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Supporting Information

for

Electrochemical radical cation aza-Wacker cyclizations

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General remarks and characterization data, including copies of ^{1}H and ^{13}C NMR spectra

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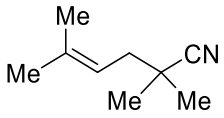
1. General remarks

All reagents and solvents were purchased from commercial sources and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) carried out on silica gel plates, with detection by UV absorption (254 nm) and by heating the plates after dipping them in a solution of 12 M molybdo(VI) phosphoric acid *n*-hydrate in 95% ethanol. Silica gel (particle size 40–50 μm, normal or reversed-phase) was used for column chromatography. ^{1}H NMR spectra were collected on a 600, 500, or 400 MHz NMR spectrometer using the deuterated solvent as an internal deuterium reference. Chemical shift data are given in δ units calibrated with residual protic solvent. The multiplicity of a signal is indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; sept, septet; m, multiplet. ^{13}C NMR spectra were collected at 150, 125, or 100 MHz with proton decoupling using the deuterated solvent as an internal carbon reference. Chemical shift data are given in δ units calibrated with residual solvent. High-resolution mass spectra (HRMS) were collected by electrospray ionization (ESI)- or direct analysis in real time (DART)-time-of-flight (TOF) spectrometers.

2. Synthetic procedure, characterization data

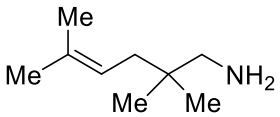
S1, **S2**–**S10**, **S12**, **S13**, **1**, and **11** were synthesized in a similar manner as previously reported by Yoon and co-workers.[1]

*Synthesis of 2,2,5-trimethylhex-4-enenitrile (**S1**) Yellow oil.*



To a solution of iPr_2NH (2.26 mL, 16.0 mmol) in THF (32 mL) stirred at 0 °C was added dropwise *n*-BuLi in hexane (1.52 M, 10.5 mL, 16.0 mmol). The resulting solution was stirred at 0 °C for 15 min, then, isobutyronitrile (1.45 mL, 16.0 mmol) was added dropwise, and the resulting solution was stirred at 0 °C for 15 min. Prenyl bromide (1.85 mL, 16.0 mmol) was added dropwise and the resulting reaction mixture was stirred at rt for 18 h. The reaction was quenched by sat. NH_4Cl aq. (80 mL) and extracted by Et_2O (40 mL × 3). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S1** (2.2 g) which was used without further purification.

*Synthesis of 2,2,5-trimethylhex-4-en-1-amine (**S2**) Colorless oil.*

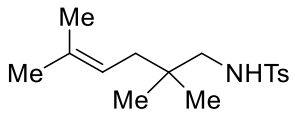


To a solution of $LiAlH_4$ (1.14 g, 30.0 mmol) in Et_2O (40 mL) stirred at 0 °C was slowly added **S1** (2.2 g) and the resulting reaction mixture was stirred at rt for 2 h. The consumption of starting materials was checked by TLC, the reaction mixture was cooled to 0 °C, and quenched by dropwise addition of 3 M NaOH aq. (10 mL).

The resulting mixture was stirred for 15 min, dried over Na_2SO_4, filtered, and concentrated in vacuo. Silica gel column chromatography (DCM/MeOH 20:1) gave the titled compound in 87% (**S2**, 1.98 g, 14.0 mmol). ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.18-5.15 (1H, m), 2.44 (2H, s), 1.89 (2H, d, J = 7.6 Hz), 1.72 (3H, s), 1.61 (3H, s), 1.20 (2H, m), 0.84 (6H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 133.0, 120.8, 52.8, 37.7, 35.8, 26.1, 24.7, 17.9; HRMS $[M + H]^+$ calculated for $[C_9H_{20}N]^+$ 142.1590, found 142.1586.

The characterization data is compatible with the reported values.[1]

*Synthesis of 4-methyl-N-(2,2,5-trimethylhex-4-en-1-yl)benzenesulfonamide (**1**) White solid.*

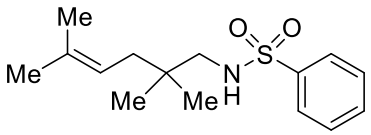


To a solution of **S2** (1.00 g, 7.08 mmol) and Et_3N (1.48 mL, 10.6 mmol) in DCM (24 mL) stirred at 0 °C was added TsCl (1.48 g, 7.76 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 84% (**1**, 1.76 g, 6.0 mmol).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (2H, d, J = 8.3 Hz), 7.31 (2H, d, J = 7.9 Hz), 5.03 (1H, tt, J = 7.9, 1.4 Hz), 4.83 (1H, t, J = 6.5 Hz), 2.66 (2H, d, J = 6.9 Hz), 2.43 (3H, s), 1.86 (2H, d, J = 7.6 Hz), 1.67 (3H, s), 1.54 (3H, s), 0.83 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.3, 137.0, 134.1, 129.7, 127.1, 119.8, 53.1, 37.9, 34.9, 26.0, 24.9, 21.6, 17.9; HRMS $[M + H]^+$ calculated for $[C_{16}H_{26}NO_2S]^+$ 296.1679, found 296.1700.

The characterization data is compatible with the reported values.[1]

*Synthesis of N-(2,2,5-trimethylhex-4-en-1-yl)benzenesulfonamide (**S3**) Colorless oil.*

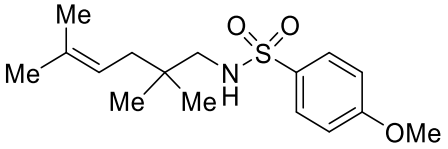


To a solution of **S2** (0.500 g, 3.54 mmol) and Et_3N (0.740 mL, 5.31 mmol) in DCM (12 mL) stirred at 0 °C was added benzenesulfonyl chloride (0.498 mL, 3.89 mmol). After stirring the resulting reaction mixture at rt for 3.5 h, the consumption of starting materials was checked by TLC, and the solution was concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 91% (**S3**, 905 mg, 3.2 mmol).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88-7.84 (2H, m), 7.61-7.49 (3H, m), 5.05 (1H, m), 4.44 (1H, m), 2.70 (2H, d, J = 6.9 Hz), 1.87 (2H, d, J = 7.6 Hz), 1.68 (3H, s), 1.55 (3H, s), 0.84 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 140.1, 134.2, 132.6, 129.1, 127.1, 119.8, 53.1, 37.9, 35.0, 26.1, 24.9, 18.0; HRMS $[M + H]^+$ calculated for $[C_9H_{20}N]^+$ 142.1590, found 142.1586.

The characterization data is compatible with the reported values.[1]

*Synthesis of 4-methoxy-N-(2,2,5-trimethylhex-4-en-1-yl)benzenesulfonamide (**S4**) White solid.*



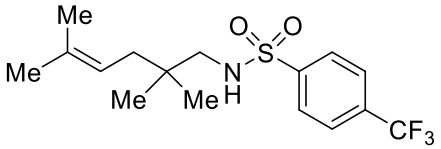
To a solution of **S2** (0.500 g, 3.54 mmol) and Et_3N (0.740 mL, 5.31 mmol) in DCM (12 mL) stirred at 0 °C was added 4-methoxybenzenesulfonyl chloride (0.802 g, 3.89 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was

concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 7:1) gave the titled compound in 94% (**S4** 1.03 g, 3.3 mmol).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (2H, d, J = 8.9 Hz), 6.98 (2H, d, J = 8.9 Hz), 5.04 (1H, m), 4.49 (1H, t, J = 6.9 Hz), 3.87 (3H, s), 2.67 (2H, d, J = 6.9 Hz), 1.86 (2H, d, J = 7.6 Hz), 1.67 (3H, s), 1.55 (3H, s), 0.83 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.7, 133.9, 131.6, 129.1, 119.8, 114.1, 55.6, 53.0, 37.8, 34.9, 26.0, 24.8, 17.8; HRMS $[M + H]^+$ calculated for $[C_{16}H_{26}NO_3S]^+$ 312.1628, found 312.1614.

The characterization data is compatible with the reported values.[1]

*Synthesis of 4-(trifluoromethyl)-N-(2,2,5-trimethylhex-4-en-1-yl)benzenesulfonamide (**S5**) White solid.*

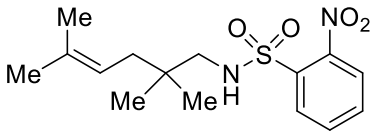


To a solution of **S2** (0.500 g, 3.54 mmol) and Et_3N (0.740 mL, 5.31 mmol) in DCM (12 mL) stirred at 0 °C was added 4-(trifluoromethyl)benzenesulfonyl chloride (0.949 g, 3.89 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 91% (**S5**, 1.13 g, 3.23 mmol).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (2H, d, J = 8.3 Hz), 7.80 (2H, d, J = 8.3 Hz), 5.05 (1H, tt, J = 1.7, 8.4 Hz), 4.47 (1H, t, J = 6.2 Hz), 2.74 (2H, d, J = 6.5 Hz), 1.88 (2H, d, J = 7.9 Hz), 1.68 (3H, s), 1.56 (3H,s), 0.86 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.7, 134.4, 127.6, 126.3, 126.3, 119.5, 53.2, 37.9, 35.0, 26.0, 24.9, 17.9; HRMS $[M + H]^+$ calculated for $[C_{16}H_{23}F_3NO_2S]^+$ 350.1396, found 350.1389.

The characterization data is compatible with the reported values.[1]

*Synthesis of 2-nitro-N-(2,2,5-trimethylhex-4-en-1-yl)benzenesulfonamide (**S6**) Colorless oil.*

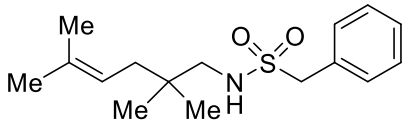


To a solution of **S2** (0.500 g, 3.54 mmol) and Et_3N (0.740 mL, 5.31 mmol) in DCM (12 mL) stirred at 0 °C was added 2-NsCl (0.862 g, 3.89 mmol). After stirring the resulting reaction mixture at rt for 1 h, the consumption of starting materials was checked by TLC, and the solution was removed by vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 7:1) gave the titled compound in 80% (**S6**, 0.920 g, 2.8 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13-8.10 (1H, m), 7.87-7.85 (1H, m), 7.76-7.73 (2H, m), 5.30 (1H, t, J = 6.4 Hz), 5.12-5.08 (1H, m), 2.84 (2H, d, J = 6.9 Hz), 1.93 (2H, d, J = 7.8 Hz), 1.70 (3H, s), 1.59 (3H,s), 0.89 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.1, 134.5, 133.7, 133.5, 132.8, 131.1, 125.4, 119.5, 53.5, 37.9, 35.1, 26.1, 25.0, 17.9; HRMS $[M + H]^+$ calculated for $[C_{15}H_{23}N_2O_4S]^+$ 327.1373, found 327.1392.

The characterization data is compatible with the reported values.[1]

*Synthesis of 1-phenyl-N-(2,2,5-trimethylhex-4-en-1-yl)methanesulfonamide (**S7**) White solid.*

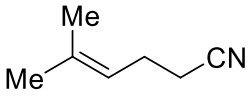


To a solution of **S2** (0.500 g, 3.54 mmol) and Et_3N (0.740 mL, 5.31 mmol) in DCM (12 mL) stirred at 0 °C was added benzylsulfonyl chloride (0.742 g, 3.89 mmol). After stirring the resulting reaction mixture at rt for 1 h, the consumption of starting materials was checked by TLC, and the solution was removed by vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 84% (**S7**, 875 mg, 2.96 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.36 (5H, m), 5.10-5.06 (1H, m), 4.38 (1H, t, J = 6.4 Hz), 4.23 (2H, s), 2.75 (2H, d, J = 6.4 Hz), 1.86 (2H, d, J = 7.3 Hz), 1.69 (3H, s), 1.54 (3H,s), 0.85 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 134.0, 130.6, 129.5, 128.8, 128.7, 119.7, 58.5, 53.5, 37.8, 35.1, 26.0, 24.7, 17.9; HRMS [M + H]$^+$ calculated for $[C_{16}H_{26}NO_2S]^+$ 296.1679, found 296.1650.

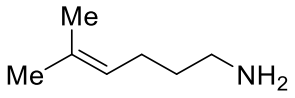
The characterization data is compatible with the reported values.[1]

*Synthesis of 5-methylhex-4-enenitrile (**S8**) Yellow oil.*



To a solution of MeCN (0.370 mL, 7.04 mmol) in THF (18 mL) stirred at −78 °C was added dropwise *n*-BuLi in hexane (1.52 M, 4.77 mL, 7.25 mmol). The resulting solution was stirred at −78 °C for 15 min, prenyl bromide (1.00 mL, 8.66 mmol) in THF (9 mL) was added dropwise and the resulting reaction mixture was stirred at rt for 2 h. The reaction was quenched by sat. NH_4Cl aq. (50 mL) and extracted by Et_2O (40 mL × 3). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S8** (2.2 g) which was used without further purification.

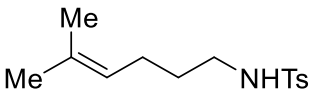
*Synthesis of 5-methylhex-4-en-1-amine (**S9**) Yellow oil.*



To a solution of $LiAlH_4$ (1.29 g, 33.9 mmol) in Et_2O (75 mL) stirred at 0 °C was slowly added **S8** (2.2 g) in Et_2O. After stirring the resulting reaction mixture at rt for 2 h the consumption of starting materials was checked by TLC, the reaction mixture was cooled to 0 °C, and quenched by dropwise addition of 3 M aq.

NaOH (10 mL). The resulting mixture was stirred 15 min, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S9** (1.3 g) which was used without further purification.

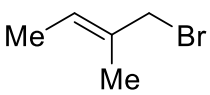
*Synthesis of 4-methyl-N-(5-methylhex-4-en-1-yl)benzenesulfonamide (**S10**) Colorless oil.*



To a solution of **S9** (1.3 g) and Et_3N (2.40 mL, 17.2 mmol) in DCM (30 mL) stirred at 0 °C was added TsCl (2.68 g, 14 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 58% (**S10**, 1.09 g, 4.1 mmol) over 3 steps.

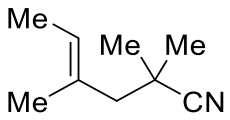
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.77 (2H, d, J = 8.9 Hz), 7.30 (2H, d, J = 7.6 Hz), 5.21-5.15 (1H, m), 4.98 (1H, t, J = 6.9 Hz), 2.91 (2H, q, J = 6.9 Hz), 2.42 (3H, s), 1.94 (2H, q, J = 7.6 Hz), 1.64 (3H, s), 1.52 (3H,s), 1.48 (2H, quint, J = 7.6 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 143.3, 137.1, 132.6, 129.7, 127.2, 123.2, 43.0, 29.6, 25.7, 25.1, 21.6, 17.7; HRMS $[M + H]^+$ calculated for $[C_{14}H_{22}NO_2S]^+$ 268.1366, found 268.1390. The characterization data is compatible with the reported values.[1]

*Synthesis of (E)-1-Bromo-2-methylbut-2-ene (**S11**). Colorless oil.*



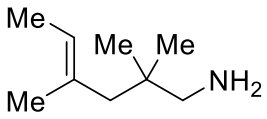
Synthesized according to the method reported by Alexanian and co-workers.[2] The characterization data is compatible with the reported values.[3]

*Synthesis of (E)-2,2,4-trimethylhex-4-enenitrile (**S12**) Yellow oil.*



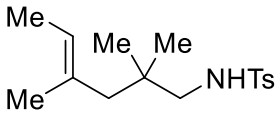
To a solution of iPr_2NH (1.13 mL, 8.00 mmol) in THF (20 mL) stirred at 0 °C was added dropwise *n*-BuLi in hexane (1.52 M, 5.25 mL, 8.00 mmol). The resulting solution was stirred at 0 °C for 15 min, isobutyronitrile (0.725 mL, 8.00 mmol) was added dropwise, and the resulting solution was stirred at 0 °C for 15 min. Then, **S11** (1.19 g, 8.00 mmol) in THF (5 mL) was added dropwise and the resulting reaction mixture was stirred at rt for 18 h. The reaction was quenched by sat. NH_4Cl aq. (40 mL) and extracted by Et_2O (40 mL × 3). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S12** (1.3 g) which was used without further purification.

*Synthesis of (E)-2,2,4-trimethylhex-4-en-1-amine (**S13**) Colorless oil.*



To a solution of $LiAlH_4$ (0.500 g, 13.2 mmol) in THF (20 mL) stirred at 0 °C was slowly added **S12** (1.3 g). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, the reaction mixture was cooled to 0 °C, and quenched by dropwise addition of 3 M aq. NaOH (10 mL). The resulting mixture was stirred 15 min, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S13** (1.0 g) which was used without further purification.

*Synthesis of (E)-4-methyl-N-(2,2,4-trimethylhex-4-en-1-yl)benzenesulfonamide (**11**) White solid.*

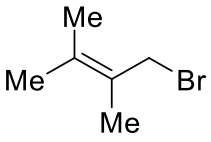


To a solution of **S13** (1.0 g) and Et_3N (1.48 mL, 10.6 mmol) in DCM (24 mL) stirred at 0 °C was added TsCl (1.48 g, 7.76 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 34% (**11**, 800 mg, 2.7 mmol, *cis*/*trans* = 1:15) over 3 steps.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (2H, d, J = 8.3 Hz), 7.31 (2H, d, J = 8.3 Hz), 5.12 (1H, q, J = 7.6 Hz), 4.46 (1H, t, J = 6.9 Hz), 2.68 (2H, d, J = 6.9 Hz), 2.43 (3H, s), 1.89 (2H, s), 1.60 (3H, s), 1.54 (3H,d, J = 6.9 Hz), 0.85 (6H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 143.4, 137.0, 132.8, 129.7, 127.2, 123.5, 53.5, 49.5, 35.1, 25.7, 21.6, 18.6, 13.7; HRMS $[M + H]^+$ calculated for $[C_{16}H_{26}NO_2S]^+$ 296.1679, found 296.1671.

The characterization data is compatible with the reported values.[1]

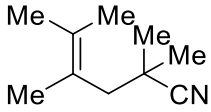
*Synthesis of 1-bromo-2,3-dimethylbut-2-ene (**S14**) Colorless oil.*



Synthesized according to the method reported by Zhang and co-workers.[3] The characterization data is compatible with the reported values.[3]

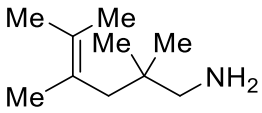
All spectral data were in agreement with previously reported values.

*Synthesis of 2,2,4,5-tetramethylhex-4-enenitrile (**S15**) Yellow oil.*



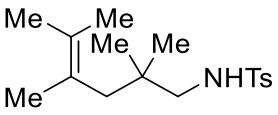
To a solution of iPr_2NH (1.13 mL, 8.00 mmol) in THF (16 mL) stirred at 0 °C was added dropwise *n*-BuLi in hexane (1.52 M, 5.25 mL, 8.00 mmol). The resulting solution was stirred at 0 °C for 15 min, isobutyronitrile (0.725 mL, 8.00 mmol) was added dropwise, and the resulting solution was stirred at 0 °C for 15 min. Then, **S14** (1.30 g, 8.00 mmol) in THF (5 mL) was added dropwise and the resulting reaction mixture was stirred at rt for 18 h. The reaction mixture was quenched by sat. NH_4Cl aq. (40 mL) and extracted by Et_2O (40 mL × 3). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S15** (0.46 g) which was used without further purification.

*Synthesis of 2,2,4,5-tetramethylhex-4-en-1-amine (**S16**) Yellow oil.*



To a solution of $LiAlH_4$ (171 mg, 4.50 mmol) in THF (20 mL) stirred at 0 °C was slowly added **S15** (0.46 g) in THF (5 mL). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, the reaction mixture was cooled to 0 °C, and quenched by dropwise addition of 3 M NaOH aq. (10 mL). The resulting mixture was stirred 15 min, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S16** (0.47 g) which was used without further purification.

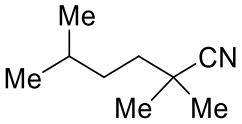
*Synthesis of 4-methyl-N-(2,2,4,5-tetramethylhex-4-en-1-yl)benzenesulfonamide (**14**) White solid.*



To a solution of **S16** (0.47 g) and Et_3N (0.630 mL, 4.50 mmol) in DCM (12 mL) stirred at 0 °C was added TsCl (0.630 g, 3.30 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solution was removed by vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 20% (**14**, 491 mg, 1.6 mmol) over 3 steps.

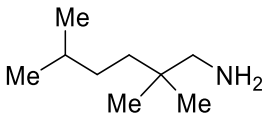
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 (2H, d, J = 8.3 Hz), 7.31 (2H, d, J = 7.6 Hz), 4.74 (1H, t, J = 6.2 Hz), 2.70 (2H, d, J = 6.9 Hz), 2.43 (3H, s), 2.00 (2H, s), 1.63 (6H, s), 1.57 (3H,s), 0.87 (6H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 143.3, 137.1, 129.7, 128.4, 127.2, 125.0, 54.4, 44.4, 36.7, 25.7, 21.8, 21.7, 21.6, 21.0; HRMS $[M + H]^+$ calculated for $[C_{17}H_{28}NO_2S]^+$ 310.1835, found 310.1860.

*Synthesis of 2,2,5-trimethylhexanenitrile (**S17**) Yellow oil.*



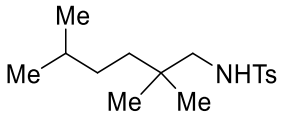
To a solution of iPr_2NH (2.26 mL, 16.0 mmol) in THF (32 mL) stirred at 0 °C was added dropwise *n*-BuLi in hexane (1.52M, 10.5 mL, 16.0 mmol). The resulting solution was stirred at 0 °C for 15 min, isobutyronitrile (1.45 mL, 16.0 mmol) was added dropwise, and the resulting solution was stirred at 0 °C for 15 min. A solution of 1-bromo-3-methylbutane (2.01 mL, 16.0 mmol) in THF (2 mL) was added dropwise and the resulting reaction mixture was stirred at rt for 18 h. The reaction was quenched by sat. NH_4Cl aq. (80 mL) and extracted by Et_2O (40 mL × 3). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S17** (3.1 g) which was used without further purification.

*Synthesis of 2,2,5-trimethylhexan-1-amine (**S18**) Yellow oil.*



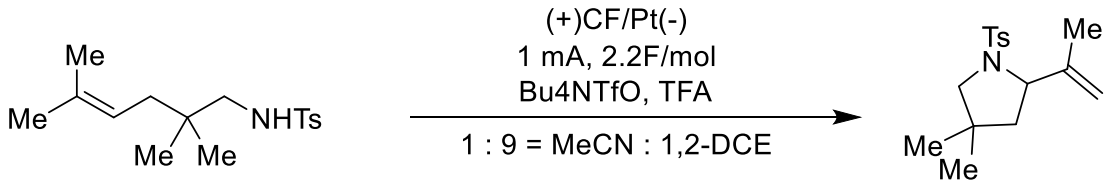
To a solution of $LiAlH_4$ (1.34 g, 35.2 mmol) in Et_2O (40 mL) stirred at 0 °C was slowly added **S17** (3.1 g). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, the reaction mixture was cooled to 0 °C, and quenched by dropwise addition of 3 M NaOH aq. (10 mL). The resulting mixture was stirred 15 min, dried over Na_2SO_4, filtered, and concentrated in vacuo to give crude **S18** (2.4 g) which was used without further purification.

*Synthesis of 4-methyl-N-(2,2,5-trimethylhexyl)benzenesulfonamide (**S19**) White solid.*



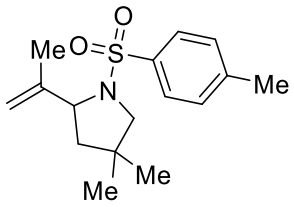
To a solution of **S18** (2.4 g) and Et_3N (3.35 mL, 24.0 mmol) in DCM (30 mL) stirred at 0 °C was added TsCl (3.66 g, 19.2 mmol). After stirring the resulting reaction mixture at rt for 2 h, the consumption of starting materials was checked by TLC, and the solvent was removed by vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the titled compound in 49% (**S19**, 2.32 g, 7.8 mmol) over 3 steps.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (2H, d, J = 8.2 Hz), 7.31 (2H, d, J = 8.2 Hz), 4.73 (1H, t, J = 6.4 Hz), 2.66 (2H, d, J = 6.9 Hz), 2.43 (3H, s), 1.37 (1H, sept, J = 6.4 Hz), 1.16-1.12 (2H, m), 1.03-0.97 (2H, m), 0.83-0.82 (12H, m); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.3, 137.1, 129.7, 127.2, 53.1, 37.2, 33.7, 32.7, 28.7, 24.9, 22.7, 21.6; HRMS $[M + H]^+$ calculated for $[C_{16}H_{28}NO_2S]^+$ 298.1835, found 298.1856.



General procedure for electrochemical aza-Wacker cyclizations. The appropriate alkene (0.20 mmol), TFA (0.20 mmol), and CH_3CN (0.4 mL) were added to a solution of Bu_4NOTf/1,2-DCE (0.10 M, 3.6 mL) while stirring at room temperature. The resulting reaction mixture was electrolyzed at 1 mA using CF anode (10 mm × 10 mm) and Pt cathode (10 mm × 20 mm) in an undivided cell with stirring. Then, the solution was diluted with sat. aq. $NaHCO_3$ and extracted with dichloromethane. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the corresponding ring compounds.

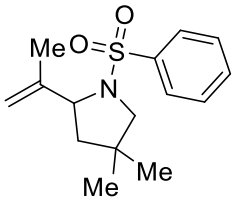
*4,4-Dimethyl-2-(prop-1-en-2-yl)-1-tosylpyrrolidine (**2**) White solid.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (2H, d, J = 8.2 Hz), 7.30 (2H, d, J = 7.8 Hz), 4.96 (1H, s), 4.82 (1H, m), 4.06 (1H, t, J = 8.7 Hz), 3.26 (1H, d, J = 11.0 Hz), 3.19 (1H, d, J = 11.0 Hz), 2.42 (3H, s), 1.72 (3H, s), 1.66-1.59 (2H, m), 1.03 (3H,s), 0.57 (3H,s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 145.3, 143.2, 135.3, 129.5, 127.6, 112.0, 65.6, 62.0, 46.3, 37.4, 26.3, 25.9, 21.6, 17.0; HRMS $[M + H]^+$ calculated for $[C_{16}H_{24}NO_2S]^+$ 294.1522, found 294.1504.

The characterization data is compatible with the reported values.[1]

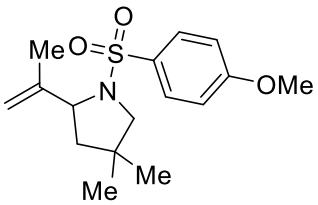
*4,4-Dimethyl-1-(phenylsulfonyl)-2-(prop-1-en-2-yl)pyrrolidine (**4**) Colorless oil.*



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83-7.82 (2H, m), 7.58 (1H, tt, J = 6.9, 2.1 Hz), 7.52 (2H, t, J = 7.6 Hz), 4.97 (1H, s), 4.83 (1H, m), 4.09 (1H, dd, J = 9.6, 7.6 Hz), 3.28 (1H, dd, J = 11.0, 1.4 Hz), 3.20 (1H, d, J = 11.0 Hz), 1.71 (3H, s), 1.68-1.61 (2H, m), 1.03 (3H, s), 0.55 (3H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 145.2, 138.3, 132.6, 128.9, 127.5, 112.1, 65.7, 62.0, 46.3, 37.5, 26.2, 25.8, 17.0; HRMS $[M + H]^+$ calculated for $[C_{15}H_{22}NO_2S]^+$ 280.1366, found 280.1339.

The characterization data is compatible with the reported values.[1]

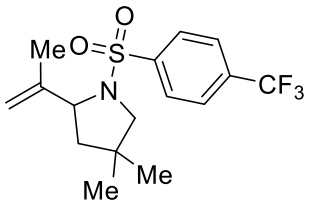
*1-((4-Methoxyphenyl)sulfonyl)-4,4-dimethyl-2-(prop-1-en-2-yl)pyrrolidine (**5**) White solid.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.34 (2H, m), 6.99-6.96 (2H, m), 4.96 (1H, s), 4.82 (1H, t, J = 1.4 Hz), 4.04 (1H, t, J = 8.7 Hz), 3.87 (3H, s), 3.22 (2H, q, J = 10.5 Hz), 1.73 (3H, s), 1.69-1.60 (2H, m), 1.04 (3H,s), 0.60 (3H,s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.8, 145.4, 130.1, 129.6, 114.0, 111.9, 65.6, 62.0, 55.7, 46.4, 37.4, 26.3, 26.0, 17.0; HRMS $[M + H]^+$ calculated for $[C_{16}H_{24}NO_3S]^+$ 310.1471, found 310.1479.

The characterization data is compatible with the reported values.[1]

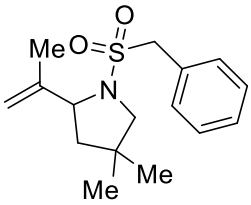
*4,4-Dimethyl-2-(prop-1-en-2-yl)-1-((4-(trifluoromethyl)phenyl)sulfonyl)pyrrolidine (**6**) White solid.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (2H, d, J = 8.2 Hz), 7.78 (2H, d, J = 8.2 Hz), 4.95 (1H, s), 4.83 (1H, t, J = 1.4 Hz), 4.16 (1H, dd, J = 9.6, 7.3 Hz), 3.35 (1H, dd, J = 10.5, 1.8 Hz), 3.19 (1H, d, J = 11.0 Hz), 1.73 (1H, ddd, J = 12.8, 7.8, 1.4 Hz), 1.67-1.61 (1H, m), 1.64 (3H, s), 1.06 (3H,s), 0.67 (3H,s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 144.5, 142.3, 134.2 (d, J = 33.6 Hz), 127.9, 126.0 (q, J = 3.8 Hz), 123.4 (d, J = 272.2 Hz), 112.8, 65.8, 62.0, 46.1, 37.7, 25.9, 25.8, 16.9; HRMS $[M + H]^+$ calculated for $[C_{16}H_{21}F_3NO_2S]^+$ 348.1240, found 348.1225.

The characterization data is compatible with the reported values.[1]

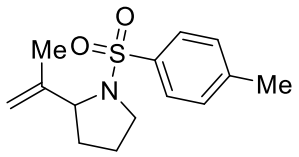
*1-(Benzylsulfonyl)-4,4-dimethyl-2-(prop-1-en-2-yl)pyrrolidine (**8**) White solid.*



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44-7.33 (5H, m), 5.03 (1H, s), 4.88 (1H, t, J = 2.1 Hz), 4.49 (1H, dd, J = 9.6, 7.6 Hz), 4.23 (1H, d, J = 13.1 Hz), 4.13 (1H, d, J = 13.8 Hz), 3.30 (1H, dd, J = 10.3, 2.1 Hz), 2.77 (1H, d, J = 10.3 Hz), 1.82 (1H, ddd, J = 12.4, 7.6, 1.4 Hz), 1.71 (3H, s), 1.57 (1H, dd, J = 12.4, 9.6 Hz), 1.03 (3H,s), 1.02 (3H,s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 144.8, 130.9, 129.3, 128.8, 128.7, 128.6, 112.8, 65.3, 61.9, 59.4, 46.0, 38.5, 25.7, 25.7, 17.8; HRMS $[M + H]^+$ calculated for $[C_{16}H_{24}NO_2S]^+$ 294.1522, found 294.1552.

The characterization data is compatible with the reported values.[1]

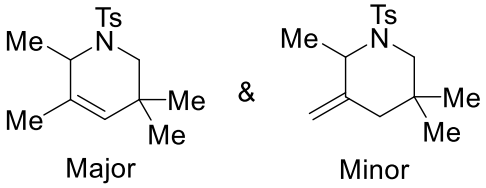
*2-(Prop-1-en-2-yl)-1-tosylpyrrolidine (**10**) Colorless oil.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (2H, d, J = 8.2 Hz), 7.31 (2H, d, J = 8.2 Hz), 5.00 (1H, s), 4.86 (1H, s), 4.04 (1H, t, J = 6.4 Hz), 3.49-3.43 (1H, m), 3.28 (1H, dt, J = 10.1, 7.3 Hz), 2.43 (3H, s), 1.85-1.76 (1H, m), 1.74 (3H, s), 1.72-1.66 (2H, m), 1.59-1.50 (1H, m); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 145.1, 143.3, 135.0, 129.6, 127.6, 111.8, 65.0, 49.3, 31.5, 24.1, 21.6, 18.7; HRMS $[M + H]^+$ calculated for $[C_{14}H_{20}NO_2S]^+$ 266.1209, found 266.1217.

The characterization data is compatible with the reported values.[1]

*3,3,5,6-Tetramethyl-1-tosyl-1,2,3,6-tetrahydropyridine and 2,5,5-trimethyl-3-methylene-1-tosylpiperidine (**12**, 2:1 r.r.) White solid.*



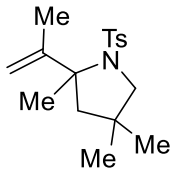
Isolated as a 2:1 mixture of regioisomers.

Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (2H, d, J = 8.3 Hz), 7.28-7.26 (2H, m), 5.14 (1H, s), 4.18 (1H, q, J = 6.9 Hz), 3.42 (1H, d, J = 12.4 Hz), 2.78 (1H, d, J = 13.1 Hz), 2.41 (3H, s), 1.63 (3H, s), 1.04 (3H, d, J = 6.9 Hz), 0.93 (3H, s), 0.91 (3H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 142.9, 138.8, 132.8, 131.1, 129.6, 127.2, 52.6, 49.7, 33.3, 27.0, 26.8, 21.6, 20.8, 16.3.

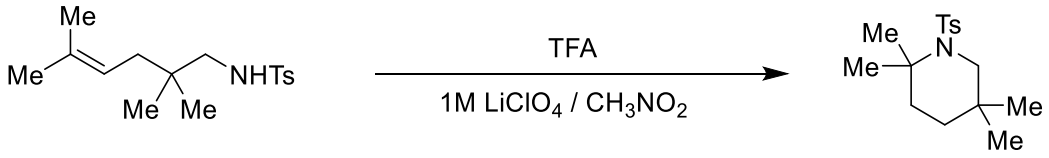
Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (2H, d, J = 8.3 Hz), 7.28-7.26 (2H, m), 4.87 (1H, s), 4.65 (1H, t, J = 2.1 Hz), 4.62 (1H, q, J = 6.9 Hz), 3.27 (1H, dd, J = 14.4, 1.4 Hz), 2.83 (1H, d, J = 13.1 Hz), 2.41 (3H, s), 2.14 (1H, d, J = 13.8 Hz), 1.81 (1H, dd, J = 15.8, 2.1 Hz), 1.09 (3H, d, J = 6.9 Hz), 0.94 (3H, s), 0.74 (3H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 144.2, 142.9, 138.4, 129.6, 127.1, 110.9, 55.2, 50.6, 42.2, 32.7, 28.0, 23.5, 21.6, 16.5.

HRMS $[M + H]^+$ calculated for $[C_{16}H_{24}NO_2S]^+$ 294.1522, found 294.1526.

*2,4,4-Trimethyl-2-(prop-1-en-2-yl)-1-tosylpyrrolidine (**15**) White solid.*

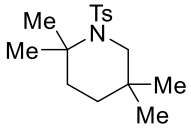


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (2H, d, J = 8.2 Hz), 7.27 (2H, d, J = 8.2 Hz), 5.09 (1H, s), 4.87 (1H, s), 3.23 (1H, d, J = 10.1 Hz), 3.18 (1H, d, J = 9.6 Hz), 2.42 (3H, s), 1.97 (1H, d, J = 13.3 Hz), 1.79 (3H, s), 1.65 (3H, s), 1.59 (1H, d, J = 13.3 Hz), 1.06 (3H, s), 0.94 (3H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 149.7, 142.8, 138.3, 129.3, 127.4, 110.9, 71.3, 62.4, 54.9, 36.5, 28.3, 27.7, 25.7, 21.6, 19.8; HRMS $[M + H]^+$ calculated for $[C_{17}H_{26}NO_2S]^+$ 308.1679, found 308.1656.



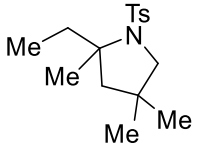
General procedure for non-electrochemical cyclization. The appropriate alkene (0.20 mmol) and TFA (0.20 mmol) were added to a solution of $LiClO_4/CH_3NO_2$ (1.0 M, 4 mL) and the resulting reaction mixture was stirred at rt for 18 h. The solution was diluted with sat. $NaHCO_3$ aq. and extracted with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. Silica gel column chromatography (*n*-hexane/EtOAc 10:1) gave the corresponding ring compounds.

*2,2,5,5-Tetramethyl-1-tosylpiperidine (**3**) White solid.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (2H, d, J = 8.7 Hz), 7.24 (2H, d, J = 8.2 Hz), 3.14 (2H, s), 2.39 (3H, s), 1.47-1.44 (2H, m), 1.33-1.30 (2H, m), 1.24 (6H, s), 0.94 (6H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 142.7, 140.3, 129.4, 127.3, 57.6, 54.2, 38.0, 33.8, 31.1, 26.3, 25.8, 21.6; HRMS $[M + H]^+$ calculated for $[C_{16}H_{16}NO_2S]^+$ 296.1679, found 296.1683.

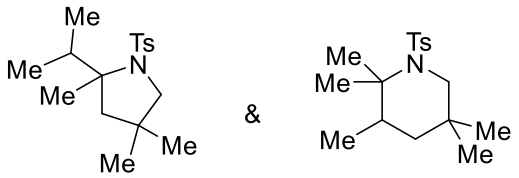
*2-Ethyl-2,4,4-trimethyl-1-tosylpyrrolidine (**13**) White solid.*



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (2H, d, J = 8.7 Hz), 7.27 (2H, d, J = 8.2 Hz), 3.09 (1H, d, J = 9.6 Hz), 3.04 (1H, d, J = 9.6 Hz), 2.41 (3H, s), 1.97 (1H, sext, J = 6.0 Hz), 1.87-1.78 (2H, m), 1.49 (1H, d, J = 12.4

Hz), 1.48 (3H, s), 1.01 (3H, s), 0.97 (3H, s), 0.89 (3H, t, J = 7.3 Hz); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 142.6, 138.7, 129.3, 127.3, 69.7, 61.7, 52.5, 36.2, 34.8, 27.7, 27.5, 27.1, 21.5, 9.5; HRMS $[M + H]^+$ calculated for $[C_{16}H_{26}NO_2S]^+$ 296.1679, found 296.1665.

*2-Isopropyl-2,4,4-trimethyl-1-tosylpyrrolidine (**16**), 2,2,3,5,5-pentamethyl-1-tosylpiperidine (**17**) White solid.*



16 **17**

*Isolated as a 1:1 mixture of **16** & **17**.*

(**16**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (2H, d, J = 8.2 Hz), 7.24-7.22 (2H, m), 3.10 (1H, d, J = 10.1 Hz), 2.98 (1H, d, J = 10.1 Hz), 2.52 (1H, sept, J = 6.9 Hz), 2.38 (3H, s), 1.85 (1H, d, J = 13.3 Hz), 1.53 (3H, s), 1.27 (1H, d, J = 13.3 Hz), 0.97 (3H, s), 0.90 (3H, d, J = 6.9 Hz), 0.84 (3H, s), 0.79 (3H, d, J = 6.9 Hz); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 142.5, 138.9, 129.3, 127.2, 73.1, 61.8, 47.6, 38.0, 36.0, 27.9, 27.0, 24.5, 18.6, 17.1.

(**17**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (2H, d, J = 8.7 Hz), 7.24-7.22 (2H, m), 3.66 (1H, dd, J = 13.3, 1.4 Hz), 2.82 (1H, d, J = 12.8 Hz), 2.38 (3H, s), 1.78 (1H, sext, J = 6.4 Hz), 1.39 (3H, s), 1.23-1.13 (2H, m), 1.03 (3H, s), 0.95 (3H, s), 0.87 (3H, s), 0.73 (3H, d, J = 6.9 Hz); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 142.5, 141.1, 129.4, 127.1, 62.6, 53.9, 43.3, 37.8, 31.2, 29.0, 27.4, 26.7, 21.5, 17.8, 16.1.

HRMS $[M + H]^+$ calculated for $[C_{17}H_{28}NO_2S]^+$ 310.1835, found 310.1837.

References

[1] Reed, N. L.; Lutovsky, G. A.; Yoon, T. P. *J. Am. Chem. Soc.* **2021**, *143*, 6065–6070.

[2] Venning, A. R. O.; Kwiatkowski, M. R.; Roque Peña, J. E.; Lainhart, B. C.; Guruparan, A. A.; Alexanian, E. J. *J. Am. Chem. Soc.* **2017**, *139*, 11595–11600.

[3] Kou, X.; Shao, Q.; Ye, C.; Yang, G.; Zhang, W. *J. Am. Chem. Soc.* **2018**, *140*, 7587–7597.

3. Cyclic Voltammetry Studies

Cyclic voltammetry experiments were performed in 1,2-DCE (4.5 mL), CH_3CN (0.5 mL) with analyte (2 mM) and Bu_4NOTf (0.1 M) using a glassy carbon working electrode, platinum wire electrode, Ag/AgCl KCl aq. reference electrode, and a scan rate of 50 mV/s.

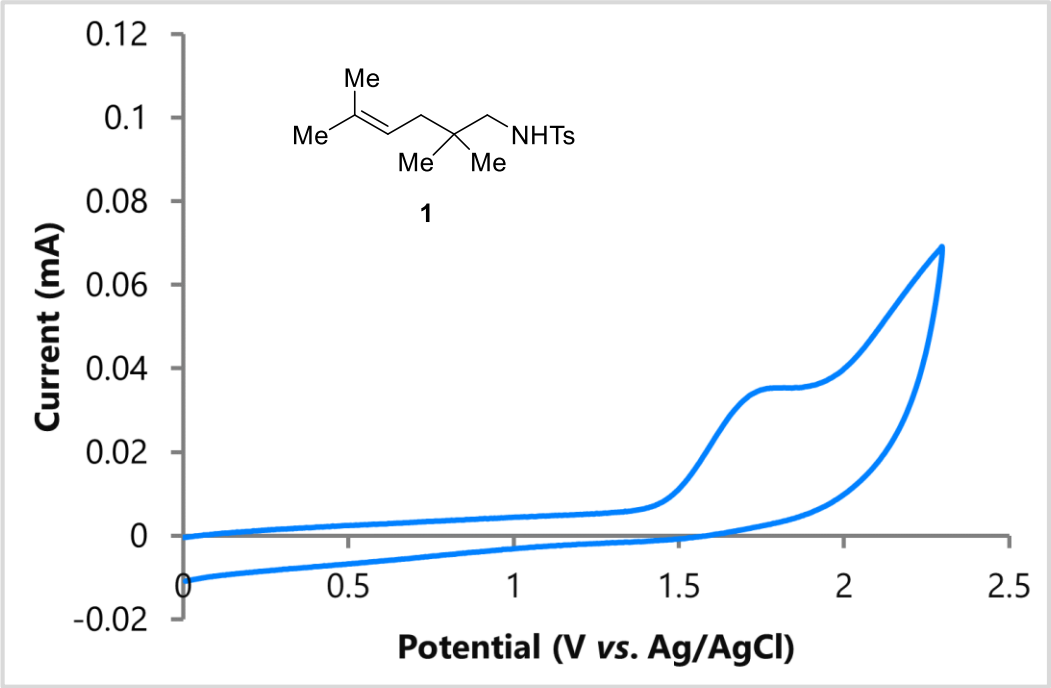


Figure S1. Cyclic voltammogram of alkene **1**, E_P = 1.77 V.

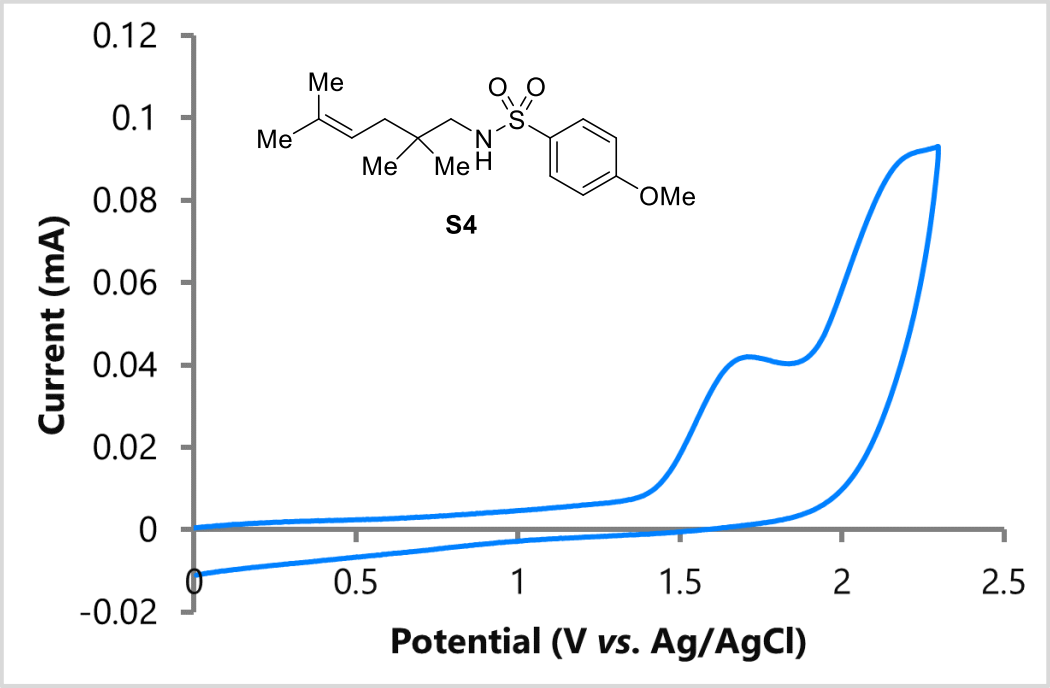


Figure S2. Cyclic voltammogram of alkene **S4**, E_P = 1.70 V.

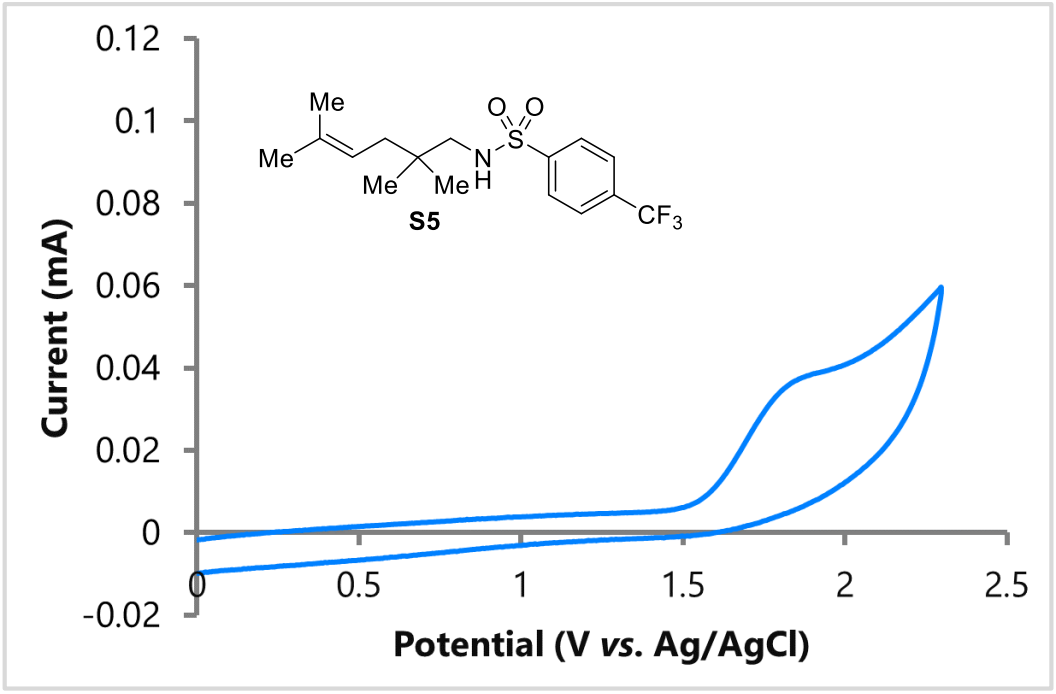


Figure S3. Cyclic voltammogram of alkene **S5**, E_P = 1.86 V.

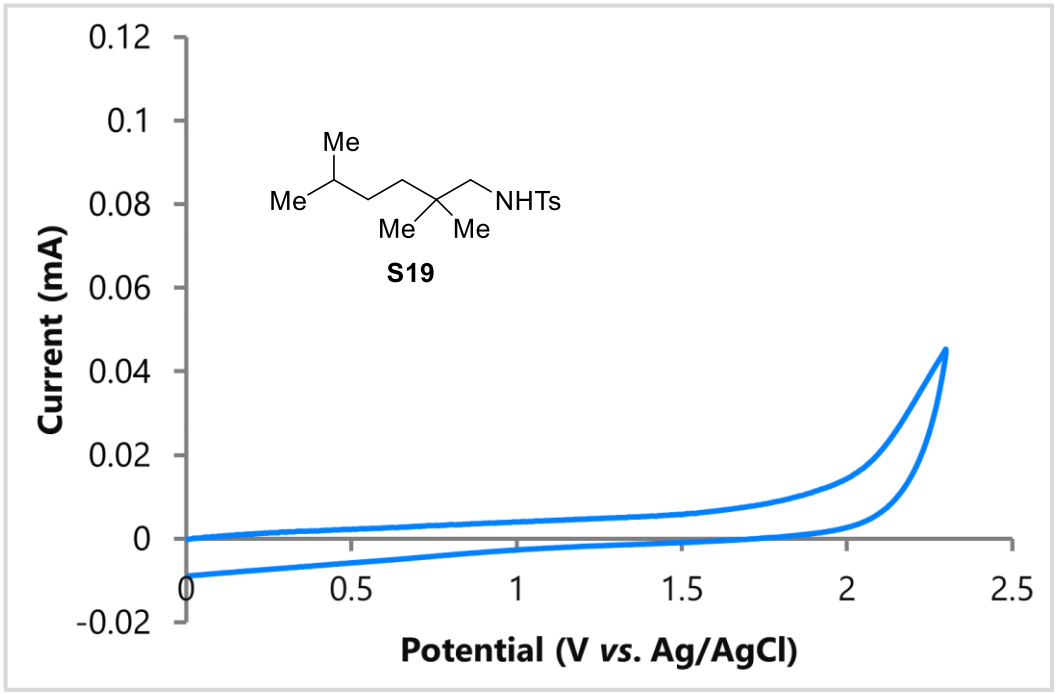
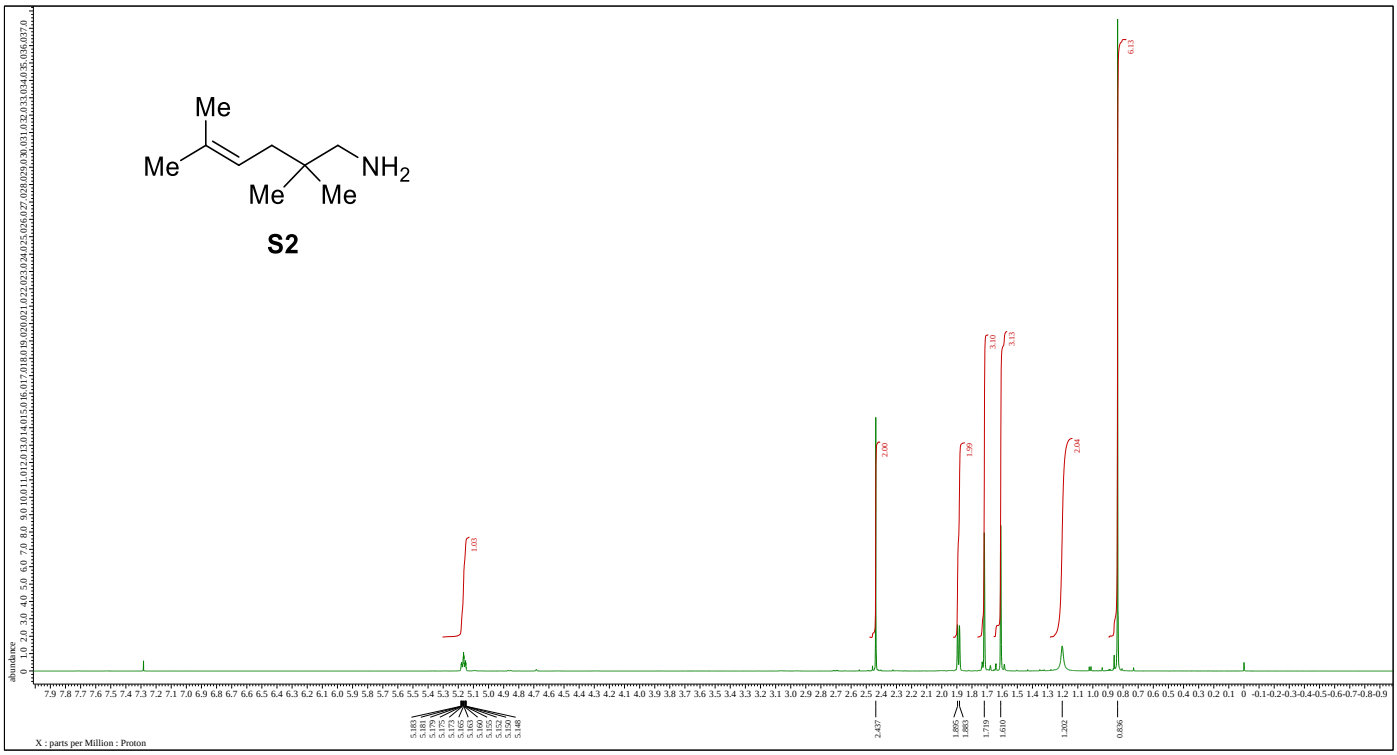
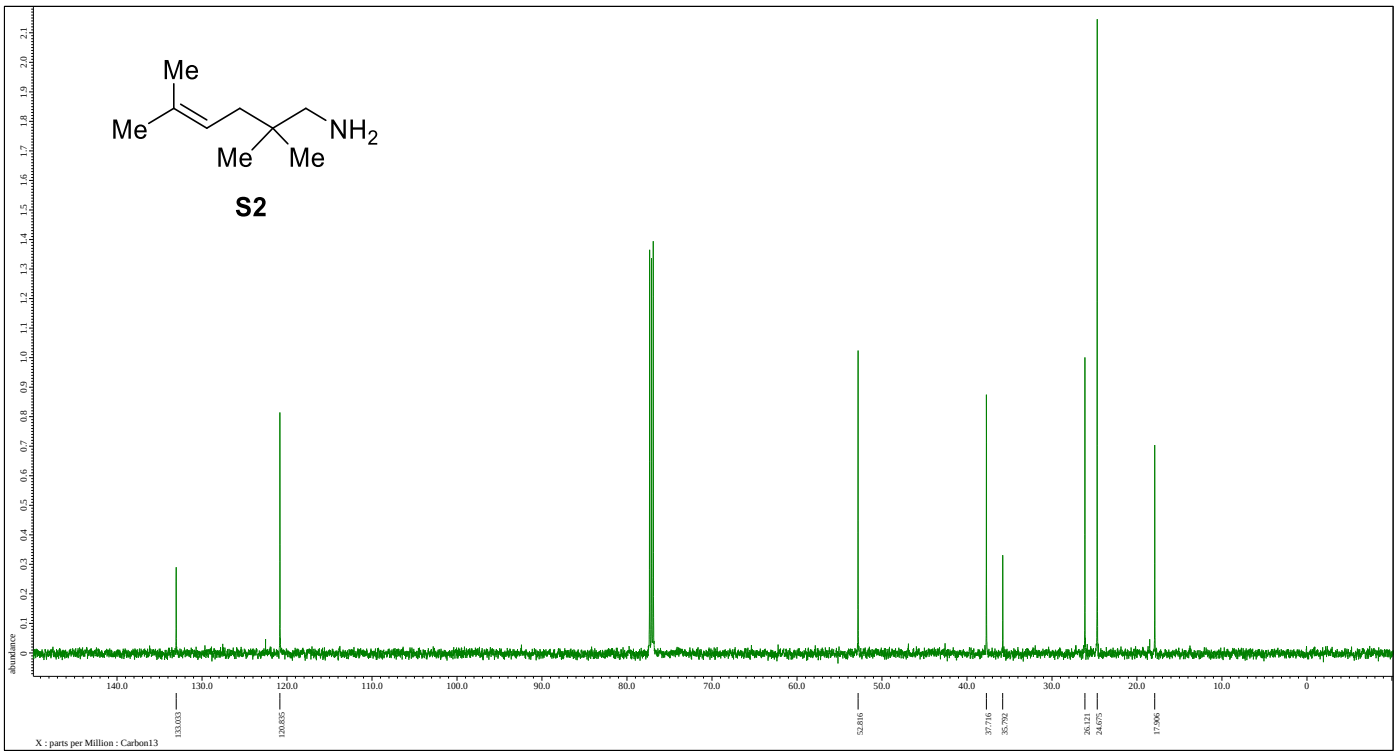
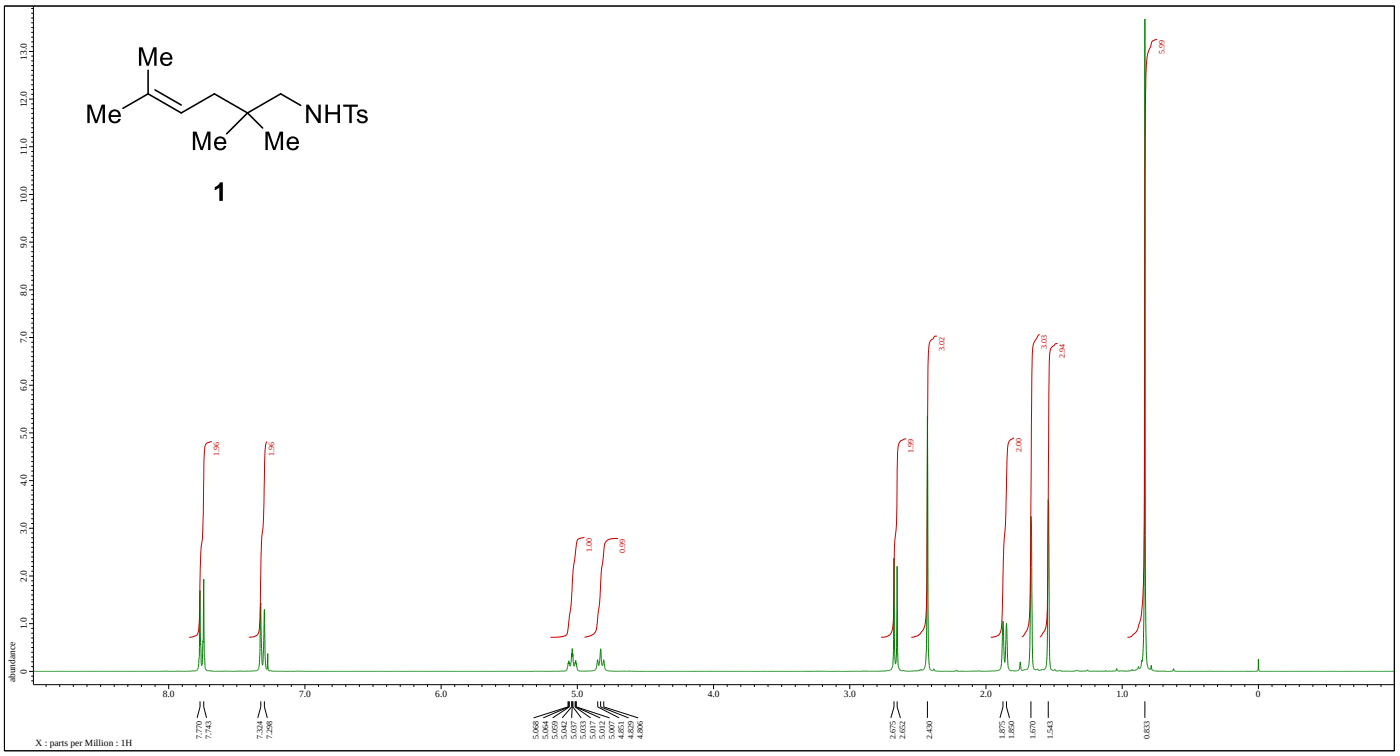


Figure S4. Cyclic voltammogram of the aryl sulfonamide without trisubstituted alkene **S19**, E_P > 2.3 V.

4. Copies of ^{1}H and ^{13}C NMR spectra



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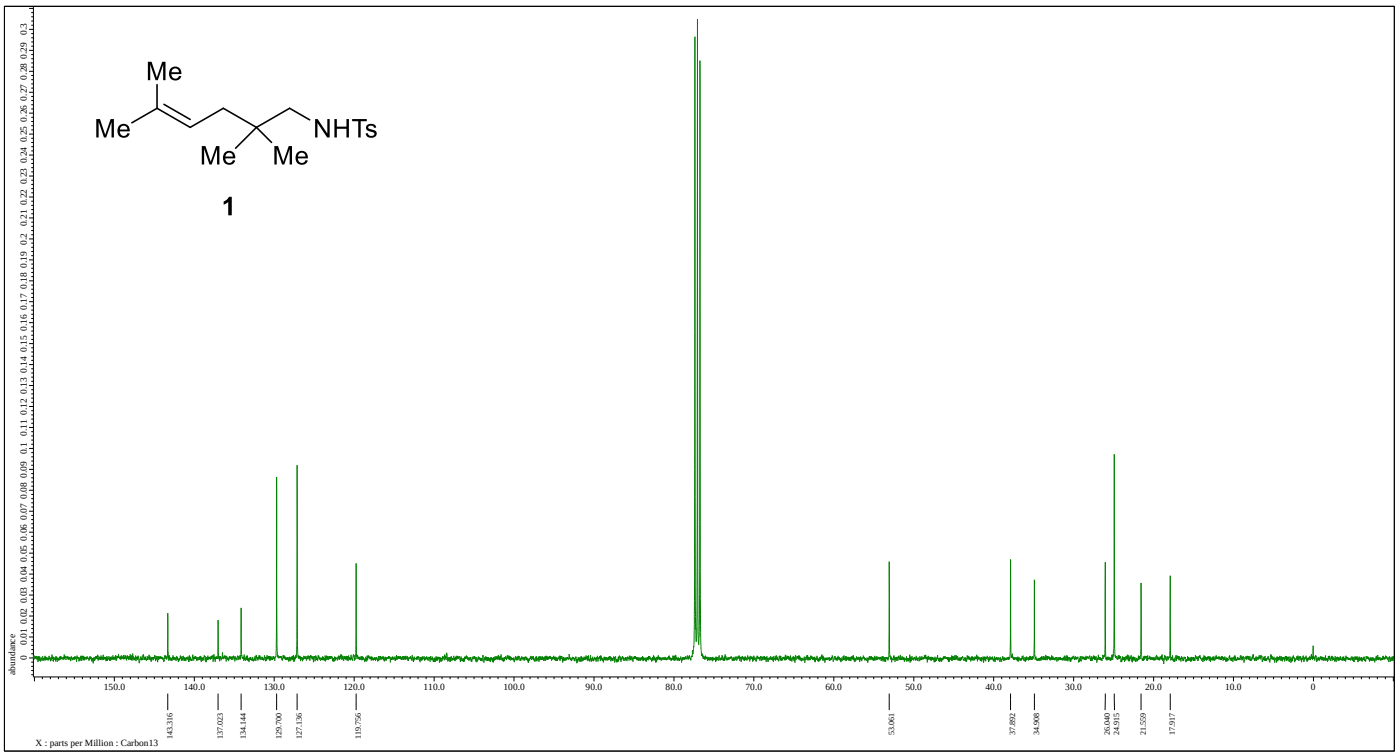
Me
NHTs
Me
Me
1
X : parts per Million : 1H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NHTs
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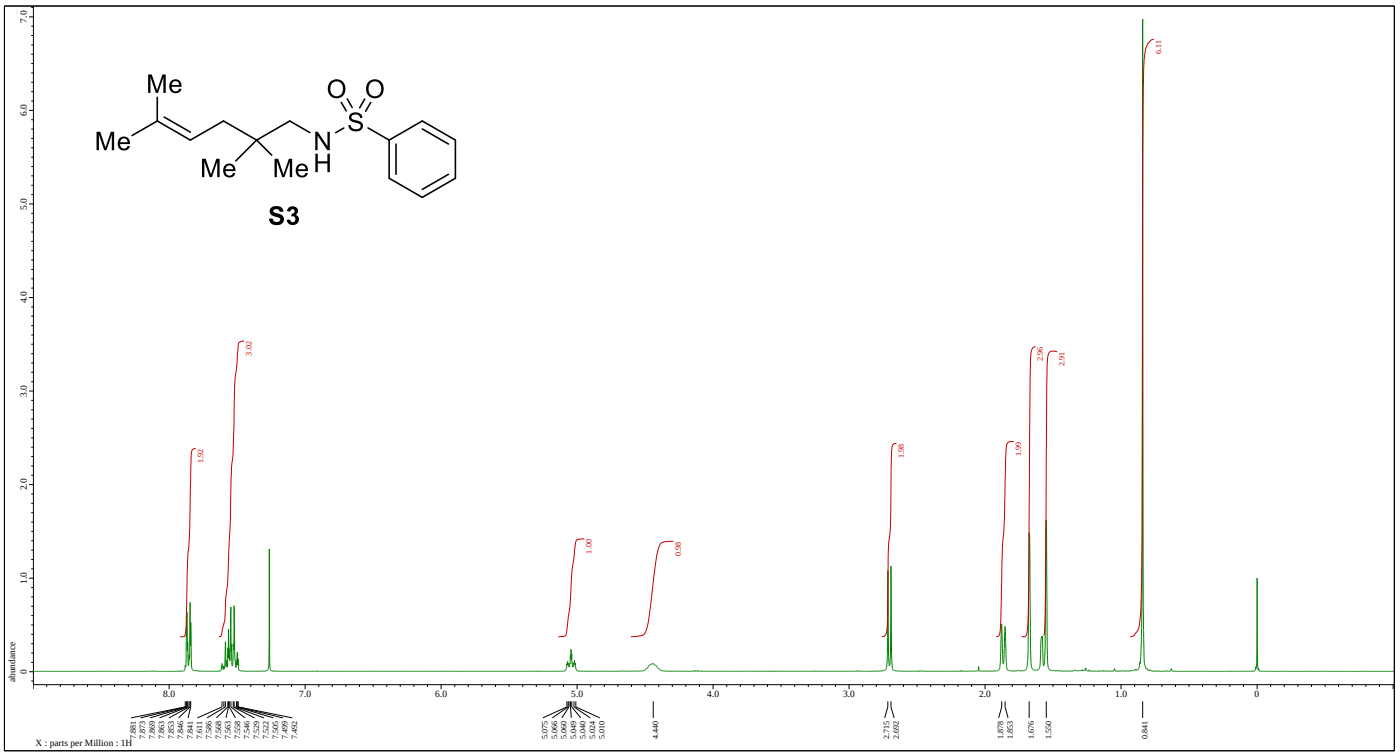
Me
1
X : parts per Million : Carbon13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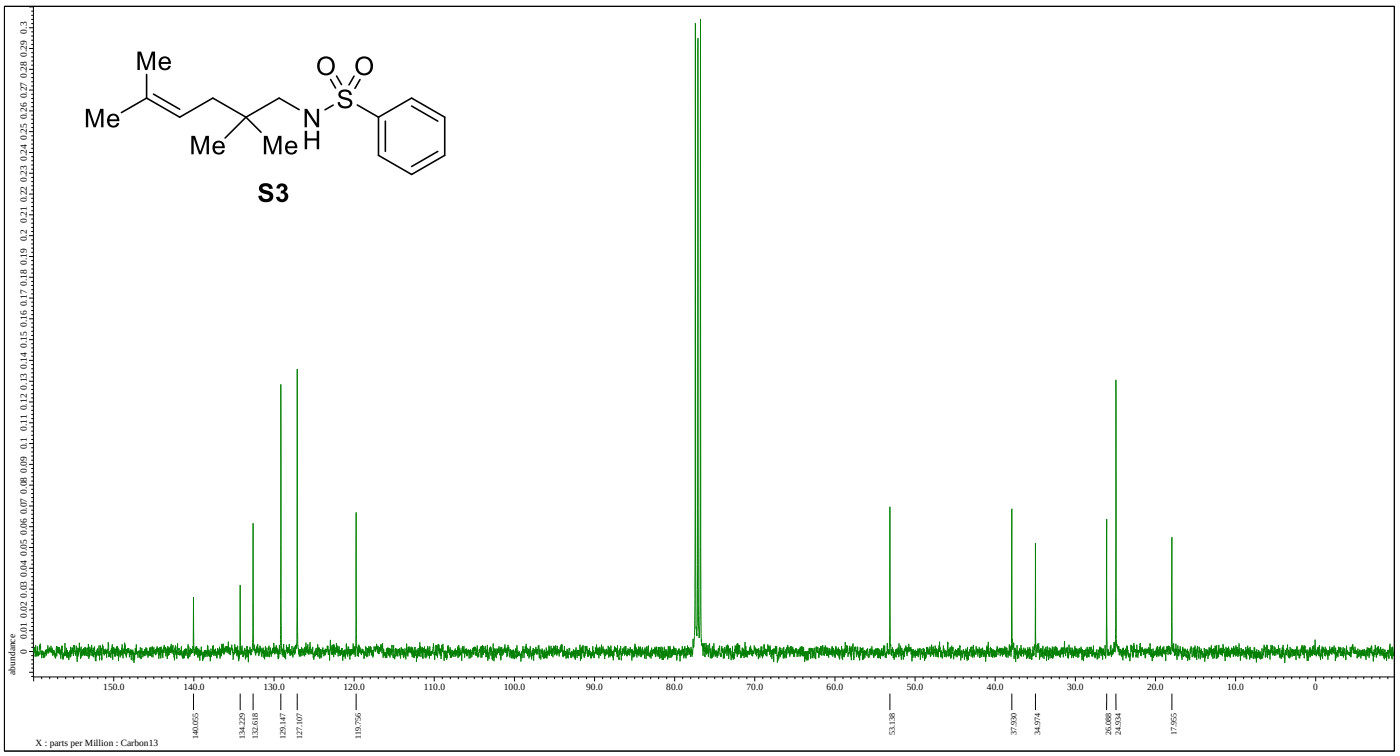
Me
Me
Me
N
H
O
O
S
S3
1.92
3.02
1.00
0.98
1.98
1.99
2.98
2.91
6.11
X : parts per Million : 1H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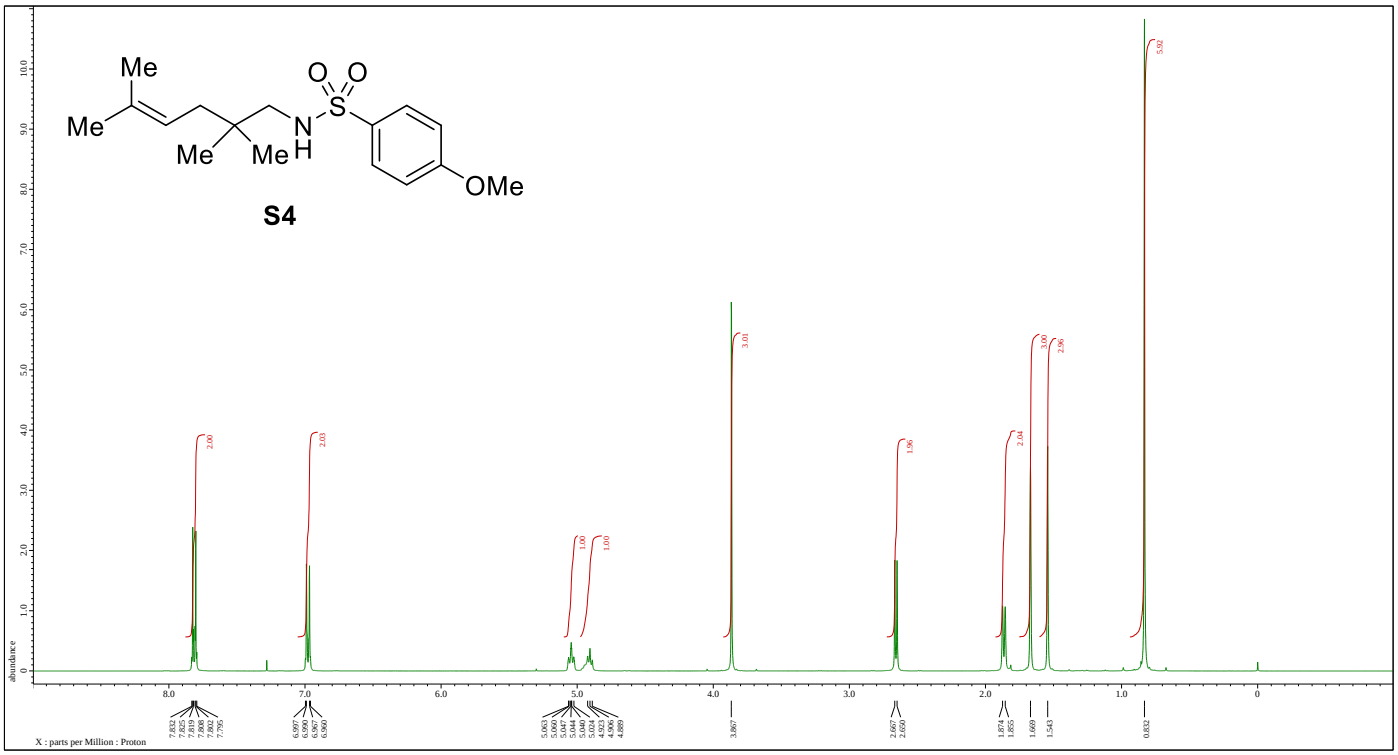
Me
Me
Me
N
H
O
O
S
S3
X : parts per Million : Carbon13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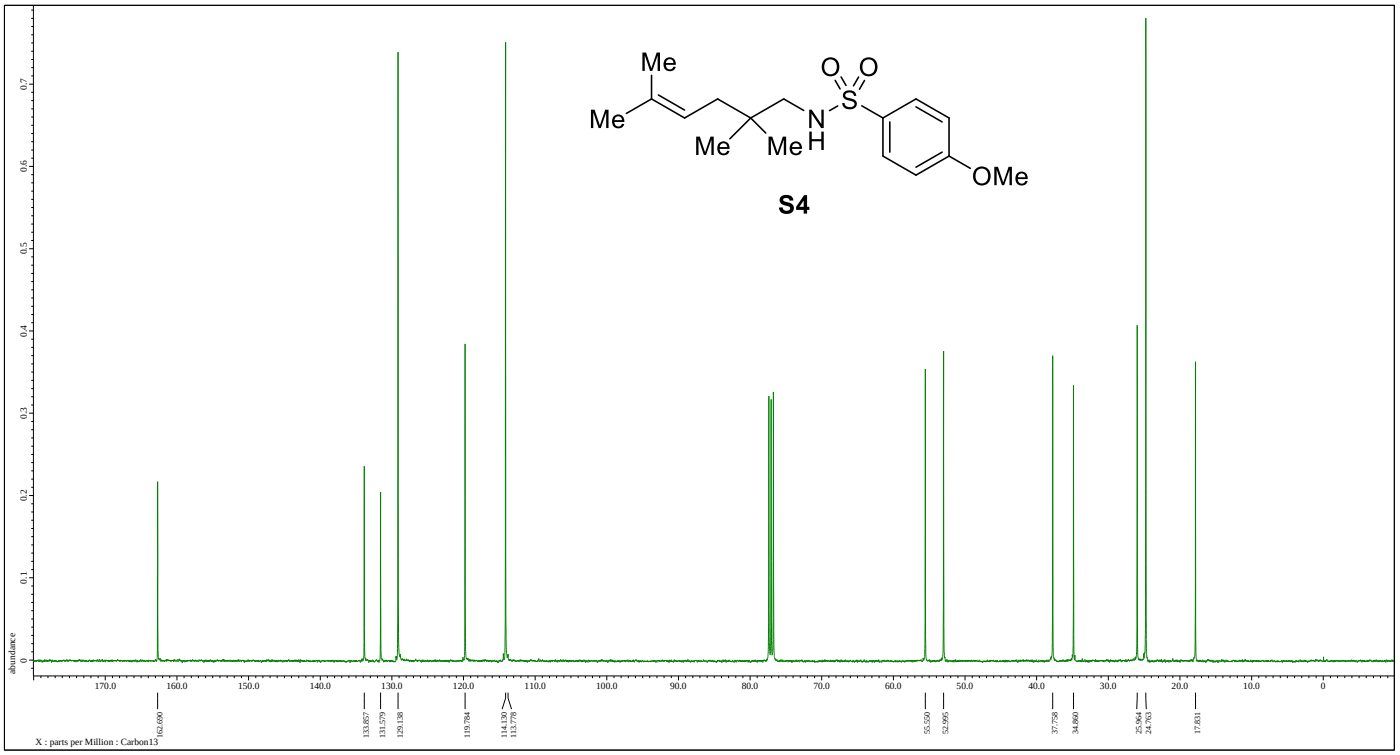
Me
Me
Me
O
O
S
N
H
OMe
S4
X : parts per Million : Proton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Me
Me
O
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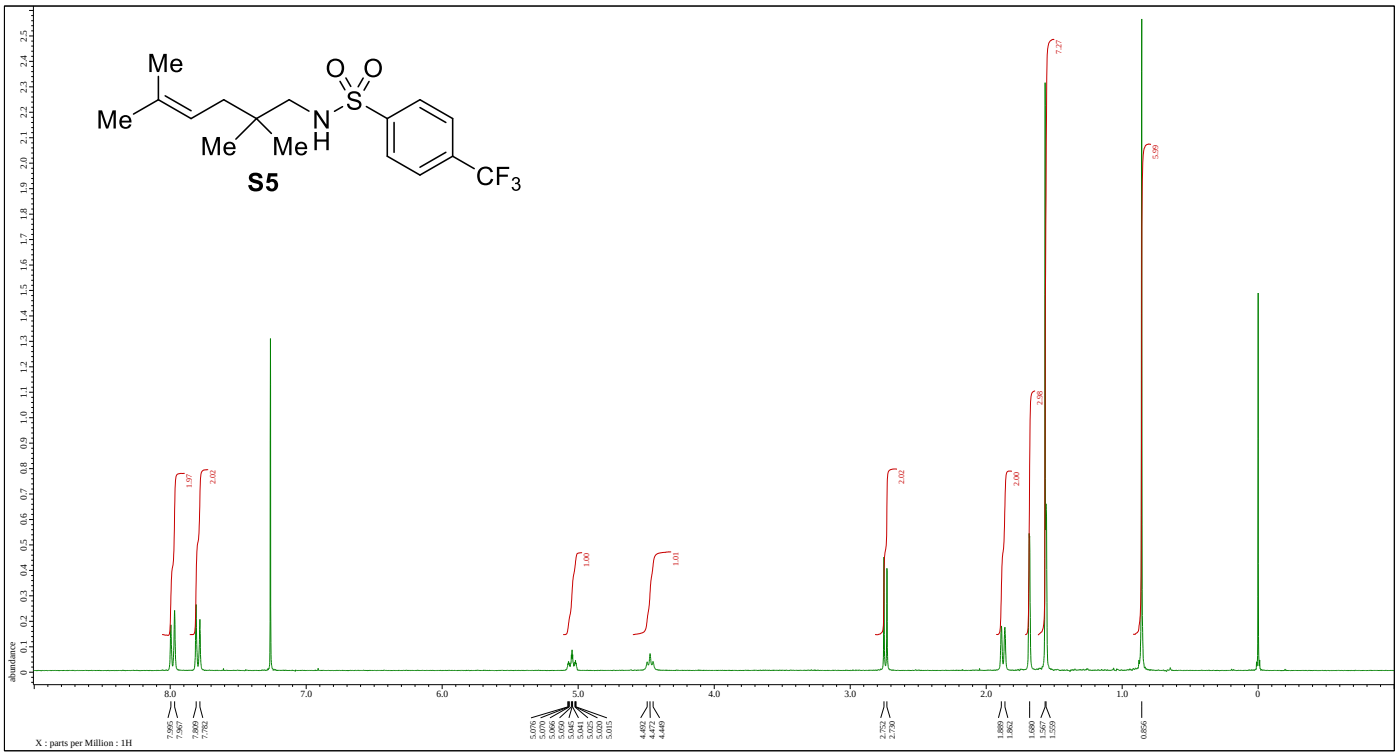
H
OMe
S4
X : parts per Million : Carbon13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CF3
S5
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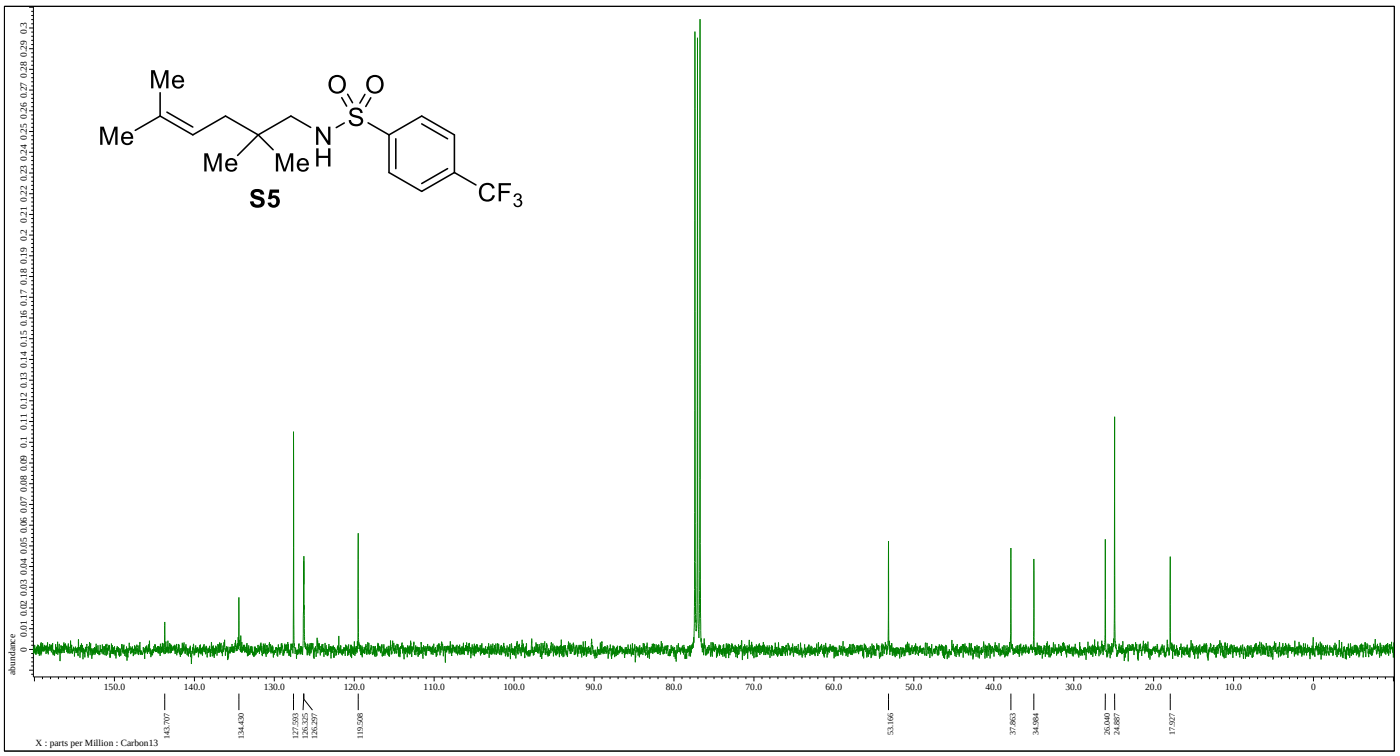
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Me
Me
N
H
O
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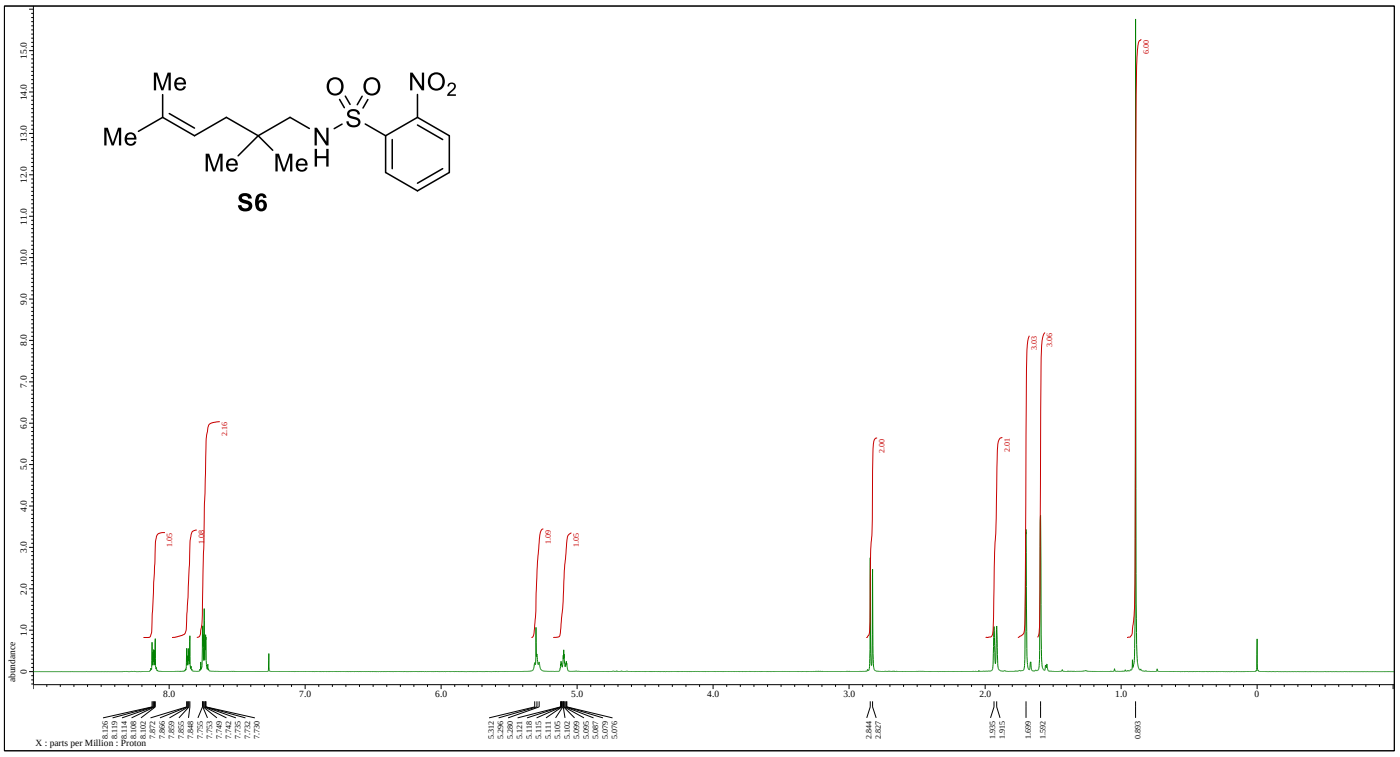
S
CF3
S5
X : parts per Million : Carbon13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S6
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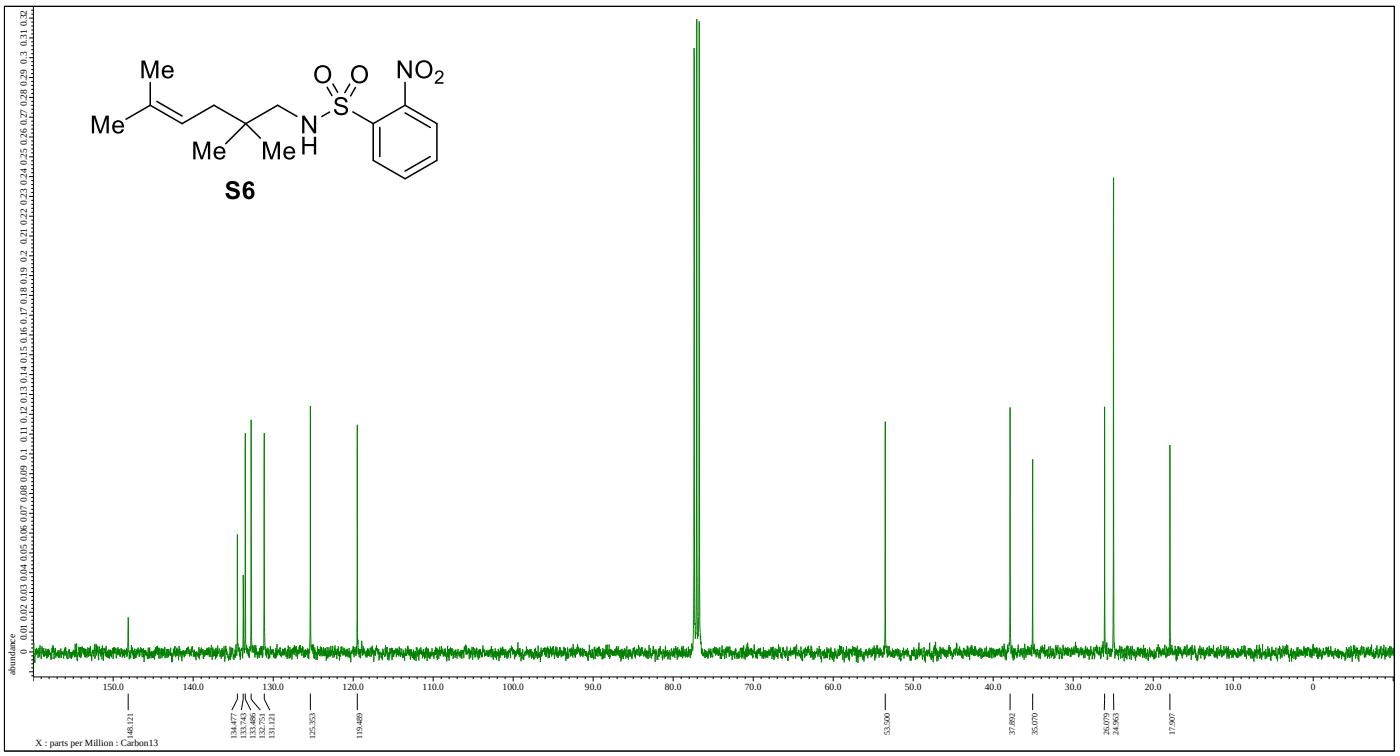
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H
O
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S
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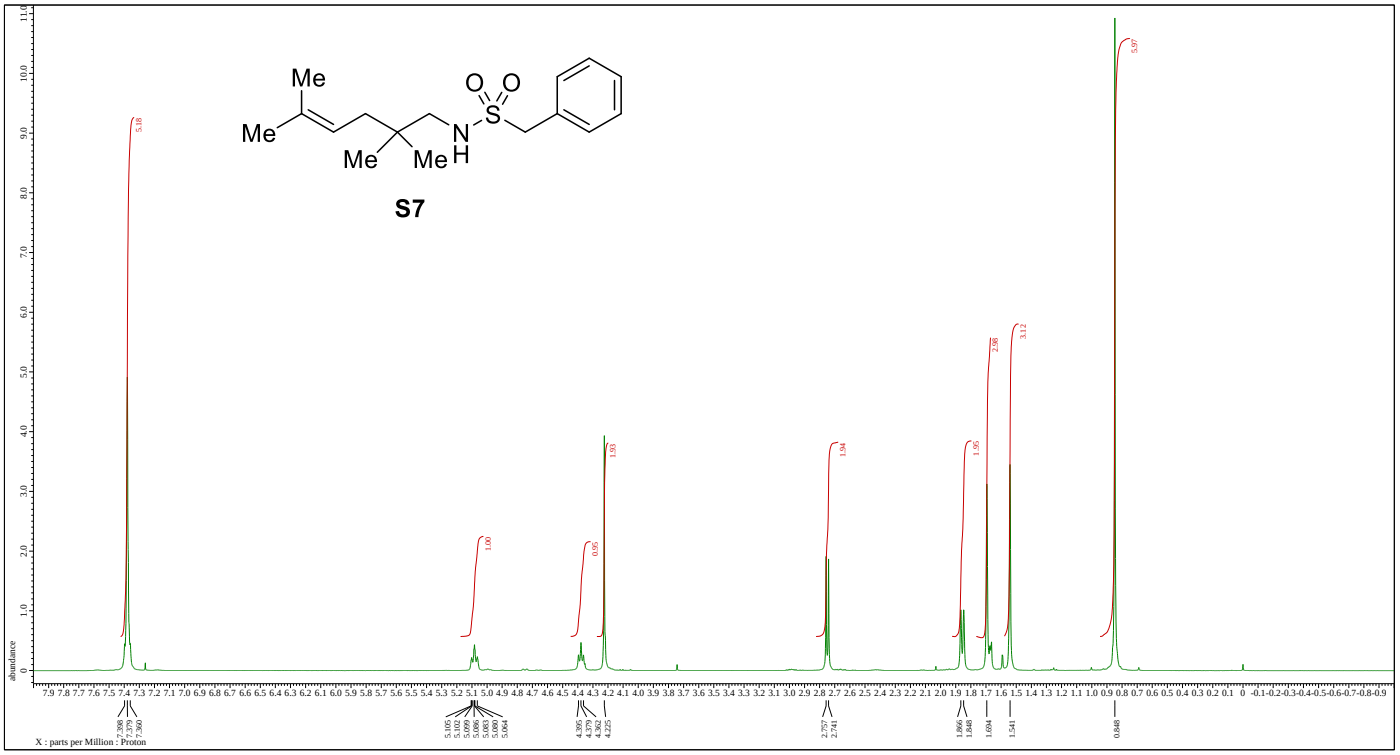
NO2
S6
X : parts per Million : Carbon13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Me
Me
O
O
S
N
H
S7
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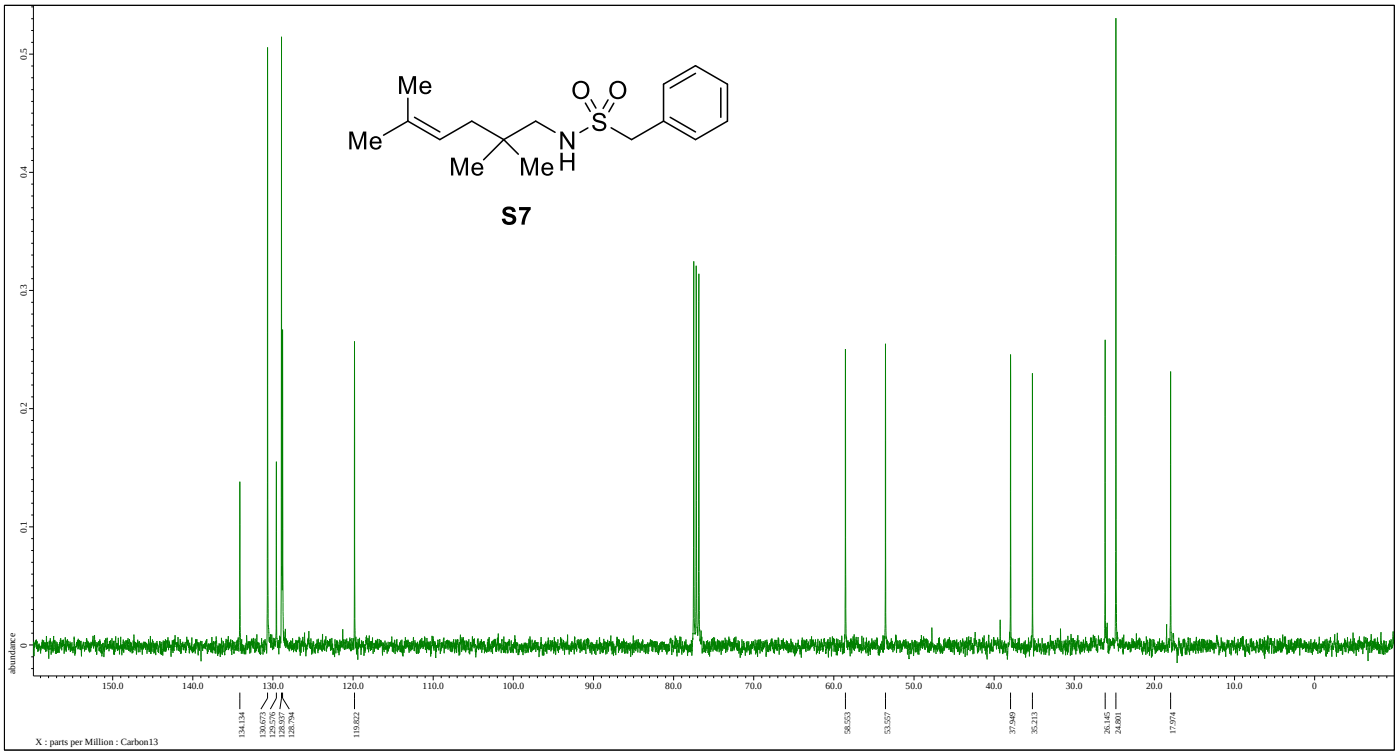
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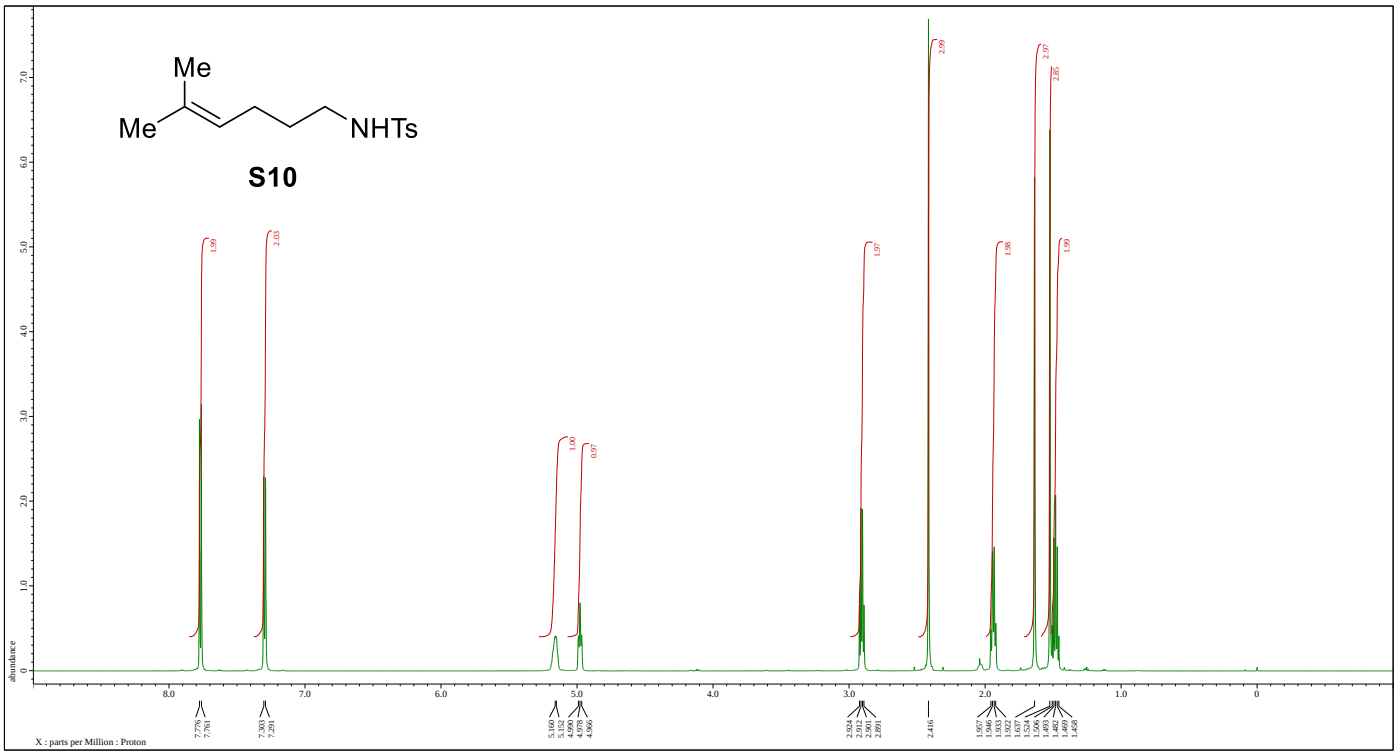
Me
Me
Me
O
O
S
N
H
S7
X : parts per Million : Carbon13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NHTs
S10
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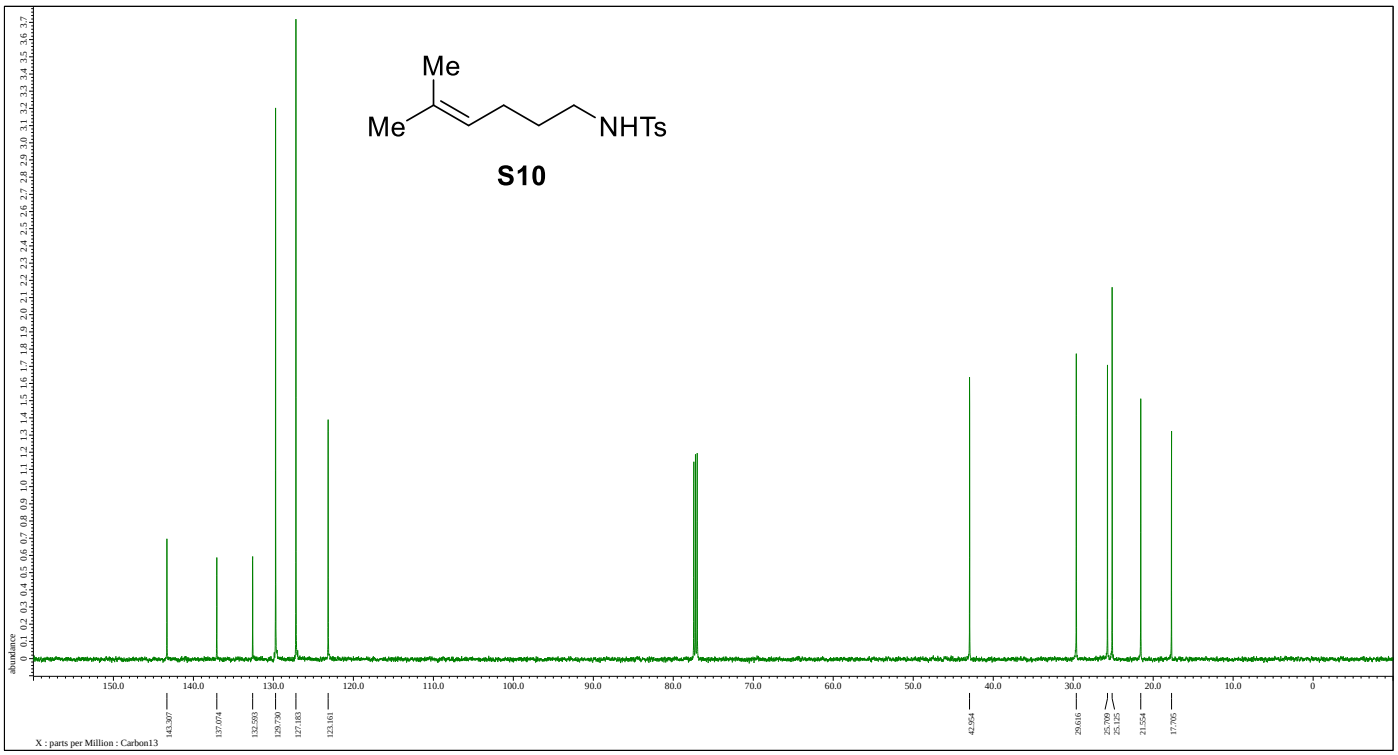
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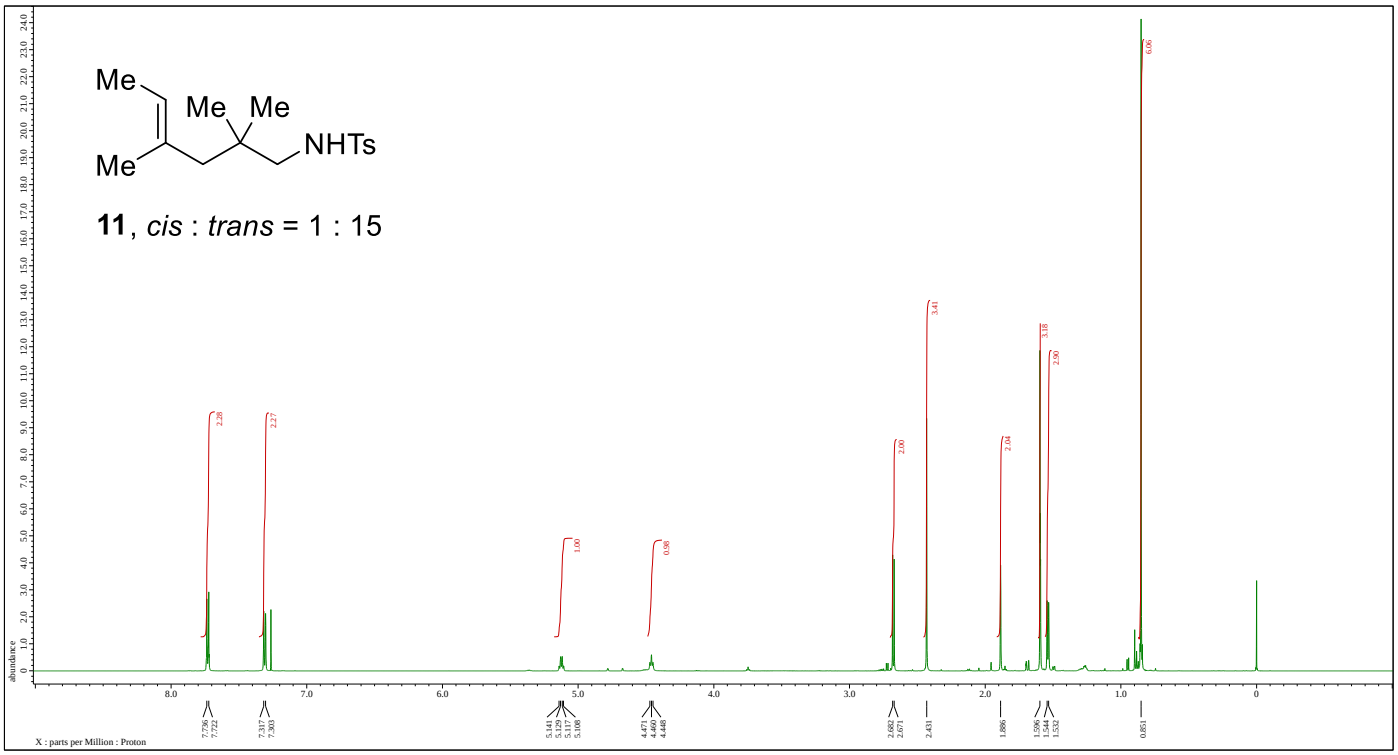
Me
NHTs
S10
X : parts per Million : Carbon13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Me
NHTs
Me
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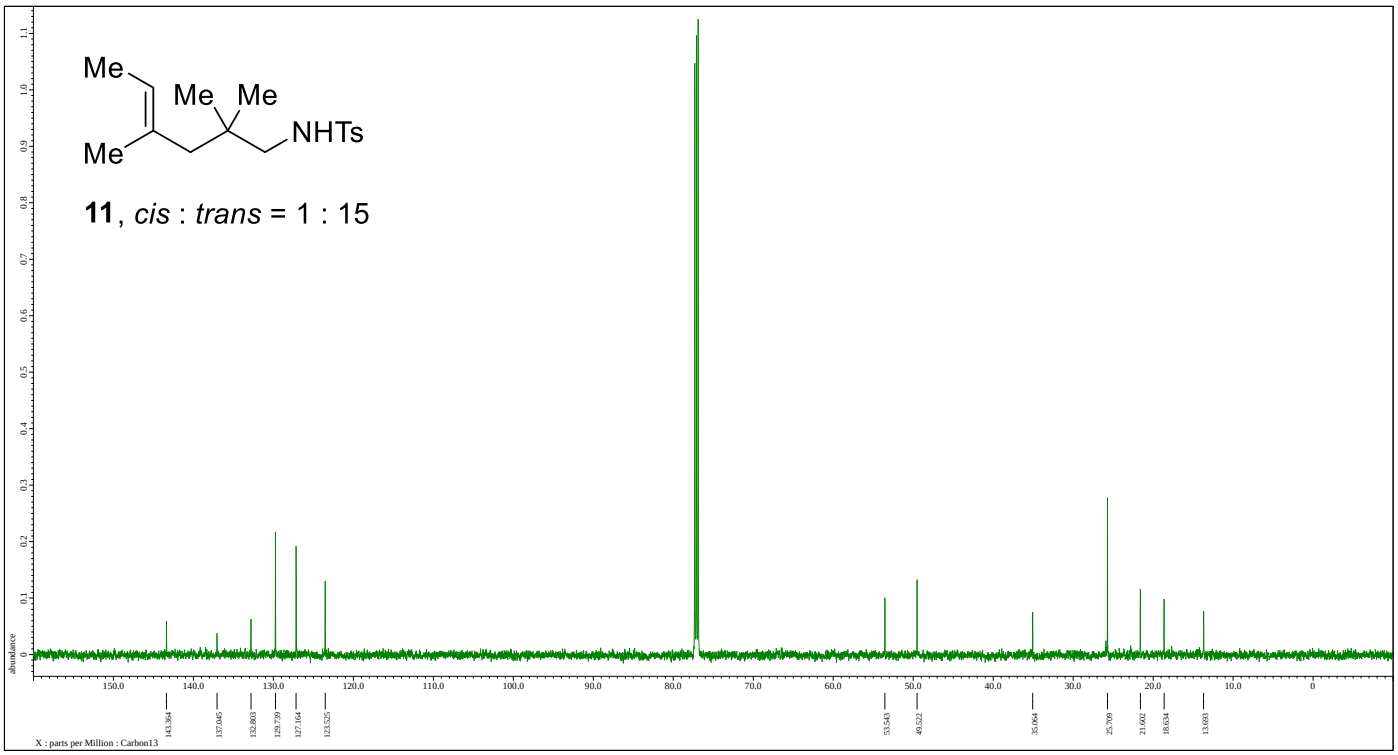
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NHTs
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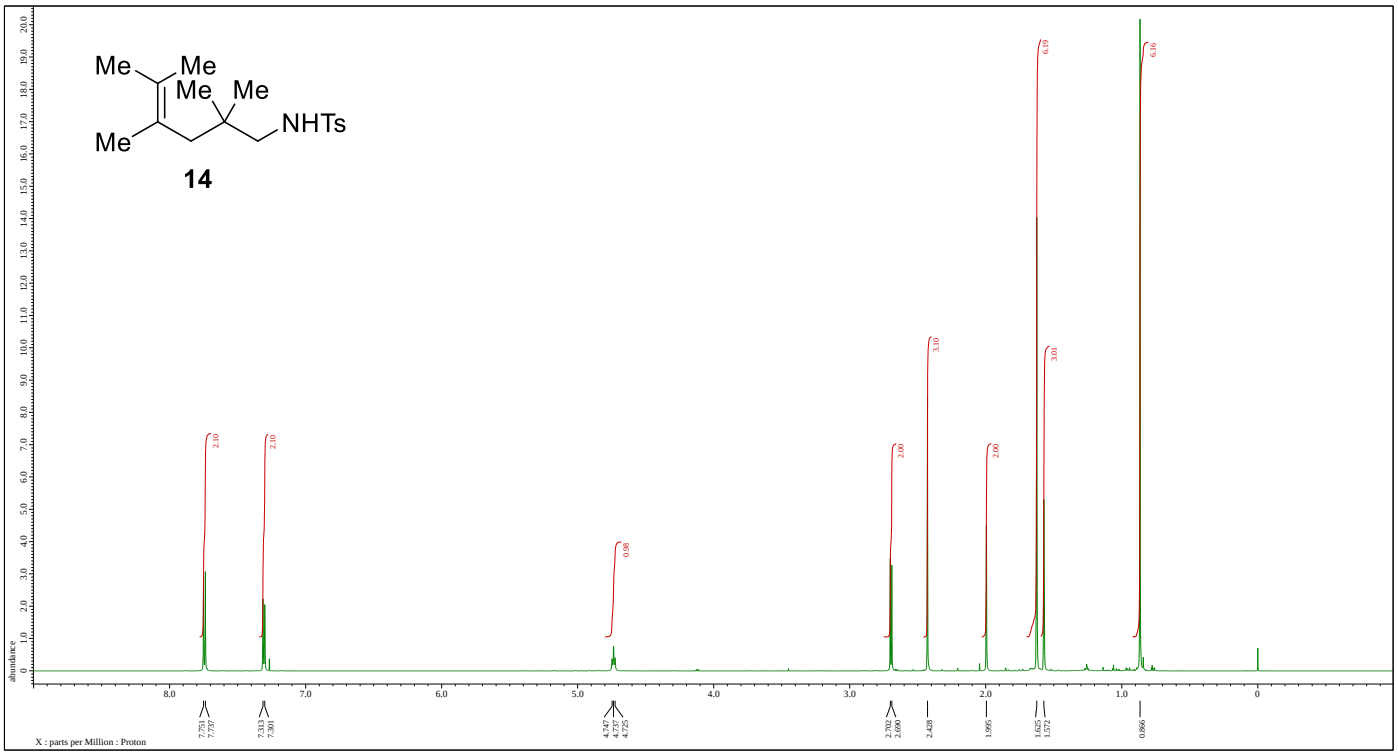
11, cis : trans = 1 : 15
X : parts per Million : Carbon13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Me
Me
NHTs
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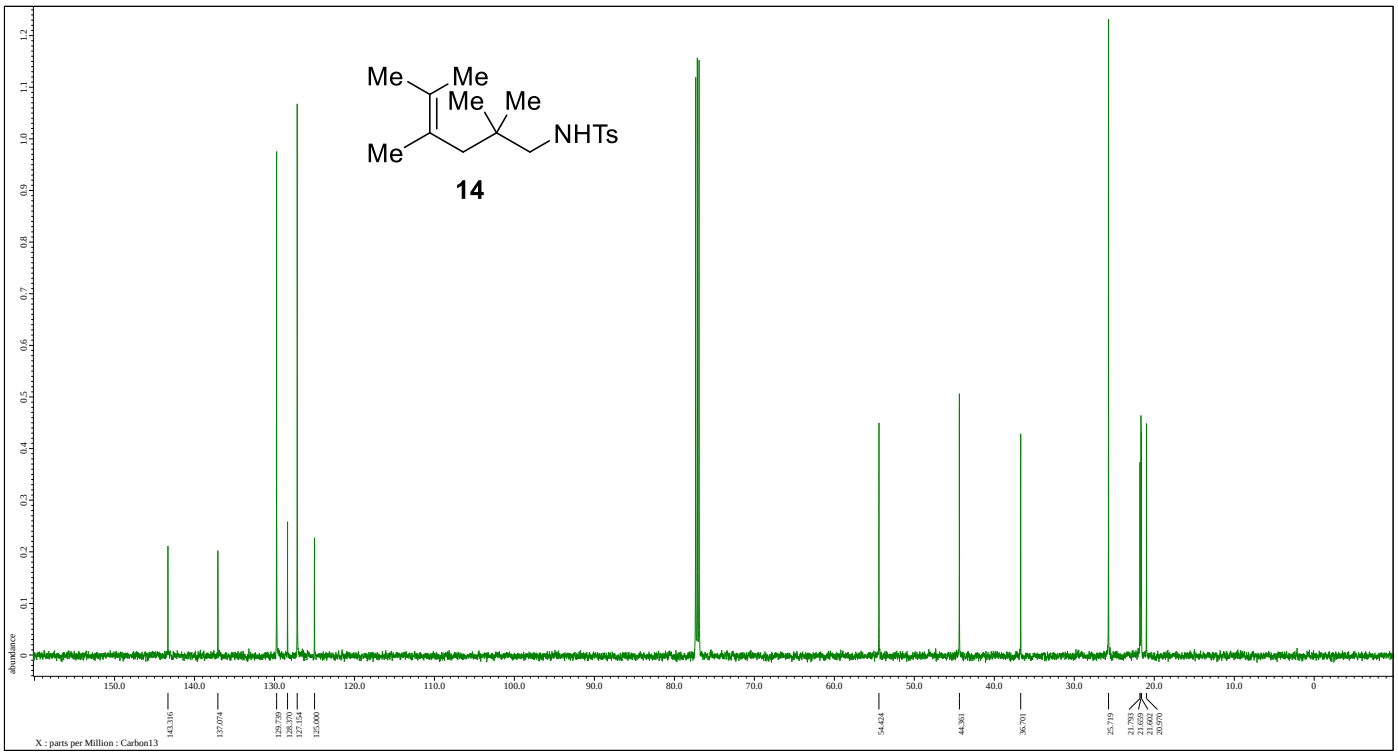
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Me
Me
NHTs
14
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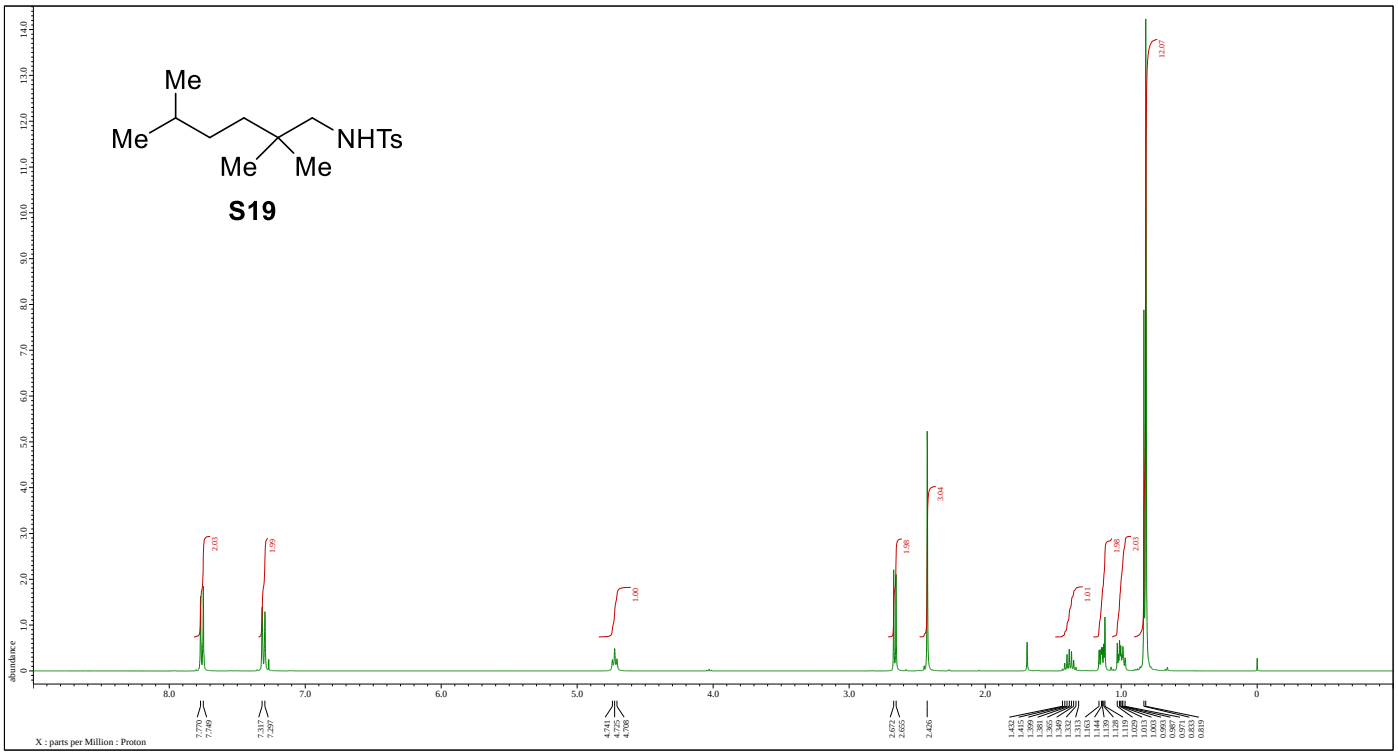
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NHTs
Me
Me
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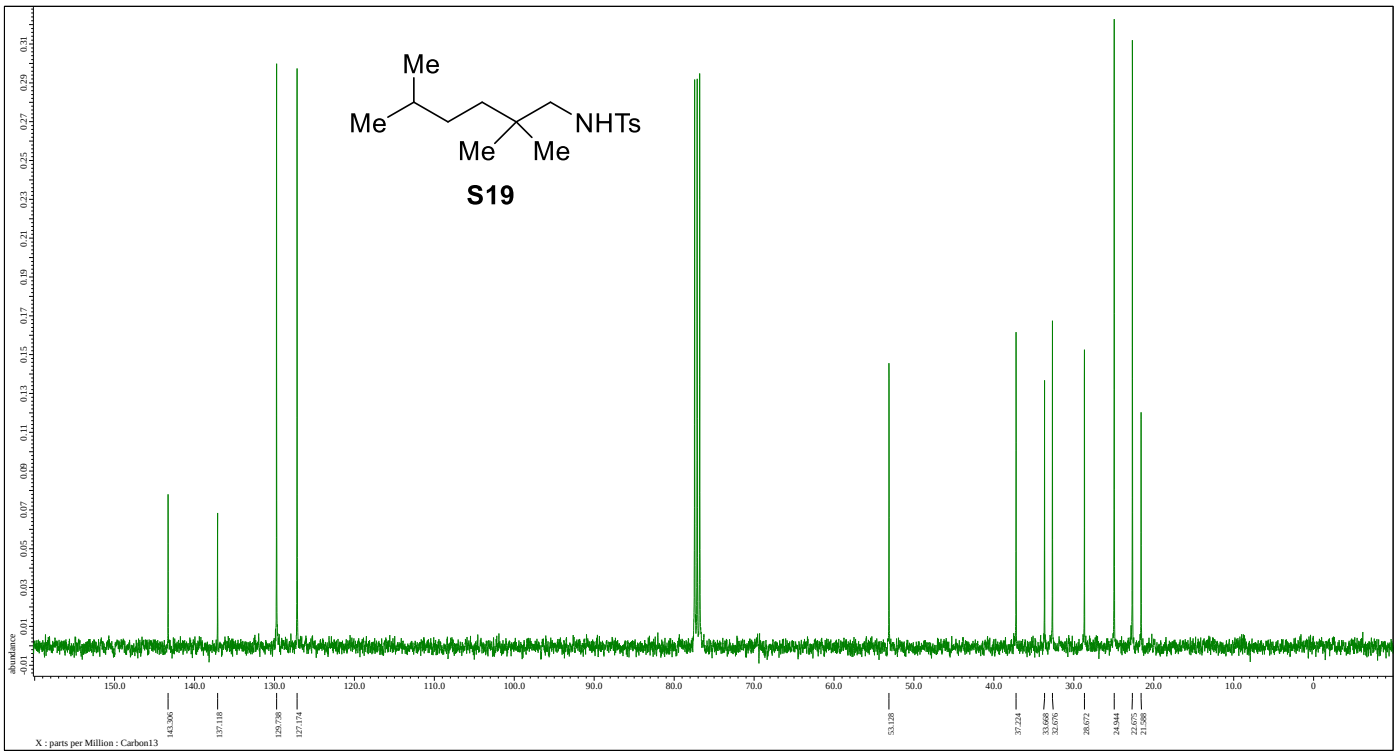
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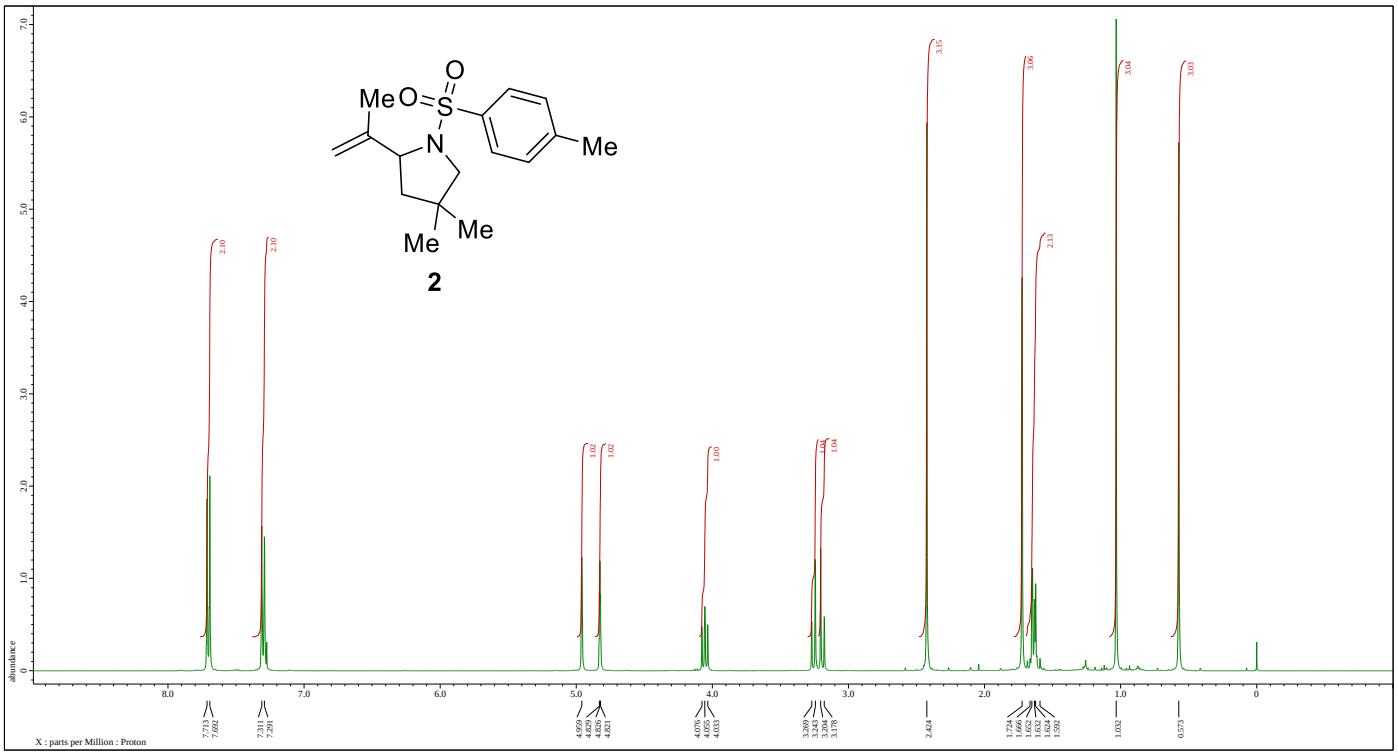
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abundance
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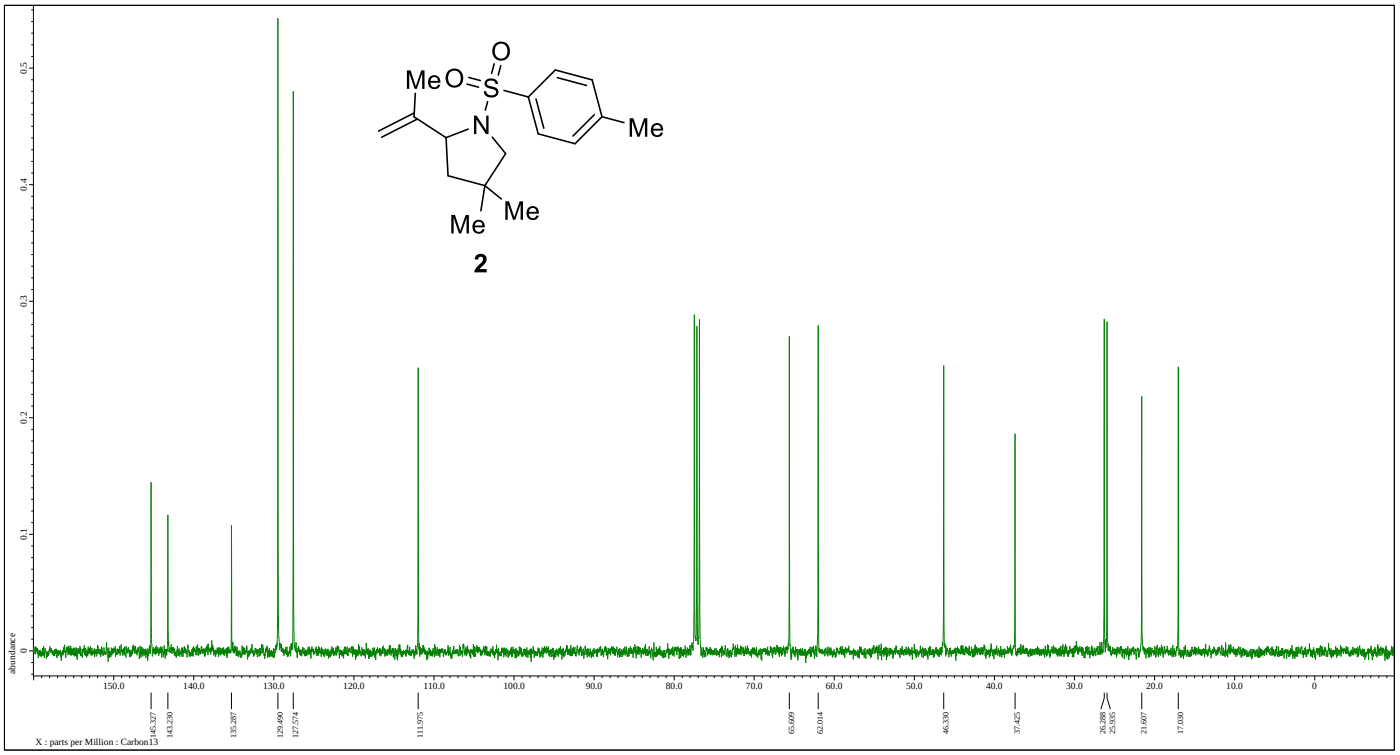
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abundance
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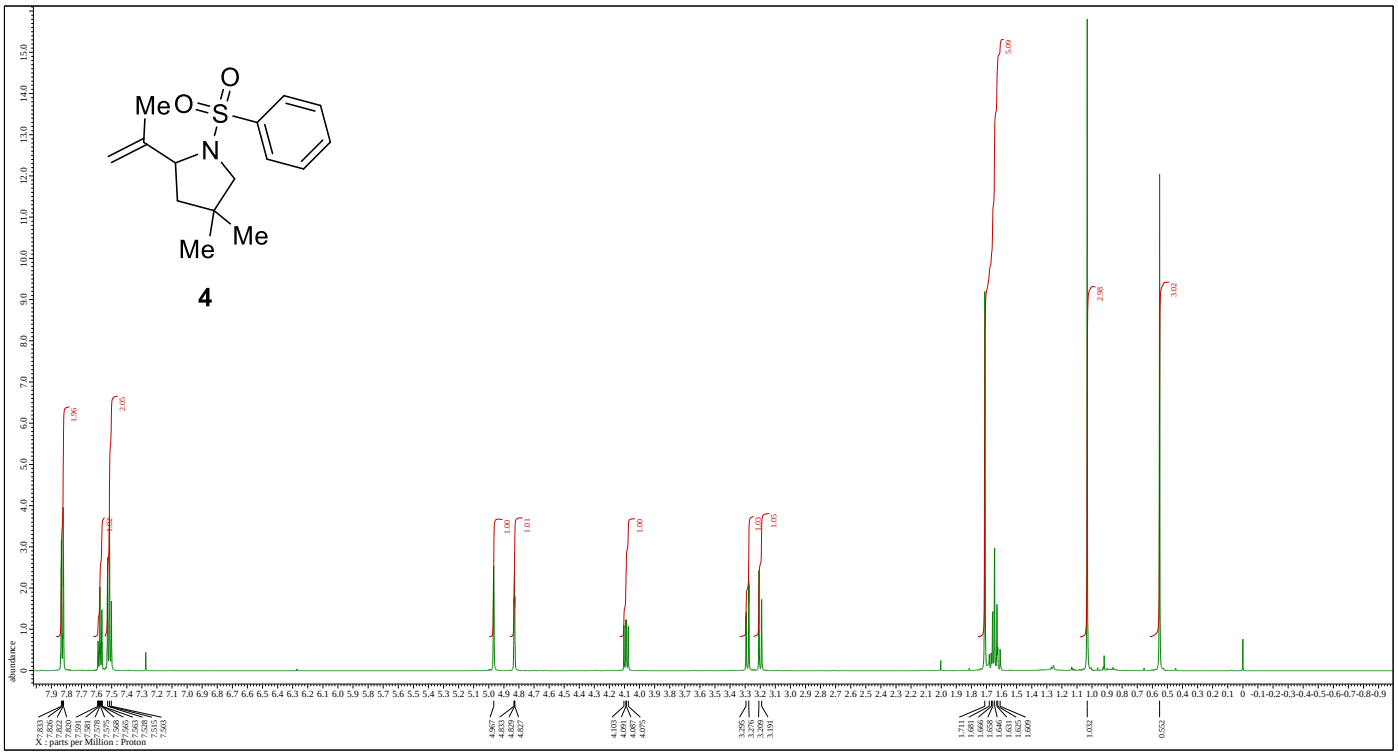
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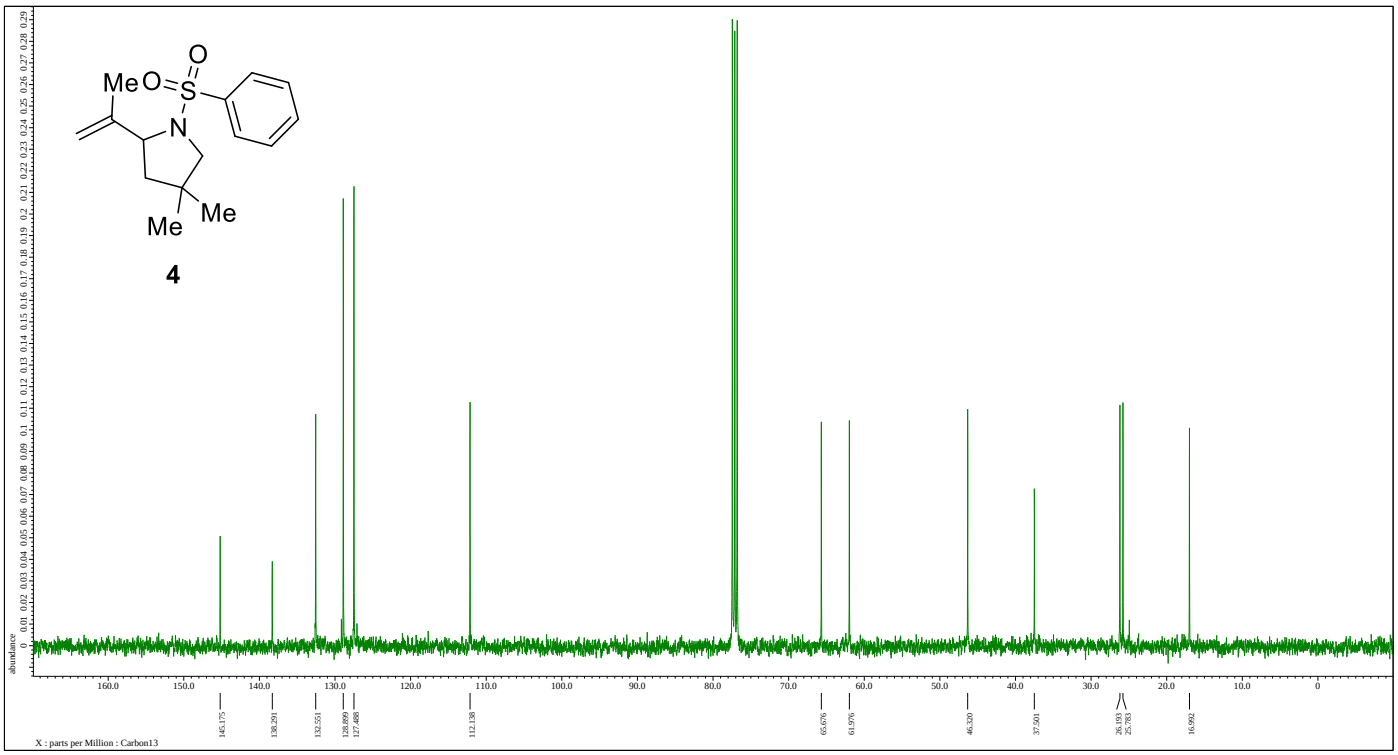
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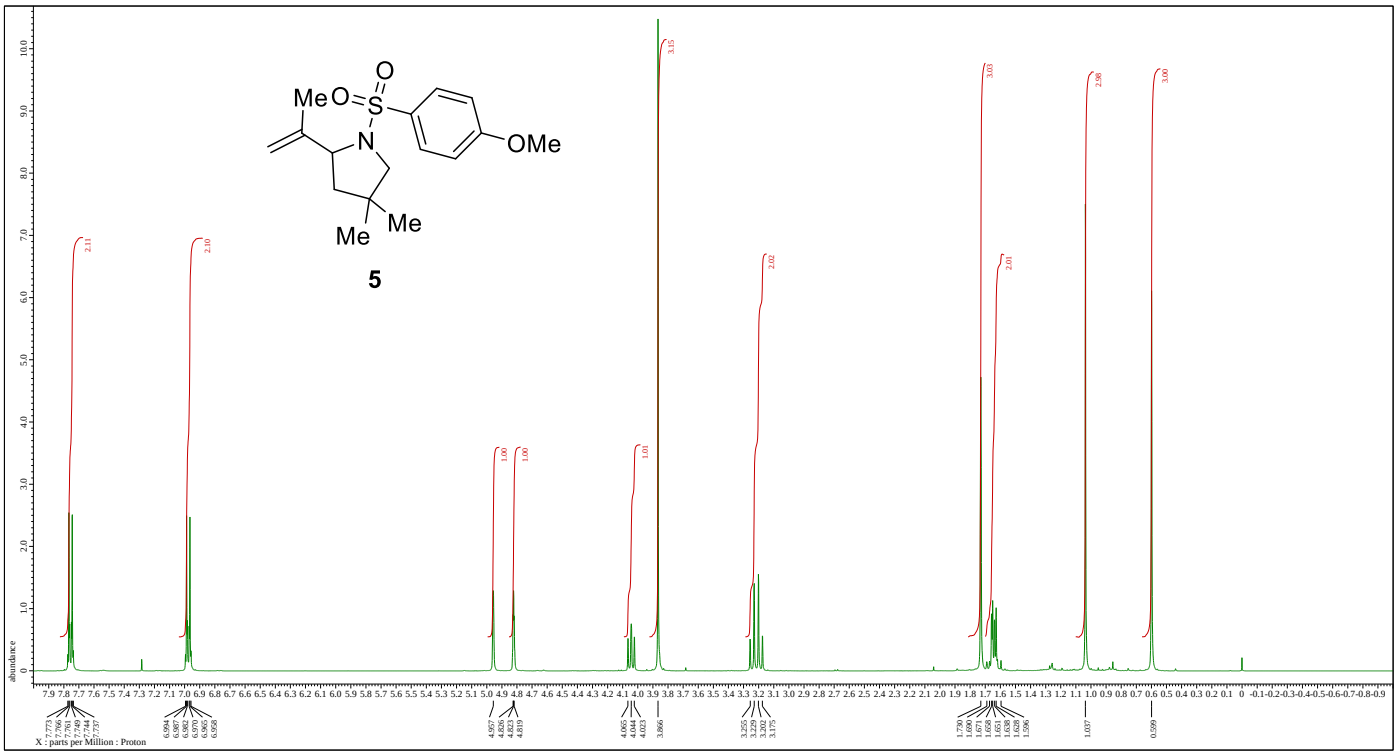
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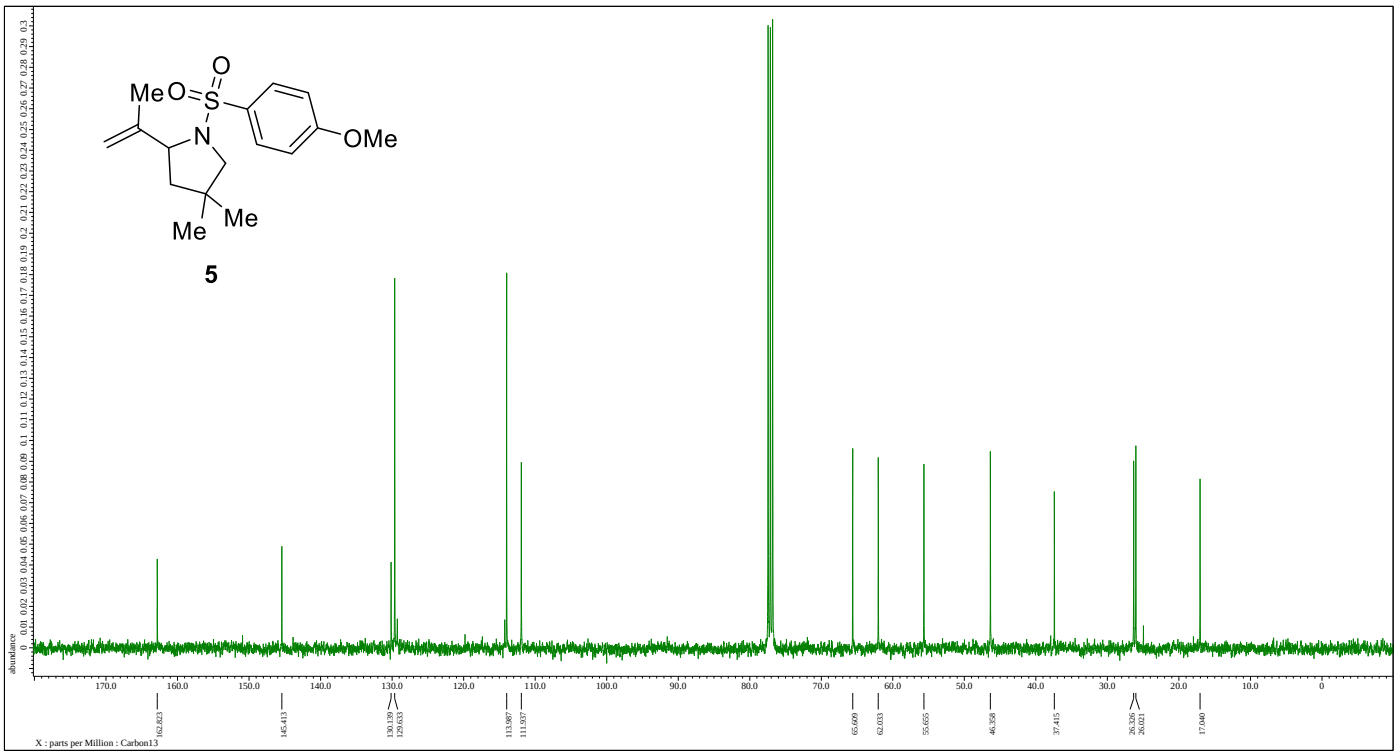
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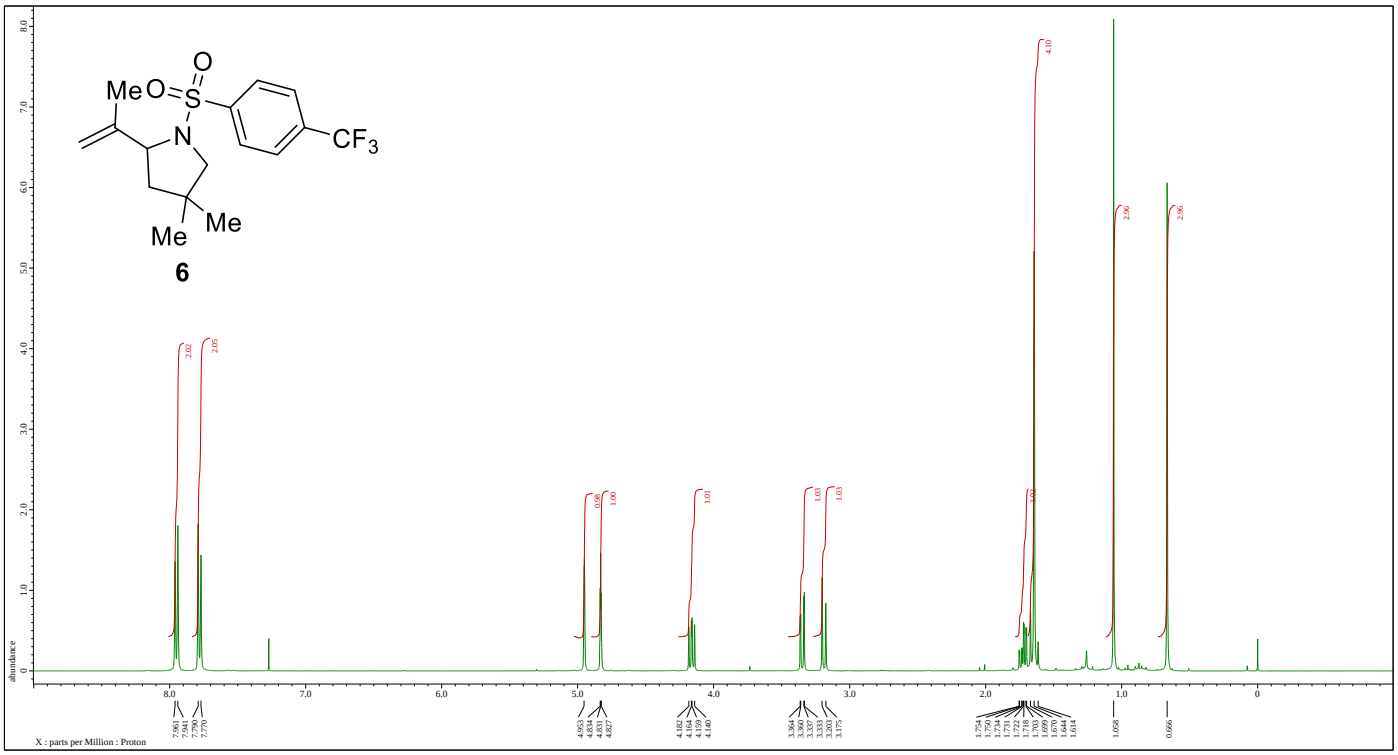
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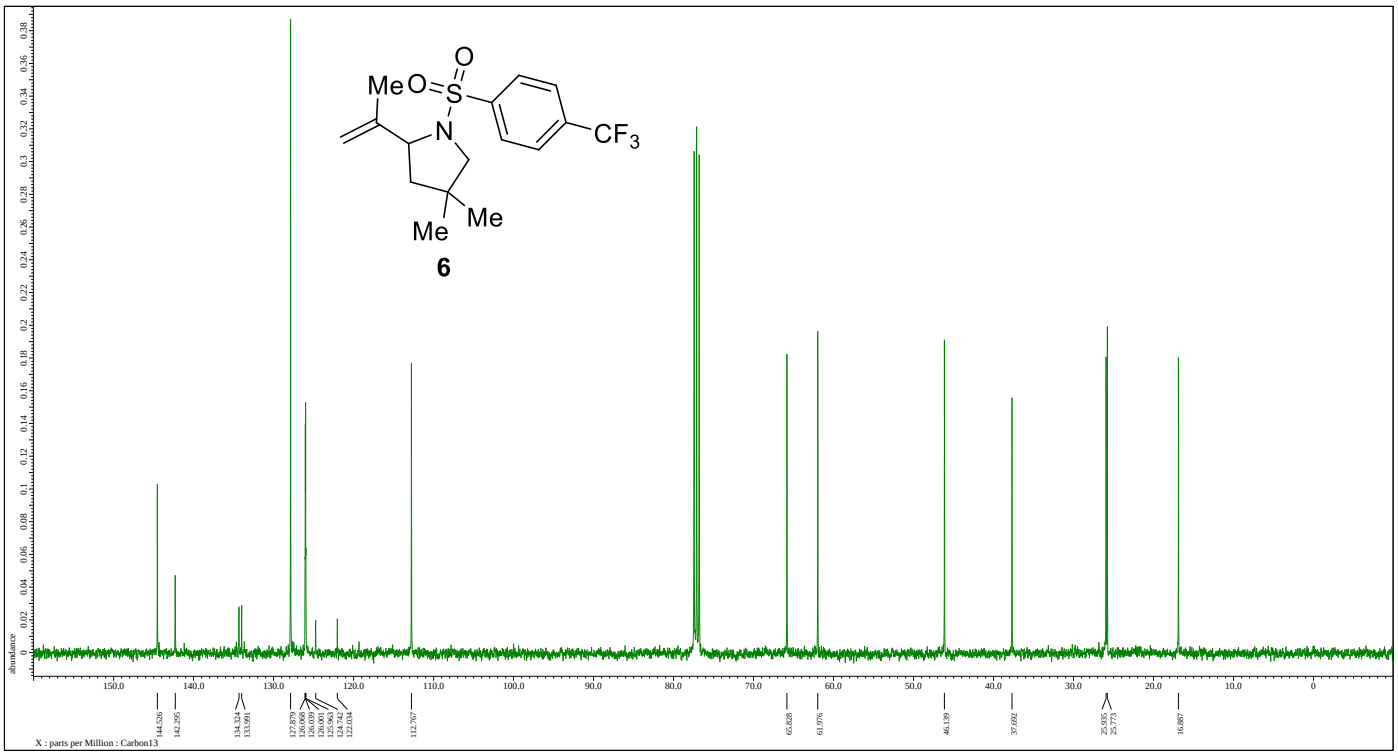
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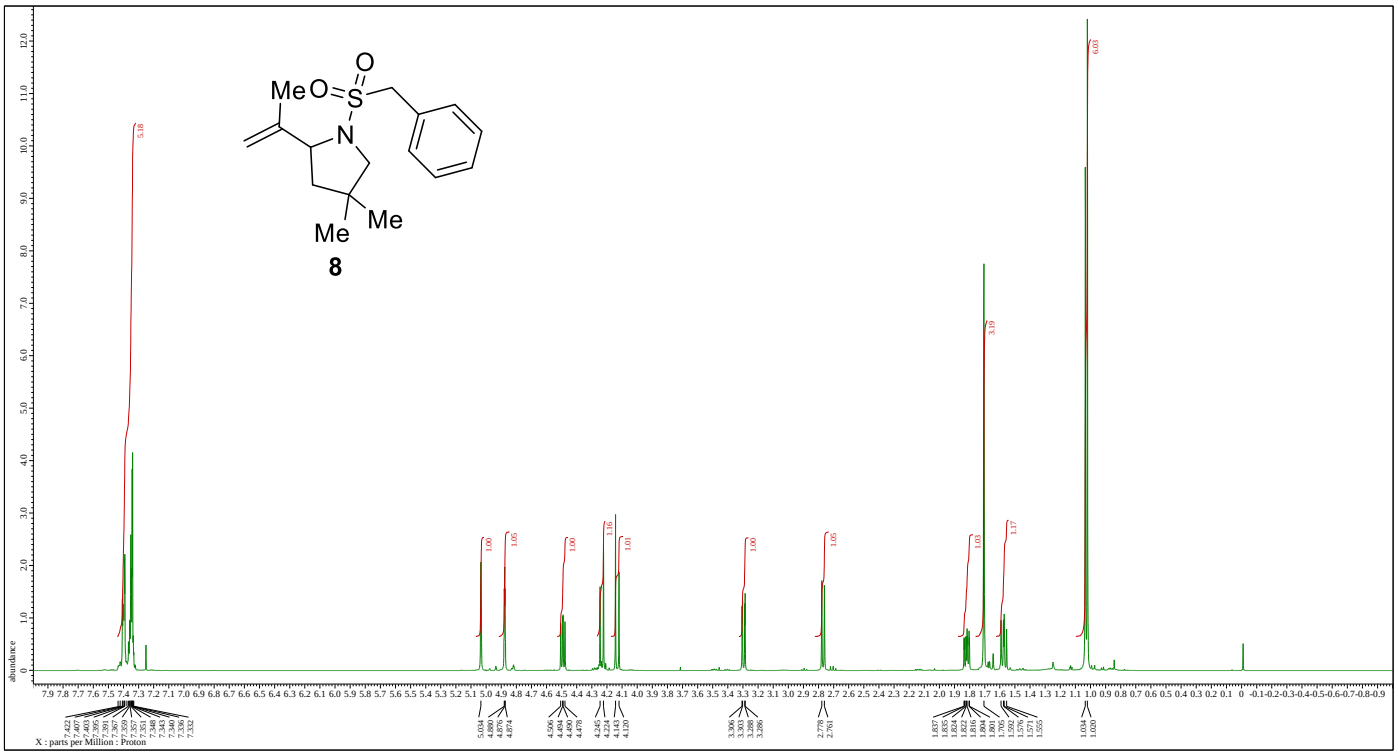
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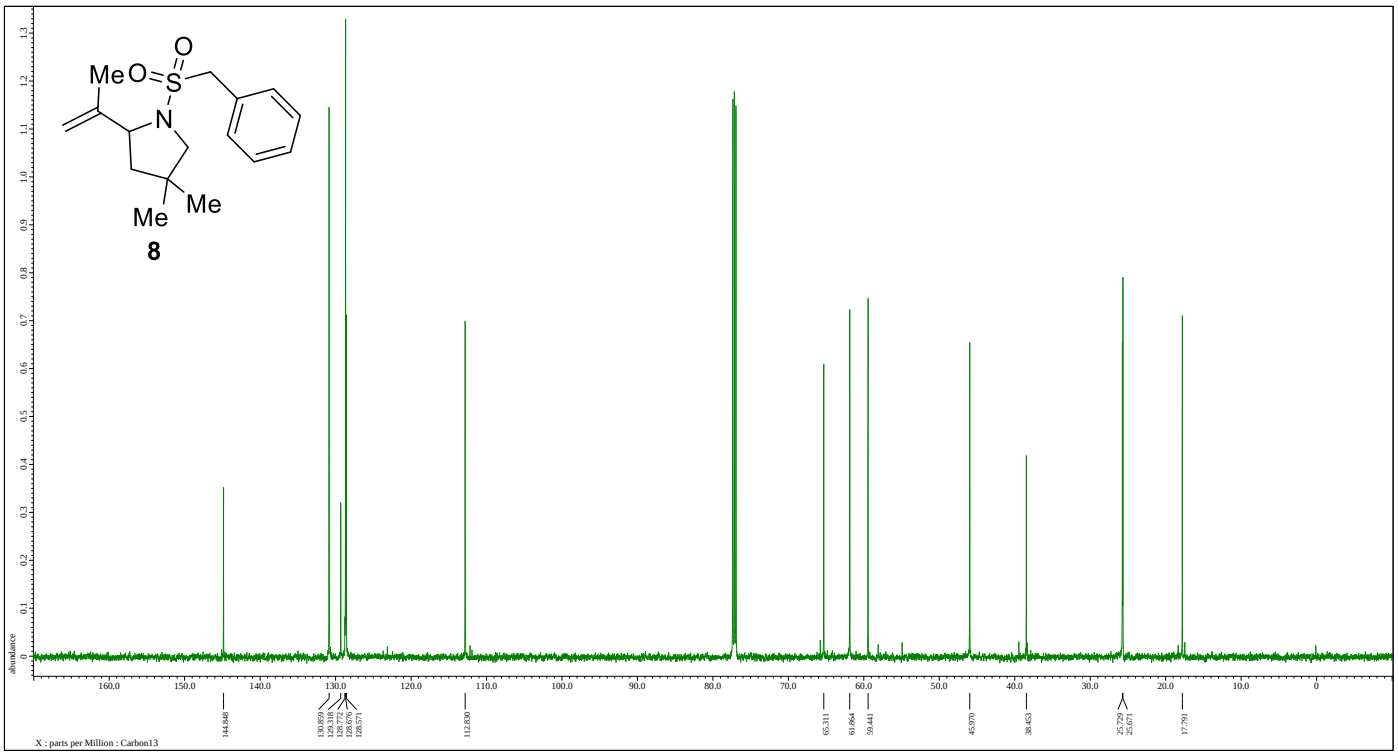
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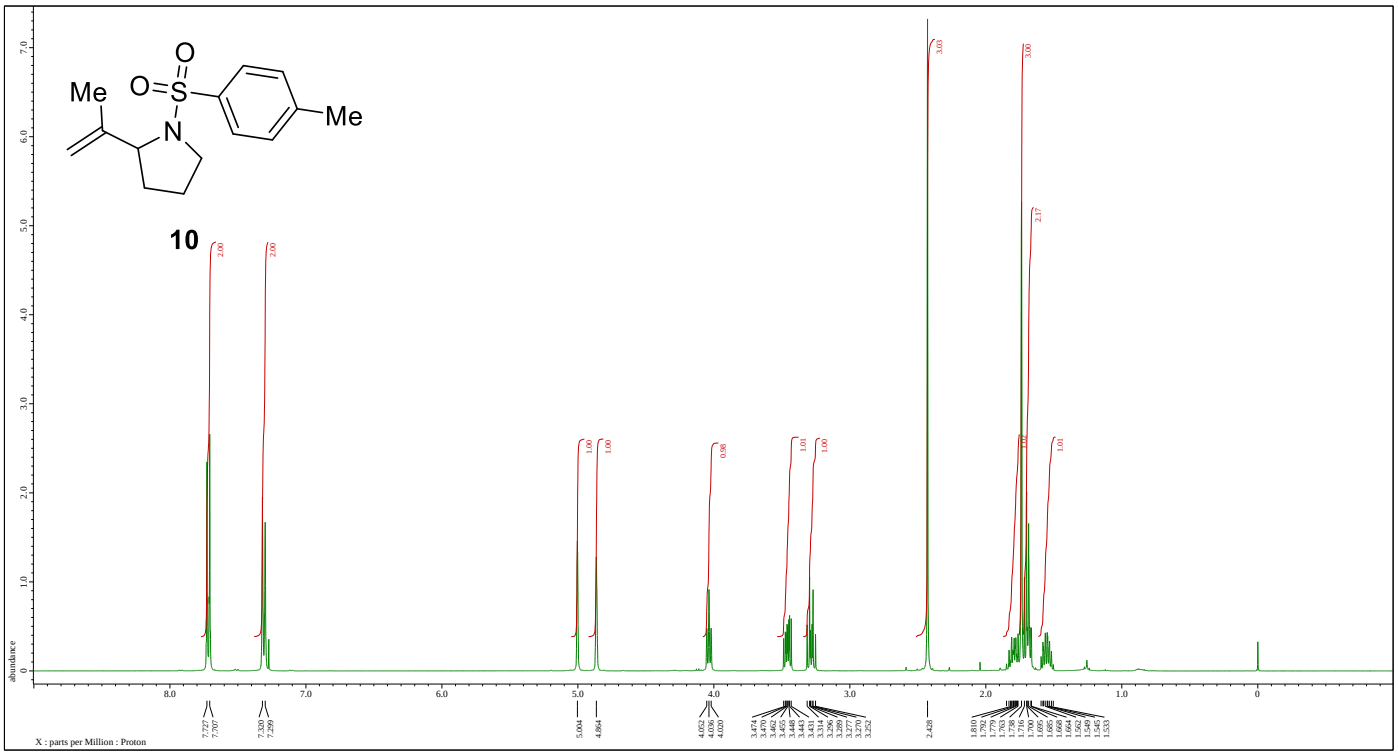
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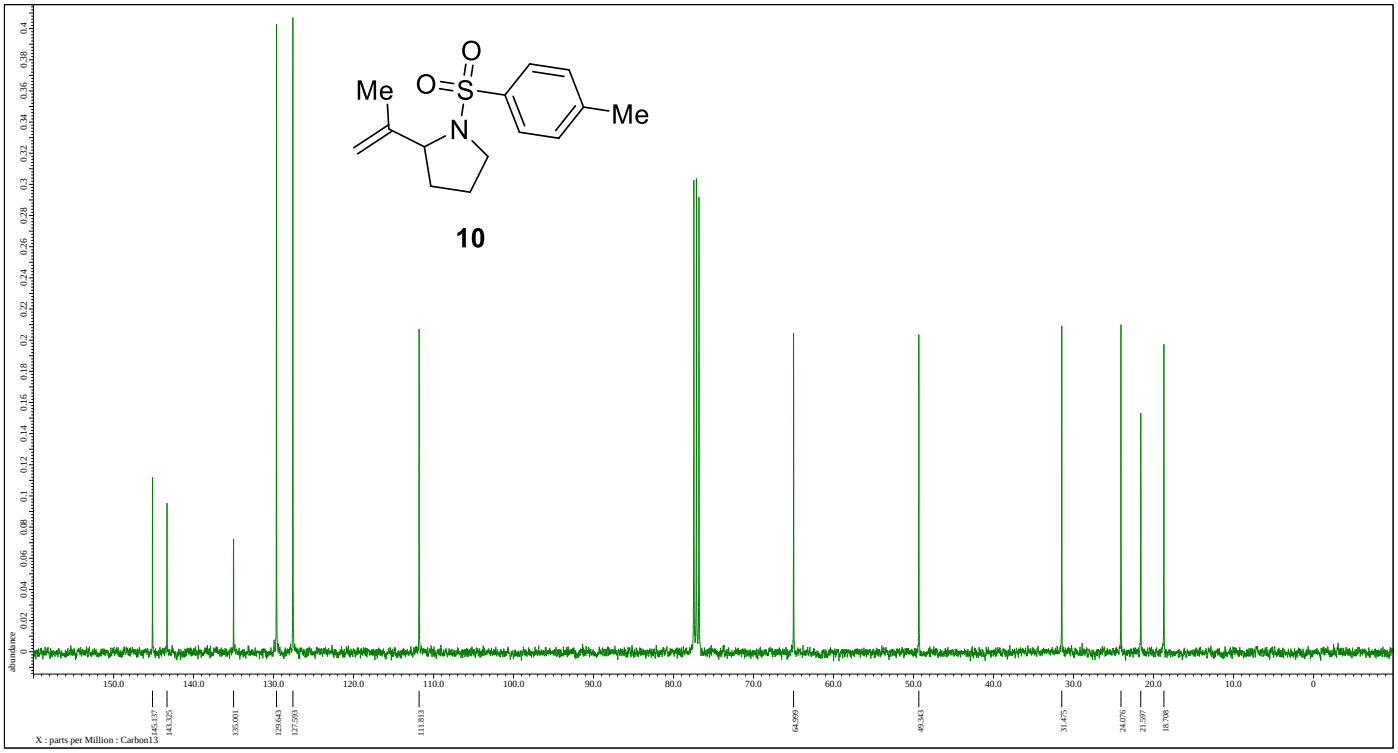
O
O=S
N
Me
10
abundance
X : parts per Million : Proton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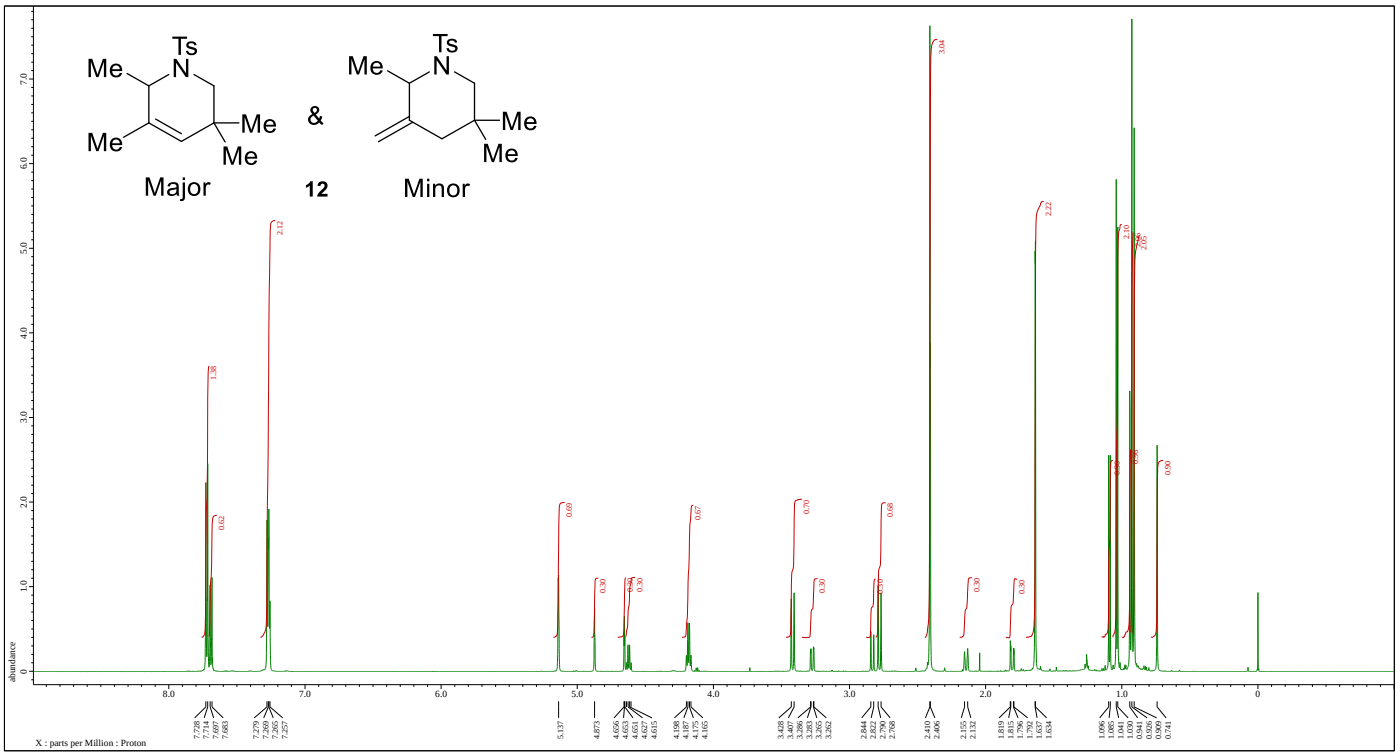
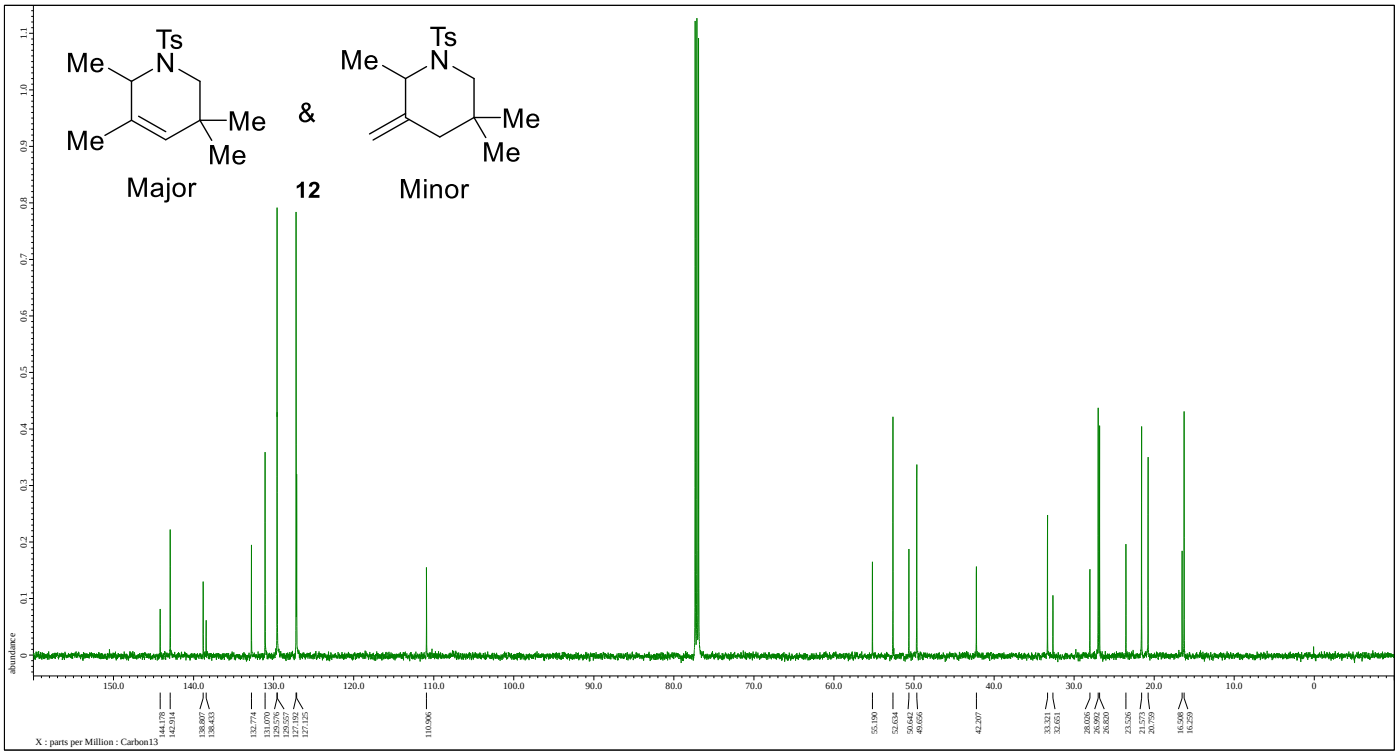
10
abundance
X : parts per Million : Carbon13


Ts
N
Me
Me
Me
Me
Major
&
12
Minor
X : parts per Million : Proton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N
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Me
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Me
Major
&
12
Minor
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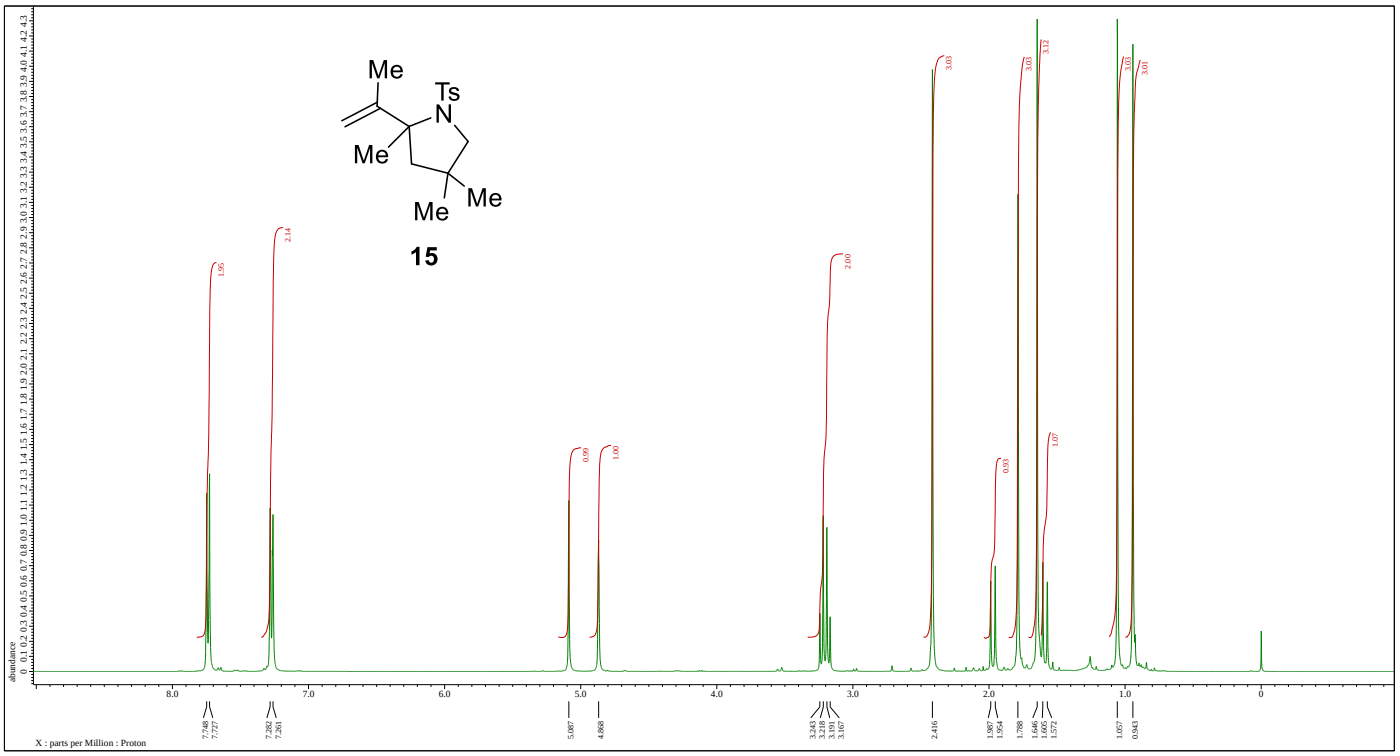
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15
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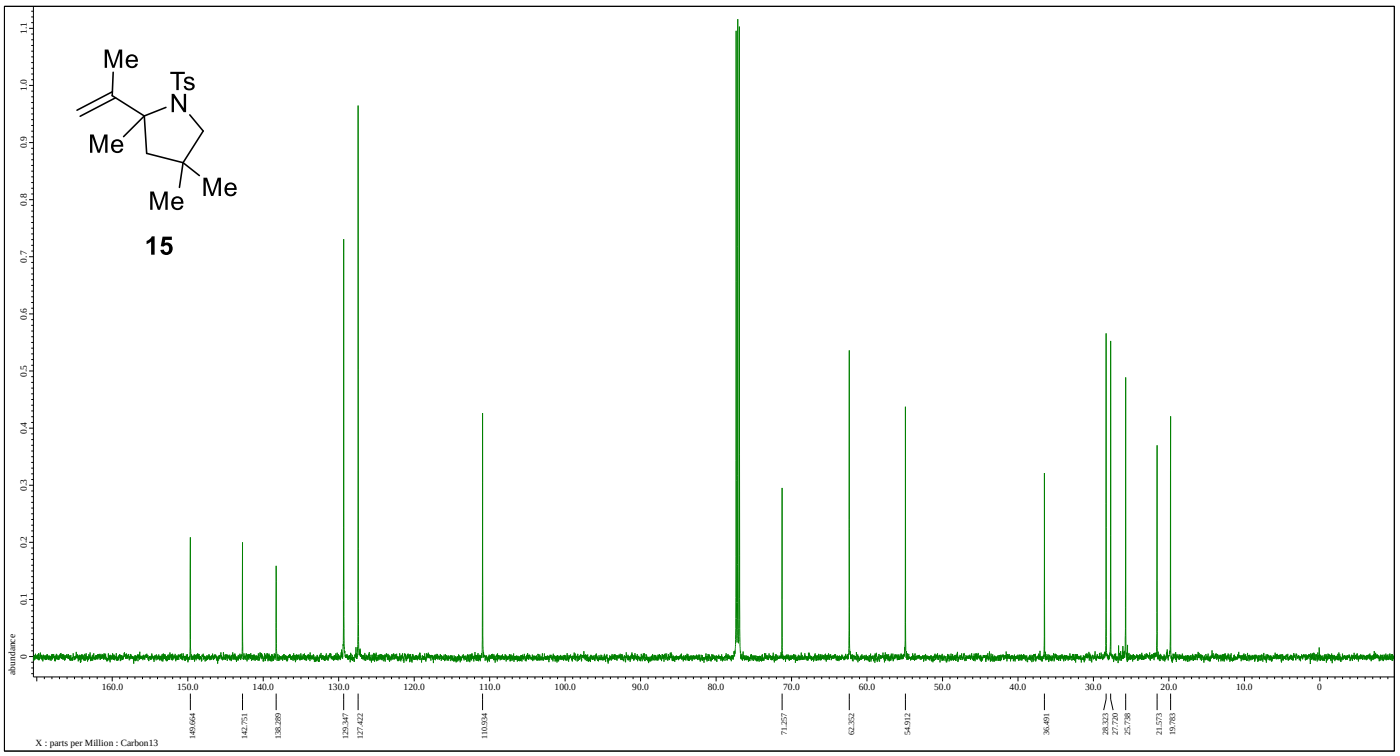
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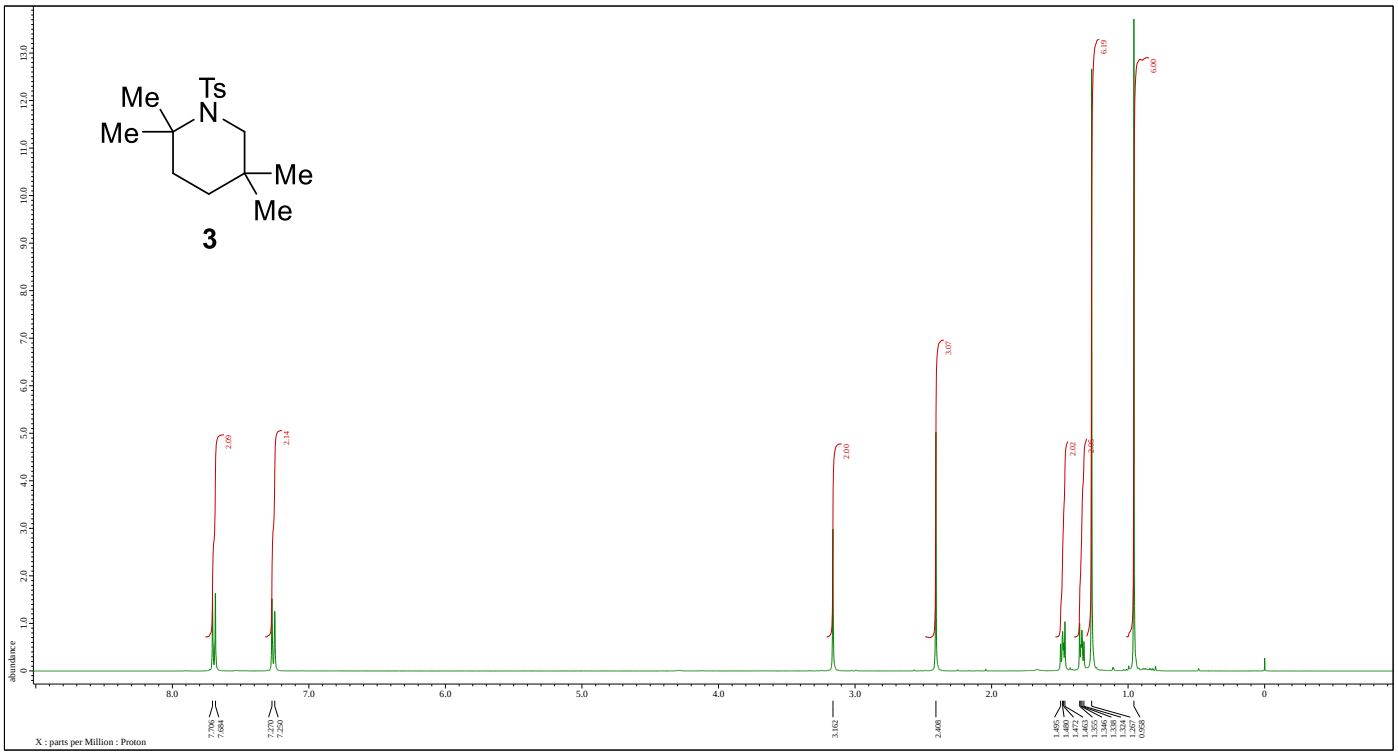
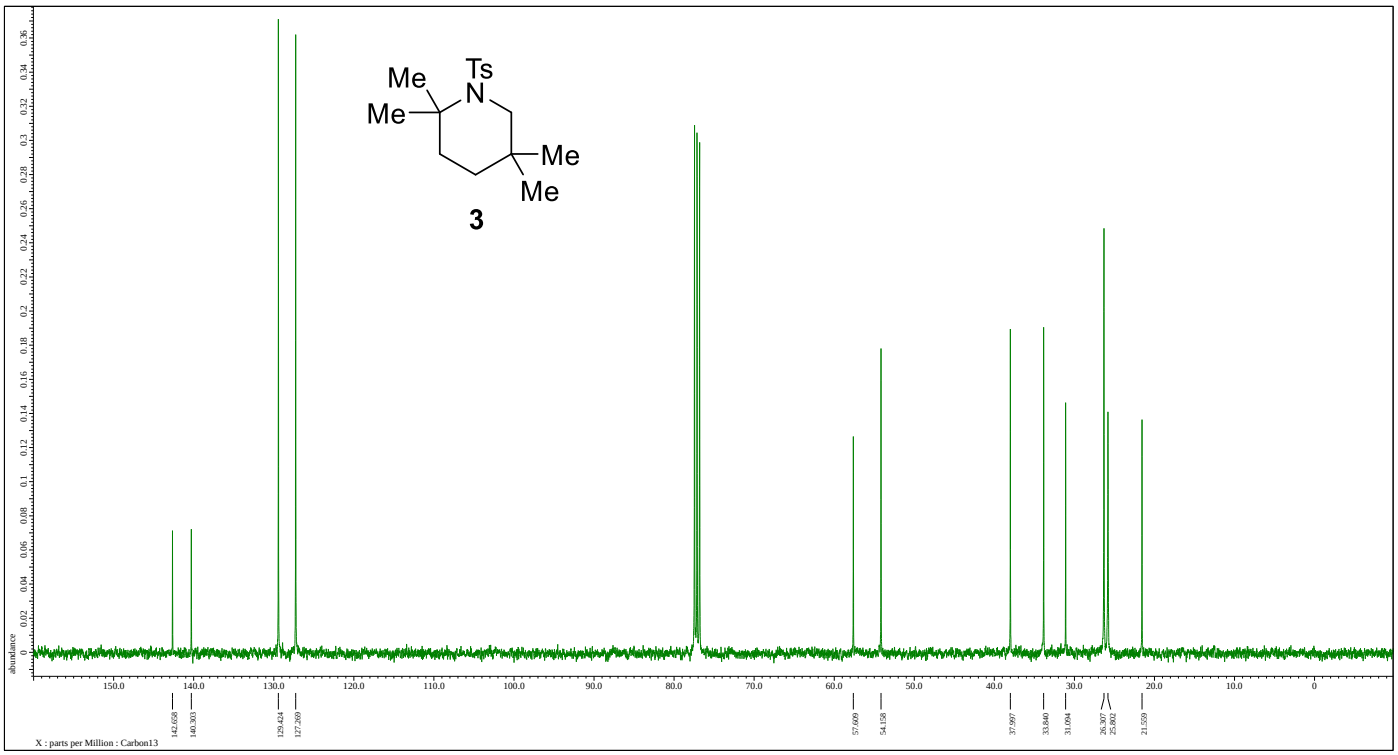
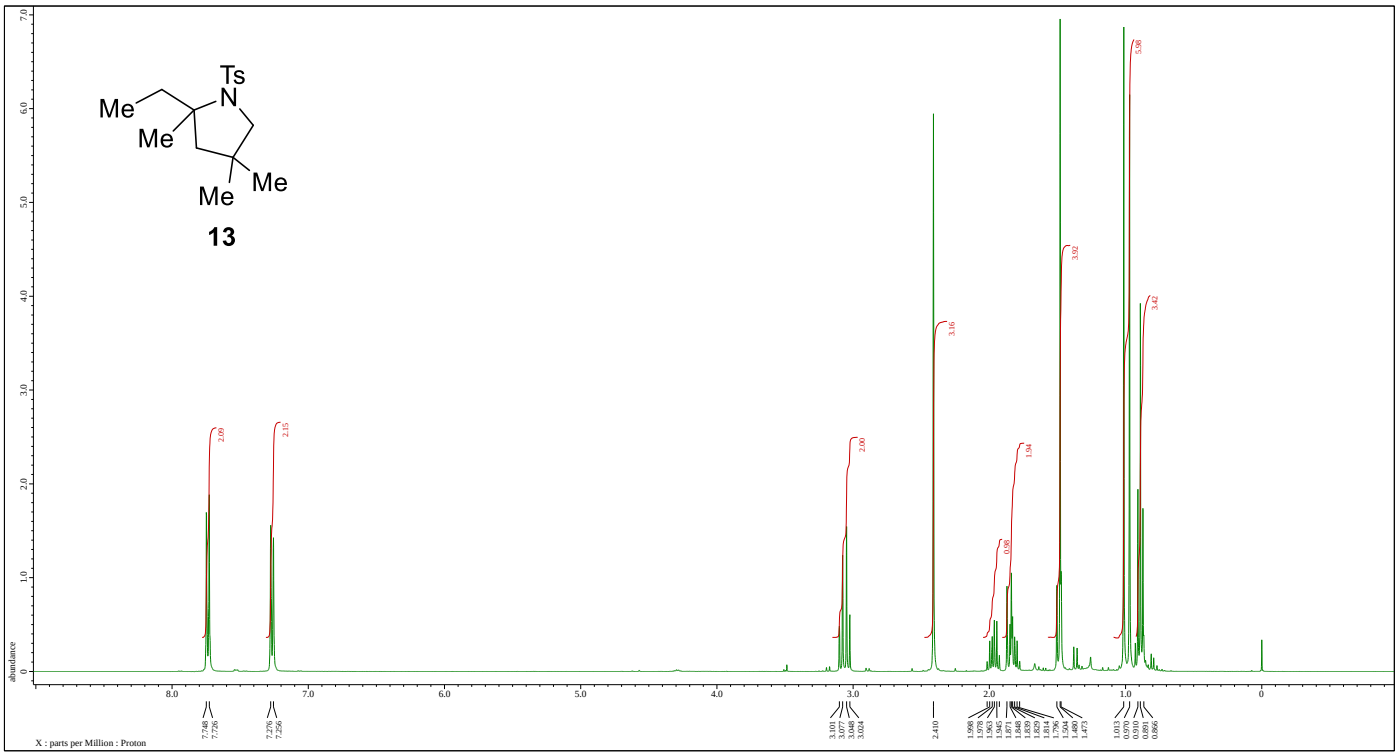
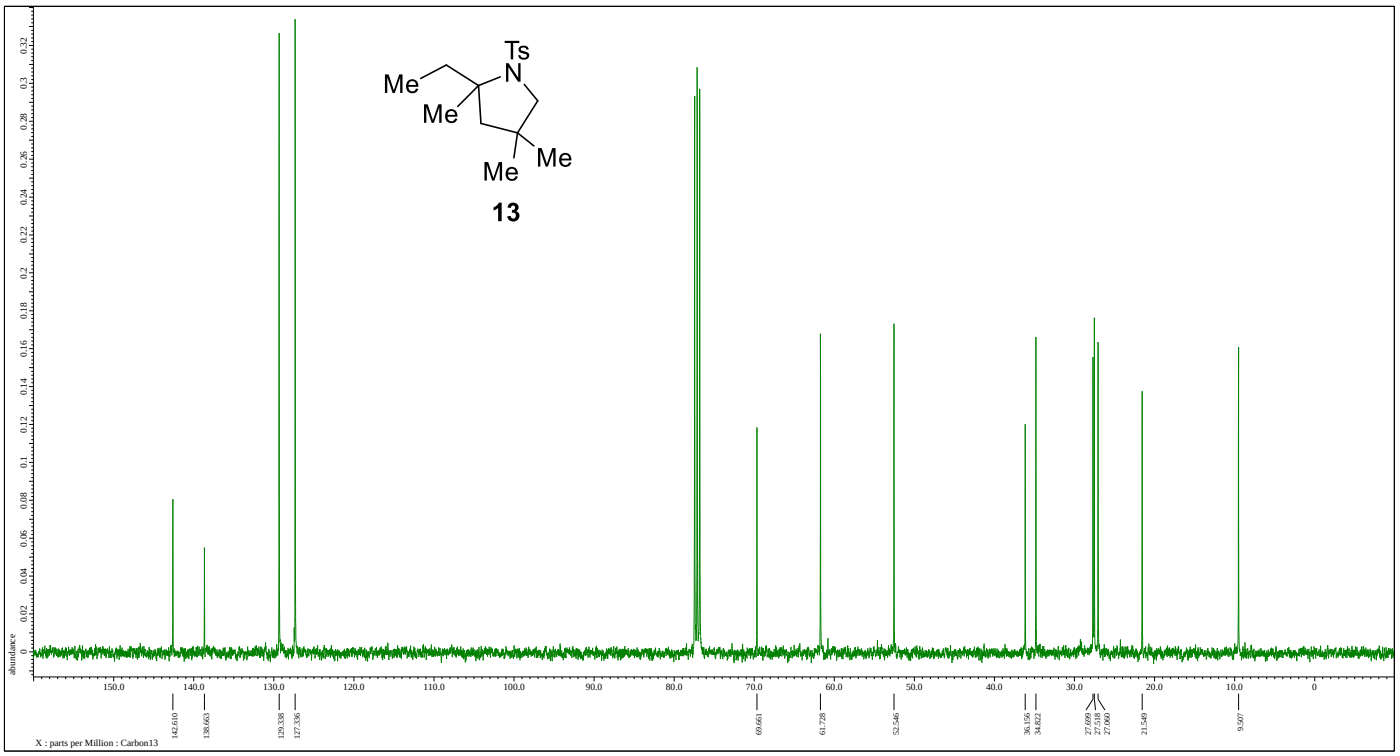
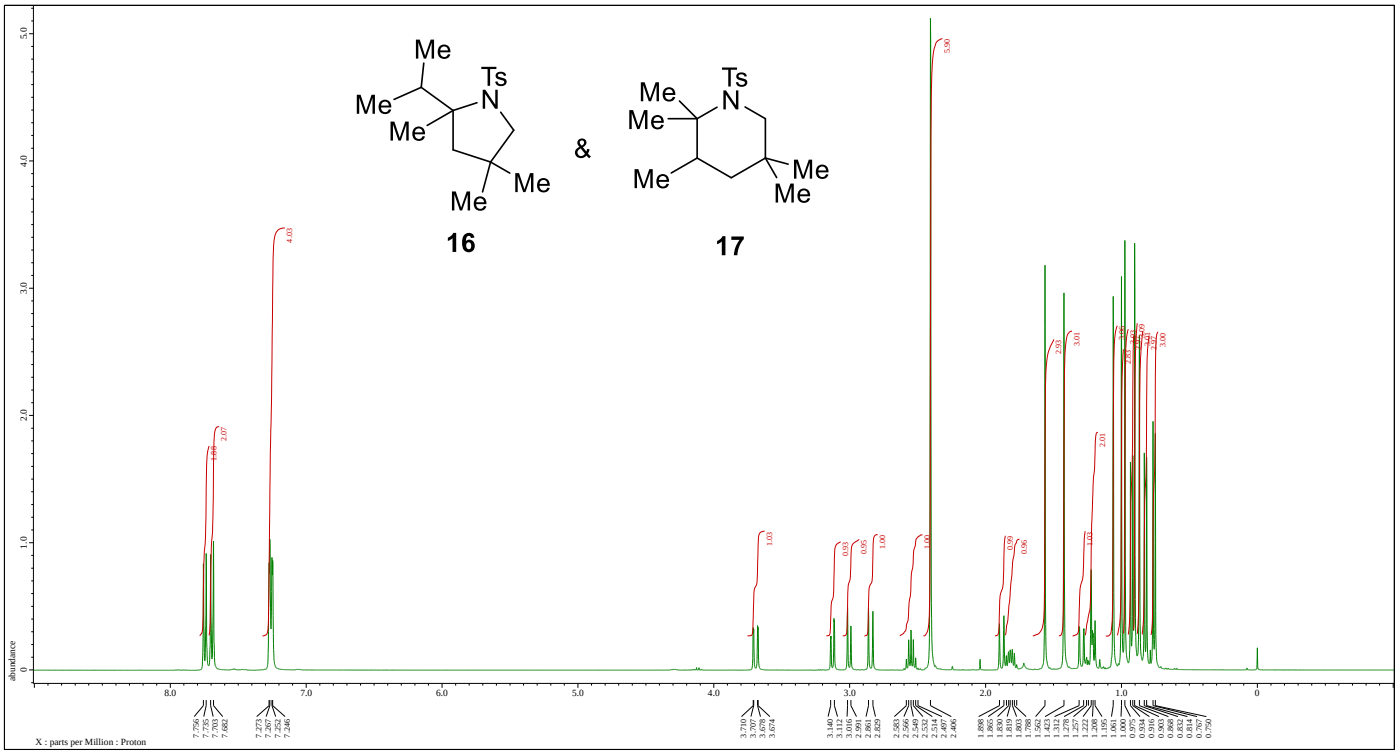
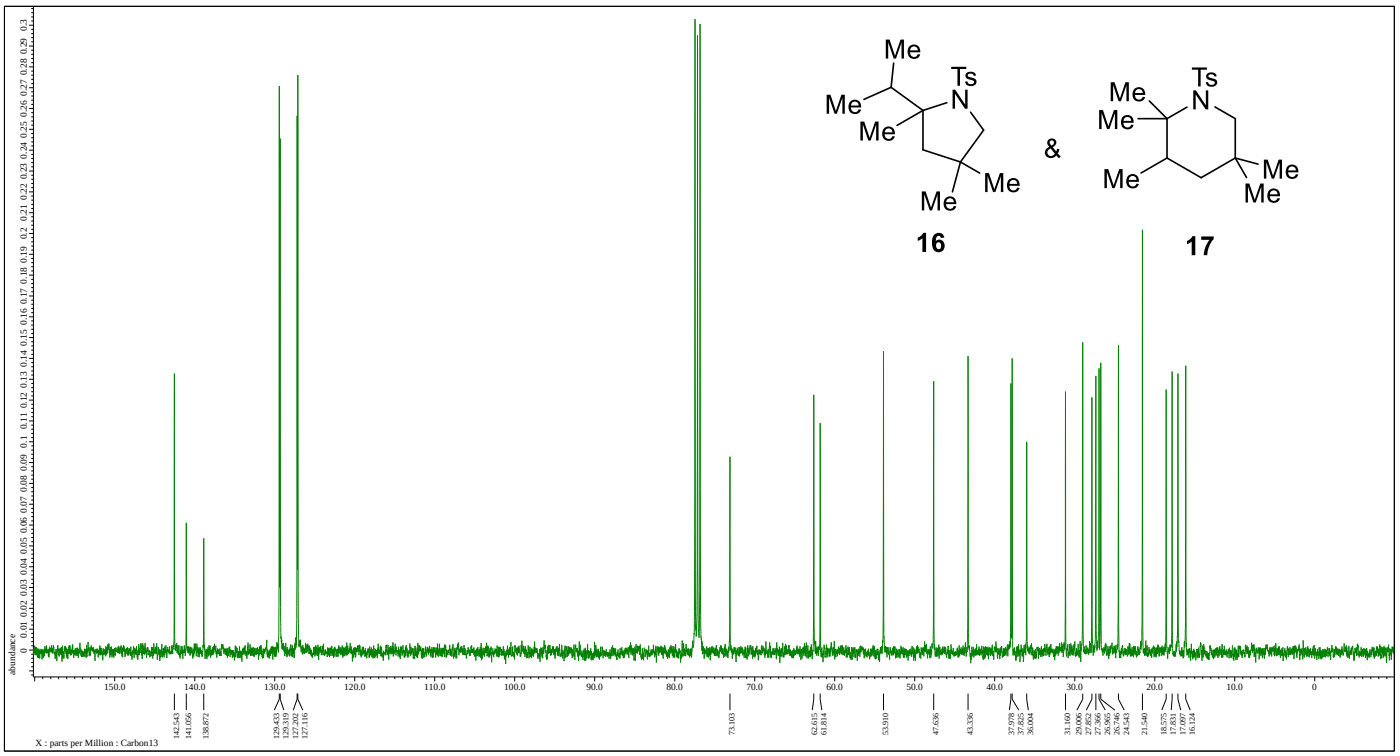

Me
Ts
N
Me
Me
Me
3
X : parts per Million : Proton


Me
Ts
N
Me
Me
Me
3
X : parts per Million : Carbon13


Ts
N
Me
Me
Me
Me
13
abundance
X : parts per Million : Proton


Ts
N
Me
Me
Me
Me
13
abundance
X : parts per Million : Carbon13


Me
Ts
N
Me
Me
Me
Me
16
&
Me
Ts
N
Me
Me
Me
Me
17
X : parts per Million : Proton


Me
Ts
N
Me
Me
Me
16
&
Me
Ts
N
Me
Me
Me
Me
17
X : parts per Million : Carbon13