Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Palladium-Catalyzed Intramolecular Reductive Olefin Hydrocarbonation: Benzylic Hydrogen Playing as a New Hydrogen Donor

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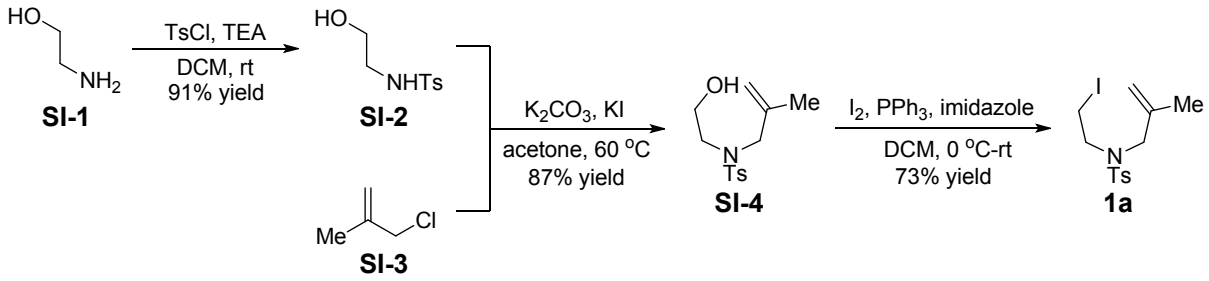
1. General Information

Organic solvents (Aldrich) were used without further purification. Purifications of reactions products were carried out by flash chromatography using Merck silica gel (40-63 μm). ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz) were measured on a Brucker Avance 400 MHz spectrometer. Chemical shifts are reported in parts per million (ppm, δ) downfield from residual solvents peaks and coupling constants are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), doublet (d), triplet (t), …. Splitting patterns that could not be interpreted or easily visualized are designated as multiplet (m). Electrospray mass spectra were obtained using an ESI/TOF Mariner Mass Spectrometer. Unless otherwise noted, all other commercially available reagents and solvents were used without further purification.

2. Preparation of Starting Material.

General Procedure I:

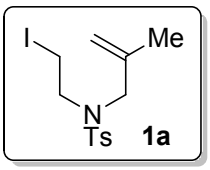


Preparation of alcohol SI-2: To a solution of 2-Aminoethanol **SI-1** (12.2 g, 200 mmol, 1.0 equiv) in DCM (150 mL) at room temperature was added TsCl (38.0 g, 200 mmol, 1.0 equiv). Then a solution of triethylamine (22.2 g, 220 mmol, 1.1 equiv) in 50 mL DCM was added dropwise by constant pressure funnel under vigorous stirring. The mixture was stirred at room temperature over night and then washed with water (50 mL × 3). The organic layer was dried over Na_2SO_4 and concentrated to afford the crude alcohol **SI-2** (39.3 g, 183 mmol, 91% yield) as a white solid, which was used without further purification.

Preparation of alcohol SI-4: The **SI-2** (2.15 g, 10 mmol, 1.0 equiv) was dissolved in 40 mL acetone, and **SI-3** (1.2 mL, 12 mmol, 1.2 equiv), K_2CO_3 (1.66 g, 12 mmol, 1.2 equiv), KI (0.33 g, 2 mmol, 0.2 equiv) was added successively. The mixture was stirred and refluxed at 60 °C over night. Then the reaction system was quenched with water, the organic layer was exacted three times with AcOEt (20 mL). Drying over Na_2SO_4 and concentration in vacuo afforded the crude material, which was then purified by silica gel column chromatography (PE:EA, 2:1) to give the alcohol **SI-4** (2.33 g, 8.65 mmol, 87% yield) as light yellow solid.

Preparation of alkyl iodide 1a: To a solution of **SI-4** (1.66 g, 5 mmol, 1.0 equiv) in 40 mL DCM was cooled to 0 °C. PPh_3 (3.93 g, 15 mmol, 3.0 equiv), imidazole (1.02 g, 15 mmol, 3.0 equiv), I_2 (3.81 g, 15 mmol, 3.0 equiv) was added successively. Then the reaction mixture was warmed to room temperature and stirred for 6 h. The precipitate was removed by filtration. After the concentration of the filtrate *in vacuo*, purification by column chromatography (PE:EA, 20:1) afforded the iodide **1a** (1.39 g, 3.67 mmol, 73% yield) as a white solid.

Analytical Data:



***N*-(2-iodoethyl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{13}H_{18}INO_2S$

The title compound was prepared according to **general procedure I**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.93 (s, 1H), 4.88 (s, 1H), 3.67 (s, 2H), 3.39 (t, *J* = 8.4 Hz, 2H), 3.19 (t, *J* = 8.4 Hz, 2H), 2.43 (s, 3H), 1.73 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.6, 140.5, 136.2, 129.8, 127.1, 115.3, 55.5, 50.8, 21.5, 19.7, 1.7.

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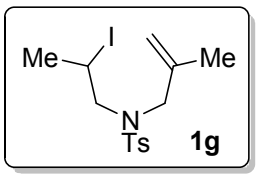

***N*-(2-iodopropyl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{14}H_{20}INO_2S$

The title compound was prepared according to **general procedure I**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 4.91 (s, 1H), 4.85 (s, 1H), 4.31-4.26 (m, 1H), 3.78 (d, *J* = 14.8 Hz, 1H), 3.55-3.47 (m, 2H), 3.36 (dd, *J* = 14.4, 5.6 Hz, 1H), 2.43 (s, 3H), 1.90 (d, *J* = 6.8 Hz, 3H), 1.68 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.6, 140.5, 136.0, 129.7, 127.3, 115.6, 58.3, 56.5, 25.4, 24.0, 21.5, 19.9.

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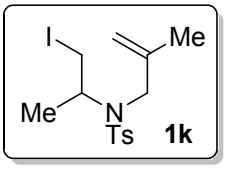

***N*-(1-iodopropan-2-yl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{14}H_{20}INO_2S$

The title compound was prepared according to **general procedure I**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 4.94 (s, 1H), 4.91 (s, 1H), 4.09-4.00 (m, 1H), 3.85 (d, *J* = 15.6 Hz, 1H), 3.64 (d, *J* = 15.6 Hz, 1H), 3.34-3.30 (m, 1H), 3.14 (t, *J* = 9.6 Hz, 1H), 2.42 (s, 3H), 1.78 (s, 3H), 1.23 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.4, 142.6, 137.5, 129.7, 127.1, 114.3, 56.1, 50.5, 21.5, 19.9, 17.0, 8.5.

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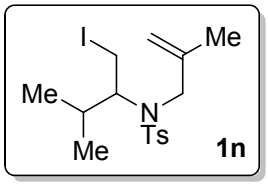

***N*-(1-iodo-3-methylbutan-2-yl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{16}H_{24}INO_2S$

The title compound was prepared according to **general procedure I**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.77 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 4.97 (s, 1H), 4.90 (s, 1H), 3.81 (d, *J* = 16.0 Hz, 1H), 3.74-3.68 (m, 2H), 3.33 (dd, *J* = 11.2, 4.8 Hz, 1H), 3.21 (dd, *J* = 10.8, 6.8 Hz, 1H), 2.42 (s, 3H), 2.06-1.97 (m, 1H), 1.71 (s, 3H), 1.02 (d, *J* = 6.4 Hz, 3H), 0.90 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.3, 142.1, 137.7, 129.4, 127.9, 115.2, 66.2, 51.3, 32.3, 21.5, 20.8, 20.6, 20.5, 5.2.

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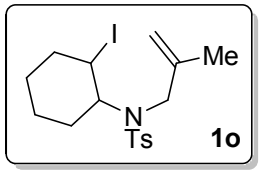

***N*-(2-iodocyclohexyl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{17}H_{24}INO_2S$

The title compound was prepared according to **general procedure I**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.99 (s, 1H), 4.90 (s, 1H), 3.92 (d, *J* = 15.2 Hz, 1H), 3.70 (d, *J* = 15.6 Hz, 1H), 3.39-3.34 (m, 1H), 2.63-2.55 (m, 1H), 2.42 (s, 3H), 2.04-1.99 (m, 1H), 1.84-1.77 (m, 2H),1.76 (s, 3H), 1.60-1.39 (m, 4H), 1.20-1.13 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.3, 142.9, 137.9, 129.6, 127.1, 112.1, 60.2, 55.6, 50.4, 36.3, 28.7, 26.1, 24.4, 21.8, 20.5.

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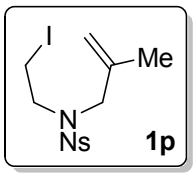


***N*-(2-iodoethyl)-*N*-(2-methylallyl)-4-nitrobenzenesulfonamide**

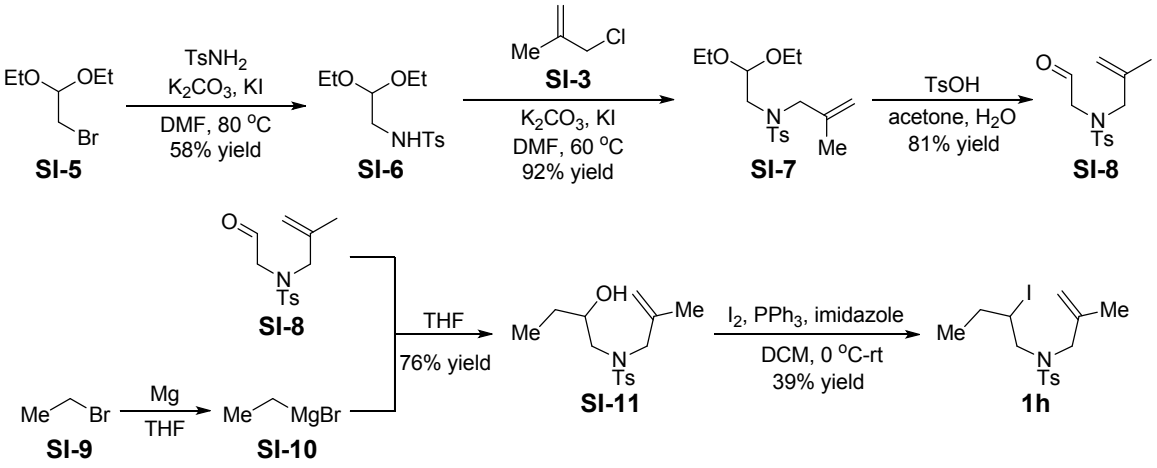
$C_{12}H_{15}IN_2O_4S$

The title compound was prepared according to **general procedure I**, the NsCl (4-nitrobenzenesulphonyl chloride) was employed instead of TsCl in the process of preparing **SI-2**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.37 (d, *J* = 8.8 Hz, 2H), 8.01 (d, *J* = 8.8 Hz, 2H), 4.97 (s, 1H), 4.88 (s, 1H), 3.77 (s, 2H), 3.47 (t, *J* = 8.0 Hz, 2H), 3.21 (t, *J* = 8.8 Hz, 2H), 1.71 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 150.1, 145.2, 139.6, 128.3, 124.5, 115.9, 55.3, 50.7, 19.7, 0.7.

General Procedure II:



Preparation of amide SI-6: To a solution of 2-bromo-1,1-diethoxyethane **SI-5** (1.5 mL, 10 mmol, 1.0 equiv) in *N,N*-dimethylformamide (20 mL) at room temperature was added $TsNH_2$ (5.14 g, 30 mmol, 3.0 equiv). Then the K_2CO_3 (3.46 g, 25 mmol, 2.5 equiv) and KI (0.83 g, 5 mmol, 0.5 equiv) were added successively. The mixture was stirred and refluxed at 80 °C over night. Then the reaction system was quenched with water, the organic layer was exacted with 80 mL AcOEt. The organic extracts were washed three times with water (25 mL). Drying over Na_2SO_4 and concentration *in vacuo*, the crude material was purified by silica gel column chromatography (PE:EA, 4:1) to give the amide **SI-6** (1.67 g, 5.81 mmol, 58% yield) as white solid.

Preparation of amide SI-7: The amide **SI-6** (1.67 g, 5.8 mmol, 1.0 equiv) was dissolved in *N,N*-dimethylformamide (15 mL), and then **SI-3** (0.85 mL, 8.7 mmol, 1.5 equiv), K_2CO_3 (2.40 g, 17.4 mmol, 3.0 equiv), KI (0.48 g, 2.9 mmol, 0.5 equiv) was added successively. The mixture was stirred and refluxed at 60 °C for 8 h. Then the reaction system was quenched with water, the organic layer was exacted with 80 mL AcOEt. The organic extracts were washed three times with water (25 mL). Drying over Na_2SO_4 and concentration *in vacuo*, the crude material was purified by silica gel column chromatography (PE:EA, 8:1) to give the amide **SI-7** (1.82 g, 5.34 mmol, 92% yield) as colorless oil.

Preparation of aldehyde SI-8: The amide **SI-7** (1.82 g, 5.34 mmol, 1.0 equiv) was dissolved in a mixture of 15 mL acetone and 3 mL H_2O was added TsOH (0.19 g, 1.1 mmol, 0.2 equiv). The colorless solution was stirred at 80 °C for 4 h. After cooling the reaction mixture to room temperature, 20 mL H_2O were added. The reaction mixture was extracted with AcOEt (20 mL

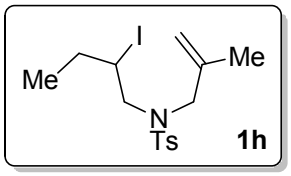
× 3). After drying over Na_2SO_4, the extract was concentrated *in vacuo* to give the crude product **SI-8** (1.16 g, 4.34 mmol, 81% yield). The crude product was used without further purification for the next reaction.

Preparation of Grignard Reagent SI-10: In a 100 mL oven-dried three-necked round bottom flask, Mg (1.09 g, 45 mmol, 1.0 equiv) and dry THF (5 mL) was added under a nitrogen atmosphere. Then 0.3 mL of EtBr (4.0 mmol, 0.09 equiv) was added by a syringe. The mixture was heated at 70 ^oC until the colorless solution turned to light taupe, then the hot plate was removed. A solution of EtBr (3.1 mL, 41 mmol, 0.91 equiv) in dry THF (40 mL) was added dropwise. All of the above operations were carried out under a nitrogen atmosphere. The resulting mixture was stirred at room temperature for 1.5 h to give the solution of Grignard reagent **SI-10** (1 mol/L).

Preparation of alcohol SI-11: In a 50 mL oven-dried round bottom flask, aldehyde **SI-8** (267.3 mg, 1.00 mmol, 1.0 equiv) was dissolved in dry THF (15 mL), the solution of Grignard reagent **SI-10** (1 mol/L) (1.5 mL, 1.50 mmol, 1.5 equiv) was added dropwise at 0 ^oC. Then the resulting mixture was wormed to room temperature and stirred for 30 min before quenching with H_2O. All of the above operations were carried out under a nitrogen atmosphere. Then the organic layer was exacted with dichloromethane (40 mL × 3). After drying over Na_2SO_4, the extract was filtrated through celite and washed with DCM. Concentration *in vacuo* afforded a light brown oil. The crude material was purified by silica gel column chromatography (PE:EA, 5:1) to obtain the alcohol **SI-11** (227.4 mg, 0.76 mmol, 76% yield).

Preparation of alkyl iodide 1h: To a solution of **SI-11** (227.4 mg, 0.76 mmol, 1.0 equiv) in 15 mL DCM was cooled to 0 ^oC. PPh_3 (598.0 mg, 2.28 mmol, 3.0 equiv), imidazole (155.2 mg, 2.28 mmol, 3.0 equiv), I_2 (578.7 mg, 2.28 mmol, 3.0 equiv) was added successively. Then the mixture was warmed to room temperature and stirred for 6 h. Then the precipitate was removed by filtration. After the concentration of the filtrate *in vacuo*, purification by column chromatography (PE:EA, 20:1) afforded the iodide **1h** (120.2 mg, 0.30 mmol, 39% yield) as a colorless oil.

Analytical Data:



***N*-(2-iodobutyl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{15}H_{22}INO_2S$

The title compound was prepared according to **general procedure II**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 4.91 (s, 1H), 4.86 (s, 1H), 4.24-4.14 (m, 1H), 3.84 (d, *J* = 14.8 Hz, 1H), 3.64 (dd, *J* = 14.8, 10.0 Hz, 1H), 3.50 (d, *J* = 14.4 Hz, 1H), 3.37 (dd, *J* = 14.4, 5.2 Hz, 1H), 2.43 (s, 3H), 1.99-1.90 (m, 1H), 1.68 (s, 3H), 1.67-1.62 (m, 1H), 1.02 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.6, 140.7, 136.1, 129.7, 127.3, 115.4, 57.0, 56.6, 36.6, 29.9, 21.5, 19.9, 14.3.

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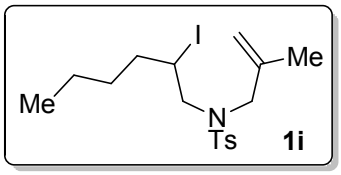


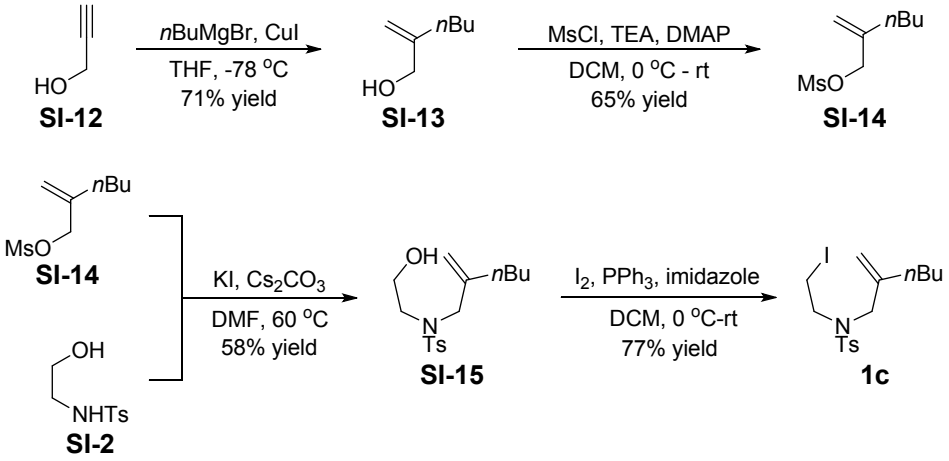
***N*-(2-iodohexyl)-4-methyl-*N*-(2-methylallyl)benzenesulfonamide**

$C_{17}H_{26}INO_2S$

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.91 (s, 1H), 4.86 (s, 1H), 4.25-4.17 (m, 1H), 3.83 (d, *J* = 14.8 Hz, 1H), 3.62 (dd, *J* = 14.4, 9.6 Hz, 1H), 3.53 (d, *J* = 14.4 Hz, 1H), 3.40 (dd, *J* = 14.4, 5.6 Hz, 1H), 2.43 (s, 3H), 1.88-1.80 (m, 1H), 1.69 (s, 3H), 1.67-1.50 (m, 3H), 1.36-1.30 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.6, 140.6, 136.2, 129.7, 127.3, 115.4, 57.2, 56.5, 36.4, 34.5, 31.9, 21.8, 20.0, 13.9.

General Procedure III:



Preparation of alcohol SI-13:

All of the following operations were carried out under a nitrogen atmosphere.

(1) In a 250 mL oven-dried three-necked round bottom flask, Mg (2.19 g, 90 mmol, 2.5 equiv) and dry THF (5 mL) was added under a nitrogen atmosphere. Then 0.3 mL of *n*BuBr was added by a syringe. The mixture was heated at 70 °C until the colorless solution turned to light taupe, then the hot plate was removed. A solution of *n*BuBr (9.6 mL, 90 mmol, 2.5 equiv) in dry THF (90 mL) was added dropwise. The resulting mixture was stirred at room temperature for 2 h, until the Mg was completely consumed, to give the solution of *n*BuMgBr (90 mmol, 2.5 equiv).

(2) In another 250 mL oven-dried three-necked round bottom flask, propargyl alcohol **SI-12** (2.1 mL, 36 mmol, 1.0 equiv) was dissolved in dry THF (40 mL). Then CuI (0.343g, 1.8 mmol, 0.1 equiv) was added under stirring. The suspension was cooled to –78 °C. Then a solution of *n*BuMgBr (90 mmol, 2.5 equiv) in THF (95 mL) was added dropwise by constant pressure funnel under vigorous stirring. The resulting mixture held at –78 °C for 1 h. Then it was warmed to room temperature and stirred for 18 h. The mixture was cooled to –78 °C again and quenched slowly with H_2O (10 mL). After the suspension was warmed to room temperature, dilute HCl solution (1 N, 150 mL) was added and the aqueous layer was extracted with EtOAc (70 mL × 3). The combined organic layers were washed brine (50 mL), dried with Na_2SO_4, and concentrated *in vacuo*. Purification by column chromatography (PE:EA, 3:1) afforded the **SI-13** (2.90 g, 25.4 mmol, 71%) as a light yellow liquid.

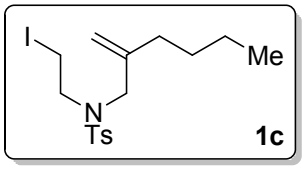
Preparation of alkene SI-14: In a 50 mL oven-dried round bottom flask **SI-13** (571.0 mg, 5.0 mmol, 1.0 equiv) was dissolved in DCM (12 mL). TEA (1.4 mL, 10.0 mmol, 2.0 equiv),

DMAP (61.1 mg, 0.5 mmol, 0.1 equiv) was added successively to this solution. Then a solution of MsCl (0.43 mL, 5.5 mmol, 1.1 equiv) in 3 mL DCM was added dropwise at 0 °C. The resulting mixture was stirred at room temperature for 1.5 h and quenched with aq. NH_4Cl. Then dilute HCl solution (1 N, 15 mL) was added and the aqueous layer was extracted with DCM (10 mL × 3). The combined organic layers were washed brine (10 mL), dried with Na_2SO_4, and concentrated *in vacuo*. Purification by column chromatography (PE:EA, 3:1) afforded the **SI-14** (630.6 mg, 3.3 mmol, 65%) as a colorless oil.

Preparation of alcohol SI-15: In a 50 mL oven-dried round bottom flask **SI-14** (630.6 mg, 3.3 mmol, 1.0 equiv) was dissolved in *N,N*-dimethylformamide (20 mL), and then **SI-2** (861.1 mg, 4.0 mmol, 1.2 equiv), Cs_2CO_3 (2.69 g, 8.3 mmol, 2.5 equiv), KI (116.2 mg, 0.7 mmol, 0.2 equiv) was added successively. The mixture was stirred and refluxed at 60 °C for 8 h. Then the reaction system was quenched with water, the organic layer was exacted with 80 mL AcOEt. The organic extracts were washed with water (25 mL × 3). Drying over Na_2SO_4 and concentration *in vacuo*, the crude material was purified by silica gel column chromatography (PE:EA, 3:1) to give the alcohol **SI-15** (601.1 mg, 1.9 mmol, 58% yield) as colorless oil.

Preparation of alkyl iodide 1c: To a solution of **SI-15** (601.1 mg, 1.9 mmol, 1.0 equiv) in 20 mL DCM was cooled to 0 °C. PPh_3 (1.50 g, 5.7 mmol, 3.0 equiv), imidazole (388.1 mg, 5.7 mmol, 3.0 equiv), I_2 (1.45 g, 5.7 mmol, 3.0 equiv) was added successively. Then the reaction mixture was warmed to room temperature and stirred for 6 h. Then the precipitate was removed by filtration. After the concentration of the filtrate *in vacuo*, purification by column chromatography (PE:EA, 20:1) afforded the iodide **1c** (616.4 mg, 1.5 mmol, 77% yield) as a white solid.

Analytical Data:



***N*-(2-iodoethyl)-4-methyl-*N*-(2-methylenehexyl)benzenesulfonamide**

$C_{16}H_{24}INO_2S$

The title compound was prepared according to **general procedure III**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.93 (s, 1H), 4.90 (s, 1H), 3.68 (s, 2H), 3.40-3.63 (m, 2H), 3.20-3.16 (m, 2H), 2.43 (2, 3H), 2.00 (t, *J* = 7.6 Hz, 2H), 1.42-1.35 (m, 2H), 1.33-1.24 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 144.6, 143.6, 136.3, 129.8, 127.2, 114.2, 54.2, 50.7, 32.5, 29.6, 22.4, 21.5, 13.9, 1.7.

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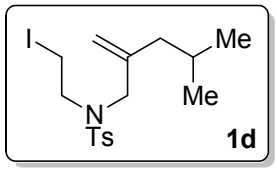


***N*-(2-iodoethyl)-4-methyl-*N*-(4-methyl-2-methylenepentyl)benzenesulfonamide**

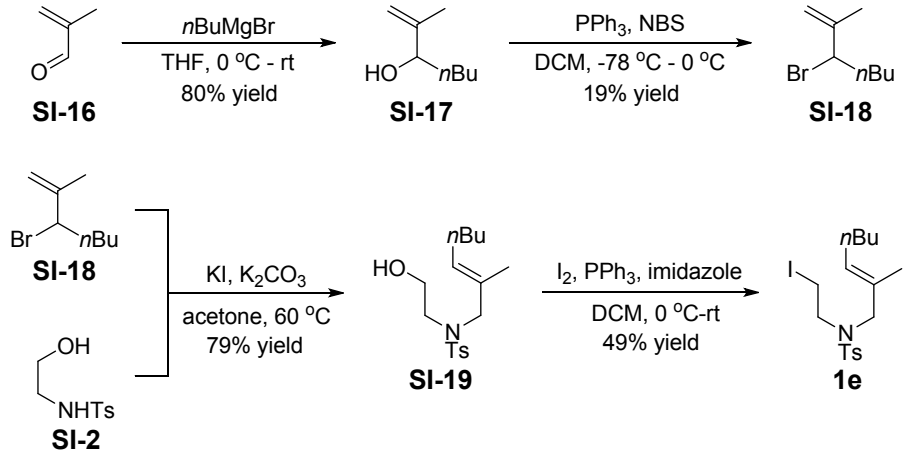
$C_{16}H_{24}INO_2S$

The title compound was prepared according to **general procedure III**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.69 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.94 (s, 1H), 4.91 (s, 1H), 3.65 (s, 2H), 3.41-3.37 (m, 2H), 3.21-3.17 (m, 2H), 2.43 (s, 3H), 1.89 (d, *J* = 7.2 Hz, 2H), 1.79-1.69 (m, 1H), 0.85 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.6, 143.2, 136.2, 129.8, 127.2, 115.7, 53.9, 50.7, 42.4, 27.8, 25.9, 22.3, 21.5, 1.6.

General Procedure IV:



Preparation of alcohol SI-17:

All of the following operations were carried out under a nitrogen atmosphere.

(1) In a 100 mL oven-dried three-necked round bottom flask, Mg (291.6 mg, 12 mmol, 1.2 equiv) and dry THF (3 mL) was added under a nitrogen atmosphere. Then 0.2 mL of *n*BuBr was added by a syringe. The mixture was heated at 70 °C until the colorless solution turned to light taupe, then the hot plate was removed. A solution of *n*BuBr (1.3 mL, 12 mmol, 1.2 equiv) in dry THF (30 mL) was added dropwise. The resulting mixture was stirred at room temperature for 2 h, until the Mg was completely consumed, to give the solution of *n*BuMgBr (12 mmol, 1.2 equiv).

(2) In a 100 mL oven-dried round bottom flask, aldehyde **SI-16** (0.82 mL, 10 mmol, 1.0 equiv) was dissolved in dry THF (5 mL), the solution of *n*BuMgBr (12 mmol, 1.2 equiv) was added dropwise at 0 °C. Then the resulting mixture was wormed to room temperature and stirred for 30 min before quenching with H_2O. Then the organic layer was exacted with dichloromethane (40 mL × 3). After drying over Na_2SO_4, the extract was filtrated through celite and washed with DCM. Concentration *in vacuo* afforded a light yellow oil. The crude material was purified by silica gel column chromatography (PE:EA, 5:1) to obtain the alcohol **SI-17** (1.024 g, 8.0 mmol, 80% yield).

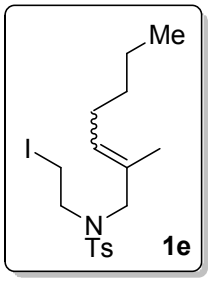
Preparation of bromide SI-18: In a 50 mL oven-dried round bottom flask **SI-17** (1.024 g, 8.0 mmol, 1.0 equiv) was dissolved in DCM (20 mL), and then PPh_3 (2.5 g, 9.6 mmol, 1.2 equiv) was added at 0 °C. The resulting mixture was stirred for 10 min before cooling to -78 °C. Then *N*-Bromosuccinimide (1.5 g, 8.8 mmol, 1.1 equiv) was added to the above reaction

mixture. The solution was stirred at -78 °C for 30 min, which was then warmed to 0 °C and stirred for an additional 10 min. After the concentration *in vacuo*, purification by column chromatography (PE only) afforded the bromide **SI-18** (291.6 mg, 1.5 mmol, 19% yield) as a light yellow liquid.

Preparation of bromide SI-19: In a 50 mL oven-dried round bottom flask, the **SI-18** (291.6 mg, 1.5 mmol, 1.0 equiv) was dissolved in 10 mL acetone, and **SI-2** (473.6 mg, 2.2 mmol, 1.5 equiv), K_2CO_3 (525.2 g, 3.8 mmol, 2.5 equiv), KI (49.8 mg, 0.3 mmol, 0.2 equiv) was added successively. The resulting mixture was stirred and refluxed at 60 ^oC over night. Then the reaction system was quenched with water, the organic layer was exacted with AcOEt (10 mL × 3). Drying over Na_2SO_4 and concentration in vacuo afforded the crude material, which was then purified by silica gel column chromatography (PE:EA, 2:1) to give the alcohol **SI-19** (385.6 mg, 1.2 mmol, 79% yield) as a colorless oil.

Preparation of alkyl iodide 1e: To a solution of **SI-19** (385.6 mg, 1.2 mmol, 1.0 equiv) in 15 mL DCM was cooled to 0 ^oC. PPh_3 (918.1 mg, 3.5 mmol, 3.0 equiv), imidazole (238.3 mg, 3.5 mmol, 3.0 equiv), I_2 (888.3 mg, 3.5 mmol, 3.0 equiv) was added successively. Then the reaction mixture was warmed to room temperature and stirred for 6 h. Then the precipitate was removed by filtration. After the concentration of the filtrate *in vacuo*, purification by column chromatography (PE:EA, 20:1) afforded the iodide **1e** (251.3 mg, 0.58 mmol, 49% yield) as a white solid.

Analytical Data:



***N*-(2-iodoethyl)-4-methyl-*N*-(2-methylhept-2-en-1-yl)benzenesulfonamide**

$C_{17}H_{26}INO_2S$

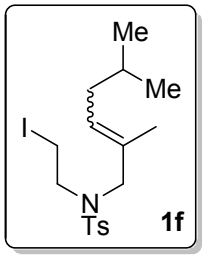
E/Z = 19:31

The title compound was prepared according to **general procedure IV**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71-7.70 (m, 2H), 7.33-7.30 (m, 2H), 5.41 (t, *J* = 7.6 Hz, 0.4H), 5.31 (t, *J* = 7.6 Hz, 0.6H), 3.74 (s, 0.8H), 3.60 (s, 1.2H), 3.36-3.32 (m, 2H), 3.21-3.13 (m, 2H), 2.43 (s, 1.1H), 2.43 (s, 1.9H), 2.07-1.93 (m, 2H), 1.66 (s, 1.1H), 1.62 (s, 1.9H), 1.32-1.27 (m, 4H), 0.91-0.86 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.5, 143.5, 136.4, 136.3, 131.9, 131.3, 130.0, 129.8, 129.8, 129.5, 127.1, 127.1, 57.5, 50.6, 50.4, 48.7, 32.1, 31.4, 27.6, 27.4, 22.4, 22.3, 21.5, 14.0, 13.9, 1.9, 1.8.

……..



***N*-(2,5-dimethylhex-2-en-1-yl)-*N*-(2-iodoethyl)-4-methylbenzenesulfonamide**

$C_{17}H_{26}INO_2S$

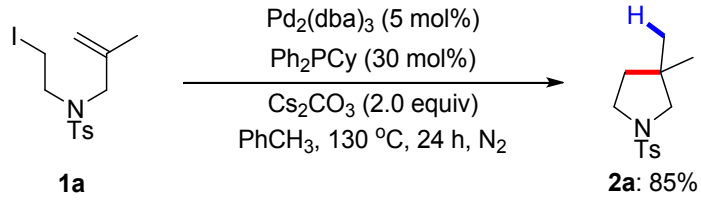
E/Z = 9:16

The title compound was prepared according to **general procedure IV**.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71-7.68 (m, 2H), 7.33-7.30 (m, 2H), 5.43 (t, *J* = 7.6 Hz, 0.4H), 5.34 (t, *J* = 7.6 Hz, 0.6H), 3.74 (s, 0.7H), 3.63 (s, 1.3H), 3.37-3.32 (m, 2H), 3.22-3.14 (m, 2H), 2.44 (s, 1.1H), 2.43 (s, 1.9H), 1.93-1.86 (m, 2H), 1.68 (s, 1.1H), 1.62 (s, 1.9H), 1.59-1.58 (m, 1H), 0.89-0.86 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.5, 143.5, 136.5, 136.3, 130.7, 130.3, 130.1, 129.8, 129.8, 127.1, 127.1, 57.6, 50.5, 50.3, 48.8, 37.1, 36.7, 28.8, 28.6, 22.4, 22.4, 21.6, 21.5, 14.1, 1.8, 1.8.

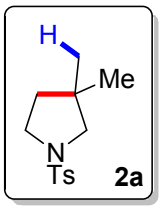
3. Typical Procedure and Analytical Data for Palladium-Catalyzed Intramolecular Hydrocarbonation Reaction.



Typical Procedure:

Unactivated alkyl iodide **1a** (75.9 mg, 0.20 mmol, 1.0 equiv), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 0.05 equiv), Ph_2PCy (16.1 mg, 0.06 mmol, 0.3 equiv) and Cs_2CO_3 (130.3 mg, 0.40 mmol, 2.0 equiv) were added to a reaction tube and vacuum purged three times, backfilling with N_2. Then the toluene (3 mL) was added under nitrogen atmosphere. The resulting mixture was stirred at 130 °C for 24 h. After cooling the reaction mixture at rt, it was quenched with a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with EtOAc (10 mL × 3). The combined organic phase was sequentially washed with saturated aqueous solution of NaCl and then concentrated *in vacuo*. The mixture was purified by silica gel column chromatography (PE:EA, 20:1) to give the reductive product **2a** (43.2 mg, 0.17 mmol, 85% yield).

Analytical Data:



3,3-dimethyl-1-tosylpyrrolidine

$C_{13}H_{19}NO_2S$ **MW:** 253.36 g·mol^{-1}

Off-White Solid

Isolated Amount: 43.2 mg **Yield:** 85%

^{1}H NMR (600 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 3.32 (t, *J* = 7.2 Hz, 2H), 2.97 (s, 2H), 2.42 (s, 3H), 1.54 (t, *J* = 7.2 Hz, 2H), 0.91 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.2, 134.1, 129.5, 127.4, 60.2, 47.0, 39.1, 38.6, 26.0, 21.5.

MS (EI) m/z 253 (M+); **HRMS (ESI)** Calcd for $C_{13}H_{19}NO_2S$+H 254.1215, Found 254.1217.

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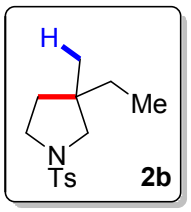


3-ethyl-3-methyl-1-tosylpyrrolidine

$C_{14}H_{21}NO_2S$ **MW:** 267.39 g·mol^{-1}

Off-White Solid

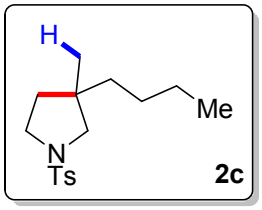
Isolated Amount: 29.3 mg **Yield:** 55%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 3.33-3.27 (m, 2H), 2.98 (s, 2H), 2.43 (s, 3H), 1.58-1.51 (m, 2H), 1.29-1.23 (m, 2H), 0.82 (s, 3H), 0.79 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.1, 134.1, 129.5, 127.4, 58.8, 46.8, 42.1, 37.3, 31.9, 22.6, 21.5, 9.1.

MS (EI) m/z 267 (M+); **HRMS (ESI)** Calcd for $C_{14}H_{21}NO_2S$+H 268.1371, Found 268.1374.

……



3-butyl-3-methyl-1-tosylpyrrolidine

$C_{16}H_{25}NO_2S$ **MW:** 295.44 g·mol^{-1}

Off-White Solid

Isolated Amount: 35.2 mg **Yield:** 60%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 3.32-3.28 (m, 2H), 2.98 (s, 2H), 2.43 (s, 3H), 1.58-1.53 (m, 2H), 1.20-1.11 (m, 6H), 0.86-0.83 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.2, 134.2, 129.5, 127.4, 59.1, 46.7, 41.8, 39.2, 37.8, 27.0, 23.3, 23.2, 21.5, 13.9.

MS (EI) m/z 295 (M+); **HRMS (ESI)** Calcd for $C_{16}H_{25}NO_2S$+H 296.1684, Found 296.1682.

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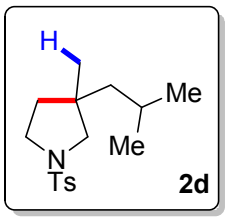


3-isobutyl-3-methyl-1-tosylpyrrolidine

$C_{16}H_{25}NO_2S$ **MW:** 295.44 g·mol^{-1}

Off-white solid

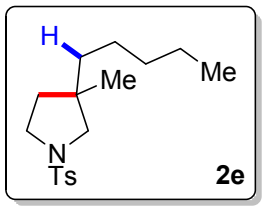
Isolated Amount: 30.1 mg **Yield**: 51%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 3.33-3.26 (m, 2H), 3.06 (d, *J* = 9.6 Hz, 1H), 2.94 (d, *J* = 9.6 Hz, 1H), 2.43 (s, 3H), 1.66-1.55 (m, 3H), 1.17 (dd, *J* = 6.0, 4.4 Hz, 2H), 0.86-0.84 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.2, 134.3, 129.5, 127.4, 59.9, 48.6, 46.4, 42.0, 38.8, 25.0, 24.5, 24.2, 23.2, 21.5.

MS (EI) m/z 295 (M+); **HRMS (ESI)** Calcd for $C_{16}H_{25}NO_2S$+H 296.1684, Found 296.1685.

……



3-methyl-3-pentyl-1-tosylpyrrolidine

$C_{17}H_{27}NO_2S$ **MW:** 309.47 g·mol^{-1}

White Solid

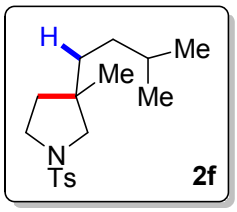
Isolated Amount: 49.3 mg **Yield:** 80%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.31-3.28 (m, 2H), 2.98 (s, 2H), 2.42 (s, 3H), 1.59-1.52 (m, 2H), 1.29-1.21 (m, 2H), 1.18-1.09 (m, 6H), 0.87-0.83 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.1, 134.2, 129.5, 127.4, 59.1, 46.7, 41.8, 39.4, 37.8, 32.4, 24.5, 23.2, 22.5, 21.5, 14.0.

MS (EI) m/z 309 (M+); **HRMS (ESI)** Calcd for $C_{17}H_{27}NO_2S$+H 310.1841, Found 310.1843.

……



3-isopentyl-3-methyl-1-tosylpyrrolidine

$C_{17}H_{27}NO_2S$ **MW:** 309.47 g·mol^{-1}

Off-White Solid

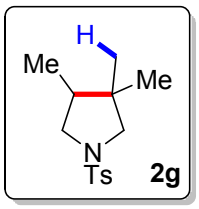
Isolated Amount: 43.7 mg **Yield:** 71%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.34-3.25 (m, 2H), 2.98 (s, 2H), 2.42 (s, 3H), 1.59-1.48 (m, 2H), 1.39-1.32 (m, 1H), 1.19-1.12 (m, 2H), 1.07-0.98 (m, 2H), 0.82 (s, 3H), 0.82-0.79 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.2, 134.2, 129.5, 127.4, 59.2, 46.7, 41.8, 37.7, 37.1, 33.8, 28.5, 23.2, 22.5, 22.4, 21.5.

MS (EI) m/z 309 (M+); **HRMS (ESI)** Calcd for $C_{17}H_{27}NO_2S$+H 310.1841, Found 310.1840.

……



3,3,4-trimethyl-1-tosylpyrrolidine

$C_{14}H_{21}NO_2S$ **MW:** 267.39 g·mol^{-1}

Colorless Oil

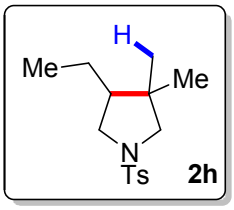
Isolated Amount: 36.4 mg **Yield:** 68%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.47 (dd, *J* = 9.6, 7.6 Hz, 1H), 3.16 (d, *J* = 9.2 Hz, 1H), 2.96 (d, *J* = 9.2 Hz, 1H), 2.89 (t, *J* = 9.6 Hz, 1H), 2.43 (s, 3H), 1.74-1.68 (m, 1H), 0.90 (s, 3H), 0.78 (d, *J* = 7.2 Hz, 3H), 0.65 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.1, 134.2, 129.5, 127.3, 61.0, 53.5, 42.4, 40.3, 24.7, 21.5, 20.0, 11.4.

MS (EI) m/z 267 (M+); **HRMS (ESI)** Calcd for $C_{14}H_{21}NO_2S$+H 268.1371, Found 268.1370.

……



4-ethyl-3,3-dimethyl-1-tosylpyrrolidine

$C_{15}H_{23}NO_2S$ **MW**: 281.41 g·mol^{-1}

Off-White Solid

Isolated Amount: 39.2 mg **Yield**: 70%

^{1}H NMR (400 MHz, DMSO, δ ppm): 7.71 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.53 (dd, *J* = 9.6, 8.0 Hz, 1H), 3.14 (d, *J* = 9.6 Hz, 1H), 2.95-2.87 (m, 2H), 2.43 (s, 3H), 1.56-1.48 (m, 1H), 1.40-1.33 (m, 1H), 1.05-0.97 (m, 1H), 0.93 (s, 3H), 0.84 (t, *J* = 7.2 Hz, 3H), 0.66 (s, 3H).

^{13}C NMR (100 MHz, DMSO, δ ppm): 143.2, 134.1, 129.5, 127.4, 58.8, 46.8, 42.1, 37.3, 31.9, 22.6, 21.5, 9.1.

MS (EI) m/z 281 (M+); **HRMS (ESI)** Calcd for $C_{15}H_{23}NO_2S$+H 282.1528, Found 282.1527.

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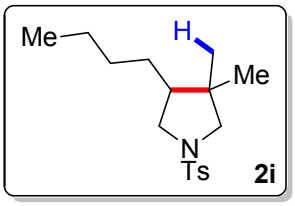


4-butyl-3,3-dimethyl-1-tosylpyrrolidine

$C_{17}H_{27}NO_2S$ **MW**: 309.47 g·mol^{-1}

White Solid

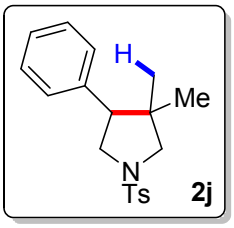
Isolated Amount: 39.7 mg **Yield**: 64%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.51 (dd, *J* = 9.6, 7.6 Hz, 1H), 3.14 (d, *J* = 9.6 Hz, 1H), 2.97-2.86 (m, 2H), 2.43 (s, 3H), 1.60-1.53 (m, 1H), 1.28-1.12 (m, 5H), 1.02-0.95 (m, 1H), 0.92 (s, 3H), 0.86 (t, *J* = 6.8 Hz, 3H), 0.65 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.1, 134.3, 129.5, 127.3, 61.4, 52.2, 48.1, 40.3, 30.8, 27.0, 24.9, 22.9, 21.5, 20.5, 13.9.

MS (EI) m/z 309 (M+); **HRMS (ESI)** Calcd for $C_{17}H_{27}NO_2S$+H 310.1841, Found 310.1844.

……



3,3-dimethyl-4-phenyl-1-tosylpyrrolidine

$C_{19}H_{23}NO_2S$ **MW:** 329.46 g·mol^{-1}

Off-White Solid

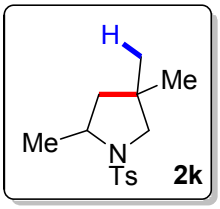
Isolated Amount: 39.4 mg **Yield:** 60%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.78 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 2H), 7.25-7.21 (m, 3H), 7.02 (dd, *J* = 7.6, 2.4 Hz, 2H), 3.71-3.63 (m, 2H), 3.30 (d, *J* = 9.6 Hz, 1H), 3.12 (d, *J* = 9.6 Hz, 1H), 2.85 (t, *J* = 8.4 Hz, 1H), 2.46 (s, 3H), 0.93 (s, 3H), 0.59 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.4, 137.4, 134.4, 129.7, 128.4, 128.2, 127.5, 127.1, 60.6, 54.0, 51.3, 42.0, 25.8, 21.5.

MS (EI) m/z 329 (M+); **HRMS (ESI)** Calcd for $C_{19}H_{23}NO_2S$+H 330.1528, Found 330.1530.

……



2,4,4-trimethyl-1-tosylpyrrolidine

$C_{14}H_{21}NO_2S$ **MW:** 267.39 g·mol^{-1}

Colorless Oil

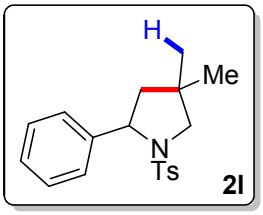
Isolated Amount: 34.7 mg **Yield**: 65%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.68-3.59 (m, 1H), 3.15 (d, *J* = 10.4 Hz, 1H), 3.05 (d, *J* = 10.4 Hz, 1H), 2.41 (s, 3H), 1.74-1.69 (m, 1H), 1.41-1.36 (m, 4H), 1.02 (s, 3H), 0.54 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.0, 135.3, 129.4, 127.4, 61.4, 55.9, 48.9, 37.1, 26.5, 25.9, 22.6, 21.4.

MS (EI) m/z 267 (M+); **HRMS (ESI)** Calcd for $C_{14}H_{21}NO_2S$+H 268.1371, Found 268.1368.

……



4,4-dimethyl-2-phenyl-1-tosylpyrrolidine

$C_{19}H_{23}NO_2S$ **MW:** 329.46 g·mol^{-1}

Off-White Solid

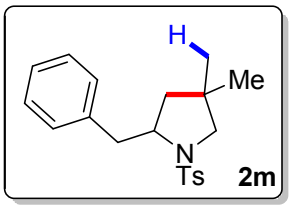
Isolated Amount: 41.4 mg **Yield**: 63%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.54 (d, *J* = 8.0 Hz, 2H), 7.28-7.26 (m, 3H), 7.24-7.19 (m, 4H), 4.69 (dd, *J* = 9.2, 7.2 Hz, 1H), 3.44 (d, *J* = 10.4 Hz, 1H), 3.34 (d, *J* = 10.4 Hz, 1H), 2.40 (s, 3H), 2.01 (ddd, *J* = 12.8, 7.2, 1.2 Hz, 1H), 1.72 (dd, *J* = 12.8, 9.6 Hz, 1H), 1.05 (s, 3H), 0.75 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.0, 142.9, 135.7, 129.3, 128.2, 127.3, 127.0, 126.5, 63.8, 61.8, 51.5, 38.1, 26.0, 25.6, 21.5.

MS (EI) m/z 329 (M+); **HRMS (ESI)** Calcd for $C_{19}H_{23}NO_2S$+H 330.1528, Found 330.1527.

……



2-benzyl-4,4-dimethyl-1-tosylpyrrolidine

$C_{20}H_{25}NO_2S$ **MW:** 343.48 g·mol^{-1}

Off-White Solid

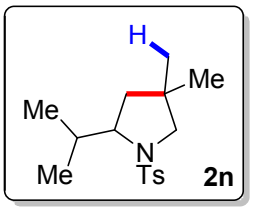
Isolated Amount: 46.5 mg **Yield:** 68%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.79 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 2H), 7.23-7.22 (m, 3H), 3.82-3.74 (m, 1H), 3.58 (dd, *J* = 13.2, 3.6 Hz, 1H), 3.12 (s, 2H), 2.77 (dd, *J* = 13.2, 10.0 Hz, 1H), 2.43 (s, 3H), 1.48 (dd, *J* = 8.4, 6.0 Hz, 2H), 0.98 (s, 3H), 0.44 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.3, 138.5, 135.2, 129.6, 129.5, 128.4, 127.5, 126.3, 61.6, 61.5, 45.7, 42.9, 37.2, 26.4, 25.8, 21.5.

MS (EI) m/z 343 (M+); **HRMS (ESI)** Calcd for $C_{20}H_{25}NO_2S$+H 344.1684, Found 344.1681.

……



2-isopropyl-4,4-dimethyl-1-tosylpyrrolidine

$C_{16}H_{25}NO_2S$ **MW:** 295.44 g·mol^{-1}

Colorless Oil

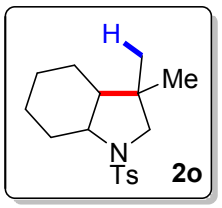
Isolated Amount: 44.3 mg **Yield:** 75%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.72 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.71-3.65 (m, 1H), 3.27 (dd, *J* = 10.8, 1.2 Hz, 1H), 3.01 (d, *J* = 10.8 Hz, 1H), 2.54-2.47 (m, 1H), 2.42 (s, 3H), 1.50-1.43 (m, 2H), 1.00 (s, 3H), 0.84-0.80 (m, 6H), 0.49 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 142.9, 136.4, 129.4, 127.2, 64.9, 61.8, 39.3, 37.1, 30.2, 26.1, 25.8, 19.3, 14.5.

MS (EI) m/z 295 (M+); **HRMS (ESI)** Calcd for $C_{16}H_{25}NO_2S$+H 296.1684, Found 296.1682.

……



3,3-dimethyl-1-tosyloctahydro-1*H*-indole

$C_{17}H_{25}NO_2S$ **MW:** 307.45 g·mol^{-1}

White Solid

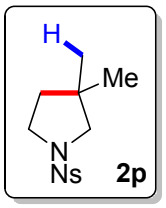
Isolated Amount: 39.8 mg **Yield:** 65%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.71 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.30 (d, *J* = 10.8 Hz, 1H), 3.05 (d, *J* = 10.4 Hz, 1H), 2.55-2.46 (m, 1H), 2.42 (s, 3H), 1.69-1.64 (m, 1H), 1.59-1.42 (m, 5H), 1.25-1.16 (m, 3H), 0.90 (s, 3H), 0.46 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 143.1, 134.2, 129.4, 127.5, 60.4, 58.9, 47.8, 38.9, 28.9, 27.0, 24.6, 24.3, 21.9, 21.5, 20.3.

MS (EI) m/z 307 (M+); **HRMS (ESI)** Calcd for $C_{17}H_{25}NO_2S$+H 308.1684, Found 308.1685.

……



3,3-dimethyl-1-((4-nitrophenyl)sulfonyl)pyrrolidine

$C_{12}H_{16}N_2O_4S$ **MW:** 284.33 g·mol^{-1}

Yellow Solid

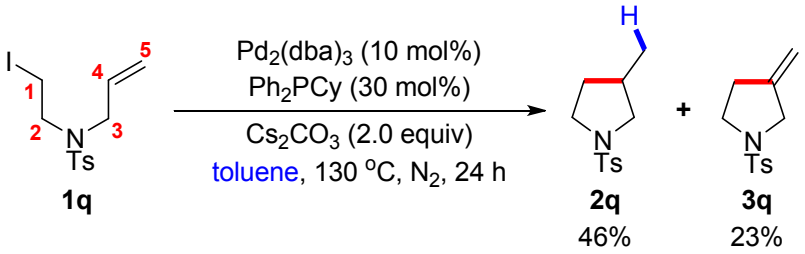
Isolated Amount: 37.9 mg **Yield:** 67%

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.38 (d, *J* = 8.8 Hz, 2H), 8.01 (d, *J* = 9.2 Hz, 2H), 3.40 (t, *J* = 7.2 Hz, 2H), 3.03 (s, 2H), 1.61 (t, *J* = 7.2 Hz, 2H), 0.94 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 150.0, 143.3, 128.4, 124.3, 60.3, 47.1, 39.1, 38.8, 25.8.

MS (EI) m/z 284 (M+); **HRMS (ESI)** Calcd for $C_{12}H_{16}N_2O_4S$+H 285.0909, Found 285.0911.

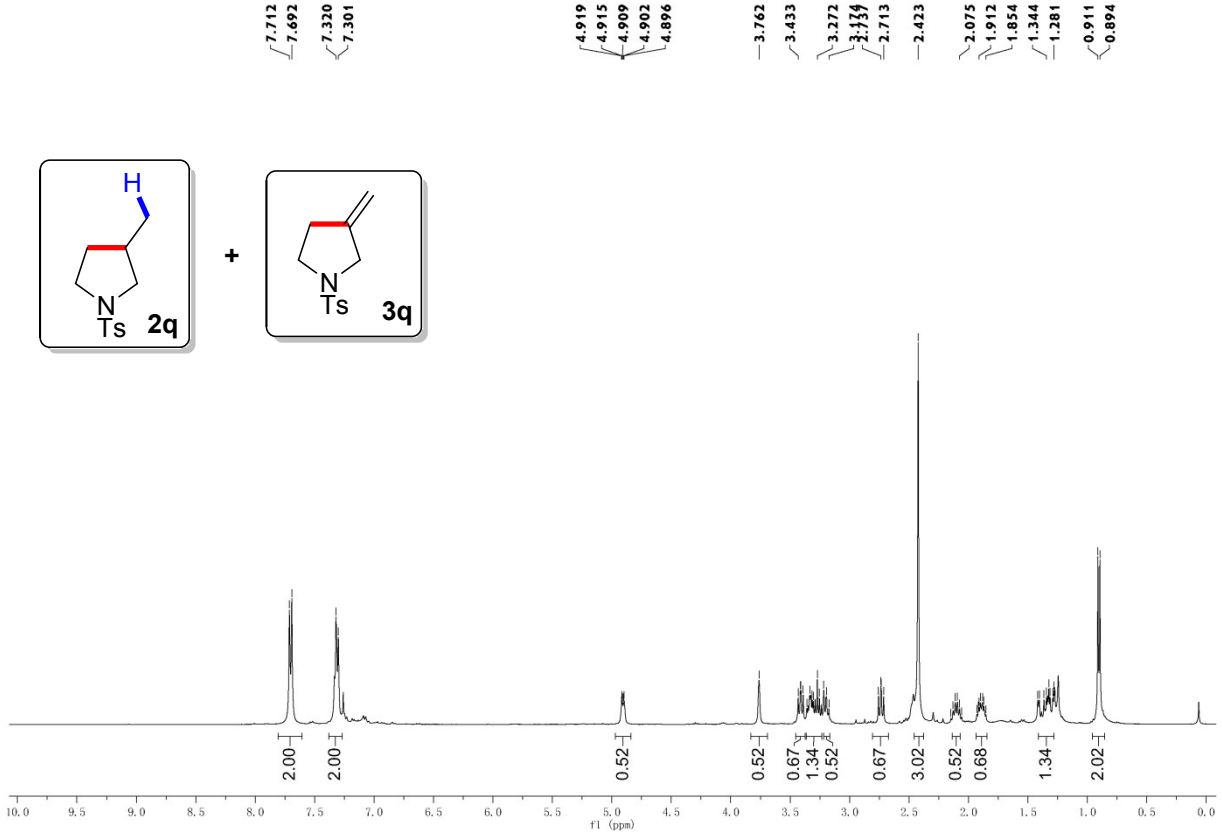
4. Reaction of substrate 1q.

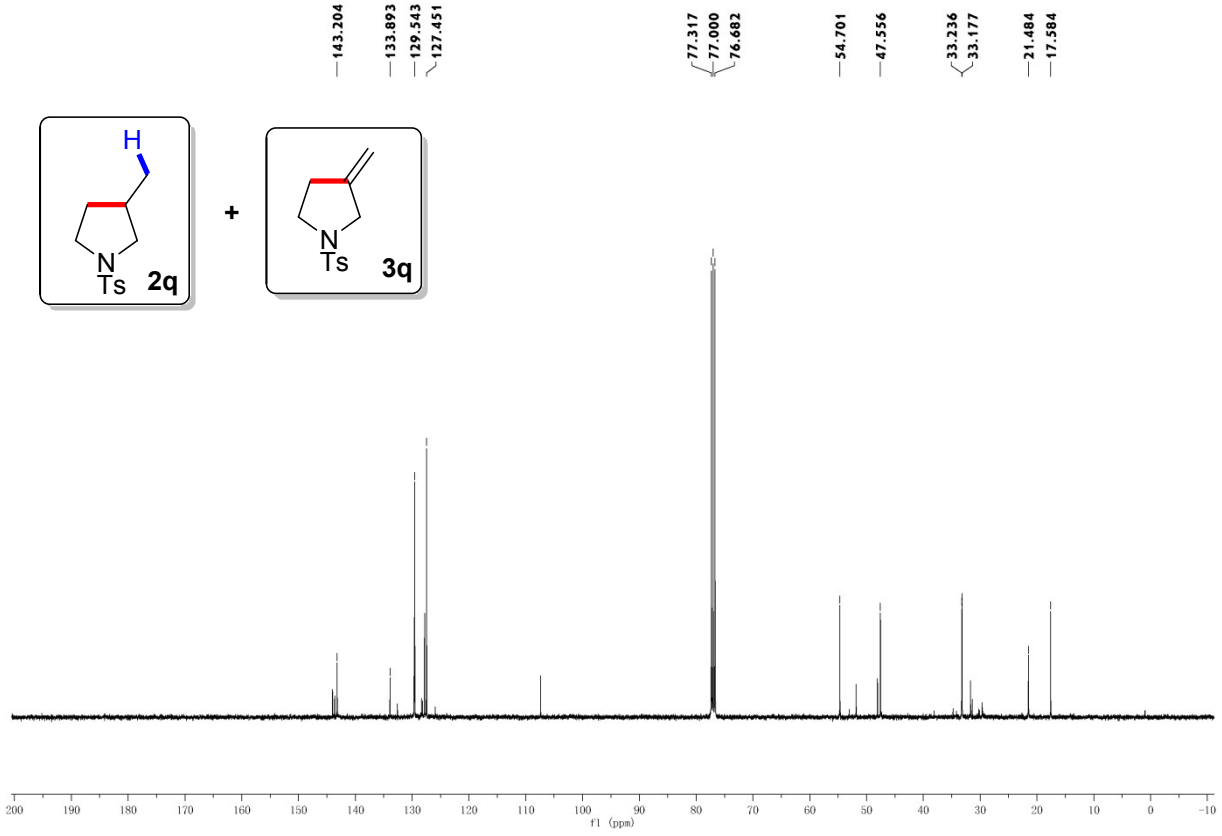


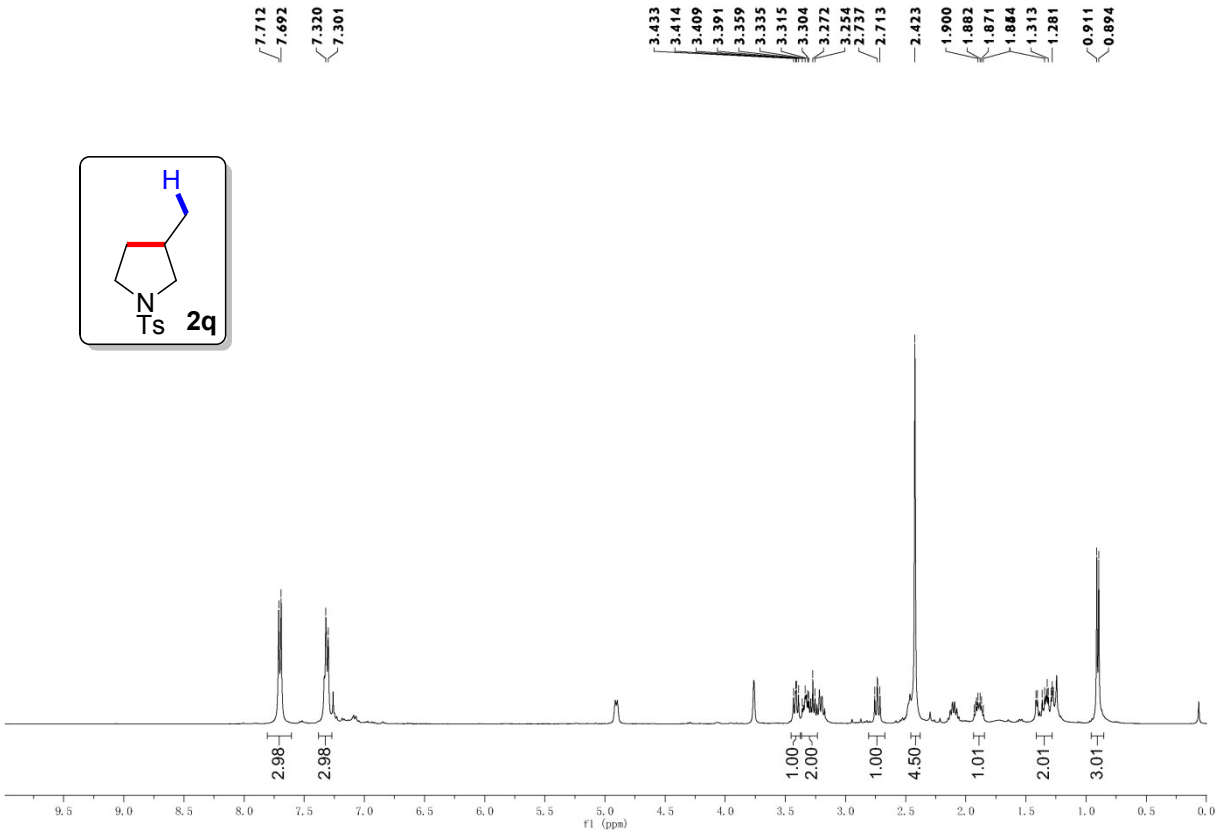
Unactivated alkyl iodide **1q** (73.0 mg, 0.20 mmol, 1.0 equiv), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 0.05 equiv), Ph_2PCy (16.1 mg, 0.06 mmol, 0.3 equiv) and Cs_2CO_3 (130.3 mg, 0.40 mmol, 2.0 equiv) were added to a reaction tube and vacuum purged three times, backfilling with N_2. Then the toluene (3 mL) was added under nitrogen atmosphere. The resulting mixture was stirred at 130 °C for 24 h. After cooling the reaction mixture at rt, it was quenched with a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with EtOAc (10 mL × 3). The combined organic phase was sequentially washed with saturated aqueous solution of NaCl and then concentrated *in vacuo*. The mixture was purified by silica gel column chromatography (PE:EA, 20:1) to give an inseparable mixture (33.2 mg) of the reductive product **2q** (22.2 mg, 0.09 mmol, 46% yield) and eliminated product **3q** (11.0 mg, 0.05 mmol, 23% yield) (the proportion of **2q** and **3q** in mixture was determined by **^{1}H NMR**).

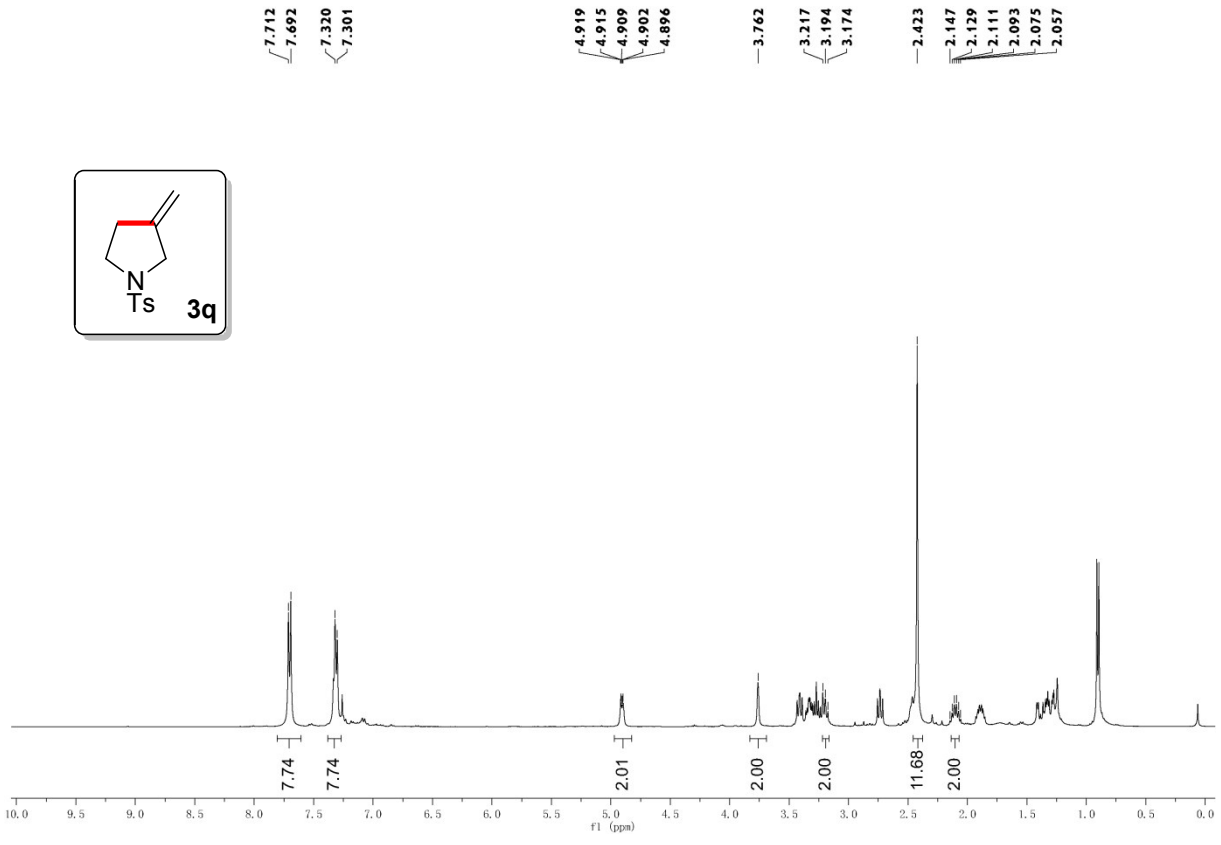
Analytical data for **2q**: **^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):** 7.70 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 7.6 Hz, 2H), 3.41 (dd, *J* = 9.6, 7.6 Hz, 1H), 3.36-3.25 (m, 2H), 2.74 (dd, *J* = 9.2, 8.0 Hz, 1H), 2.42 (s, 3H), 1.93-1.85 (m, 1H), 1.42-1.28 (m, 2H), 0.90 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$, δ ppm):** 143.2, 133.9, 129.5, 127.5, 54.7, 47.6, 33.2, 33.2, 21.5, 17.6.

Copies of the ^{1}H NMR and ^{13}C NMR for the reaction of substrate 1q:

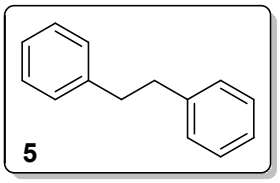





7.712
7.692
7.320
7.301
3.433
3.414
3.409
3.391
3.359
3.335
3.315
3.304
3.272
3.254
2.737
2.713
2.423
1.900
1.882
1.871
1.854
1.313
1.281
0.911
0.894
H
N
Ts
2q
2.98
2.98
1.00
2.00
1.00
4.50
1.01
2.01
3.01
f1 (ppm)


7.712
7.692
7.320
7.301
4.919
4.915
4.909
4.902
4.896
3.762
3.217
3.194
3.174
2.423
2.147
2.129
2.111
2.093
2.075
2.057
N
Ts
3q
7.74
7.74
2.01
2.00
2.00
11.68
2.00
f1 (ppm)

5. Data of 1,2-diphenylethane.



1,2-diphenylethane

$C_{14}H_{14}$

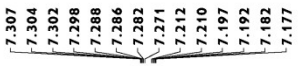
The title compound was isolated by silica gel column chromatography (PE only) from the standard reaction system.

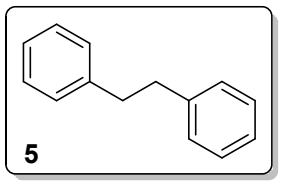
^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.31-7.26 (m, 4H), 7.21-7.18 (m, 6H), 2.93 (s, 4H).

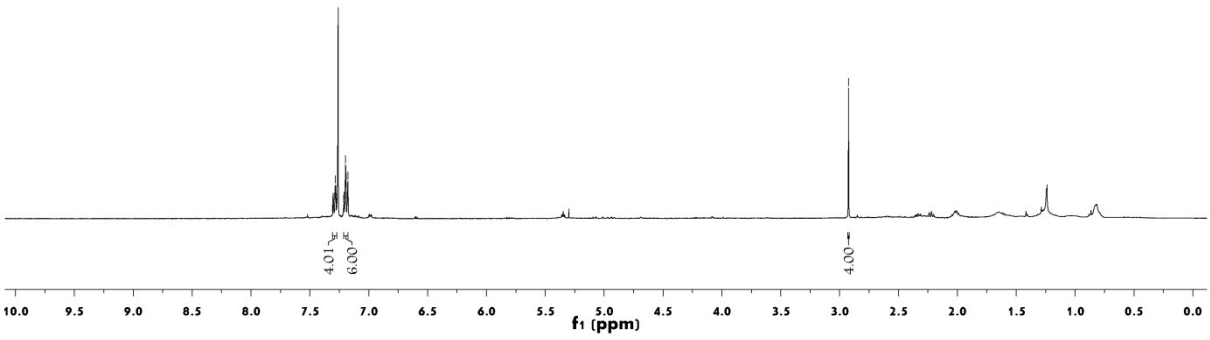
^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 141.7, 128.4, 128.3, 125.9, 37.9.

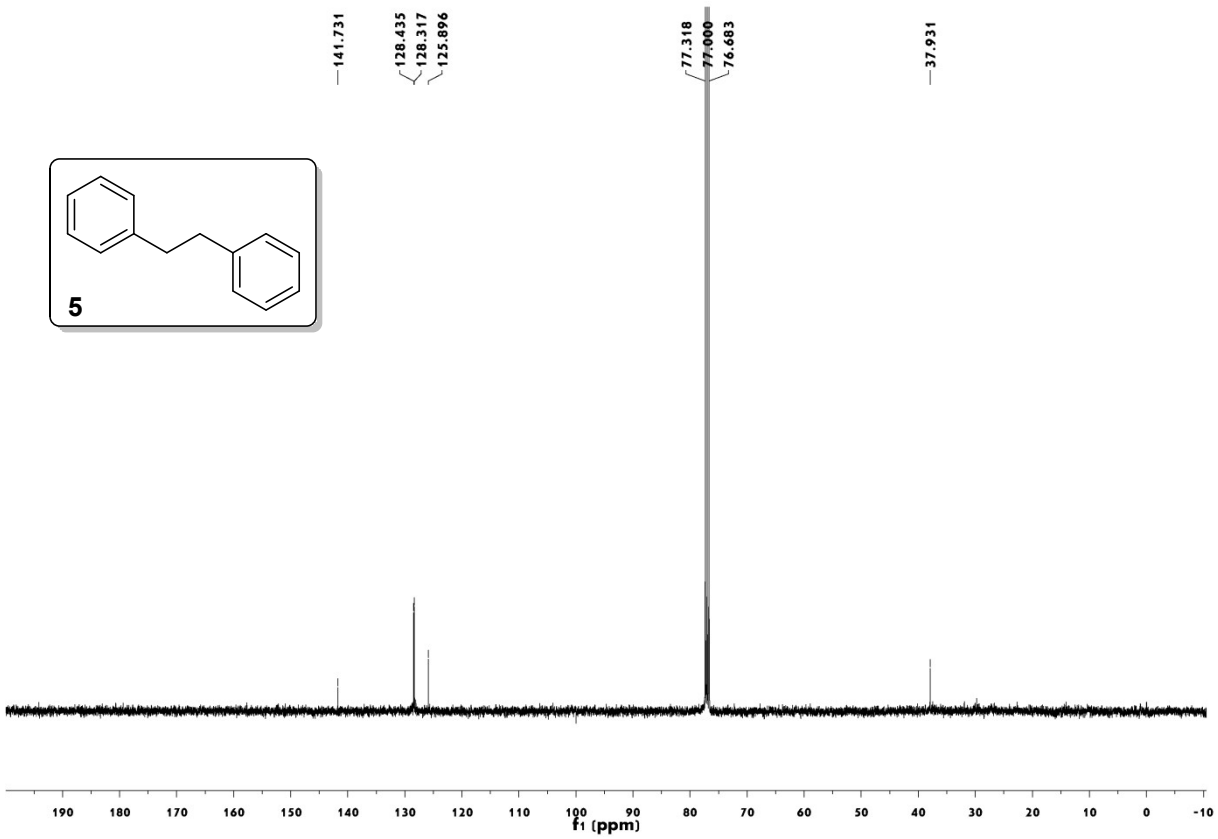
MS (EI) m/z 182 (M+).

Copies of the ^{1}H NMR and ^{13}C NMR:

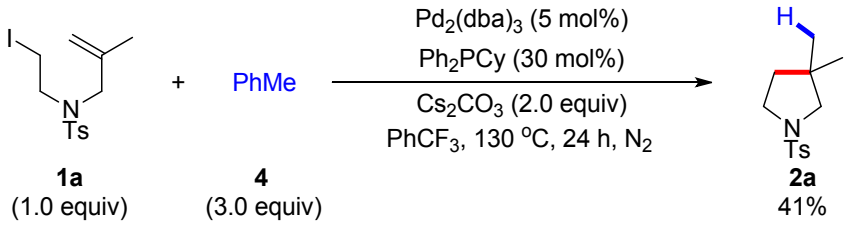







141.731
128.435
128.317
125.896
77.318
77.000
76.683
37.931
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

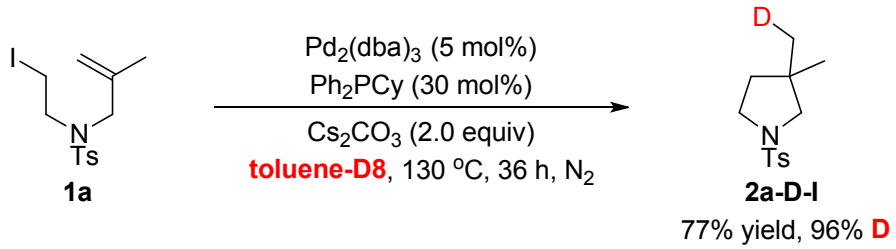
6. Reaction of Stoichiometric Hydrogen Donor.



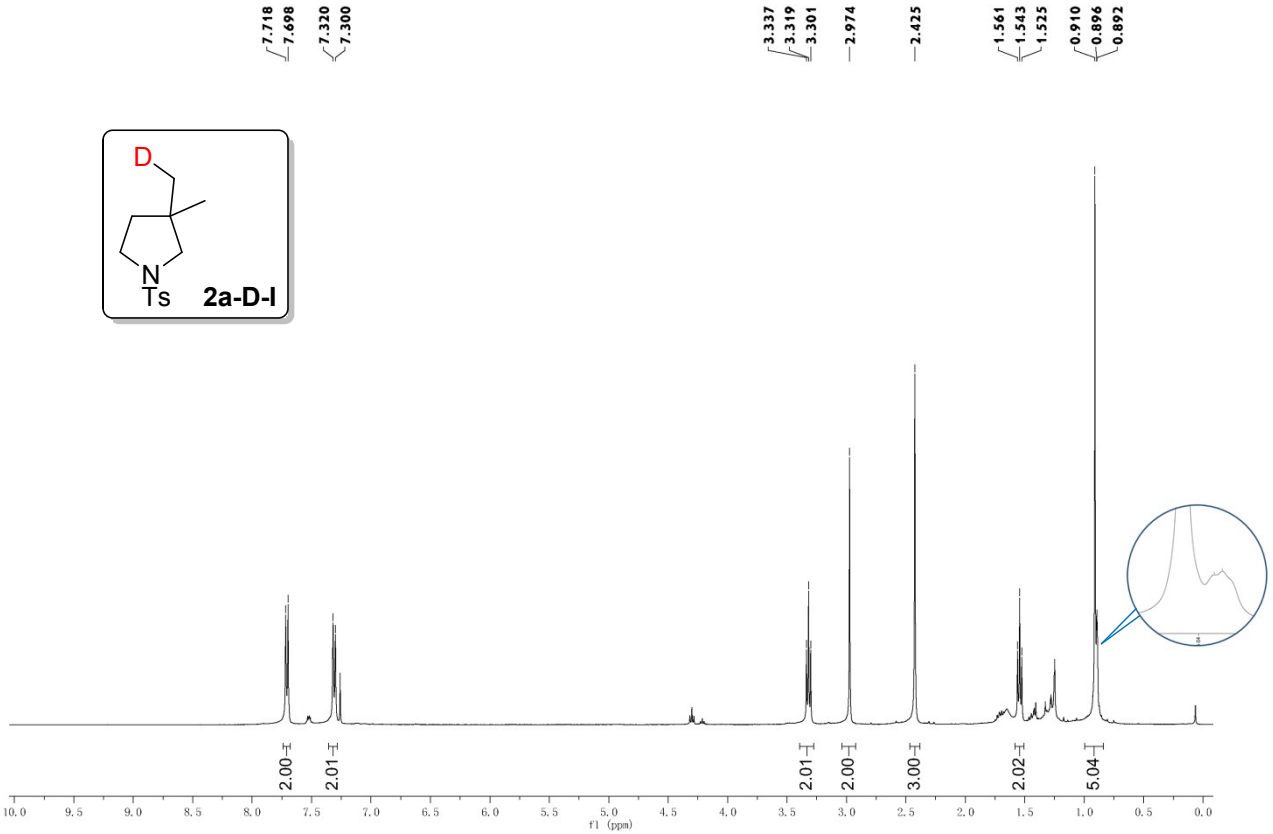
1a (1.0 equiv) **4** (3.0 equiv) **2a** 41%

Unactivated alkyl iodide **1a** (75.9 mg, 0.20 mmol, 1.0 equiv), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 0.05 equiv), Ph_2PCy (16.1 mg, 0.06 mmol, 0.3 equiv) and Cs_2CO_3 (130.3 mg, 0.40 mmol, 2.0 equiv) were added to a reaction tube and vacuum purged three times, backfilling with N_2. Then the trifluorotoluene (3 mL) and PhMe (63.6 μL, 0.6 mmol, 3.0 equiv) was added successively under nitrogen atmosphere. The resulting mixture was stirred at 130 °C for 24 h. After cooling the reaction mixture at rt, it was quenched with a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with EtOAc (10 mL × 3). The combined organic phase was sequentially washed with saturated aqueous solution of NaCl and then concentrated *in vacuo*. The mixture was purified by silica gel column chromatography (PE:EA, 20:1) to give the reductive product **2a** (20.6 mg, 0.08 mmol, 41% yield).

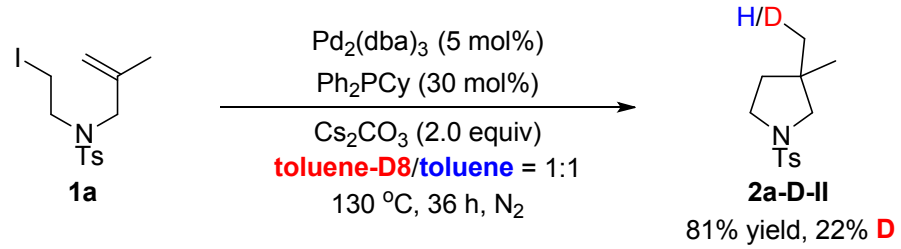
7. Deuterium Scrambling Investigation



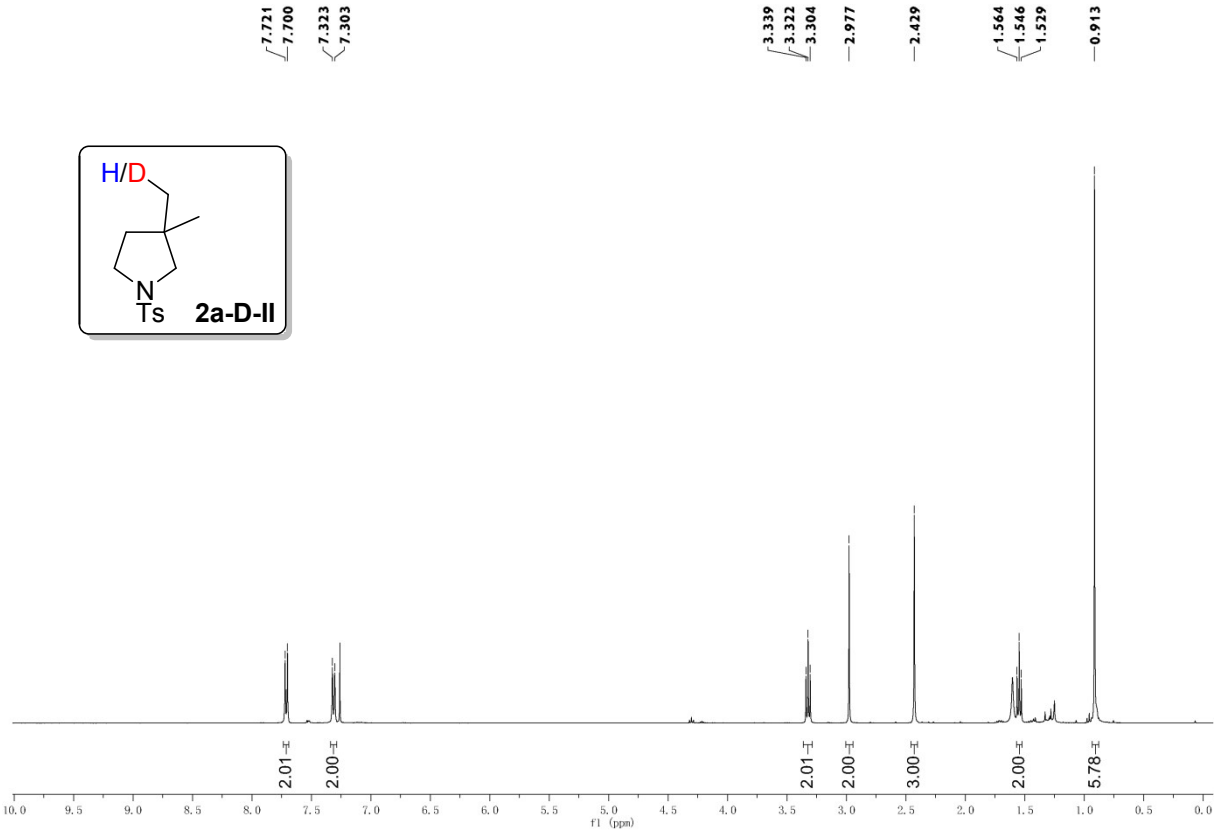
Unactivated alkyl iodide **1a** (75.9 mg, 0.20 mmol, 1.0 equiv), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 0.05 equiv), Ph_2PCy (16.1 mg, 0.06 mmol, 0.3 equiv) and Cs_2CO_3 (130.3 mg, 0.40 mmol, 2.0 equiv) were added to a reaction tube and vacuum purged three times, backfilling with N_2. Then the toluene-D8 (3 mL) was added under nitrogen atmosphere. The resulting mixture was stirred at 130 °C for 24 h. After cooling the reaction mixture at rt, it was quenched with a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with EtOAc (10 mL × 3). The combined organic phase was sequentially washed with saturated aqueous solution of NaCl and then concentrated *in vacuo*. The mixture was purified by silica gel column chromatography (PE:EA, 20:1) to give the reductive product **2a-D-I** with 96% deuterium (39.2 mg, 0.15 mmol, 77% yield). **^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):** 7.71 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.32 (t, *J* = 7.2 Hz, 2H), 2.97 (s, 2H), 2.43 (s, 3H), 1.54 (t, *J* = 7.2 Hz, 2H), 0.91-0.89 (m, 5.04H).



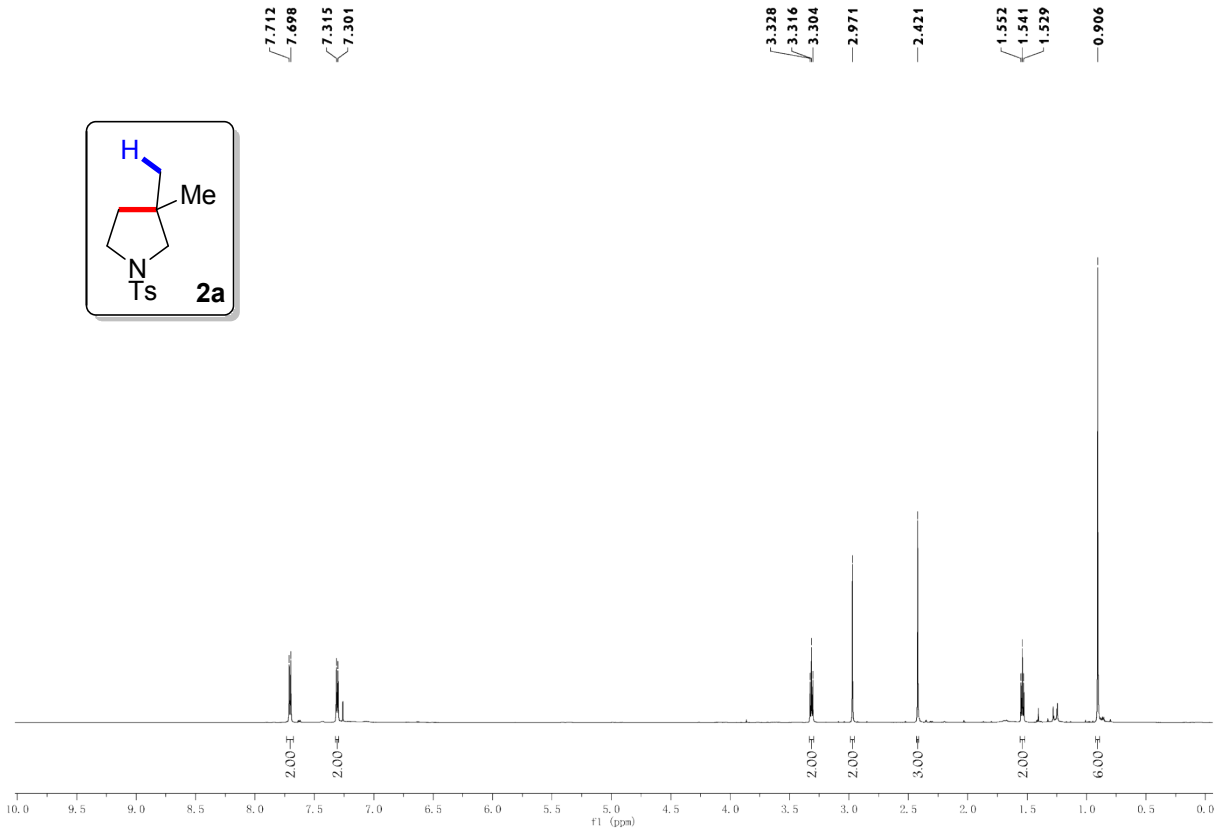
8. Kinetic Isotopic Effect Experiment.

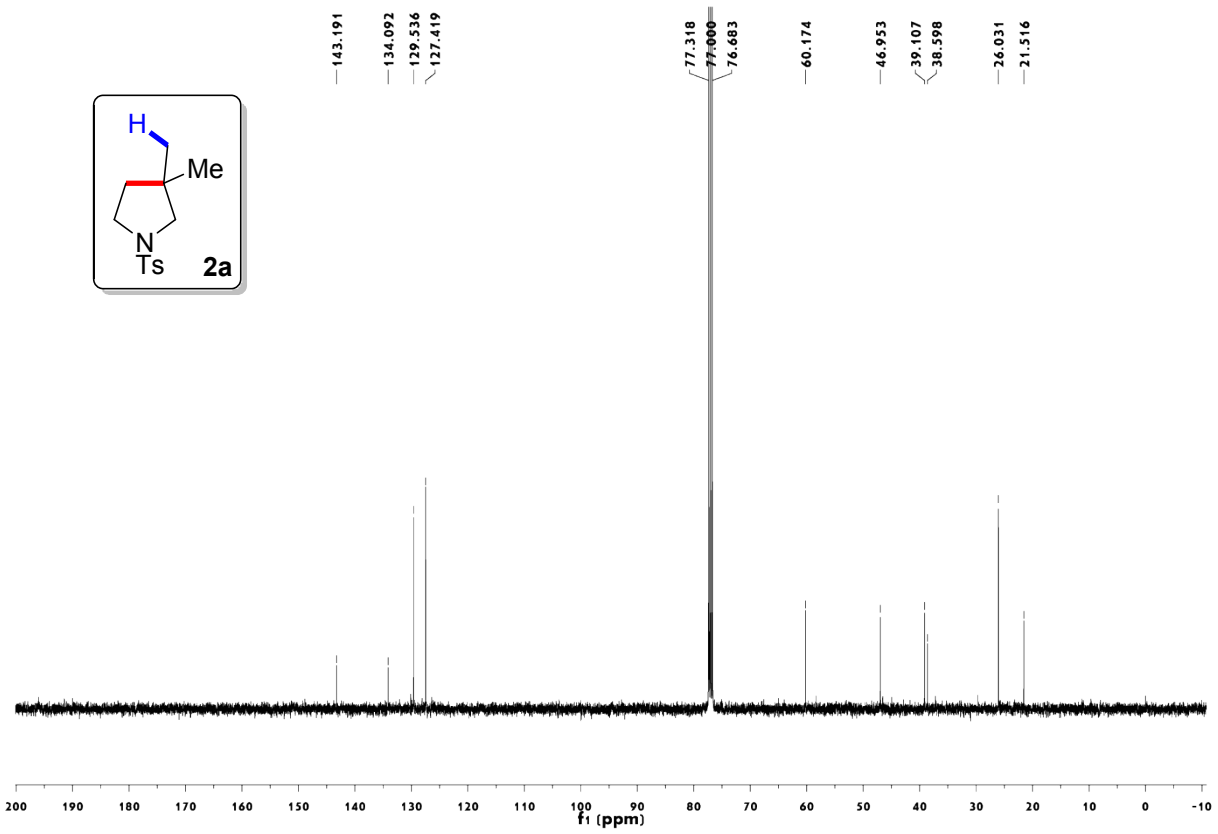


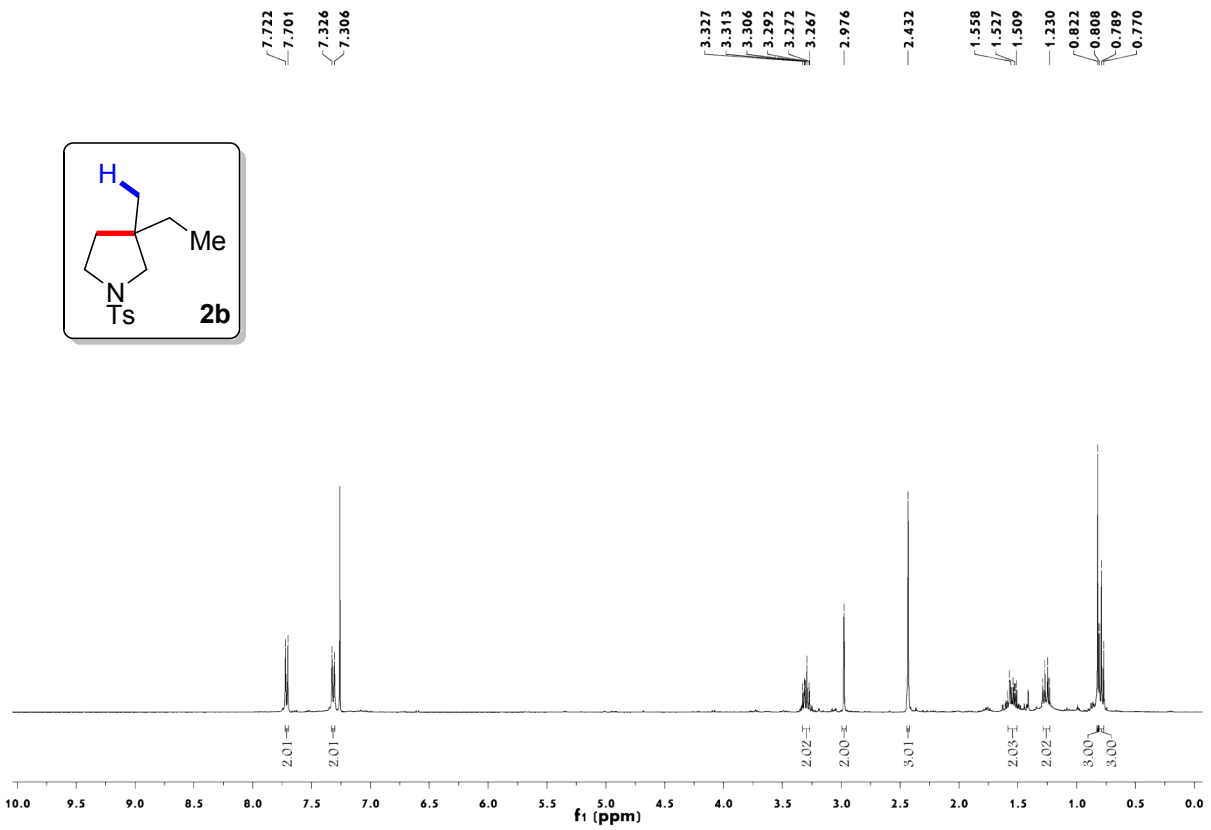
Unactivated alkyl iodide **1a** (75.9 mg, 0.20 mmol, 1.0 equiv), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 0.05 equiv), Ph_2PCy (16.1 mg, 0.06 mmol, 0.3 equiv) and Cs_2CO_3 (130.3 mg, 0.40 mmol, 2.0 equiv) were added to a reaction tube and vacuum purged three times, backfilling with N_2. Then the toluene-D8 (1.5 mL) and PhMe (1.5 mL) was added under nitrogen atmosphere. The resulting mixture was stirred at 130 °C for 24 h. After cooling the reaction mixture at rt, it was quenched with a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with EtOAc (10 mL × 3). The combined organic phase was sequentially washed with saturated aqueous solution of NaCl and then concentrated *in vacuo*. The mixture was purified by silica gel column chromatography (PE:EA, 20:1) to give the reductive product **2a-D-II** (41.2 mg, 0.16 mmol, 81% yield). **^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):** 7.71 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 3.32 (t, J = 6.8 Hz, 2H), 2.98 (s, 2H), 2.43 (s, 3H), 1.55 (t, J = 7.2 Hz, 2H), 0.91 (s, 5.78H). **Kinetic Isotopic Effect:** k_H/k_D = 0.78/(1-0.78) = 3.5

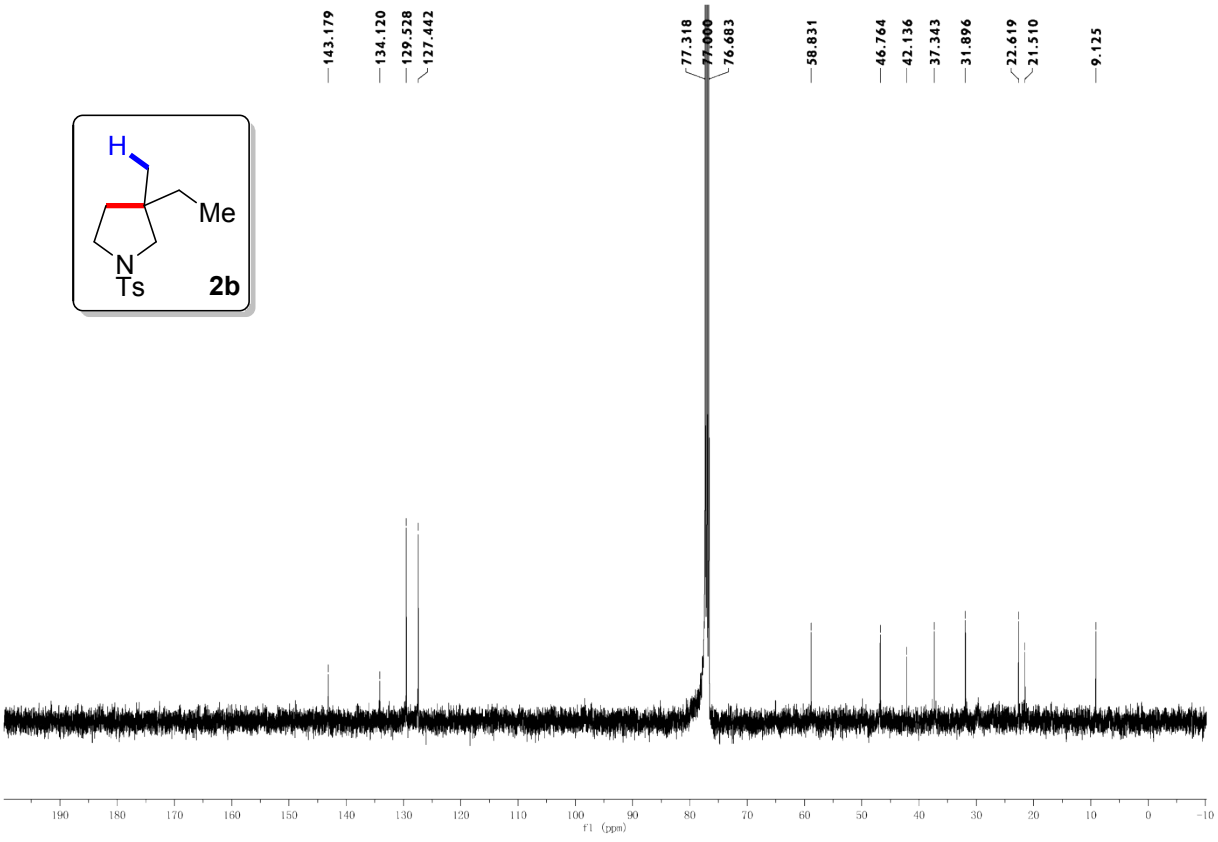


9. Copies of the ^{1}H NMR and ^{13}C NMR for Redcutive Product.






7.722
7.701
7.326
7.306
3.327
3.313
3.306
3.292
3.272
3.267
2.976
2.432
1.558
1.527
1.509
1.230
0.822
0.808
0.789
0.770
H
Me
N
Ts
2b
2.01
2.01
2.02
2.00
3.01
2.03
2.02
3.00
3.00
f1 (ppm)


143.179
134.120
129.528
127.442
77.318
77.000
76.683
58.831
46.764
42.136
37.343
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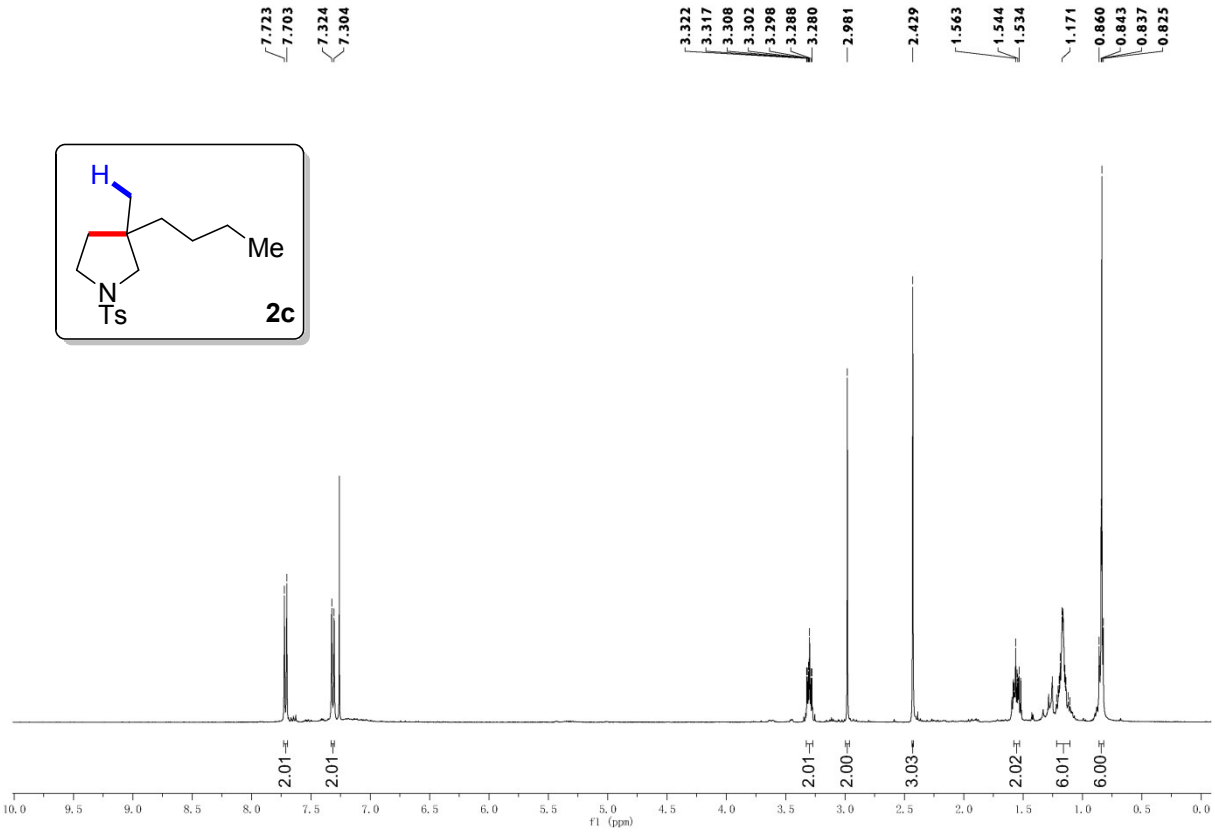
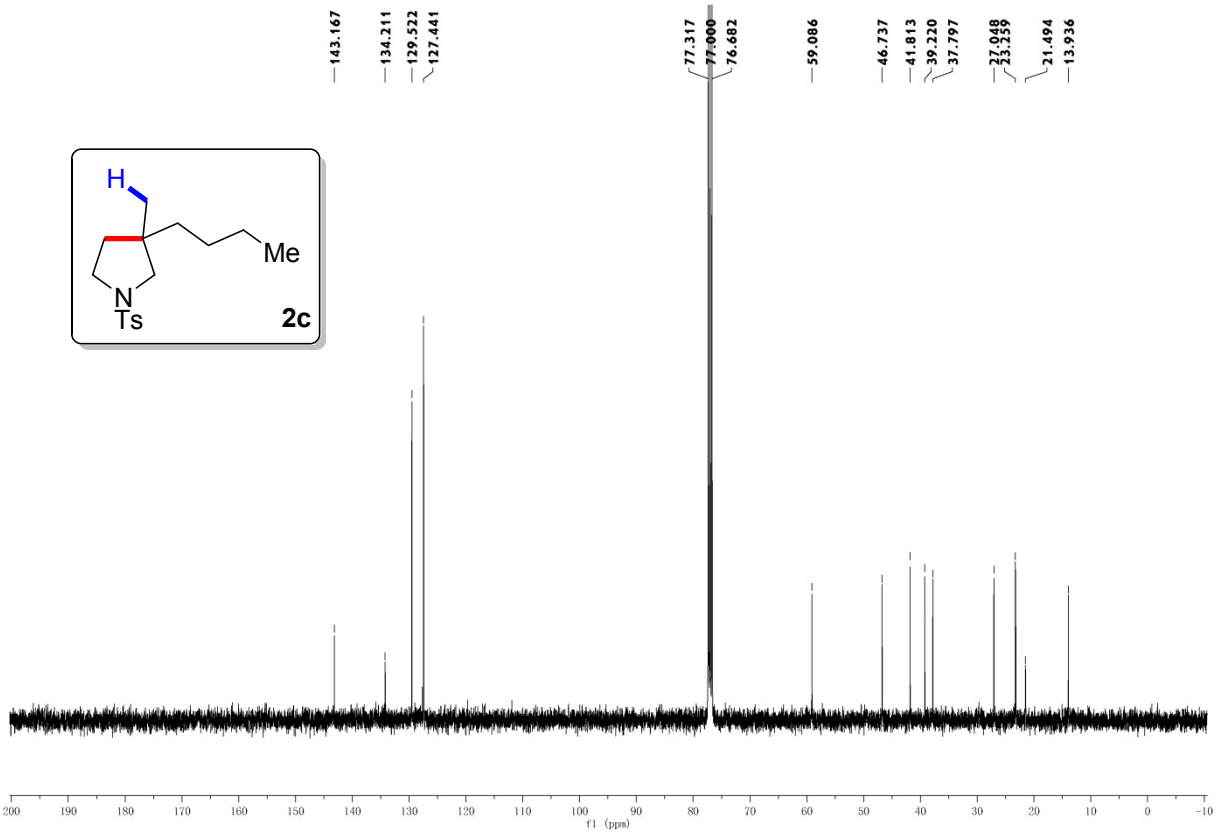
22.619
21.510
9.125
H
Me
N
Ts
2b
f1 (ppm)


7.723
7.703
7.324
7.304
3.322
3.317
3.308
3.302
3.298
3.288
3.280
2.981
2.429
1.563
1.544
1.534
1.171
0.860
0.843
0.837
0.825
H
Me
N
Ts
2c
2.01
2.01
2.01
2.00
3.03
2.02
6.01
6.00
f1 (ppm)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129.522
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77.000
76.682
59.086
46.737
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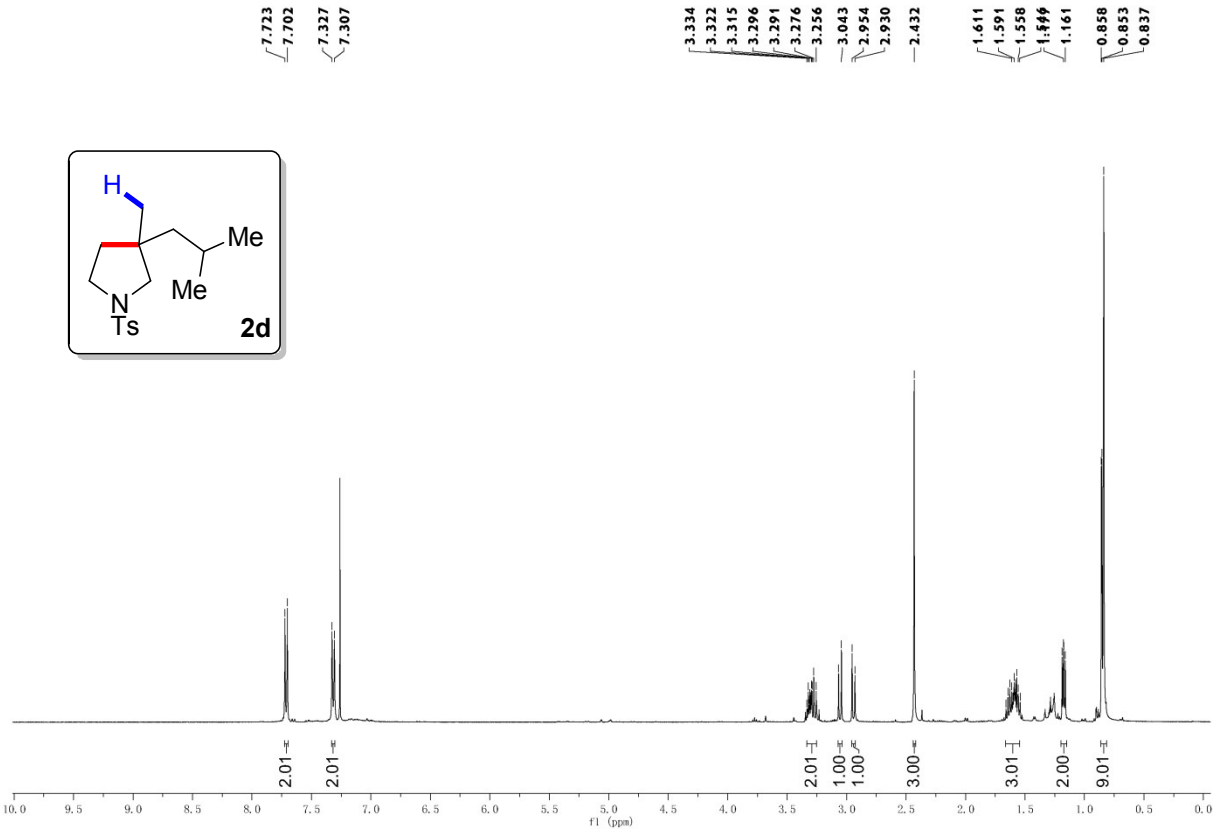
37.797
27.048
23.259
21.494
13.936
H
Me
N
Ts
2c
f1 (ppm)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7.327
7.307
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3.322
3.315
3.296
3.291
3.276
3.256
3.043
2.954
2.930
2.432
1.611
1.591
1.558
1.161
0.858
0.853
0.837
H
Me
Me
N
Ts
2d
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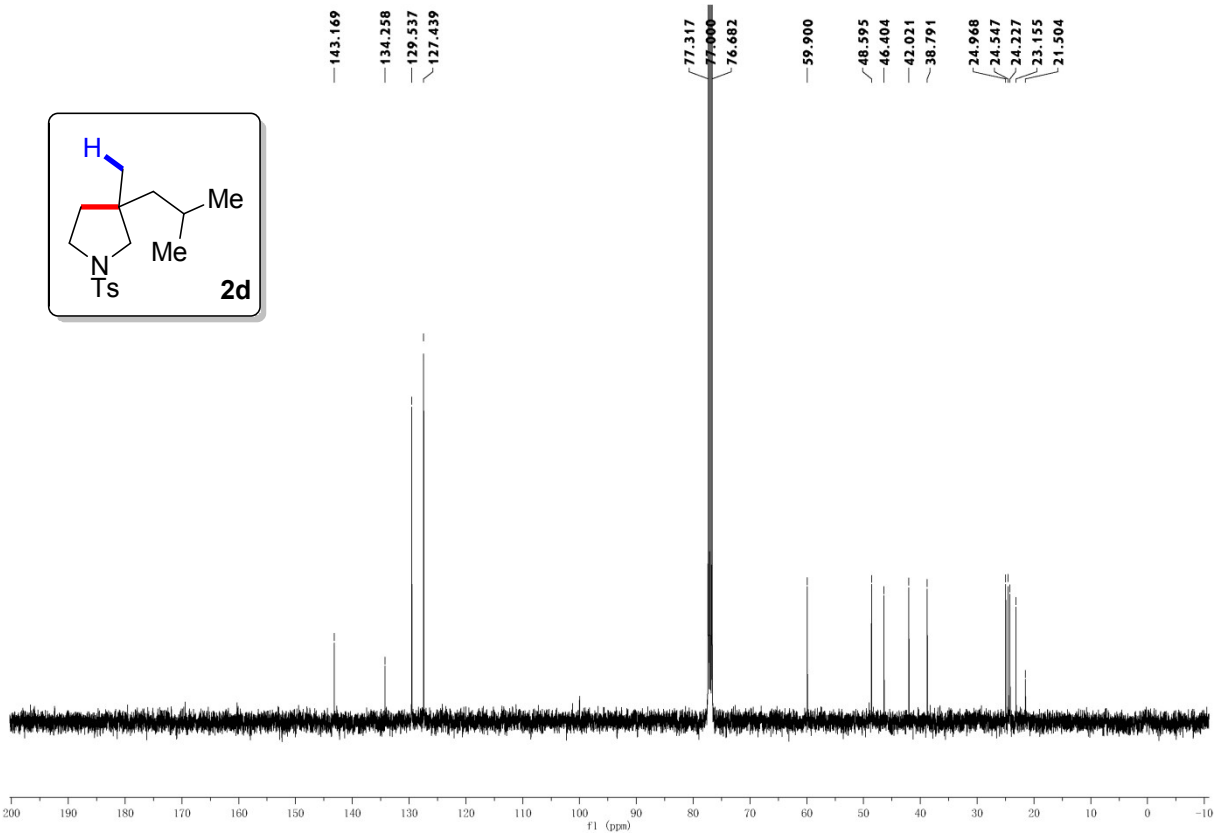
2.01
2.01
1.00
1.00
3.00
3.01
2.00
9.01
f1 (ppm)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134.258
129.537
127.439
77.317
77.000
76.682
59.900
48.595
46.404
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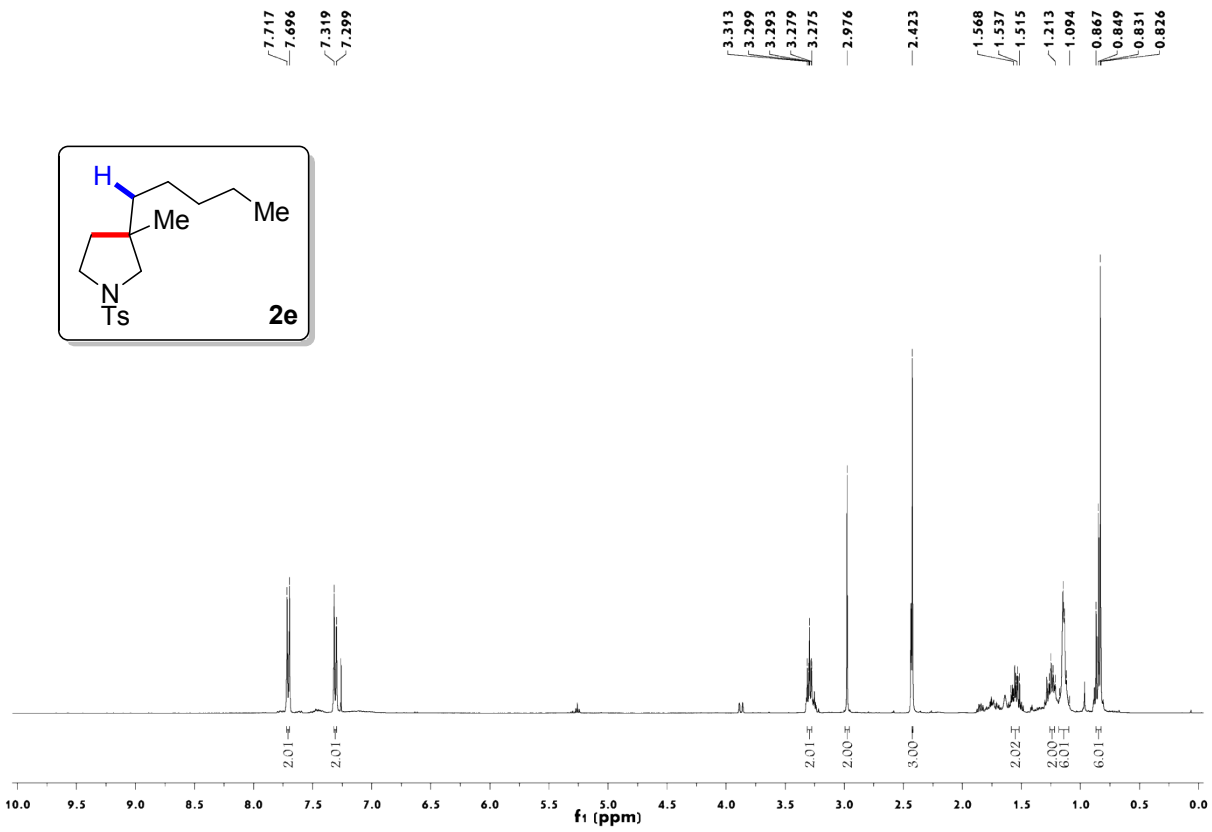
24.547
24.227
23.155
21.504
H
Me
Me
N
Ts
2d
f1 (ppm)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7.696
7.319
7.299
3.313
3.299
3.293
3.279
3.275
2.976
2.423
1.568
1.537
1.515
1.213
1.094
0.867
0.849
0.831
0.826
H
Me
Me
N
Ts
2e
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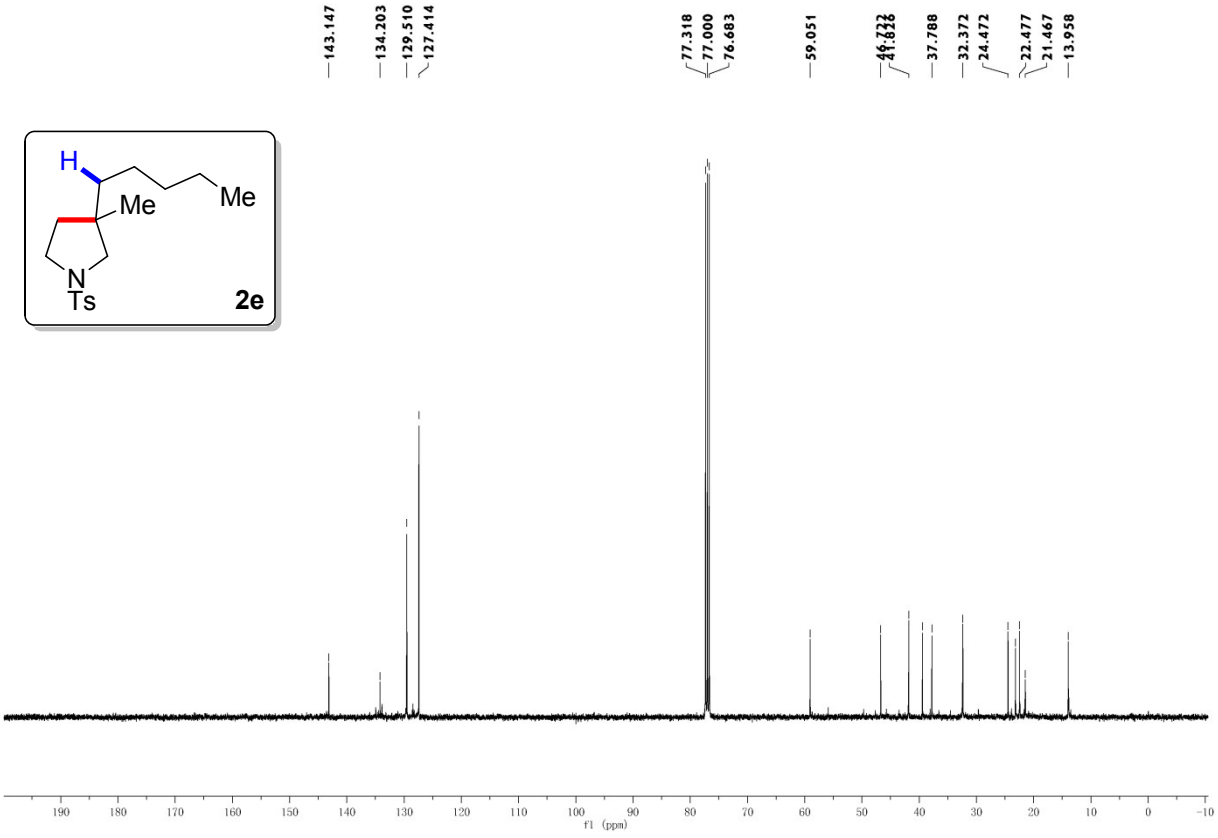
2.01
2.01
2.00
3.00
2.02
2.00
6.01
6.01
f1 (ppm)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134.203
129.510
127.414
77.318
77.000
76.683
59.051
46.722
41.828
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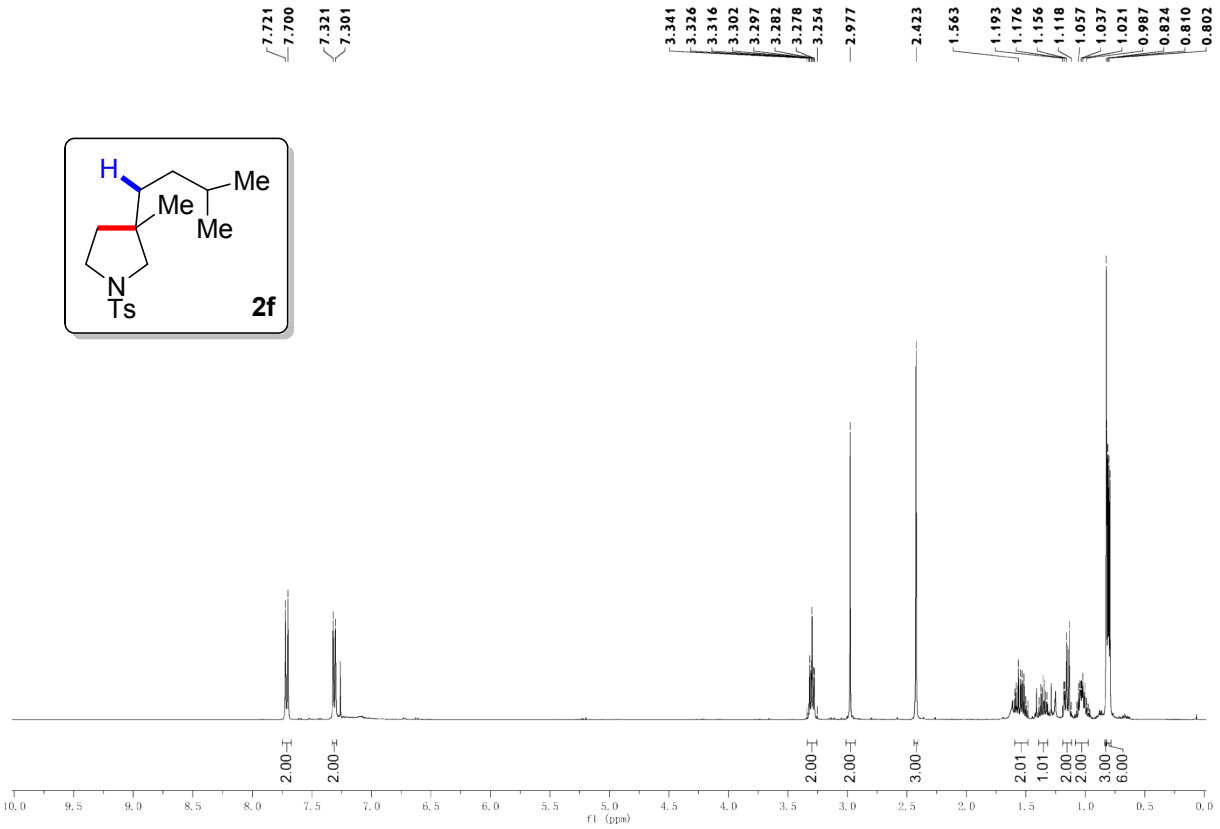
24.472
22.477
21.467
13.958
H
Me
Me
N
Ts
2e
f1 (ppm)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7.700
7.321
7.301
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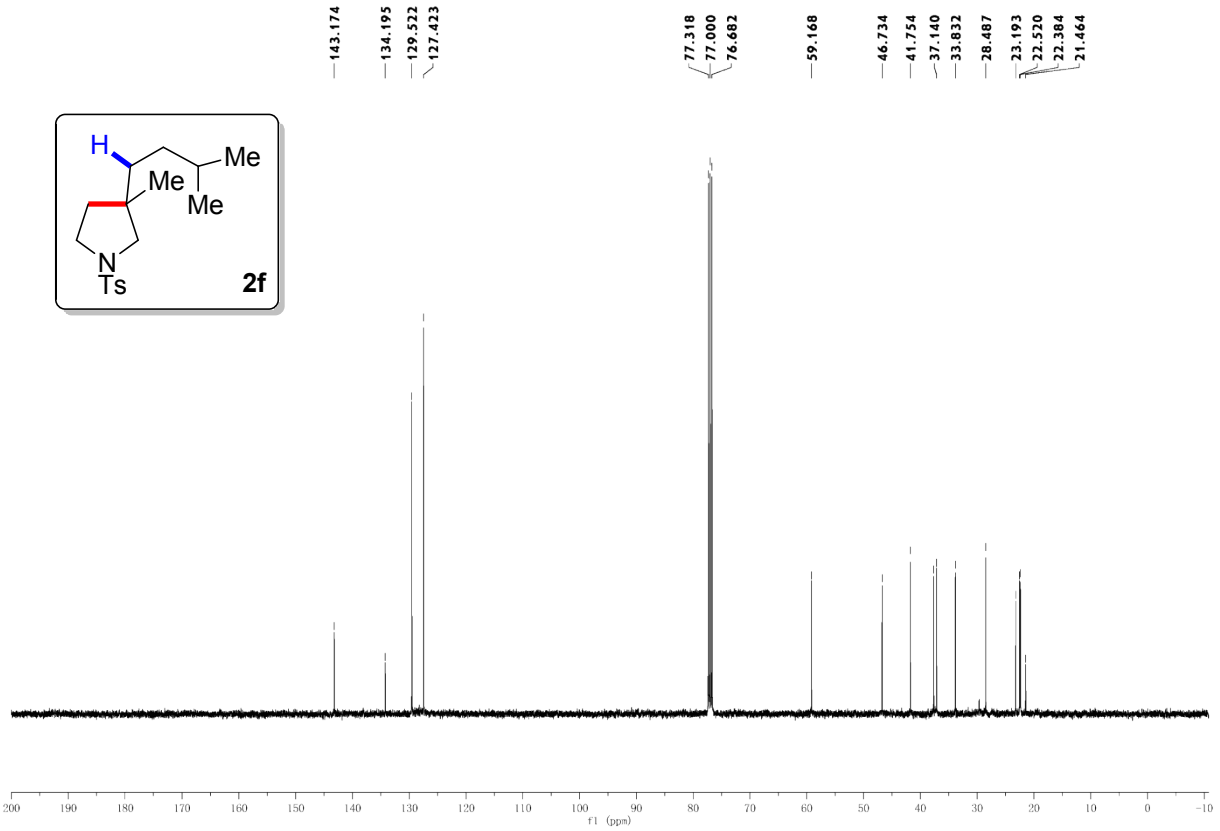
3.326
3.316
3.302
3.297
3.282
3.278
3.254
2.977
2.423
1.563
1.193
1.176
1.156
1.118
1.057
1.037
1.021
0.987
0.824
0.810
0.802
H
Me
Me
Me
N
Ts
2f
2.00
2.00
2.00
2.00
3.00
2.01
1.01
2.00
2.00
3.00
6.00
f1 (ppm)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134.195
129.522
127.423
77.318
77.000
76.682
59.168
46.734
41.754
37.140
33.832
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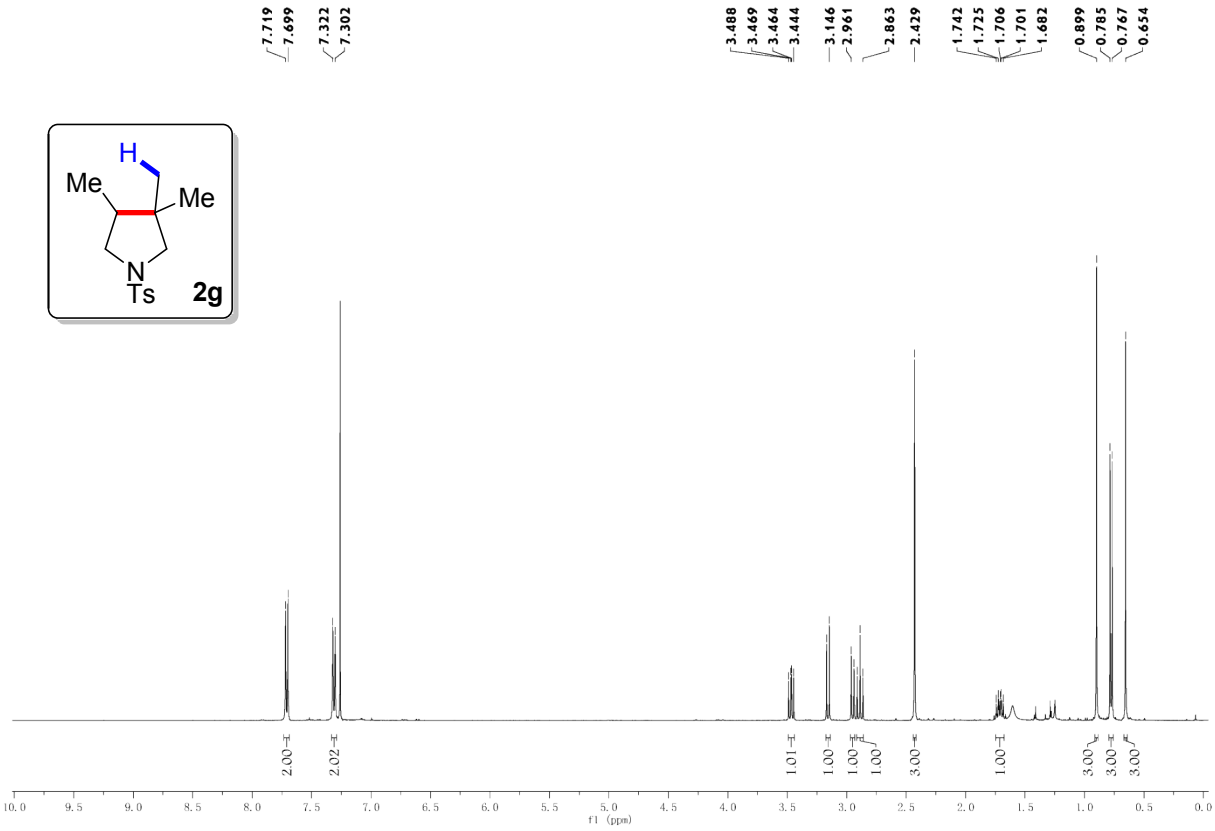
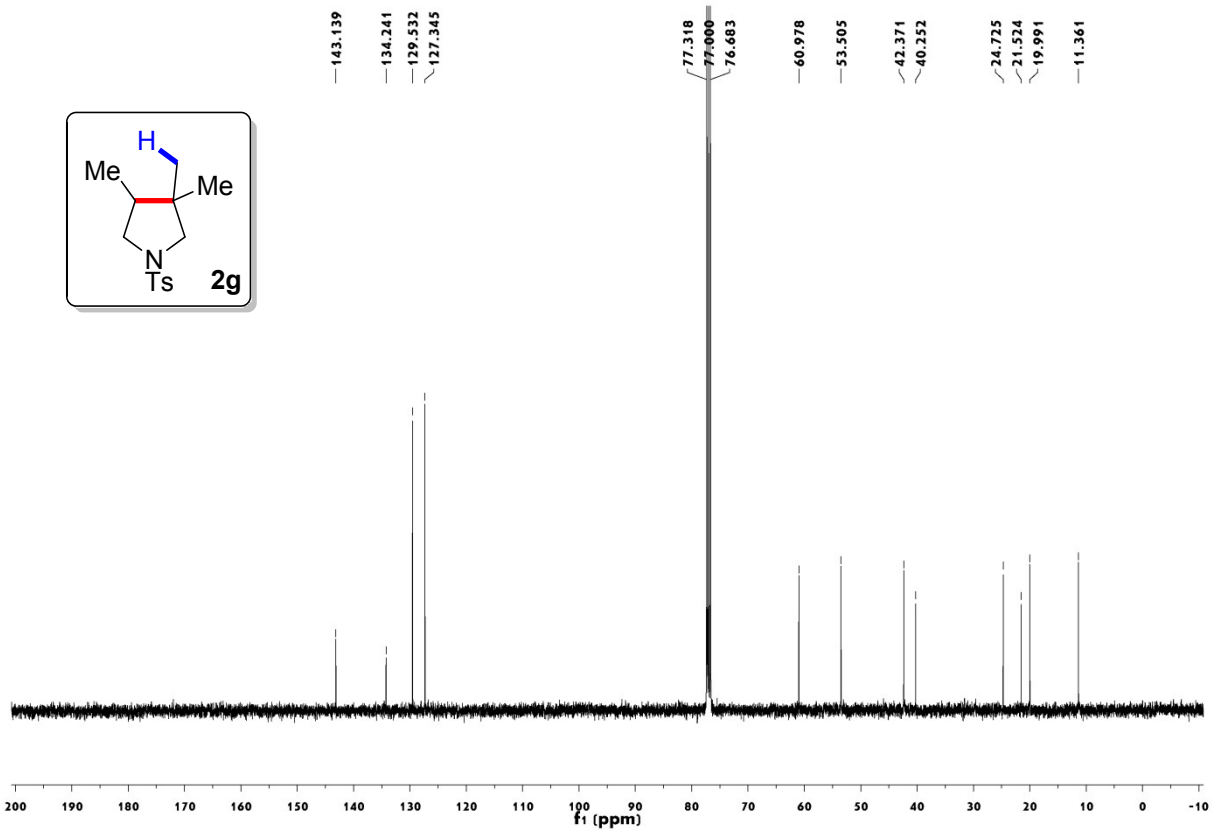
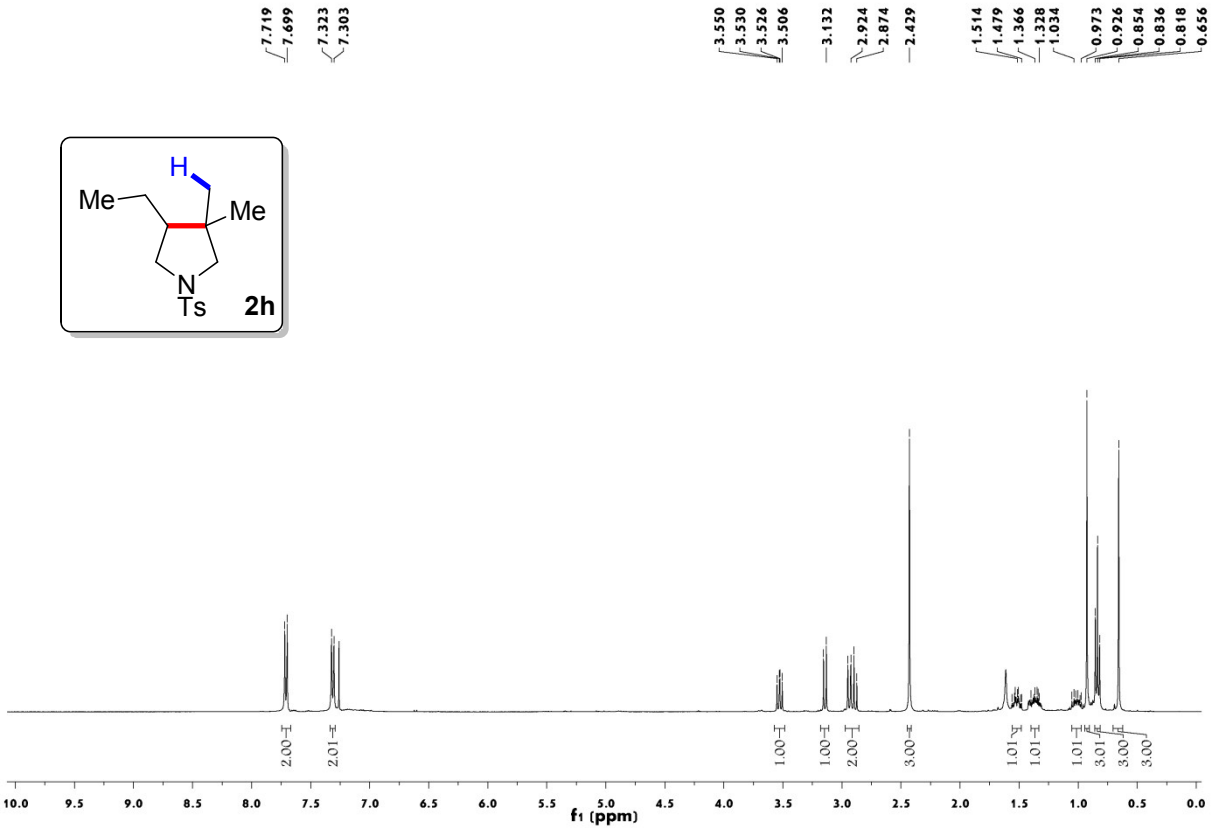
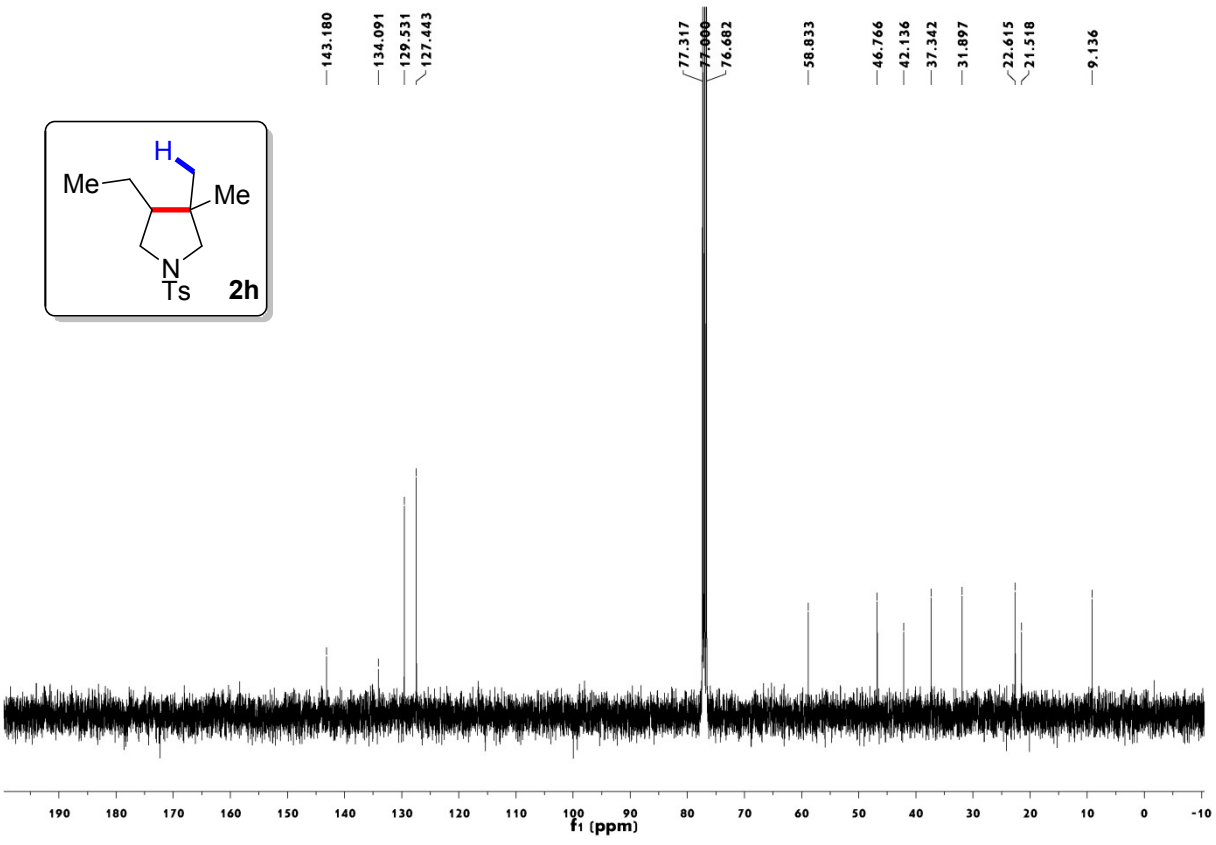
23.193
22.520
22.384
21.464
H
Me
Me
Me
N
Ts
2f
f1 (ppm)


7.719
7.699
7.322
7.302
3.488
3.469
3.464
3.444
3.146
2.961
2.863
2.429
1.742
1.725
1.706
1.701
1.682
0.899
0.785
0.767
0.654
H
Me
Me
N
Ts
2g
2.00
2.02
1.01
1.00
1.00
1.00
3.00
1.00
3.00
3.00
3.00
f1 (ppm)


143.139
134.241
129.532
127.345
77.318
77.000
76.683
60.978
53.505
42.371
40.252
24.725
21.524
19.991
11.361
H
Me
Me
N
Ts
2g
f1 (ppm)


7.719
7.699
7.323
7.303
3.550
3.530
3.526
3.506
3.132
2.924
2.874
2.429
1.514
1.479
1.366
1.328
1.034
0.973
0.926
0.854
0.836
0.818
0.656
H
Me
Me
N
Ts
2h
2.00
2.01
1.00
1.00
2.00
3.00
1.01
1.01
1.01
3.01
3.00
3.00
f1 (ppm)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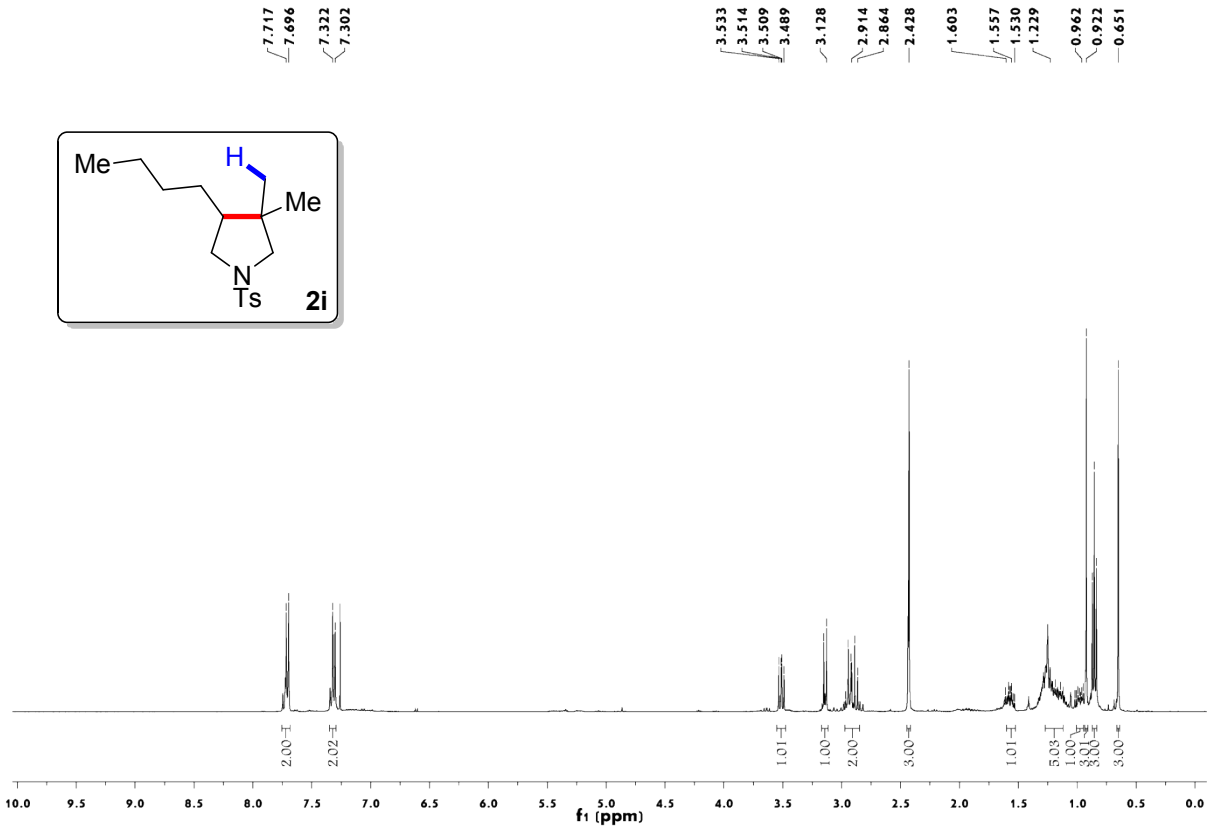
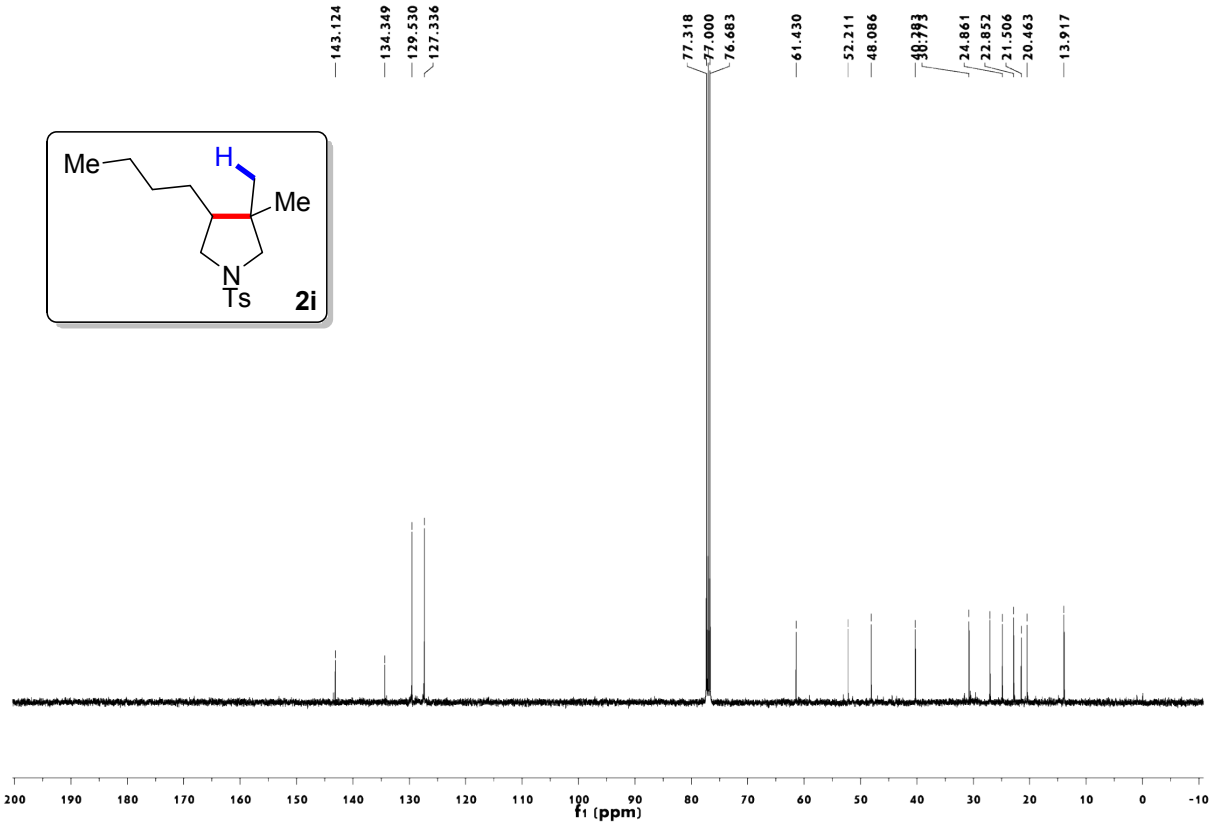
134.091
129.531
127.443
77.317
77.000
76.682
58.833
46.766
42.136
37.342
31.897
22.615
21.518
9.136
H
Me
Me
N
Ts
2h
f1 (ppm)


7.717
7.696
7.322
7.302
3.533
3.514
3.509
3.489
3.128
2.914
2.864
2.428
1.603
1.557
1.530
1.229
0.962
0.922
0.651
Me
H
Me
N
Ts
2i
2.00
2.02
1.01
1.00
2.00
3.00
1.01
5.03
1.00
3.00
3.00
f1 (ppm)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134.349
129.530
127.336
77.318
77.000
76.683
61.430
52.211
48.086
40.393
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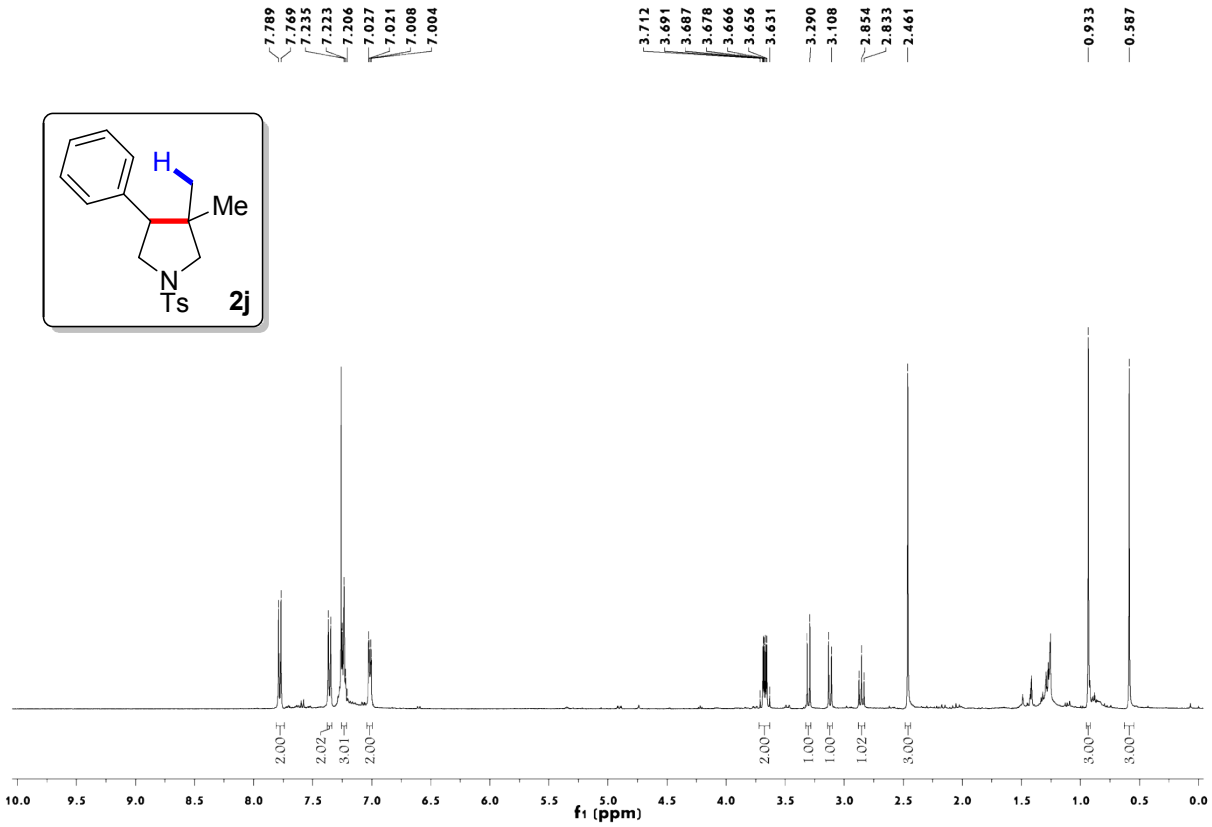
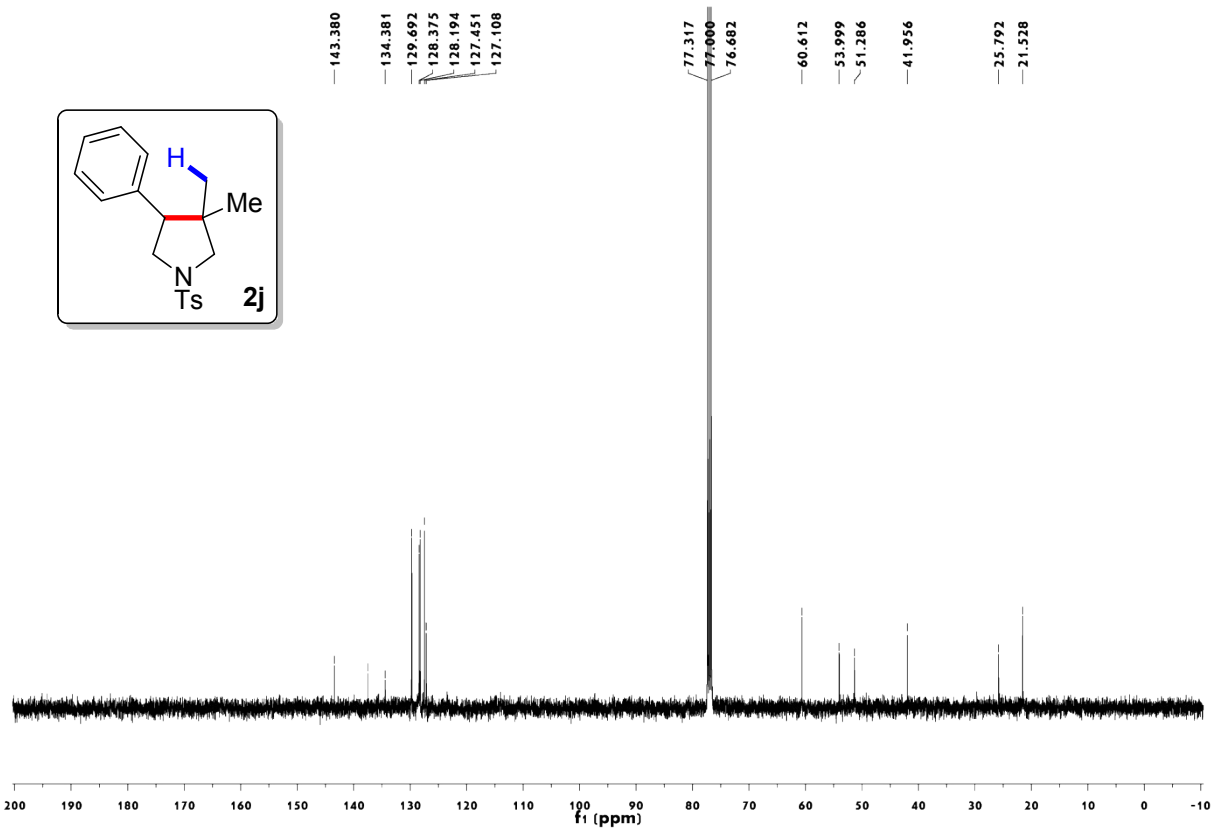
24.861
22.852
21.506
20.463
13.917
Me
H
Me
N
Ts
2i
f1 (ppm)


7.789
7.769
7.235
7.223
7.206
7.027
7.021
7.008
7.004
3.712
3.691
3.687
3.678
3.666
3.656
3.631
3.290
3.108
2.854
2.833
2.461
0.933
0.587
H
Me
N
Ts
2j
2.00
2.02
3.01
2.00
2.00
1.00
1.00
1.02
3.00
3.00
3.00
f1 (ppm)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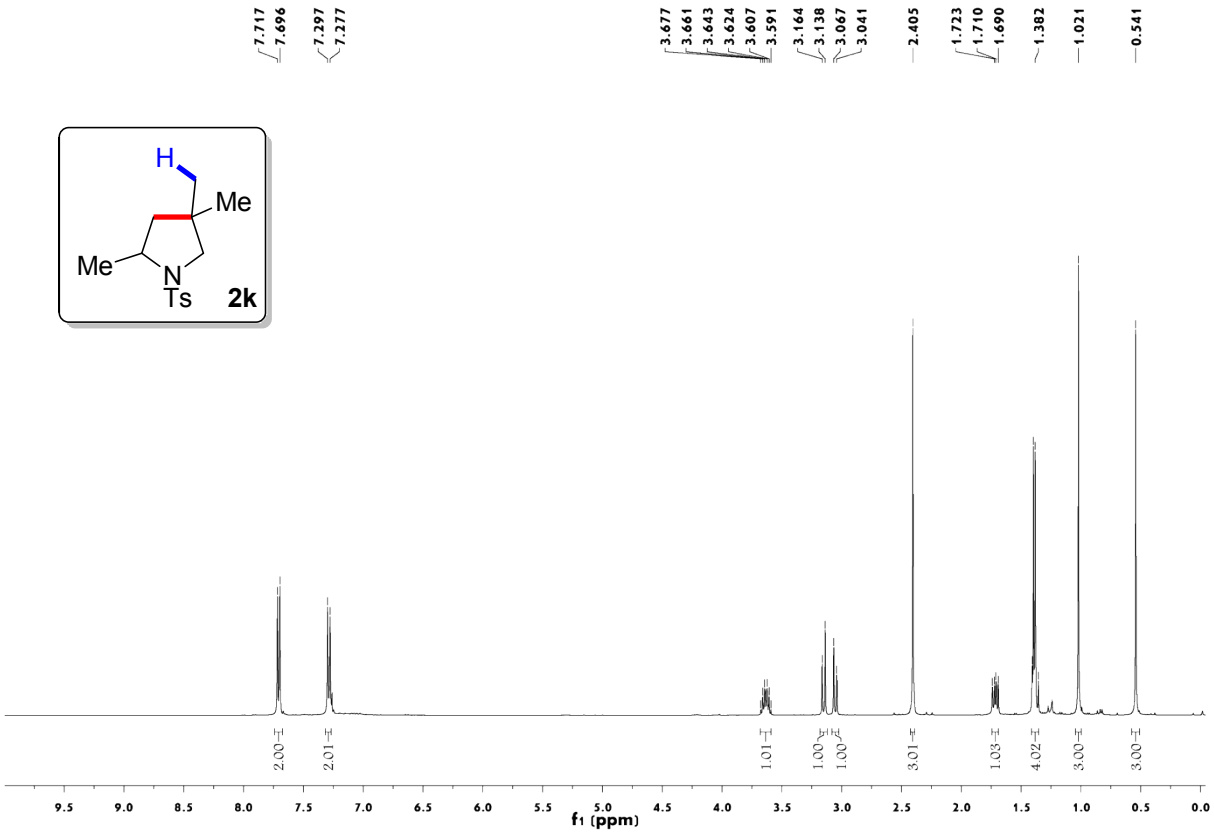
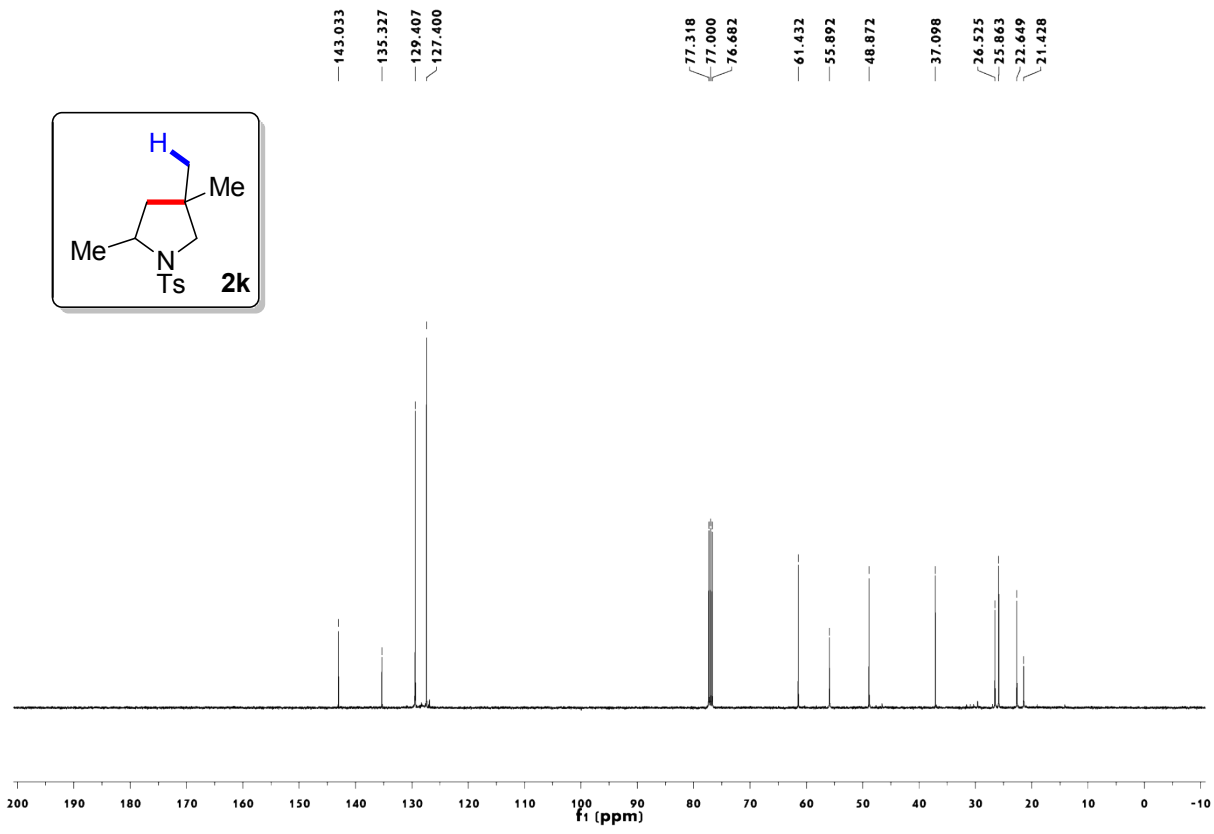
129.692
128.375
128.194
127.451
127.108
77.317
77.000
76.682
60.612
53.999
51.286
41.956
25.792
21.528
H
Me
N
Ts
2j
f1 (ppm)


7.717
7.696
7.297
7.277
3.677
3.661
3.643
3.624
3.607
3.591
3.164
3.138
3.067
3.041
2.405
1.723
1.710
1.690
1.382
1.021
0.541
H
Me
Me
N
Ts
2k
2.00
2.01
1.01
1.00
1.00
3.01
1.03
4.02
3.00
3.00
f1 (ppm)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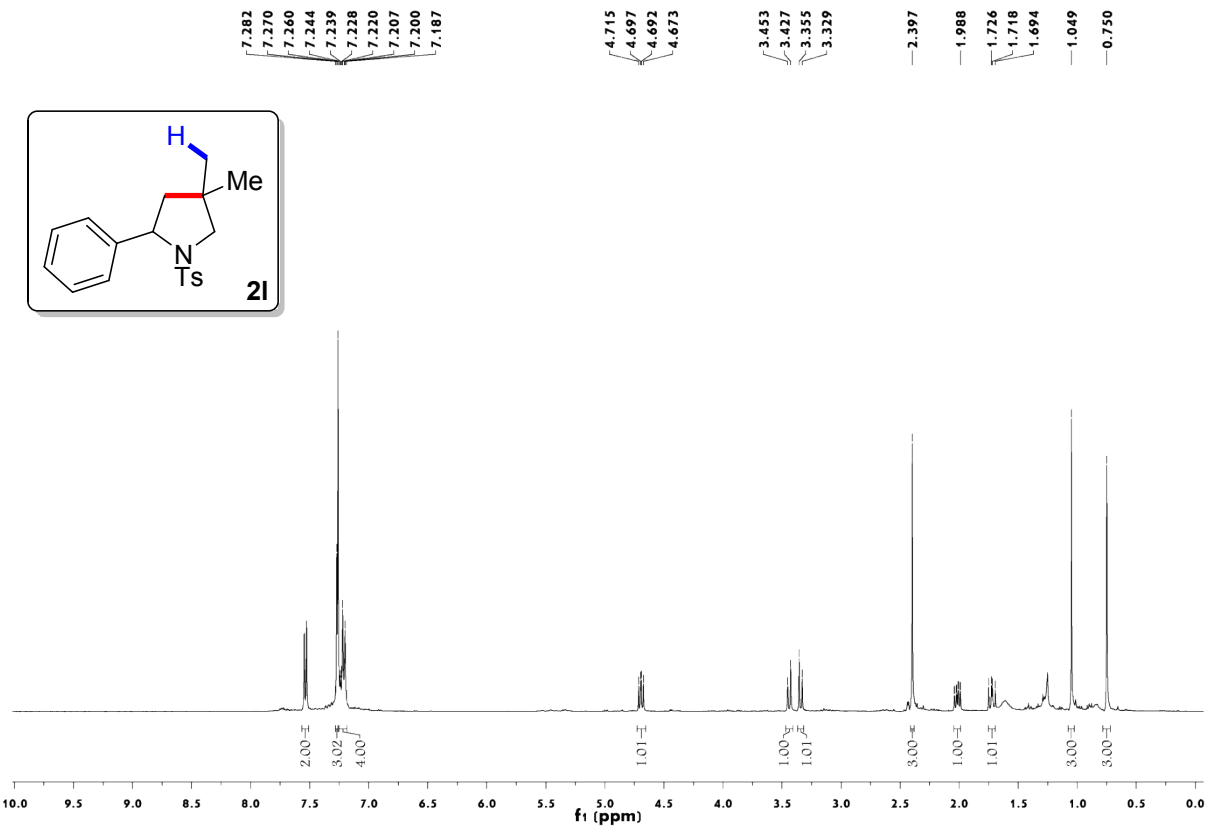
127.400
77.318
77.000
76.682
61.432
55.892
48.872
37.098
26.525
25.863
22.649
21.428
H
Me
Me
N
Ts
2k
f1 (ppm)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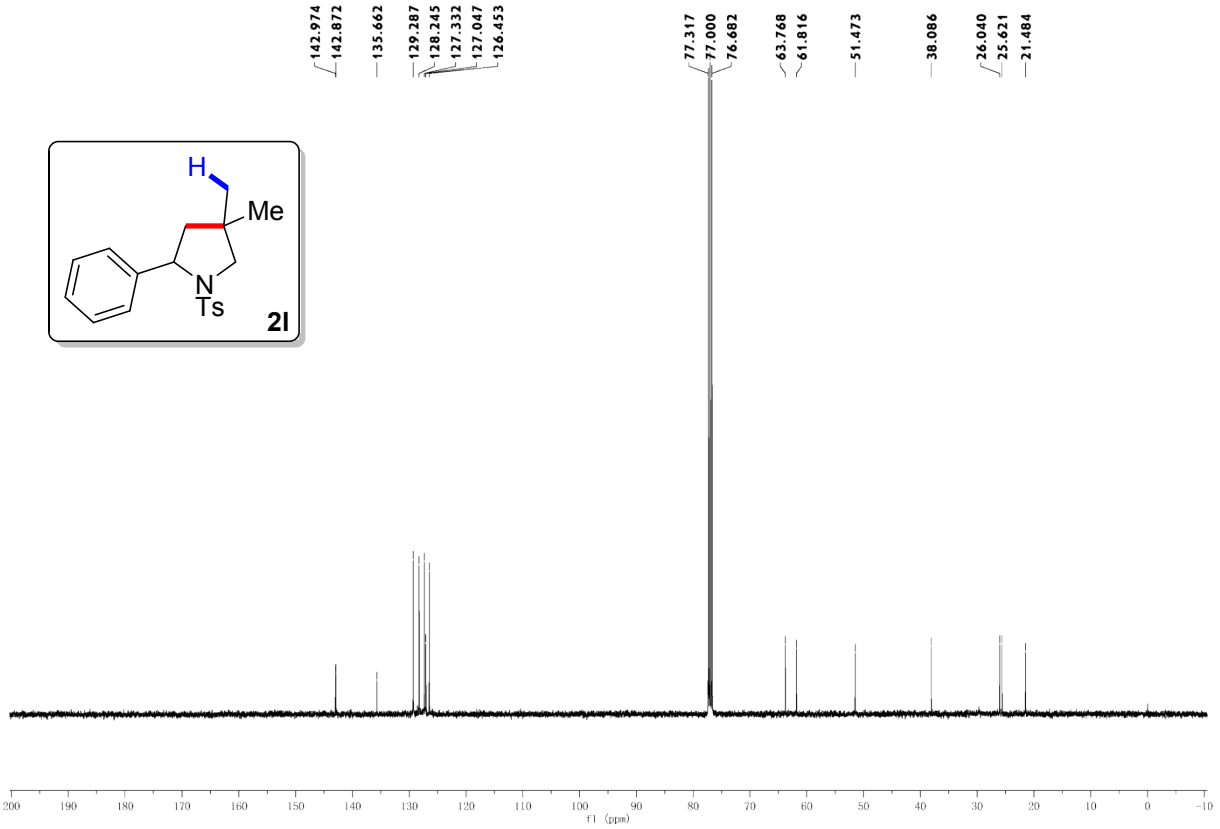
7.270
7.260
7.244
7.239
7.228
7.220
7.207
7.200
7.187
4.715
4.697
4.692
4.673
3.453
3.427
3.355
3.329
2.397
1.988
1.726
1.718
1.694
1.049
0.750
H
Me
N
Ts
2l
2.00
3.02
4.00
1.01
1.00
1.01
3.00
1.00
1.01
3.00
3.00
f1 (ppm)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128.245
127.332
127.047
126.453
77.317
77.000
76.682
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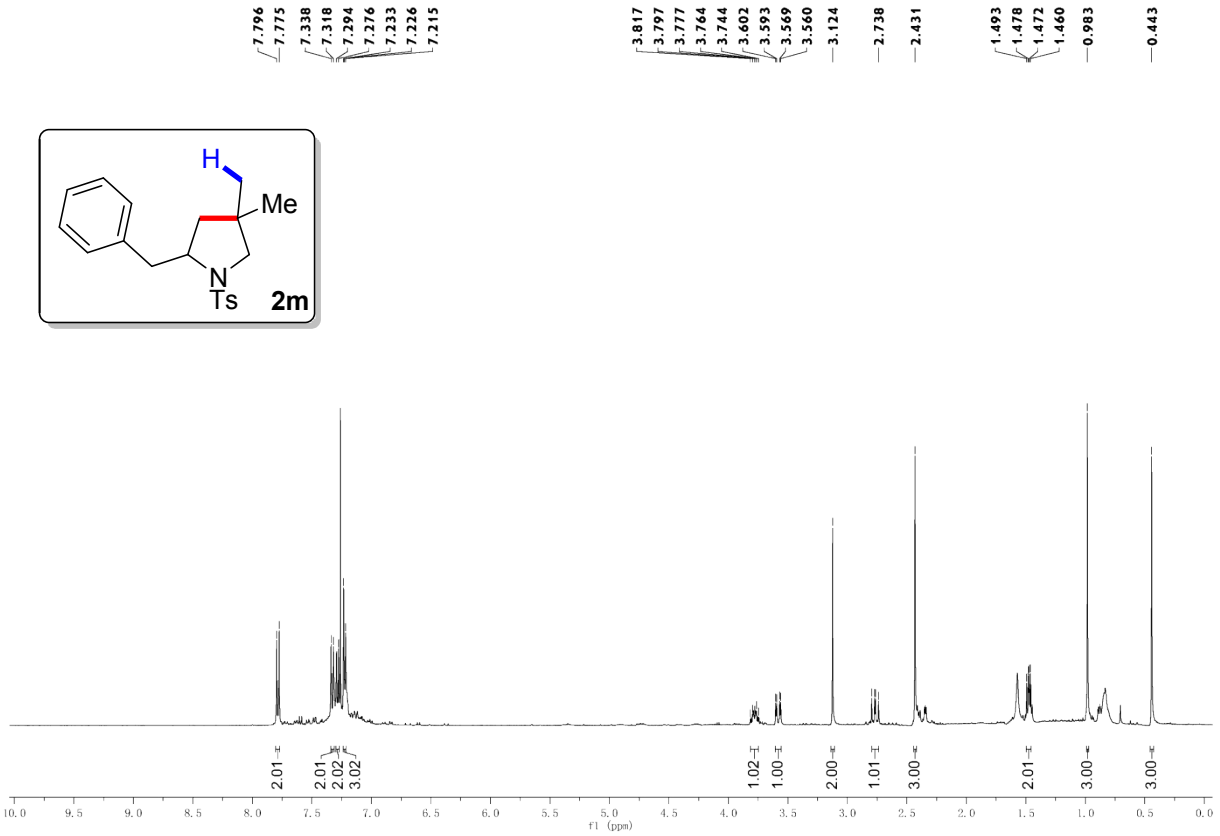
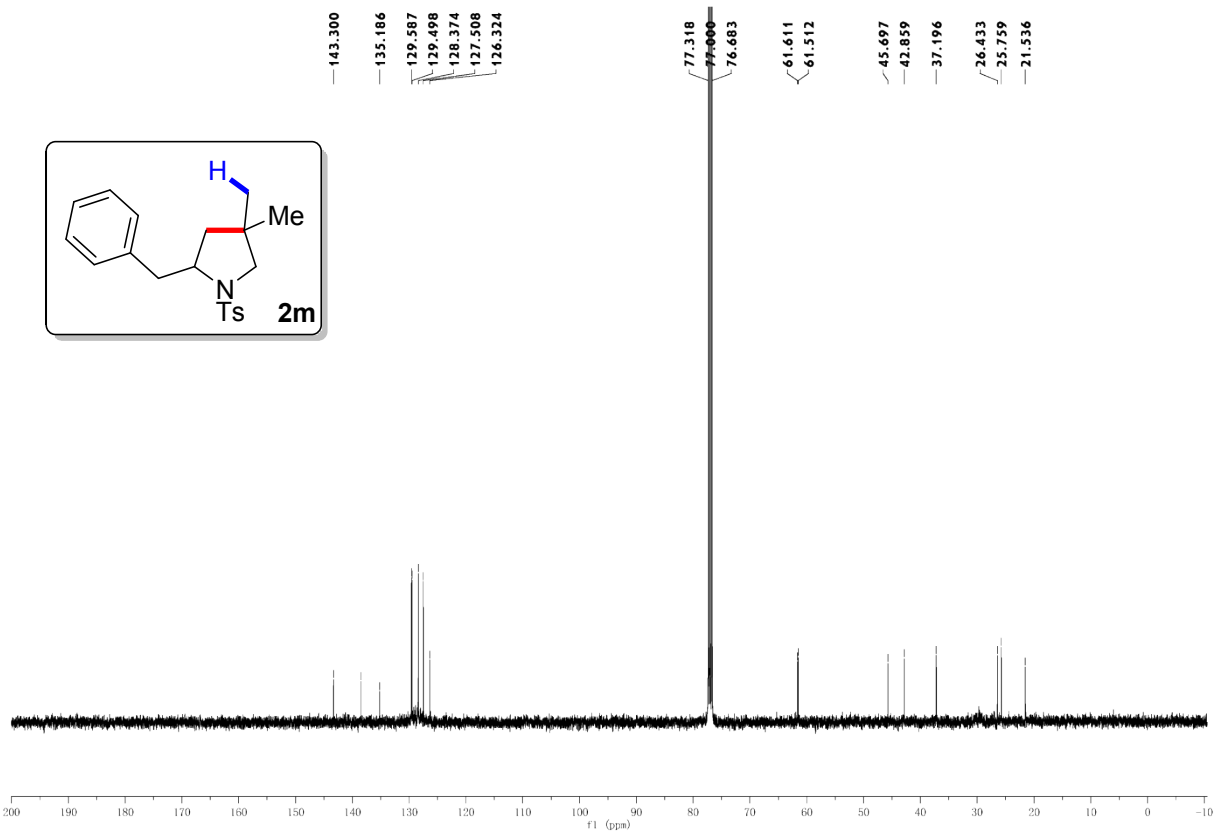
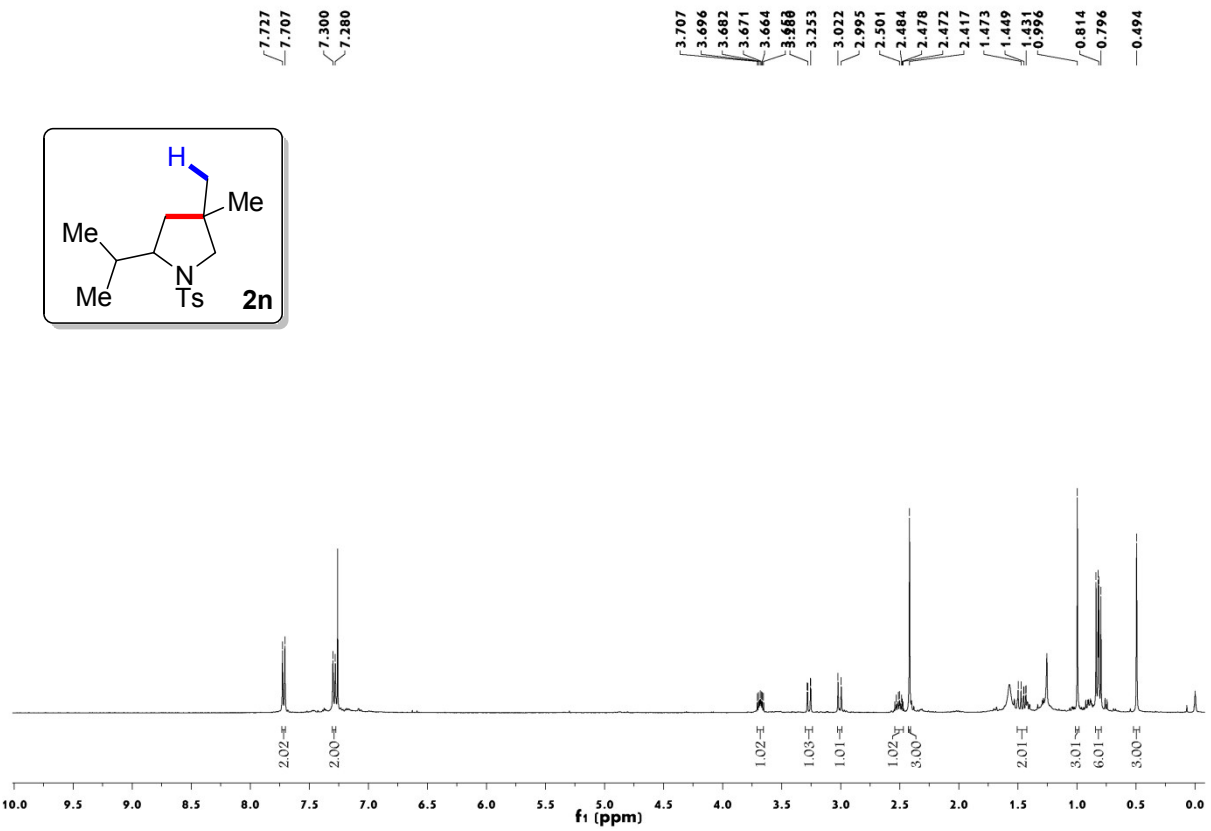
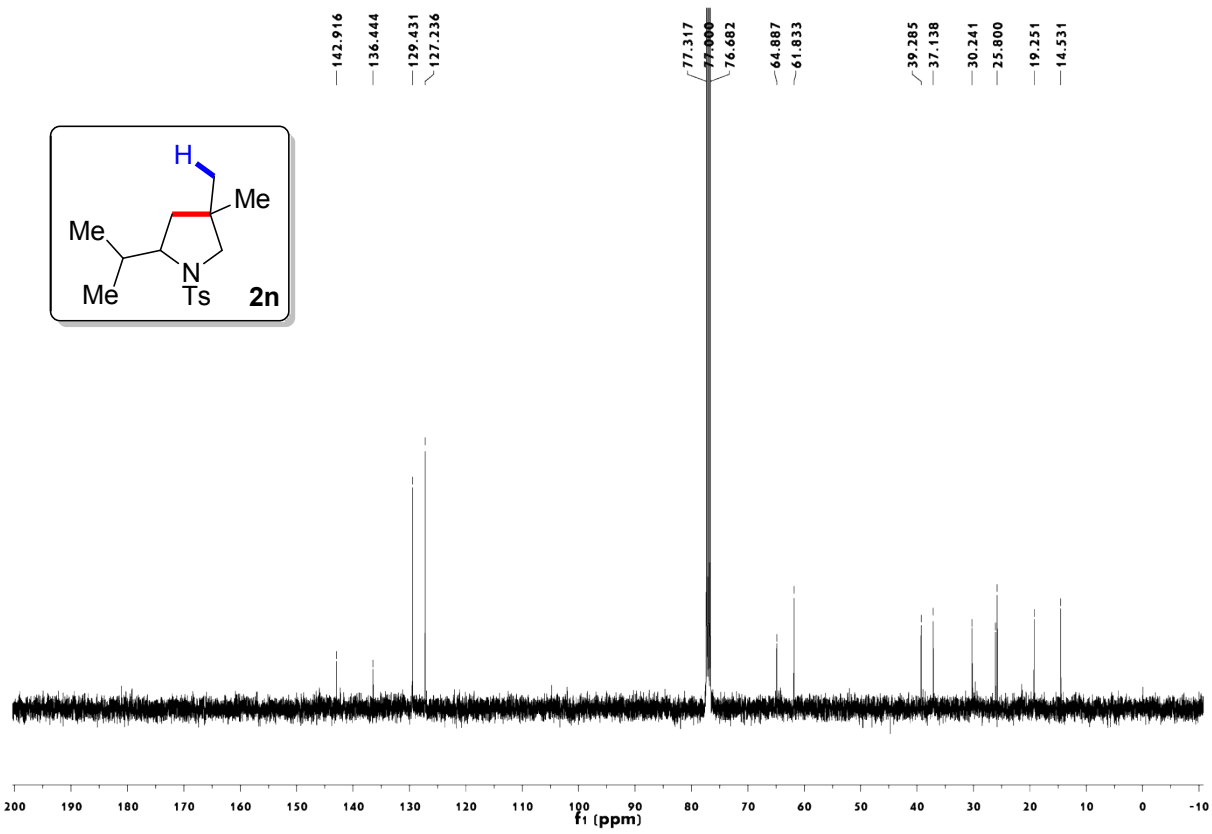
51.473
38.086
26.040
25.621
21.484
H
Me
N
Ts
2l
f1 (ppm)


7.796
7.775
7.338
7.318
7.294
7.276
7.233
7.226
7.215
3.817
3.797
3.777
3.764
3.744
3.602
3.593
3.569
3.560
3.124
2.738
2.431
1.493
1.478
1.472
1.460
0.983
0.443
H
Me
N
Ts
2m
2.01
2.01
2.02
3.02
1.02
1.00
2.00
1.01
3.00
2.01
3.00
3.00
f1 (ppm)


143.300
135.186
129.587
129.498
128.374
127.508
126.324
77.318
77.000
76.683
61.611
61.512
45.697
42.859
37.196
26.433
25.759
21.536
H
Me
N
Ts
2m
f1 (ppm)


7.727
7.707
7.300
7.280
3.707
3.696
3.682
3.671
3.664
3.253
3.022
2.995
2.501
2.484
2.478
2.472
2.417
1.473
1.449
1.431
0.996
0.814
0.796
0.494
H
Me
Me
Me
N
Ts
2n
2.02
2.00
1.02
1.03
1.01
1.02
3.00
2.01
3.01
6.01
3.00
f1 (ppm)


142.916
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129.431
127.236
77.317
77.000
76.682
64.887
61.833
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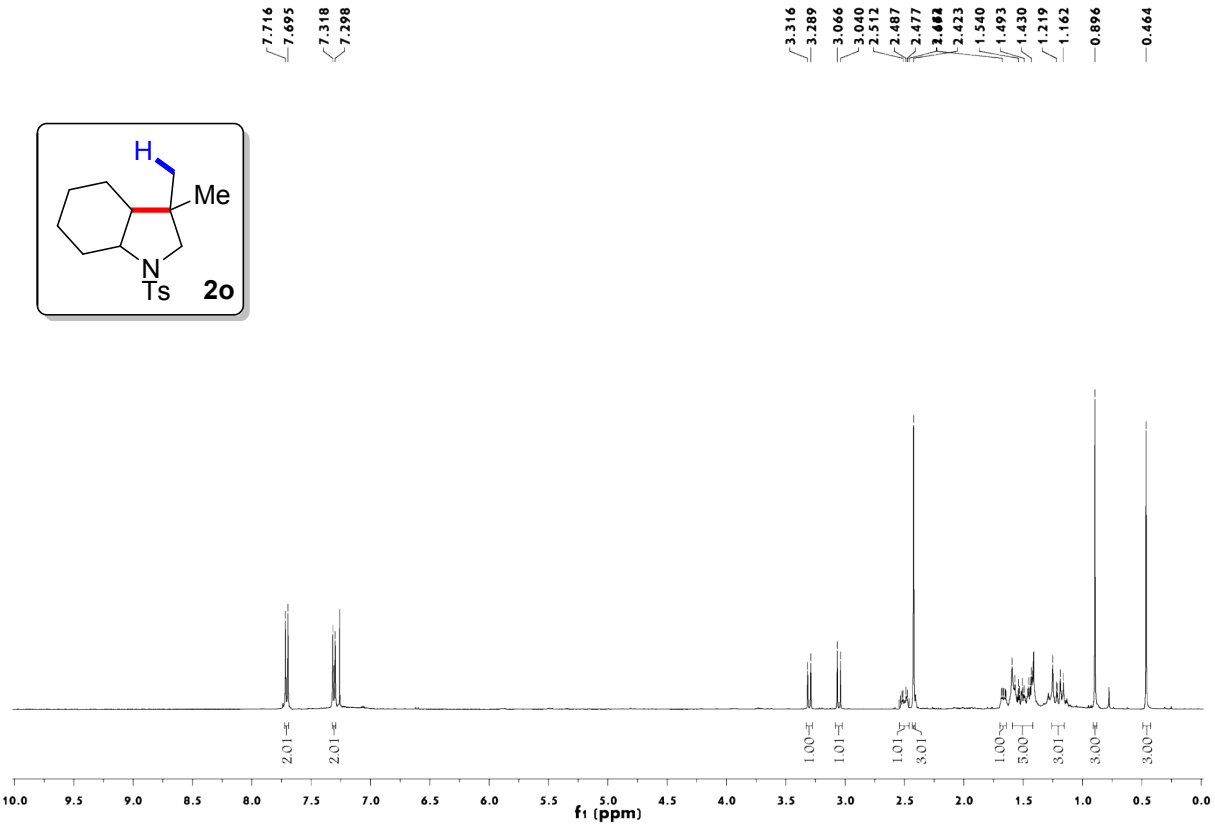
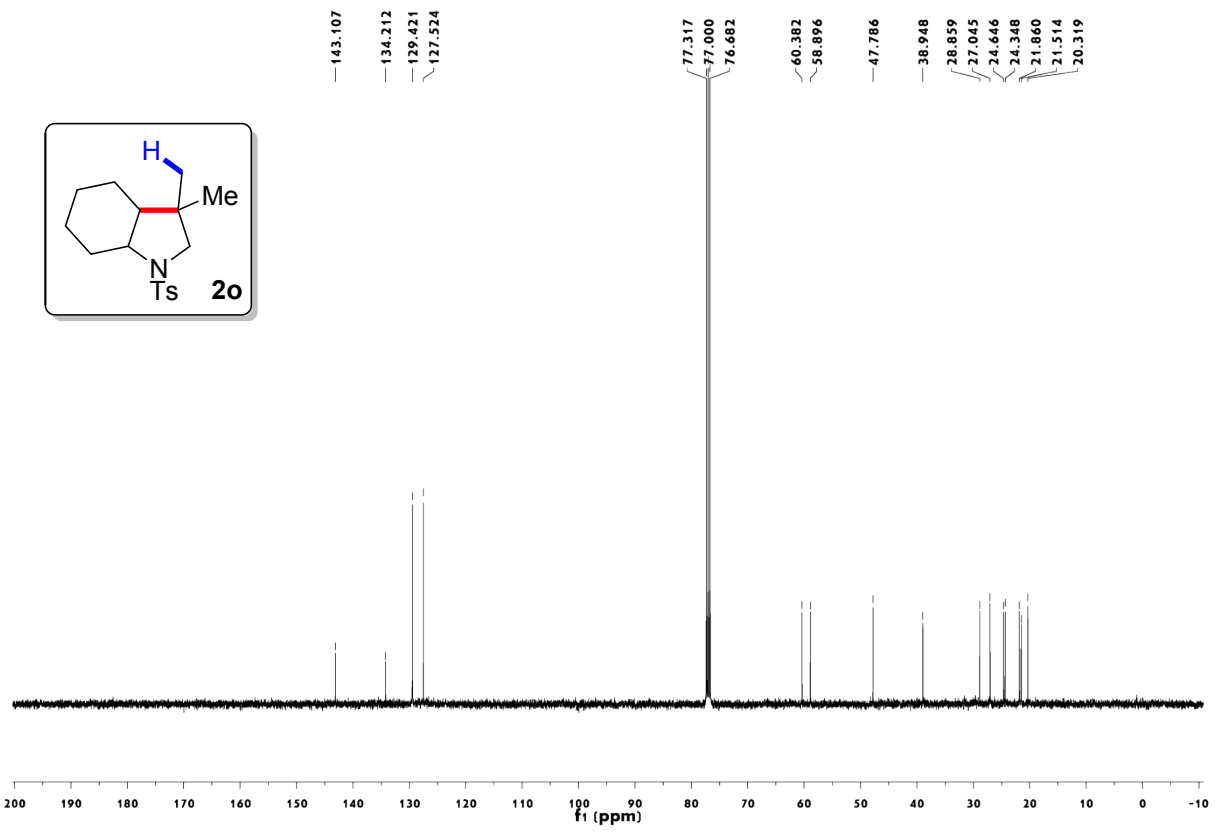
25.800
19.251
14.531
H
Me
Me
Me
N
Ts
2n
f1 (ppm)


7.716
7.695
7.318
7.298
3.316
3.289
3.066
3.040
2.512
2.487
2.477
2.423
1.540
1.493
1.430
1.219
1.162
0.896
0.464
H
Me
N
Ts
2o
2.01
2.01
1.00
1.01
1.01
3.01
1.00
5.00
3.01
3.00
3.00
f1 (ppm)


143.107
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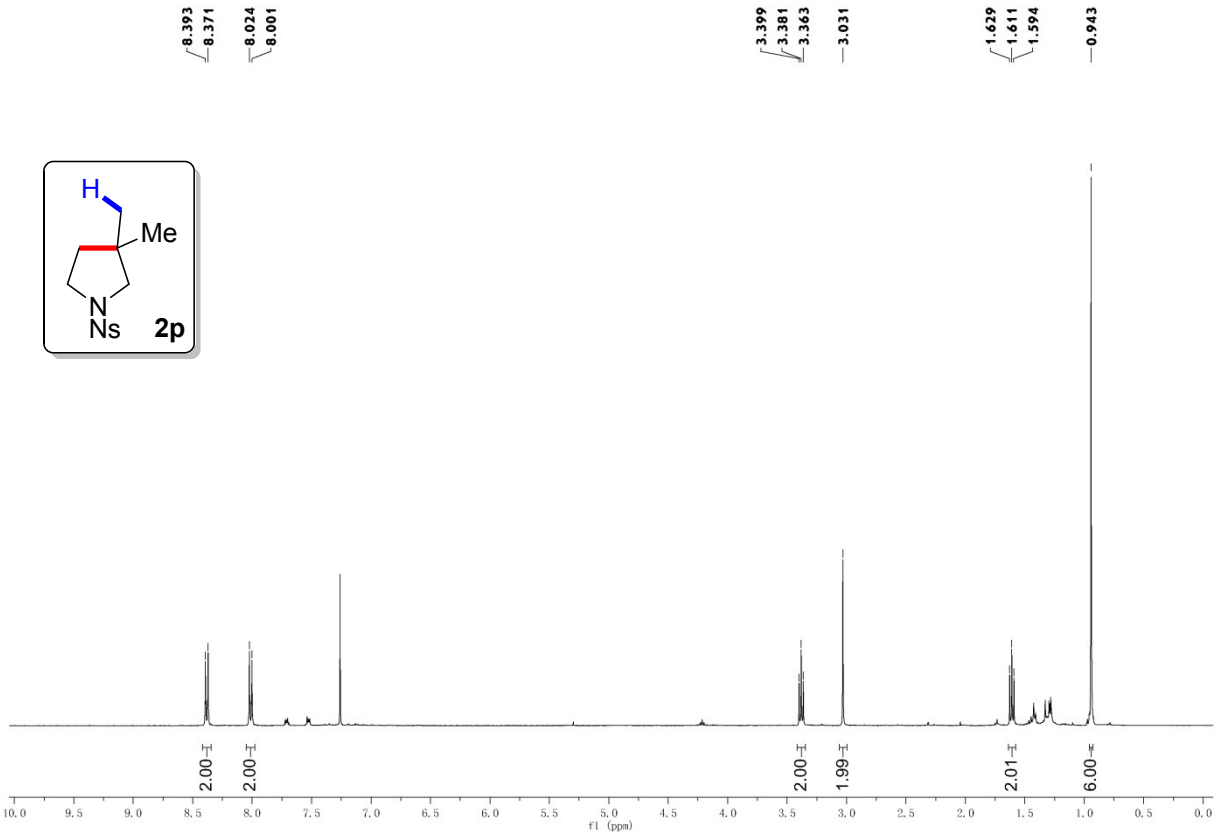
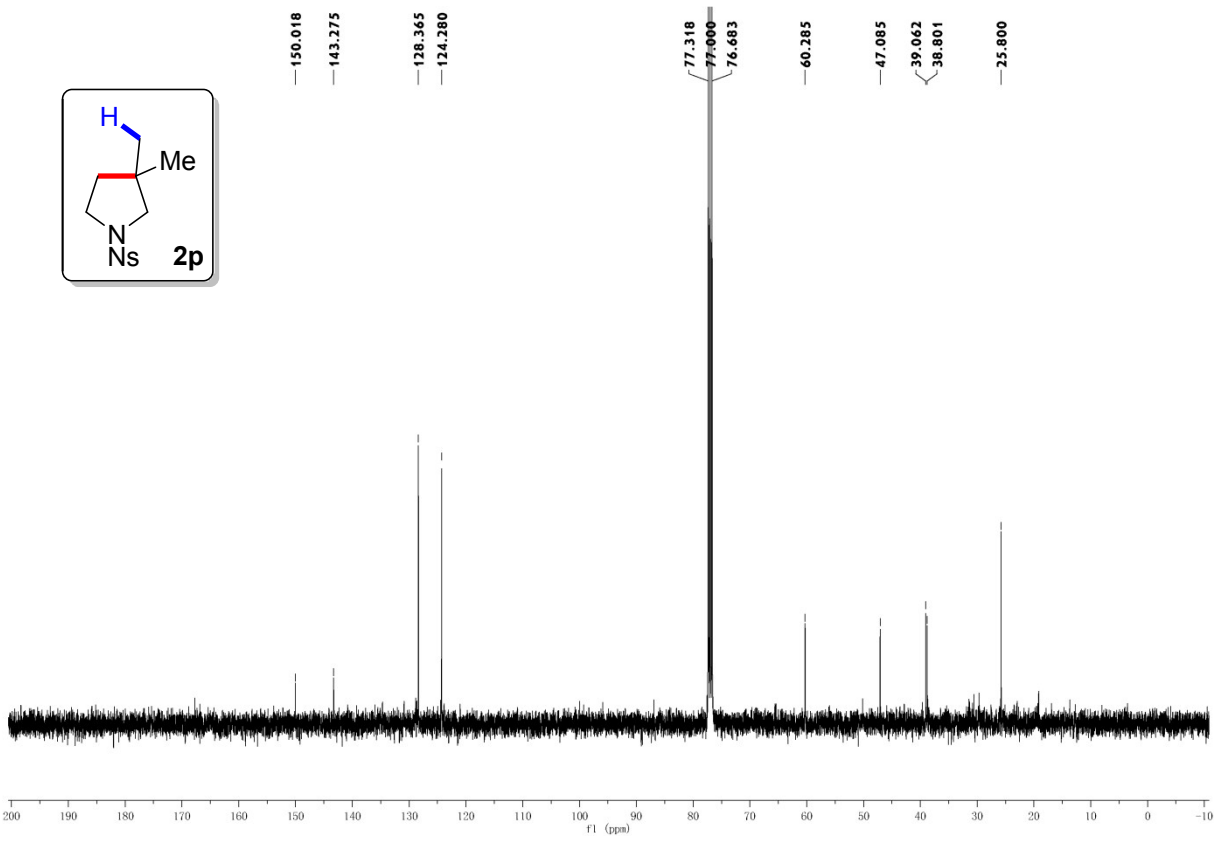
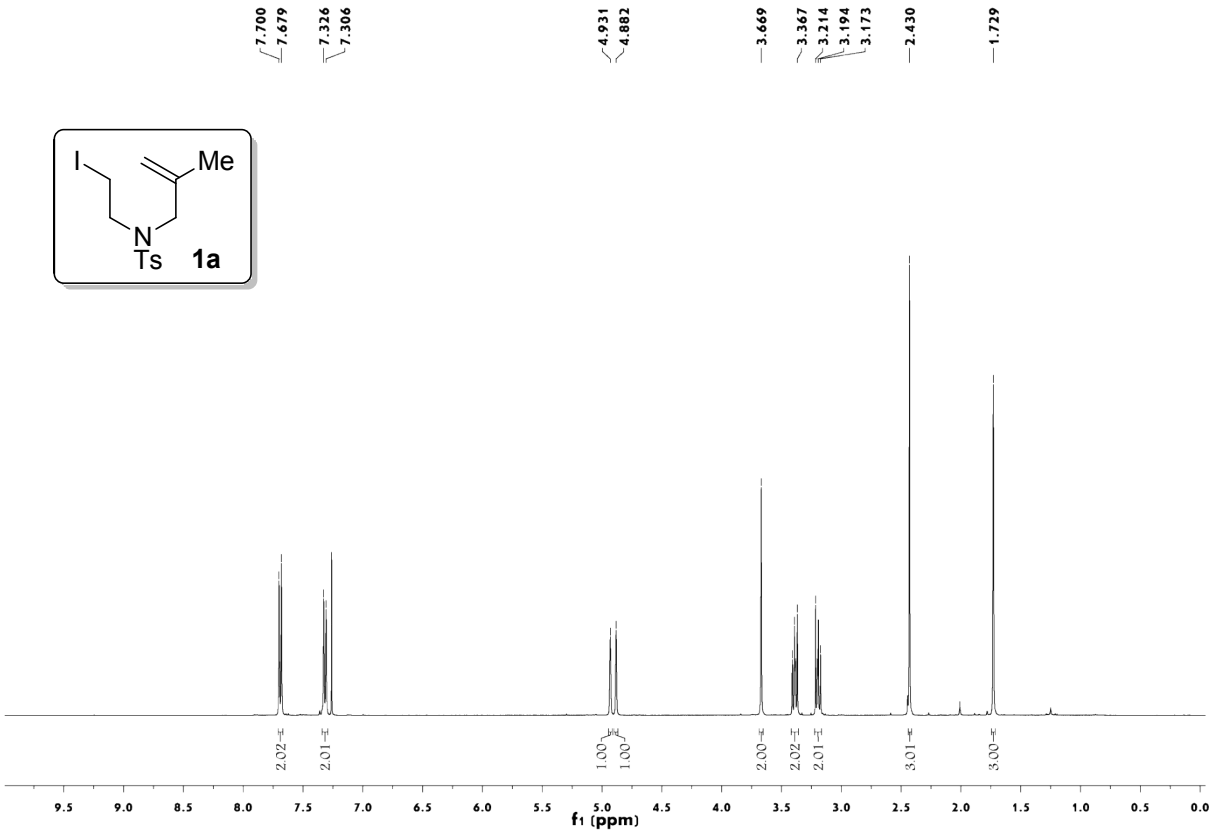
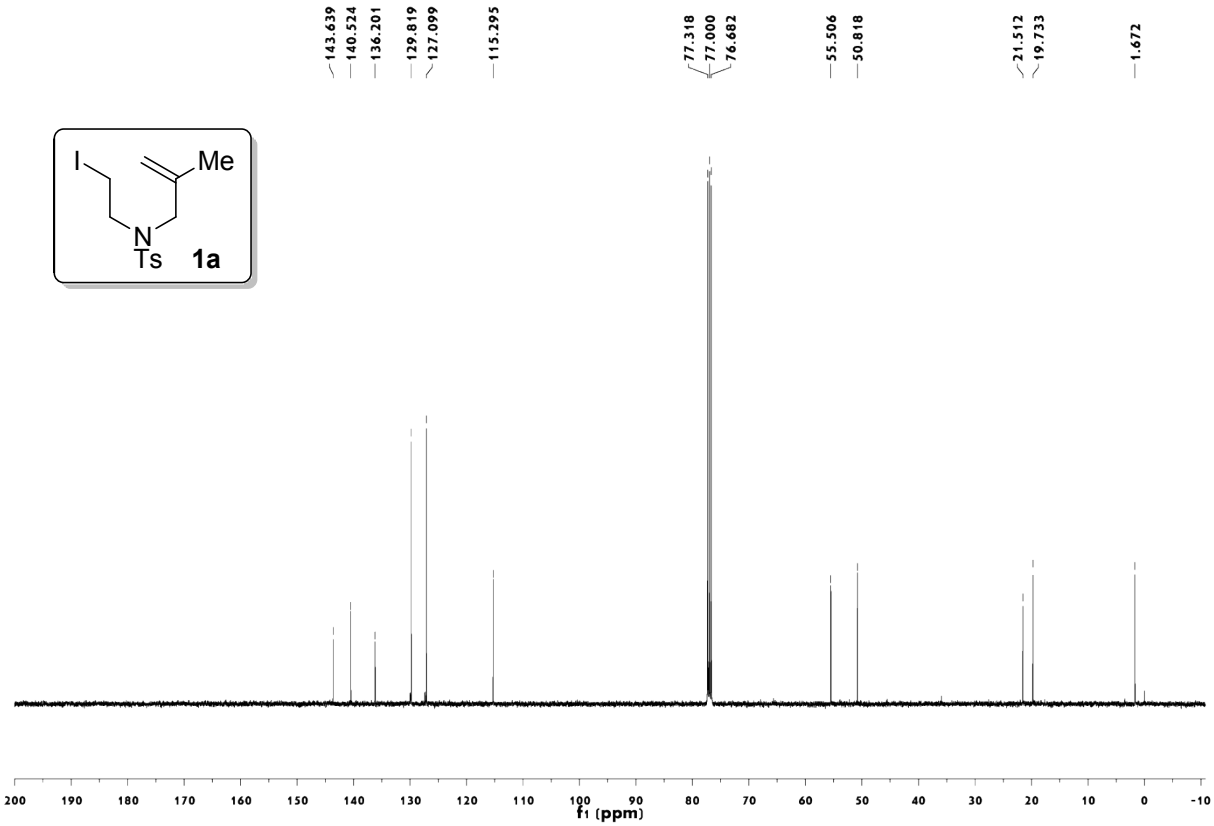
129.421
127.524
77.317
77.000
76.682
60.382
58.896
47.786
38.948
28.859
27.045
24.646
24.348
21.860
21.514
20.319
H
Me
N
Ts
2o
f1 (ppm)

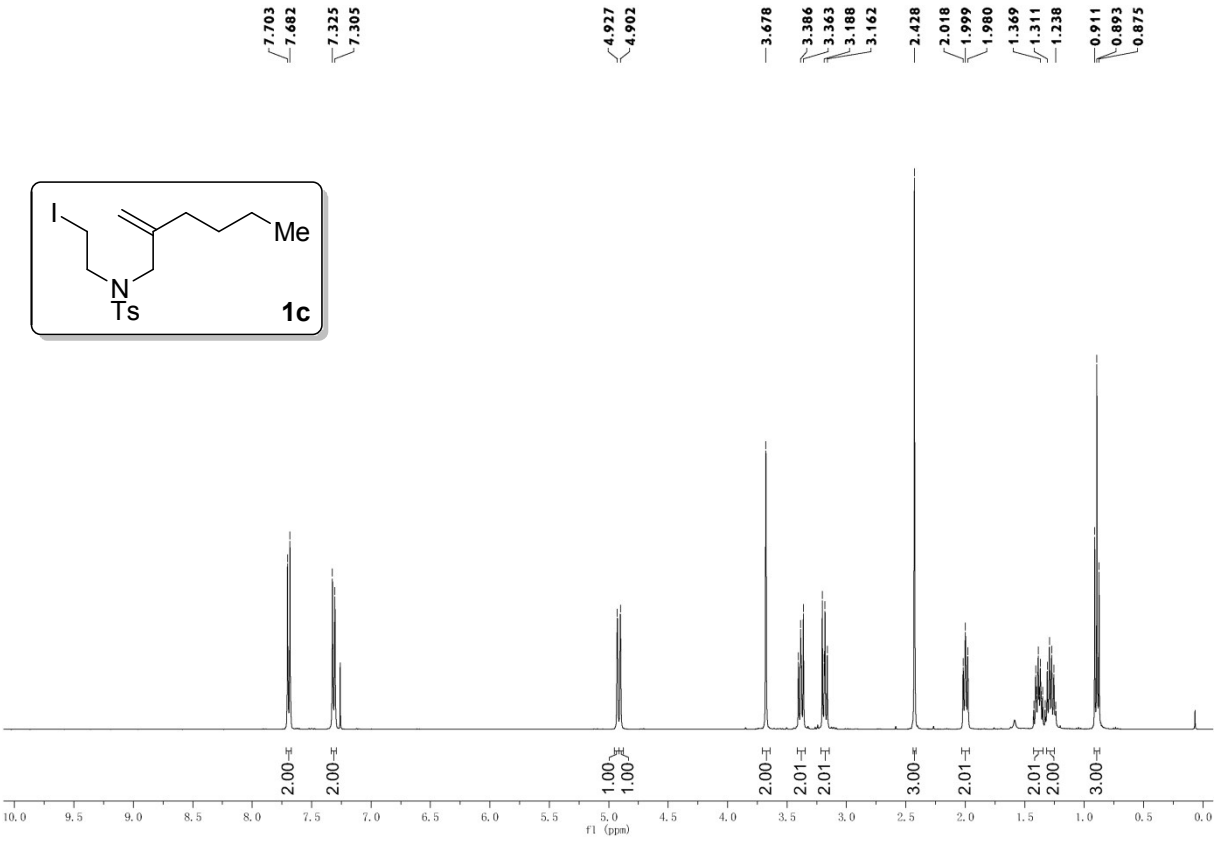

8.393
8.371
8.024
8.001
3.399
3.381
3.363
3.031
1.629
1.611
1.594
0.943
H
Me
N
Ns
2p
2.00
2.00
2.00
1.99
2.01
6.00
f1 (ppm)

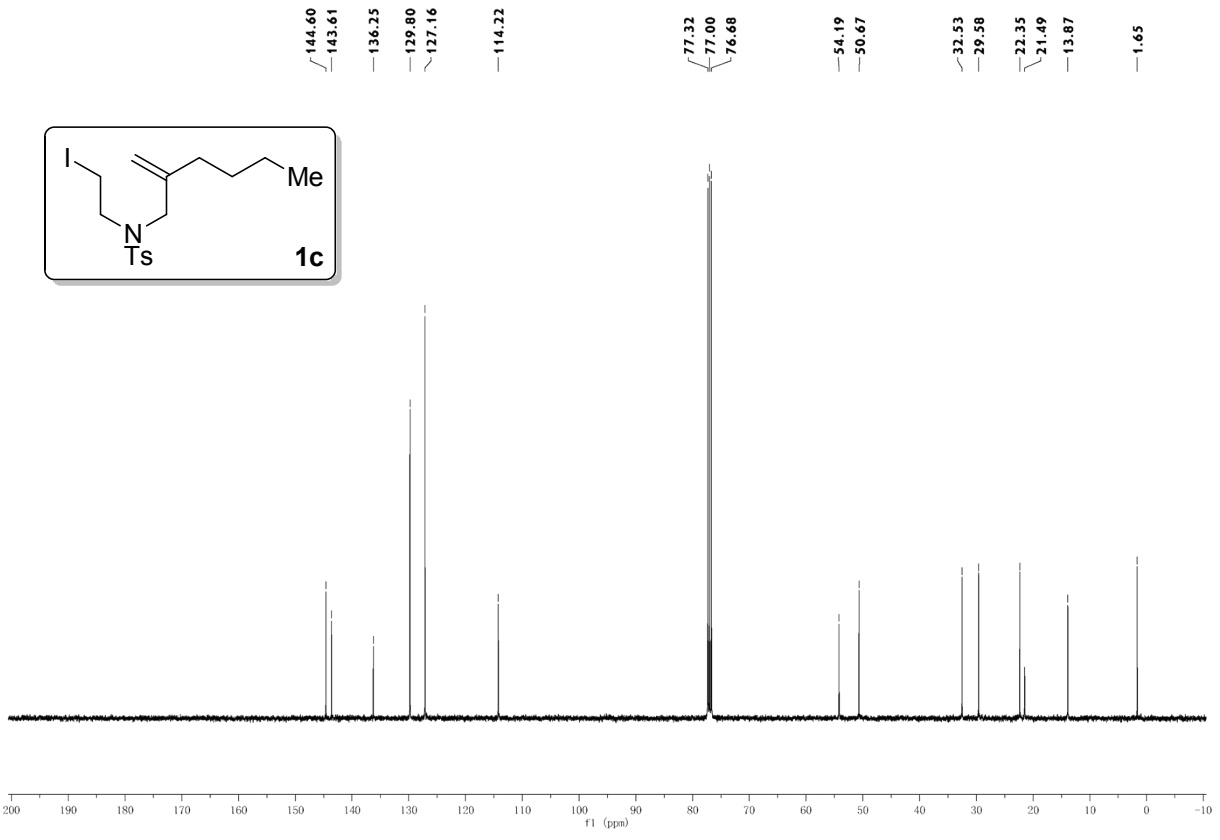

150.018
143.275
128.365
124.280
77.318
77.000
76.683
60.285
47.085
39.062
38.801
25.800
H
Me
N
Ns
2p
f1 (ppm)

10. Copies of the ^{1}H NMR and ^{13}C NMR for Partial Material






7.703
7.682
7.325
7.305
4.927
4.902
3.678
3.386
3.363
3.188
3.162
2.428
2.018
1.999
1.980
1.369
1.311
1.238
0.911
0.893
0.875
I
Me
N
Ts
1c
2.00
2.00
1.00
1.00
2.00
2.01
2.01
3.00
2.01
2.01
2.00
3.00
f1 (ppm)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143.61
136.25
129.80
127.16
114.22
77.32
77.00
76.68
54.19
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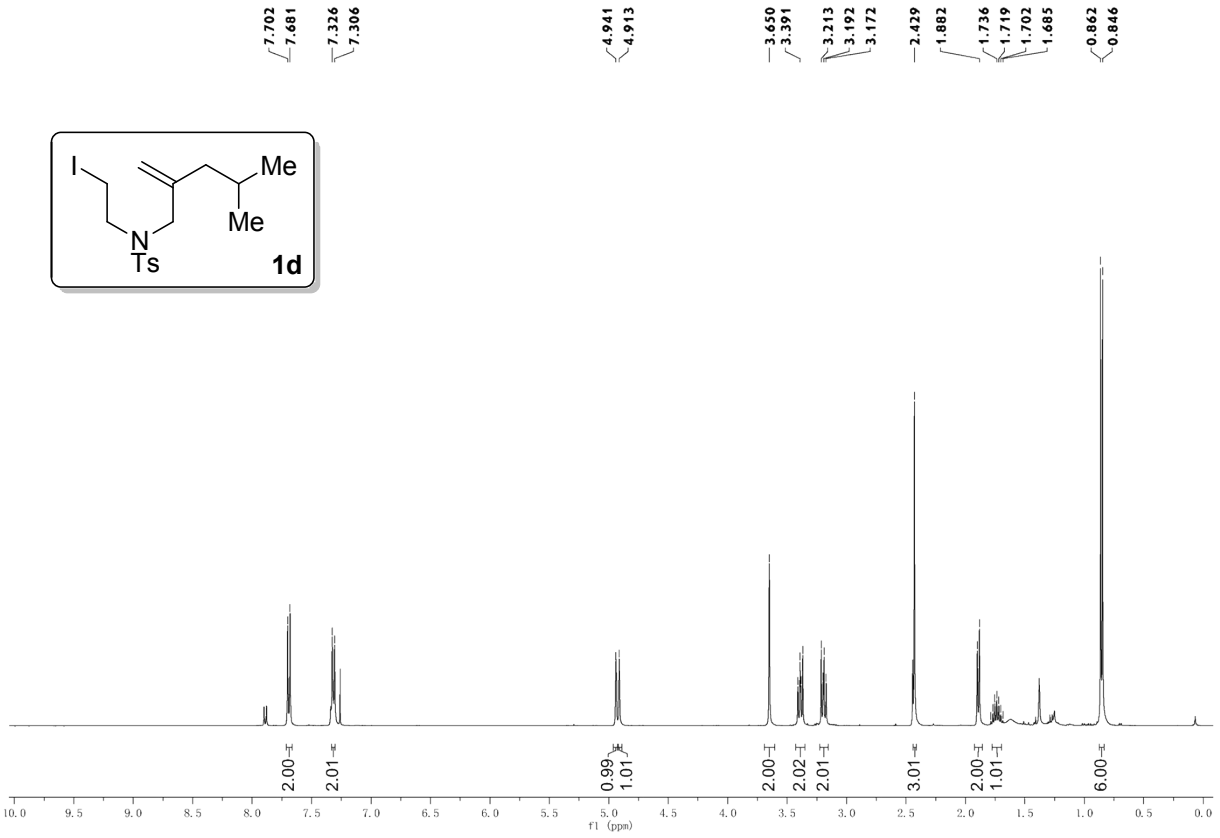
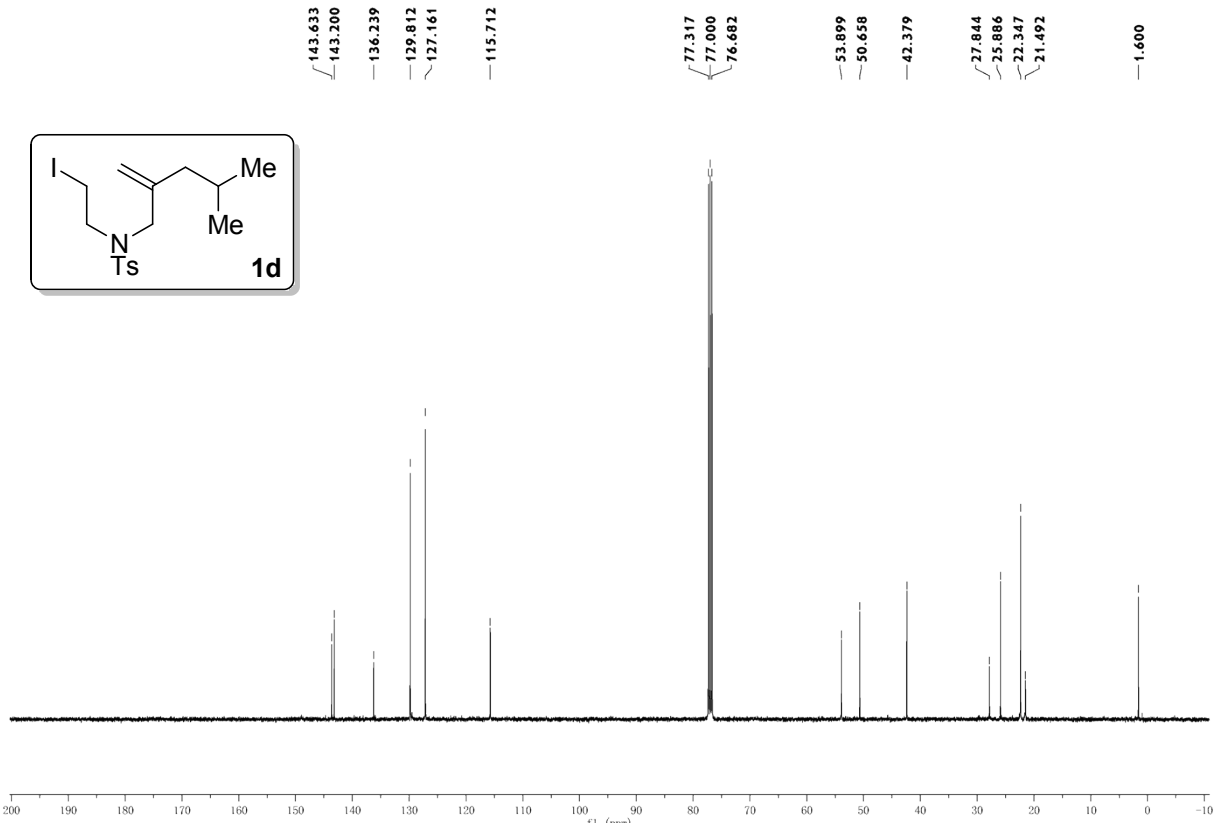
32.53
29.58
22.35
21.49
13.87
1.65
I
Me
N
Ts
1c
f1 (ppm)


7.702
7.681
7.326
7.306
4.941
4.913
3.650
3.391
3.213
3.192
3.172
2.429
1.882
1.736
1.719
1.702
1.685
0.862
0.846
I
Me
Me
N
Ts
1d
2.00
2.01
0.99
1.01
2.00
2.02
2.01
3.01
2.00
1.01
6.00
f1 (ppm)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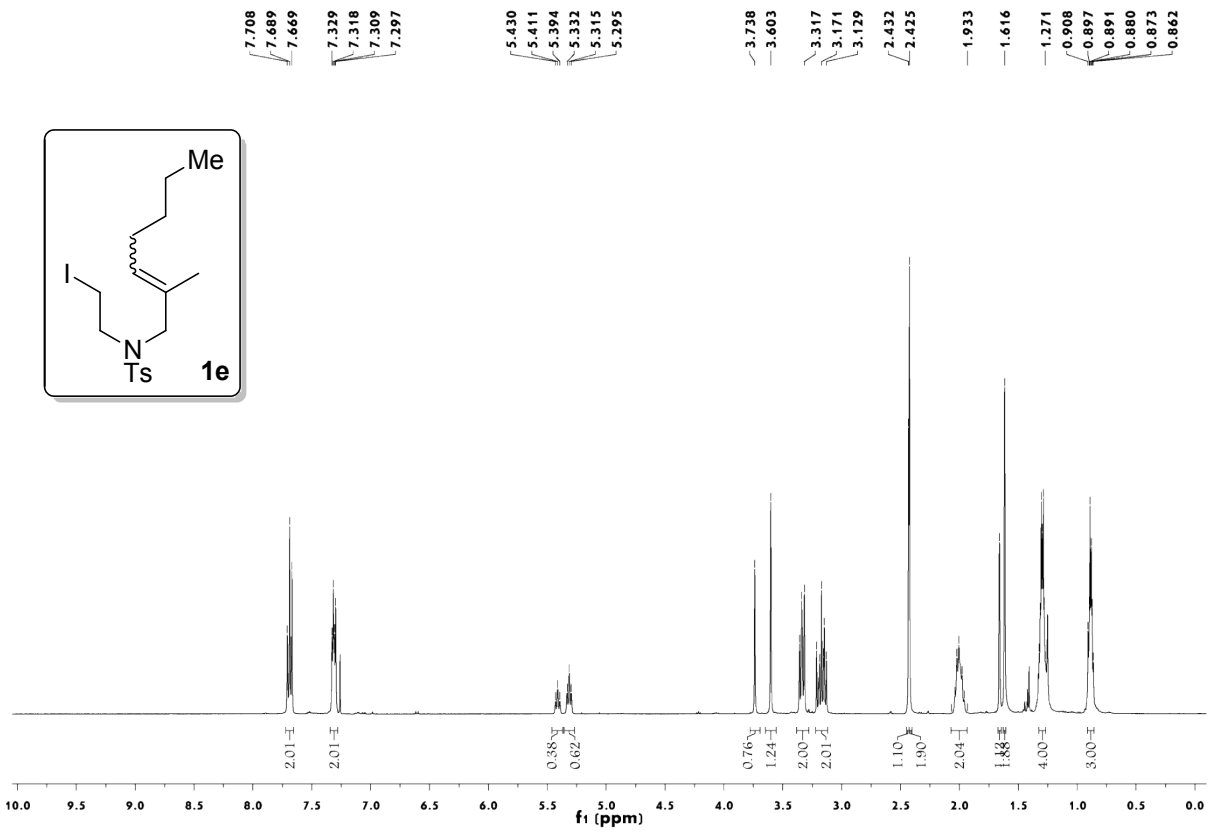
143.200
136.239
129.812
127.161
115.712
77.317
77.000
76.682
53.899
50.658
42.379
27.844
25.886
22.347
21.492
1.600
I
Me
Me
N
Ts
1d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7.689
7.669
7.329
7.318
7.309
7.297
5.430
5.411
5.394
5.332
5.315
5.295
3.738
3.603
3.317
3.171
3.129
2.432
2.425
1.933
1.616
1.271
0.908
0.897
0.891
0.880
0.873
0.862
Me
I
N
Ts
1e
2.01
2.01
0.38
0.62
0.76
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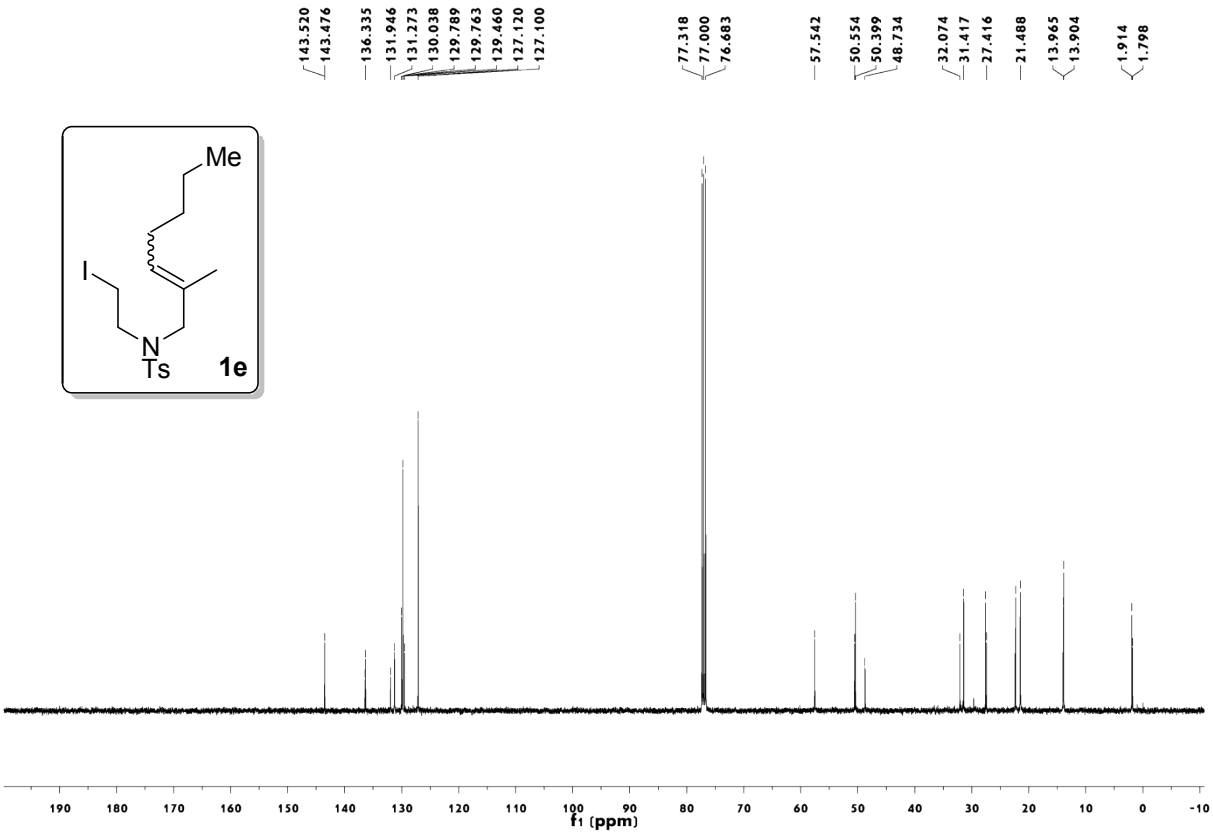
2.00
2.01
1.10
1.90
2.04
1.88
4.00
3.00
f1 (ppm)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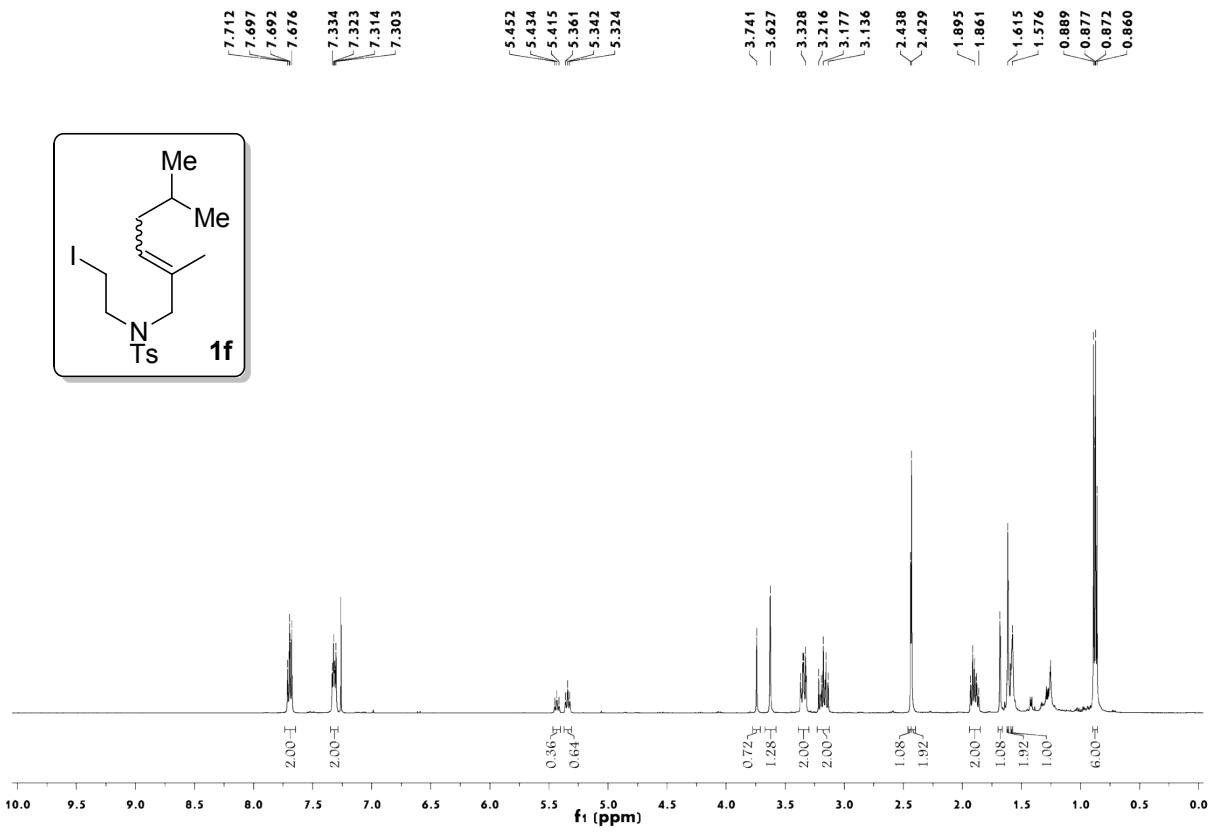
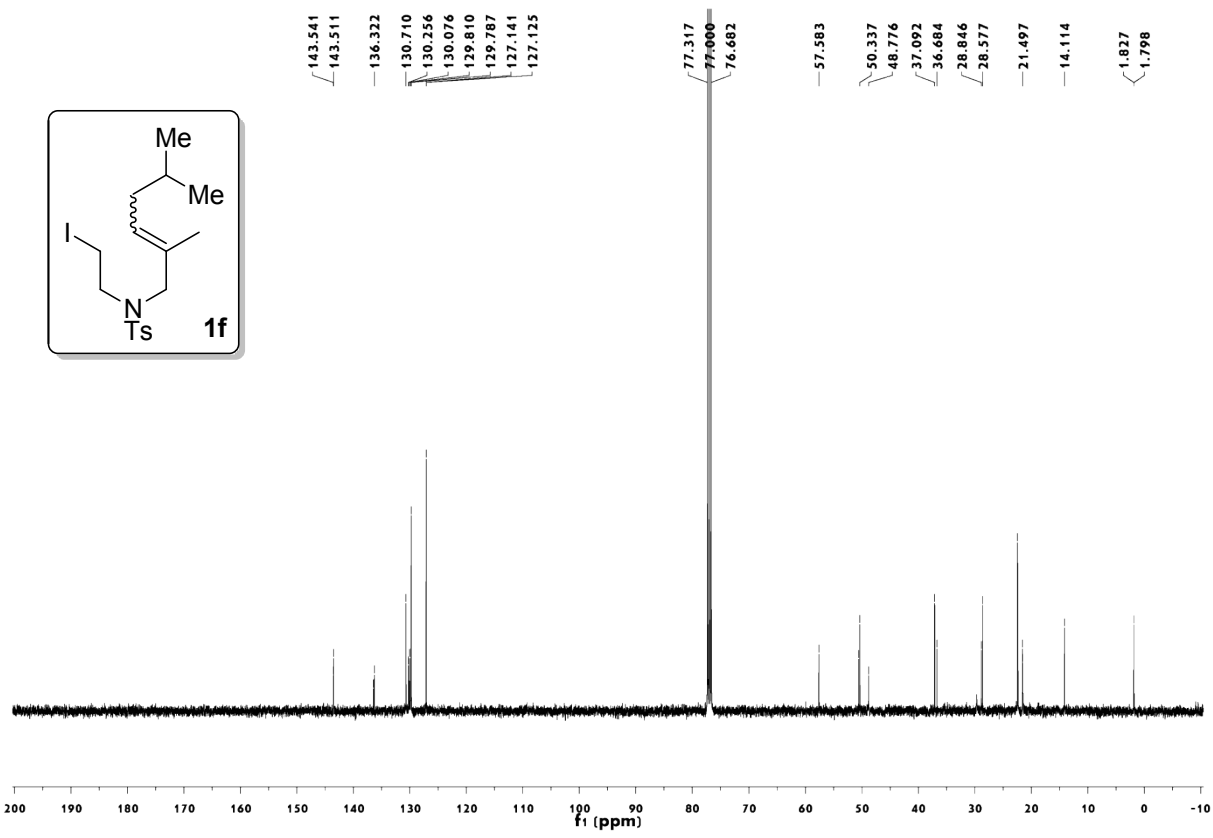
143.476
136.335
131.946
131.273
130.038
129.789
129.763
129.460
127.120
127.100
77.318
77.000
76.683
57.542
50.554
50.399
48.734
32.074
31.417
27.416
21.488
13.965
13.904
1.914
1.798
Me
I
N
Ts
1e
f1 (ppm)


7.712
7.697
7.692
7.676
7.334
7.323
7.314
7.303
5.452
5.434
5.415
5.361
5.342
5.324
3.741
3.627
3.328
3.216
3.177
3.136
2.438
2.429
1.895
1.861
1.615
1.576
0.889
0.877
0.872
0.860
Me
Me
I
N
Ts
1f
2.00
2.00
0.36
0.64
0.72
1.28
2.00
2.00
1.08
1.92
2.00
1.08
1.92
1.00
6.00
f1 (ppm)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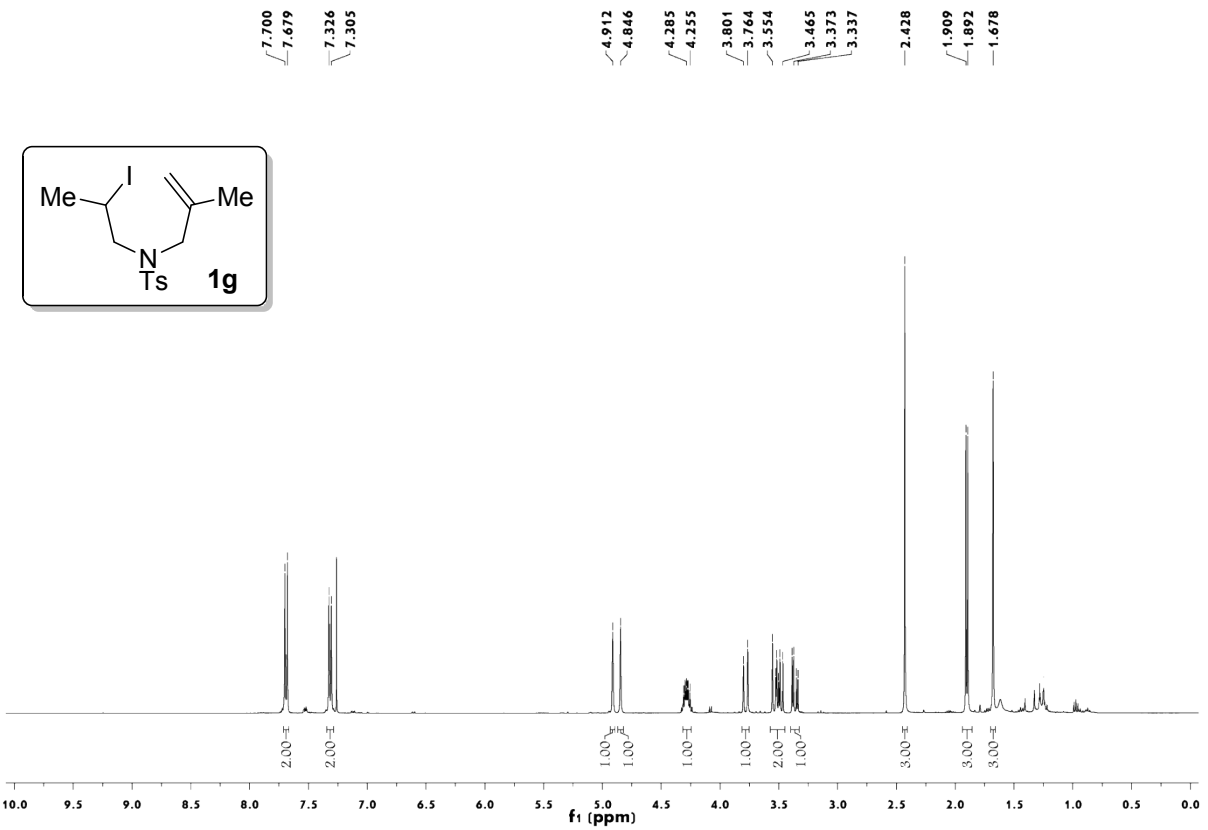
143.511
136.322
130.710
130.256
130.076
129.810
129.787
127.141
127.125
77.317
77.000
76.682
57.583
50.337
48.776
37.092
36.684
28.846
28.577
21.497
14.114
1.827
1.798
Me
Me
I
N
Ts
1f
f1 (ppm)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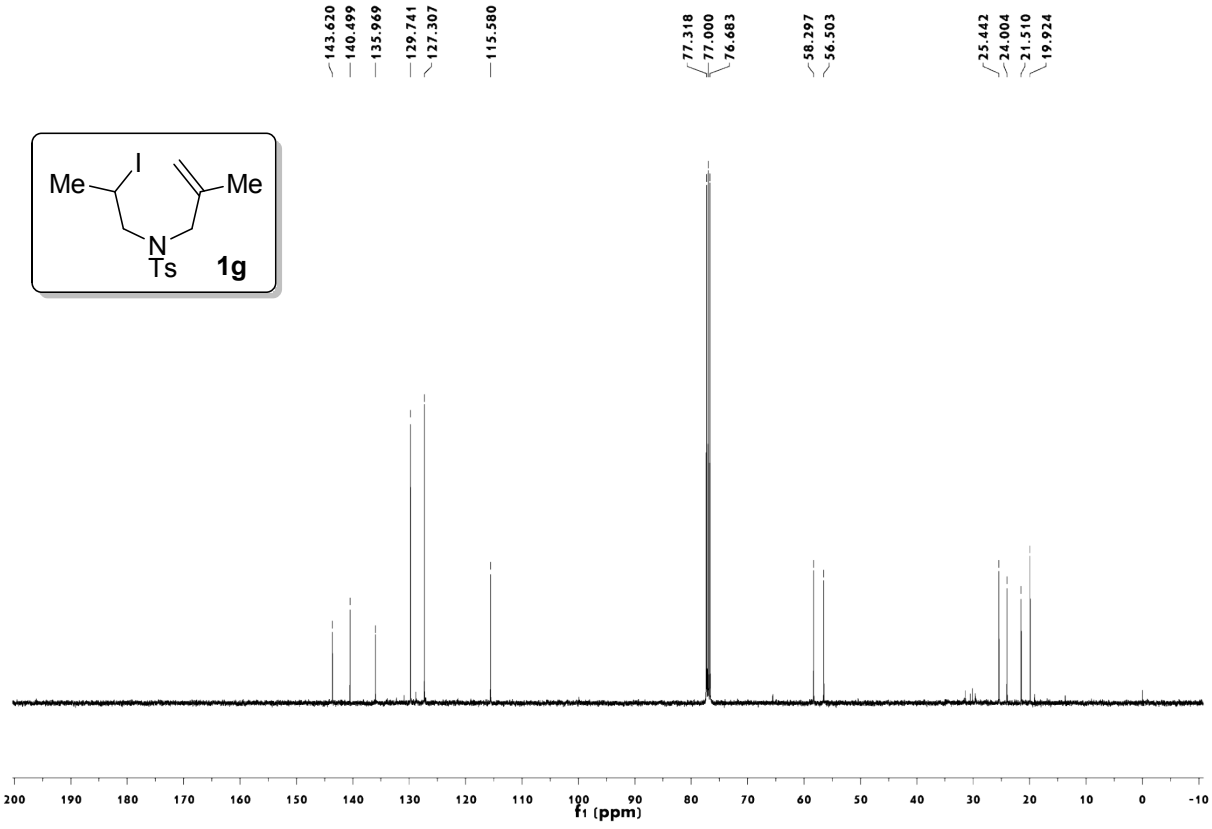
7.679
7.326
7.305
4.912
4.846
4.285
4.255
3.801
3.764
3.554
3.465
3.373
3.337
2.428
1.909
1.892
1.678
Me
I
Me
N
Ts
1g
2.00
2.00
1.00
1.00
1.00
1.00
2.00
1.00
3.00
3.00
3.00
f1 (ppm)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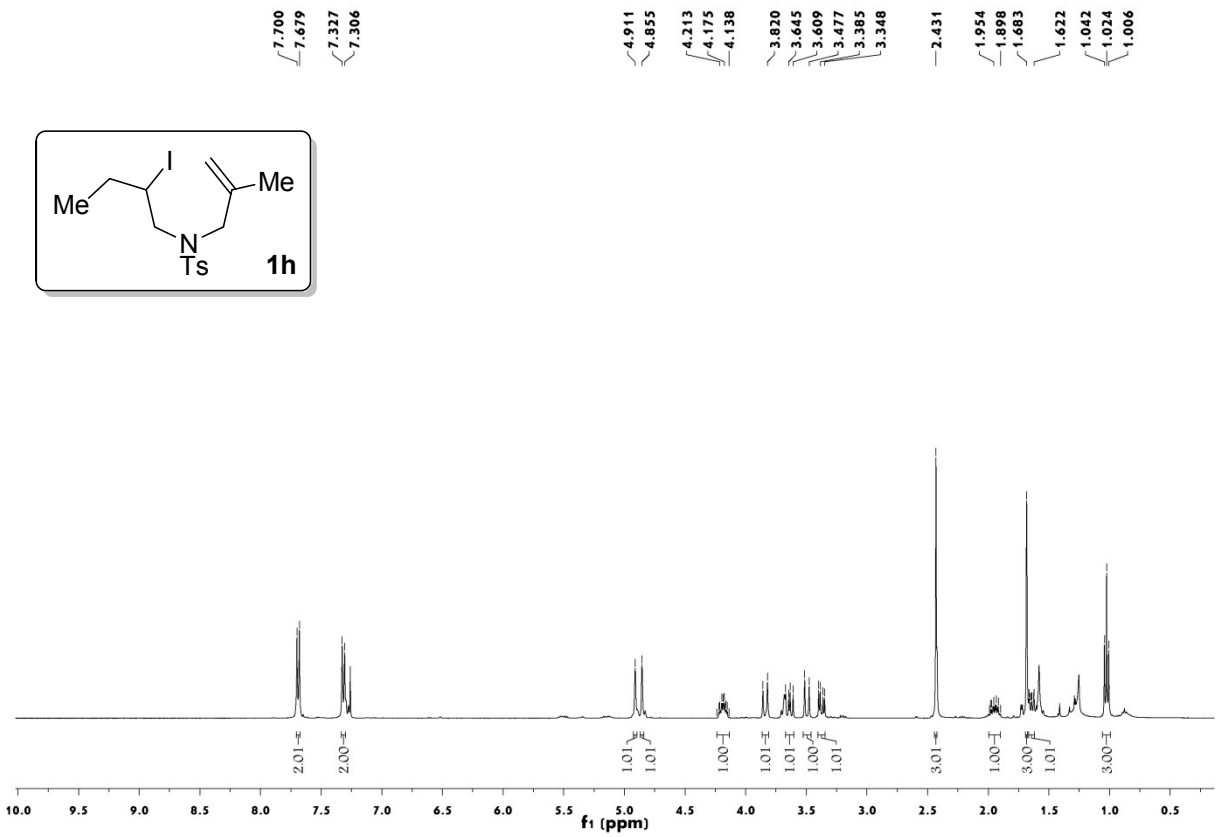
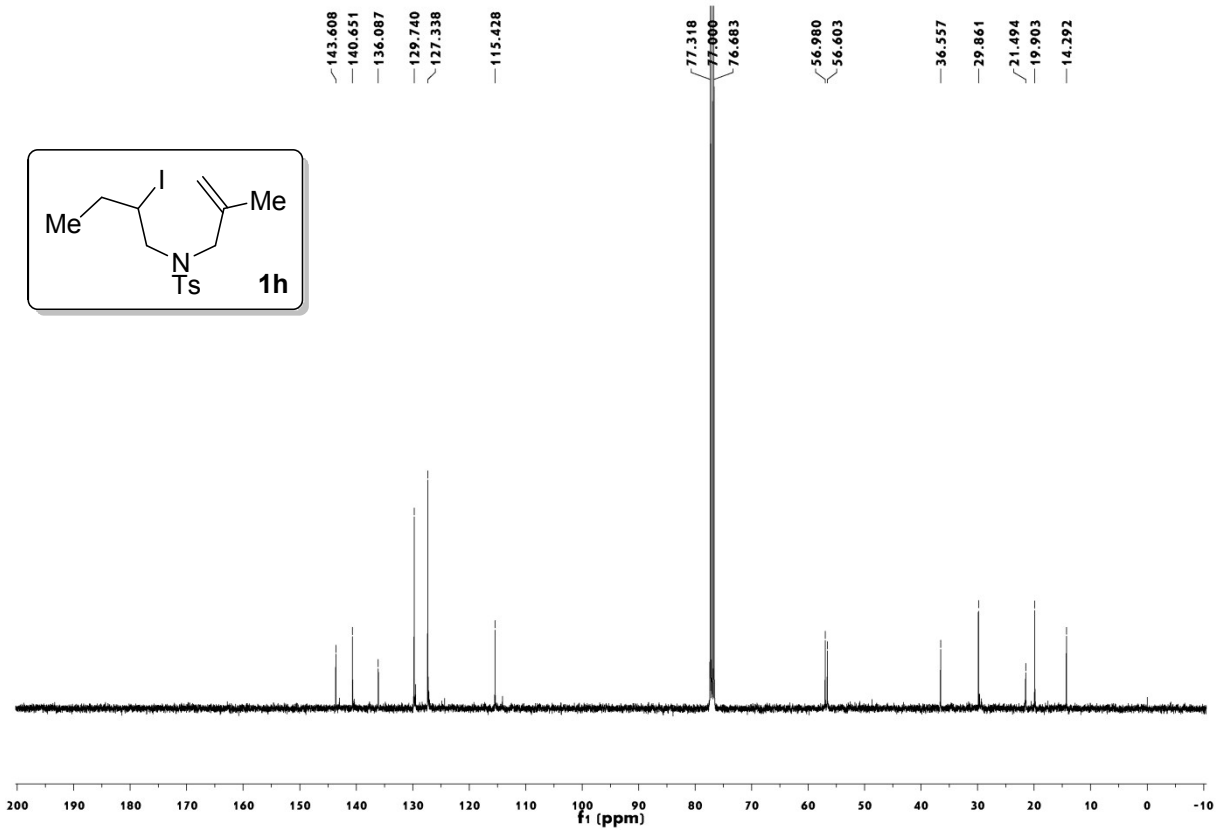
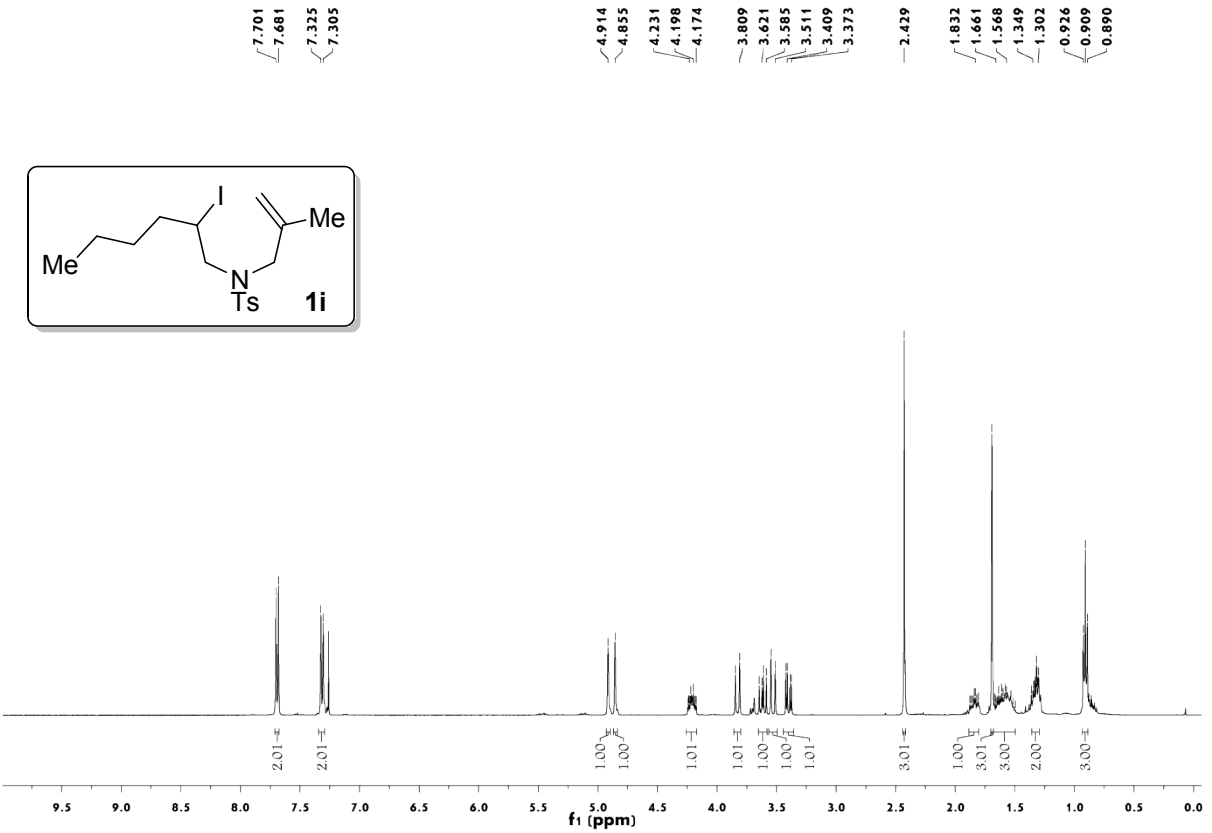
140.499
135.969
129.741
127.307
115.580
77.318
77.000
76.683
58.297
56.503
25.442
24.004
21.510
19.924
Me
I
Me
N
Ts
1g
f1 (ppm)


7.700
7.679
7.327
7.306
4.911
4.855
4.213
4.175
4.138
3.820
3.645
3.609
3.477
3.385
3.348
2.431
1.954
1.898
1.683
1.622
1.042
1.024
1.006
I
Me
Me
N
Ts
1h
2.01
2.00
1.01
1.01
1.00
1.01
1.01
1.00
1.01
3.01
1.00
3.00
1.01
3.00
f1 (ppm)


143.608
140.651
136.087
129.740
127.338
115.428
77.318
77.000
76.683
56.980
56.603
36.557
29.861
21.494
19.903
14.292
I
Me
Me
N
Ts
1h
f1 (ppm)


7.701
7.681
7.325
7.305
4.914
4.855
4.231
4.198
4.174
3.809
3.621
3.585
3.511
3.409
3.373
2.429
1.832
1.661
1.568
1.349
1.302
0.926
0.909
0.890
I
Me
Me
N
Ts
1i
2.01
2.01
1.00
1.00
1.01
1.01
1.00
1.00
1.01
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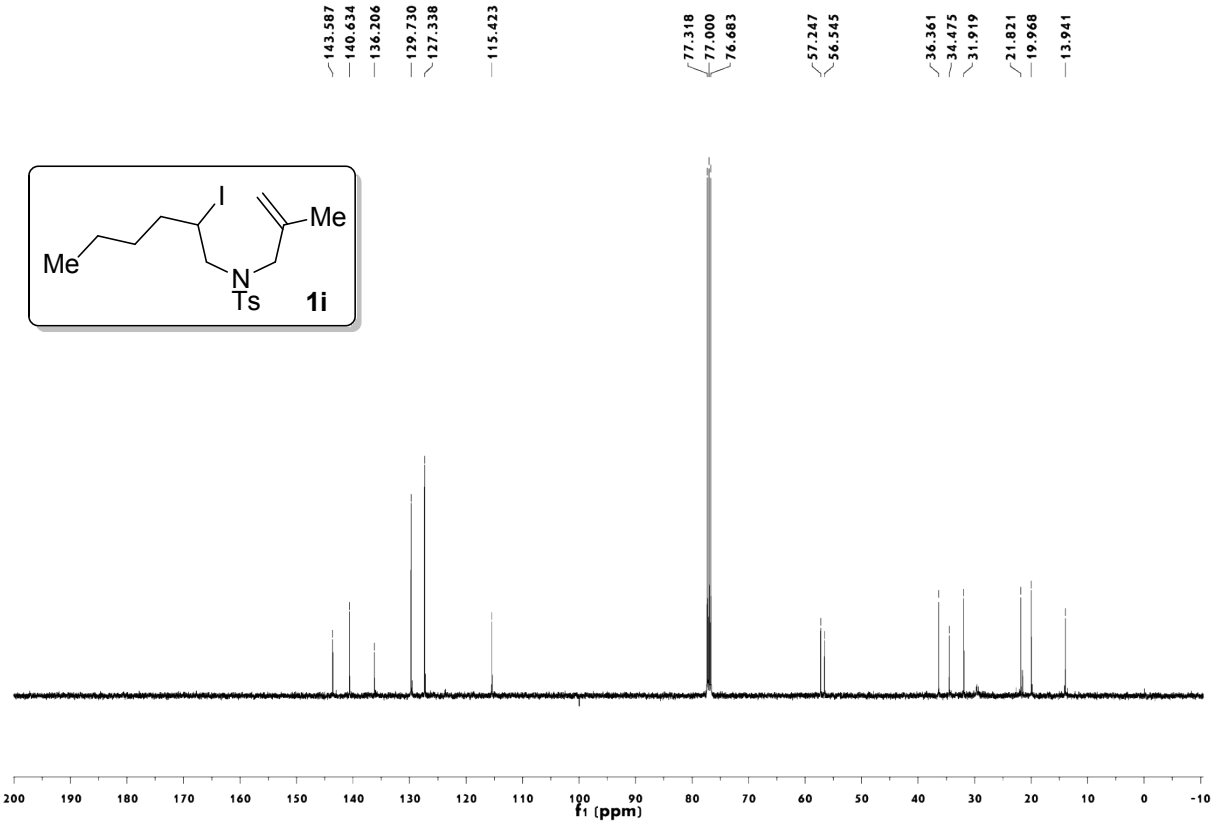
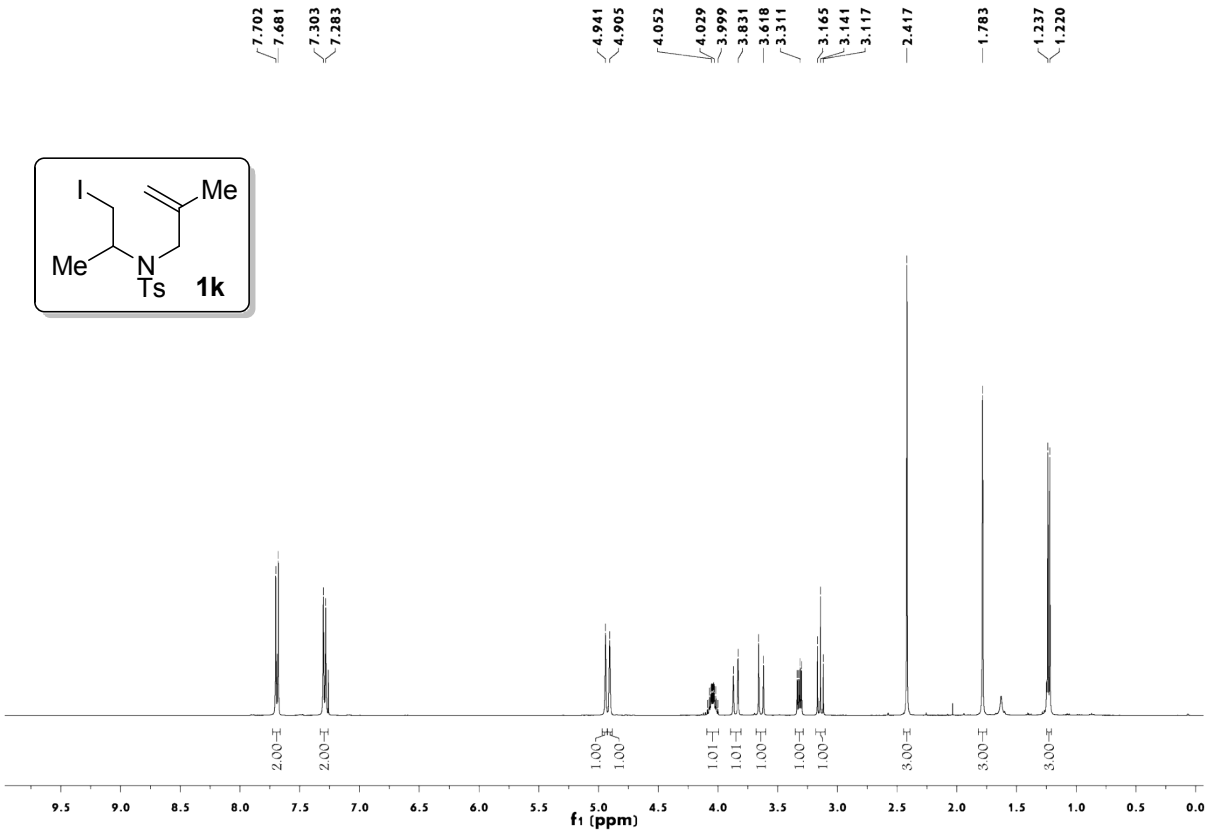
3.01
3.00
2.00
3.00
f1 (ppm)


143.587
140.634
136.206
129.730
127.338
115.423
77.318
77.000
76.683
57.247
56.545
36.361
34.475
31.919
21.821
19.968
13.941
I
Me
Me
N
Ts
1i
f1 (ppm)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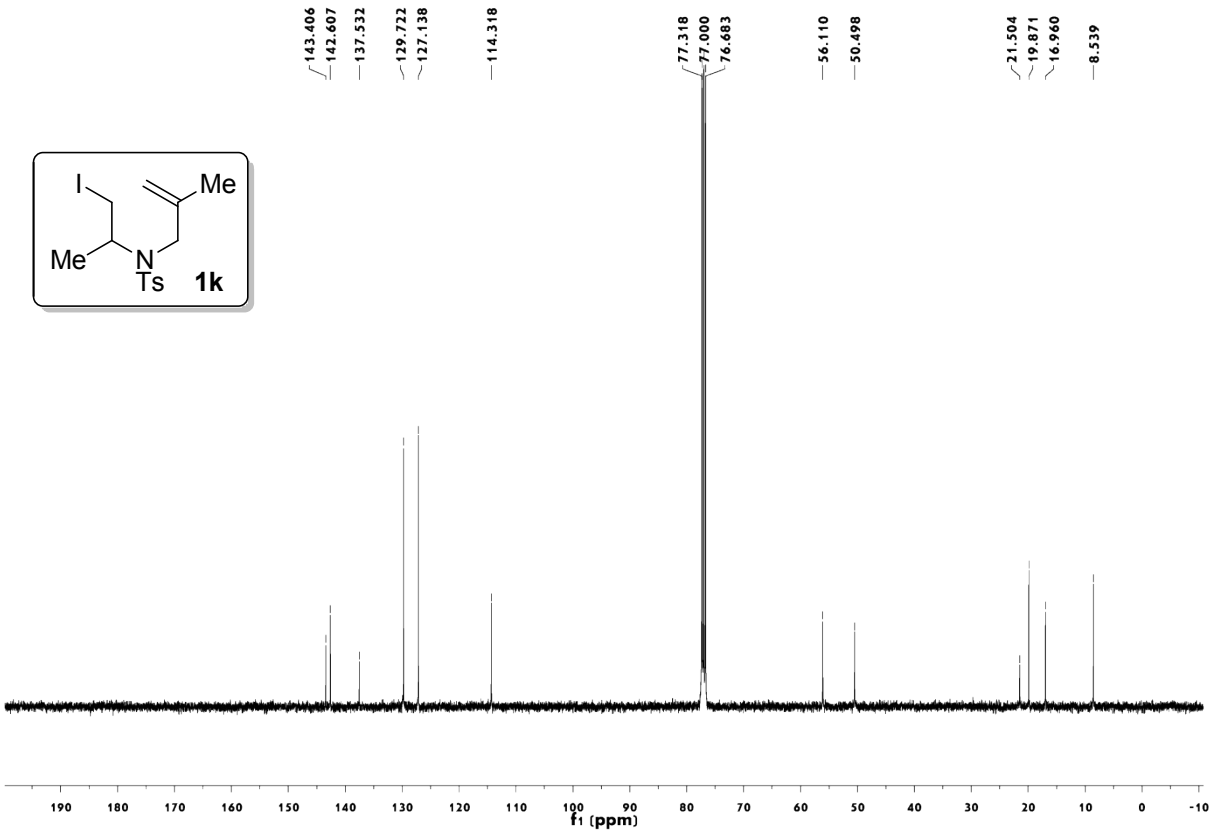
7.681
7.303
7.283
4.941
4.905
4.052
4.029
3.999
3.831
3.618
3.311
3.165
3.141
3.117
2.417
1.783
1.237
1.220
I
Me
Me
N
Ts
1k
2.00
2.00
1.00
1.00
1.01
1.01
1.00
1.00
1.00
3.00
3.00
3.00
f1 (ppm)


143.406
142.607
137.532
129.722
127.138
114.318
77.318
77.000
76.683
56.110
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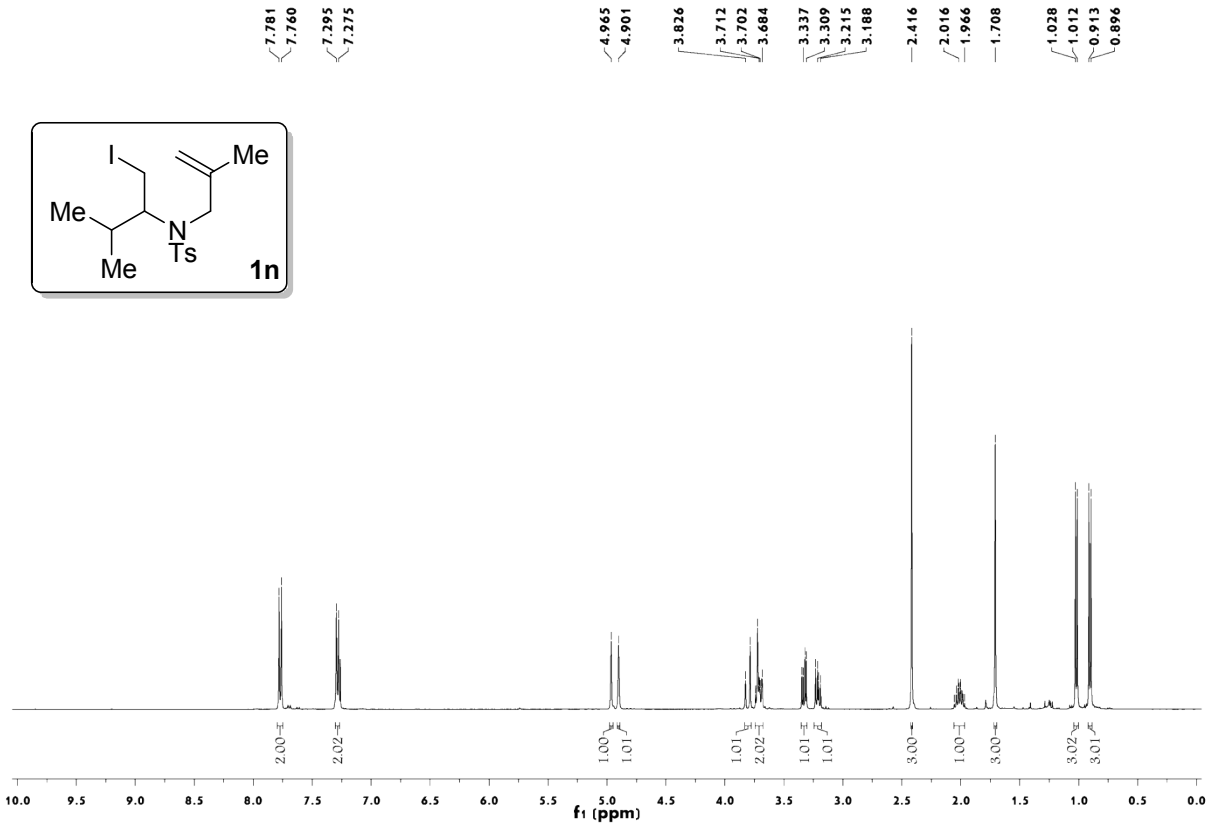
21.504
19.871
16.960
8.539
I
Me
Me
N
Ts
1k
f1 (ppm)


7.781
7.760
7.295
7.275
4.965
4.901
3.826
3.712
3.702
3.684
3.337
3.309
3.215
3.188
2.416
2.016
1.966
1.708
1.028
1.012
0.913
0.896
I
Me
Me
N
Ts
Me
1n
2.00
2.02
1.00
1.01
1.01
2.02
1.01
1.01
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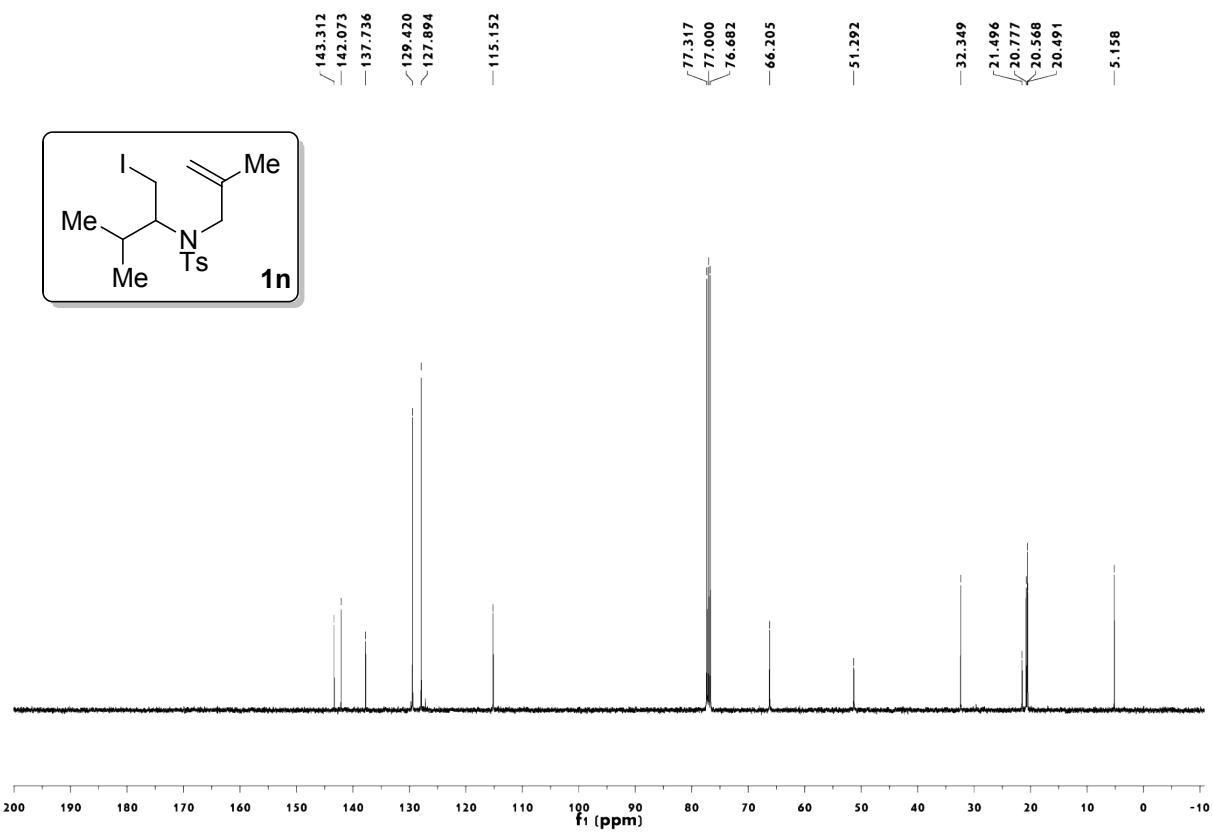
1.00
3.00
3.02
3.01
f1 (ppm)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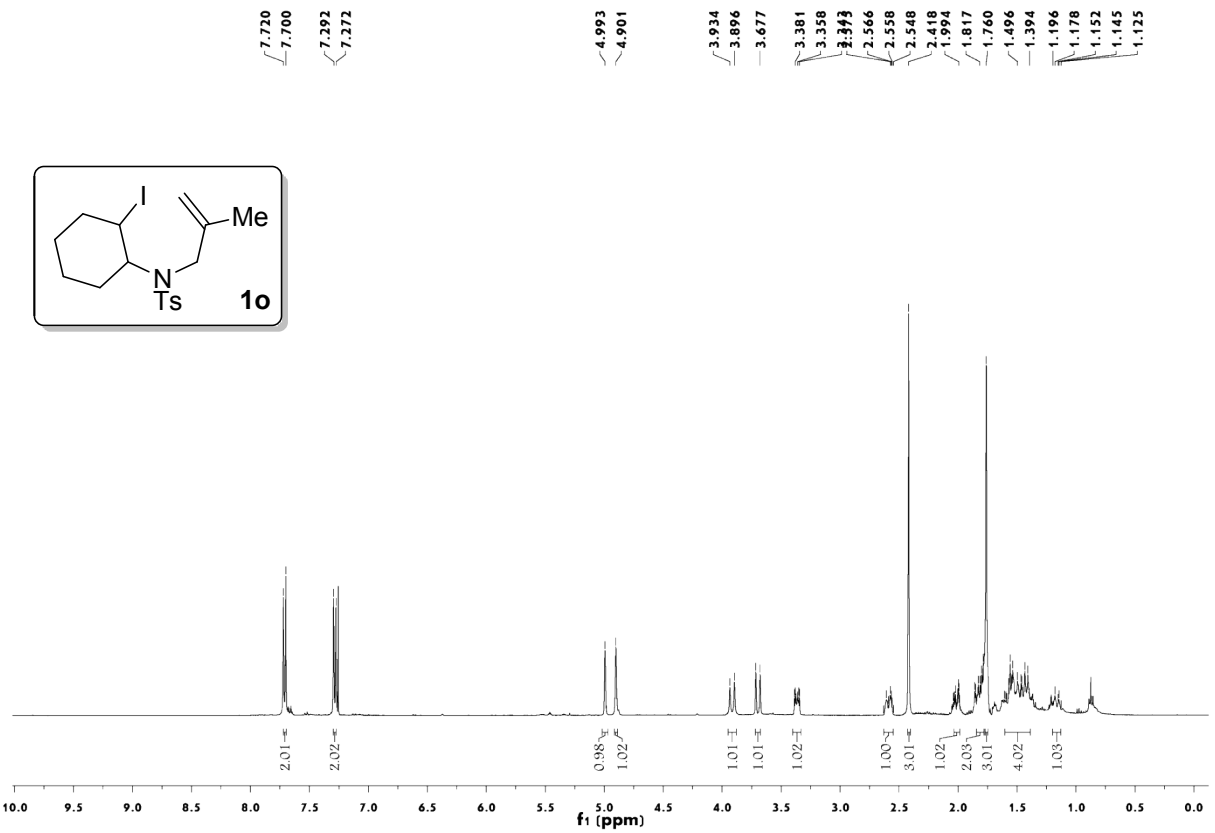
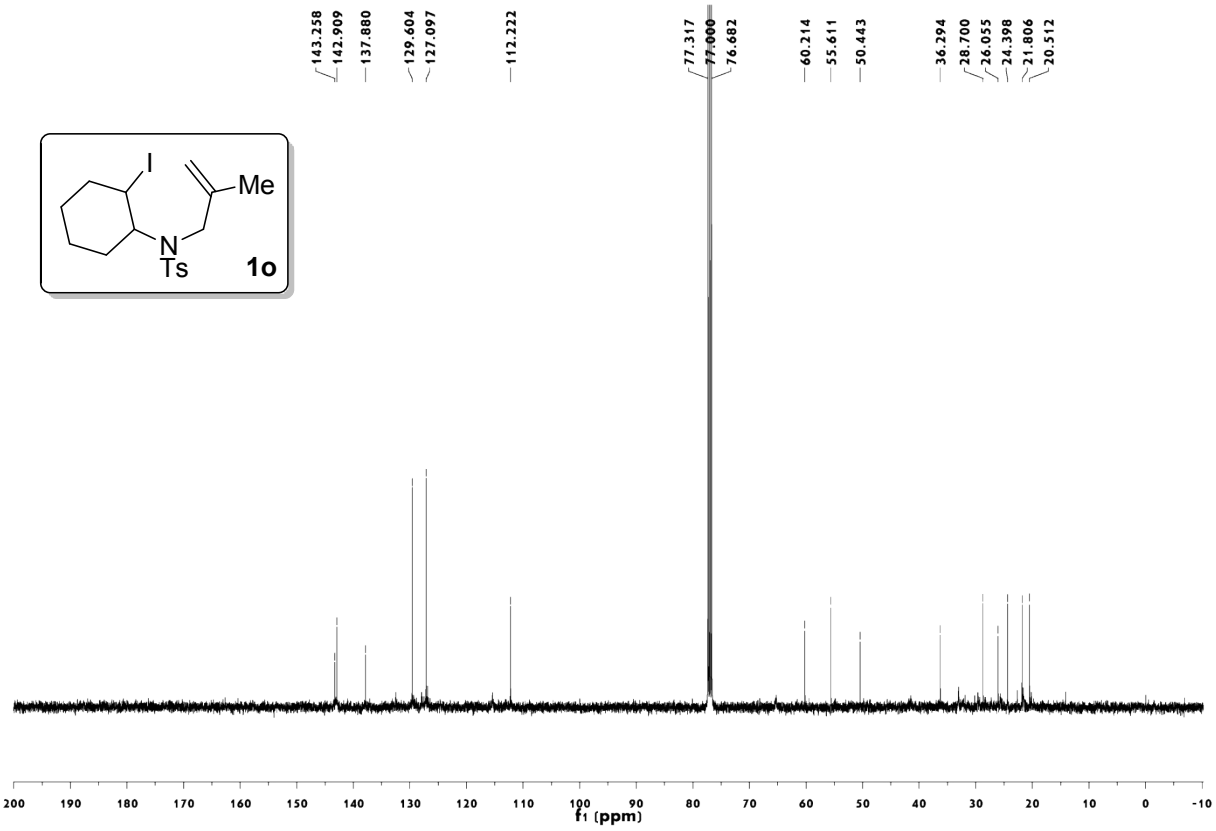
142.073
137.736
129.420
127.894
115.152
77.317
77.000
76.682
66.205
51.292
32.349
21.496
20.777
20.568
20.491
5.158
I
Me
Me
N
Ts
Me
1n
f1 (ppm)


7.720
7.700
7.292
7.272
4.993
4.901
3.934
3.896
3.677
3.381
3.358
2.566
2.558
2.548
2.418
1.994
1.817
1.760
1.496
1.394
1.196
1.178
1.152
1.145
1.125
I
Me
N
Ts
1o
2.01
2.02
0.98
1.02
1.01
1.01
1.02
1.00
3.01
1.02
2.03
3.01
4.02
1.03
f1 (ppm)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137.880
129.604
127.097
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77.317
77.000
76.682
60.214
55.611
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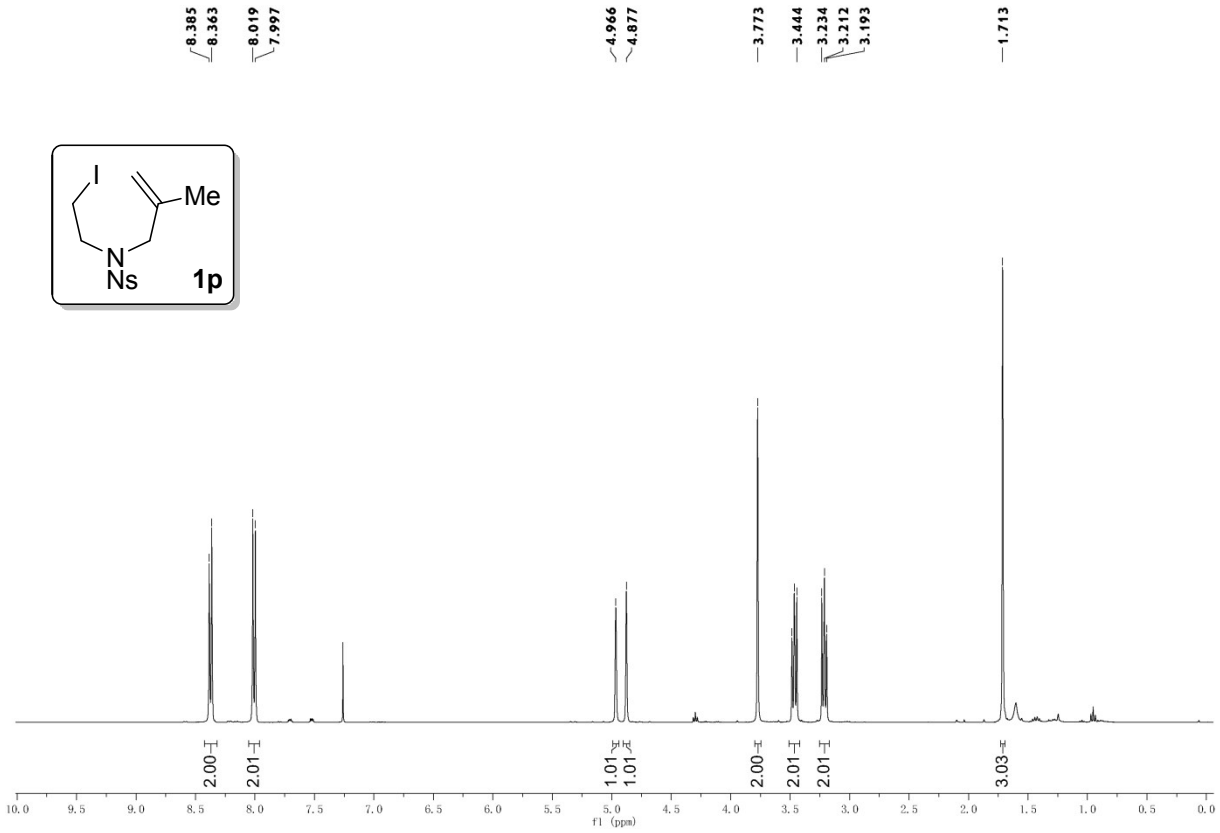
36.294
28.700
26.055
24.398
21.806
20.512
I
Me
N
Ts
1o
f1 (ppm)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8.363
8.019
7.997
4.966
4.877
3.773
3.444
3.234
3.212
3.193
1.713
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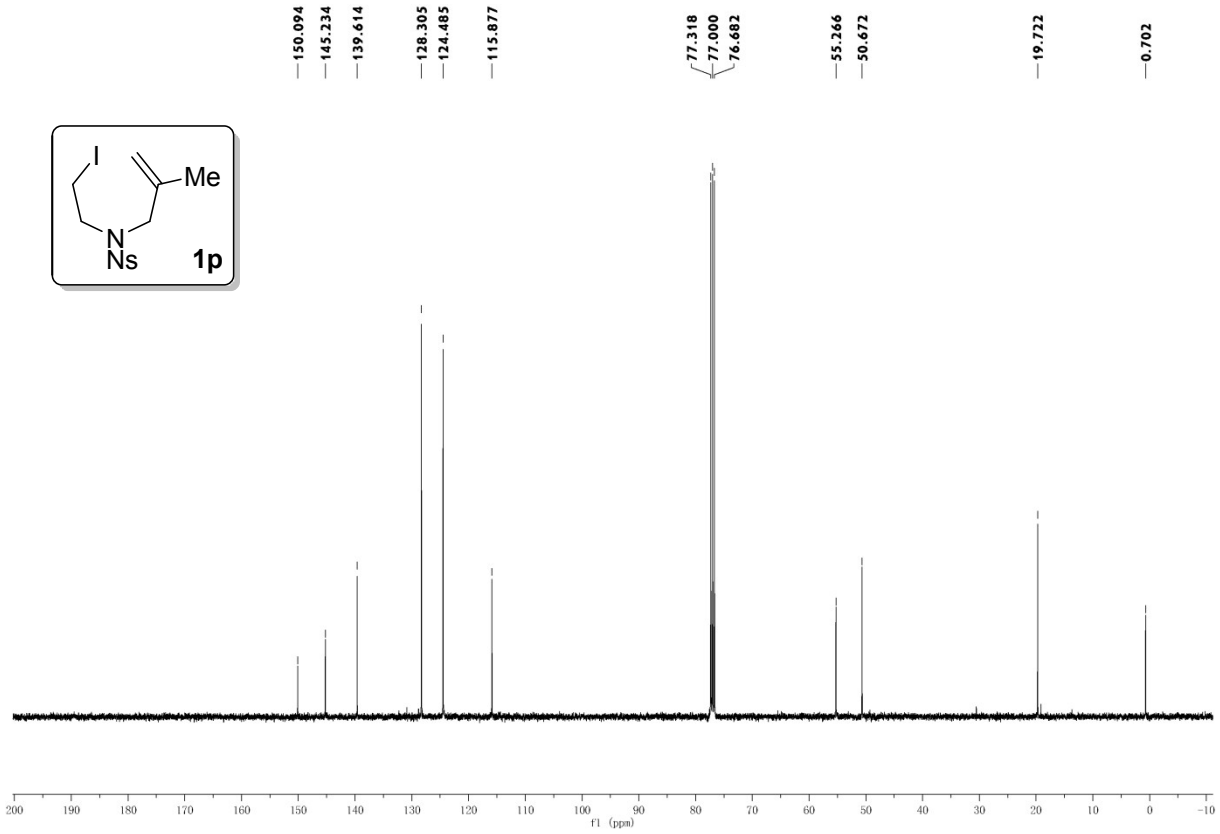
Me
N
Ns
1p
2.00
2.01
1.01
1.01
2.00
2.01
2.01
3.03
f1 (ppm)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145.234
139.614
128.305
124.485
115.877
77.318
77.000
76.682
55.266
50.672
19.722
0.702
I
Me
N
Ns
1p
f1 (ppm)