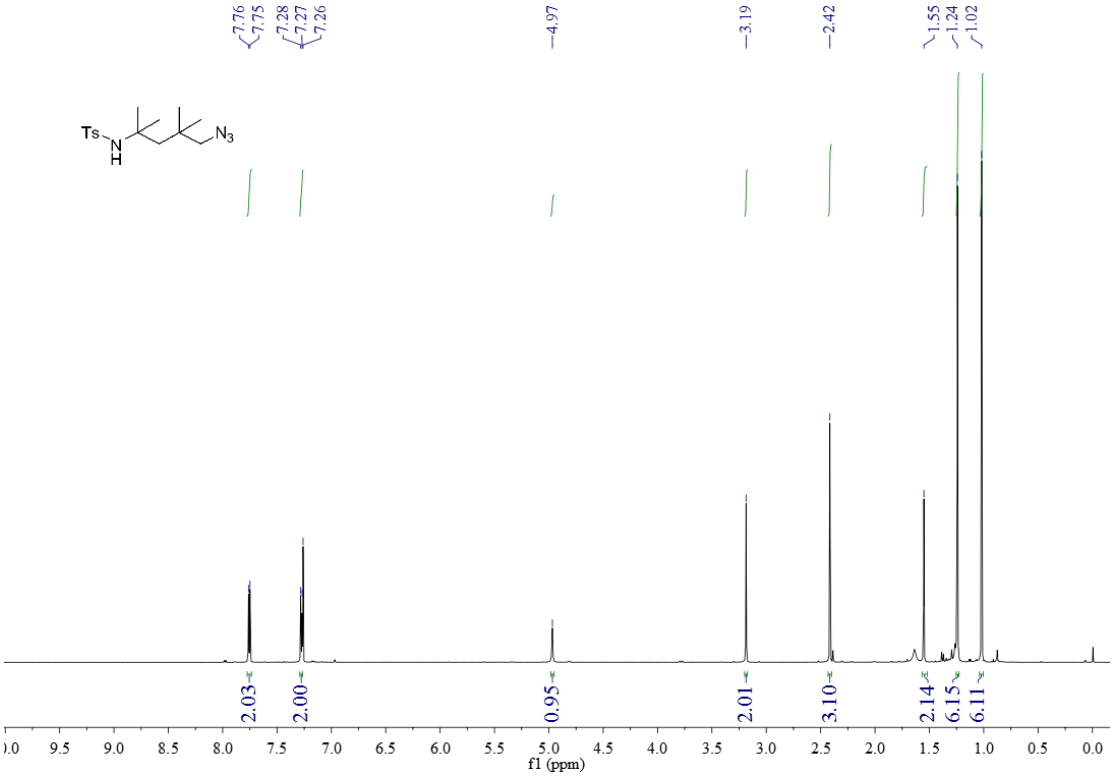
^{1}H NMR of 2l: 400 MHz, $CDCl_3$



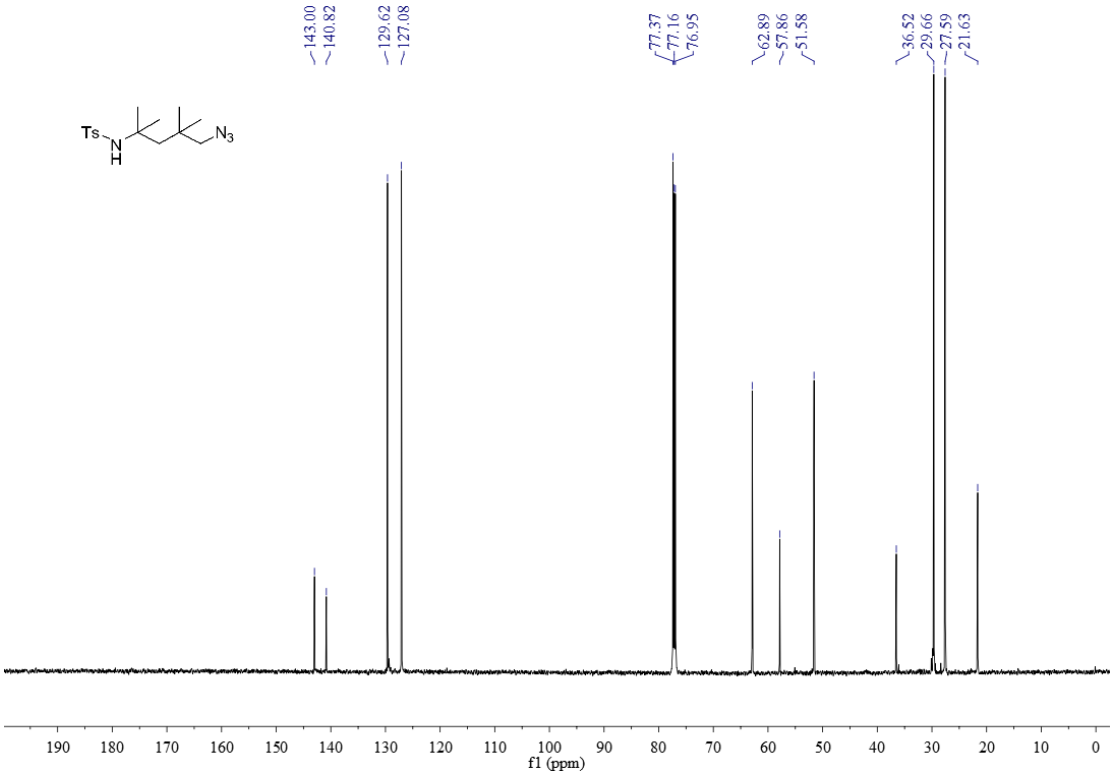
^{13}C NMR of 2l: 150 MHz, $CDCl_3$



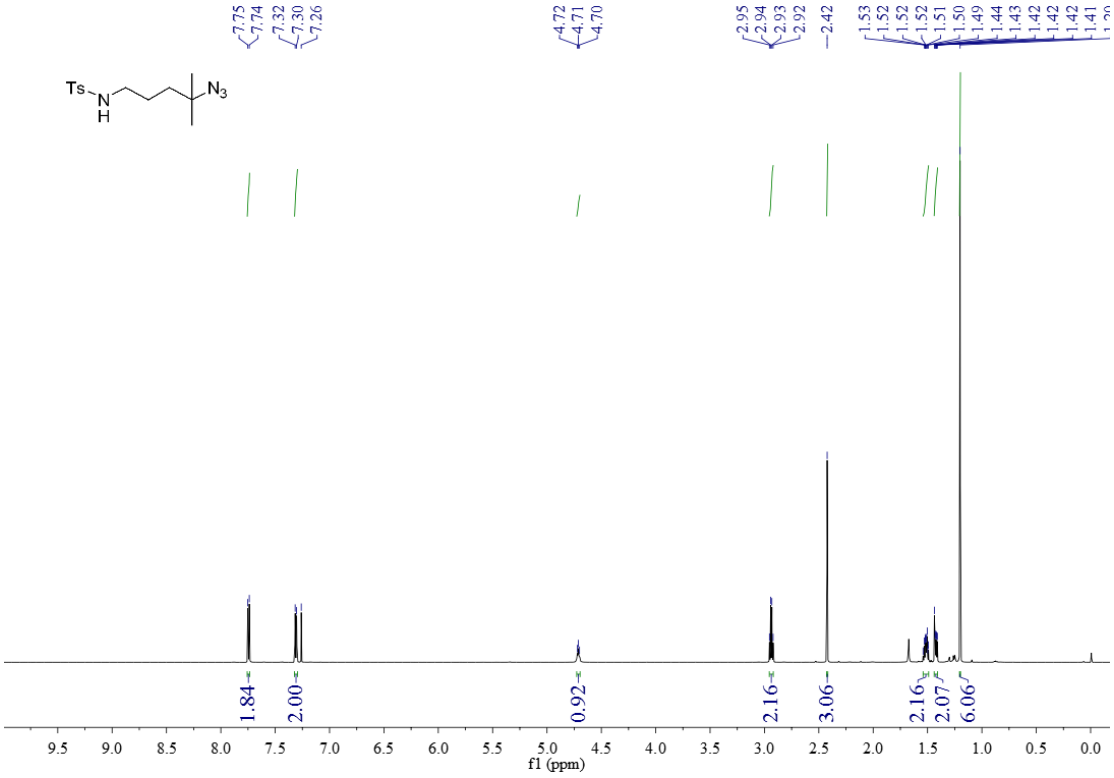
^{1}H NMR of 2m: 600 MHz, $CDCl_3$



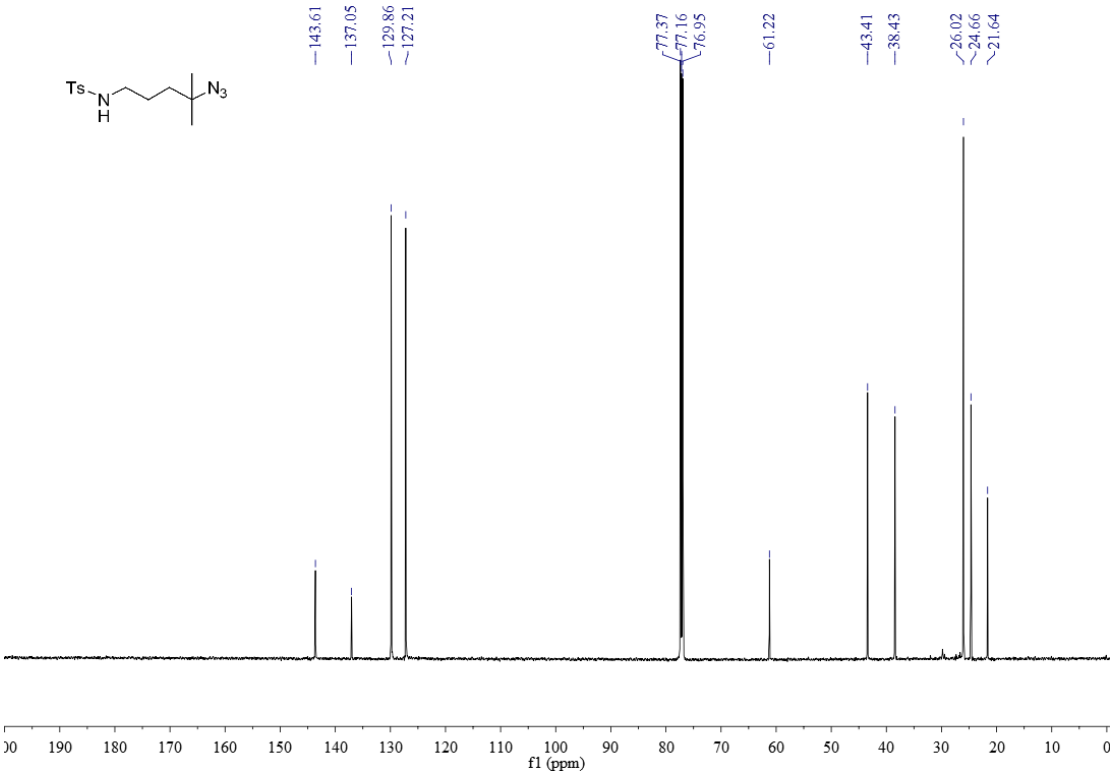
^{13}C NMR of 2m: 150 MHz, $CDCl_3$



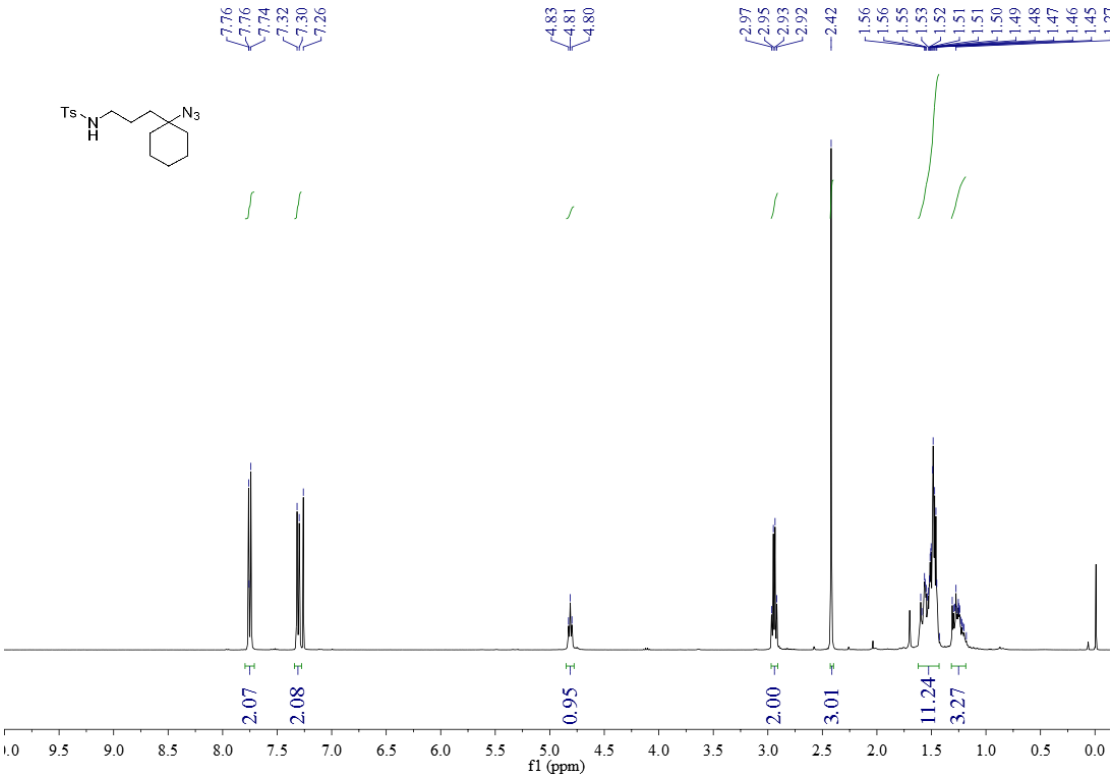
^{1}H NMR of 2n: 600 MHz, $CDCl_3$



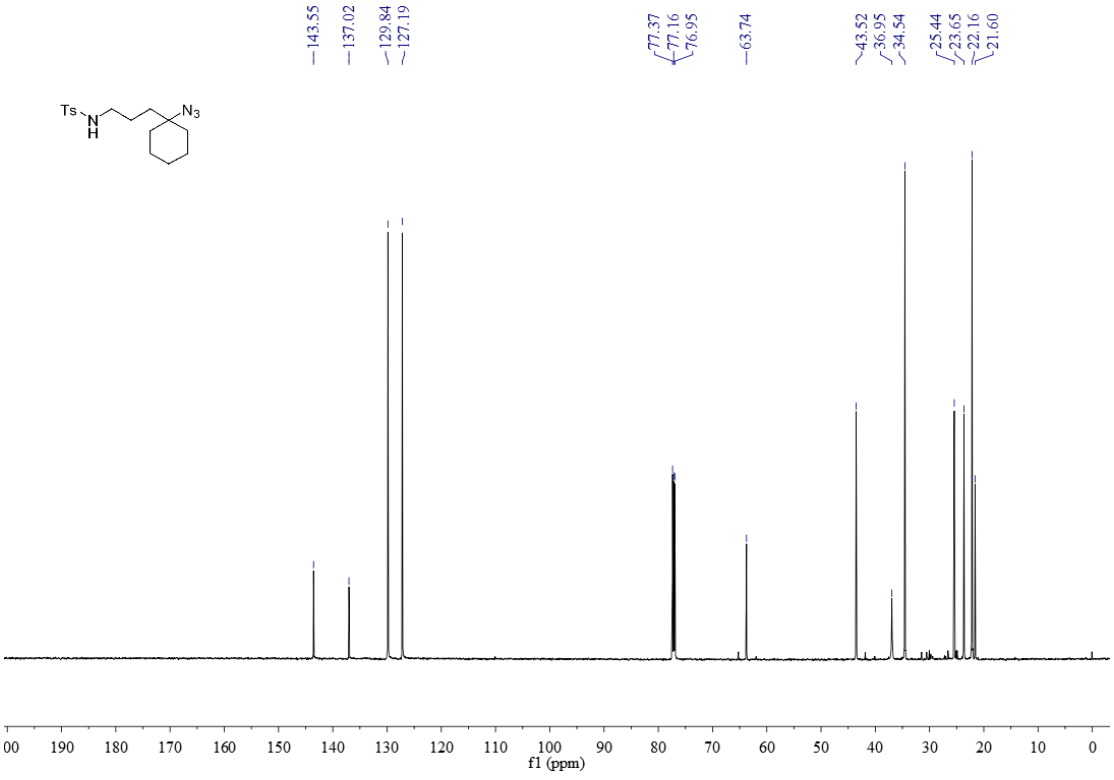
^{13}C NMR of 2n: 150 MHz, $CDCl_3$



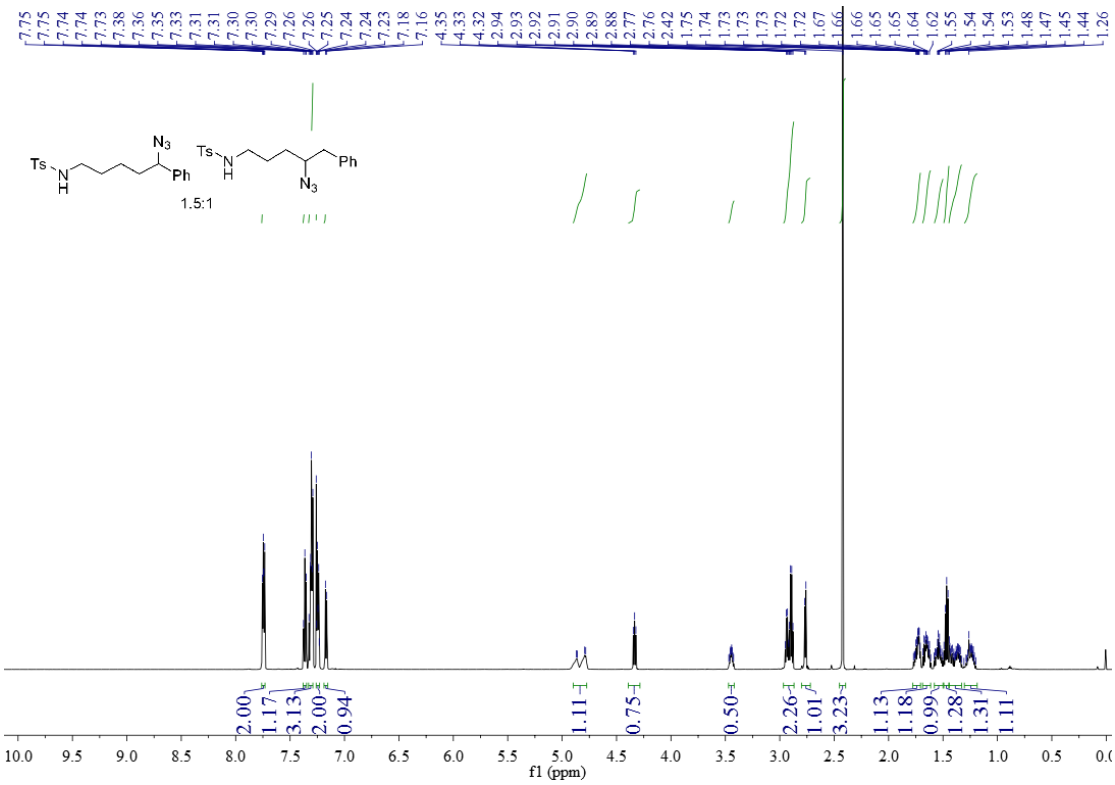
^{1}H NMR of 2o: 400 MHz, $CDCl_3$



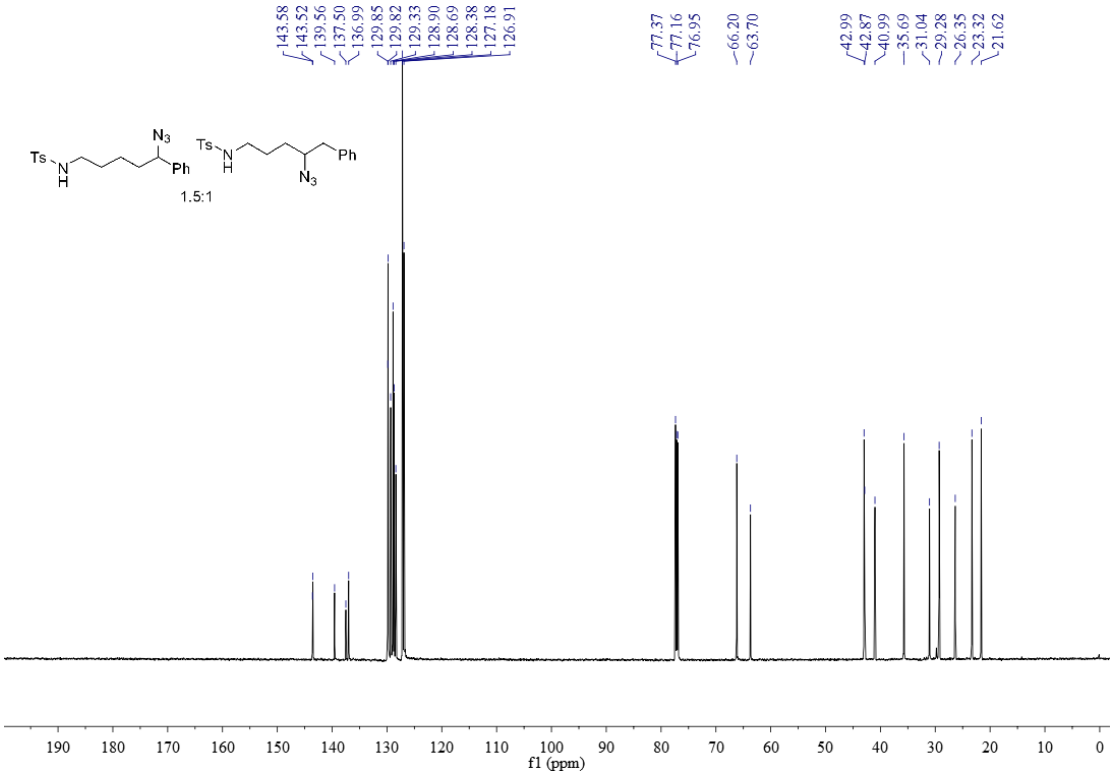
^{13}C NMR of 2o: 150 MHz, $CDCl_3$



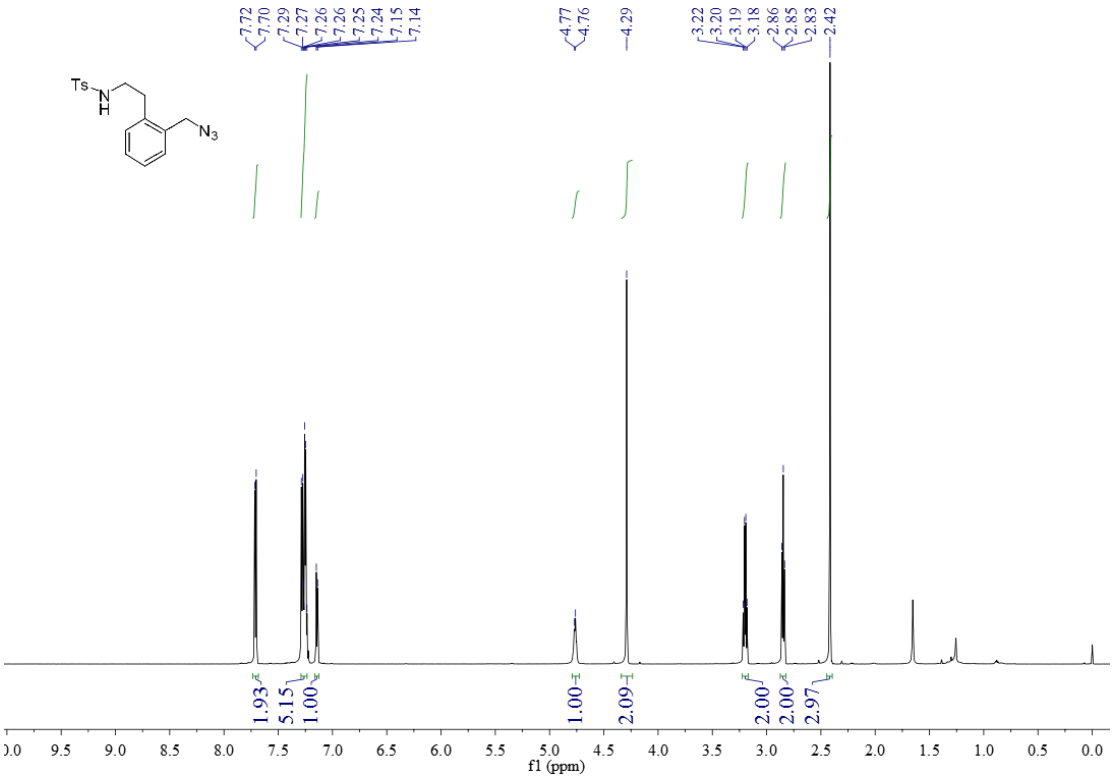
^{1}H NMR of 2p: 600 MHz, $CDCl_3$



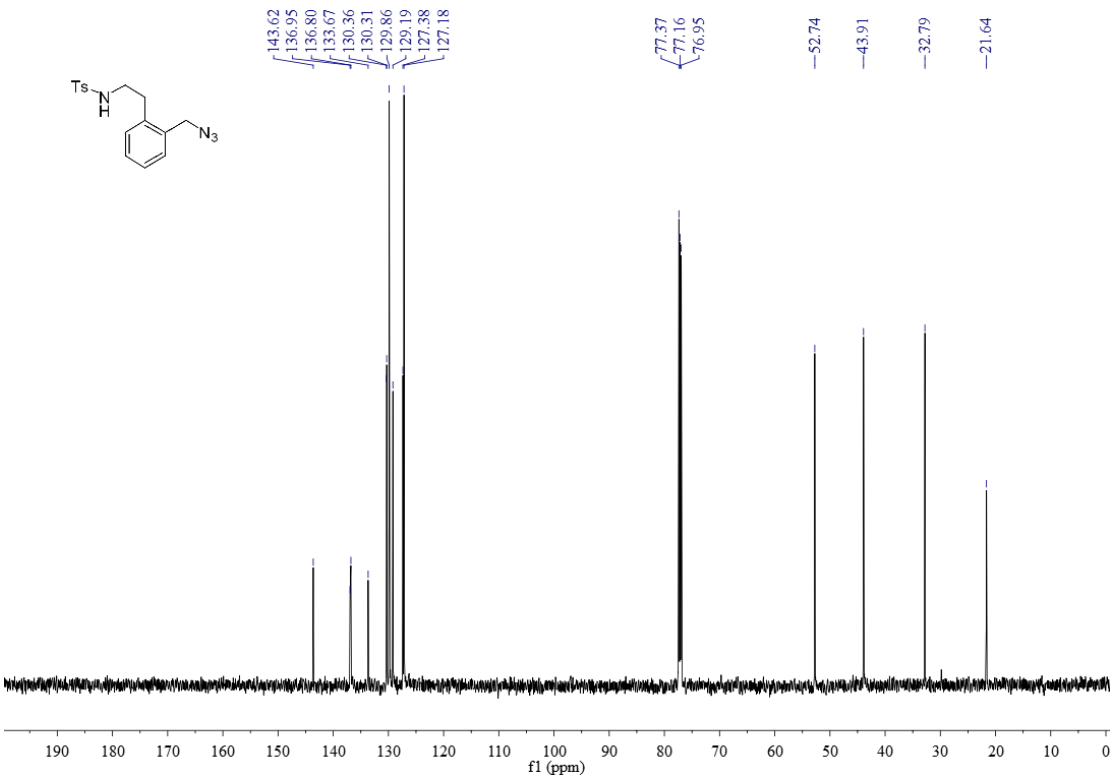
^{13}C NMR of 2p: 150 MHz, $CDCl_3$



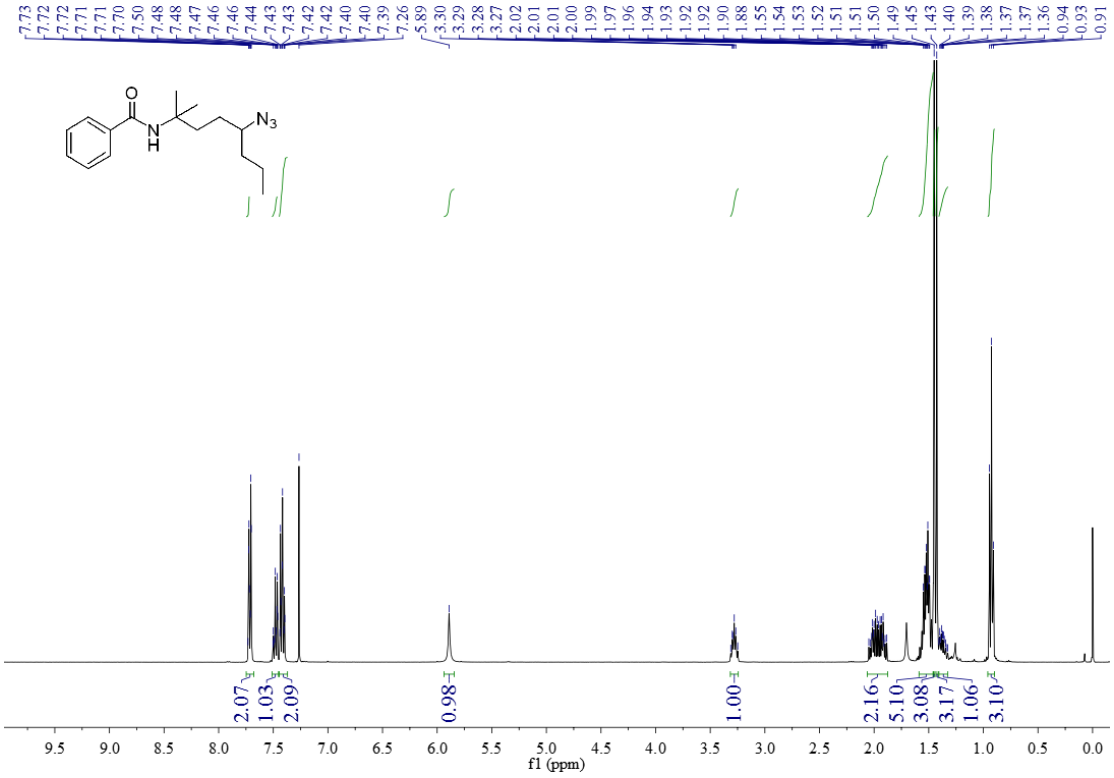
^{1}H NMR of 2q: 600 MHz, $CDCl_3$



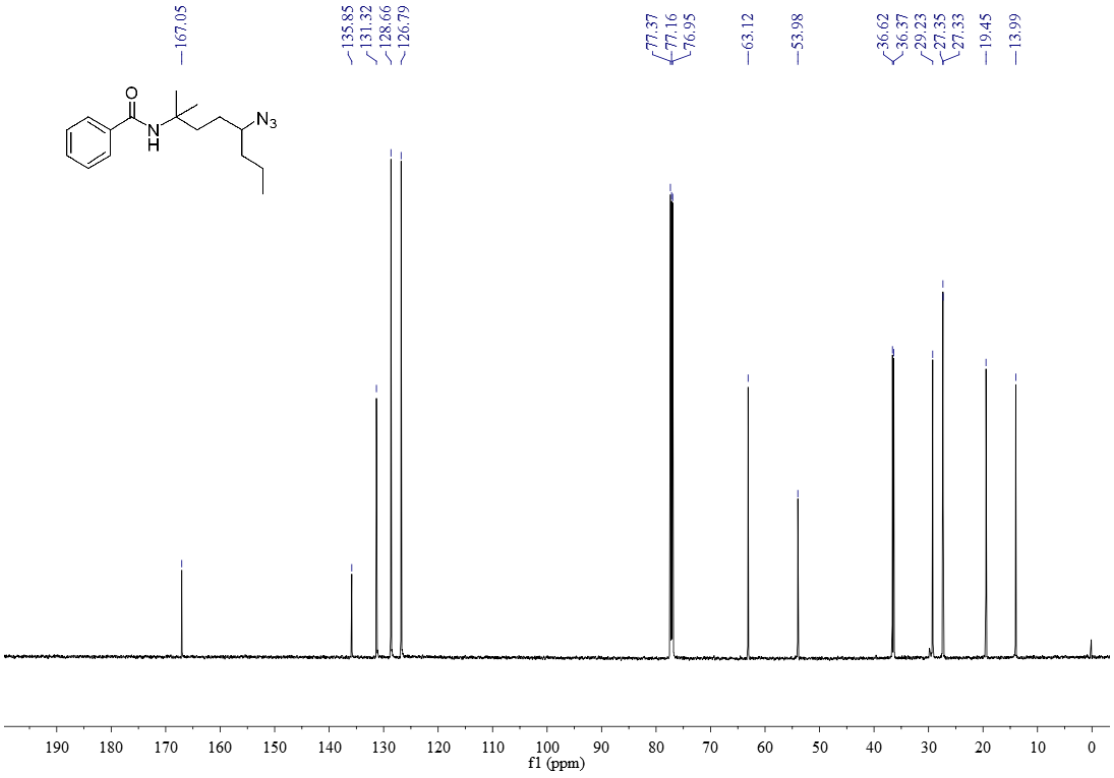
^{13}C NMR of 2q: 150 MHz, $CDCl_3$



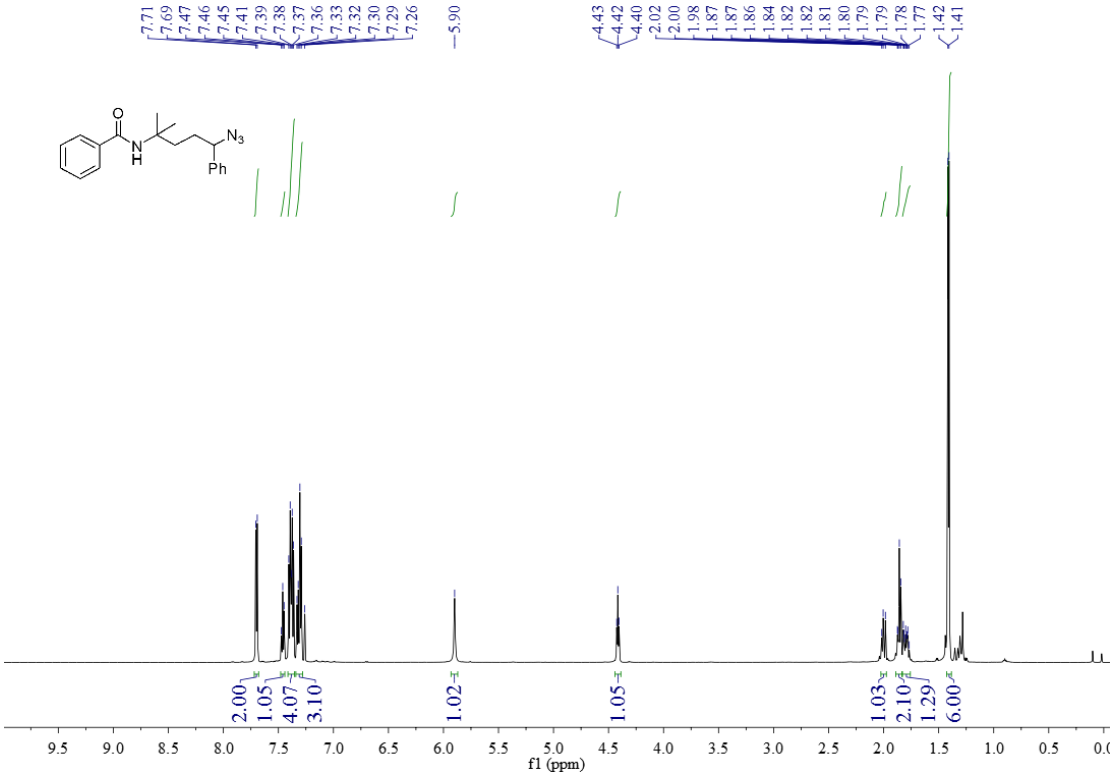
^{1}H NMR of 3a: 400 MHz, $CDCl_3$



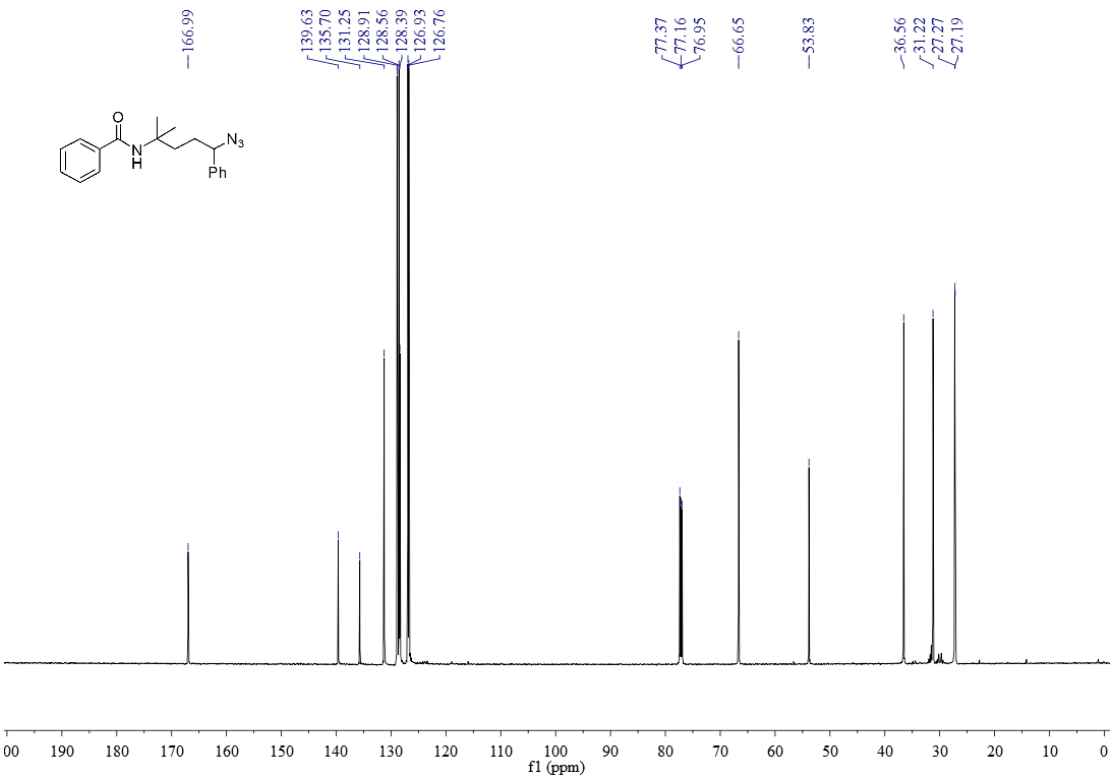
^{13}C NMR of 3a: 150 MHz, $CDCl_3$



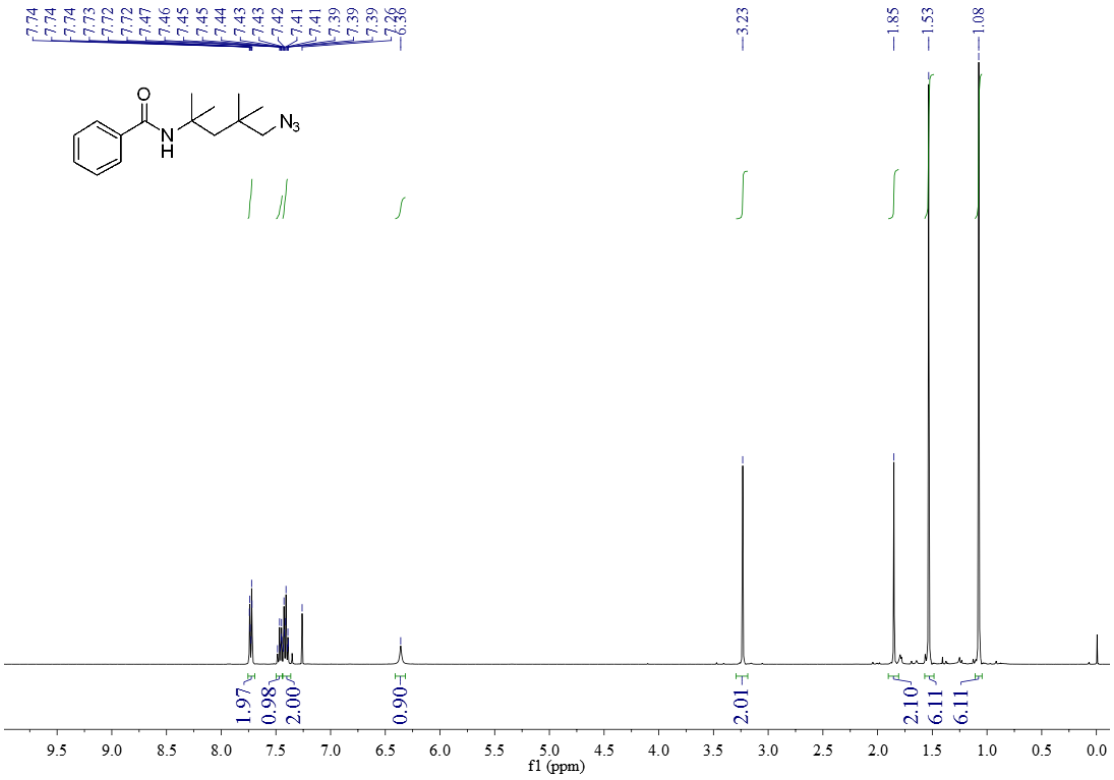
^{1}H NMR of 3b: 600 MHz, $CDCl_3$



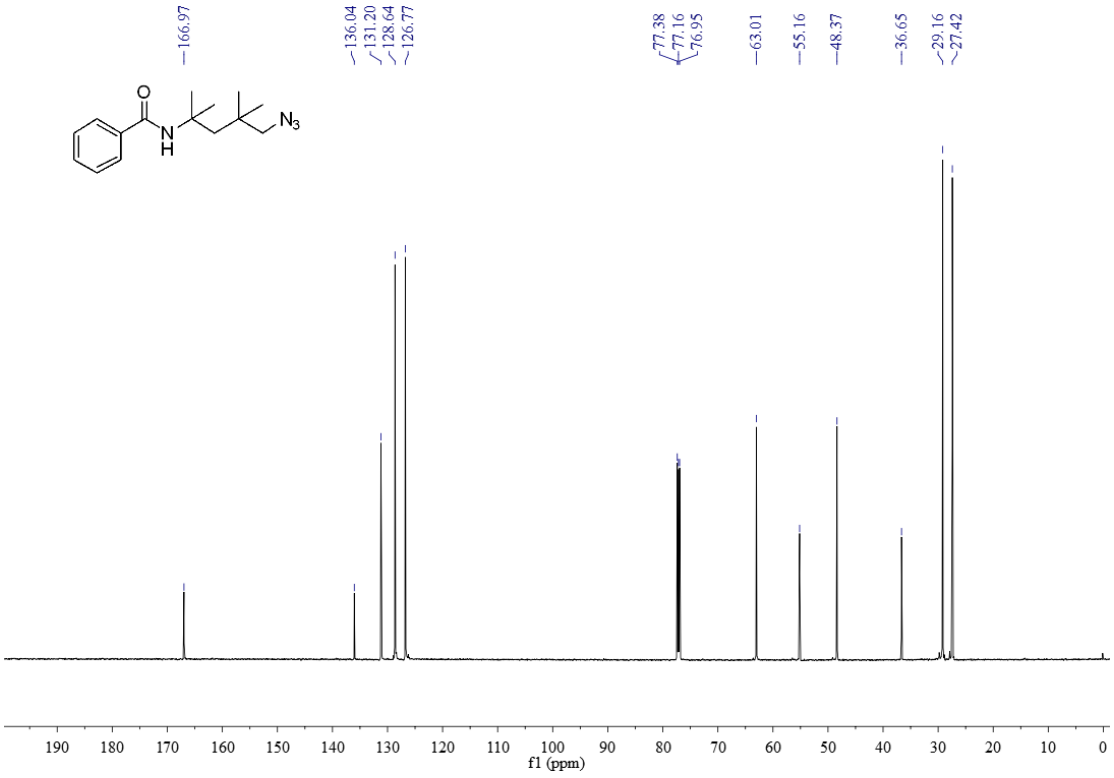
^{13}C NMR of 3b: 150 MHz, $CDCl_3$



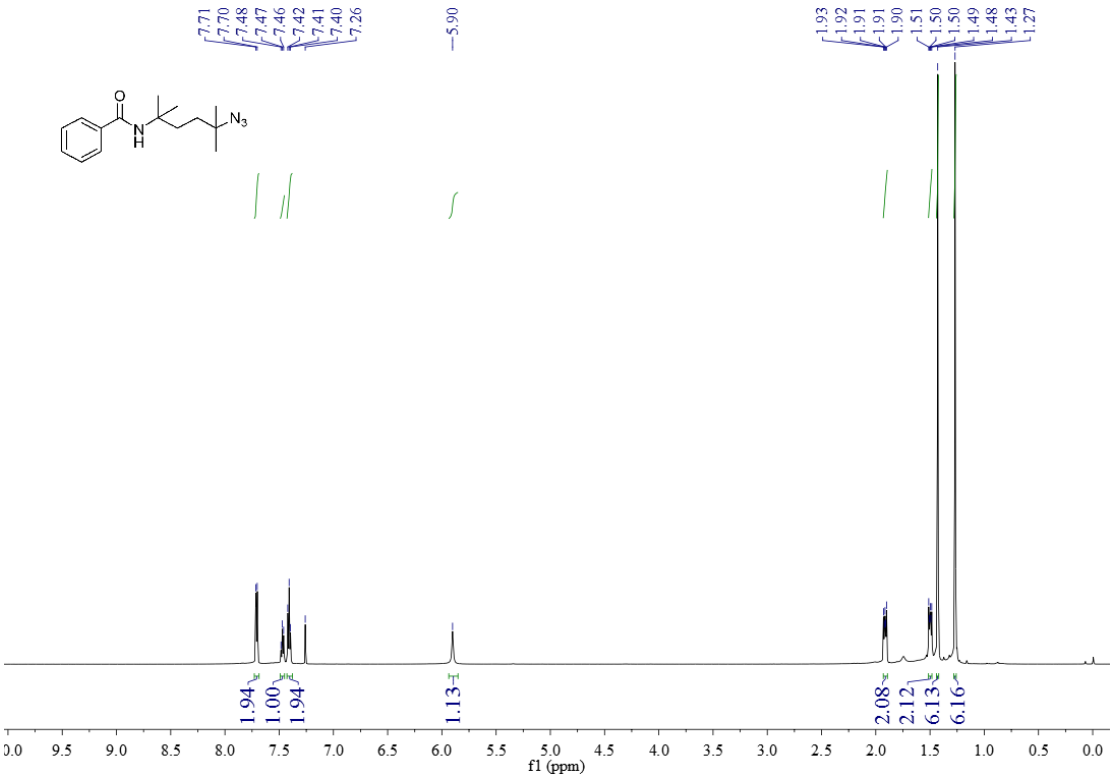
^{1}H NMR of 3c: 400 MHz, $CDCl_3$



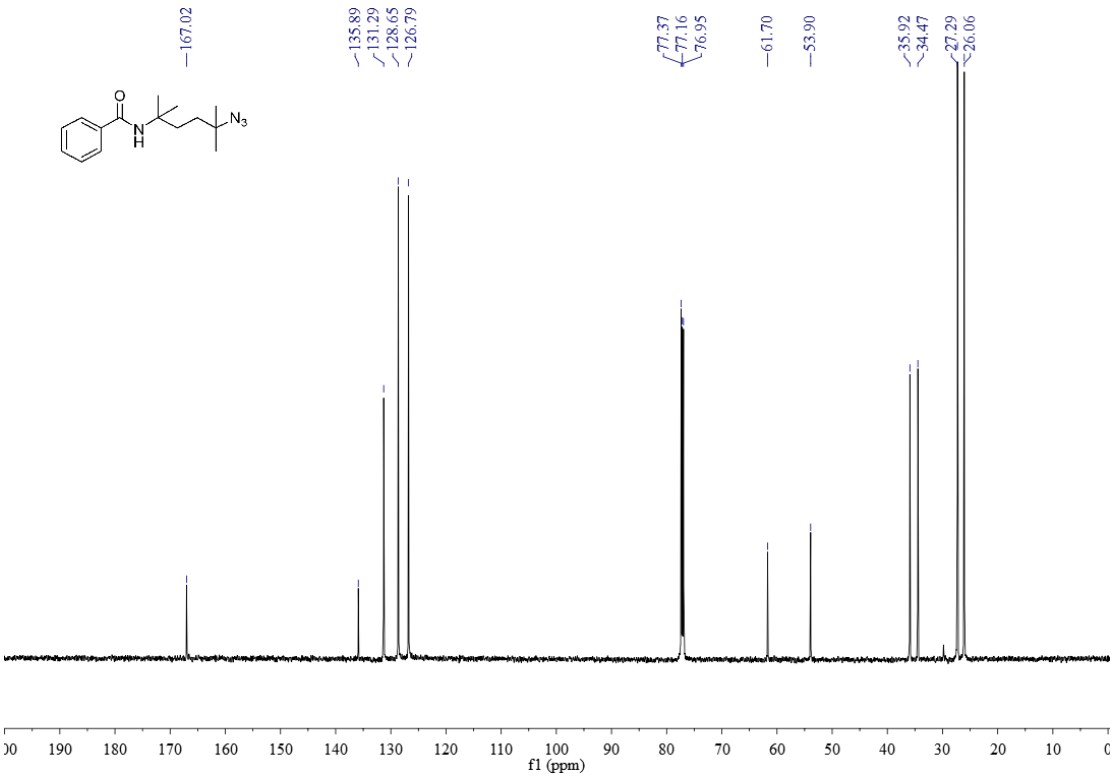
^{13}C NMR of 3c: 150 MHz, $CDCl_3$



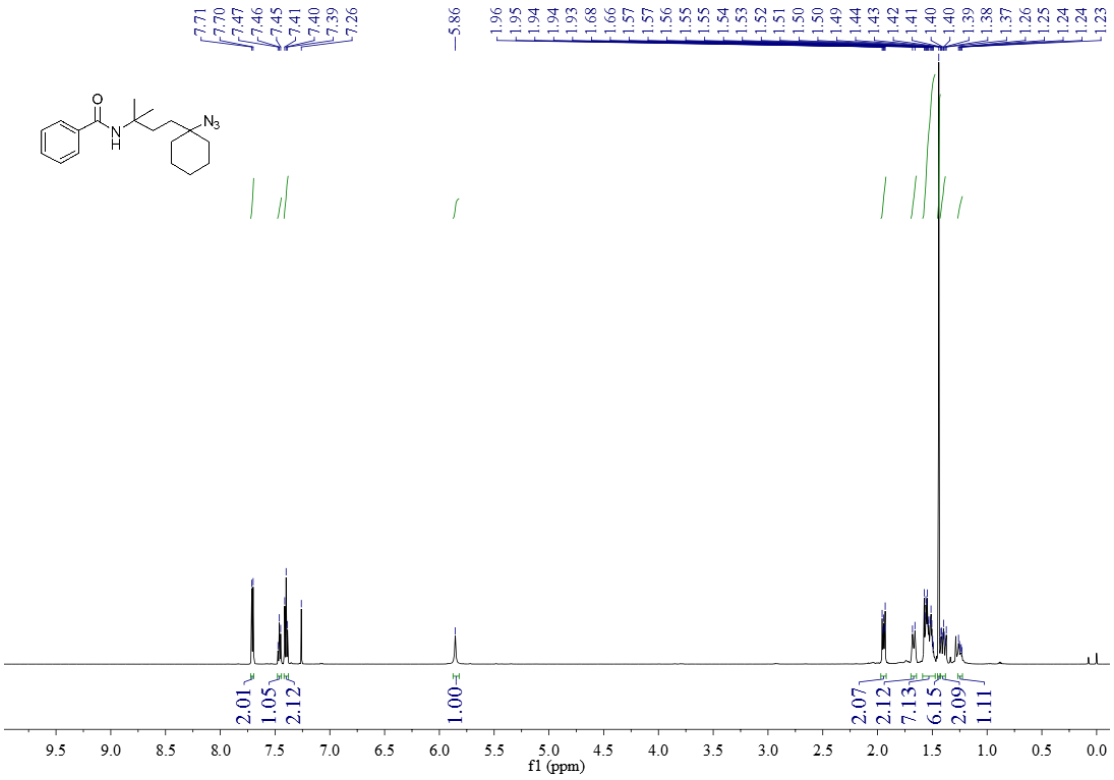
^{1}H NMR of 3d: 600 MHz, $CDCl_3$



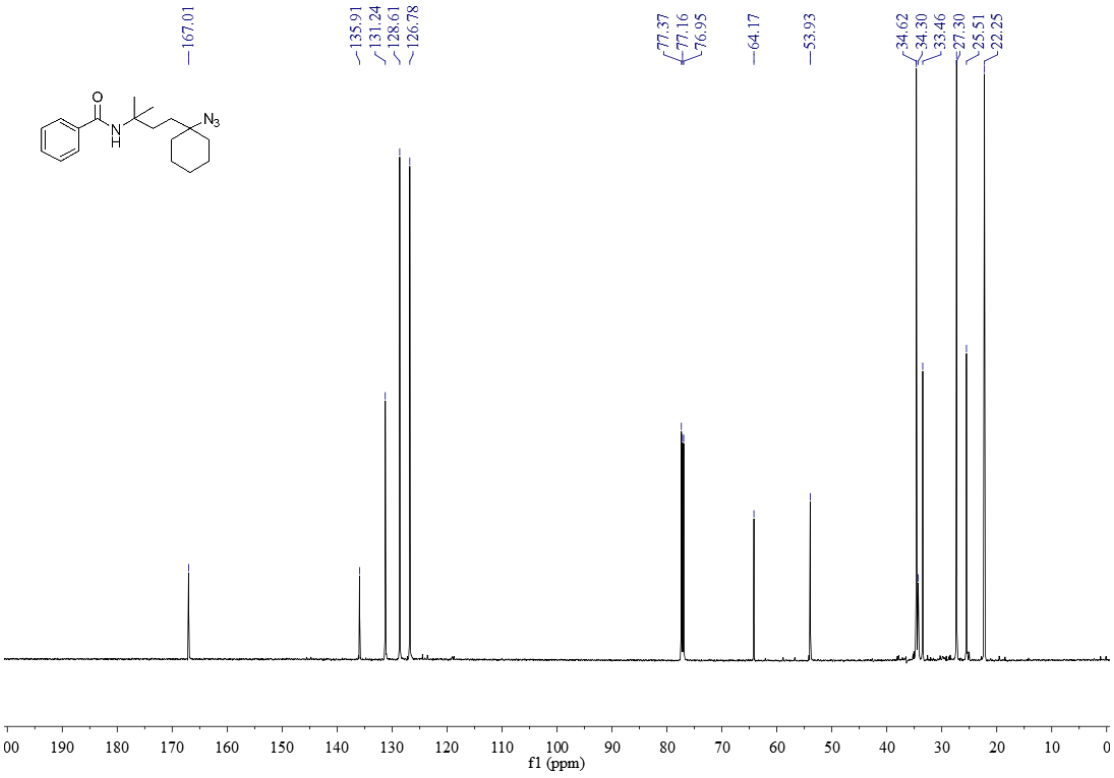
^{13}C NMR of 3d: 150 MHz, $CDCl_3$



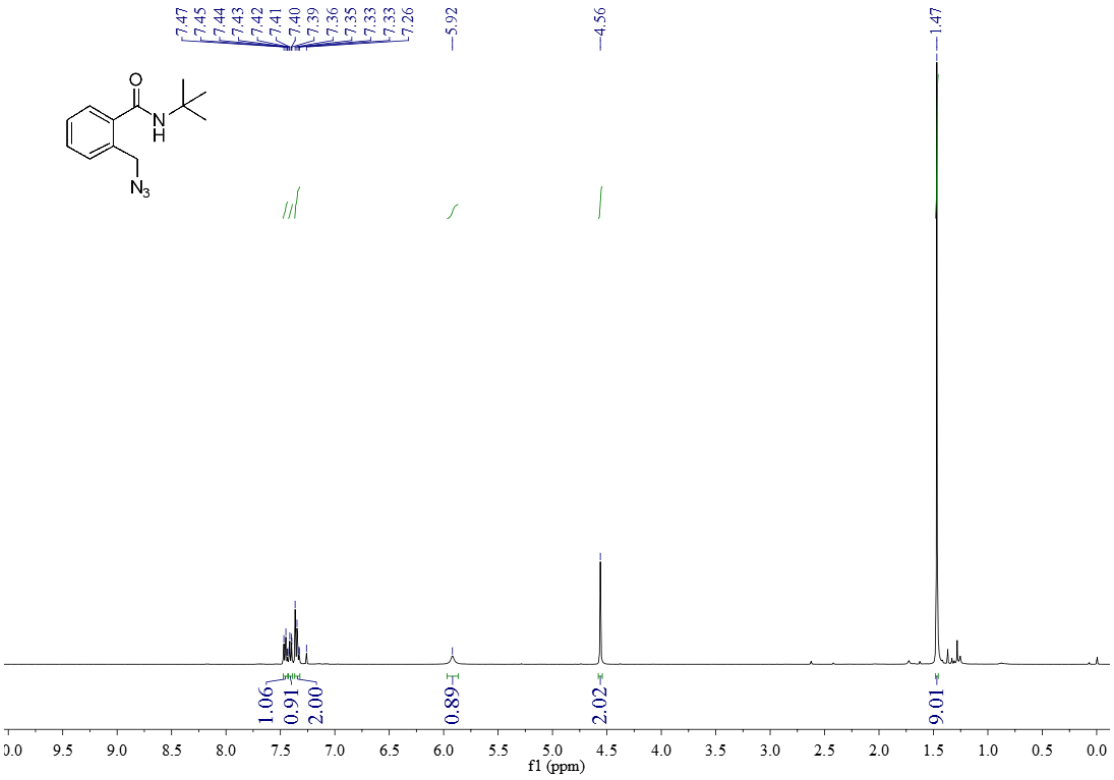
^{1}H NMR of 3e: 600 MHz, $CDCl_3$



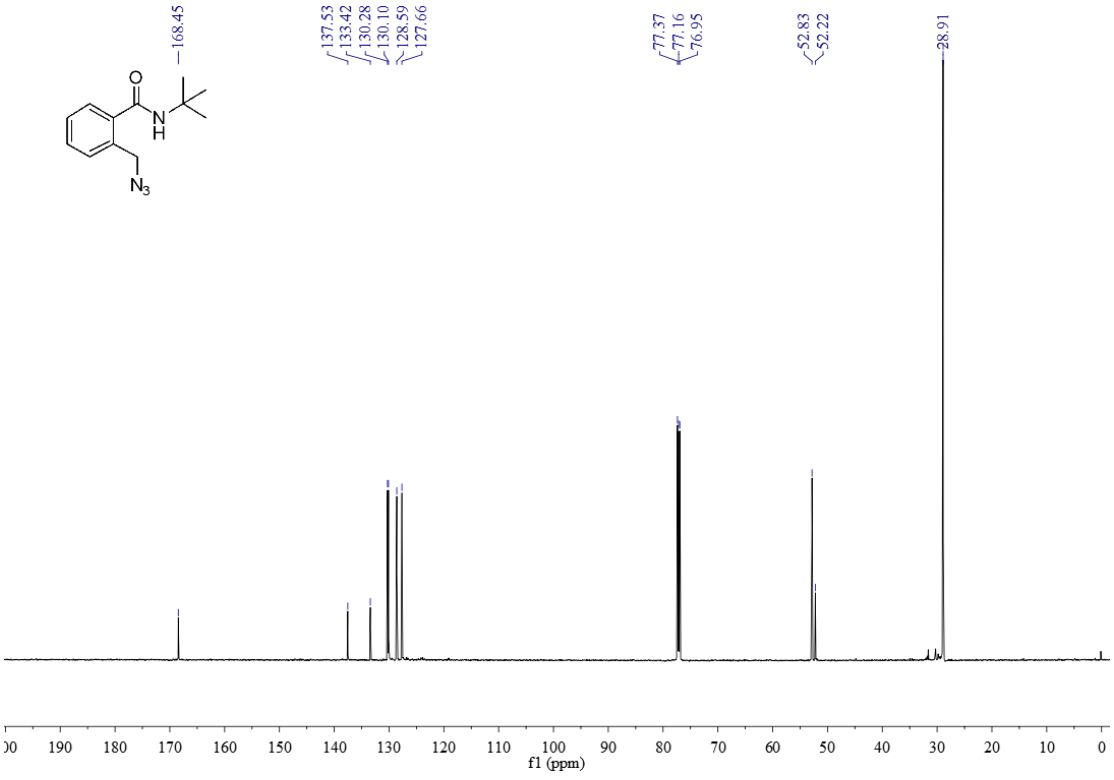
^{13}C NMR of 3e: 150 MHz, $CDCl_3$



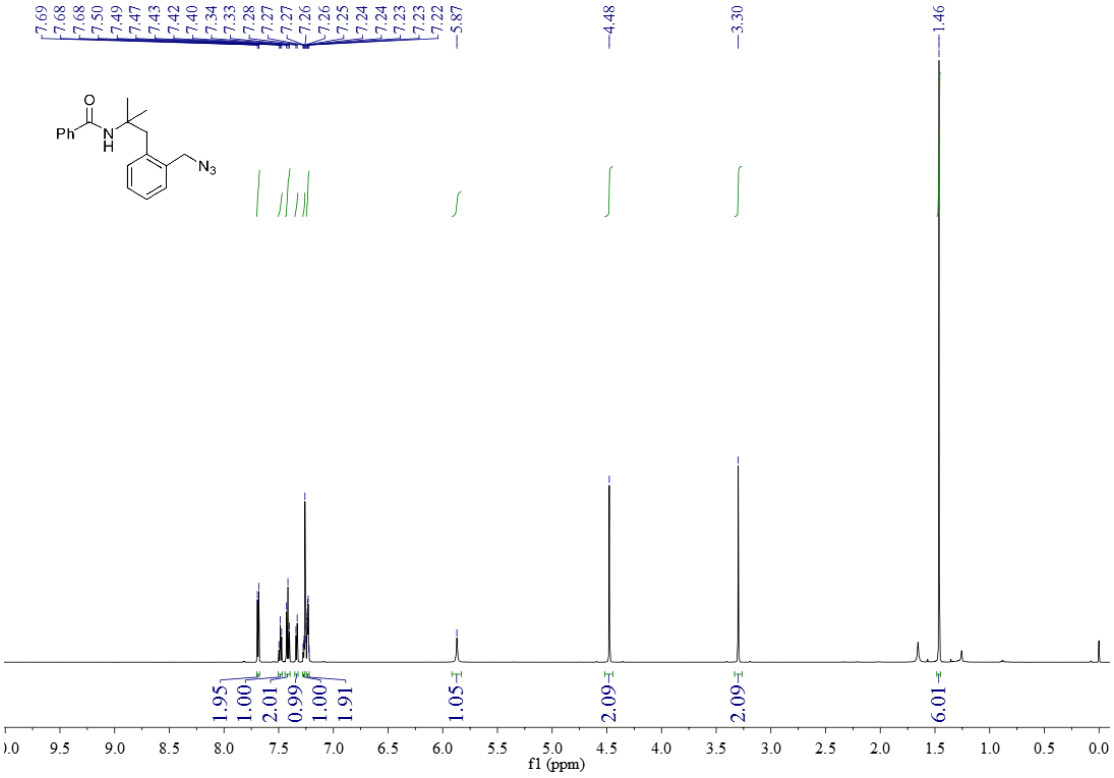
^{1}H NMR of 3f: 400 MHz, $CDCl_3$



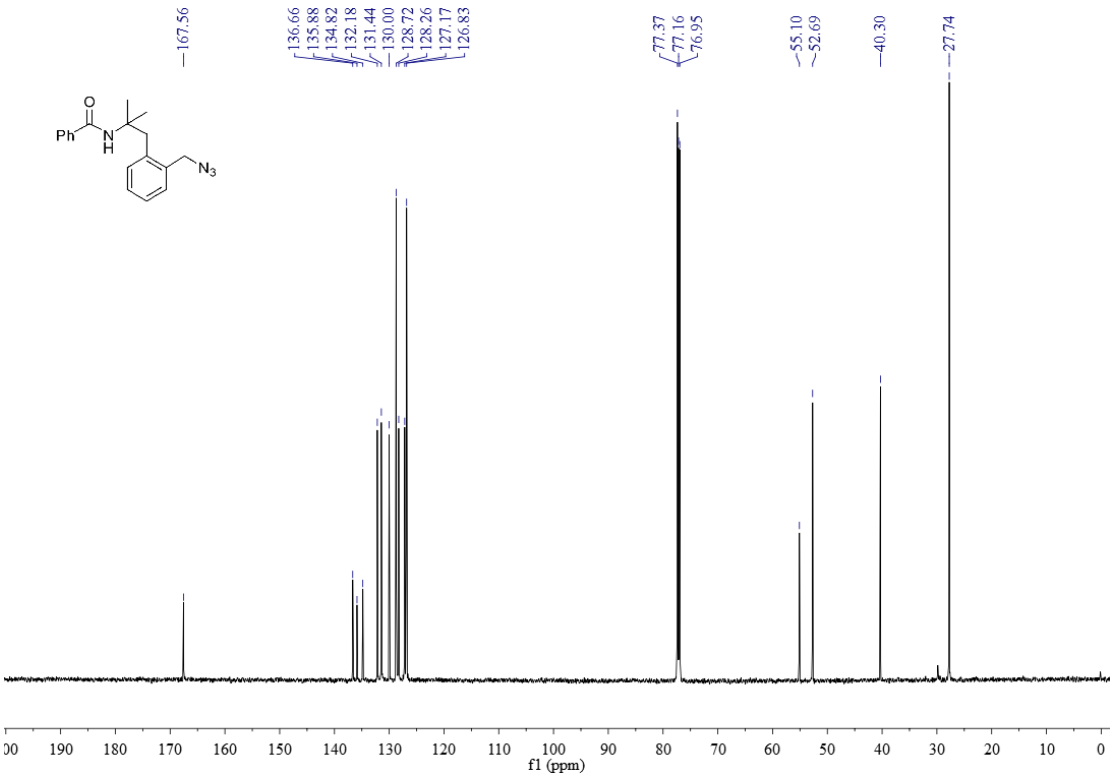
^{13}C NMR of 3f: 150 MHz, $CDCl_3$



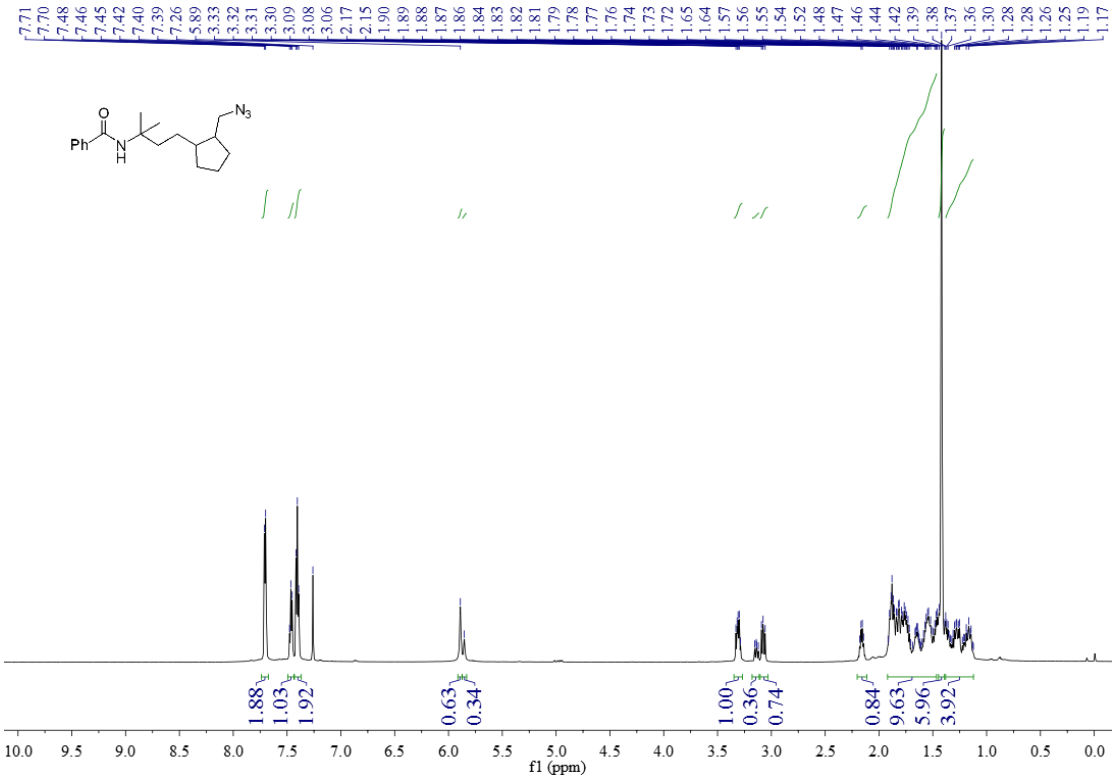
^{1}H NMR of 3g: 600 MHz, $CDCl_3$



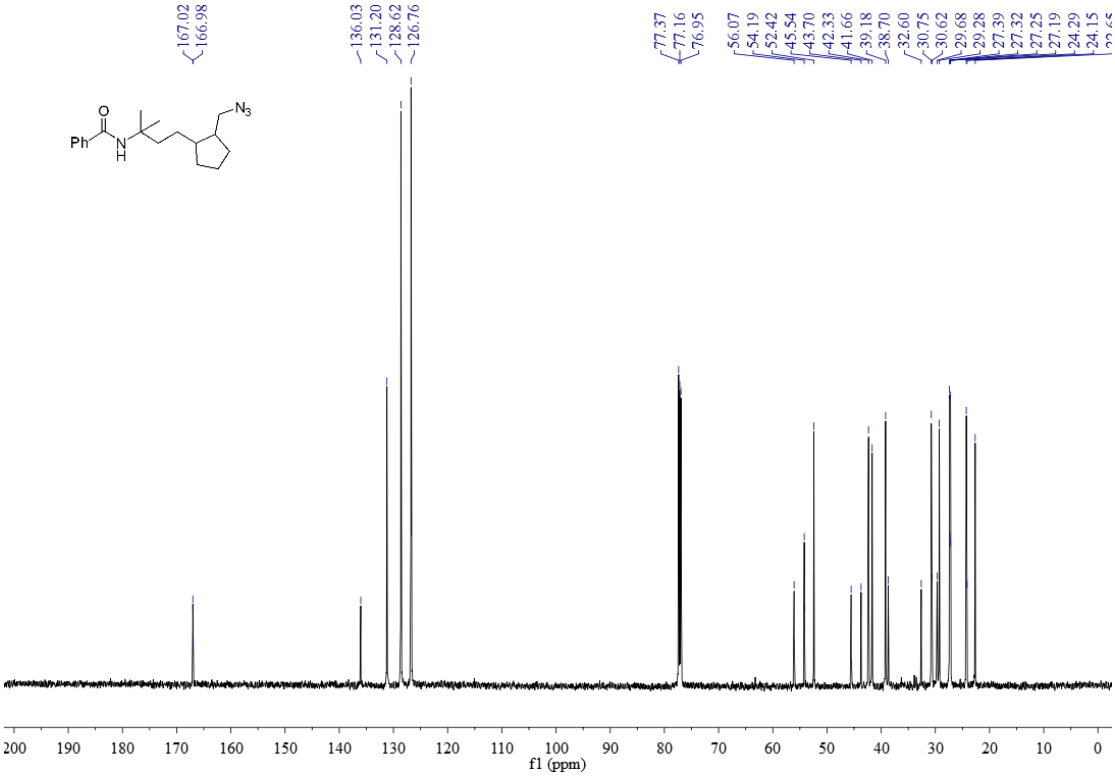
^{13}C NMR of 3g: 150 MHz, $CDCl_3$



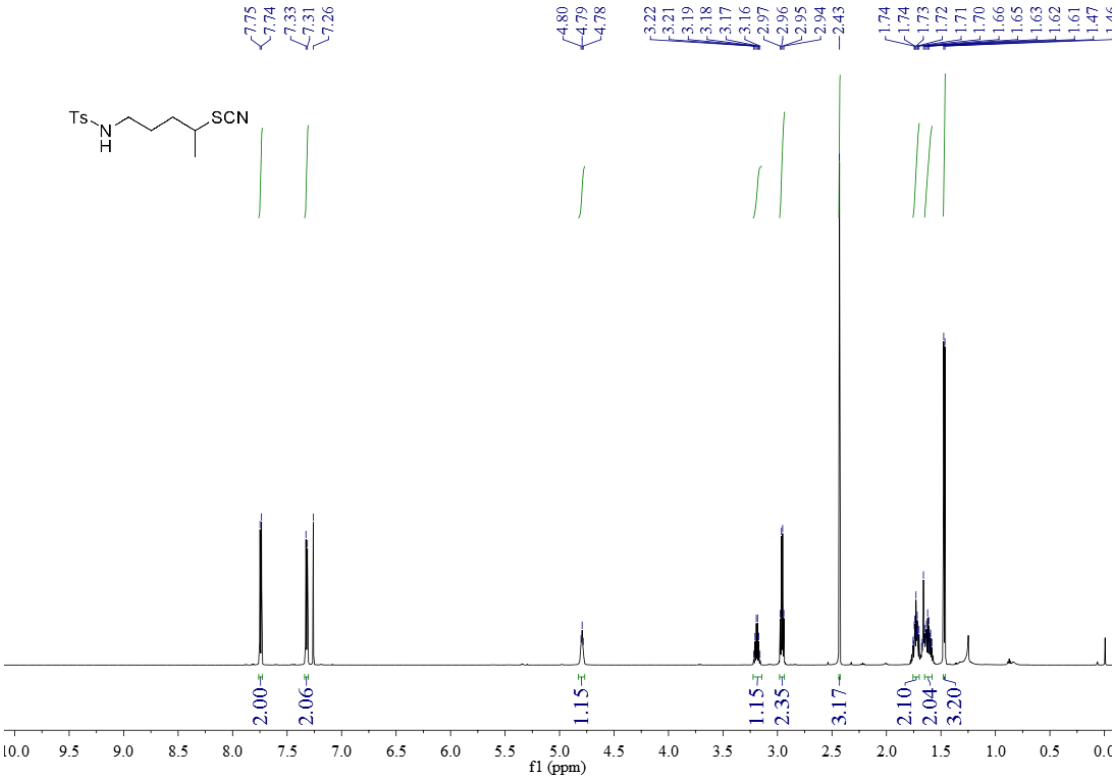
^{1}H NMR of 4: 600 MHz, $CDCl_3$



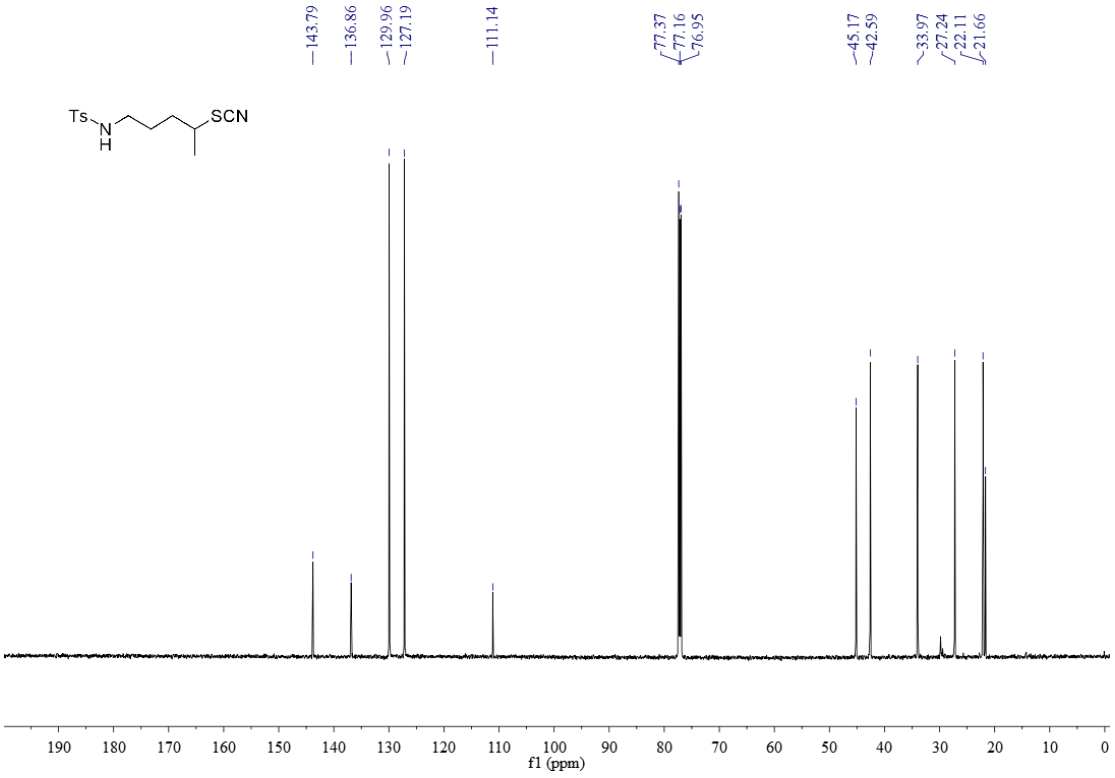
^{13}C NMR of 4: 150 MHz, $CDCl_3$



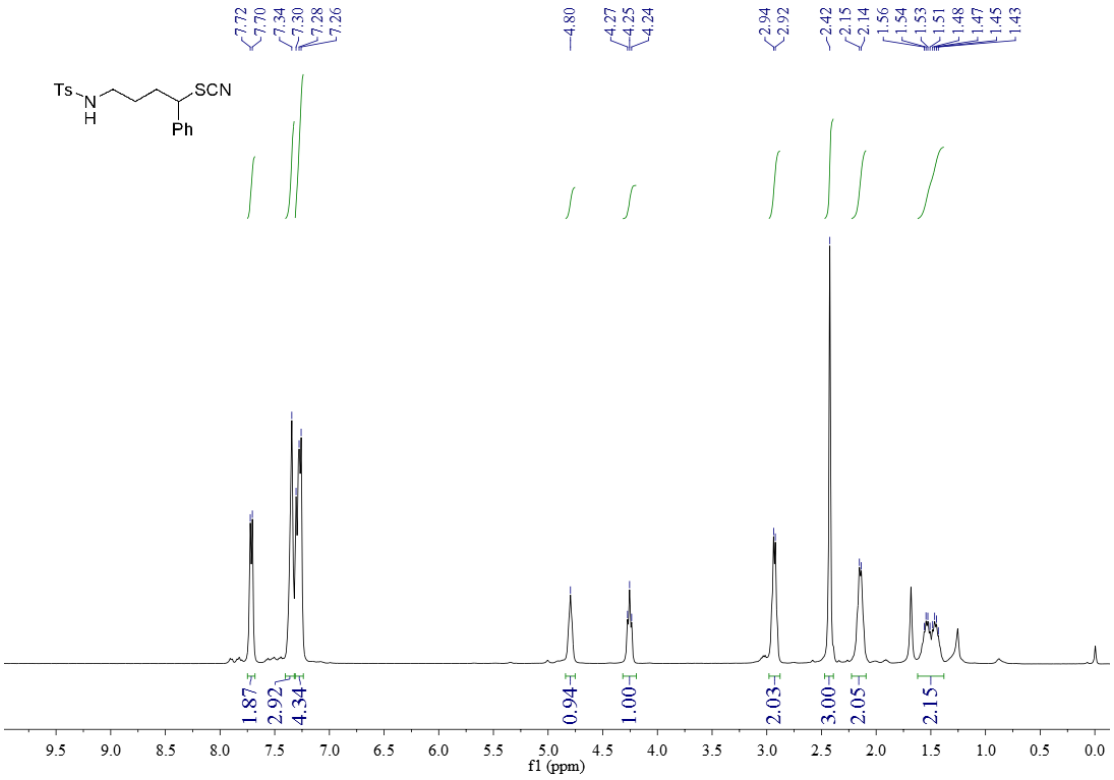
^{1}H NMR of 5a: 600 MHz, $CDCl_3$



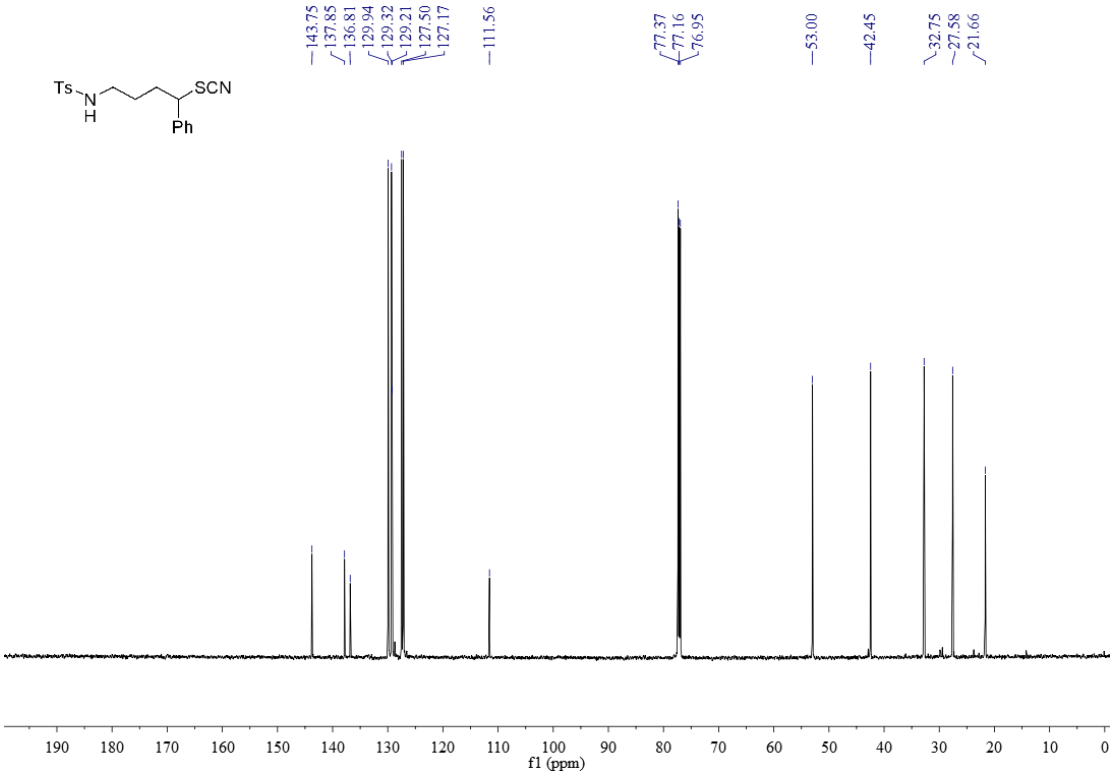
^{13}C NMR of 5a: 150 MHz, $CDCl_3$



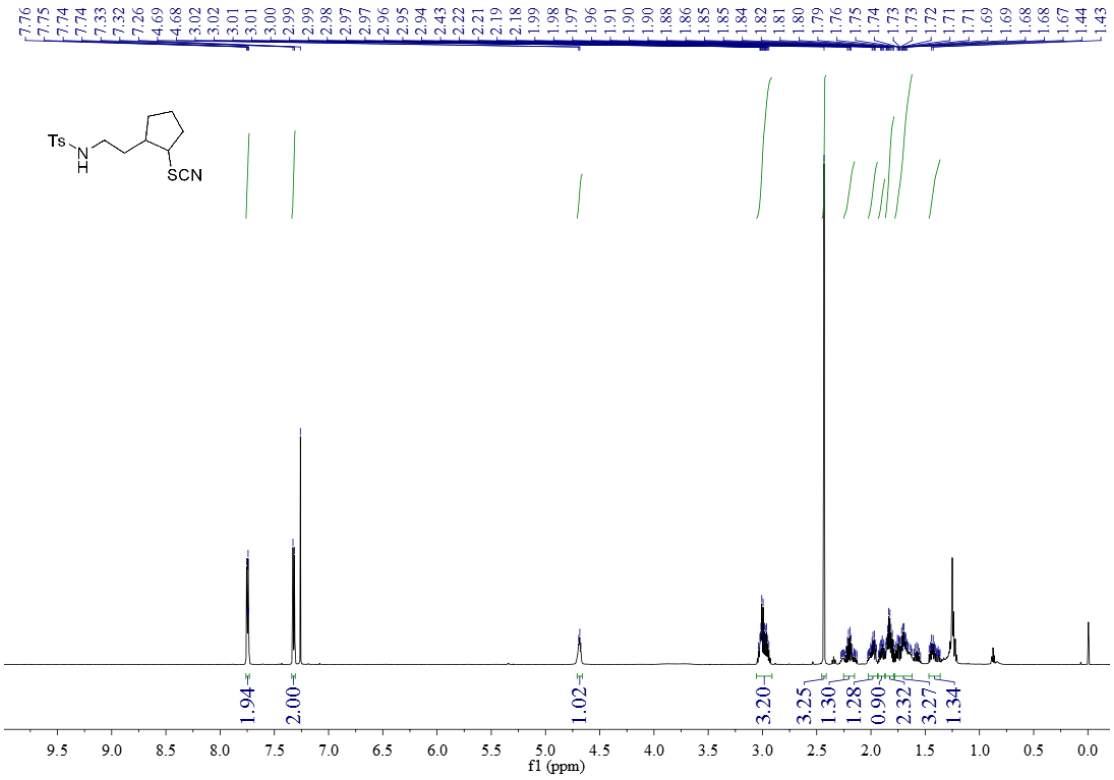
^{1}H NMR of 5b: 400 MHz, $CDCl_3$



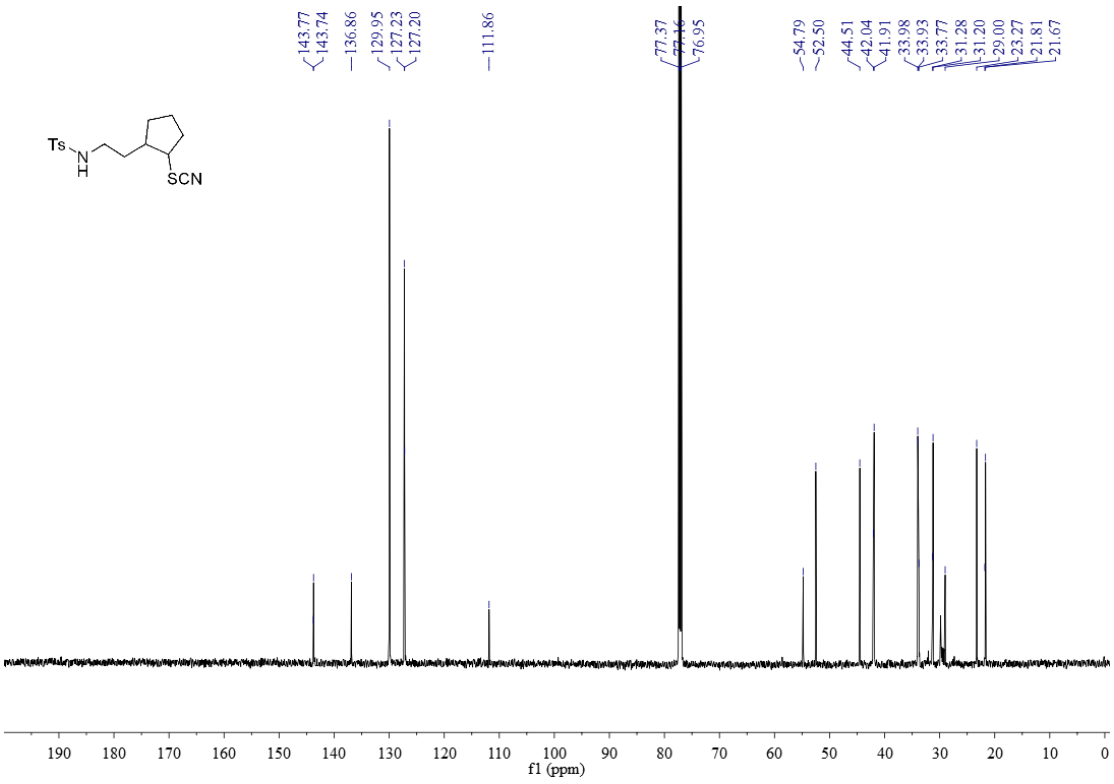
^{13}C NMR of 5b: 150 MHz, $CDCl_3$



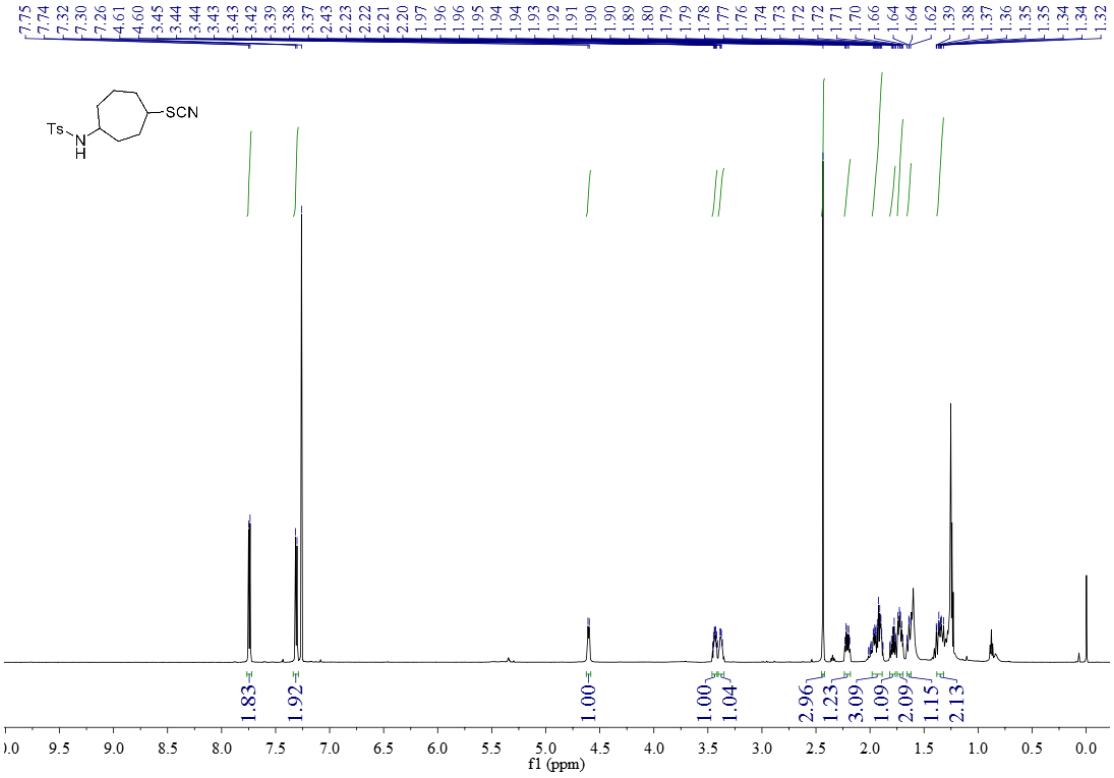
^{1}H NMR of 5c: 600 MHz, $CDCl_3$



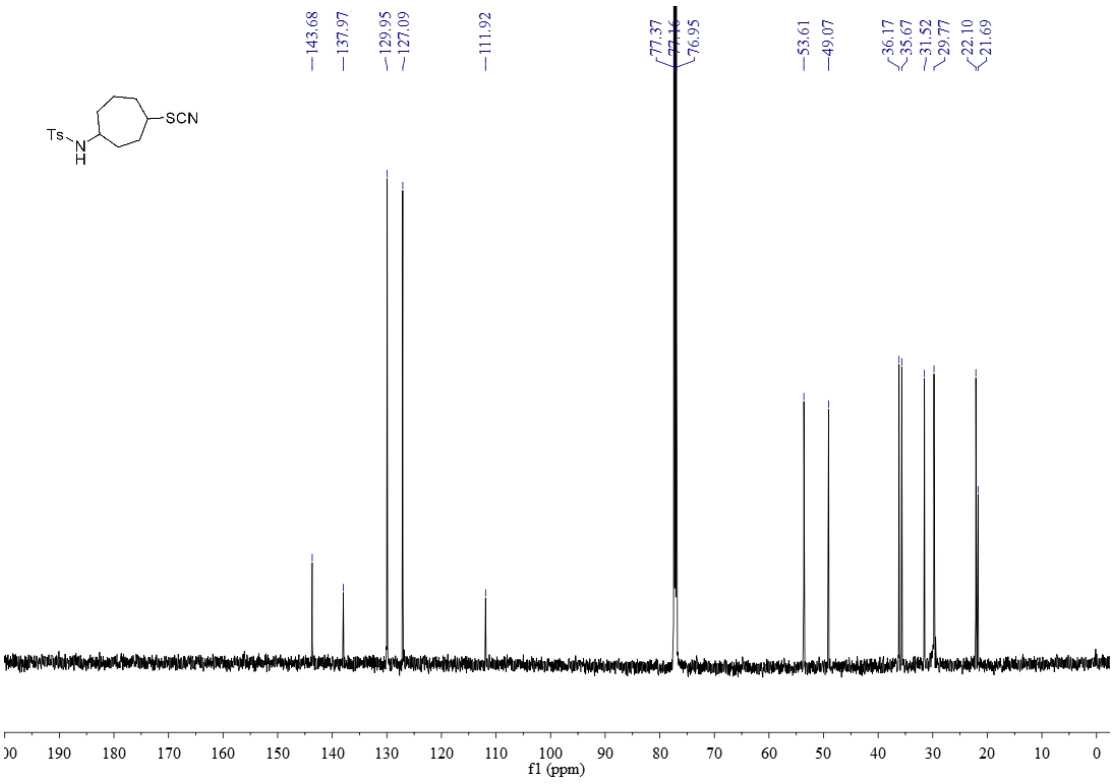
^{13}C NMR of 5c: 150 MHz, $CDCl_3$



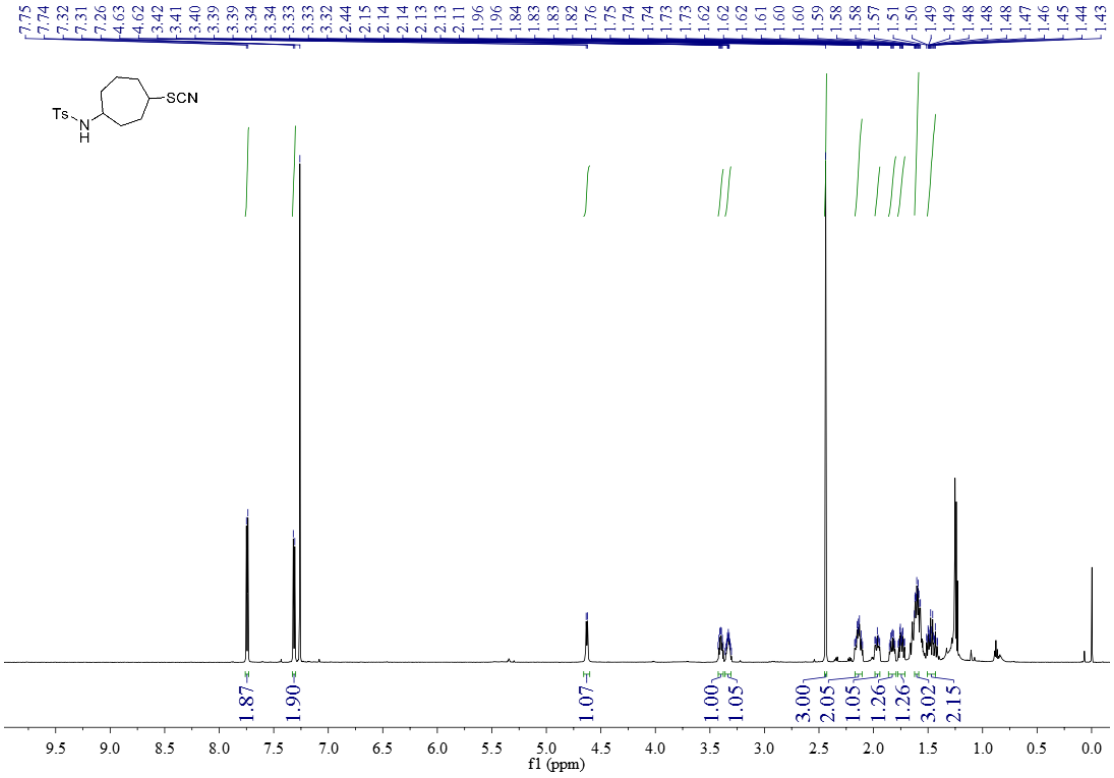
^{1}H NMR of 5d-a: 600 MHz, $CDCl_3$



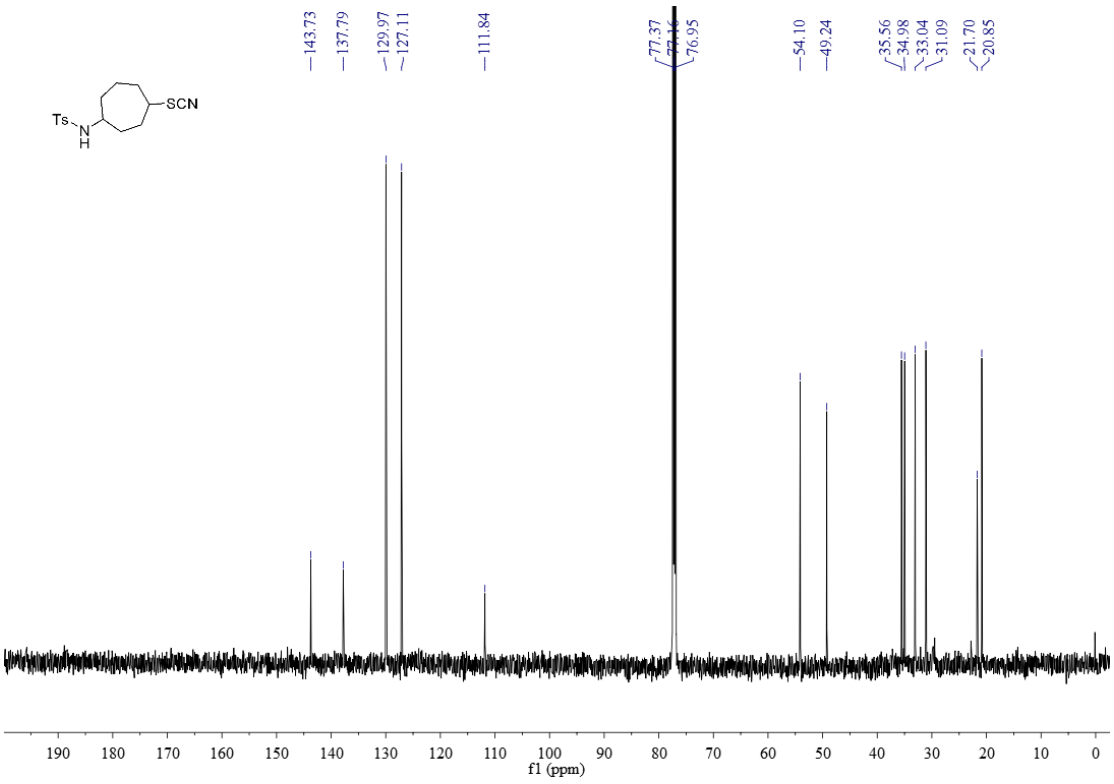
^{13}C NMR of 5d-a: 150 MHz, $CDCl_3$



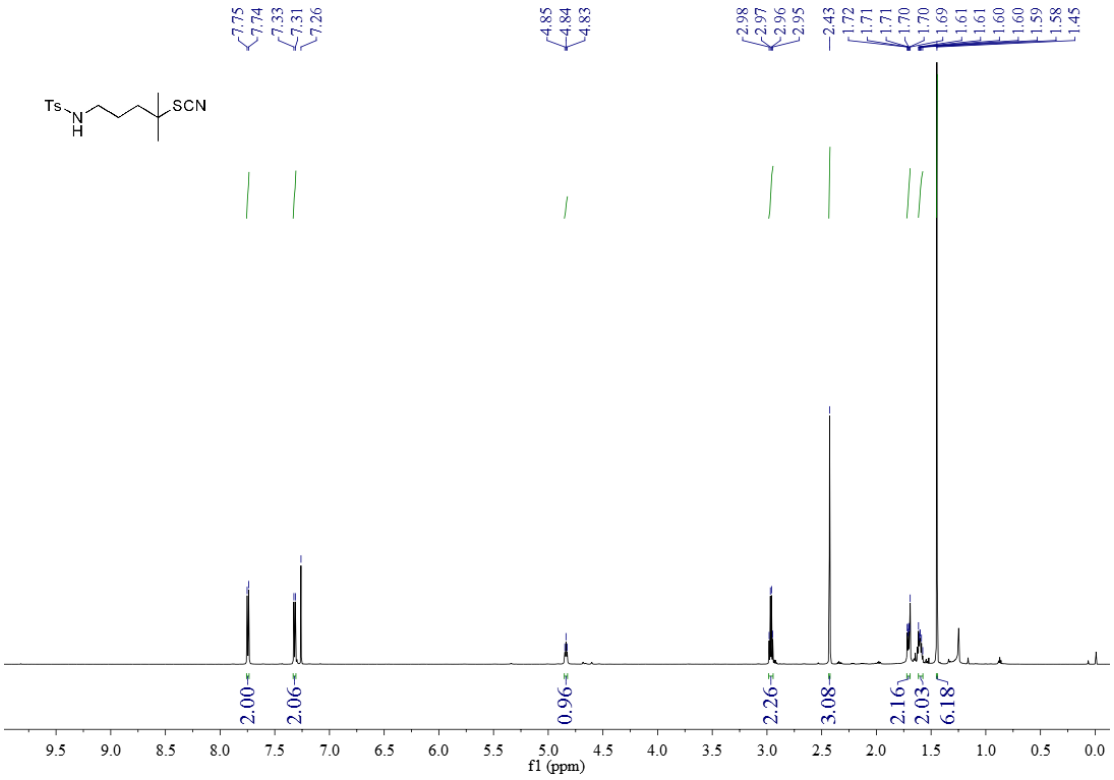
^{1}H NMR of 5d-b: 600 MHz, $CDCl_3$



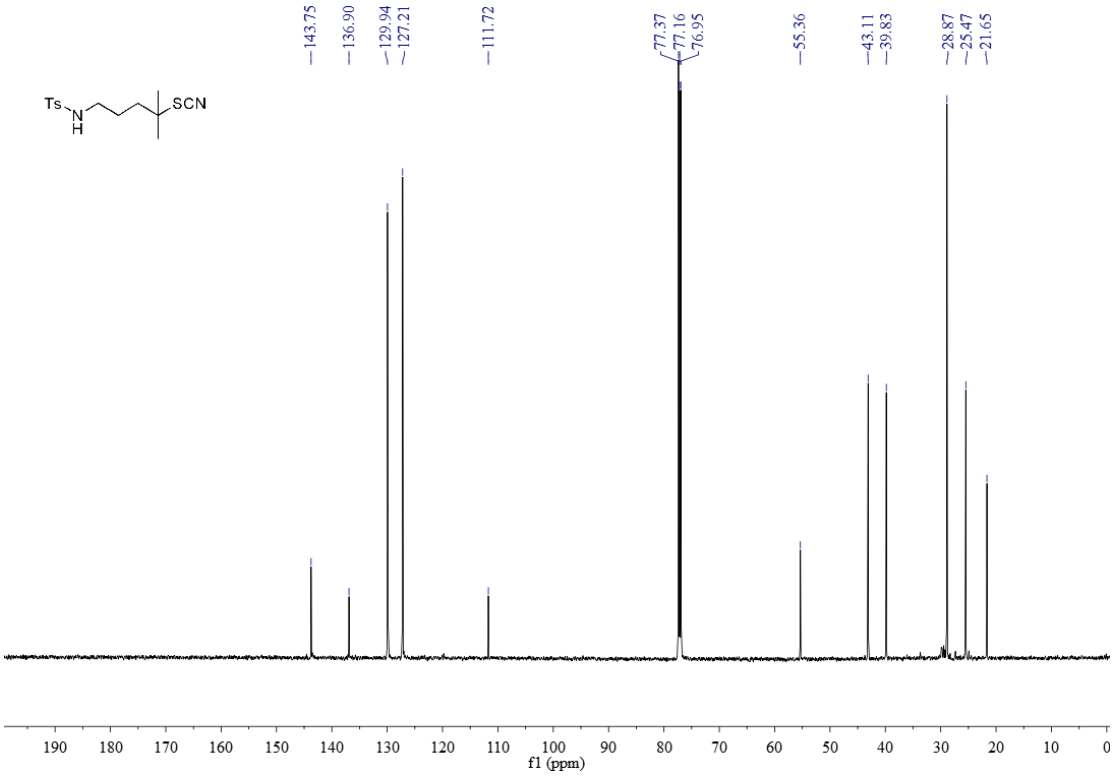
^{13}C NMR of 5d-b: 150 MHz, $CDCl_3$



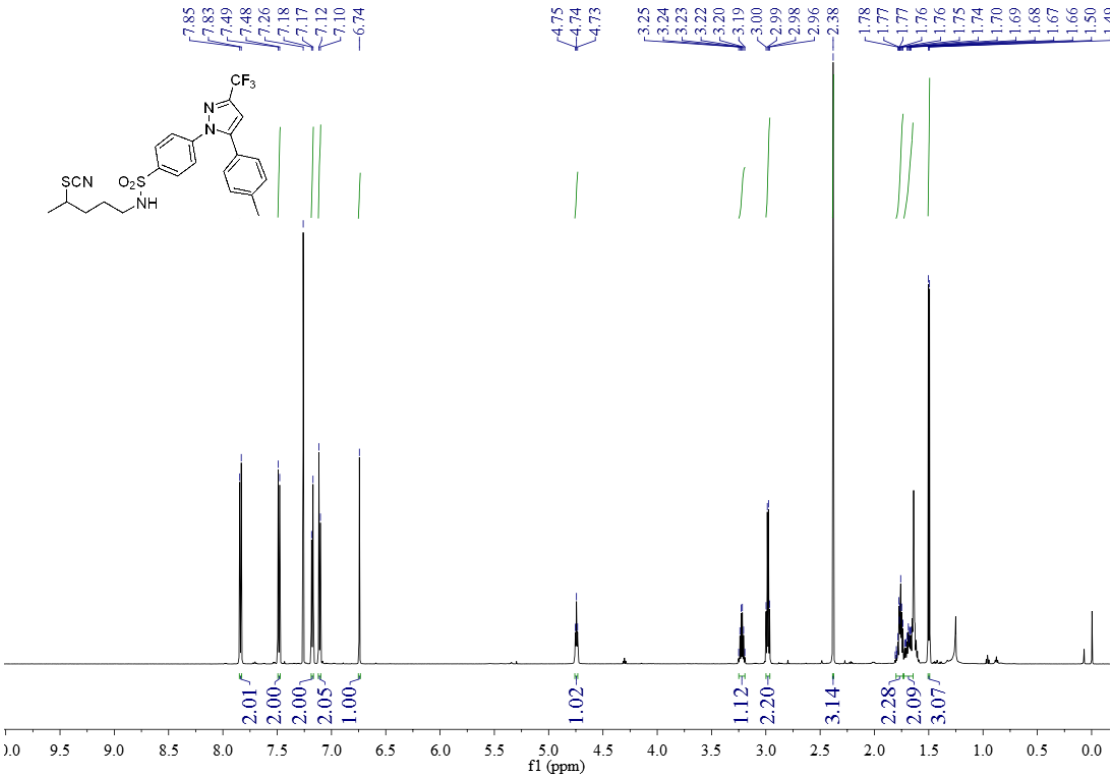
^{1}H NMR of 5e: 600 MHz, $CDCl_3$



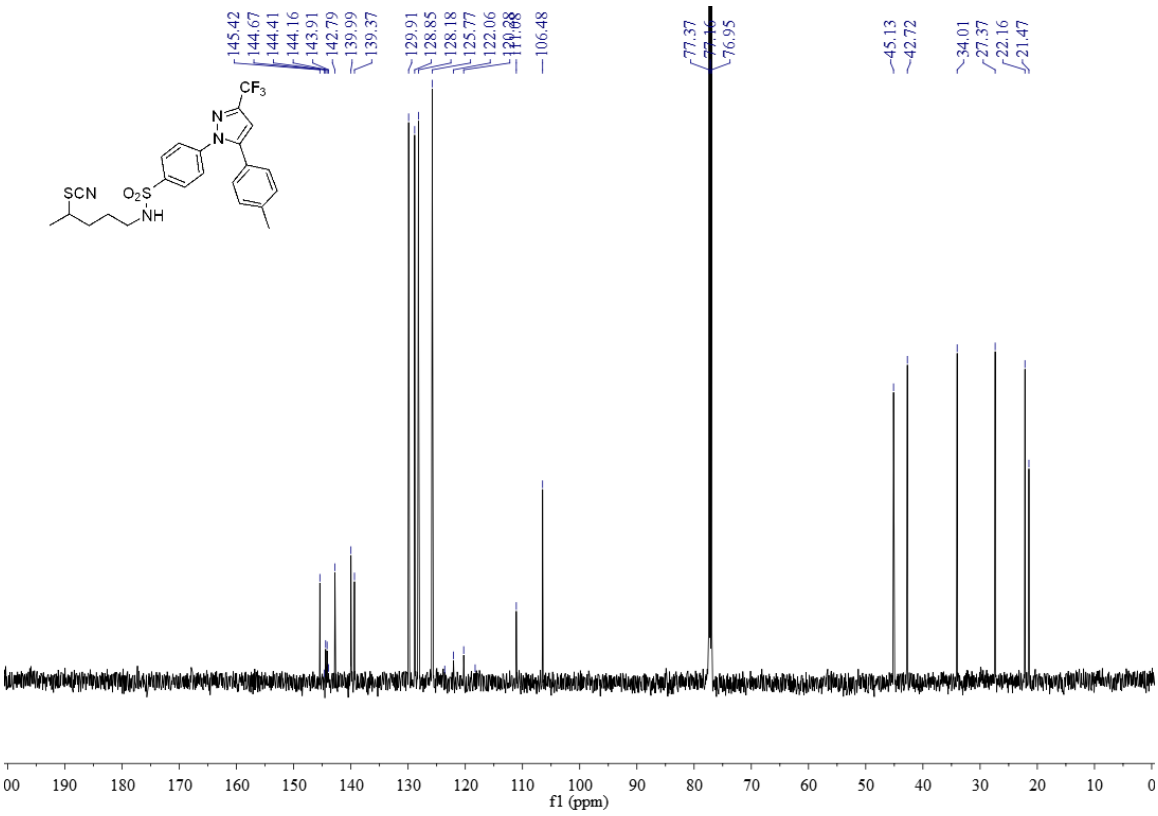
^{13}C NMR of 5e: 150 MHz, $CDCl_3$



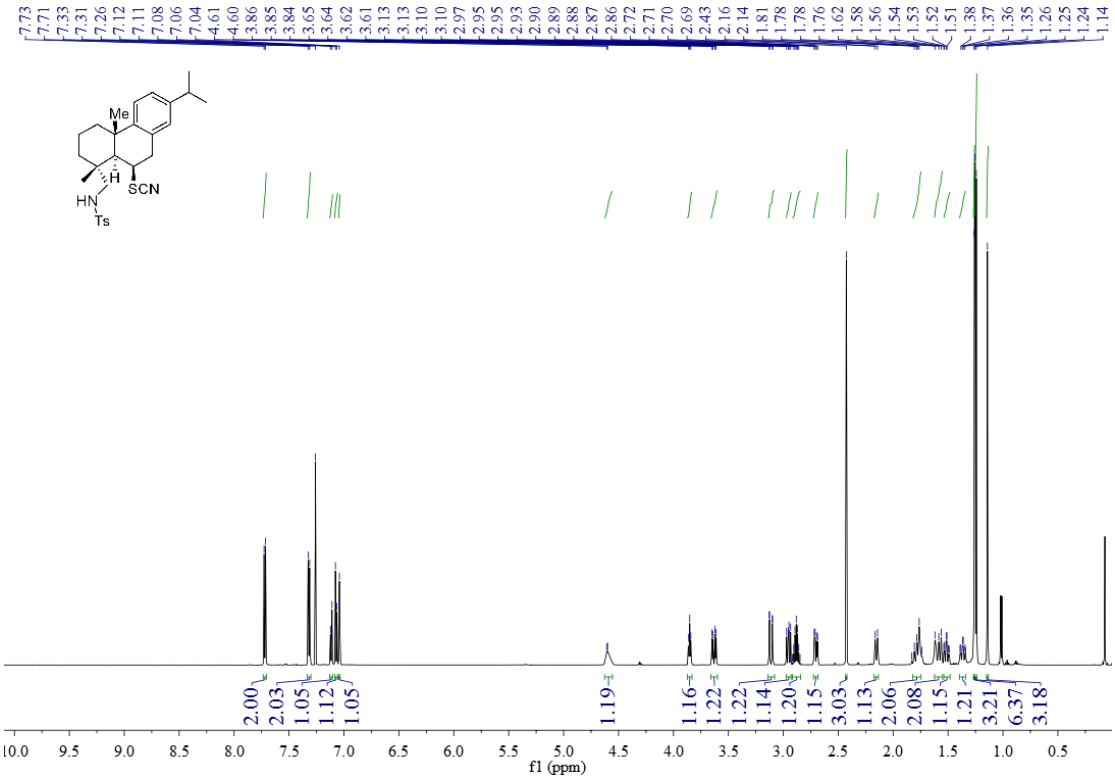
^{1}H NMR of 5f: 600 MHz, $CDCl_3$



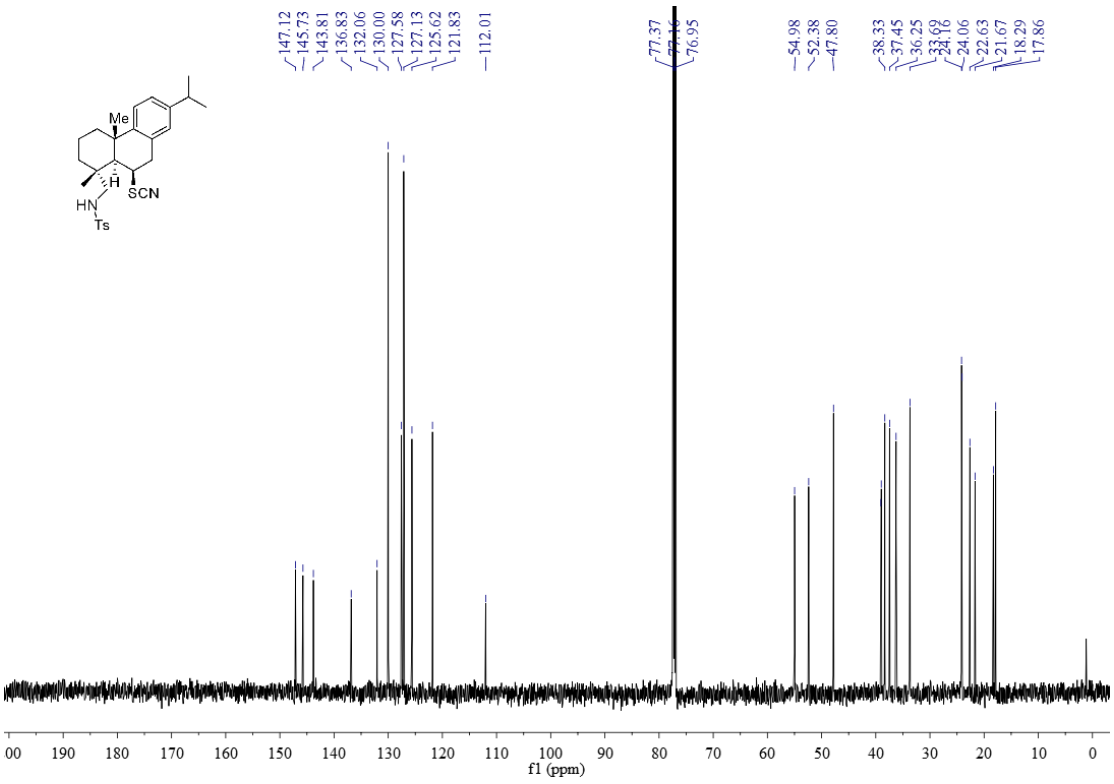
^{13}C NMR of 5f: 150 MHz, $CDCl_3$



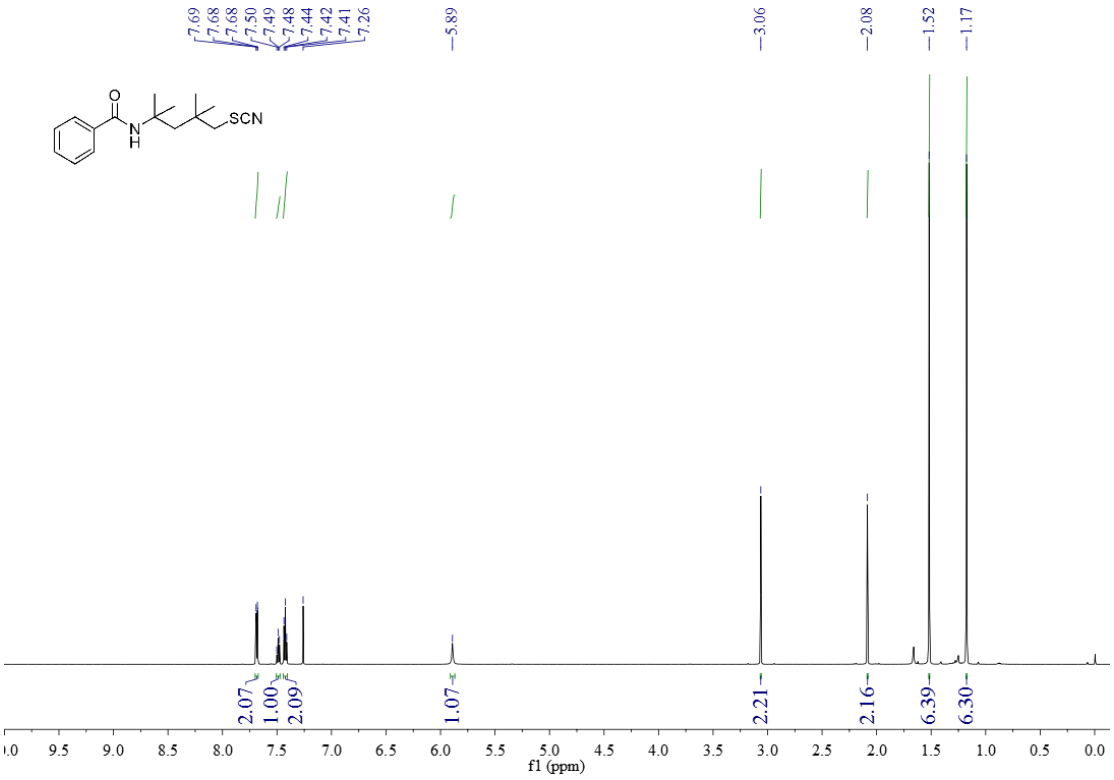
^{1}H NMR of 5g: 600 MHz, $CDCl_3$



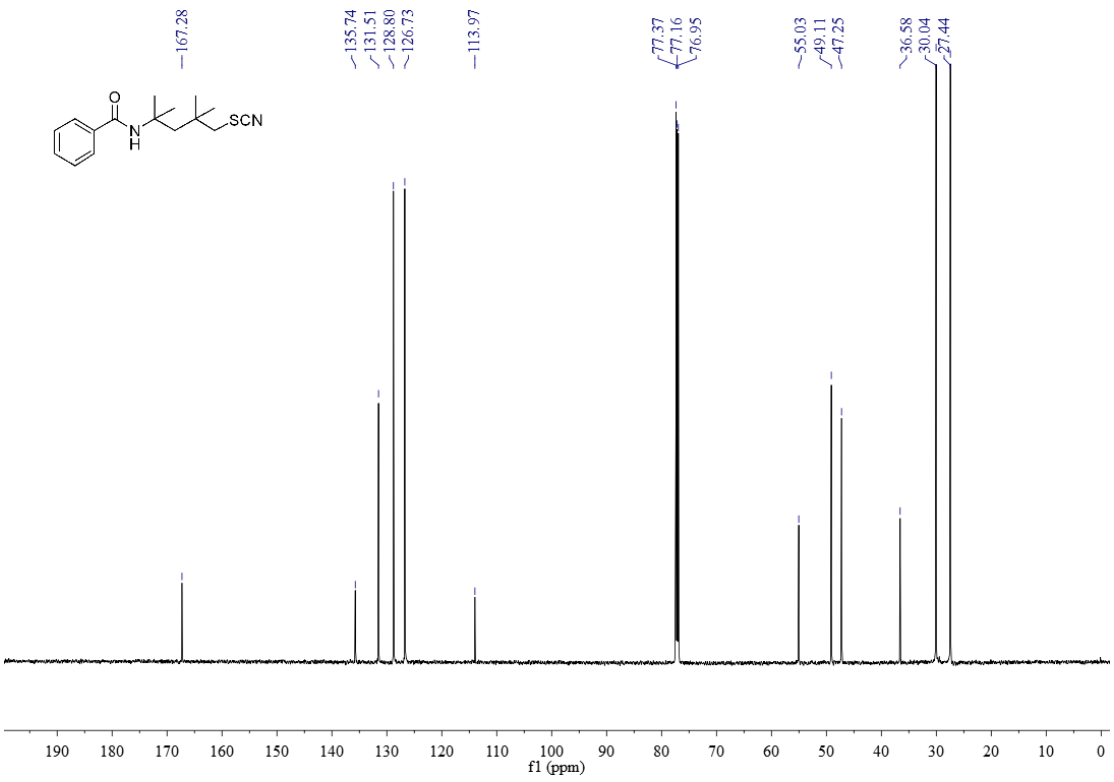
^{13}C NMR of 5g: 150 MHz, $CDCl_3$



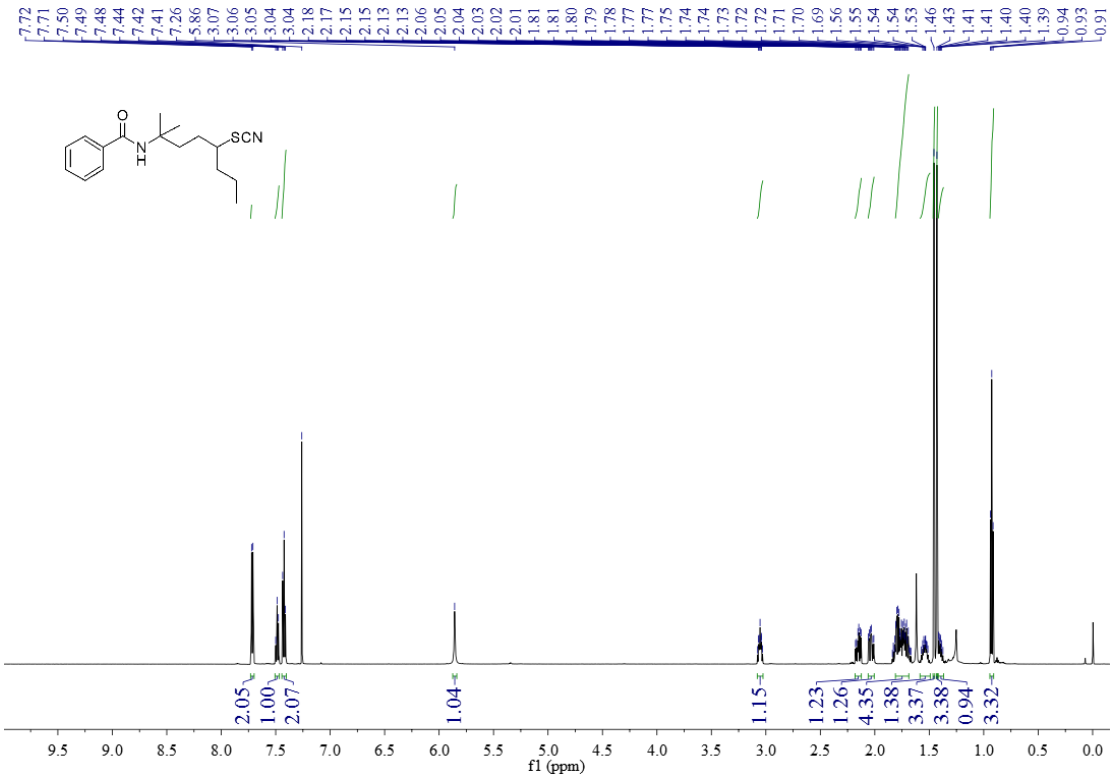
^{1}H NMR of 5h: 600 MHz, $CDCl_3$



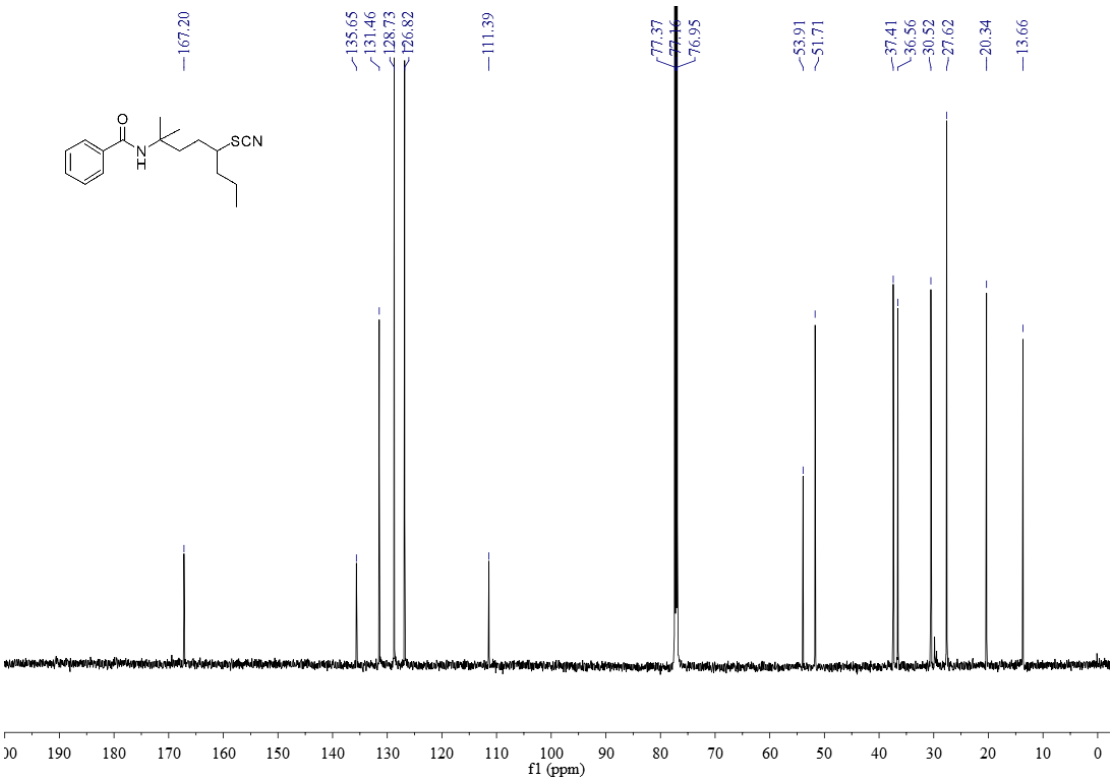
^{13}C NMR of 5h: 150 MHz, $CDCl_3$



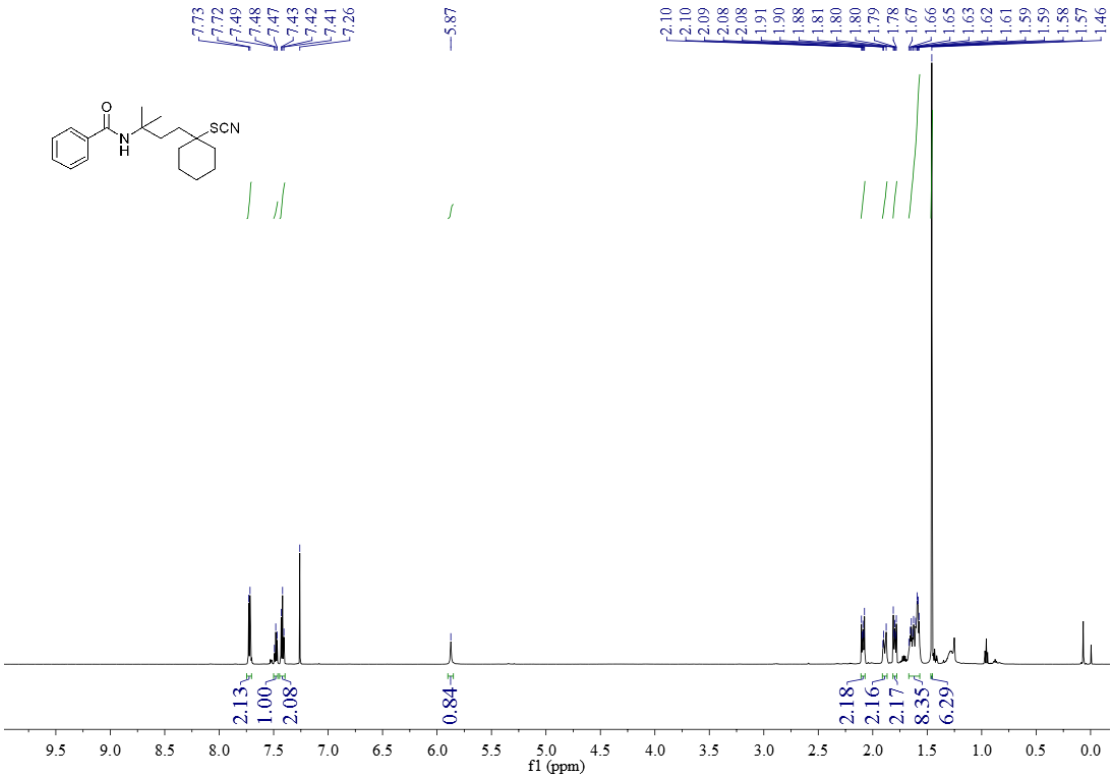
^{1}H NMR of 5i: 600 MHz, $CDCl_3$



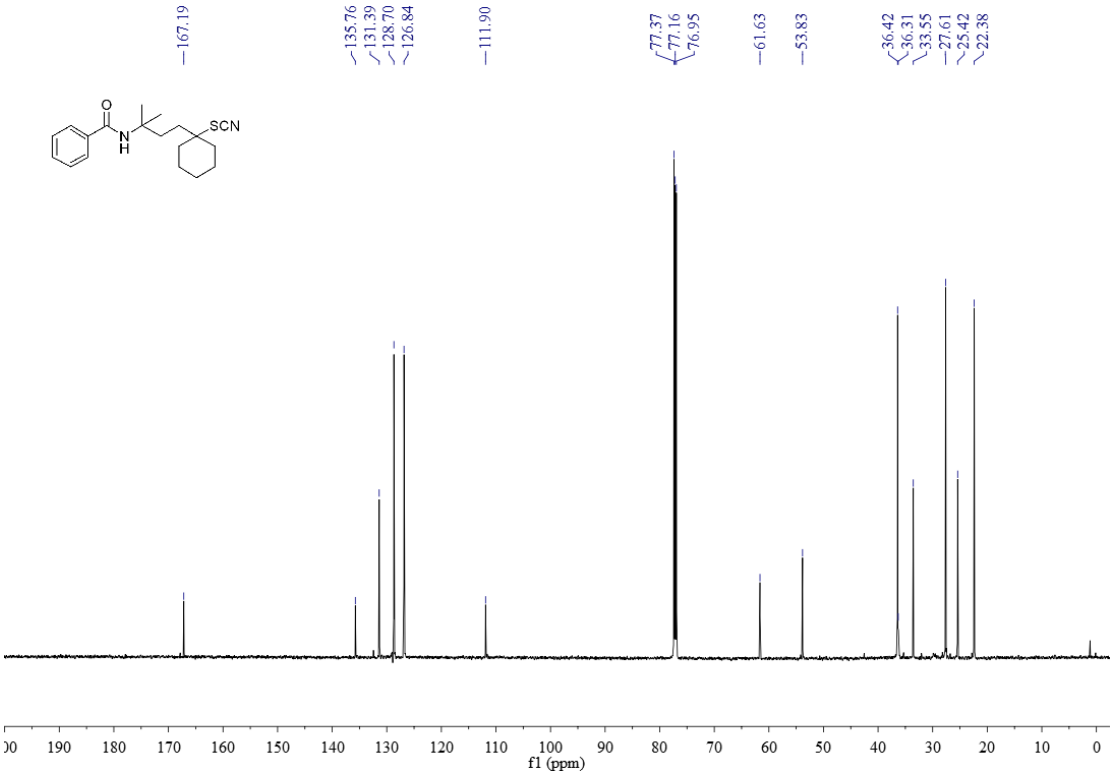
^{13}C NMR of 5i: 150 MHz, $CDCl_3$



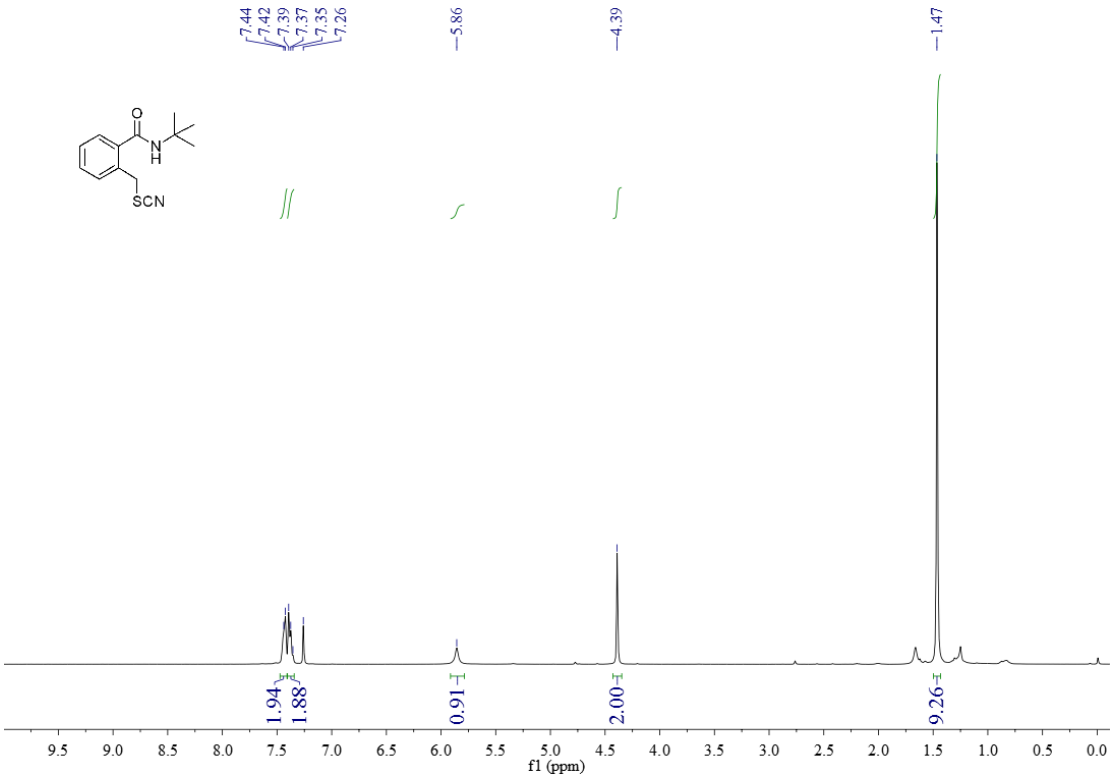
^{1}H NMR of 5j: 600 MHz, $CDCl_3$



^{13}C NMR of 5j: 150 MHz, $CDCl_3$



^{1}H NMR of 5k: 400 MHz, $CDCl_3$



^{13}C NMR of 5k: 150 MHz, $CDCl_3$