Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information for

Meyer−Schuster-type Rearrangement for the Synthesis of α-Selanyl-α,β-Unsaturated Thioesters

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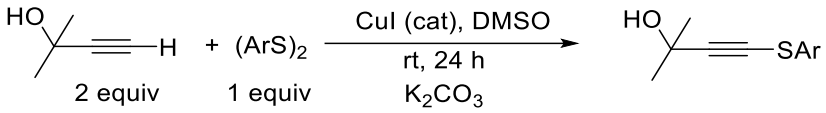
1. General Information

Unless otherwise stated, all glassware was dried before use and all reactions were performed under an atmosphere of argon. All solvents were distilled from appropriate drying agents prior to use. All reagents were used as received from commercial suppliers unless otherwise stated. Reaction progress was monitored by thin layer chromatography (TLC) performed on aluminum plates coated with silica gel F254 with 0.2 mm thickness. Chromatograms were visualized by fluorescence quenching with UV light at 254 nm, iodine and by staining using vanillin solution. Flash column chromatography was performed using silica (230-400 mesh, Merck and co.). Neat infra-red spectra were recorded using a Perkin-Elmer Spectrum 100 FT-IR spectrometer. Wavenumbers (= 1/l) are reported in cm^{-1}. Mass spectra were obtained using a Finnigan MAT 8200 or (70 eV) or an Agilent 5973 (70 eV) spectrometer, using electrospray ionization (ESI). All ^{1}H NMR and ^{13}C NMR spectra were recorded using a 400 MHz spectrometer at 298K (frequency for ^{1}H). Chemical shifts were given in parts per million (ppm, δ), referenced to TMS, defined at δ = 0.0 ppm (^{1}H NMR) and to the solvent peak of $CDCl_3$, defined at δ = 77.0 (^{13}C NMR). Coupling constants are quoted in Hz (*J*). ^{1}H NMR splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q), quintet (qt), sextet (sext), heptet (hept), septet (se) and nonet (n). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m).

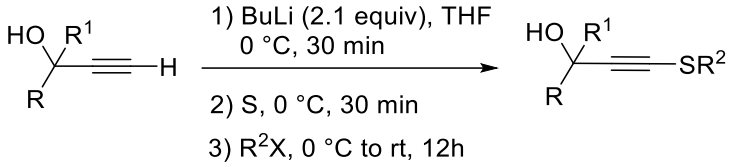
2. General Procedure for the Synthesis of the Starting Materials

Method A is based on the use of disulfides and terminal alkynes, through copper catalysis.[1]



Procedure: To a 25 mL open flask were added dissulfide (2.5 mmol), alkyne (5.0 mmol), 20 mL of undried DMSO, K_2CO_3 (10 mmol) and CuI (0.25 mmol). The solution was stirred at room temperature for 24 hours. After this period, the work up was performed using NH_4Cl (saturated solution) and ethyl acetate. The crude products were purified by flash column chromatography on silica using gradient of hexane and ethyl acetate.

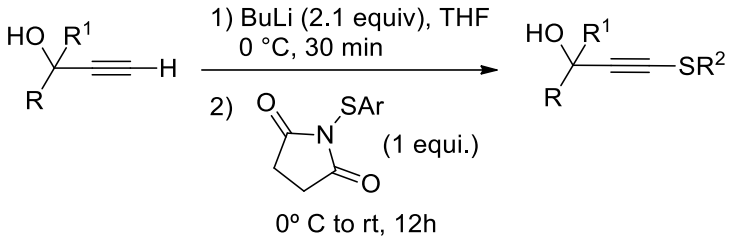
Method B is based on the use of terminal alkynes, *n*-BuLi, sulfur and alkyl halides.



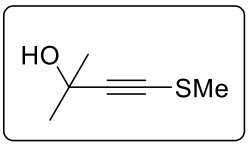
Procedure: To a flame-dried two-necked flask under argon atmosphere were added 8 mL of THF and terminal alkyne (2 mmol). The solution was cooled to 0° C and *n*-BuLi (2.1 equiv, 4.2 mmol) was added. The reaction kept stirring for 30 minutes at 0 °C. After it, S powder (2 mmol, 158 mg) was slowly added (2-3 portions) and the solution kept stirring for 30 minutes at 0° C. Then, the electrophile (1.2 equiv., 2.4 mmol) was added at 0° C and the reaction kept stirring for 12 hours at room temperature. After this period, the work up was performed using NH_4Cl (saturated solution) and ethyl acetate. The crude products were purified by flash column chromatography on silica using gradient of hexane and ethyl acetate.

[1] Bieber, L. W.; Silva, M. F.; Menezes, P. H. *Tetrahedron Lett.* **2004**, *45*, 2735.

Method C is based on the use of terminal alkynes, *n*-BuLi and an electrophilic sulfur source.

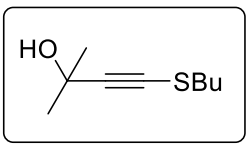


Procedure: To a flame-dried two-necked flask under argon atmosphere were added 8 mL of THF and terminal alkyne (2 mmol). The solution was cooled to 0° C and *n*-BuLi (2.1 equiv, 4.2 mmol) was added. The reaction kept stirring for 30 minutes at 0 °C. After it, an electrophilic sulfur source (1.0 equiv., 2 mmol) was added at 0° C and the reaction kept stirring for 12 hours at room temperature. After this period, the work up was performed using NH_4Cl (saturated solution) and ethyl acetate. The crude products were purified by flash column chromatography on silica using gradient of hexane and ethyl acetate.



2-methyl-4-(methylthio)but-3-yn-2-ol (s1)

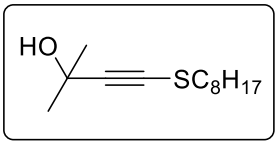
Following the method B, the reaction was performed with 2-methylbut-3-yn-2-ol (1.95 mL, 20 mmol), sulfur (0.67 g, 21 mmol) and iodomethane (1.37 mL, 22 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **77%** yield (2.0 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.67 (s, 1H); 2.31 (s, 3H); 1.46 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 96.9; 73.7; 65.7; 31.2; 19.0. **IR (neat) vmax**: 3346; 2980; 2929; 1362; 1219; 1158; 976; 925; 806. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_6H_{10}NaSO$) requires *m*/*z* 153.0350, found: *m*/*z* 153.0349.



4-(butylthio)-2-methylbut-3-yn-2-ol (s2)

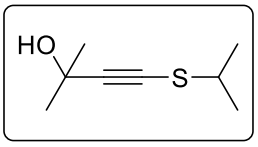
Following the method B, the reaction was performed with 2-methylbut-3-yn-2-ol (0.2 mL, 2 mmol), sulfur (0.067 g, 2.1 mmol) and 1-bromobutane (0.216 mL, 2.2 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **67%** yield (0.23 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.70 (t, *J* = 7.3

Hz, 2H); 2.14 (s, 1H); 1.70 (qt, *J* = 7.3 Hz, 2H); 1.53 (s, 6H); 1.45 (sext, *J* = 7.3 Hz, 2H); 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 98.0; 72.6; 65.9; 35.1; 31.3; 31.2; 21.3; 13.5. **IR (neat) vmax**: 3344; 2959; 2930; 2872; 1459; 1361; 1221; 1159; 978; 807; 739. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_9H_{16}NaSO$) requires *m*/*z* 195.0820, found: *m*/*z* 195.0817.



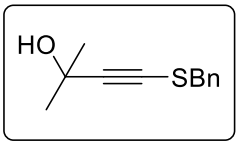
2-methyl-4-(octylthio)but-3-yn-2-ol (s3)

Following the method B, the reaction was performed with 2-methylbut-3-yn-2-ol (0.2 mL, 2 mmol), sulfur (0.067 g, 2.1 mmol) and 1-bromooctane (0.38 mL, 2.2 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **60%** yield (0.274 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.3 Hz, 2H); 1.94 (s, 1H); 1.72 (qt, *J* = 7.3 Hz, 2H); 1.53 (s, 6H); 1.44 – 1.37 (m, 2H); 1.33 – 1.21 (m, 8H); 0.89 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.9; 72.7; 66.0; 35.4; 31.8; 31.4; 29.13; 29.06; 28.2; 22.6; 14.1. **IR (neat) vmax**: 3344; 2924; 2854; 1458; 1362; 1221; 1160; 978; 927; 807; 722. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{24}NaSO$) requires *m*/*z* 251.1446, found: *m*/*z* 251.1446.



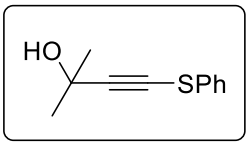
4-(isopropylthio)-2-methylbut-3-yn-2-ol (s4)

Following the method B, the reaction was performed with 2-methylbut-3-yn-2-ol (0.2 mL, 2 mmol), sulfur (0.067 g, 2.1 mmol) and 2-bromopropane (0.21 mL, 2.2 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **22%** yield (0.070 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.14 (hept, *J* = 6.7 Hz, 1H); 1.95 (s, 1H); 1.55 (s, 6H); 1.36 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 100.0; 71.6; 66.0; 39.2; 31.4; 22.8. **IR (neat) vmax**: 3357; 2977; 2928; 1453; 1365; 1222; 1156; 1053; 978; 926; 807. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_8H_{14}NaSO$) requires *m*/*z* 181.0663, found: *m*/*z* 181.0656.



4-(benzylthio)-2-methylbut-3-yn-2-ol (s5)

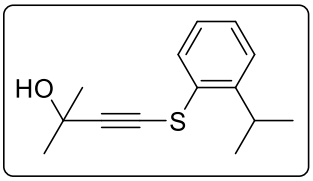
Following the method B, the reaction was performed with 2-methylbut-3-yn-2-ol (0.2 mL, 2 mmol), sulfur (0.067 g, 2.1 mmol) and benzyl bromide (0.26 mL, 2.2 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **47%** yield (0.189 g) as a red solid. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36 – 7.23 (m, 5H); 3.88 (s, 2H); 2.10 (s, 1H); 1.45 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.5; 129.1; 128.4; 127.7; 99.9; 72.1; 65.8; 39.8; 31.2. **IR (neat) vmax**: 3241; 2876; 1543; 1361; 1218; 1163; 1145; 976; 934; 763; 694. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{14}NaSO$) requires *m*/*z* 229.0663, found: *m*/*z* 229.0662.



2-methyl-4-(phenylthio)but-3-yn-2-ol (s6)[2]

Following the method A, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol), 1,2-diphenyldisulfane (0.55 g, 2.5 mmol) and copper iodine (47 mg, 0.25 mmol). Purification by flash column chromatography (from hexane to 8% AcOEt) afforded the title compound in **38%** yield (0.365 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.40 – 7.37 (m, 2H); 7.35 – 7.27 (m, 2H); 7.23 – 7.16 (m, 1H); 2.62 (s, 1H); 1.60 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 132.5; 129.1; 126.4; 125.9; 103.3; 68.7; 66.0; 31.2. **IR (neat) vmax**: 3340; 2980; 1583; 1478; 1440; 1362; 1218; 1158; 1023; 979; 925; 808; 735; 686.

4-((2-isopropylphenyl)thio)-2-methylbut-3-yn-2-ol (s7)

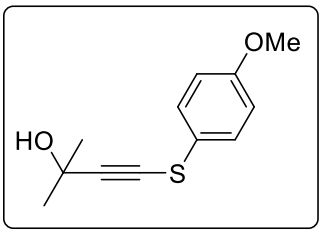


Following the method A, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol), 1,2-bis(2-isopropylphenyl)disulfane (0.76 g, 2.5 mmol) and copper iodine (47 mg, 0.25 mmol). Purification by flash column chromatography (from hexane to 8% AcOEt) afforded the title compound in **22%** yield (0.258 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 – 7.61 (m, 1H); 7.26 – 7.18 (m, 3H); 3.14 (hept, *J* = 6.8 Hz, 1H); 2.42 (s, 1H); 1.60 (s, 6H); 1.24 (d, *J* = 6.8 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 145.6;

[2] Lopes, E. F.; Dalberto, B. T.; Perin, G.; Alves, D.; Barcellos, T.; Lenardão, E. J. *Chem. Eur. J.* **2017**, *23*, 13760.

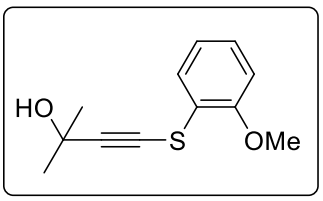
130.4; 126.9; 126.8; 125.4; 102.9; 69.4; 66.1; 31.2; 30.1; 22.9. **IR (neat) vmax**: 2963; 1471; 1363; 1220; 1160; 1044; 979; 926; 751; 730. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{14}H_{18}NaSO$) requires *m*/*z* 257.0976, found: *m*/*z* 257.0971.

4-((4-methoxyphenyl)thio)-2-methylbut-3-yn-2-ol (s8)



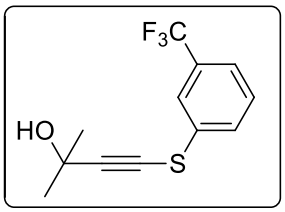
Following the method A, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol), 1,2-bis(4-methoxyphenyl)disulfane (0.696 g, 2.5 mmol) and copper iodine (47 mg, 0.25 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **25%** yield (0.278 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.9 Hz, 2H); 6.88 (d, *J* = 8.9 Hz, 2H); 3.79 (s, 3H); 2.31 (s, 1H); 1.58 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.9; 128.7; 122.7; 115.0; 101.6; 70.3; 66.0; 55.4; 31.3. **IR (neat) vmax**: 3365; 2979; 1592; 1482; 1289; 1242; 1172; 1028; 925; 821; 622. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{14}NaSO_2$) requires *m*/*z* 245.0612, found: *m*/*z* 245.0611.

4-((2-methoxyphenyl)thio)-2-methylbut-3-yn-2-ol (s9)

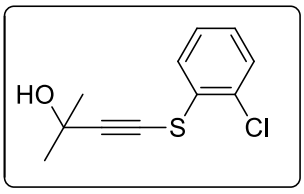


Following the method A, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol), 1,2-bis(2-methoxyphenyl)disulfane (0.696 g, 2.5 mmol) and copper iodine (47 mg, 0.25 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **60%** yield (0.67 g) as an orange oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.55 (dd, *J* = 7.8, 1.6 Hz, 1H); 7.19 (ddd, *J* = 8.1, 7.5, 1.6 Hz, 1H); 7.01 (td, *J* = 7.6, 1.2 Hz, 1H); 6.83 (dd, *J* = 8.1, 1.1 Hz, 1H); 3.87 (s, 3H); 2.21 (s, 1H); 1.63 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.1; 127.2; 126.1; 121.6; 121.2; 110.3; 103.8; 68.6; 66.1; 55.8; 31.3. **IR (neat) vmax**: 3371; 2979; 1477; 1241; 1153; 1062; 1021; 926; 745. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{14}NaSO_2$) requires *m*/*z* 245.0612, found: *m*/*z* 245.0611.

2-methyl-4-((3-(trifluoromethyl)phenyl)thio)but-3-yn-2-ol (s10)

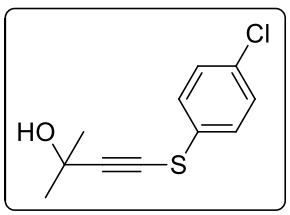


Following the method C, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol) and 1-((3-(trifluoromethyl)phenyl)thio)pyrrolidine-2,5-dione (0.551 g, 2.0 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **35%** yield (0.18 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 (s, 1H); 7.58 – 7.54 (m, 1H); 7.49 – 7.43 (m, 2H); 2.17 (s, 1H); 1.64 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 134.5; 131.7 (q, *J* = 32.5 Hz); 129.5; 128.9 (q, *J* = 1.2 Hz); 123.6 (q, *J* = 272.7 Hz); 123.2 (q, *J* = 3.7 Hz); 122.4 (q, *J* = 4 Hz); 104.9; 67.5; 66.1; 31.2. **^{19}F NMR** (376 MHz, $CDCl_3$): δ -62.9 **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{11}F_3NaSO$) requires *m*/*z* 283.0380, found: *m*/*z* 283.0378.



4-((2-chlorophenyl)thio)-2-methylbut-3-yn-2-ol (s11)

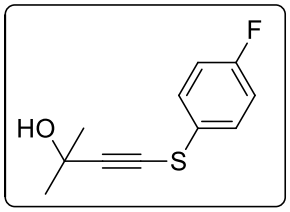
Following the method C, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol) and 1-((2-chlorophenyl)thio)pyrrolidine-2,5-dione (0.483 g, 2.0 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **51%** yield (0.23 g) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.64 (dd, *J* = 7.8, 1.3 Hz, 1H); 7.34 – 7.28 (m, 2H); 7.19 – 7.13 (m, 1H); 2.32 (s, 1H); 1.64 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 132.3; 130.2; 129.4; 127.5; 127.2; 126.6; 104.9; 67.9; 66.1; 31.2. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{11}H_{11}ClNaSO$) requires *m*/*z* 249.0117, found: *m*/*z* 249.0114.



4-((4-chlorophenyl)thio)-2-methylbut-3-yn-2-ol (s12)

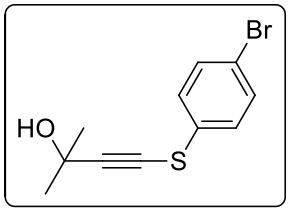
Following the method C, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol) and 1-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (0.483 g, 2.0 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **54%** yield (0.24 g) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35 – 7.27 (m, 4H); 2.36 (s, 1H); 1.61 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 132.4; 131.1; 129.3; 127.2; 103.7; 68.3; 66.1; 31.2.

HRMS (ESI+): exact mass calculated for $[M+Na]^+$ ($C_{11}H_{11}ClNaSO$) requires *m*/*z* 249.0117, found: *m*/*z* 249.0116.



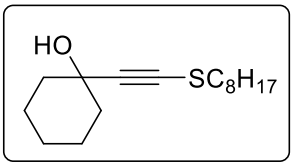
4-((4-fluorophenyl)thio)-2-methylbut-3-yn-2-ol (s13)

Following the method C, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol) and 1-((4-fluorophenyl)thio)pyrrolidine-2,5-dione (0.45 g, 2.0 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **62%** yield (0.26 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.40 – 7.34 (m, 2H); 7.09 – 7.01 (m, 2H); 2.32 (s, 1H); 1.60 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 161.7 (d, *J* = 246.4 Hz); 128.1 (d, *J* = 8.1 Hz); 127.5 (d, *J* = 3.3 Hz); 116.4 (d, *J* = 22.4 Hz); 102.9; 69.1; 66.1; 31.2. **^{19}F NMR** (376 MHz, $CDCl_3$): δ -115.4. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{11}H_{11}FNaSO$) requires *m*/*z* 233.0412, found: *m*/*z* 233.0415.



4-((4-bromophenyl)thio)-2-methylbut-3-yn-2-ol (s14)

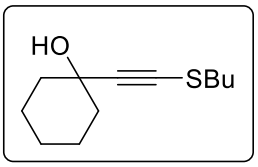
Following the method C, the reaction was performed with 2-methylbut-3-yn-2-ol (0.5 mL, 5 mmol) and 1-((4-bromophenyl)thio)pyrrolidine-2,5-dione (0.57 g, 2.0 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **45%** yield (0.19 g) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.44 (d, *J* = 8.6 Hz, 2H); 7.26 (d, *J* = 8.6 Hz, 2H); 2.32 (s, 1H); 1.61 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 132.2; 131.9; 127.5; 120.2; 103.9; 68.2; 66.1; 31.2. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{11}H_{11}BrNaSO$) requires *m*/*z* 292.9612 found: *m*/*z* 292.9613.



1-((octylthio)ethynyl)cyclohexan-1-ol (s17)

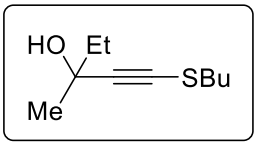
Following the method B, the reaction was performed with 1-ethynylcyclohexan-1-ol (0.65 mL, 5 mmol), sulfur (0.16 g, 5 mmol) and 1-bromooctane (0.95 mL, 5.5 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **67%** yield (0.9 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.3 Hz, 2H); 1.97 (s, 1H); 1.95 – 1.85 (m, 2H); 1.78 – 1.63 (m, 4H);

1.64 – 1.48 (m, 5H); 1.48 – 1.36 (m, 2H); 1.37 – 1.18 (m, 9H); 0.88 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.1; 74.5; 69.5; 40.0; 35.5; 31.8; 29.2; 29.12; 29.08; 28.2; 25.2; 23.3; 22.6; 14.1. **IR (neat) vmax**: 3349; 2940; 2921; 1448; 1207; 1154; 1071; 990; 722. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{16}H_{28}NaSO$) requires *m*/*z* 291.1758, found: *m*/*z* 291.1756.



1-((butylthio)ethynyl)cyclohexan-1-ol (s18)

Following the method B, the reaction was performed with 1-ethynylcyclohexan-1-ol (0.65 mL, 5 mmol), sulfur (0.16 g, 5 mmol) and 1-bromobutane (0.6 mL, 5.5 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **66%** yield (0.7 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.71 (t, *J* = 7.3 Hz, 2H); 2.07 (s, 1H); 1.96 – 1.86 (m, 2H); 1.77 – 1.64 (m, 4H); 1.61 – 1.51 (m, 5H); 1.44 (sext, *J* = 7.3 Hz, 2H); 1.35 – 1.20 (m, 1H); 0.94 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.0; 74.5; 69.5; 39.9; 35.2; 31.2; 25.1; 23.3; 21.3; 13.5. **IR (neat) vmax**: 3347; 2930; 2856; 1447; 1339; 1257; 1155; 1057; 1033; 961; 903; 781. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{20}NaSO$) requires *m*/*z* 235.1133, found: *m*/*z* 235.1132.

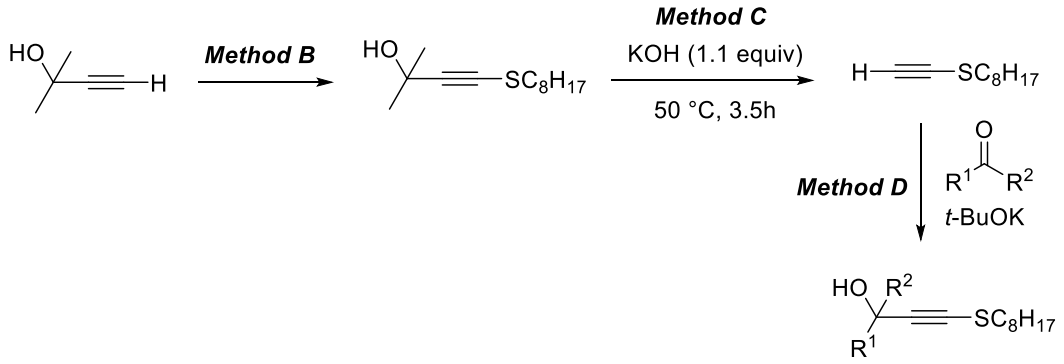


1-(butylthio)-3-methylpent-1-yn-3-ol (s21)

Following the method B, the reaction was performed with 3-methylpent-1-yn-3-ol (0.57 mL, 5 mmol), sulfur (0.16 g, 5 mmol) and bromobutane (0.6 mL, 5.5 mmol). Purification by flash column chromatography (from hexane to 10% AcOEt) afforded the title compound in **55%** yield (0.511 g) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.70 (t, *J* = 7.3 Hz, 2H); 2.05 (s, 1H); 1.75 – 1.66 (m, 4H); 1.48 (s, 3H); 1.44 (sext, *J* = 7.3 Hz, 2H); 1.03 (t, *J* = 7.4 Hz, 3H); 0.94 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.0; 73.7; 69.5; 36.5; 35.2; 31.2; 29.3; 21.3; 13.5; 9.0. **IR (neat) vmax**: 3363; 2962; 2931; 1460; 1377; 1126; 1000; 951; 908; 808. **HRMS (ESI+):** exact mass calculated for $[M+H]^+$ ($C_{10}H_{19}SO$) requires *m*/*z* 187.1157, found: *m*/*z* 187.1153.

Thioalkynes **s15, s16, s19, s22** and **s23** were prepared according to the synthetic route shown below. Compound **s3** is obtained from method B. This

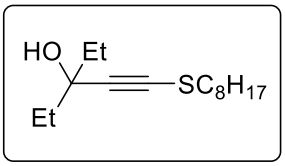
thioalkyne is used in a reaction involving the use of KOH and hexane to form terminal thioacetylene, which is then used in a reaction with different ketones to form the starting materials.



Scheme 1. Synthetic route used for substrate synthesis.

Method C:[2] In a two-necked flask with reflux condenser were added the thioalkyne **s3** (0.46 g, 2 mmol, 1 equiv), KOH (124 mg, 2.2 mmol, 1.1 equiv) and 6 mL of hexane. The system was heated to 50 ° C and stirred for 3.5 h. The crude products were purified by flash chromatography on silica using hexane as eluent.

Method D:[3] In a two-necked flask were added terminal thioacetylene (0.256 g, 1.5 mmol, 1.5 equiv.), *tert*-BuOK (112 mg, 1 mmol, 1 equiv.) and the carbonyl compound (1 mmol, 1 equiv.). The reaction was stirred at room temperature for 2 hours. The crude was purified by flash column chromatography on silica using hexane and ethyl acetate as eluent.

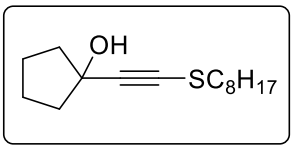


3-ethyl-1-(octylthio)pent-1-yn-3-ol (s15)

Following the general procedure, the compound **s10** was obtained in **63%** yield (162 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.4 Hz, 2H); 1.87 (s, 1H); 1.77 – 1.60 (m, 6H); 1.48 – 1.34 (m, 2H); 1.34 – 1.22 (m, 8H); 1.03 (t, *J* = 7.4 Hz, 6H); 0.88 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 96.1; 74.6; 73.0; 35.6; 34.5; 31.8; 29.2; 29.12; 29.08; 28.2; 22.6; 14.1; 8.6. **IR (neat) vmax**: 3385, 2962, 2854, 2024,

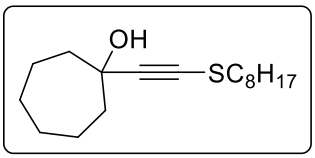
[3] Chem, S.; Yuan, F.; Zhao, H.; Li, B. *Res*. *Chem*. *Intermediat.* **2013**, *39*, 2391.

1453, 1317, 1142, 1045, 984, 951, 820, 723. **HRMS (ESI+):** exact mass calculated for $[M+H]^+$ ($C_{15}H_{29}SO$) requires *m*/*z* 257.1938, found: *m*/*z* 257.1931.



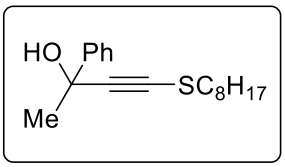
1-((octylthio)ethynyl)cyclopentan-1-ol (s16)

Following the general procedure, the compound **s11** was obtained in **60%** yield (153 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.4 Hz, 2H); 2.02 – 1.66 (m, 11H); 1.46 – 1.33 (m, 2H); 1.34 – 1.22 (m, 8H); 0.88 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.1; 75.3; 73.5; 42.5; 35.5; 31.8; 29.2; 29.13; 29.07; 28.2; 23.5; 22.6; 14.1. **IR (neat) vmax**: 3353; 2955; 2924; 2854; 1456; 1209; 1074; 992; 722. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{15}H_{26}NaSO$) requires *m*/*z* 277.1602, found: *m*/*z* 277.1595.



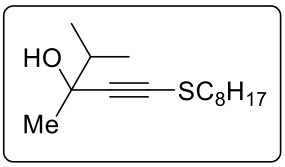
1-((octylthio)ethynyl)cycloheptan-1-ol (s19)

Following the general procedure, the compound **s14** was obtained in **60%** yield (170 mg) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.3 Hz, 2H); 2.05 – 1.97 (m, 2H); 1.94 (s, 1H); 1.87 – 1.80 (m, 2H); 1.78 – 1.48 (m, 10H); 1.47 – 1.36 (m, 2H); 1.36 – 1.21 (m, 8H); 0.88 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 98.1; 73.7; 72.6; 43.1; 35.5; 31.8; 29.2; 29.1; 29.07; 28.2; 27.9; 22.6; 22.2; 14.1. **IR (neat) vmax**: 3365; 2923; 2853; 1458; 1190; 1021; 908. **HRMS (ESI+):** exact mass calculated for $[M+H]^+$ ($C_{17}H_{31}SO$) requires *m*/*z* 283.2096, found: *m*/*z* 283.2095.



4-(octylthio)-2-phenylbut-3-yn-2-ol (s22)

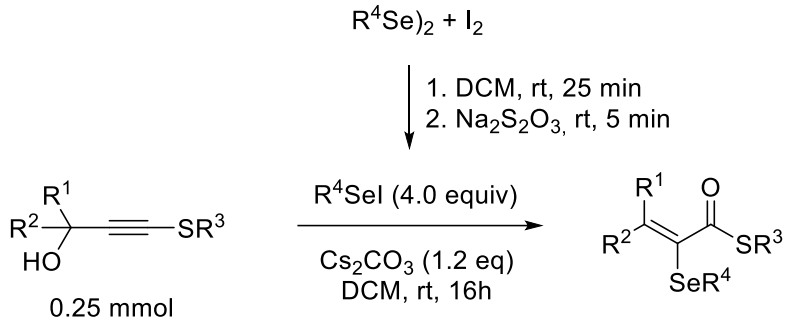
Following the general procedure, the compound **s17** was obtained in **51%** yield (148 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 – 7.62 (m, 2H); 7.39 – 7.33 (m, 2H); 7.32 – 7.26 (m, 1H); 2.74 (t, *J* = 7.3 Hz, 2H); 2.46 (s, 1H); 1.78 (s, 3H); 1.79 – 1.70 (m, 2H); 1.41 (qt, *J* = 6.8 Hz, 2H); 1.35 – 1.25 (m, 8H); 0.89 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.5; 128.2; 127.6; 124.9; 96.7; 75.7; 70.7; 35.5; 33.2; 31.7; 29.3; 29.1; 29.06; 28.2; 22.6; 14.1. **IR (neat) vmax**: 2924; 2853; 1447; 1164; 1089; 1027; 900; 762; 697. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{26}NaSO$) requires *m*/*z* 313.1602, found: *m*/*z* 313.1598.



3,4-dimethyl-1-(octylthio)pent-1-yn-3-ol (s23)

Following the general procedure, the compound **s18** was obtained in **49%** yield (126 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.68 (t, *J* = 7.3 Hz, 2H); 1.92 (s, 1H); 1.81 (hept, *J* = 6.8 Hz, 1H); 1.72 (qt, *J* = 7.2 Hz, 2H); 1.44 (s, 3H); 1.43 – 1.36 (m, 2H); 1.32 – 1.23 (m, 8H); 1.02 (d, *J* = 6.8 Hz, 3H); 0.99 (d, *J* = 6.8 Hz, 3H); 0.87 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 96.3; 74.1; 72.5; 39.0; 35.5; 31.7; 29.2; 29.1; 29.06; 28.2; 27.2; 22.6; 18.0; 17.5; 14.1. **IR (neat) vmax**: 3410; 2958; 2925; 2854; 1460; 1370; 1141; 1096; 1066; 927; 876; 723. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{15}H_{28}NaSO$) requires *m*/*z* 279.1759, found: *m*/*z* 279.1756.

3. General procedure for the synthesis of thioesters

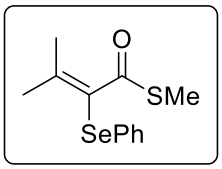


Part A) Preparation of electrophilic species:

To a flame-dried Schlenk flask under argon atmosphere were added diselenide (0.5 mmol, 2 equiv.), 3 mL of dry DCM and iodine (0.5 mmol, 2 equiv, 127 mg). The reaction kept stirring for 25 minutes. Then, $Na_2S_2O_3$ (0.15 mmol, 0.6 equiv, 24 mg) was added and the reaction kept stirring for another 5 minutes. The electrophilic species of selenium was added to the flask containing the thioalkyne.

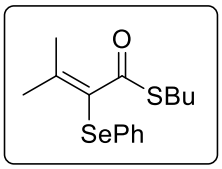
Part B) To a flame-dried Schlenk flask under argon atmosphere were added thioalkyne (0.25 mmol, 1 equiv), Cs_2CO_3 (0.3 mmol, 1.2 equiv, 98 mg) and 2.5 mL of dry DCM. The electrophilic species of selenium was added and the reaction kept stirring for 16 hours at room temperature. After this period, the work up was performed using $Na_2S_2O_3$ (saturated solution) and ethyl acetate.

The crude product was purified on flash column chromatography using a gradient of hexane and ethyl acetate as eluent (starting with 100% hexane to remove diselenide byproducts and then eluting with 2% ethyl acetate/hexane to collect the product).



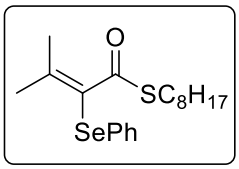
S-methyl 3-methyl-2-(phenylselanyl)but-2-enethioate (1)

Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **88%** yield (75 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.39 – 7.35 (m, 2H); 7.28 – 7.19 (m, 3H); 2.24 (s, 3H); 2.16 (s, 3H); 2.156 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 194.6; 152.3; 131.3; 130.3; 129.2; 126.6; 124.5; 26.8; 23.4; 13.4. **IR (neat) vmax**: 2923; 1737; 1649; 1576; 1475; 1437; 1366; 1127; 1068; 888; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{14}NaOSSe$) requires *m*/*z* 308.9828, found: *m*/*z* 308.9826.



S-butyl 3-methyl-2-(phenylselanyl)but-2-enethioate (2)

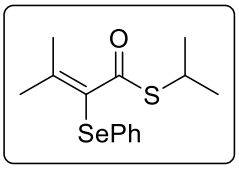
Following the general procedure, the reaction was performed with thioalkyne **s2** (43 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **78%** yield (64 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.41 – 7.36 (m, 2H); 7.27 – 7.19 (m, 3H); 2.80 (t, *J* = 7.3 Hz, 2H); 2.13 (s, 3H); 2.12 (s, 3H); 1.45 (qt, *J* = 7.3 Hz, 2H); 1.29 (sext, *J* = 7.3 Hz, 2H); 0.85 (t, *J* = 7,3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 194.3; 150.4; 131.2; 130.9; 129.1; 126.7; 124.9; 31.4; 30.0; 26.4; 23.3; 21.9; 13.6. **IR (neat) vmax**: 2925; 2852; 1651; 1577; 1471; 1366; 1127; 1073; 889; 733; 688. **HRMS (ESI+):** exact mass calculated for $[M+H]^+$ ($C_{15}H_{21}OSSe$) requires *m*/*z* 329.0478, found: *m*/*z* 329.0475.



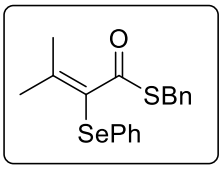
S-octyl 3-methyl-2-(phenylselanyl)but-2-enethioate (3)

Following the general procedure, the reaction was performed with thioalkyne **s3** (57 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **82%** yield (79 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42 – 7.36 (m, 2H); 7.27 – 7.17 (m, 3H); 2.79 (t, *J* = 7.2 Hz, 2H); 2.13 (s, 3H); 2.12 (s, 3H); 1.50 – 1.41 (m, 2H); 1.29 – 1.21 (m, 10H); 0.87 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.3; 150.4; 131.2; 130.8; 129.1; 126.7; 124.9; 31.7; 30.3; 29.3; 29.1; 29.06; 28.8; 26.3; 23.3; 22.6; 14.1. **IR (neat) vmax**: 2923; 2852; 1650; 1577; 1476; 1437; 1366; 1127; 1068; 1022; 889; 733; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{19}H_{28}NaOSSe$) requires *m*/*z* 407.0924, found: *m*/*z* 407.0919.

S-isopropyl 3-methyl-2-(phenylselanyl)but-2-enethioate (4)



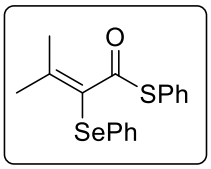
Following the general procedure, the reaction was performed with thioalkyne **s4** (40 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **75%** yield (70 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.41 – 7.38 (m, 2H); 7.26 – 7.18 (m, 3H); 3.52 (hept, *J* = 6.9 Hz, 1H); 2.12 – 2.10 (m, 6H); 1.21 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.3; 149.6; 131.1; 131.0; 129.1; 126.8; 124.9; 35.7; 26.2; 23.2; 22.7. **IR (neat) vmax**: 2961; 1645; 1577; 1476; 1438; 1366; 1242; 1032; 889; 734; 689. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{14}H_{18}NaOSSe$) requires *m*/*z* 337.0141, found: *m*/*z* 337.0138.



S-benzyl 3-methyl-2-(phenylselanyl)but-2-enethioate (5)

Following the general procedure, the reaction was performed with thioalkyne **s5** (52 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **81%** yield (73 mg) as a yellow oil. **^{1}H NMR** (400

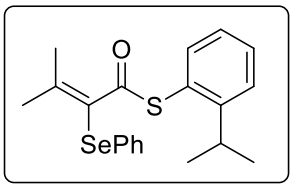
MHz, $CDCl_3$): δ 7.37 – 7.34 (m, 2H); 7.25 – 7.14 (m, 8H); 4.04 (s, 2H); 2.15 (s, 3H); 2.14 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.4; 152.2; 137.6; 131.1; 130.7; 129.2; 128.8; 128.4; 126.9; 126.8; 124.5; 34.9; 26.8; 23.5. **IR (neat) vmax**: 3056; 2913; 1642; 1590; 1474; 1452; 1125; 1071; 1026; 888; 795; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{18}NaOSSe$) requires *m*/*z* 385.0141, found: *m*/*z* 385.0139.



S-phenyl 3-methyl-2-(phenylselanyl)but-2-enethioate (6)

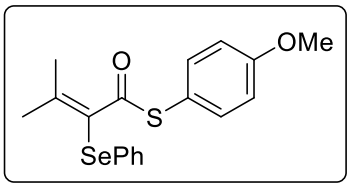
Following the general procedure, the reaction was performed with thioalkyne **s6** (48 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **82%** yield (71 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48 – 7.43 (m, 2H); 7.38 – 7.34 (m, 3H); 7.30 – 7.22 (m, 5H); 2.18 (s, 3H); 2.17 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2; 152.4; 134.5; 131.1; 129.6; 129.3; 129.1; 129.0; 127.0; 124.5; 26.8; 23.7. **IR (neat) vmax**: 3056, 2917, 1663, 1576, 1476, 1438, 1123, 1021, 955, 787, 703. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{16}NaOSSe$) requires *m*/*z* 370.9985, found: *m*/*z* 370.9983.

S-(2-isopropylphenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (7)



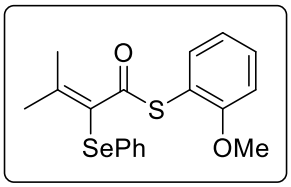
Following the general procedure, the reaction was performed with thioalkyne **s7** (59 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **84%** yield (82 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.49 – 7.44 (m, 2H); 7.38 – 7.23 (m, 5H); 7.19 – 7.12 (m, 2H); 2.95 (hept, *J* = 6.9 Hz, 1H); 2.20 (s, 3H); 2.14 (s, 3H); 1.04 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2; 152.3; 151.3; 136.2; 131.2; 131.17; 130.2; 129.2; 127.5; 126.9; 126.2; 126.1; 125.1; 30.8; 26.7; 23.6; 23.5. **IR (neat) vmax**: 2958, 2921, 2864, 1664, 1577, 1473, 1436, 1260, 1124, 1066, 883, 780, 735. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{20}H_{22}NaOSSe$) requires *m*/*z* 413.0454, found: *m*/*z* 413.0464.

S-(4-methoxyphenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (8)



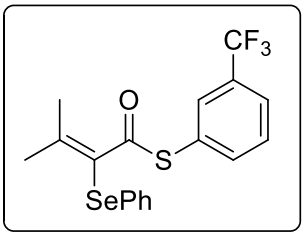
Following the general procedure, the reaction was performed with thioalkyne **s8** (56 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **65%** yield (61 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48 – 7.42 (m, 2H); 7.31 – 7.22 (m, 3H); 7.14 (d, *J* = 8.9 Hz, 2H); 6.88 (d, *J* = 8.9 Hz, 2H); 3.79 (s, 3H); 2.16 (s, 3H); 2.15 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.1; 160.4; 152.0; 136.0; 131.09; 131.07; 129.2; 126.9; 124.4; 120.2; 114.7; 55.3; 26.7; 23.6. **IR (neat) vmax**: 2908, 2835, 1662, 1591, 1573, 1492, 1476, 1437, 1285, 1122, 1022, 825, 732. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{18}NaO_2SSe$) requires *m*/*z* 401.0091, found: *m*/*z* 401.0092.

S-(2-methoxyphenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (9)



Following the general procedure, the reaction was performed with thioalkyne **s9** (56 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **60%** yield (57 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.49 – 7.44 (m, 2H); 7.39 – 7.33 (m, 1H); 7.30 – 7.18 (m, 4H); 6.95 – 6.89 (m, 2H); 3.75 (s, 3H); 2.18 (s, 3H); 2.17 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.4; 159.3; 152.2; 136.6; 131.3; 131.3; 130.9; 129.1; 126.8; 124.5; 120.9; 117.6; 111.4; 55.8; 26.6; 23.4. **IR (neat) vmax**: 2906; 2865; 1651; 1494; 1453; 1366; 1257; 1125; 1029; 890; 795. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{18}NaO_2SSe$) requires *m*/*z* 401.0091, found: *m*/*z* 401.0088.

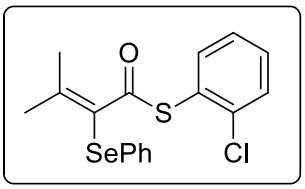
S-(3-(trifluoromethyl)phenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (10)



Following the general procedure, the reaction was performed with thioalkyne **s10** (71 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane

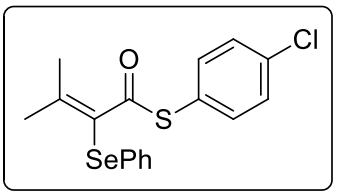
to hexane/ethyl acetate = 98:2) afforded the tittle compound in **78%** yield (81 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 7.7 Hz, 1H); 7.50 – 7.38 (m, 5H); 7.33 – 7.23 (m, 3H); 2.20 (s, 3H); 2.18 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.0; 153.6; 137.9 (q, *J* = 1.2 Hz); 131.5; 131.15 (q, *J* = 32.7 Hz); 131.14; 131.11; 130.8; 129.34; 129.3; 127.2; 125.8 (q, *J* = 3.7 Hz); 124.4; 123.6 (q, *J* = 272.7 Hz); 27.1; 23.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.6. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{15}F_3NaOSSe$) requires *m*/*z* 438.9859, found: *m*/*z* 438.9855.

S-(2-chlorophenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (11)



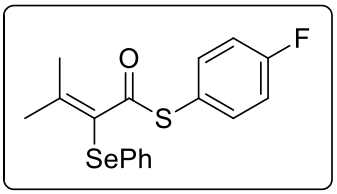
Following the general procedure, the reaction was performed with thioalkyne **s11** (57 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **90%** yield (86 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48 – 7.43 (m, 3H); 7.34 – 7.20 (m, 6H); 2.20 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 190.3; 154.2; 138.8; 137.1; 131.1; 131.0; 130.8; 130.0; 129.2; 129.2; 127.1; 126.9; 124.4; 27.2; 23.7. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{15}ClNaOSSe$) requires *m*/*z* 404.9595, found: *m*/*z* 404.9595.

S-(4-chlorophenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (12)



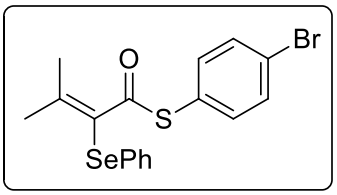
Following the general procedure, the reaction was performed with thioalkyne **s12** (57 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **81%** yield (77 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46 – 7.40 (m, 2H); 7.34 – 7.24 (m, 5H); 7.18 – 7.13 (m, 2H); 2.19 (s, 3H); 2.17 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.6; 153.5; 135.7; 135.4; 131.0; 130.96; 129.3; 129.2; 128.2; 127.0; 124.4; 27.0; 23.8. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{15}ClNaOSSe$) requires *m*/*z* 404.9595, found: *m*/*z* 404.9591.

S-(4-fluorophenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (13)



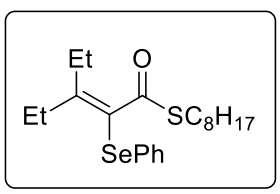
Following the general procedure, the reaction was performed with thioalkyne **s13** (53 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **81%** yield (74 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46 – 7.41 (m, 2H); 7.32 – 7.24 (m, 3H); 7.21 – 7.16 (m, 2H); 7.08 – 7.01 (m, 2H); 2.18 (s, 3H); 2.16 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.1 (d, *J* = 1.4 Hz); 163.3 (d, *J* = 249.6 Hz); 153.1; 136.5 (d, *J* = 8.5 Hz); 131.0; 130.98; 129.3; 127.0; 125.0 (d, *J* = 3.5 Hz); 124.4; 116.2 (d, *J* = 22.0 Hz); 26.9; 23.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -111.6. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{15}FNaOSSe$) requires *m*/*z* 388.9891, found: *m*/*z* 388.9890.

S-(4-bromophenyl) 3-methyl-2-(phenylselanyl)but-2-enethioate (14)



Following the general procedure, the reaction was performed with thioalkyne **s14** (68 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **60%** yield (64 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.4 Hz, 2H); 7.45 – 7.40 (m, 2H); 7.32 – 7.23 (m, 3H); 7.08 (d, *J* = 8.4 Hz, 2H); 2.19 (s, 3H); 2.17 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.5; 153.5; 136.0; 132.2; 130.98; 130.96; 129.3; 128.9; 127.0; 124.4; 123.7; 27.1; 23.8. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{15}FNaOSSe$) requires *m*/*z* 388.9891, found: *m*/*z* 388.9890.

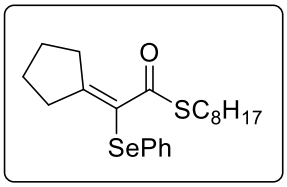
S-octyl 3-ethyl-2-(phenylselanyl)pent-2-enethioate (15)



Following the general procedure, the reaction was performed with thioalkyne **s10** (64 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **73%** yield (75 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42 – 7.37 (m, 2H); 7.27 – 7.16

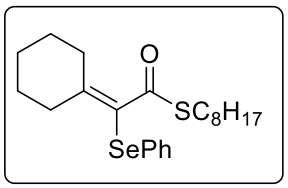
(m, 3H); 2.77 (t, *J* = 7.3 Hz, 2H); 2.51 (q, *J* = 7.6 Hz, 2H); 2.42 (q, *J* = 7.5 Hz, 2H); 1.48 – 1.37 (m, 2H); 1.35 – 1.20 (m, 10H); 1.14 (t, *J* = 7.5 Hz, 3H); 1.06 (t, *J* = 7.6 Hz, 3H); 0.88 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.0; 159.6; 131.1; 131.0; 129.0; 126.7; 124.2; 31.7; 30.2; 29.6; 29.2; 29.1; 29.0; 28.7; 27.4; 22.6; 14.1; 13.5; 12.9. **IR (neat) vmax**: 2960; 2924; 2853; 1651; 1577; 1476; 1461; 1131; 1053; 1022; 803; 763; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{21}H_{32}NaOSSe$) requires *m*/*z* 435.1237, found: *m*/*z* 435.1237.

S-octyl 2-cyclopentylidene-2-(phenylselanyl)ethanethioate (16)



Following the general procedure, the reaction was performed with thioalkyne **s11** (64 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **81%** yield (83 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36 – 7.30 (m, 2H); 7.26 – 7.14 (m, 3H); 2.91 (t, *J* = 7.2 Hz, 2H); 2.78 (t, *J* = 7.4 Hz, 2H); 2.61 (t, *J* = 7.2 Hz, 2H); 1.82 (qt, *J* = 7.0 Hz, 2H); 1.67 (qt, *J* = 7.0 Hz, 2H); 1.51 (qt, *J* = 7.2 Hz, 2H); 1.33 – 1.20 (m, 10H); 0.87 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.2; 172.1; 131.9; 129.5; 129.2; 126.3; 120.2; 39.1; 36.2; 31.7; 31.1; 29.2; 29.1; 28.9; 27.5; 25.1; 22.6; 14.1. **IR (neat) vmax**: 2922; 2852; 1645; 1575; 1476; 1156; 1084; 1021; 824; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{21}H_{30}NaOSSe$) requires *m*/*z* 433.1080, found: *m*/*z* 433.1080.

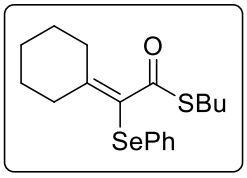
S-octyl 2-cyclohexylidene-2-(phenylselanyl)ethanethioate (17)



Following the general procedure, the reaction was performed with thioalkyne **s12** (67 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **81%** yield (86 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.45 – 7.40 (m, 2H); 7.27 – 7.18 (m, 3H); 2.80 (t, *J* = 7.3 Hz, 2H); 2.59 (t, *J* = 5.8 Hz, 2H); 2.50 (t, *J* = 5.8 Hz, 2H); 1.71 – 1.57 (m, 6H); 1.45 (qt, *J* = 6.6 Hz, 2H); 1.31 – 1.21 (m, 10H); 0.87 (t,

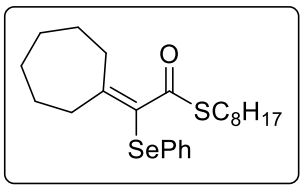
J = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.4; 155.8; 131.2; 131.1; 129.0; 126.7; 121.4; 35.6; 33.7; 31.7; 30.0; 29.3; 29.1; 29.0; 28.7; 28.4; 28.1; 26.2; 22.6; 14.1. **IR (neat) vmax**: 2923; 2852; 1662; 1577; 1476; 1438; 1115; 1058; 1021; 984; 908; 830; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{22}H_{32}NaOSSe$) requires *m*/*z* 447.1237, found: *m*/*z* 447.1244.

S-butyl 2-cyclohexylidene-2-(phenylselanyl)ethanethioate (18)



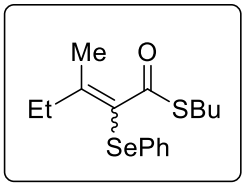
Following the general procedure, the reaction was performed with thioalkyne **s13** (53 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **73%** yield (67 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46 – 7.38 (m, 2H); 7.26 – 7.17 (m, 3H); 2.81 (t, *J* = 7.3 Hz, 2H); 2.62 – 2.57 (m, 2H); 2.53 – 2.47 (m, 2H); 1.69 – 1.59 (m, 6H); 1.48 – 1.40 (m, 2H); 1.36 – 1.24 (m, 2H); 0.86 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.5; 155.8; 131.23; 131.19; 129.1; 126.8; 121.5; 35.7; 33.7; 31.4; 29.7; 28.4; 28.1; 26.2; 21.9; 13.6. **IR (neat) vmax**: 2927; 2854; 1661; 1577; 1476; 1438; 1114; 1056; 1021; 983; 830; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{24}NaOSSe$) requires *m*/*z* 391.0611, found: *m*/*z* 391.0605.

S-octyl 2-cycloheptylidene-2-(phenylselanyl)ethanethioate (19)



Following the general procedure, the reaction was performed with thioalkyne **s14** (71 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **70%** yield (77 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.43 – 7.39 (m, 2H); 7.26 – 7.17 (m, 3H); 2.77 (t, *J* = 7.4 Hz, 2H); 2.68 – 2.64 (m, 2H); 2.62 – 2.58 (m, 2H); 1.75 – 1.68 (m, 2H); 1.68 – 1.61 (m, 2H); 1.60 – 1.51 (m, 4H); 1.46 – 1.37 (m, 2H); 1.31 – 1.19 (m, 10H); 0.87 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.1; 157.4; 131.3; 131.0; 129.0; 126.8; 124.8; 37.1; 34.4; 31.7; 30.2; 29.3; 29.1; 29.06; 29.0; 28.75; 28.73; 27.9; 26.8; 22.6; 14.1. **IR (neat) vmax**: 2921; 2851; 1649; 1476; 1438; 1079; 790; 732; 688. **HRMS (ESI+):** exact mass

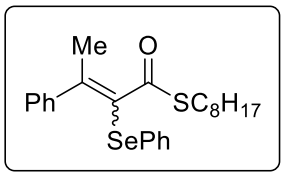
calculated for $[M+Na]^+$ ($C_{23}H_{34}NaOSSe$) requires *m*/*z* 461.1393, found: *m*/*z* 461.1384.



S-butyl 3-methyl-2-(phenylselanyl)pent-2-enethioate (21)

Following the general procedure, the reaction was performed with thioalkyne **s16** (47 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **89%** yield (76 mg) as a yellow oil. The ratio between the isomers is *1:1.25*. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42 – 7.37 (m, 4H); 7.26 – 7.17 (m, 6H); 2.83 – 2.75 (m, 4H); 2.53 (q, *J* = 7.6 Hz, 2H); 2.42 (q, *J* = 7.5 Hz, 2H); 2.09 (s, 3H); 2.08 (s, 3H); 1.48 – 1.38 (m, 4H); 1.34 – 1.24 (m, 4H); 1.14 (t, *J* = 7.5 Hz, 3H); 1.05 (t, *J* = 7.6 Hz, 3H); 0.88 – 0.82 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.4; 194.0; 155.0; 154.7; 131.3; 130.94, 130.93, 129.1; 129.06; 126.7; 124.5; 124.2; 32.9; 31.33; 31.32; 30.0; 29.93; 29.89; 23.2; 21.9; 21.8; 20.8; 13.6; 13.2; 12.4. **IR (neat) vmax**: 2959; 2929; 2871; 1659; 1577; 1476; 1461; 1437; 1128; 1046; 1021; 998; 864; 793; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{16}H_{22}NaOSSe$) requires *m*/*z* 365.0454, found: *m*/*z* 365.0448.

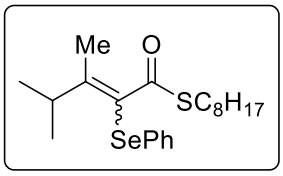
S-octyl 3-phenyl-2-(phenylselanyl)but-2-enethioate (22)



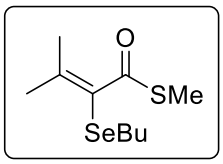
Following the general procedure, the reaction was performed with thioalkyne **s17** (73 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **45%** yield (50 mg) as a yellow oil. The ratio between the isomers is *1:1*. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.58 – 7.51 (m, 4H); 7.42 – 7.17 (m, 16H); 2.78 (t, *J* = 7.2 Hz, 2H); 2.55 (t, *J* = 7.1 Hz, 2H); 2.41 (s, 3H); 2.31 (s, 3H); 1.45 – 1.35 (m, 4H); 1.31 – 1.06 (m, 20H); 0.91 – 0.85 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.21, 194.12, 148.21, 147.50, 142.86, 141.56, 132.82, 132.59, 130.39, 129.75, 129.11, 128.91, 128.23, 128.11, 127.97, 127.90, 127.60, 127.41, 127.35, 127.16, 31.79, 30.03, 29.83, 29.25, 29.11, 29.07, 29.01, 28.91, 28.74, 28.51, 25.62, 23.93, 22.64, 22.63, 14.10. **IR (neat) vmax**: 2922; 2852; 1717; 1662;

1576; 1438; 1325; 1128; 1056; 1021; 757; 734; 688; 612. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{24}H_{30}NaOSSe$) requires *m*/*z* 469.1081, found: *m*/*z* 469.1080.

S-octyl 3,4-dimethyl-2-(phenylselanyl)pent-2-enethioate (23)

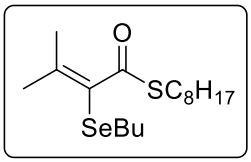


Following the general procedure, the reaction was performed with thioalkyne **s18** (64 mg, 0.25 mmol) and 1,2-diphenyldiselane (156 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **77%** yield (79 mg) as a yellow oil. The ratio between the isomers is *1:1*. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.43 – 7.38 (m, 4H); 7.27 – 7.17 (m, 6H); 3.43 (hept, *J* = 6.8 Hz, 1H); 3.09 (hept, *J* = 6.8 Hz, 1H); 2.82 – 2.75 (m, 4H); 1.95 (s, 6H); 1.43 (qt, *J* = 7.3 Hz, 4H); 1.32 – 1.20 (m, 20H); 1.08 (d, *J* = 6.8 Hz, 6H); 1.03 (d, *J* = 6.8 Hz, 6H); 0.87 (t, *J* = 6.9 Hz, 6H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.5; 194.2; 156.8; 155.9; 131.21; 131.19; 131.16, 130.7; 129.06; 129.05; 126.8; 123.6; 123.5; 35.8; 33.7; 31.76; 31.75; 30.1; 29.9; 29.3; 29.25; 29.12; 29.1; 29.06; 29.04; 28.7; 22.6; 21.0; 20.4; 17.0; 15.8; 14.1. **IR (neat) vmax**: 2958; 2923; 2853; 1653; 1476; 1438; 1120; 1063; 1022; 874; 833; 732; 688. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{21}H_{32}NaOSSe$) requires *m*/*z* 435.1237, found: *m*/*z* 435.1236.



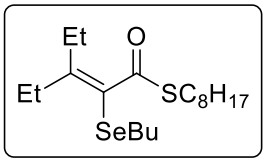
S-methyl 2-(butylselanyl)-3-methylbut-2-enethioate (24)

Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-dibutyldiselane (136 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **72%** yield (48 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.69 (t, *J* = 7.5 Hz, 2H); 2.35 (s, 3H); 2.08 (s, 3H); 2.00 (s, 3H); 1.61 (qt, *J* = 7.5 Hz, 2H); 1.39 (sext, *J* = 7.5 Hz, 2H); 0.90 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.2; 147.0; 123.7; 32.1; 28.3; 25.8; 22.9; 22.8; 13.5; 13.0. **IR (neat) vmax**: 2958; 2926; 2854; 1654; 1460; 1260; 1127; 1032; 889; 798; 722. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{10}H_{18}NaOSSe$) requires *m*/*z* 289.0141, found: *m*/*z* 289.0138.



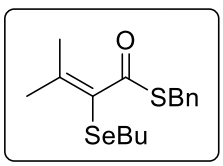
S-octyl 2-(butylselanyl)-3-methylbut-2-enethioate (25)

Following the general procedure, the reaction was performed with thioalkyne **s3** (57 mg, 0.25 mmol) and 1,2-dibutyldiselane (136 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **71%** yield (65 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.91 (t, *J* = 7.4 Hz, 2H); 2.69 (t, *J* = 7.4 Hz, 2H); 2.06 (s, 3H); 1.98 (s, 3H); 1.66 – 1.57 (m, 4H); 1.44 – 1.34 (m, 4H); 1.33 – 1.22 (m, 8H); 0.94 – 0.85 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.9; 145.8; 123.8; 32.2; 31.8; 30.1; 29.5; 29.14; 29.1; 28.9; 28.1; 25.5; 22.85; 22.79; 22.6; 14.1; 13.5. **IR (neat) vmax**: 2956; 2954; 2824; 1656; 1461; 1367; 1127; 1032; 889; 798; 722. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{17}H_{32}NaOSSe$) requires *m*/*z* 387.1237, found: *m*/*z* 387.1235.



S-octyl 2-(butylselanyl)-3-ethylpent-2-enethioate (26)

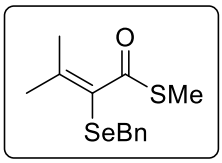
Following the general procedure, the reaction was performed with thioalkyne **s10** (64 mg, 0.25 mmol) and 1,2-dibutyldiselane (136 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **61%** yield (60 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.92 (t, *J* = 7.5 Hz, 2H); 2.68 (t, *J* = 7.5 Hz, 2H); 2.43 (q, *J* = 7.5 Hz, 2H); 2.29 (q, *J* = 7.5 Hz, 2H); 1.67 – 1.56 (m, 4H); 1.46 – 1.22 (m, 12H); 1.10 – 1.00 (m, 6H); 0.94 – 0.85 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.6; 155.4; 123.2; 32.1; 31.8; 30.0; 29.5; 29.2; 29.1; 28.9; 28.6; 27.9; 27.1; 22.8; 22.6; 14.1; 13.55; 13.49; 12.9. **IR (neat) vmax**: 2959; 2924; 2854; 1656; 1461; 1373; 1259; 1132; 1080; 1055; 803; 762; 734. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{19}H_{36}NaOSSe$) requires *m*/*z* 415.1550, found: *m*/*z* 415.1541.



***S-benzyl 2-(butylselanyl)-3-methylbut-2-enethioate* (27)**

Following the general procedure, the reaction was performed with thioalkyne **s5** (52 mg, 0.25 mmol) and 1,2-dibutyldiselane (136 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **78%** yield (67 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.34 – 7.30 (m, 2H);

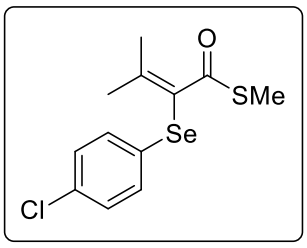
7.30 – 7.26 (m, 2H); 7.24 – 7.20 (m, 1H); 4.15 (s, 2H); 2.64 (t, *J* = 7.5 Hz, 2H); 2.07 (s, 3H); 1.99 (s, 3H); 1.64 – 1.54 (m, 2H); 1.39 – 1.30 (m, 2H); 0.86 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.9; 147.6; 137.8; 128.8; 128.5; 127.0; 123.4; 34.7; 32.1; 28.3; 25.9; 22.9; 22.8; 13.5. **IR (neat) vmax**: 2928; 1651; 1494; 1453; 1366; 1257; 1125; 1029; 890; 795; 697. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{16}H_{22}NaOSSe$) requires *m*/*z* 365.0454, found: *m*/*z* 365.0447.



S-methyl 2-(benzylselanyl)-3-methylbut-2-enethioate (28)

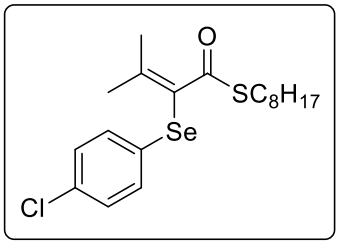
Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-dibenzyldiselane (170 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **74%** yield (55 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35 – 7.11 (m, 5H); 3.90 (s, 2H); 2.37 (s, 3H); 1.99 (s, 3H); 1.72 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.2; 151.0; 138.3; 129.0; 128.2; 126.7; 123.1; 32.4; 25.7; 22.9; 13.2. **IR (neat) vmax**: 2923; 1737; 1650; 1493; 1452; 1366; 1307; 1238; 1127; 1033; 959; 783; 757; 695. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{16}NaOSSe$) requires *m*/*z* 322.9985, found: *m*/*z* 322.9982.

S-methyl 2-((4-chlorophenyl)selanyl)-3-methylbut-2-enethioate (29)



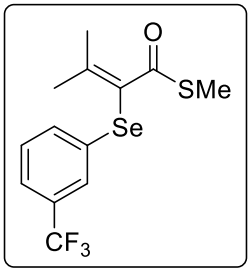
Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-bis(4-chlorophenyl)diselane (191 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **78%** yield (62 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.7 Hz, 2H); 7.21 (d, *J* = 8.7 Hz, 2H); 2.24 (s, 3H); 2.16 (s, 3H); 2.15 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.4; 152.4; 132.9; 131.8; 129.5; 129.3; 124.4; 26.7; 23.5; 13.4. **IR (neat) vmax**: 2912; 1650; 1589; 1470; 1422; 1386; 1364; 1128; 1085; 1029; 882; 810; 725. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{13}ClNaOSSe$) requires *m*/*z* 342.9439, found: *m*/*z* 342.9429.

S-octyl 2-((4-chlorophenyl)selanyl)-3-methylbut-2-enethioate (30)



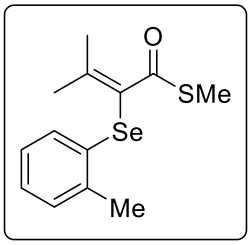
Following the general procedure, the reaction was performed with thioalkyne **s3** (57 mg, 0.25 mmol) and 1,2-bis(4-chlorophenyl)diselane (191 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **70%** yield (73 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.7 Hz, 2H); 7.20 (d, *J* = 8.7 Hz, 2H); 2.80 (t, *J* = 7.4 Hz, 2H); 2.12 (s, 3H); 2.11 (s, 3H); 1.49 – 1.41 (m, 2H); 1.32 – 1.20 (m, 10H); 0.87 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.1; 150.5; 133.0; 132.3; 129.3; 129.2; 124.7; 31.8; 30.3; 29.3; 29.1; 29.06; 28.8; 26.3; 23.3; 22.6; 14.1. **IR (neat) vmax**: 2923; 2852; 1650; 1473; 1127; 1089; 1030; 1010; 799; 728. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{19}H_{27}ClNaOSSe$) requires *m*/*z* 441.0534, found: *m*/*z* 441.0530.

S-methyl 3-methyl-2-((3-(trifluoromethyl)phenyl)selanyl)but-2-enethioate (31)

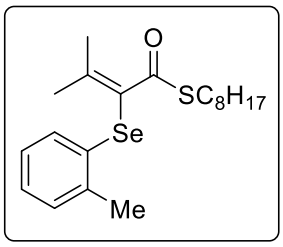


Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-bis(3-(trifluoromethyl)phenyl)diselane (224 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **75%** yield (66 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.63 (s, 1H); 7.53 (d, *J* = 7.8 Hz, 1H); 7.45 (d, *J* = 7.8 Hz, 1H); 7.36 (t, *J* = 7.8 Hz, 1H); 2.25 (s, 3H); 2.18 (s, 3H); 2.16 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.3; 153.3; 133.4 (q, *J* = 1.1 Hz); 132.6; 131.4 (q, *J* = 32.4 Hz); 129.5; 126.9 (q, *J* = 3.9 Hz); 123.8; 123.7 (q, *J* = 272.7 Hz); 123.5 (q, *J* = 3.8 Hz); 26.7; 23.5; 13.3. **^{19}F NMR** (378 MHz, $CDCl_3$): δ – 62.8. **IR (neat) vmax**: 2926; 1652; 1422; 1319; 1272; 1122; 1087; 1034; 889; 792; 692. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{13}F_3NaOSSe$) requires *m*/*z* 376.9702, found: *m*/*z* 376.9697.

S-methyl 3-methyl-2-(o-tolylselanyl)but-2-enethioate (32)



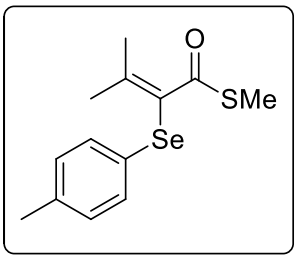
Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-di-*o*-tolyldiselane (170 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **70%** yield (52 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.26 – 7.21 (m, 1H); 7.16 – 7.03 (m, 3H); 2.38 (s, 3H); 2.21 (s, 3H); 2.18 (s, 3H); 2.14 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.6; 152.7; 137.7; 131.9; 130.1; 129.6; 126.7; 126.6; 123.9; 26.7; 23.5; 21.6; 13.4. **IR (neat) vmax**: 3048; 2916; 1640; 1563; 1452; 1355; 1122; 1030; 881; 798; 746; 684. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{16}NaOSSe$) requires *m*/*z* 322.9985, found: *m*/*z* 322.9991.



S-octyl 3-methyl-2-(o-tolylselanyl)but-2-enethioate (33)

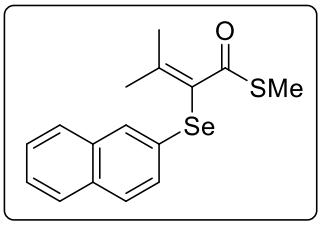
Following the general procedure, the reaction was performed with thioalkyne **s3** (57 mg, 0.25 mmol) and 1,2-di-*o*-tolyldiselane (170 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **71%** yield (71 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.28 – 7.26 (m, 1H); 7.16 – 7.03 (m, 3H); 2.77 (t, *J* = 7.4 Hz, 2H); 2.38 (s, 3H); 2.13 (s, 3H); 2.12 (s, 3H); 1.46 – 1.39 (m, 2H); 1.32 – 1.20 (m, 10H); 0.87 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.3; 150.7; 138.1; 131.7; 130.4; 130.1; 126.7; 126.6; 124.3; 31.8; 30.3; 29.3; 29.1; 29.07; 28.8; 26.3; 23.4; 22.6; 21.8; 14.1. **IR (neat) vmax**: 2922; 2852; 1651; 1456; 1127; 1031; 799; 743. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{20}H_{30}NaOSSe$) requires *m*/*z* 421.1080, found: *m*/*z* 421.1089.

S-methyl 3-methyl-2-(p-tolylselanyl)but-2-enethioate (34)



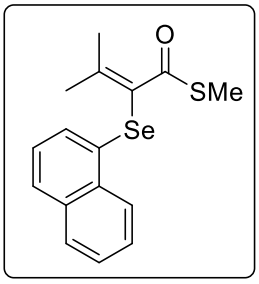
Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-di-*p*-tolyldiselane (170 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **88%** yield (66 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 8.1 Hz, 2H); 7.05 (d, *J* = 8.1 Hz, 2H); 2.29 (s, 3H); 2.22 (s, 3H); 2.14 (s, 3H); 2.13 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.7; 151.2; 136.7; 130.9; 129.9; 127.3; 124.9; 26.6; 23.4; 21.0; 13.4. **IR (neat) vmax**: 2971; 1649; 1429; 1366; 1127; 1033; 1014; 888; 797; 732. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{16}NaOSSe$) requires *m*/*z* 322.9985, found: *m*/*z* 322.9977.

S-methyl 3-methyl-2-(naphthalen-2-ylselanyl)but-2-enethioate (35)



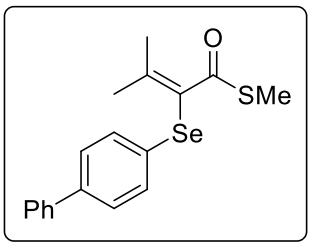
Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-di(naphthalen-2-yl)diselane (206 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **68%** yield (57 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 – 7.82 (m, 1H); 7.79 – 7.69 (m, 3H); 7.49 – 7.40 (m, 3H); 2.22 (s, 3H); 2.20 (s, 3H); 2.19 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.7; 152.6; 140.3; 139.6; 133.3; 130.6; 130.4; 128.8; 128.7; 127.8; 127.3; 126.8; 124.5; 26.9; 23.5; 13.5. **IR (neat) vmax**: 2900; 1644; 1582; 1128; 1035; 938; 889; 858; 804; 764; 740; 679. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{16}H_{16}NaOSSe$) requires *m*/*z* 358.9985, found: *m*/*z* 358.9983.

S-methyl 3-methyl-2-(naphthalen-1-ylselanyl)but-2-enethioate (36)

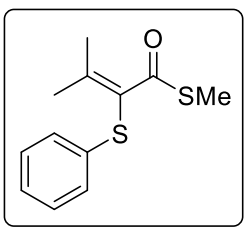


Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-di(naphthalen-1-yl)diselane (206 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **65%** yield (55 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.16 (d, *J* = 8.3 Hz, 1H); 7.83 – 7.78 (m, 1H); 7.70 (d, *J* = 8.2 Hz, 1H); 7.55 – 7.45 (m, 3H); 7.32 (t, *J* = 8.2 Hz, 1H); 2.19 (s, 6H); 2.15 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.6; 152.6; 133.9; 132.8; 130.2; 128.5; 127.4; 126.4; 126.1; 126.06; 126.0; 124.1; 26.8; 23.6; 13.4. **IR (neat) vmax**: 3048; 2918; 1646; 1499; 1374; 1250; 1197; 1133; 1021; 954; 788; 764; 648. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{16}H_{16}NaOSSe$) requires *m*/*z* 358.9985, found: *m*/*z* 358.9982.

S-methyl 2-([1,1'-biphenyl]-4-ylselanyl)-3-methylbut-2-enethioate (37)



Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-di([1,1'-biphenyl]-4-yl)diselane (232 mg, 0.5 mmol). Purification by flash column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **62%** yield (56 mg) as a yellow solid. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.57 – 7.53 (m, 2H); 7.50 – 7.39 (m, 6H); 7.35 – 7.30 (m, 1H); 2.25 (s, 3H); 2.18 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.7; 152.6; 140.3; 139.6; 133.3; 130.6; 128.7; 127.8; 127.3; 126.8; 124.5; 26.9; 23.5; 13.5. **IR (neat) vmax**: 2913; 1644; 1575; 1474; 1125; 1072; 1027; 1004; 889; 825; 795; 760; 713; 697; 680. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{18}H_{18}NaOSSe$) requires *m*/*z* 385.0141, found: *m*/*z* 385.0138.

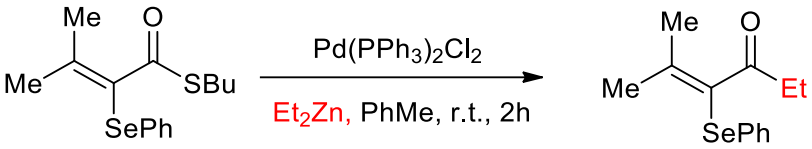


S-methyl 3-methyl-2-(phenylthio)but-2-enethioate (38)

Following the general procedure, the reaction was performed with thioalkyne **s1** (33 mg, 0.25 mmol) and 1,2-diphenyldisulfane (109 mg, 0.5 mmol). Purification by flash

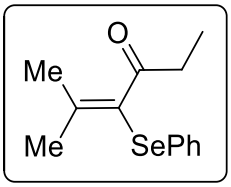
column chromatography (gradient from hexane to hexane/ethyl acetate = 98:2) afforded the tittle compound in **80%** yield (48 mg) as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.28 – 7.23 (m, 2H); 7.20 – 7.10 (m, 3H); 2.26 (s, 3H); 2.20 (s, 3H); 2.18 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.9; 156.7; 136.2; 129.0; 126.8; 126.2; 125.7; 25.7; 23.7; 13.3. **IR (neat) vmax**: 2923; 1653; 1580; 1477; 1438; 1129; 1045; 1023; 914; 804; 736; 697; 594. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{12}H_{14}NaOS_2$) requires *m/z* 261.0384, found: *m/z* 261.0382.

4. General Procedure for the Ketone Synthesis



2 **39**

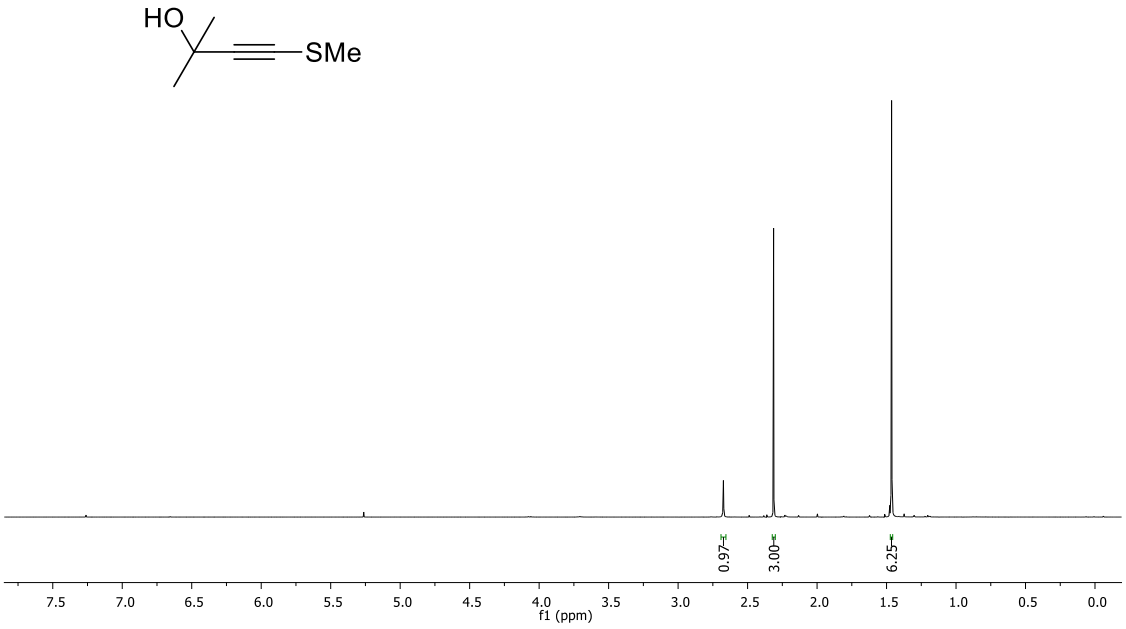
To a solution of the thiol ester **2** (0.5 mmol) and $PdCl_2(PPh_3)_2$ (10 mol%l) in toluene (2 mL) was added Et_2Zn (1.5 mmol). The reaction mixture was stirred for 2h at r.t., then quenched with 1 M HCl (10 mL). The mixture was partitioned and the aqueous layer was extracted twice with EtOAc. The combined organic extracts were washed with sat. $NaHCO_3$ and brine, dried over $MgSO_4$, and concentrated. Purification by flash column chromatography using gradient from hexane to hexane/ethyl acetate = 98:2.



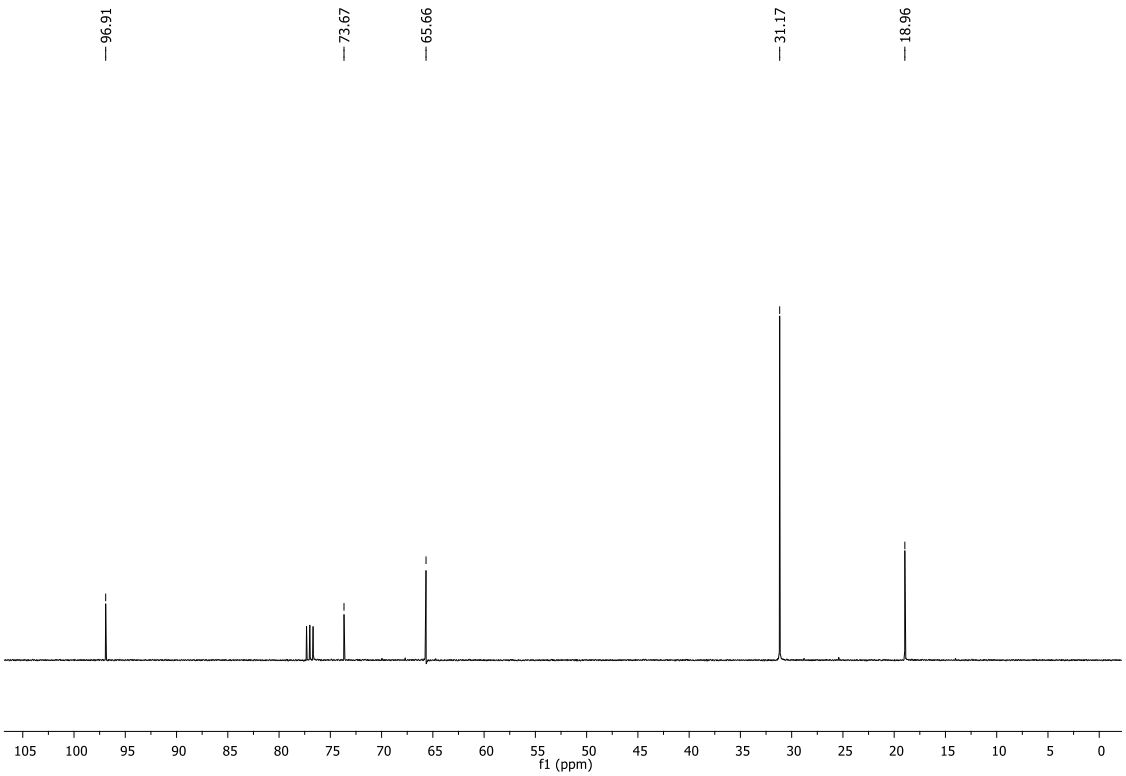
5-methyl-4-(phenylselanyl)hex-4-en-3-one (39)

The compound **39** was obtained as a yellow oil (76 mg, **57%**) **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.35 (m, 2H); 7.28-1.19 (m, 3H); 2.67 (q, *J* = 6.8 Hz, 2H); 2.12 (s, 3H); 2.01 (s, 3H); 0.97 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 205.1; 146.7; 130.7; 130.65; 129.3; 126.7; 126.3; 34.9; 25.1; 22.7; 8.2. **HRMS (ESI+):** exact mass calculated for $[M+Na]^+$ ($C_{13}H_{16}NaOSe$) requires *m/z* 291.0264, found: *m/z* 291.0260.

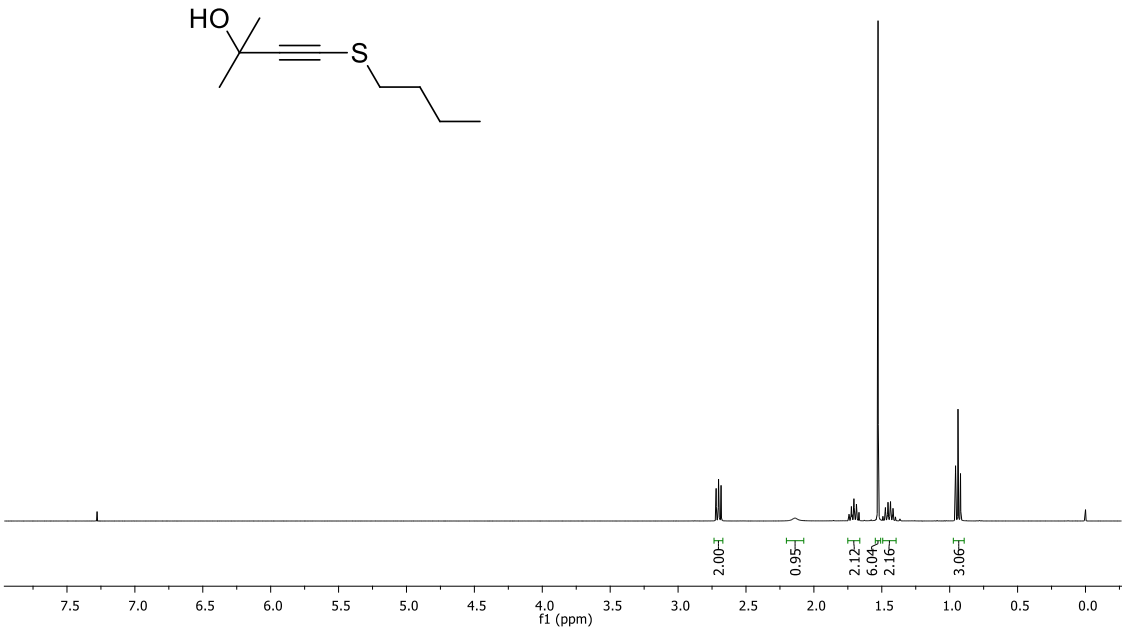
5. NMR Spectra



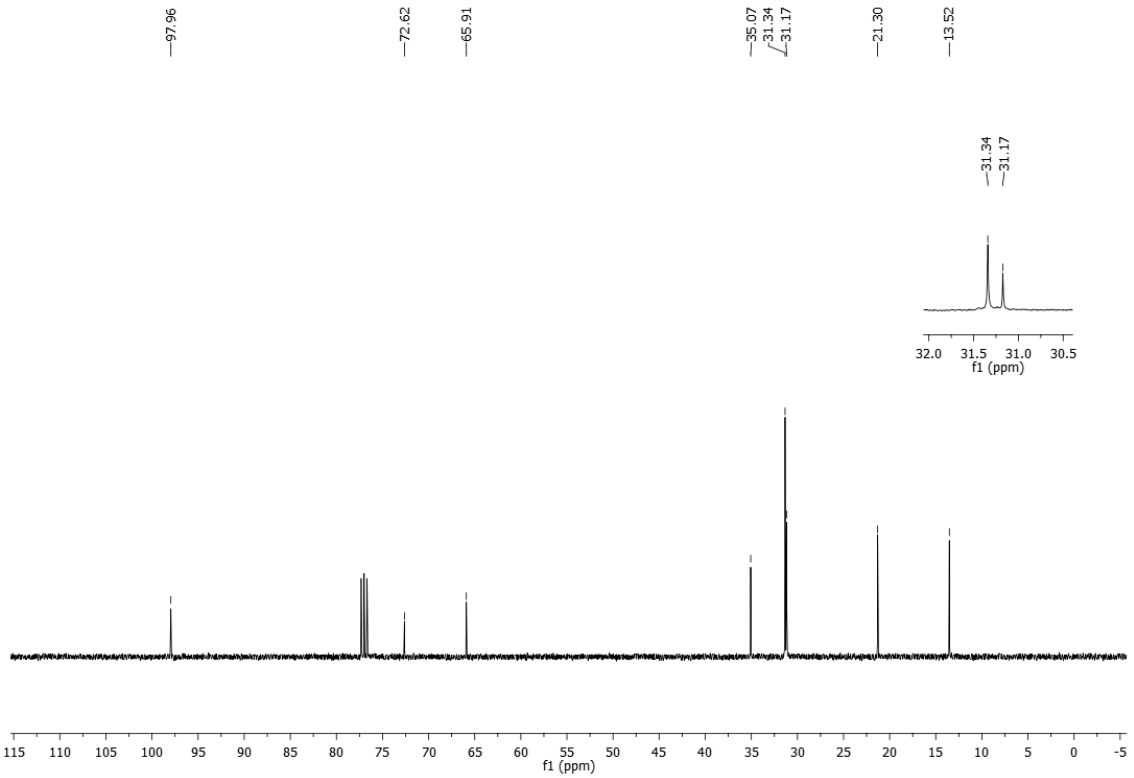
^{1}H NMR spectrum for compound **s1** ($CDCl_3$, 400 MHz)



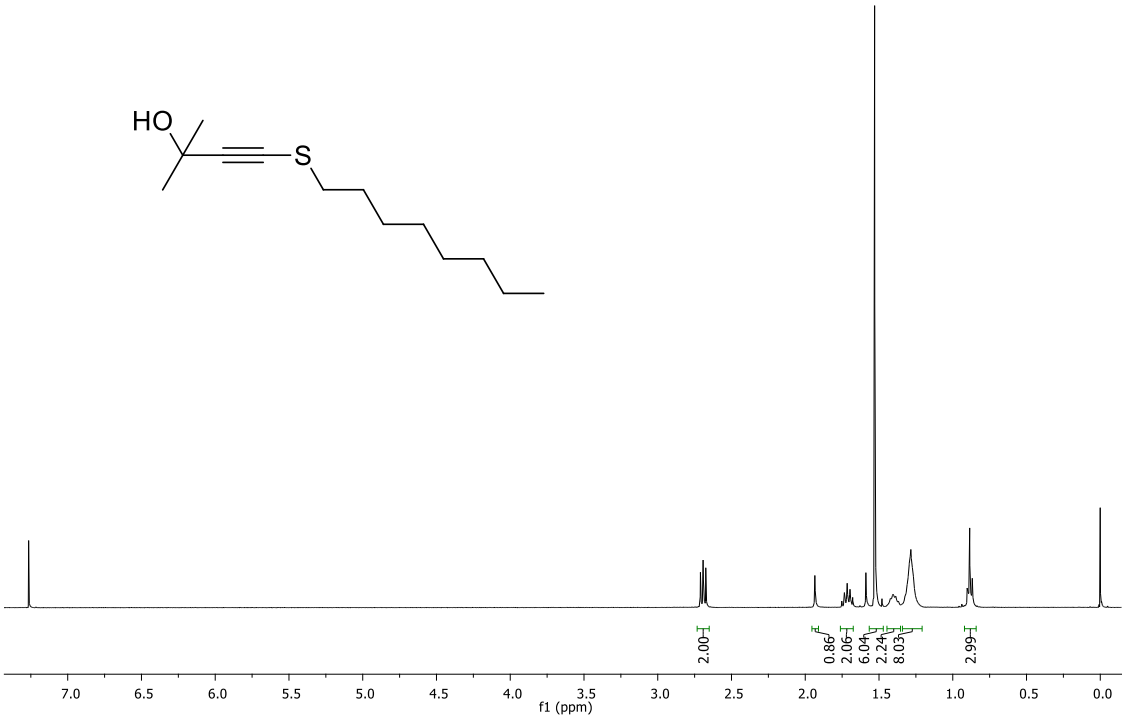
^{13}C NMR spectrum for compound **s1** ($CDCl_3$, 100 MHz)



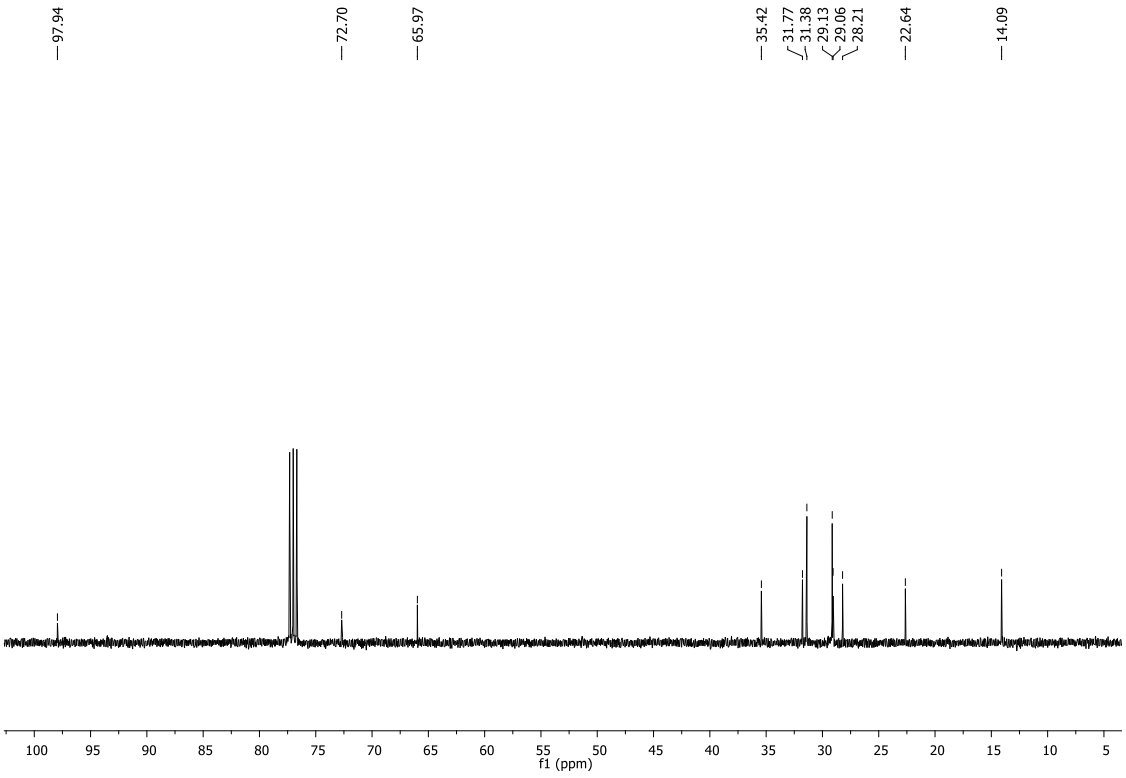
^{1}H NMR spectrum for compound **s2** ($CDCl_3$, 400 MHz)



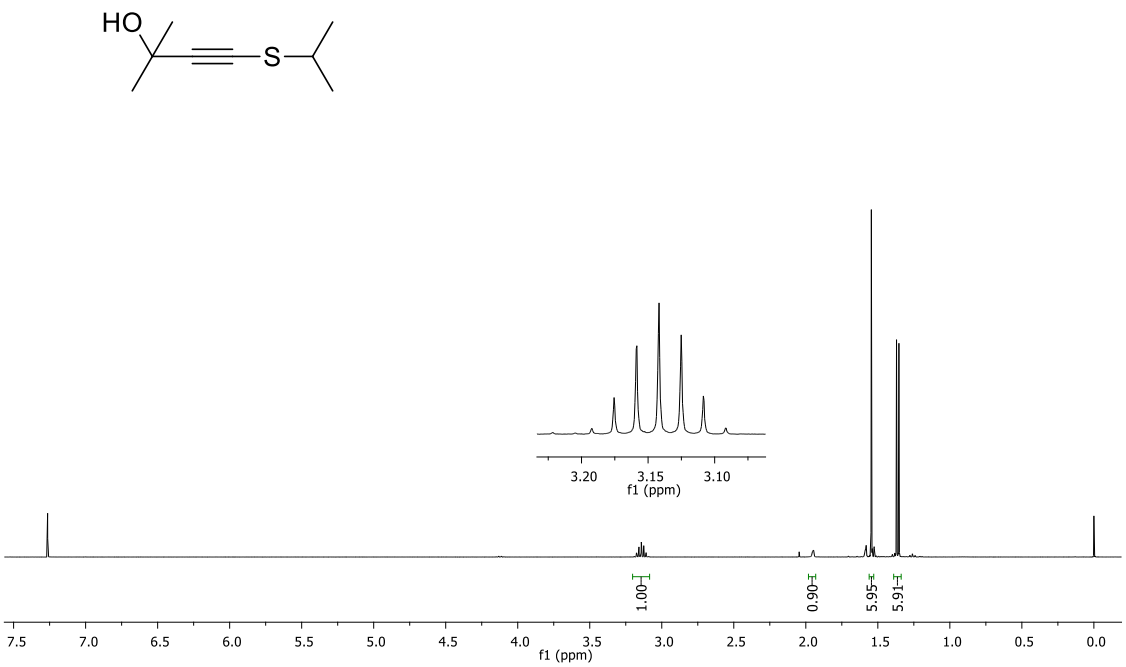
^{13}C NMR spectrum for compound **s2** ($CDCl_3$, 100 MHz)



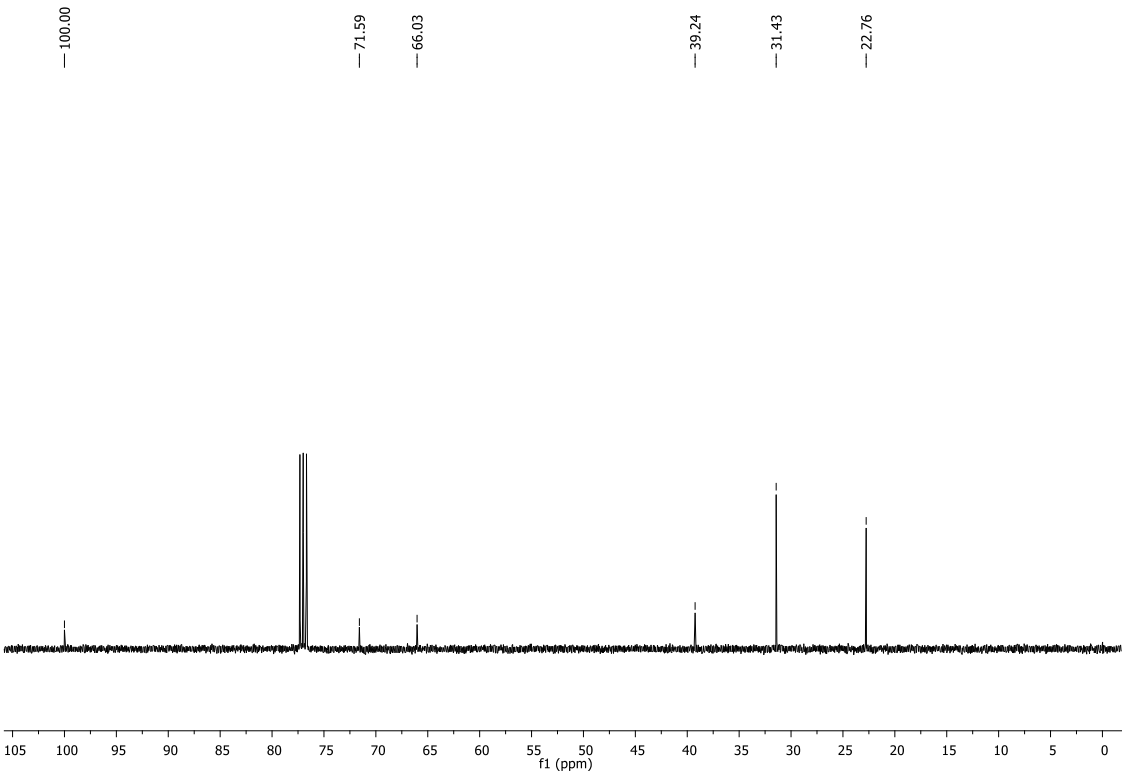
^{1}H NMR spectrum for compound **s3** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **s3** ($CDCl_3$, 100 MHz)

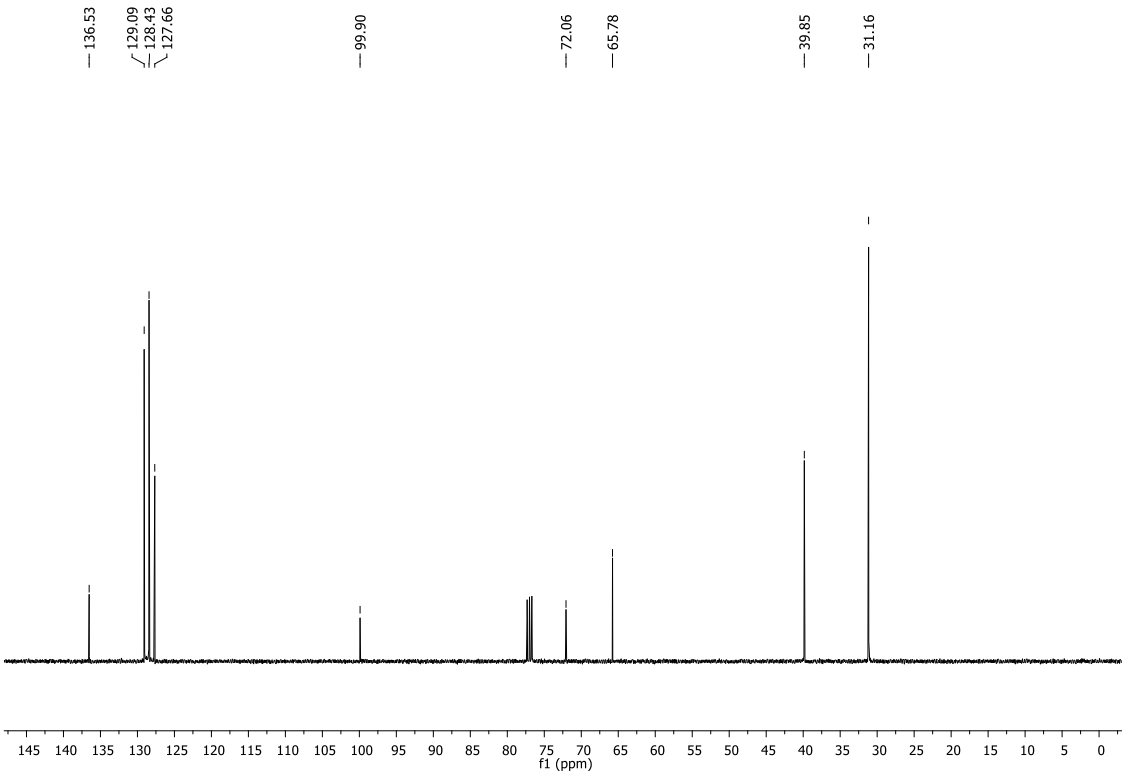


^{1}H NMR spectrum for compound **s4** ($CDCl_3$, 400 MHz)



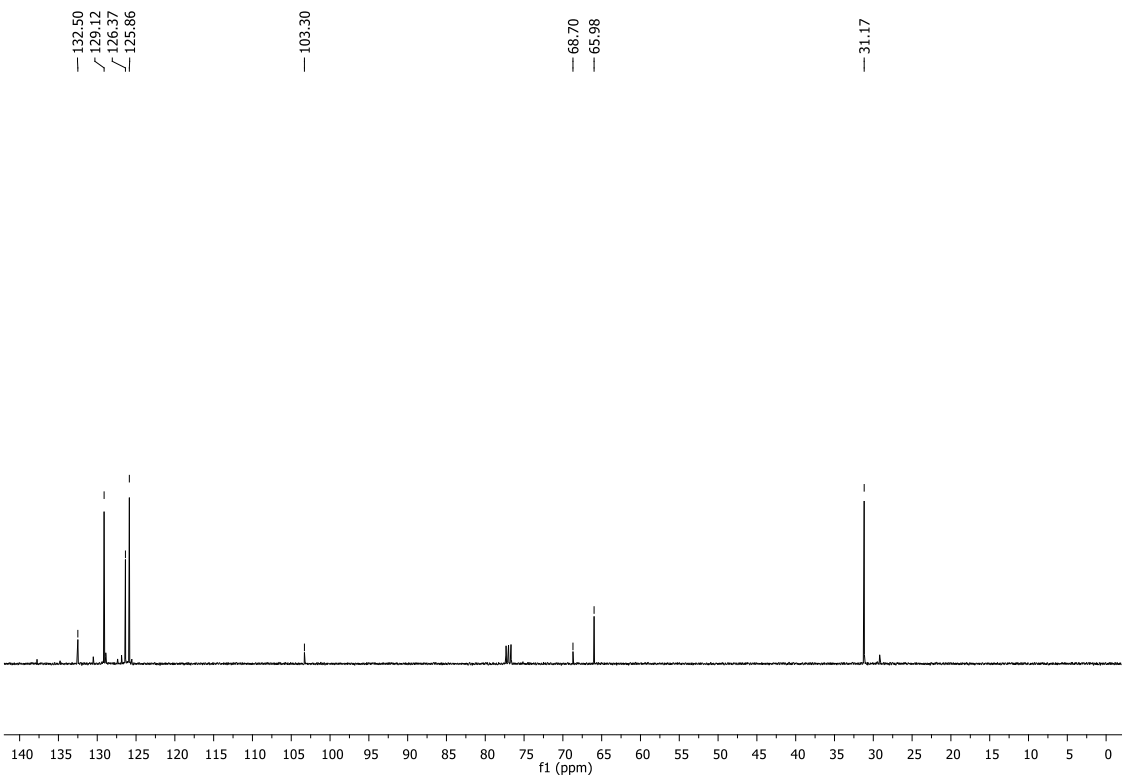
^{13}C NMR spectrum for compound **s4** ($CDCl_3$, 100 MHz)

^{1}H NMR spectrum for compound **s5** ($CDCl_3$, 400 MHz)

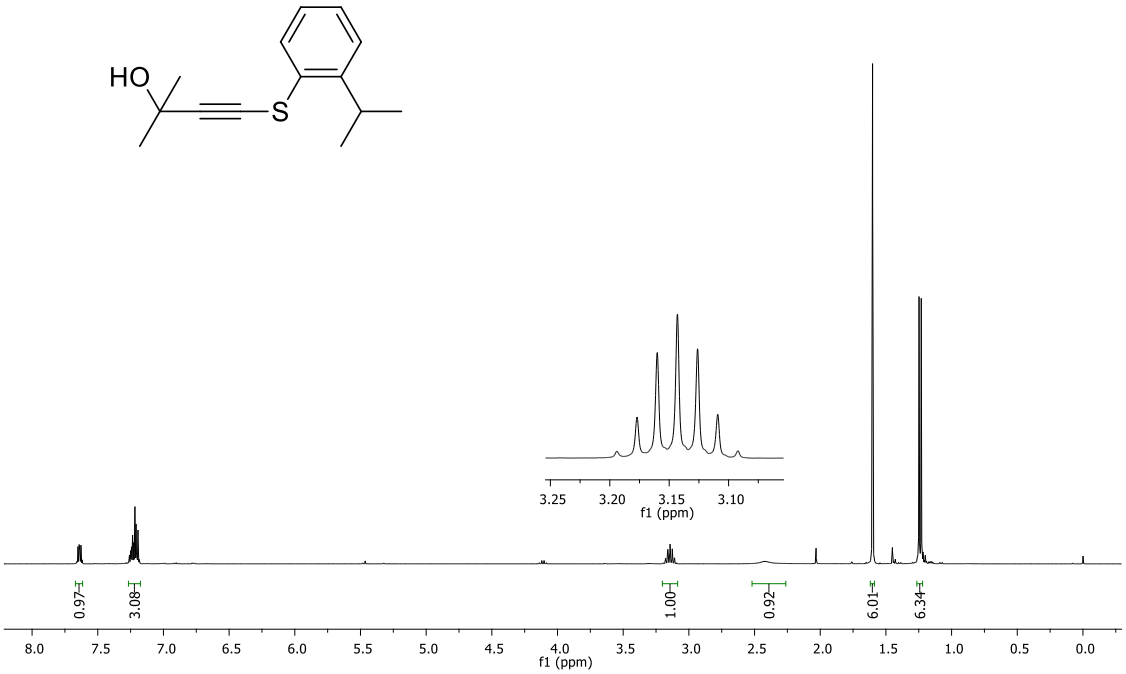


^{13}C NMR spectrum for compound **s5** ($CDCl_3$, 100 MHz)

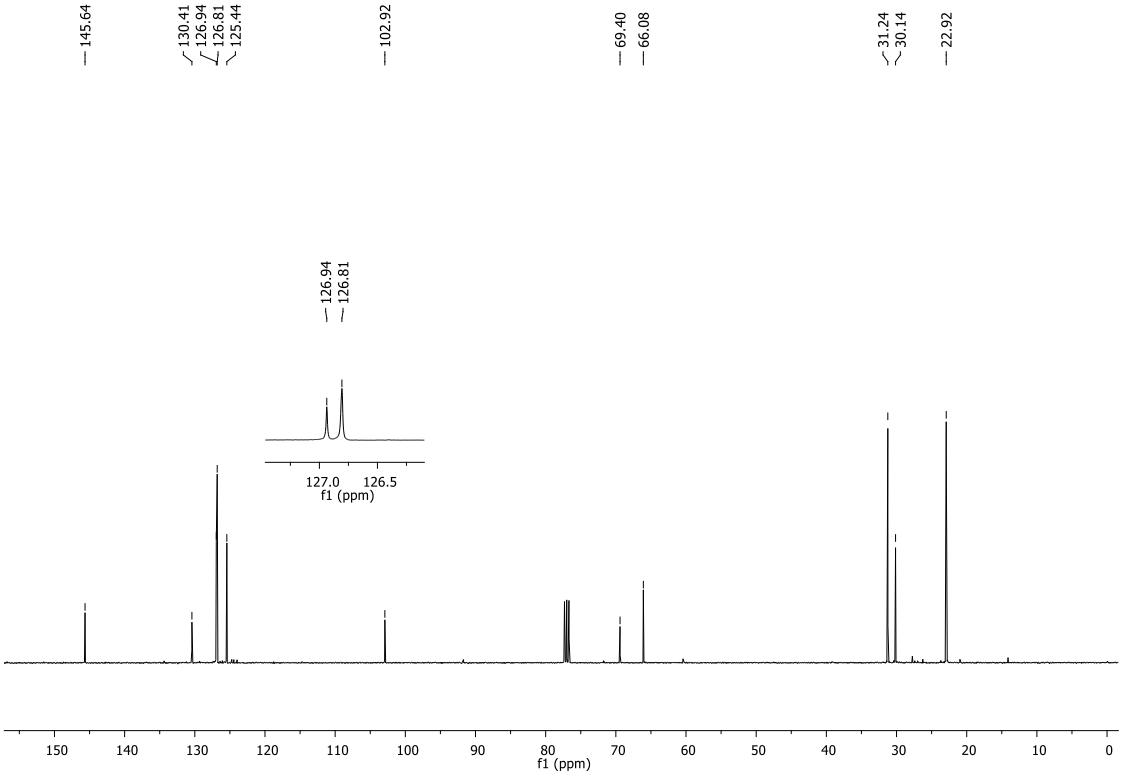
^{1}H NMR spectrum for compound **s6** ($CDCl_3$, 400 MHz)



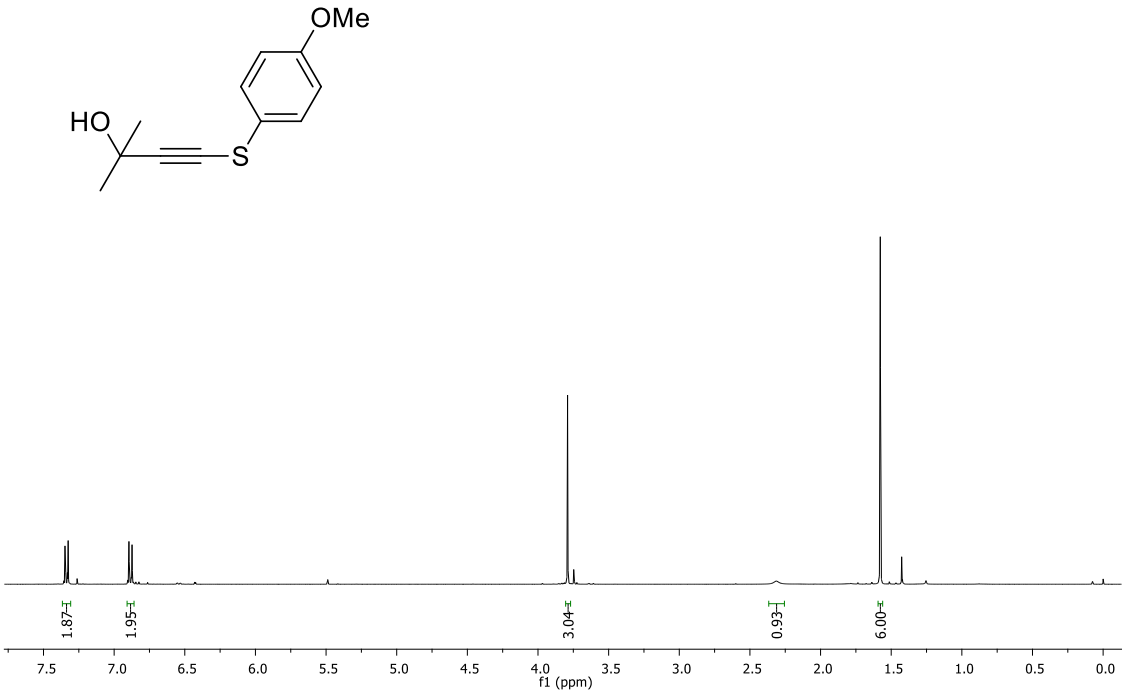
^{13}C NMR spectrum for compound **s6** ($CDCl_3$, 100 MHz)



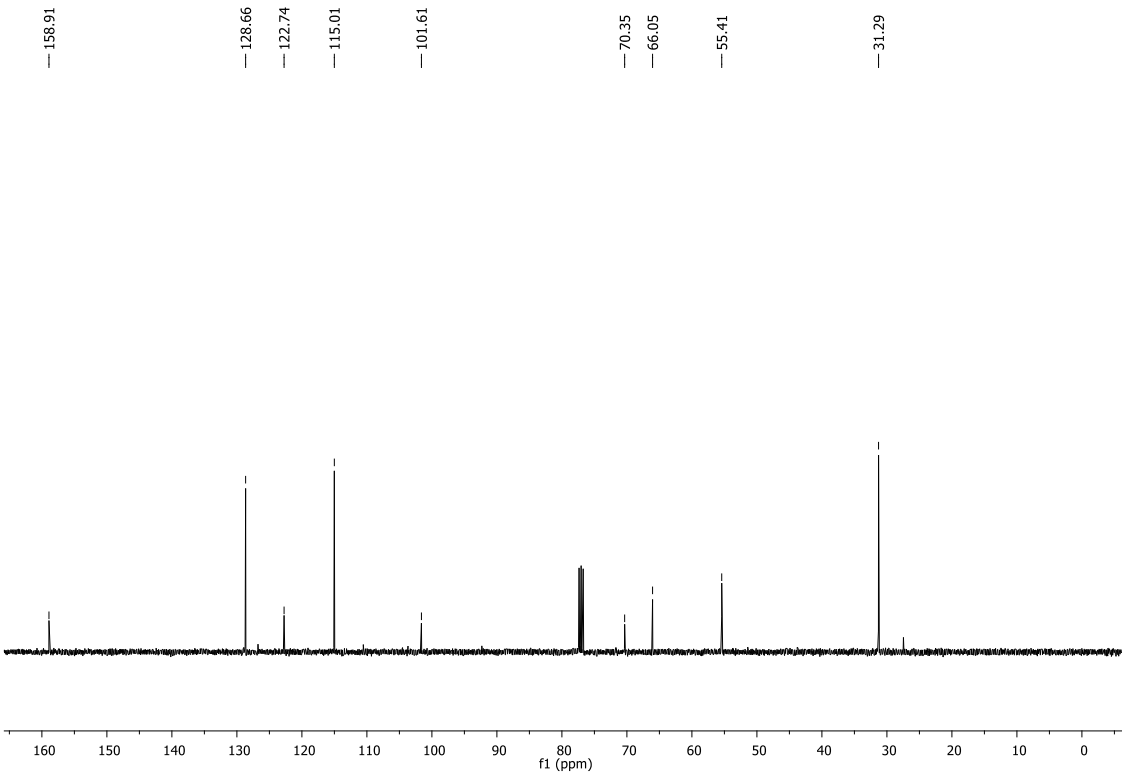
^{1}H NMR spectrum for compound **s7** ($CDCl_3$, 400 MHz)



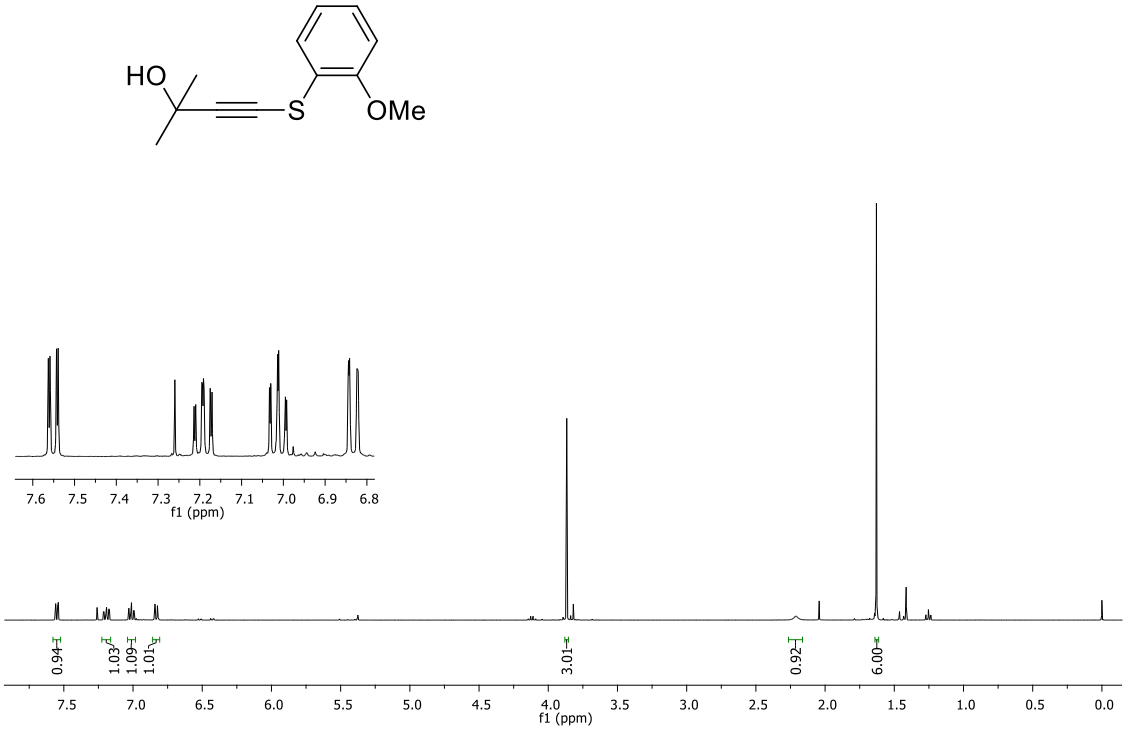
^{13}C NMR spectrum for compound **s7** ($CDCl_3$, 100 MHz)



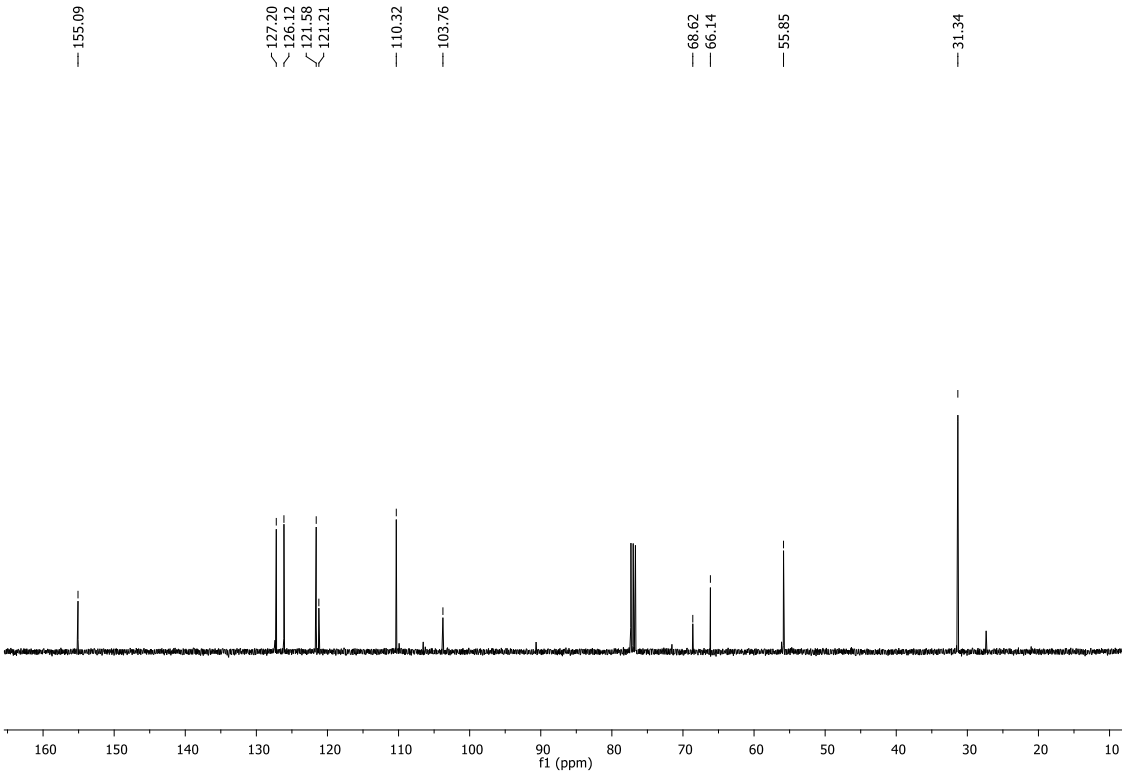
^{1}H NMR spectrum for compound **s8** ($CDCl_3$, 400 MHz)



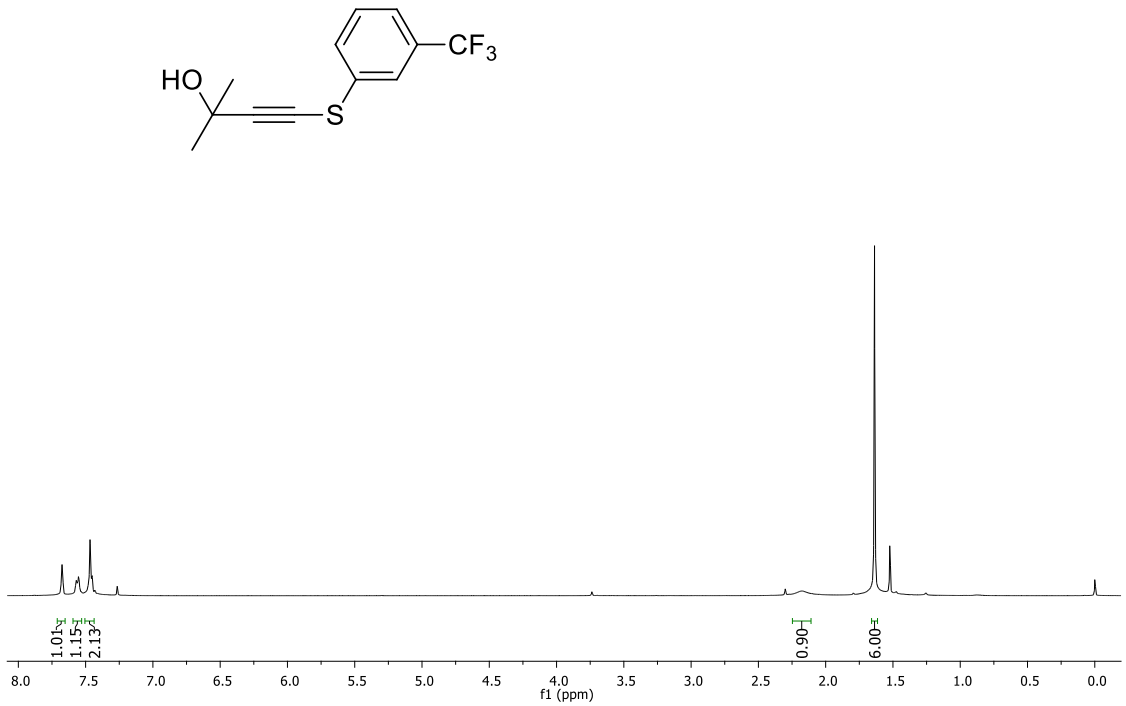
^{13}C NMR spectrum for compound **s8** ($CDCl_3$, 100 MHz)



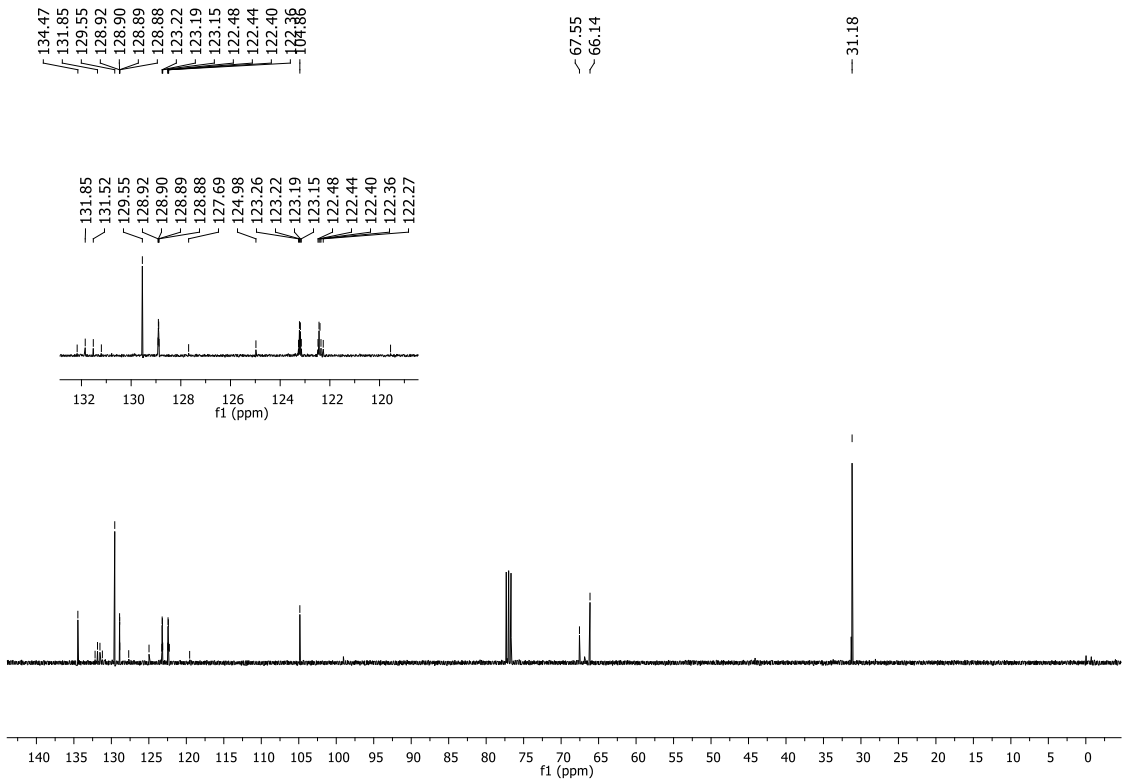
^{1}H NMR spectrum for compound **s9** ($CDCl_3$, 400 MHz)



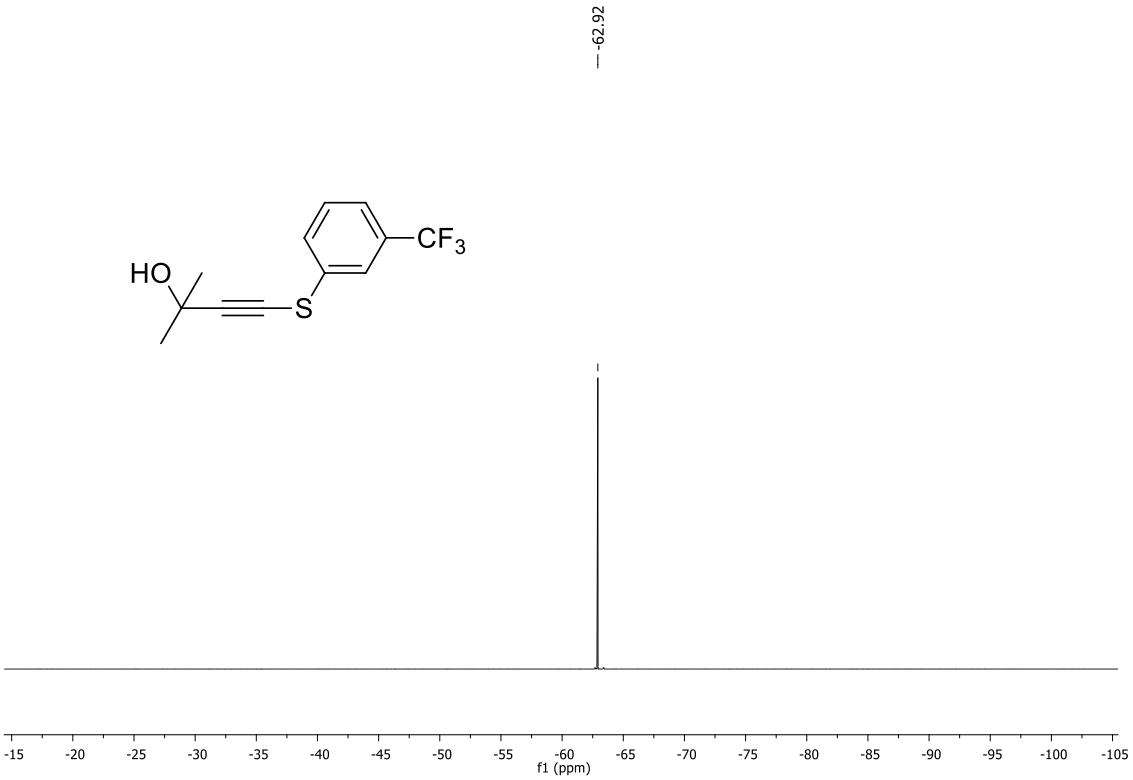
^{13}C NMR spectrum for compound **s9** ($CDCl_3$, 100 MHz)



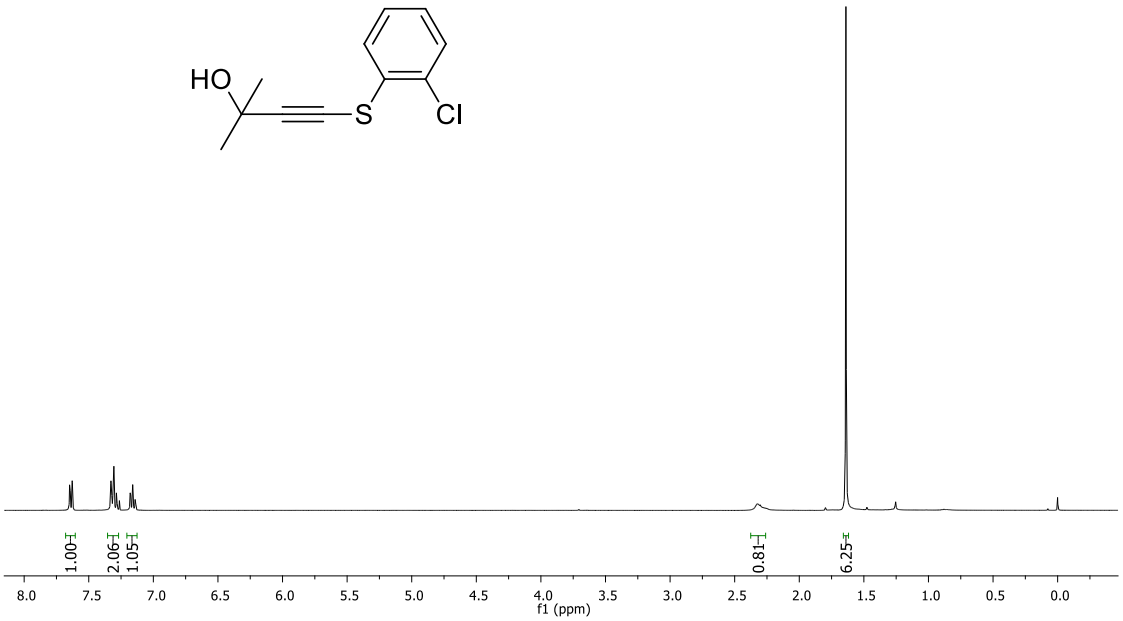
^{1}H NMR spectrum for compound **s10** ($CDCl_3$, 400 MHz)



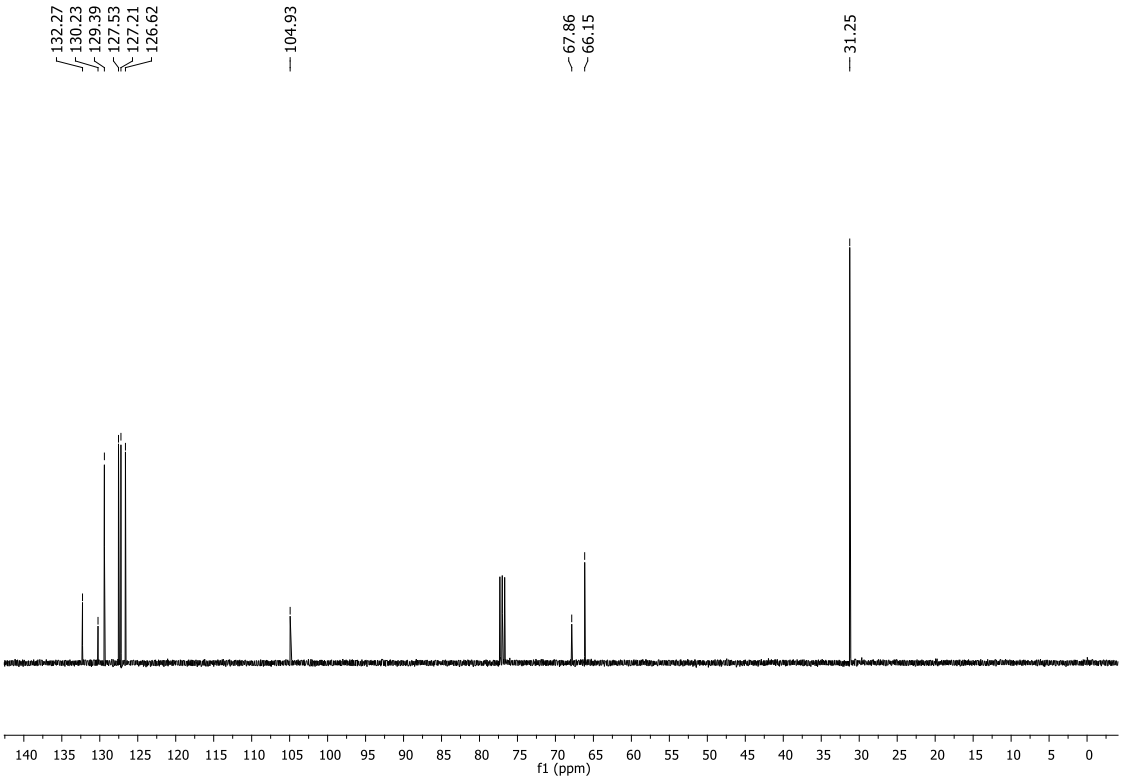
^{13}C NMR spectrum for compound **s10** ($CDCl_3$, 100 MHz)



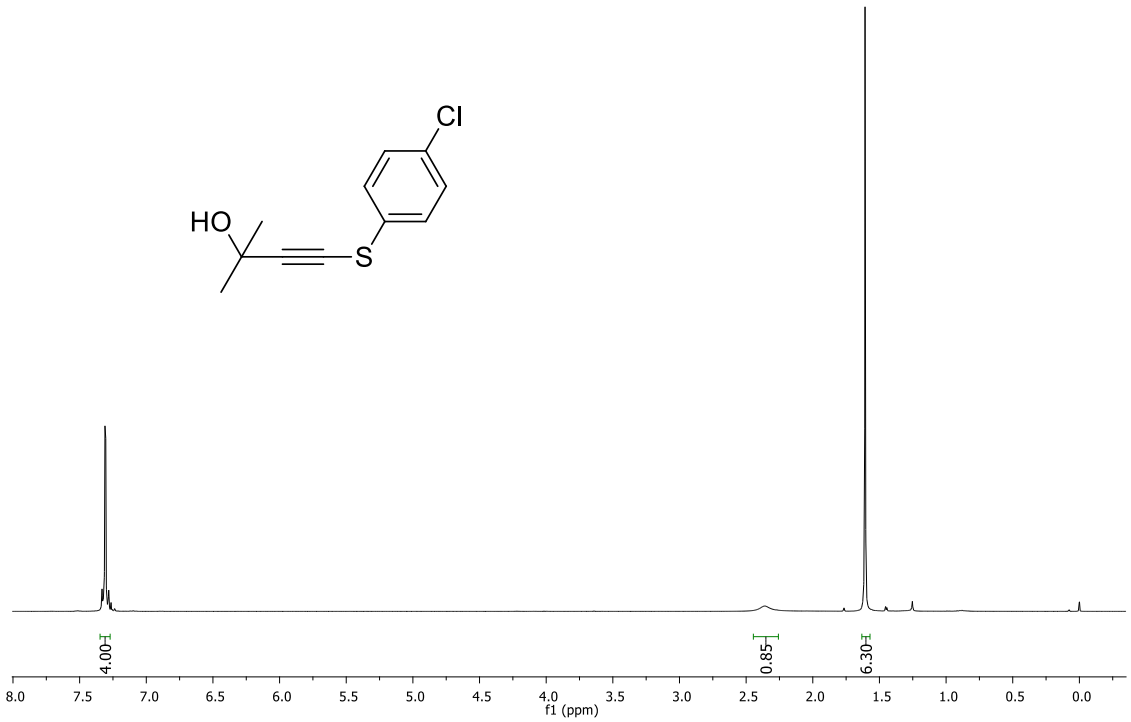
^{19}F NMR spectrum for compound **s10** ($CDCl_3$, 376 MHz)



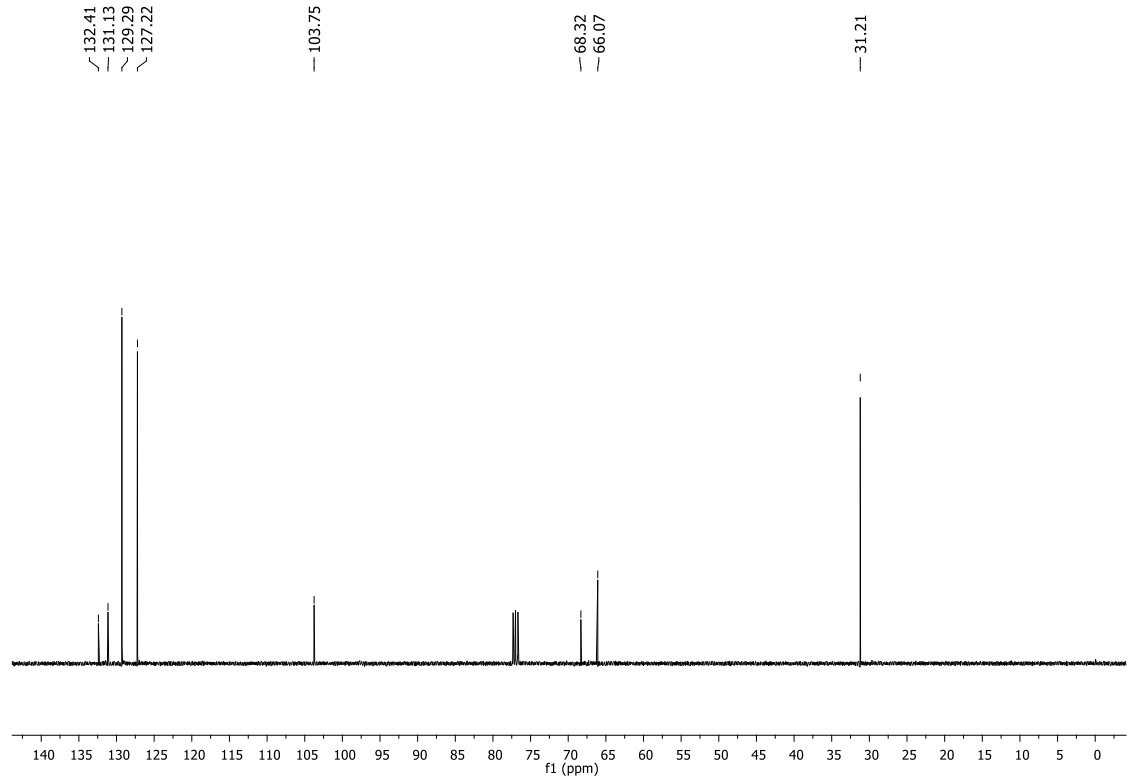
^{1}H NMR spectrum for compound **s11** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **s11** ($CDCl_3$, 100 MHz)

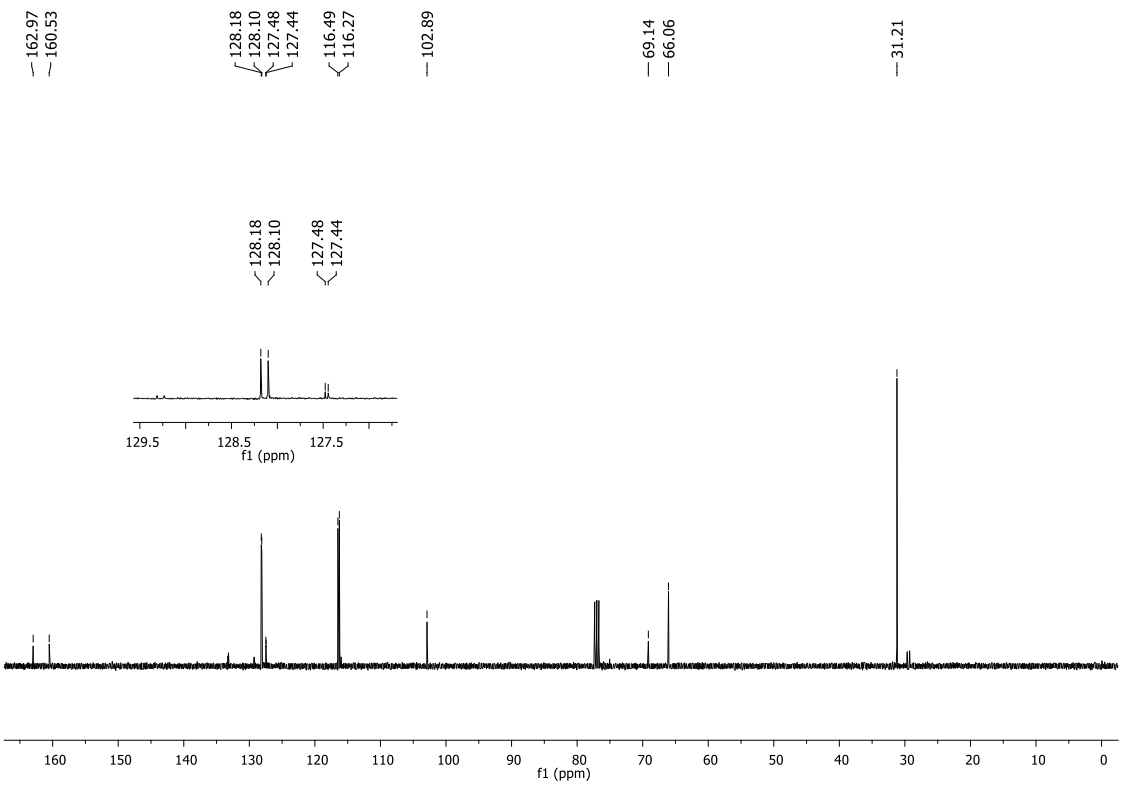


^{1}H NMR spectrum for compound **s12** ($CDCl_3$, 400 MHz)

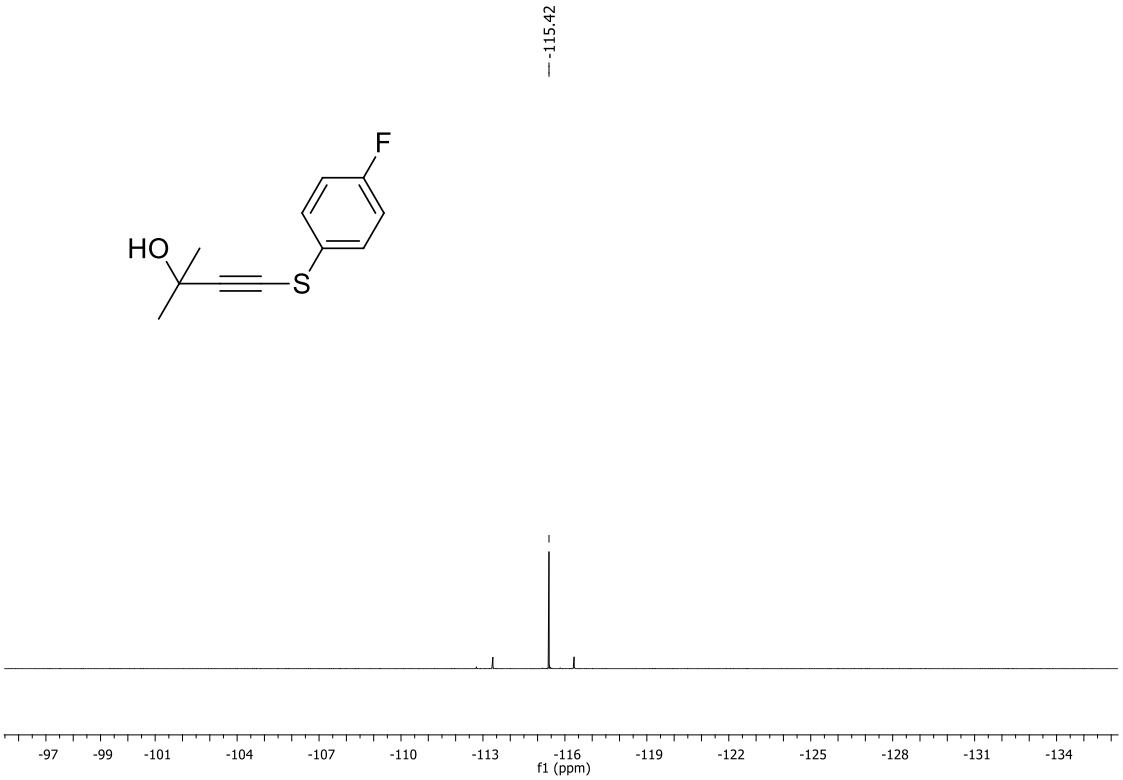


^{13}C NMR spectrum for compound **s12** ($CDCl_3$, 100 MHz)

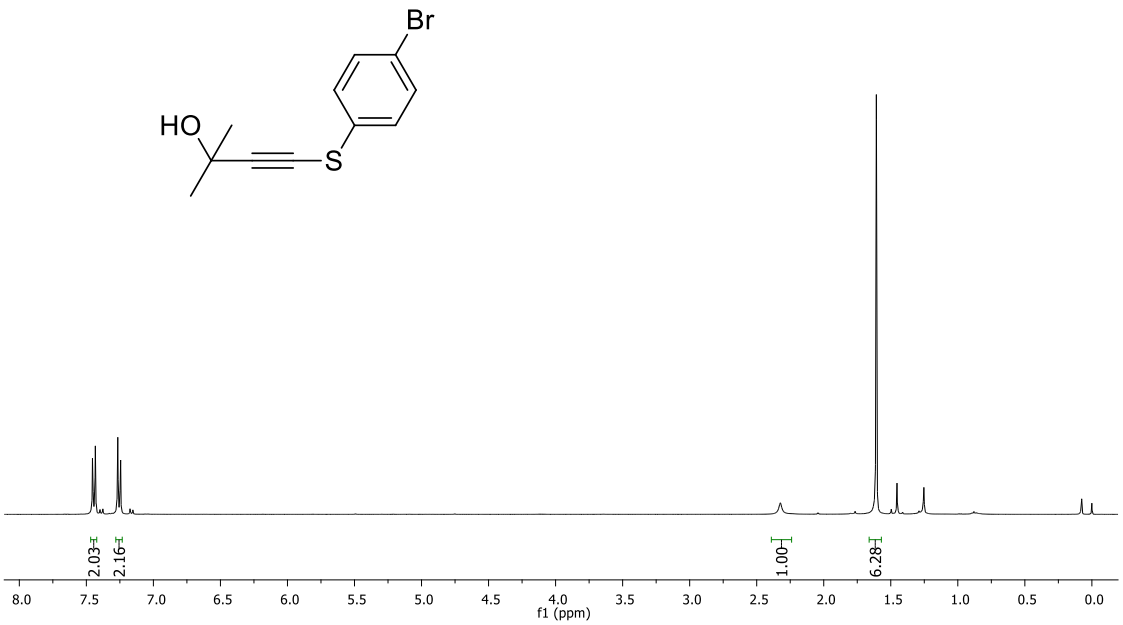
^{1}H NMR spectrum for compound **s13** ($CDCl_3$, 400 MHz)



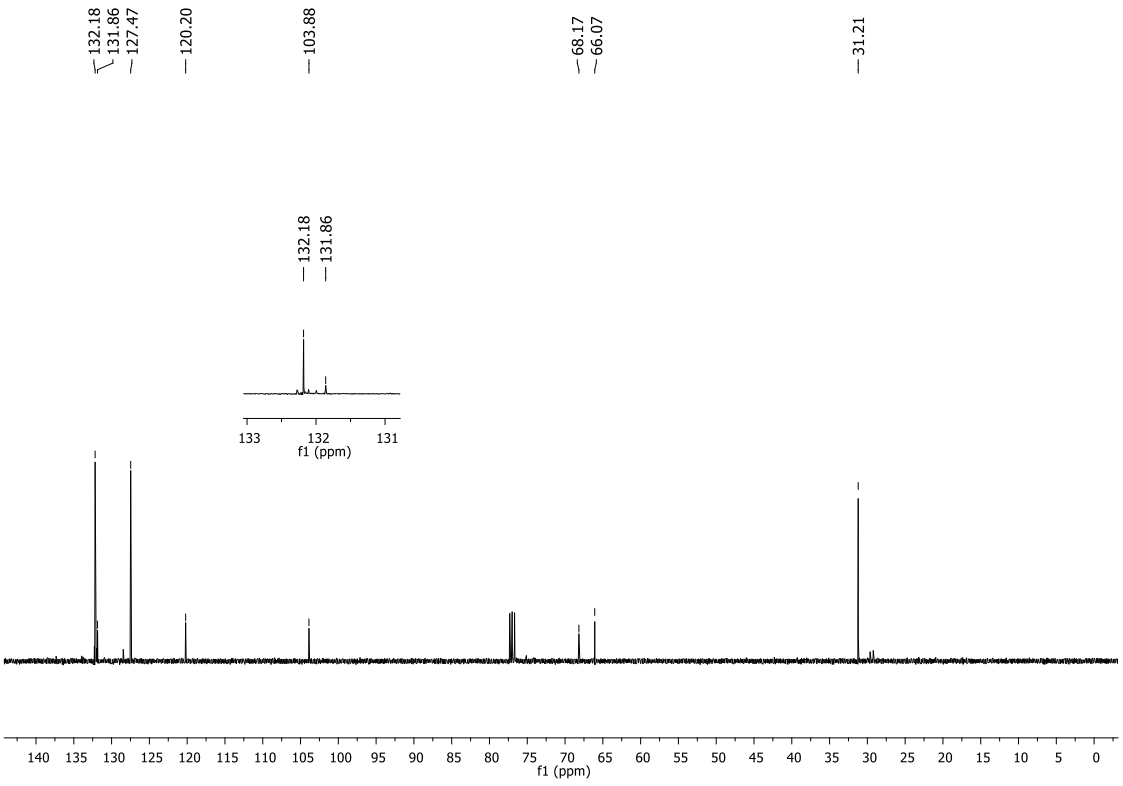
^{13}C NMR spectrum for compound **s13** ($CDCl_3$, 100 MHz)



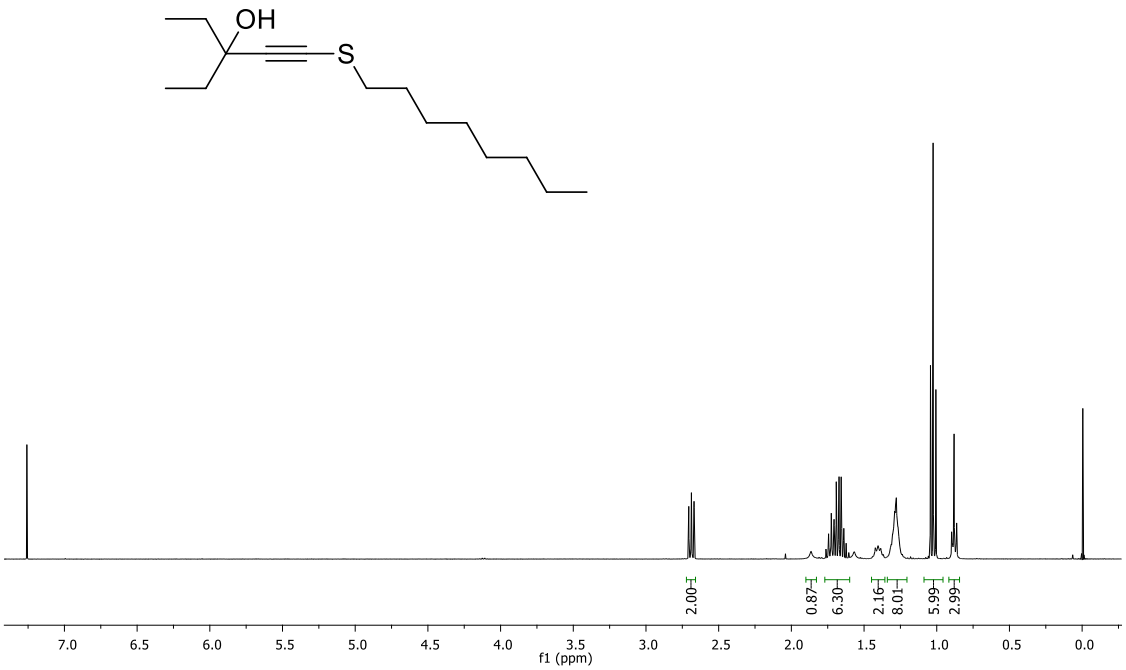
^{19}F NMR spectrum for compound **s13** ($CDCl_3$, 376 MHz)



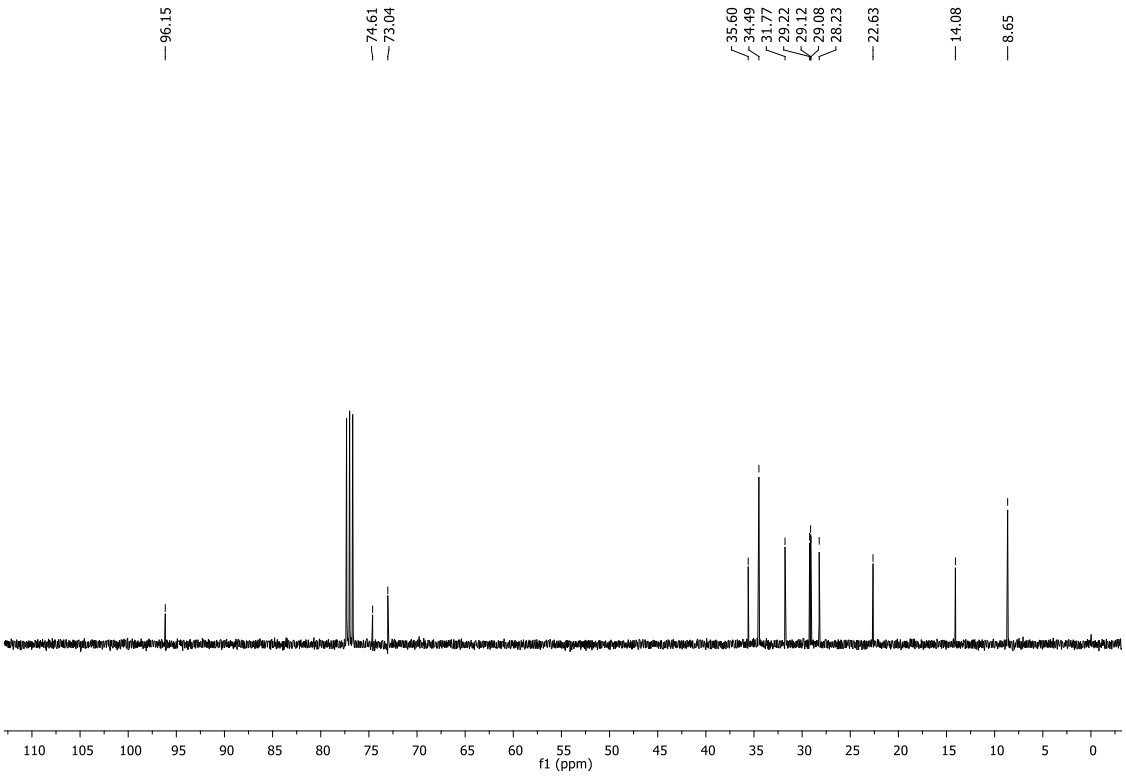
^{1}H NMR spectrum for compound **s14** ($CDCl_3$, 400 MHz)



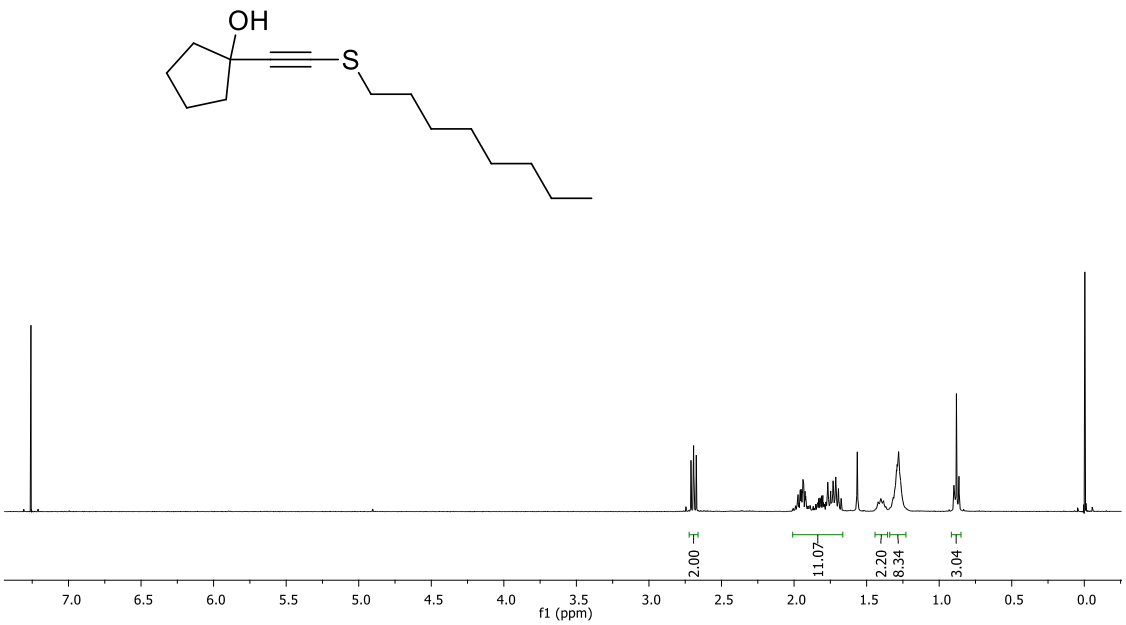
^{13}C NMR spectrum for compound **s14** ($CDCl_3$, 100 MHz)



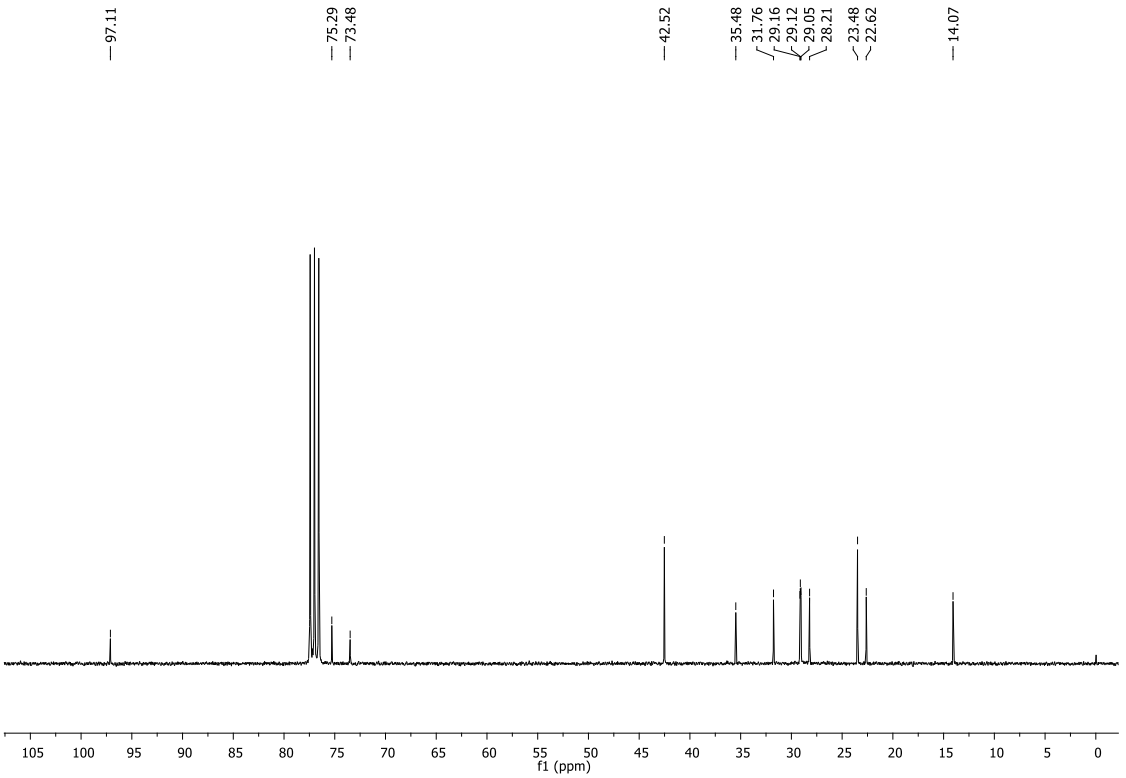
^{1}H NMR spectrum for compound **s15** ($CDCl_3$, 400 MHz)



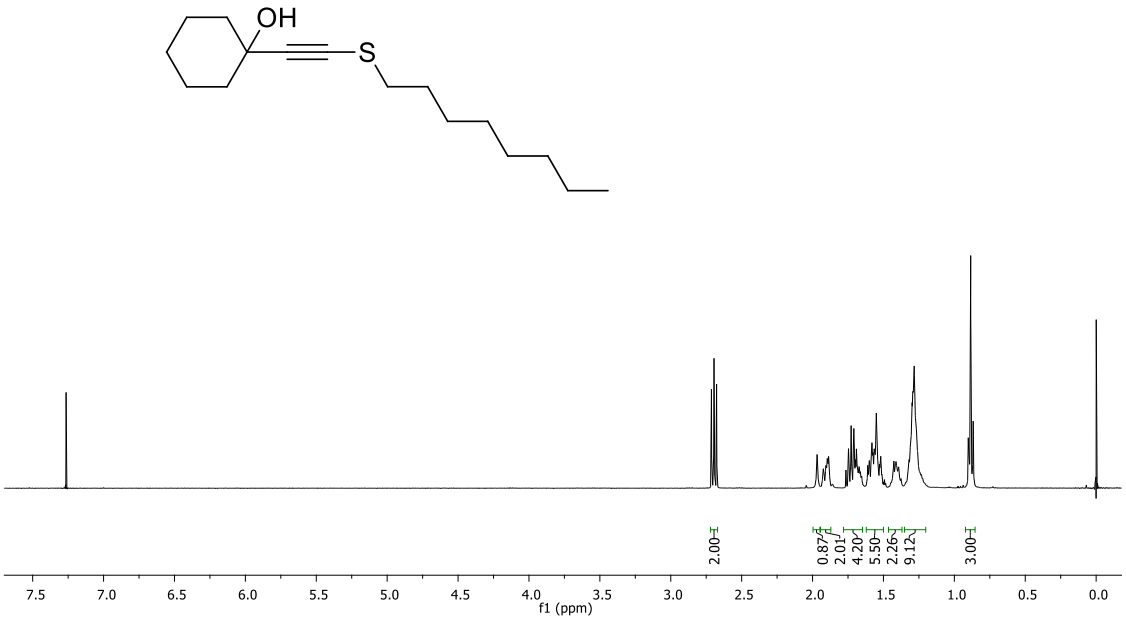
^{13}C NMR spectrum for compound **s15** ($CDCl_3$, 100 MHz)



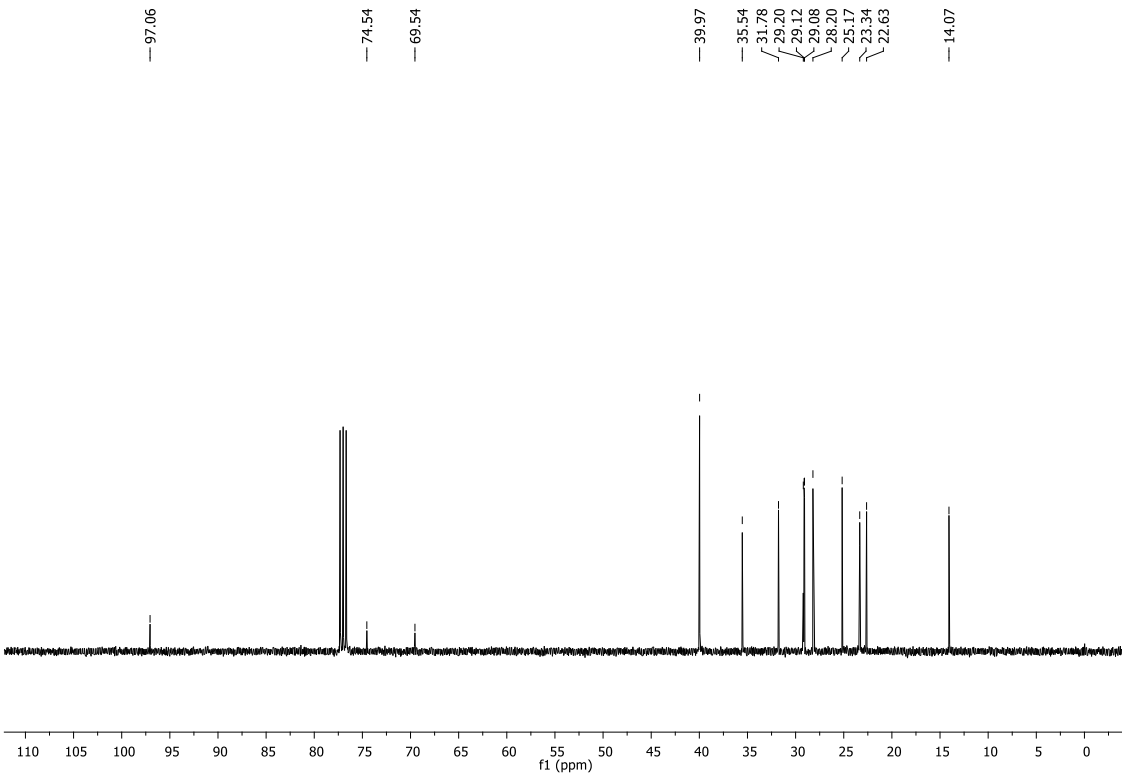
^{1}H NMR spectrum for compound **s16** ($CDCl_3$, 400 MHz)



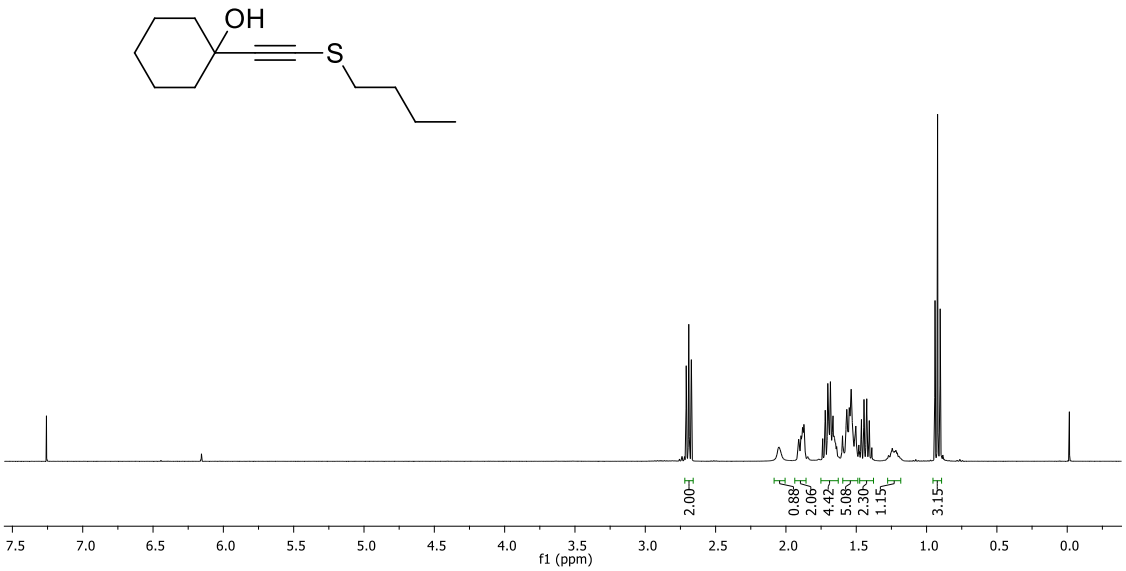
^{13}C NMR spectrum for compound **s16** ($CDCl_3$, 100 MHz)



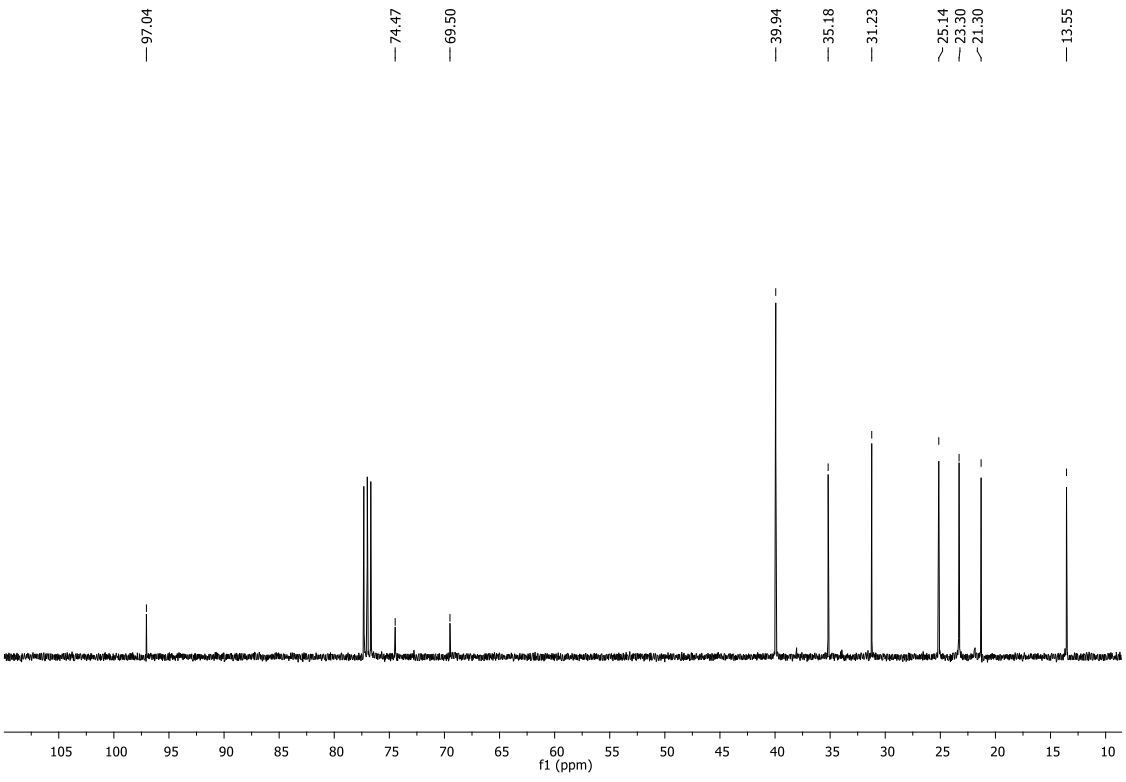
^{1}H NMR spectrum for compound **s17** ($CDCl_3$, 400 MHz)



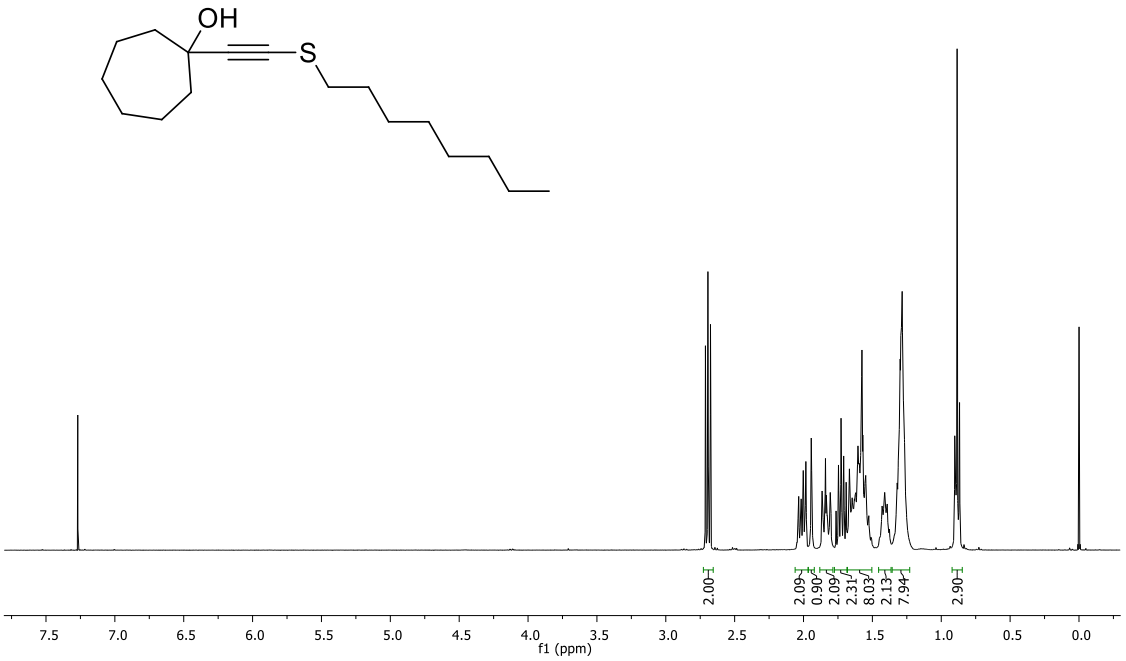
^{13}C NMR spectrum for compound **s17** ($CDCl_3$, 100 MHz)



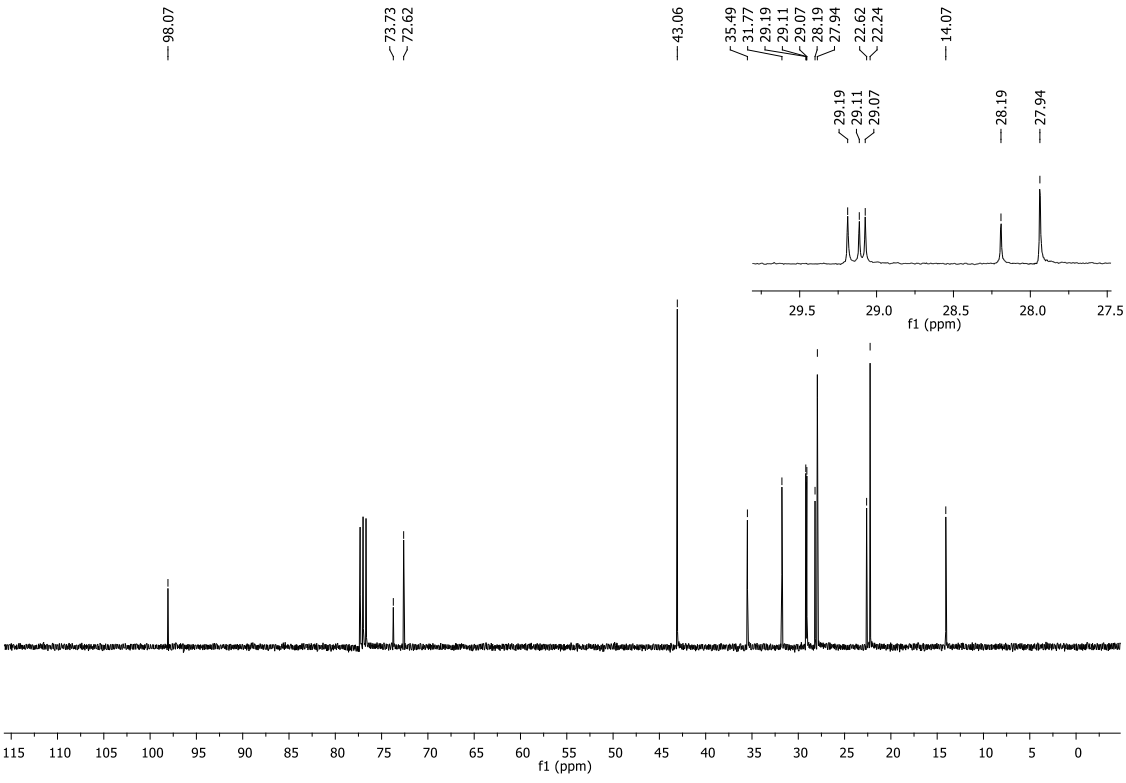
^{1}H NMR spectrum for compound **s18** ($CDCl_3$, 400 MHz)



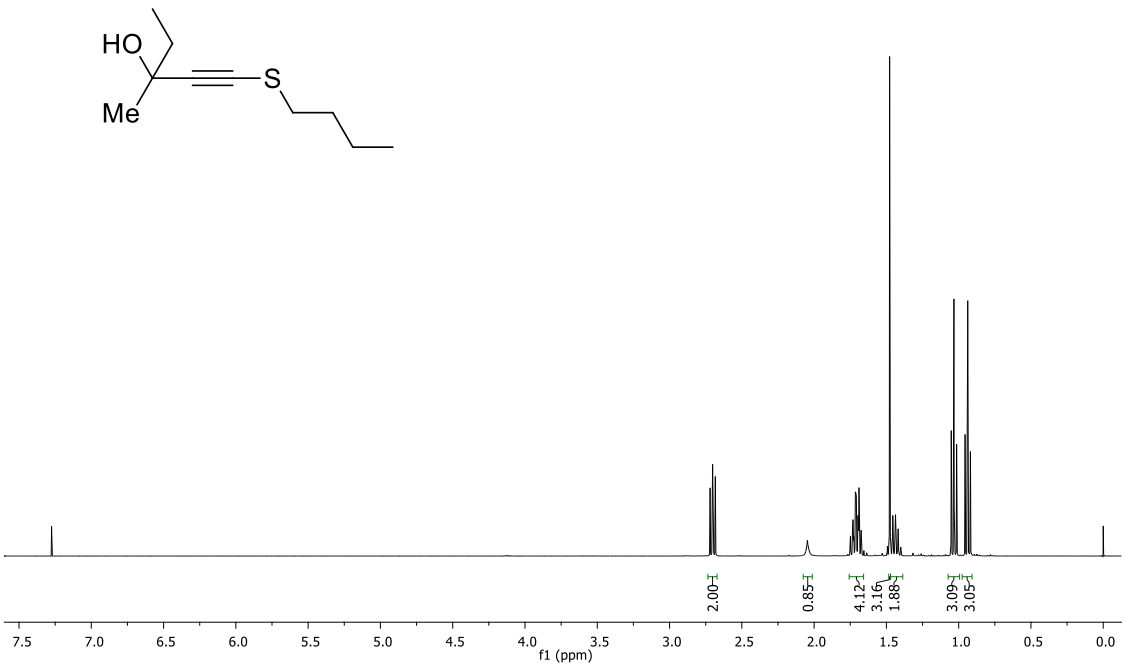
^{13}C NMR spectrum for compound **s18** ($CDCl_3$, 100 MHz)



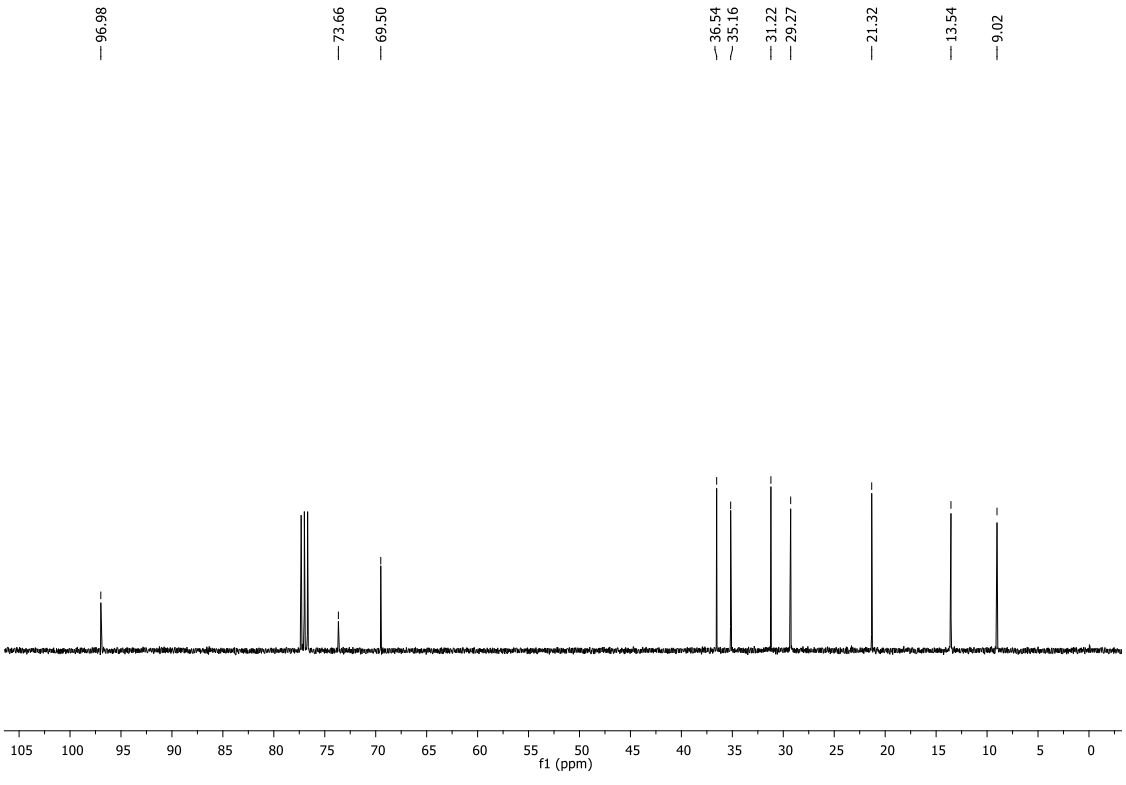
^{1}H NMR spectrum for compound **s19** ($CDCl_3$, 400 MHz)



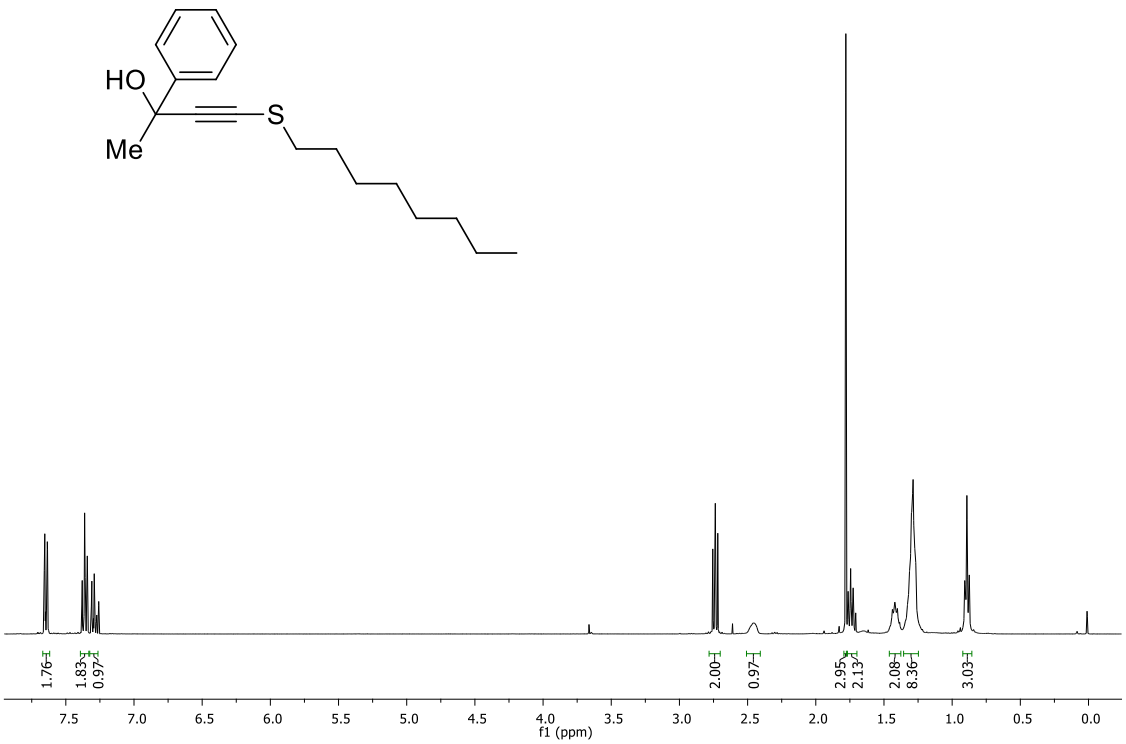
^{13}C NMR spectrum for compound **s19** ($CDCl_3$, 100 MHz)



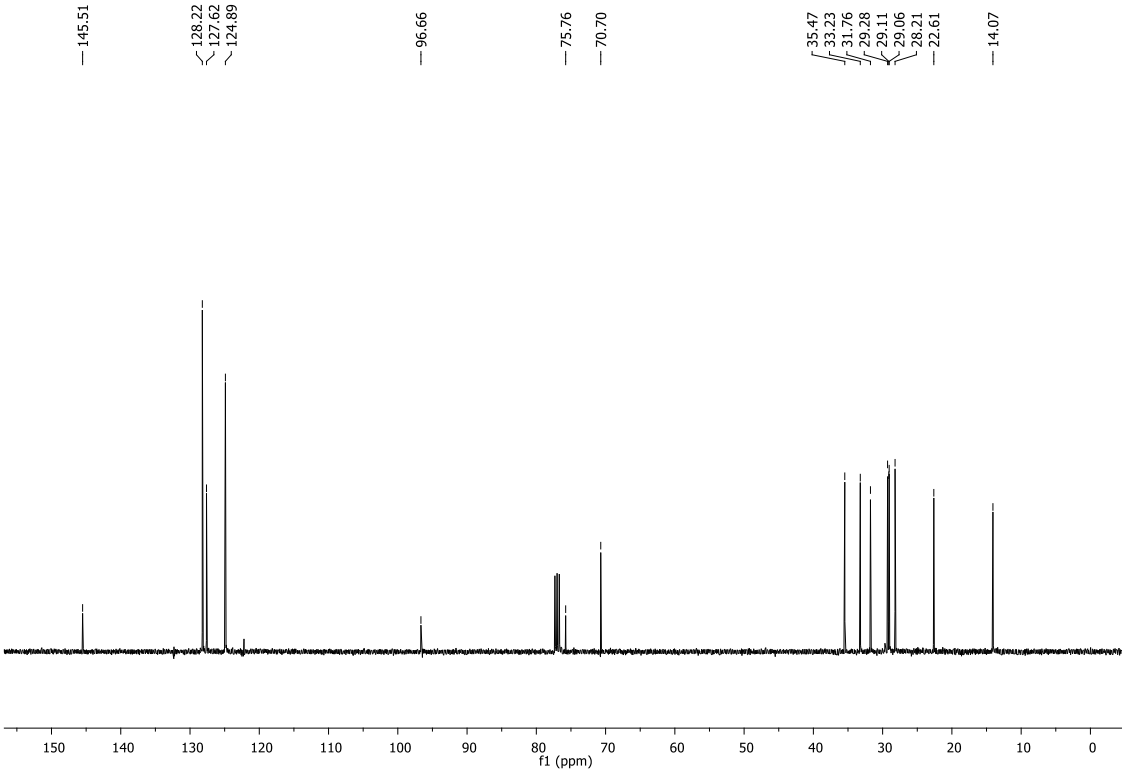
^{1}H NMR spectrum for compound **s21** ($CDCl_3$, 400 MHz)



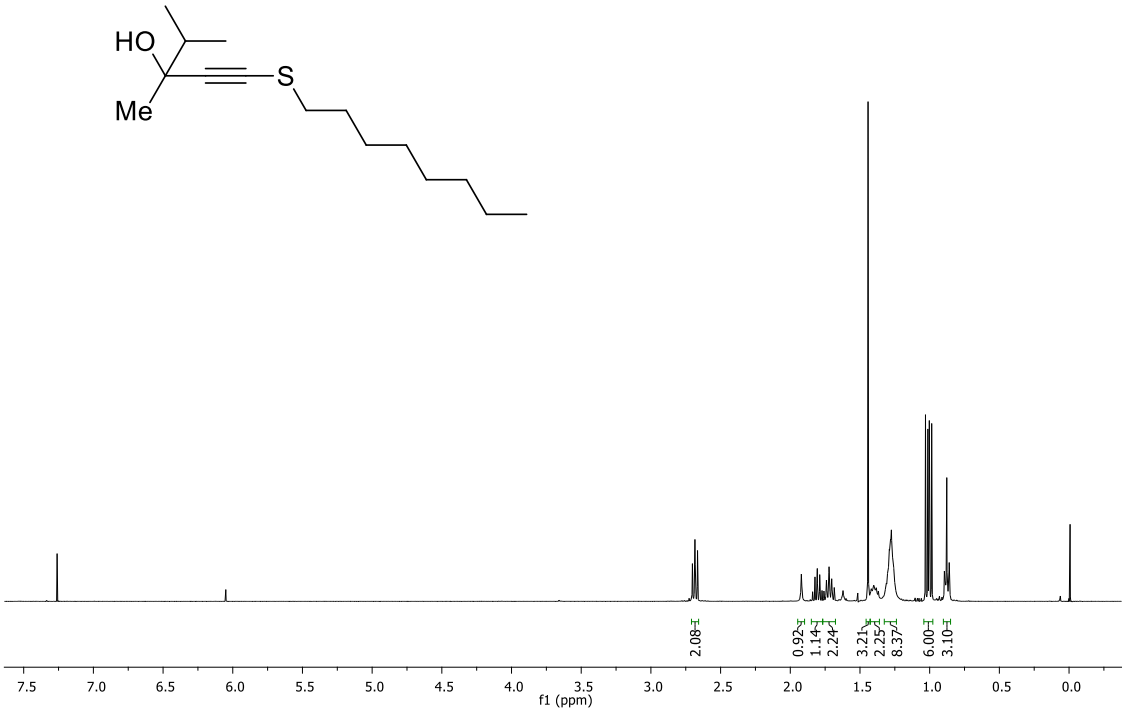
^{13}C NMR spectrum for compound **s21** ($CDCl_3$, 100 MHz)



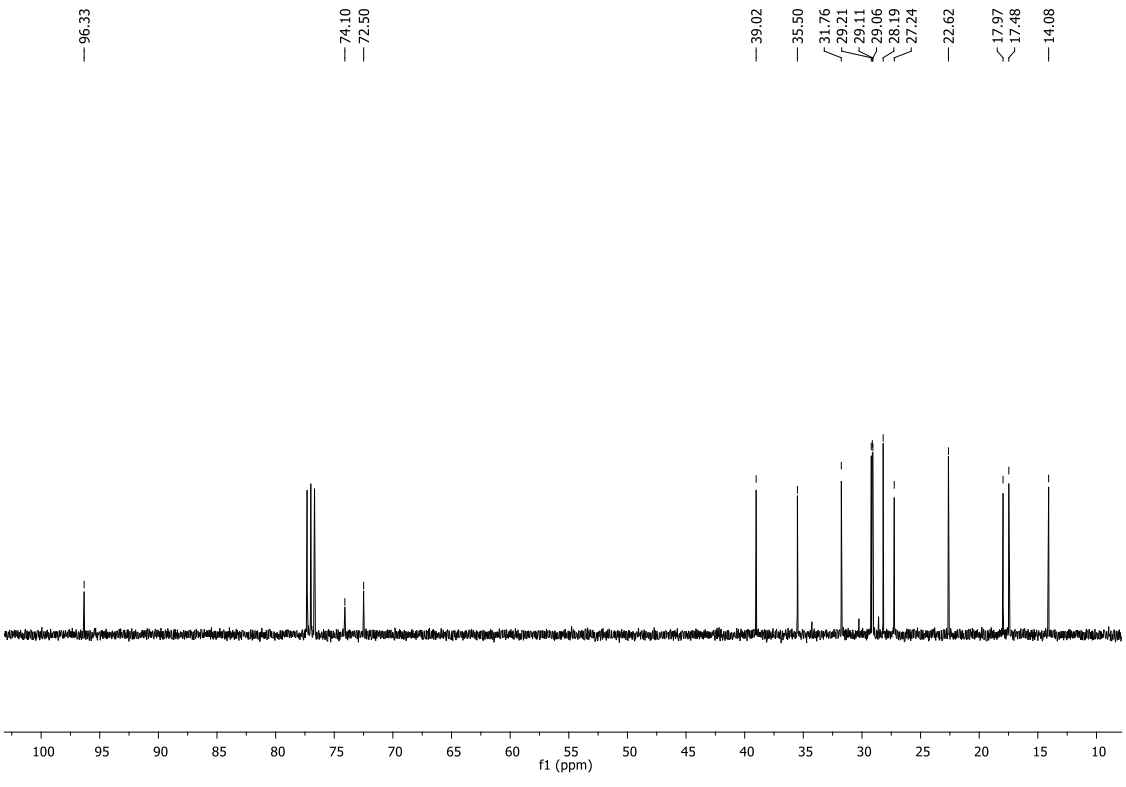
^{1}H NMR spectrum for compound **s22** ($CDCl_3$, 400 MHz)



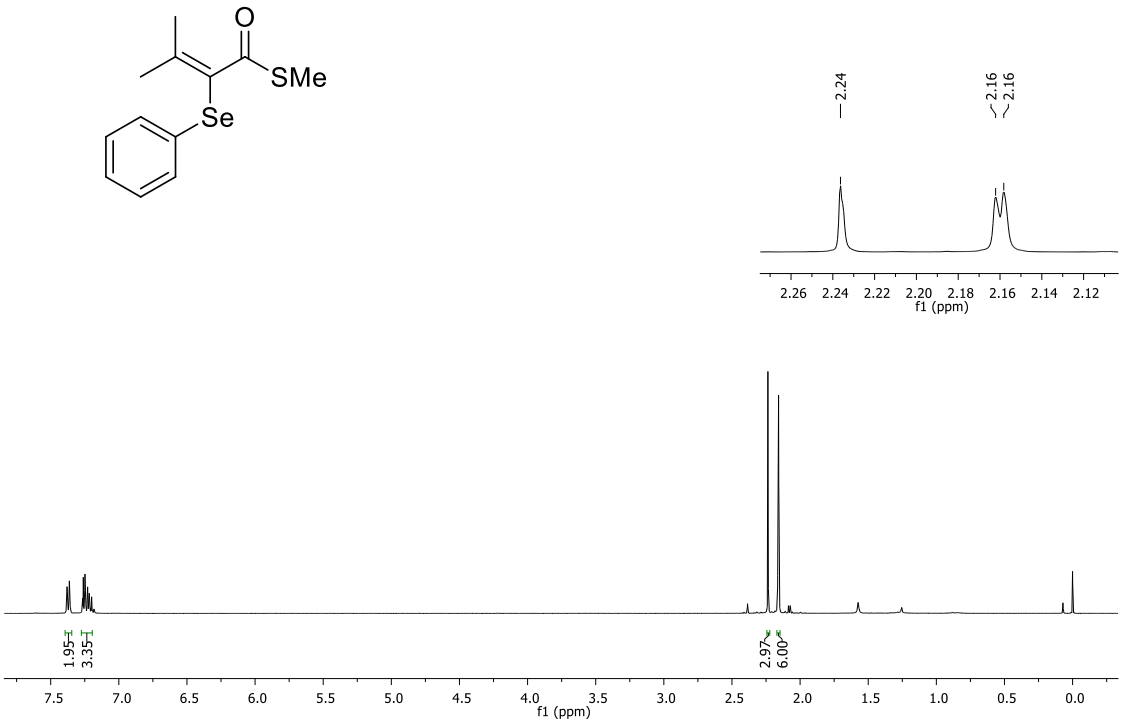
^{13}C NMR spectrum for compound **s22** ($CDCl_3$, 100 MHz)



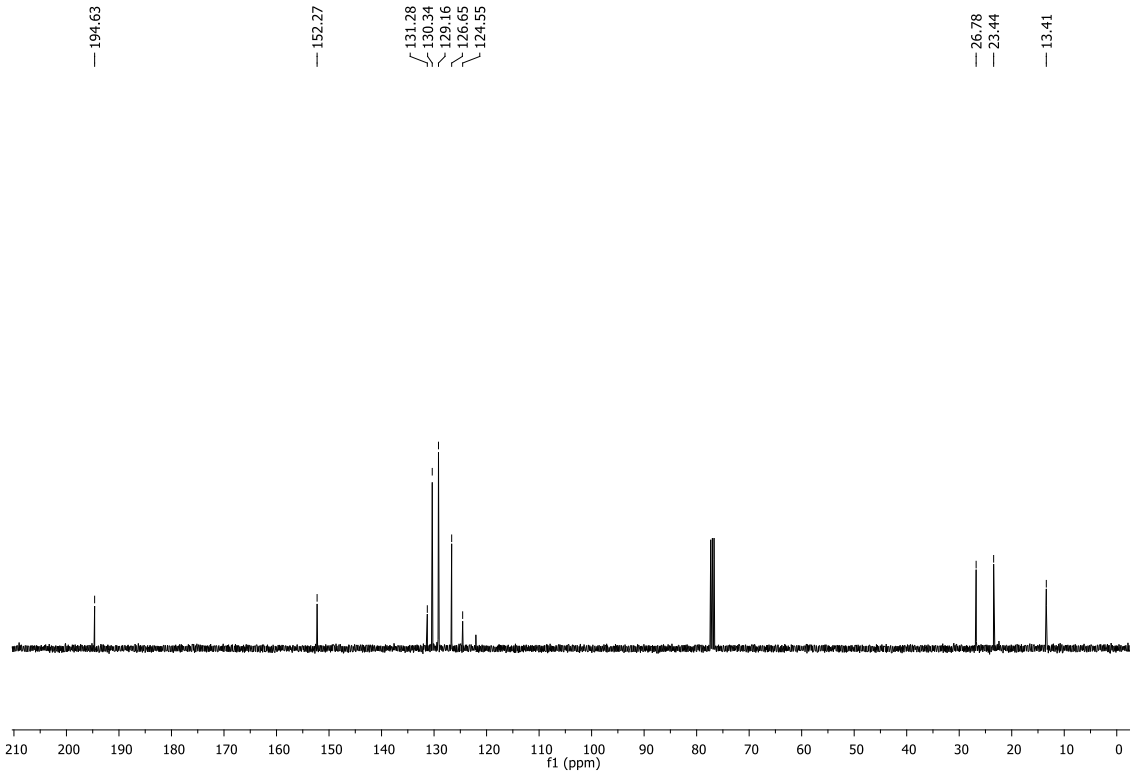
^{1}H NMR spectrum for compound **s23** ($CDCl_3$, 400 MHz)



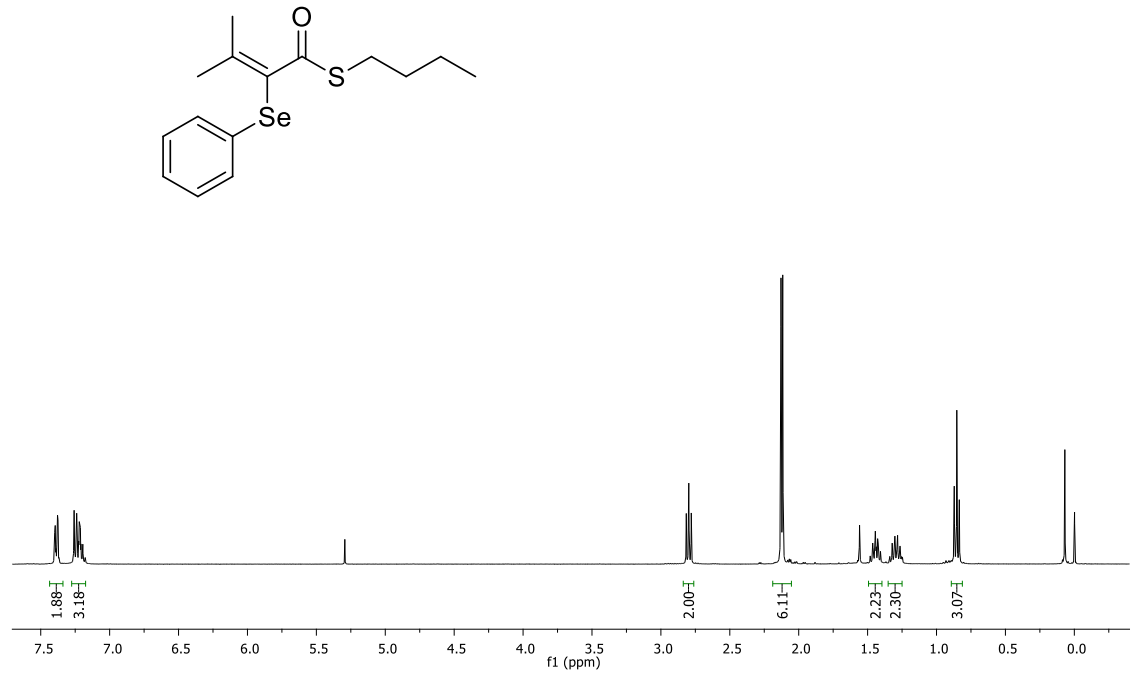
^{13}C NMR spectrum for compound **s23** ($CDCl_3$, 100 MHz)



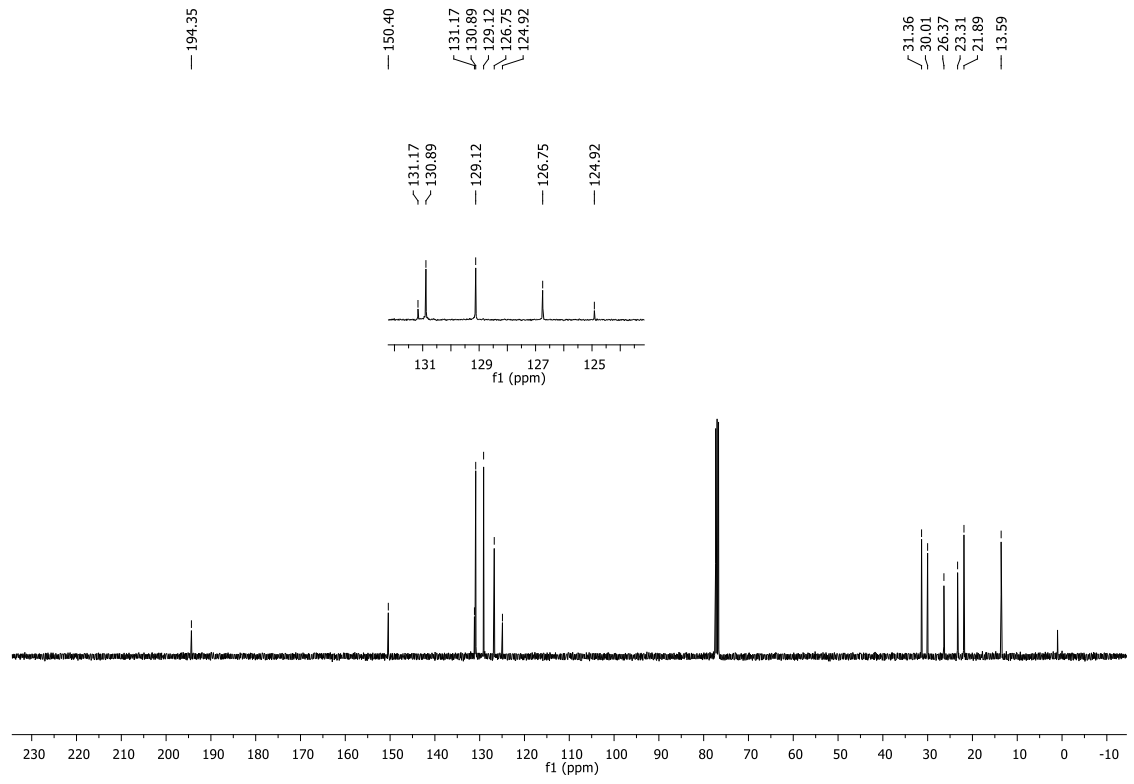
^{1}H NMR spectrum for compound **1** ($CDCl_3$, 400 MHz)



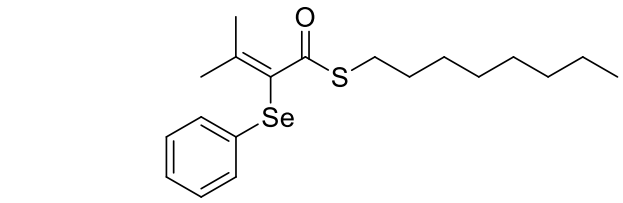
^{13}C NMR spectrum for compound **1** ($CDCl_3$, 100 MHz)

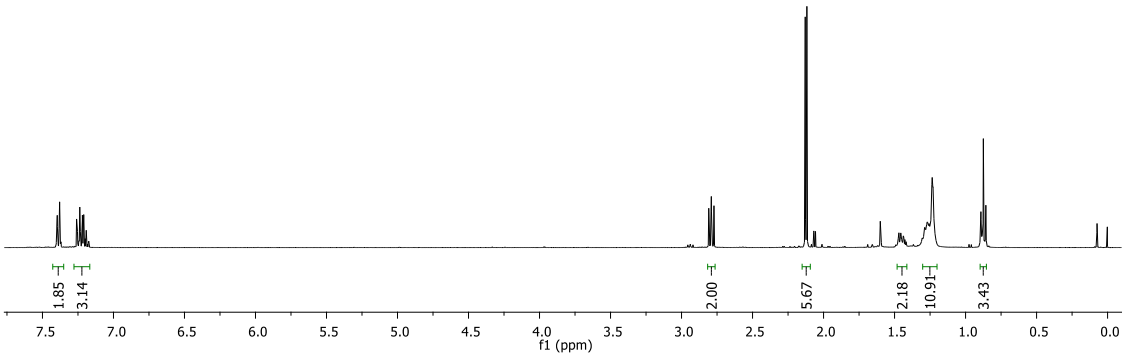


^{1}H NMR spectrum for compound **2** ($CDCl_3$, 400 MHz)

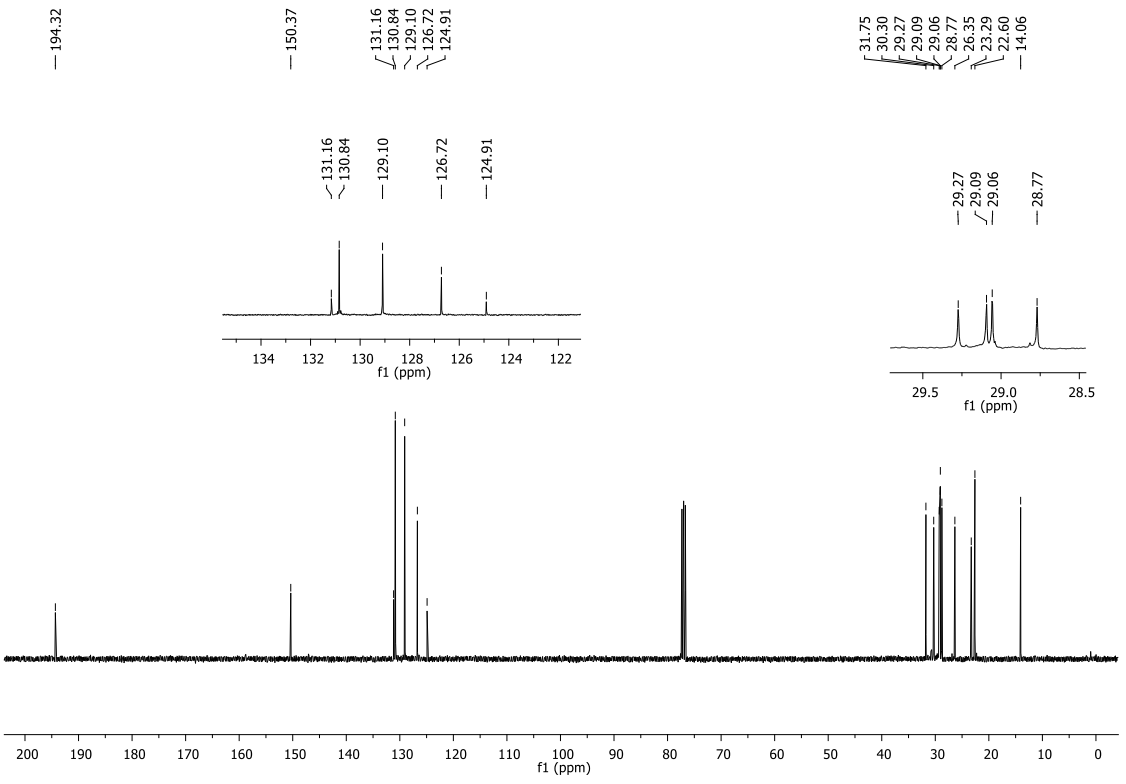


^{13}C NMR spectrum for compound **2** ($CDCl_3$, 100 MHz)

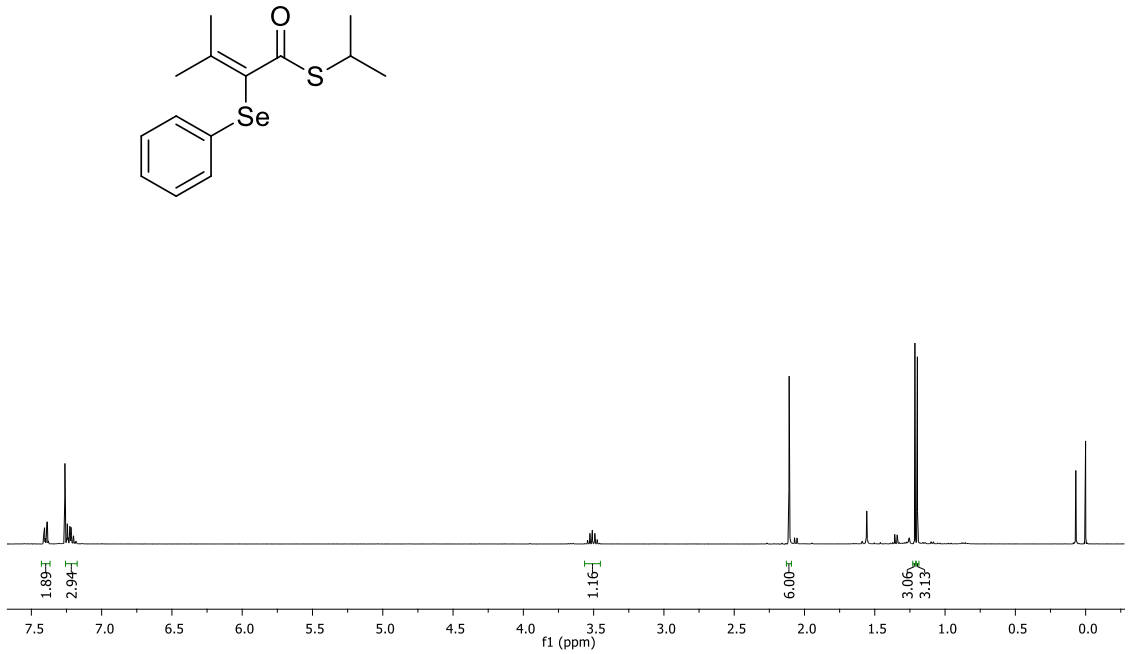




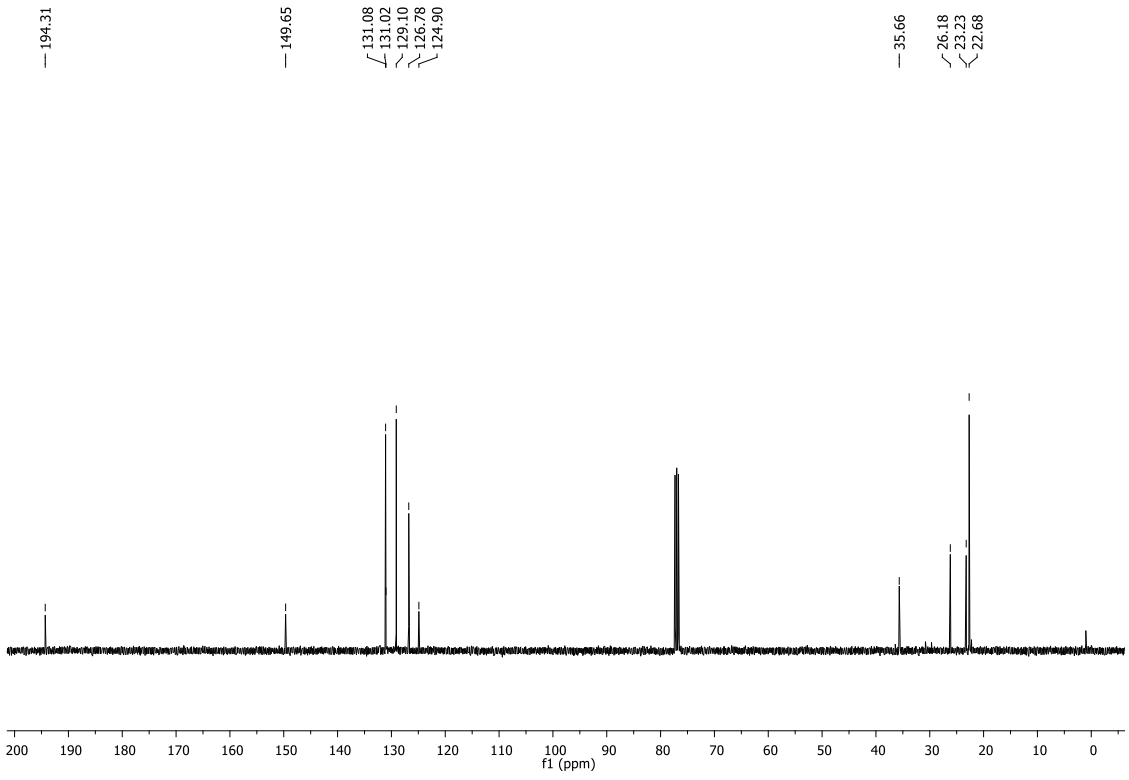
^{1}H NMR spectrum for compound **3** ($CDCl_3$, 400 MHz)



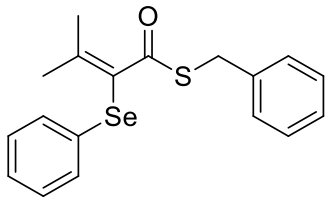
^{13}C NMR spectrum for compound **3** ($CDCl_3$, 100 MHz)

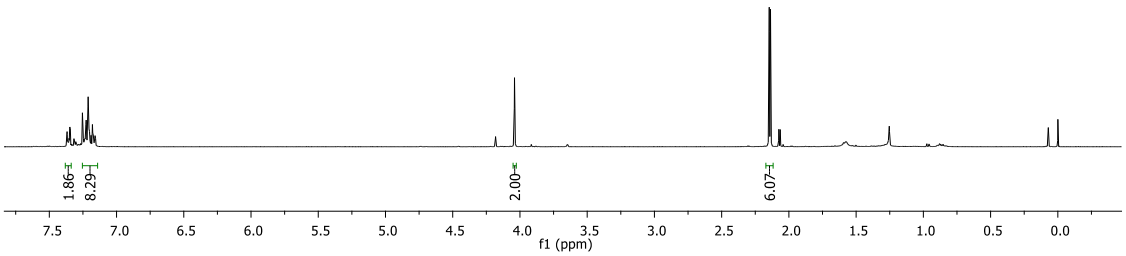


^{1}H NMR spectrum for compound **4** ($CDCl_3$, 400 MHz)

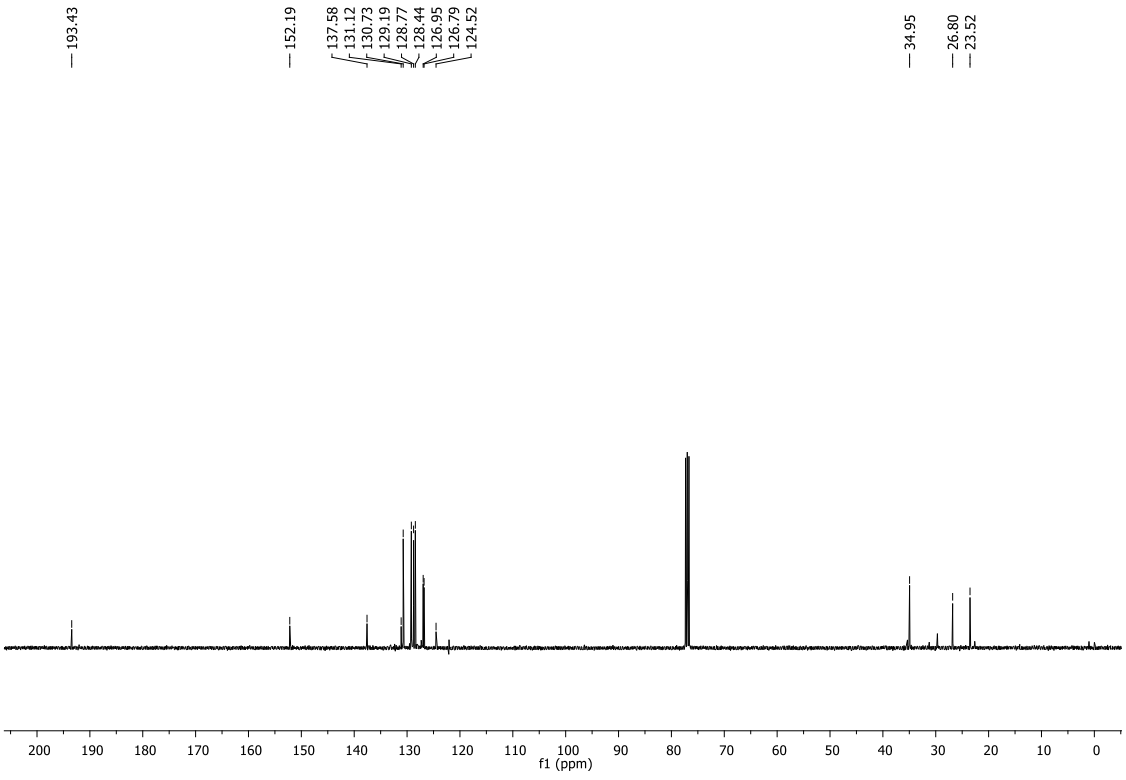


^{13}C NMR spectrum for compound **4** ($CDCl_3$, 100 MHz)

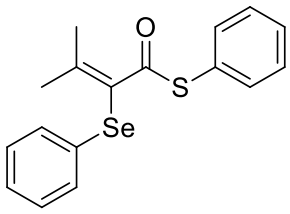


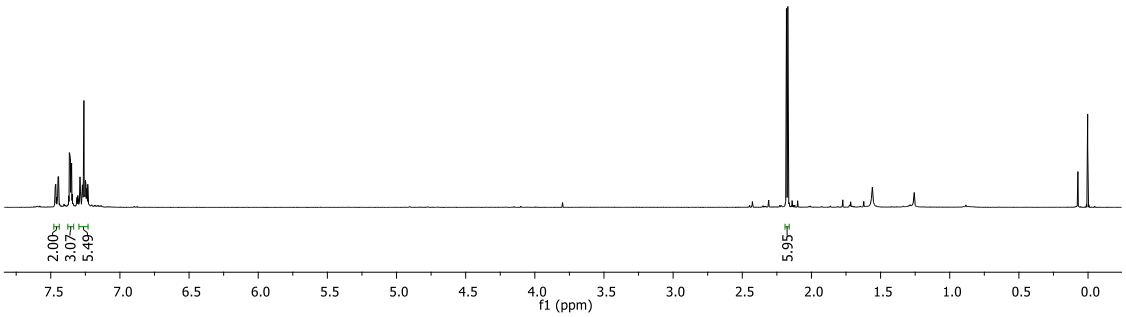


^{1}H NMR spectrum for compound **5** ($CDCl_3$, 400 MHz)

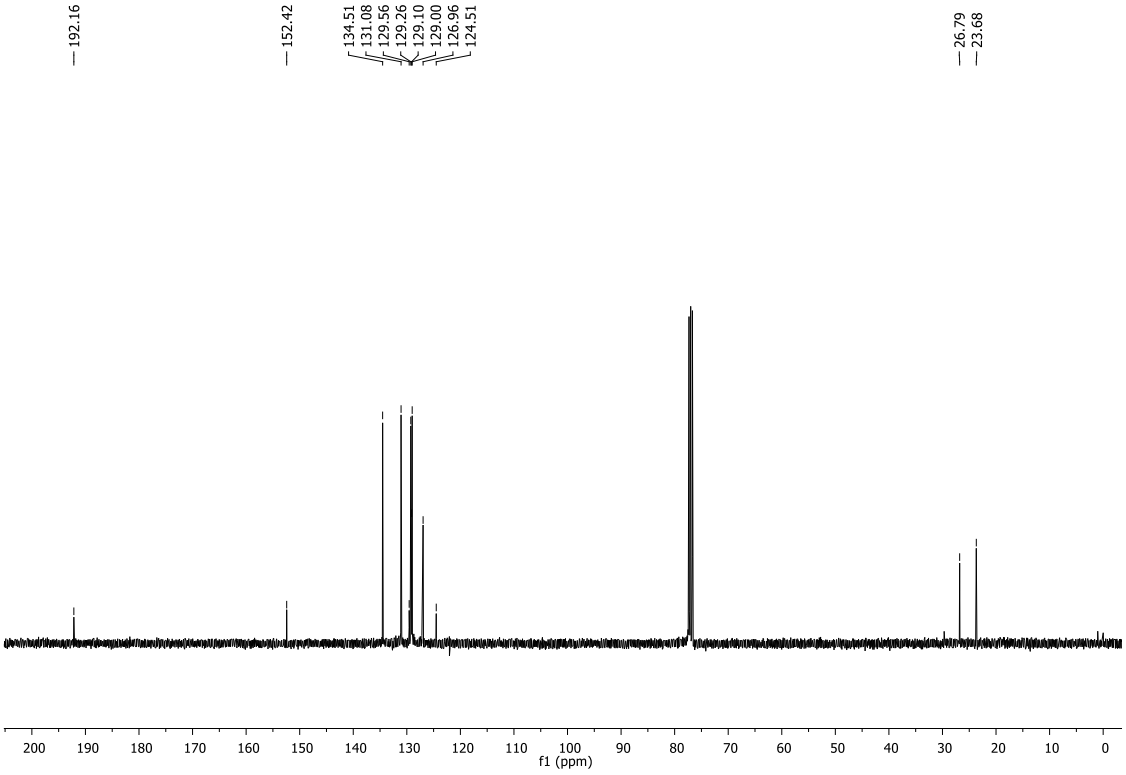


^{13}C NMR spectrum for compound **5** ($CDCl_3$, 100 MHz)

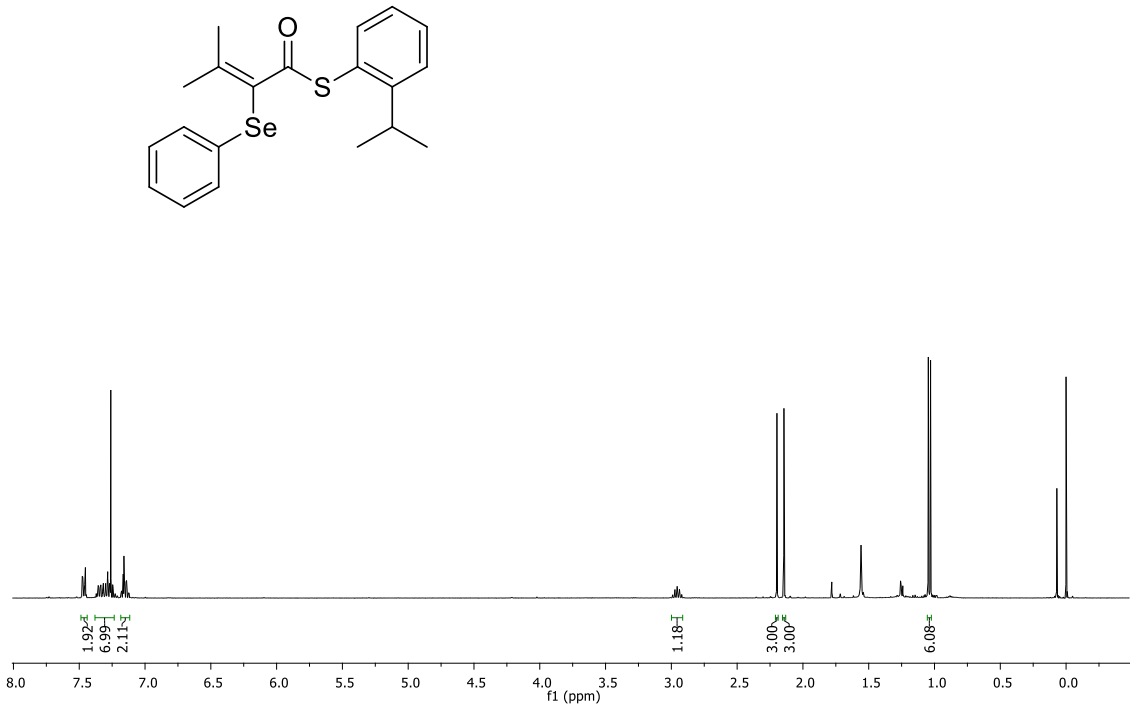




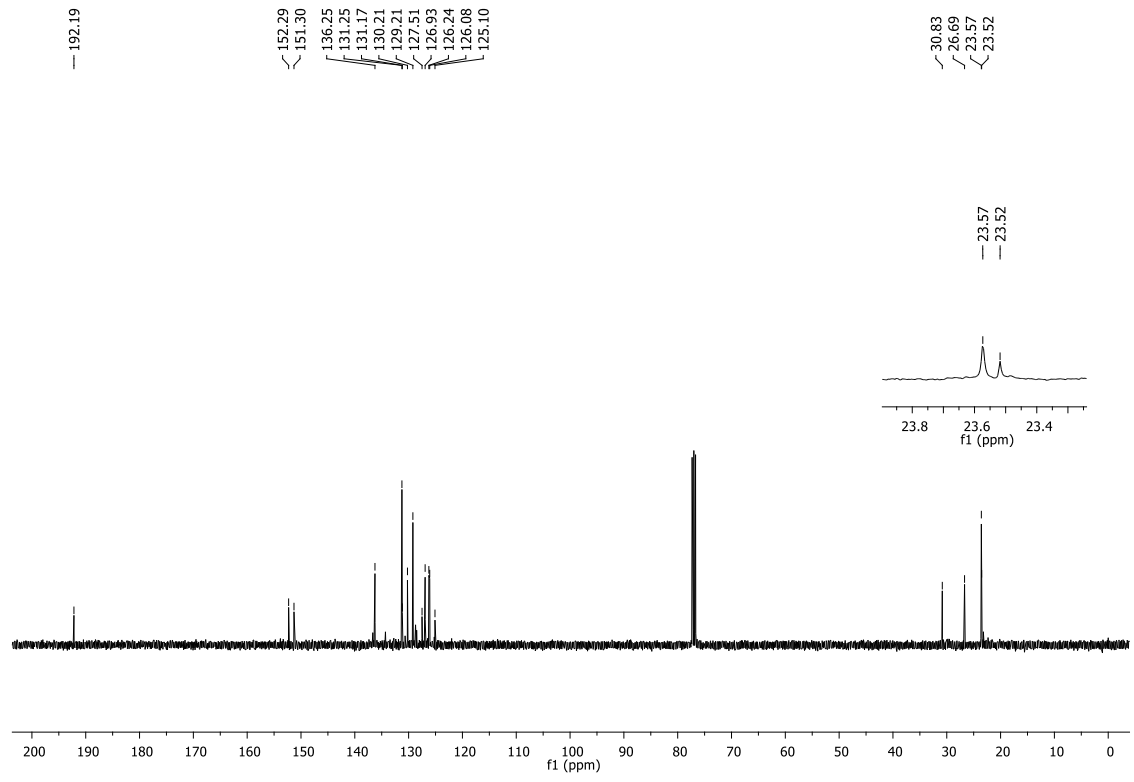
^{1}H NMR spectrum for compound **6** ($CDCl_3$, 400 MHz)



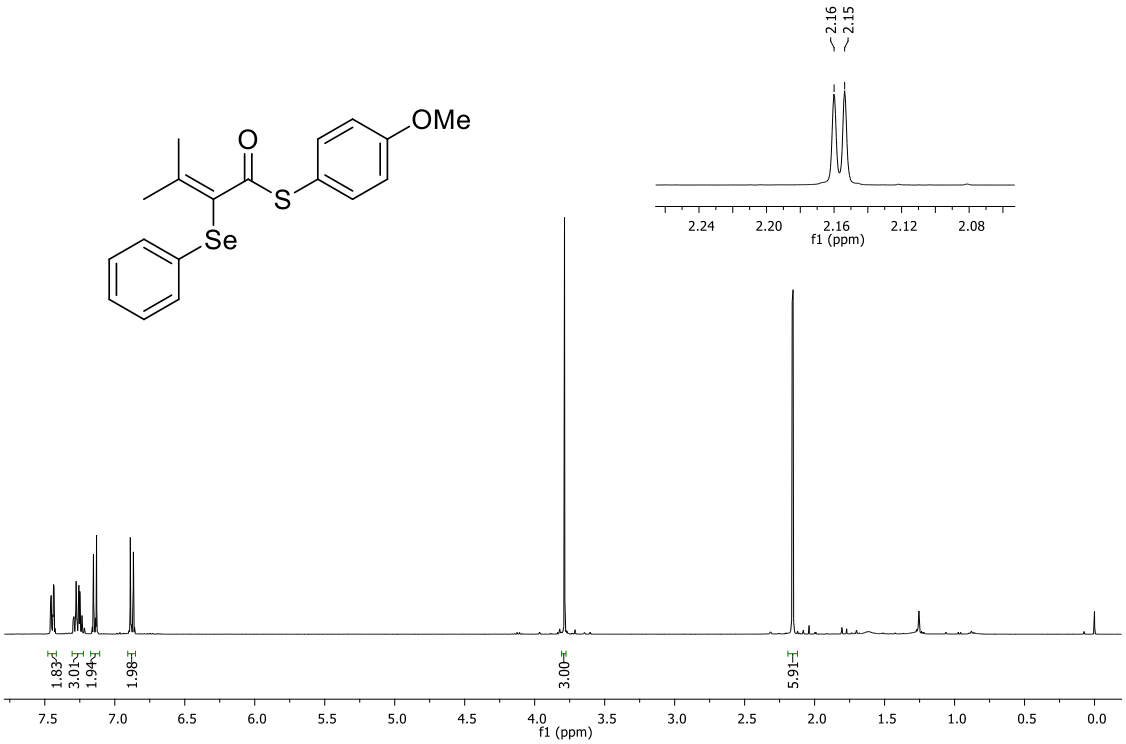
^{13}C NMR spectrum for compound **6** ($CDCl_3$, 100 MHz)



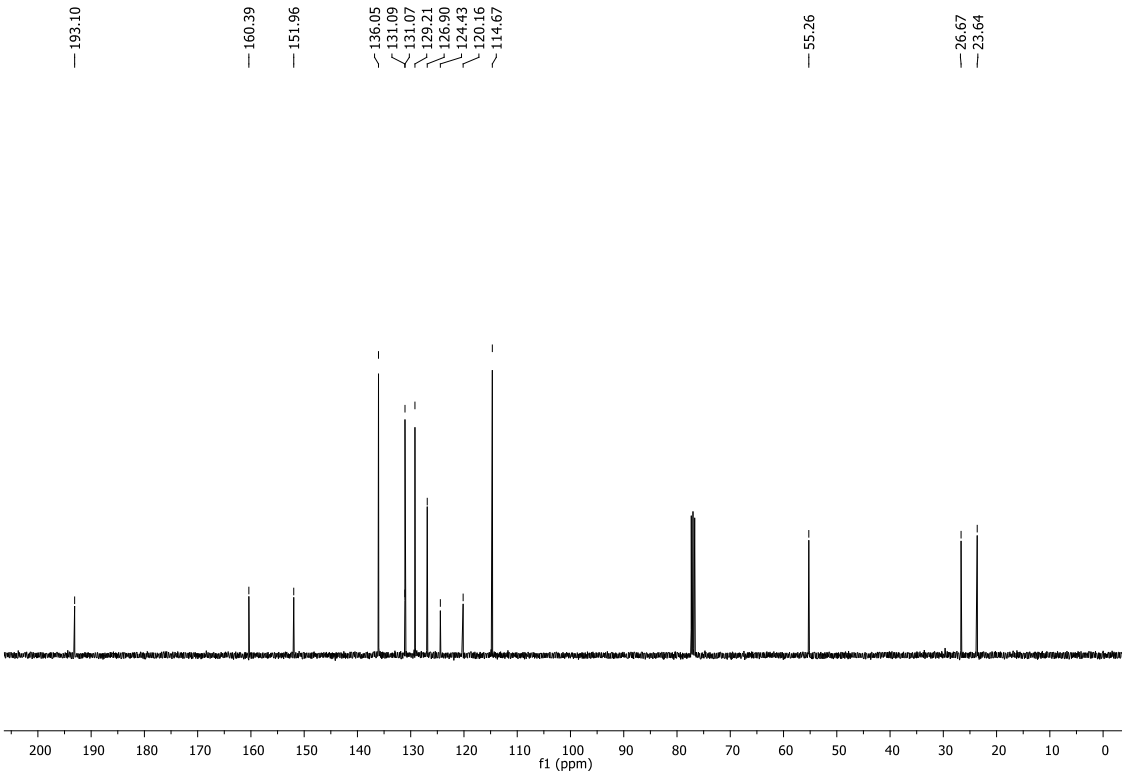
^{1}H NMR spectrum for compound **7** ($CDCl_3$, 400 MHz)



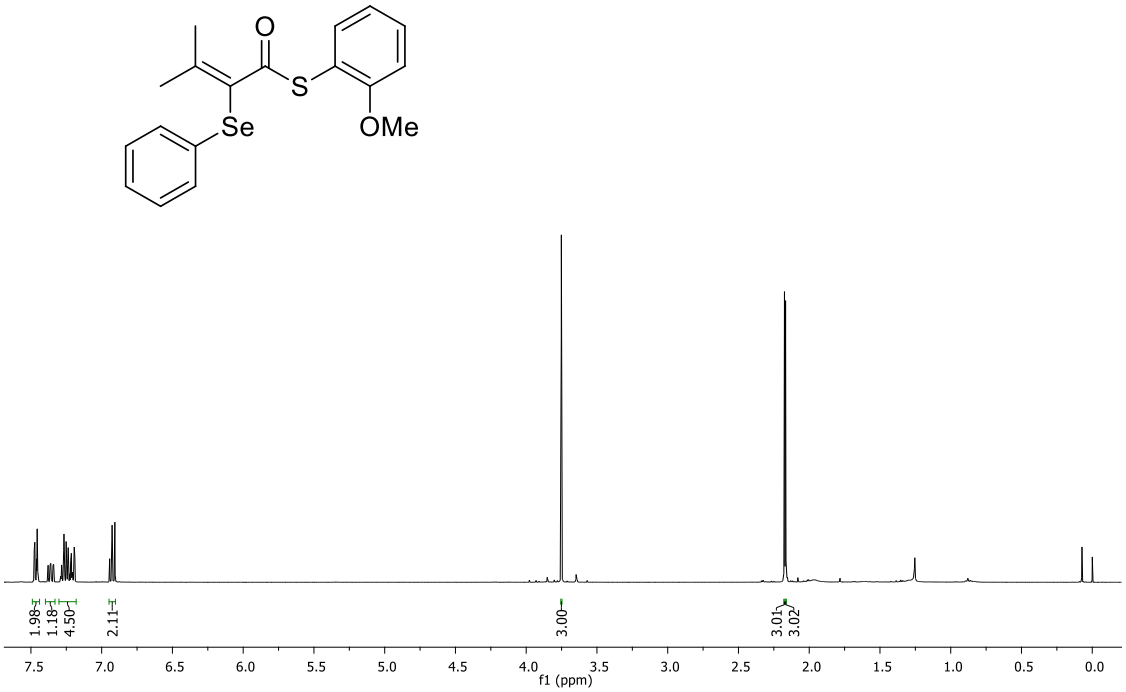
^{13}C NMR spectrum for compound **7** ($CDCl_3$, 100 MHz)



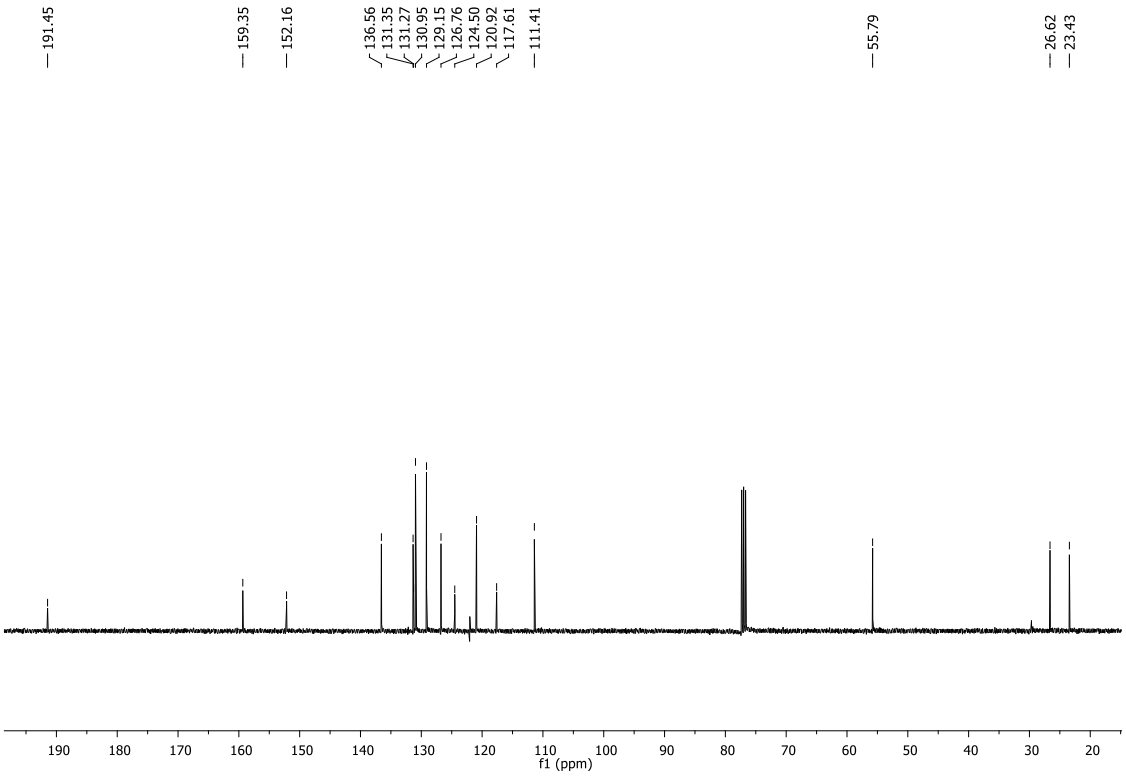
^{1}H NMR spectrum for compound **8** ($CDCl_3$, 400 MHz)



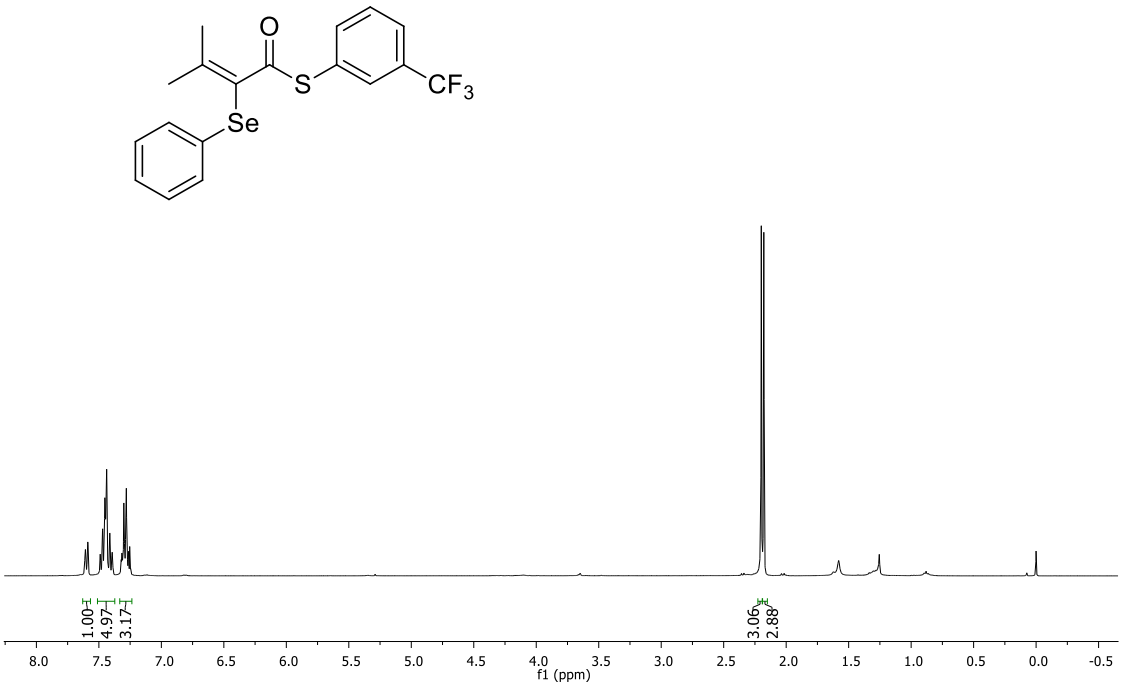
^{13}C NMR spectrum for compound **8** ($CDCl_3$, 100 MHz)



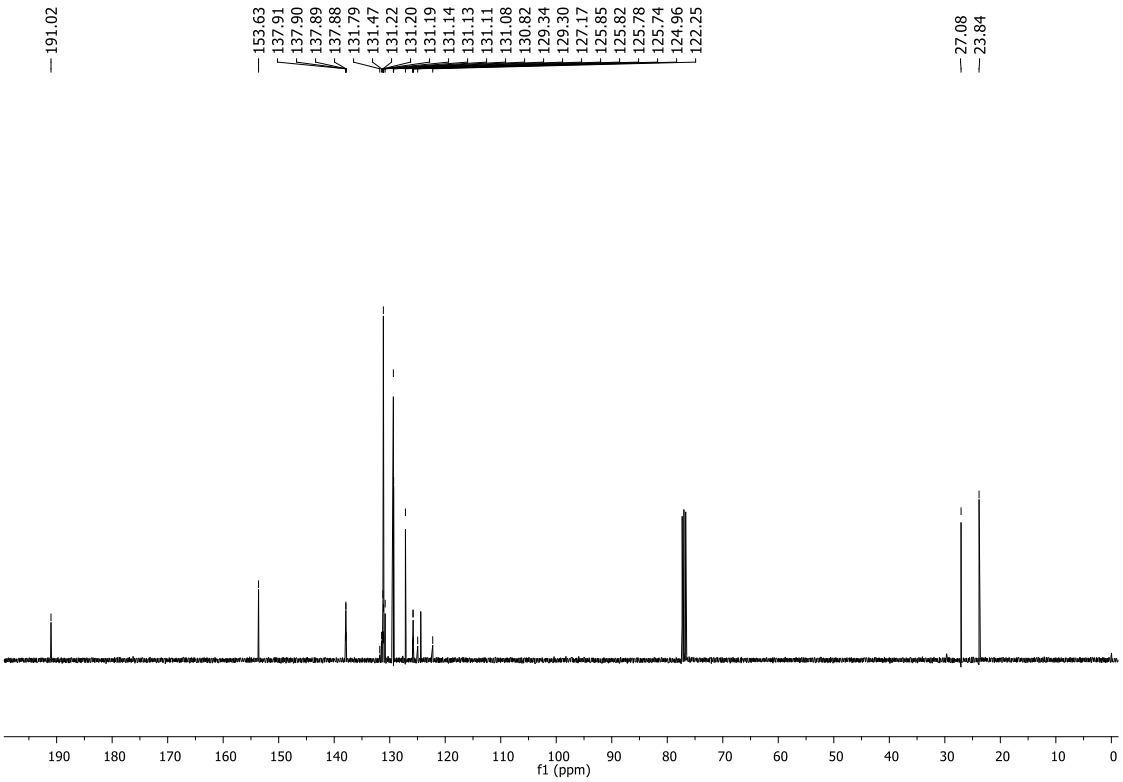
^{1}H NMR spectrum for compound **9** ($CDCl_3$, 400 MHz)



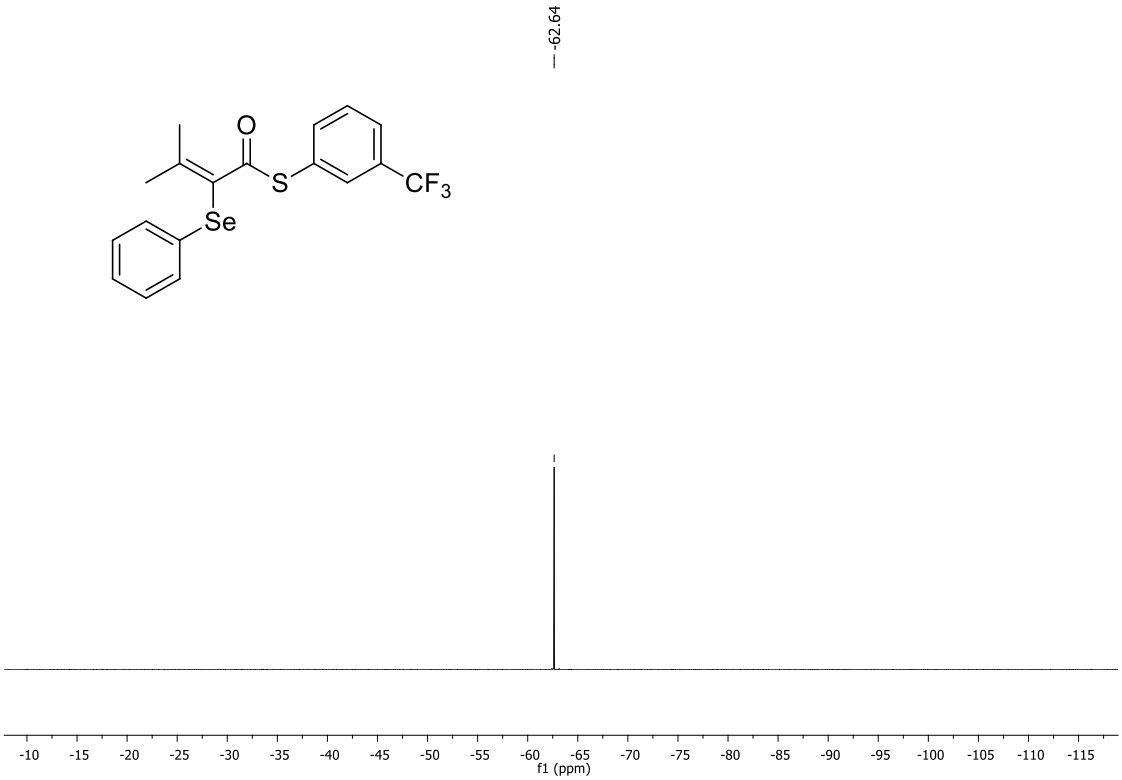
^{13}C NMR spectrum for compound **9** ($CDCl_3$, 100 MHz)



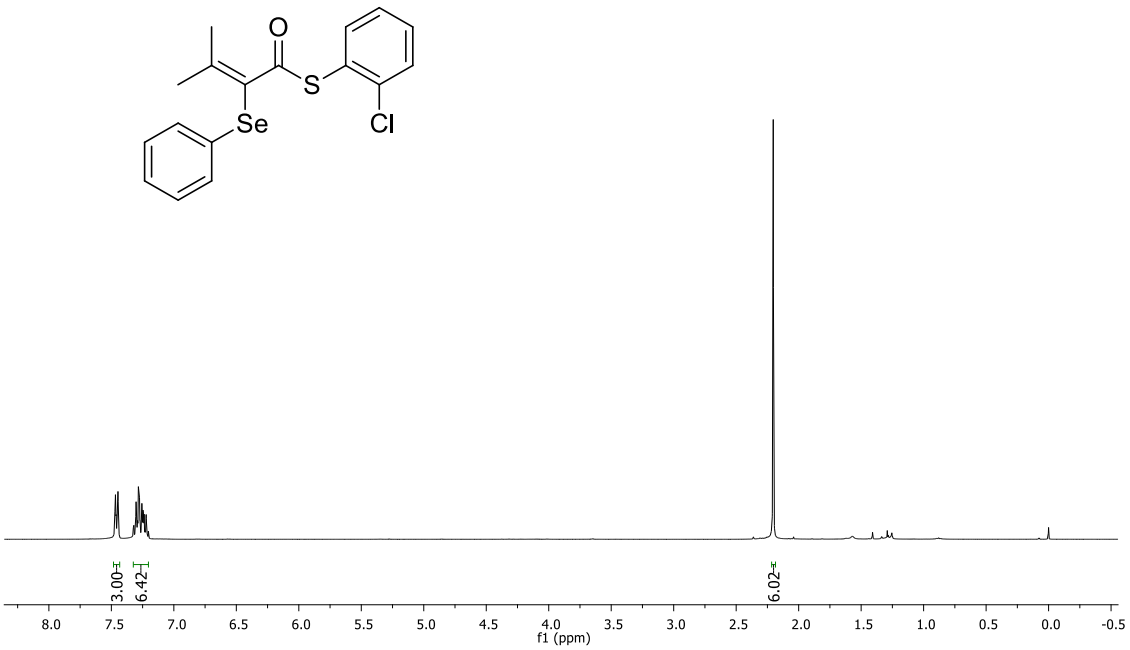
^{1}H NMR spectrum for compound **10** ($CDCl_3$, 400 MHz)



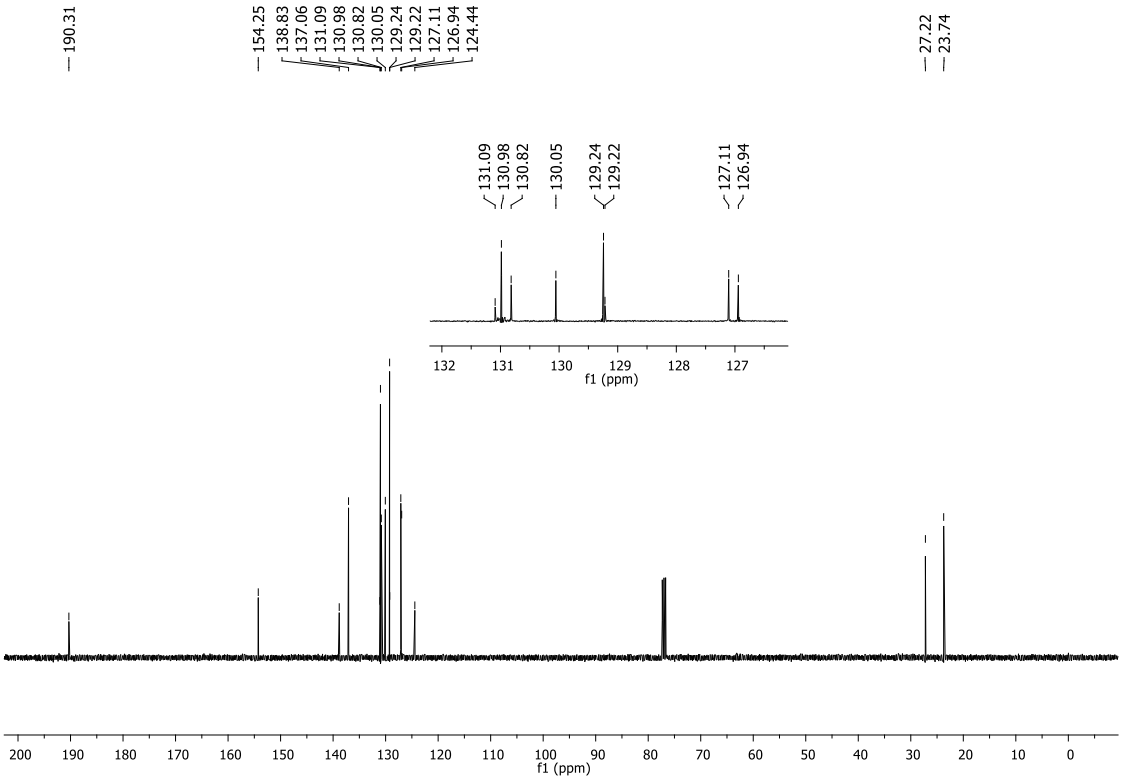
^{13}C NMR spectrum for compound **10** ($CDCl_3$, 100 MHz)



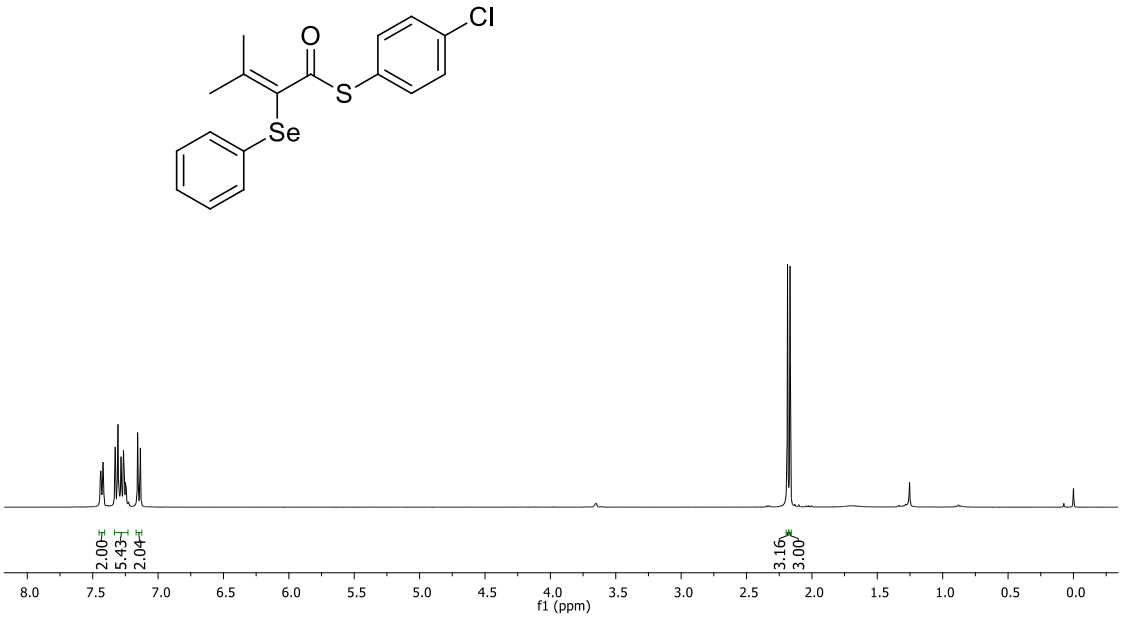
^{19}F NMR spectrum for compound **10** ($CDCl_3$, 376 MHz)



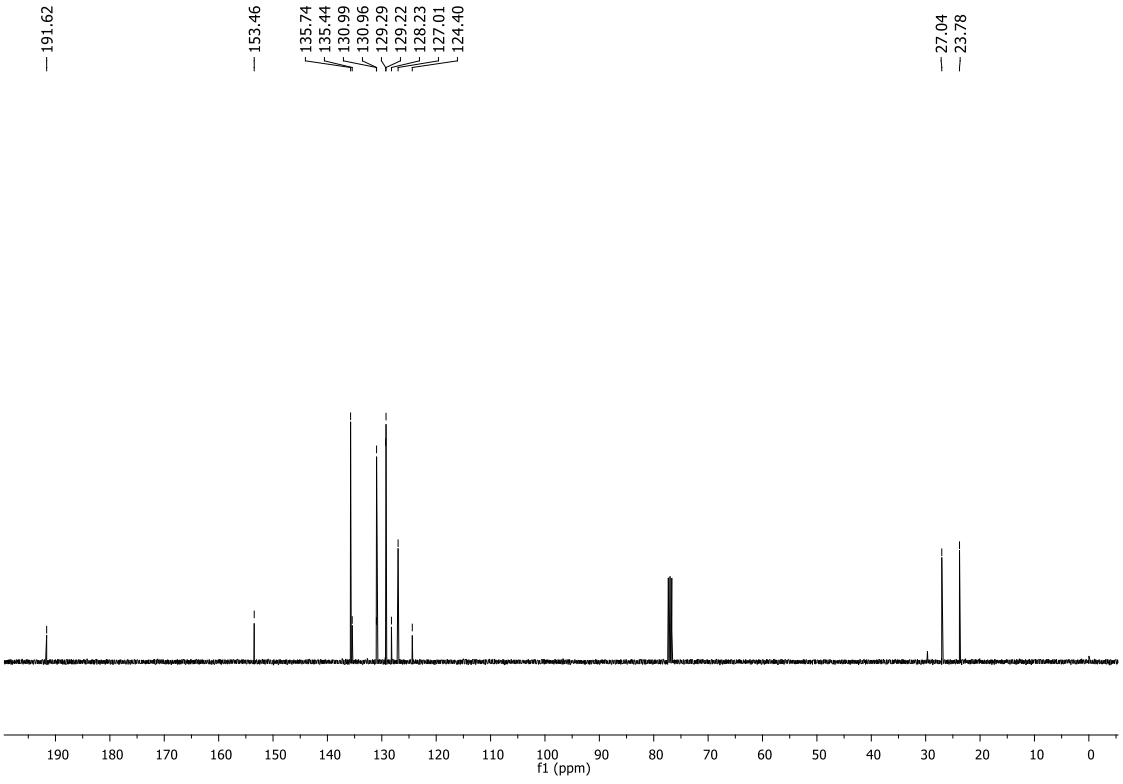
^{1}H NMR spectrum for compound **11** ($CDCl_3$, 400 MHz)



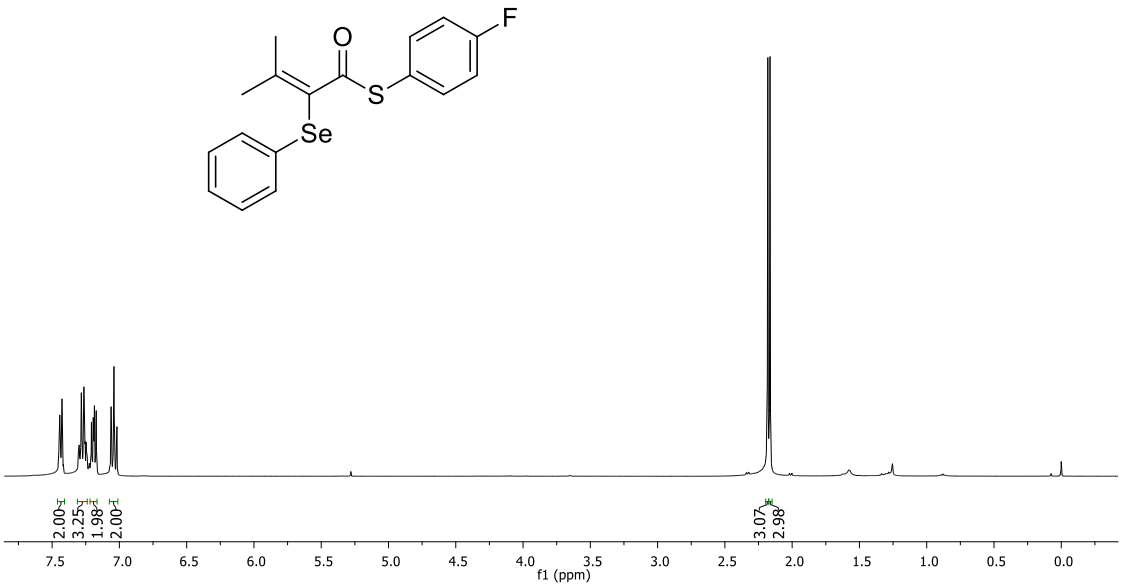
^{13}C NMR spectrum for compound **11** ($CDCl_3$, 100 MHz)



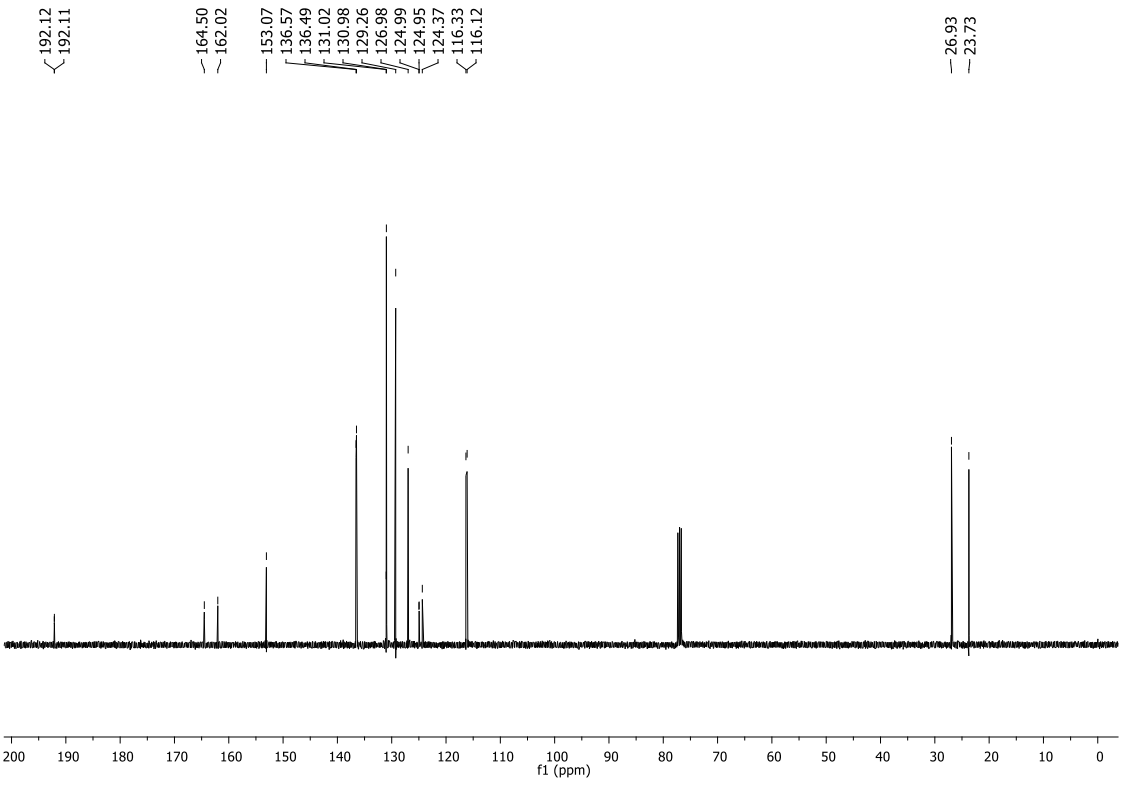
^{1}H NMR spectrum for compound **12** ($CDCl_3$, 400 MHz)



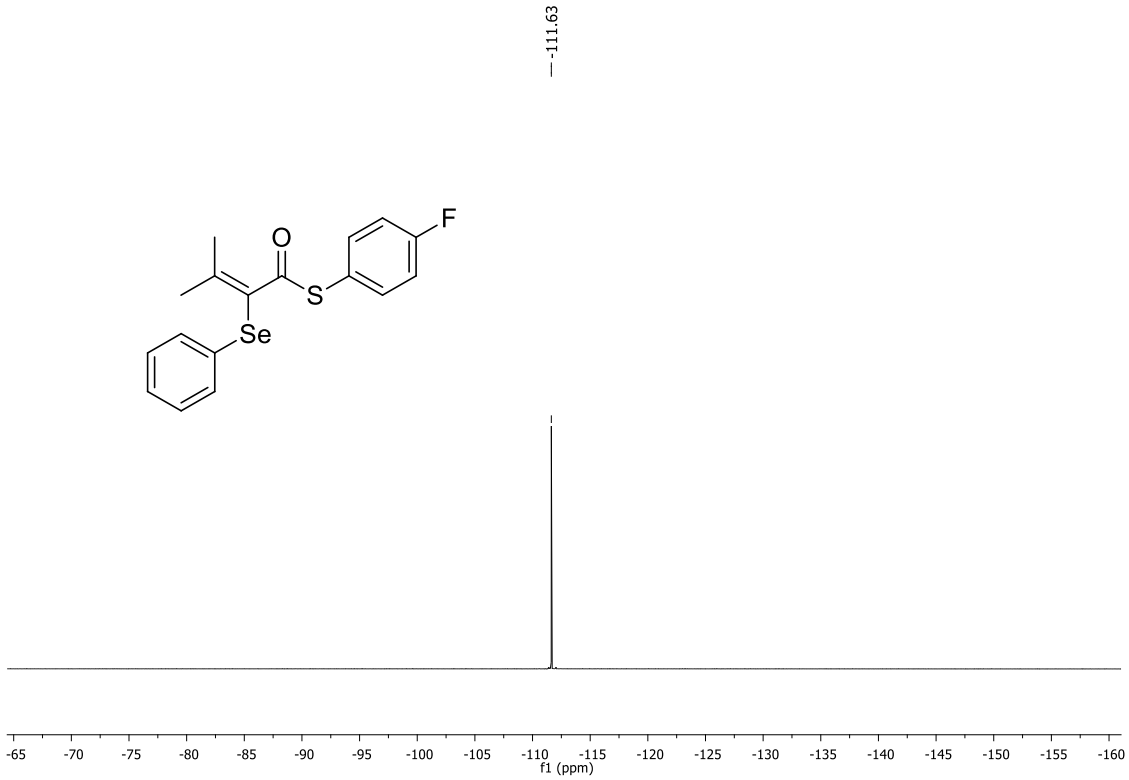
^{13}C NMR spectrum for compound **12** ($CDCl_3$, 100 MHz)



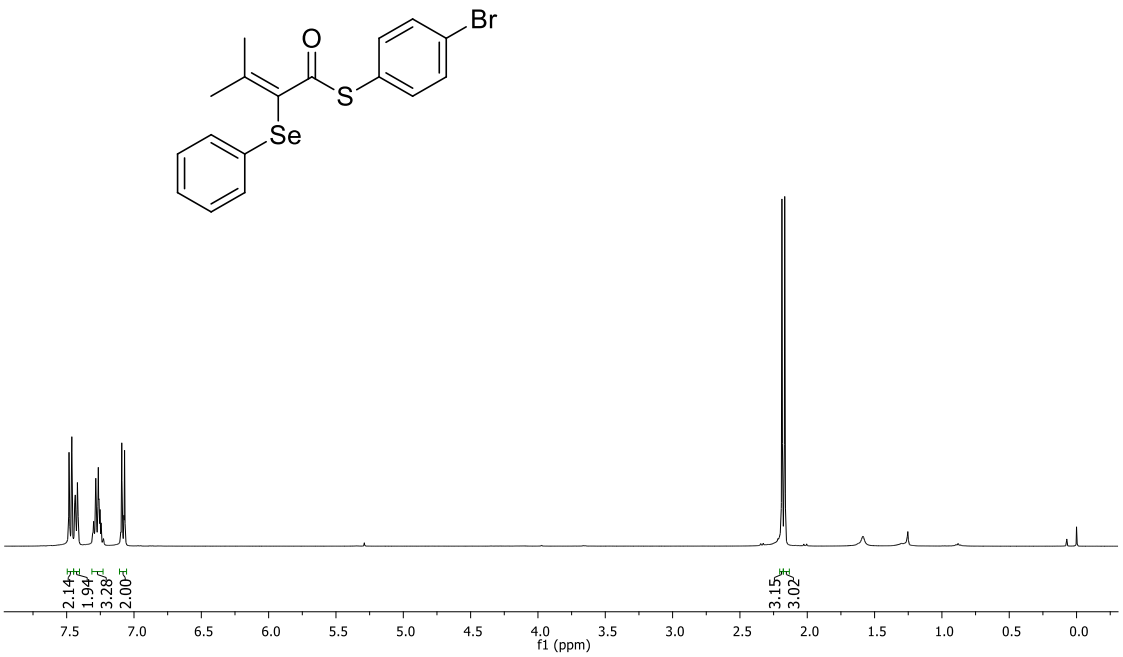
^{1}H NMR spectrum for compound **13** ($CDCl_3$, 400 MHz)



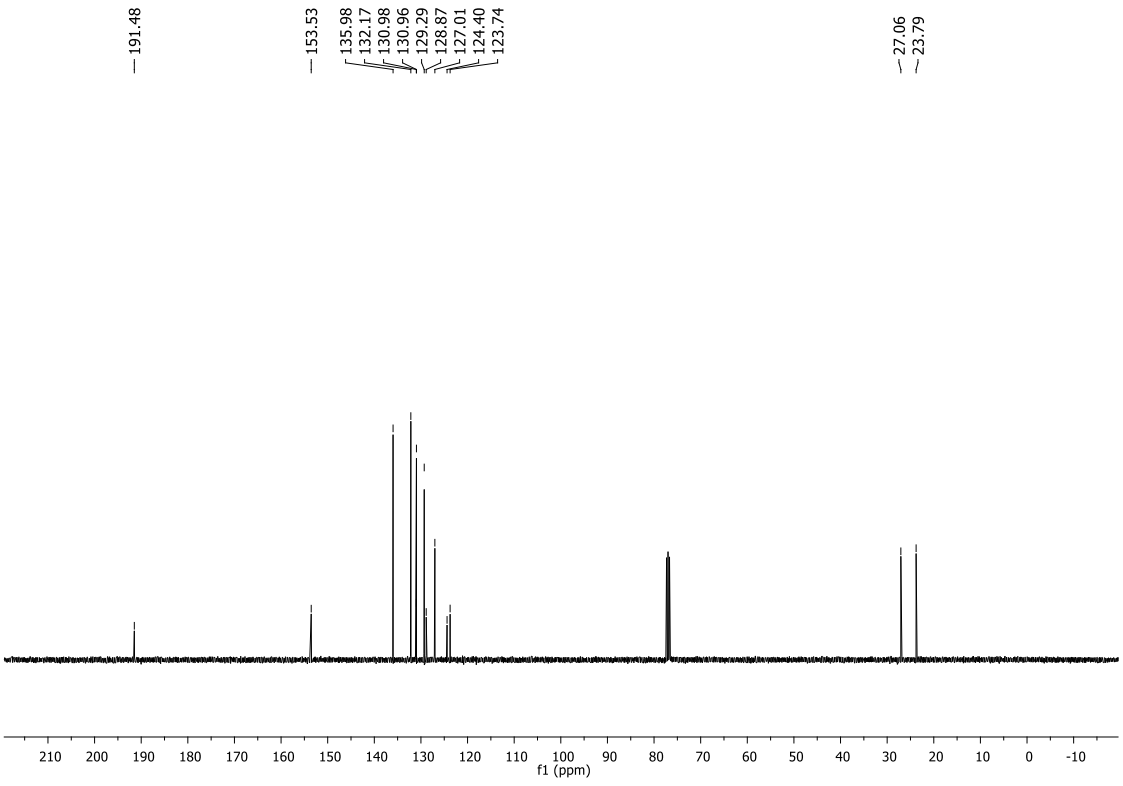
^{13}C NMR spectrum for compound **13** ($CDCl_3$, 100 MHz)



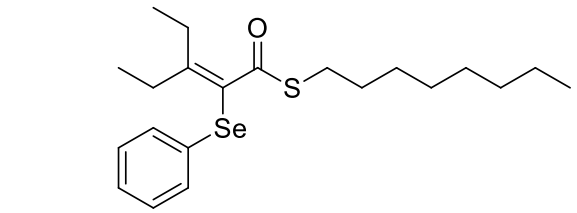
^{19}F NMR spectrum for compound **13** ($CDCl_3$, 376 MHz)

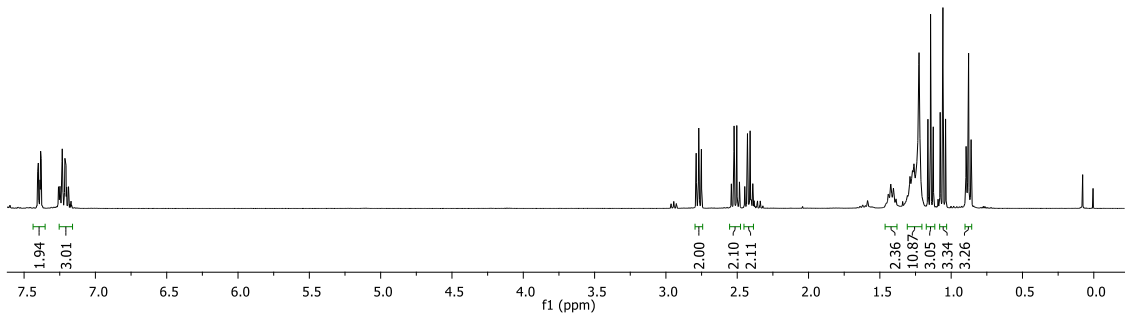


^{1}H NMR spectrum for compound **14** ($CDCl_3$, 400 MHz)

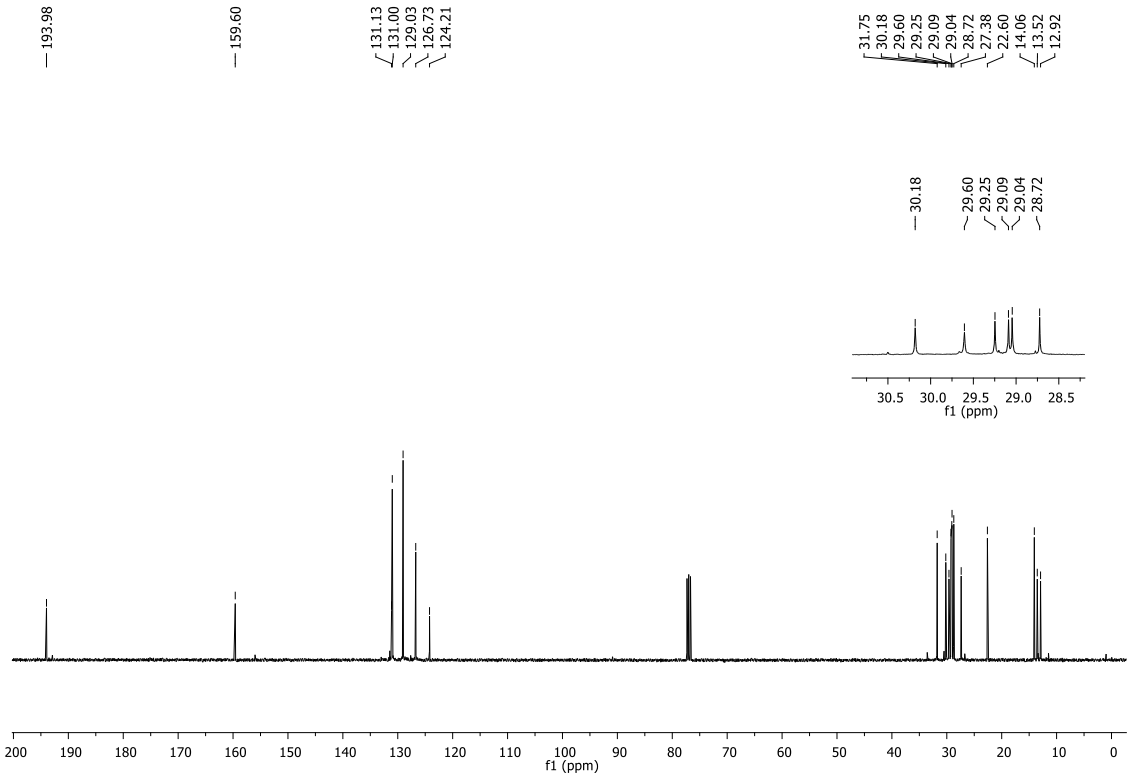


^{13}C NMR spectrum for compound **14** ($CDCl_3$, 100 MHz)

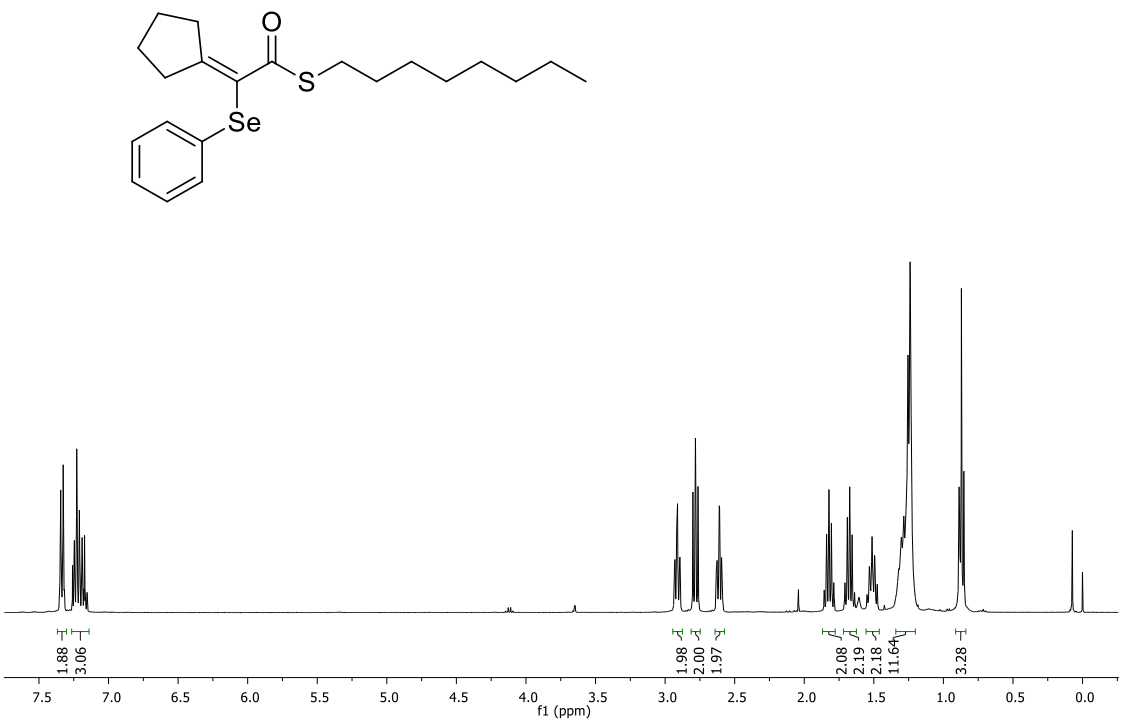




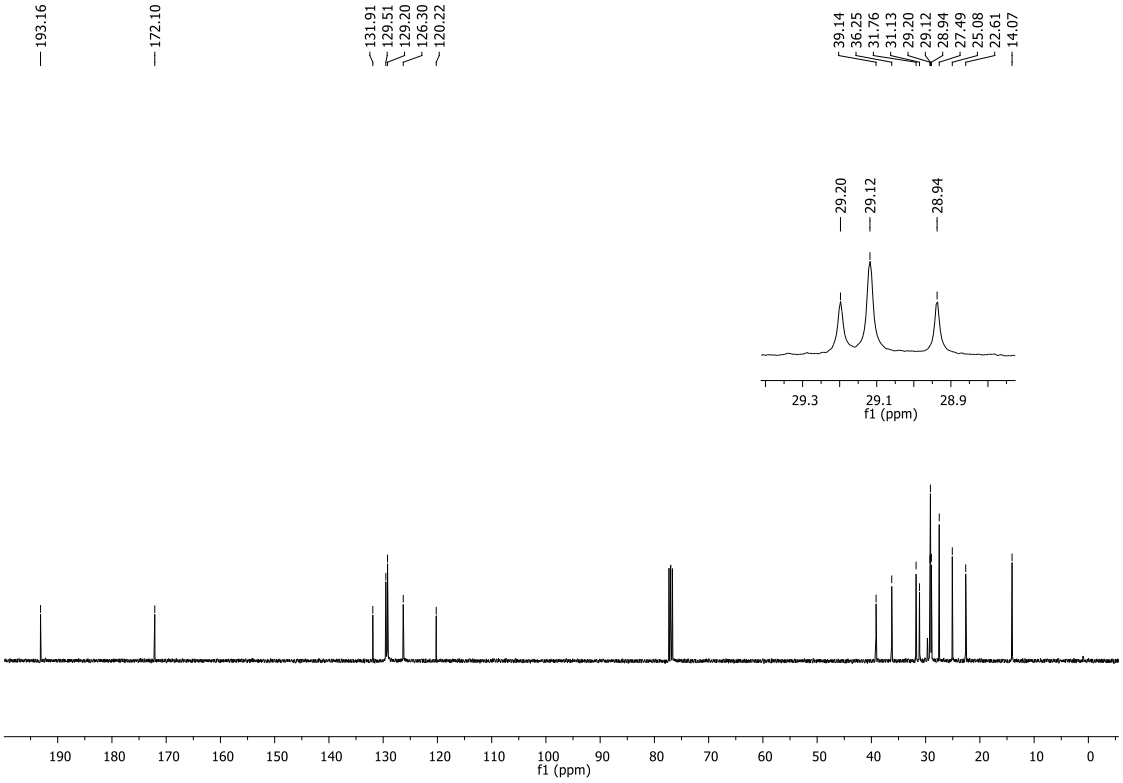
^{1}H NMR spectrum for compound **15** ($CDCl_3$, 400 MHz)



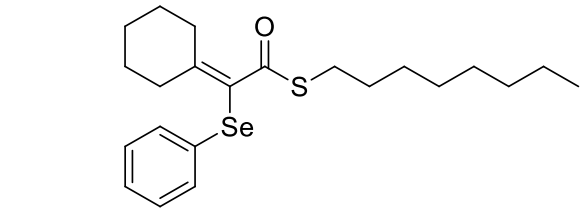
^{13}C NMR spectrum for compound **15** ($CDCl_3$, 100 MHz)

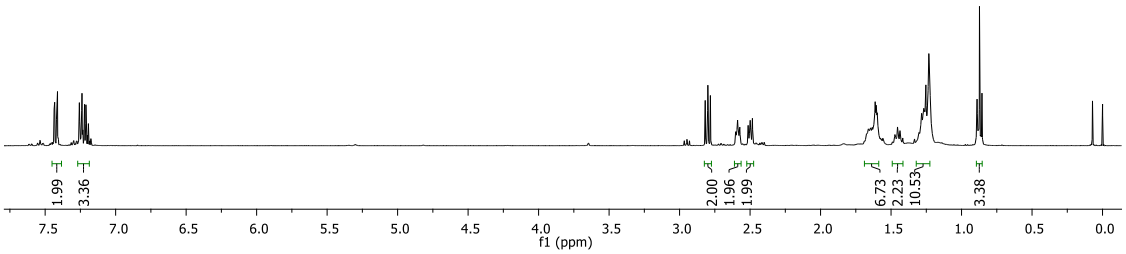


^{1}H NMR spectrum for compound **16** ($CDCl_3$, 400 MHz)

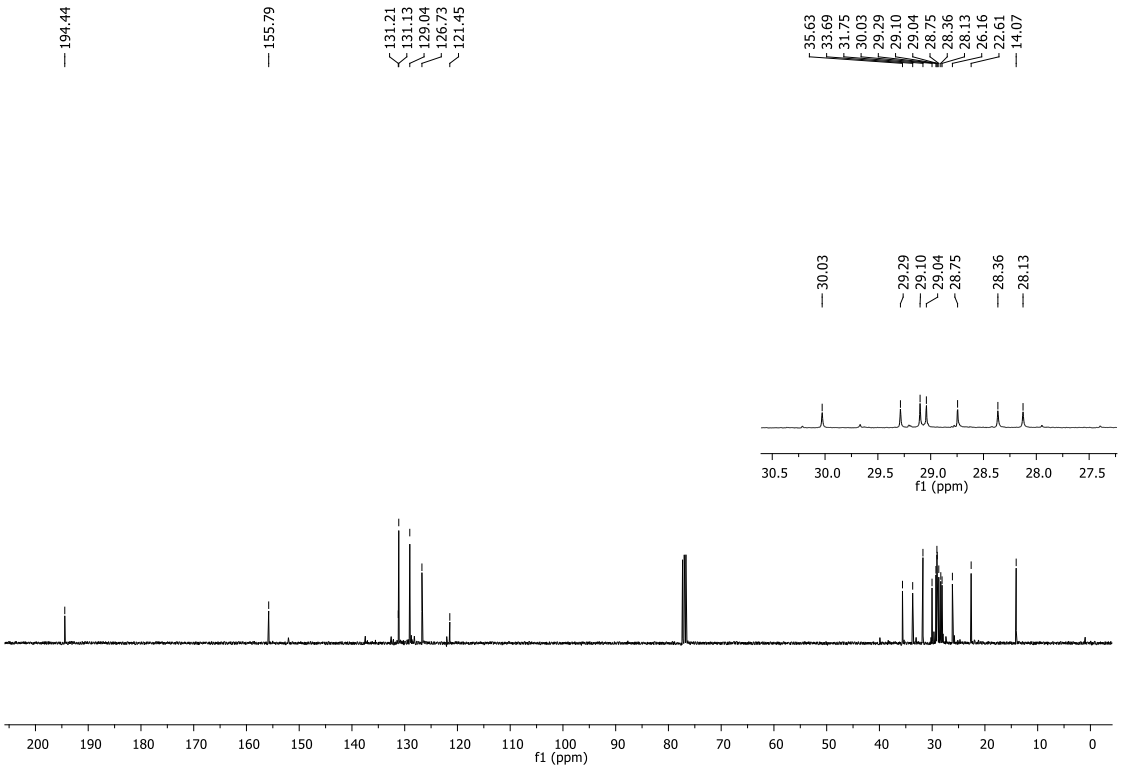


^{13}C NMR spectrum for compound **16** ($CDCl_3$, 100 MHz)

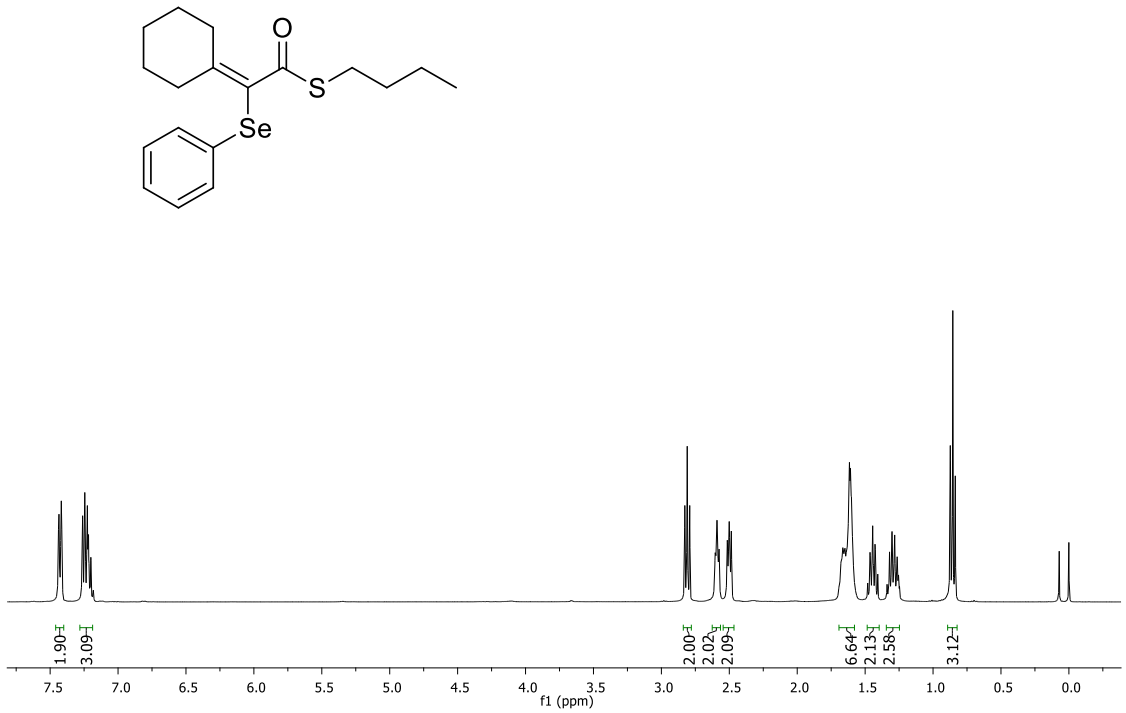




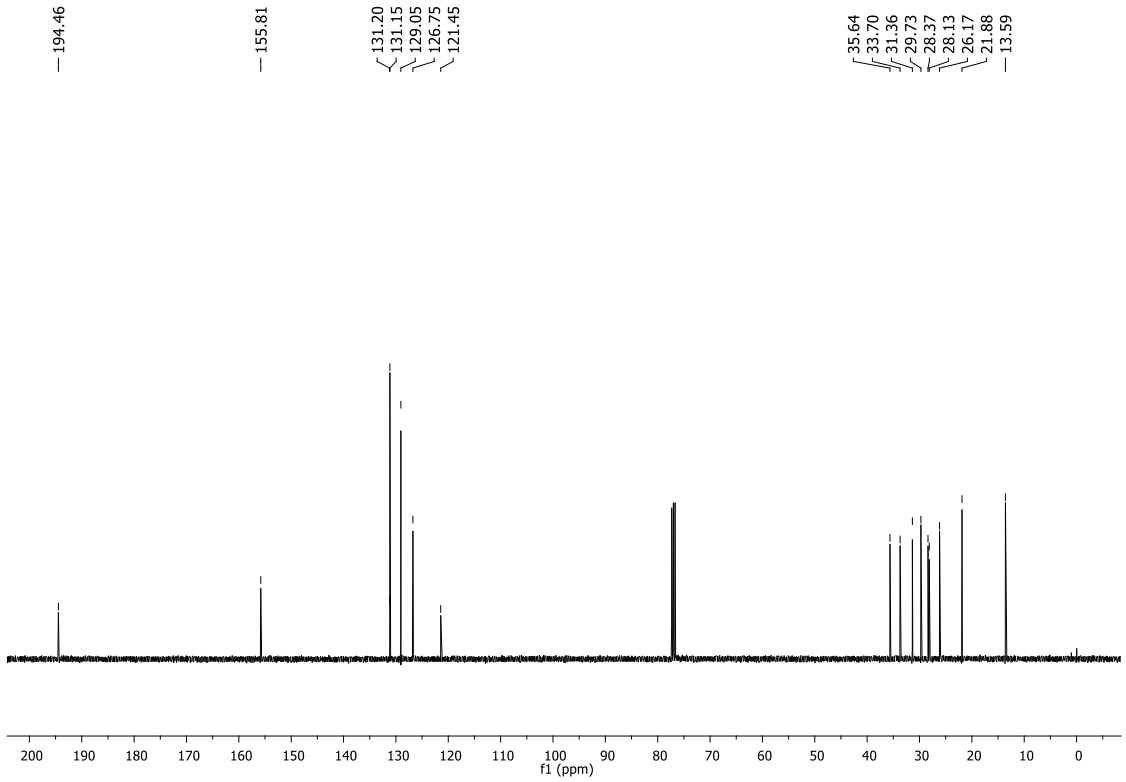
^{1}H NMR spectrum for compound **17** ($CDCl_3$, 400 MHz)



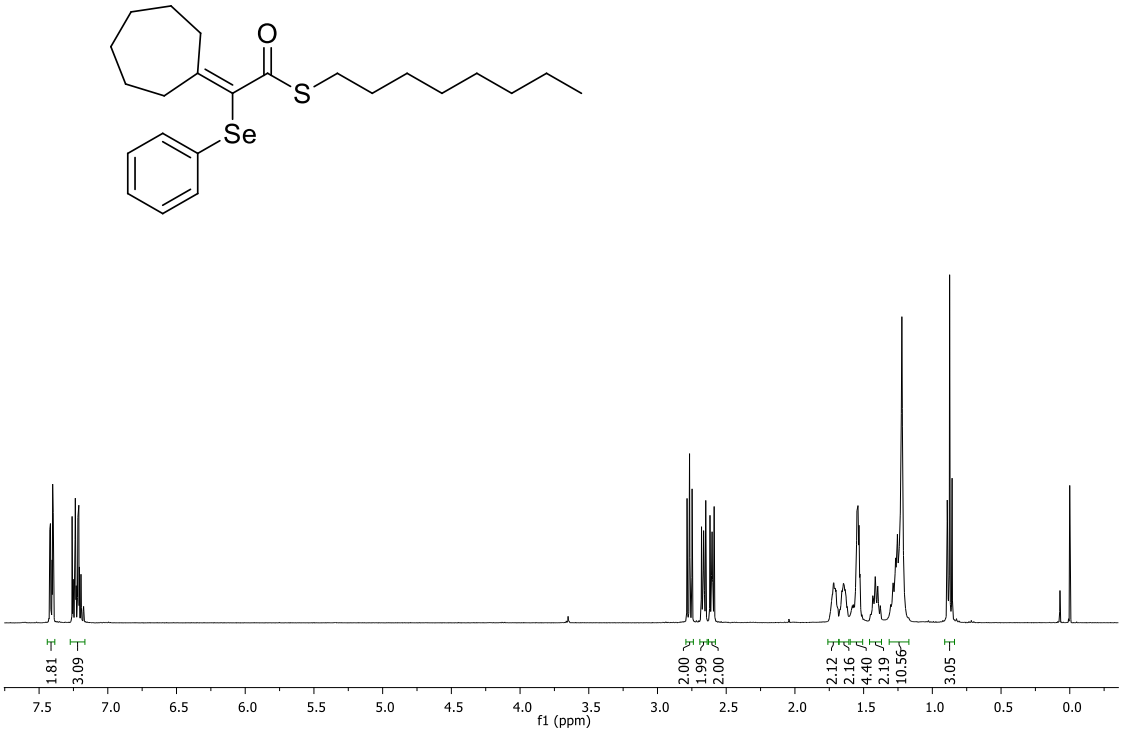
^{13}C NMR spectrum for compound **17** ($CDCl_3$, 100 MHz)



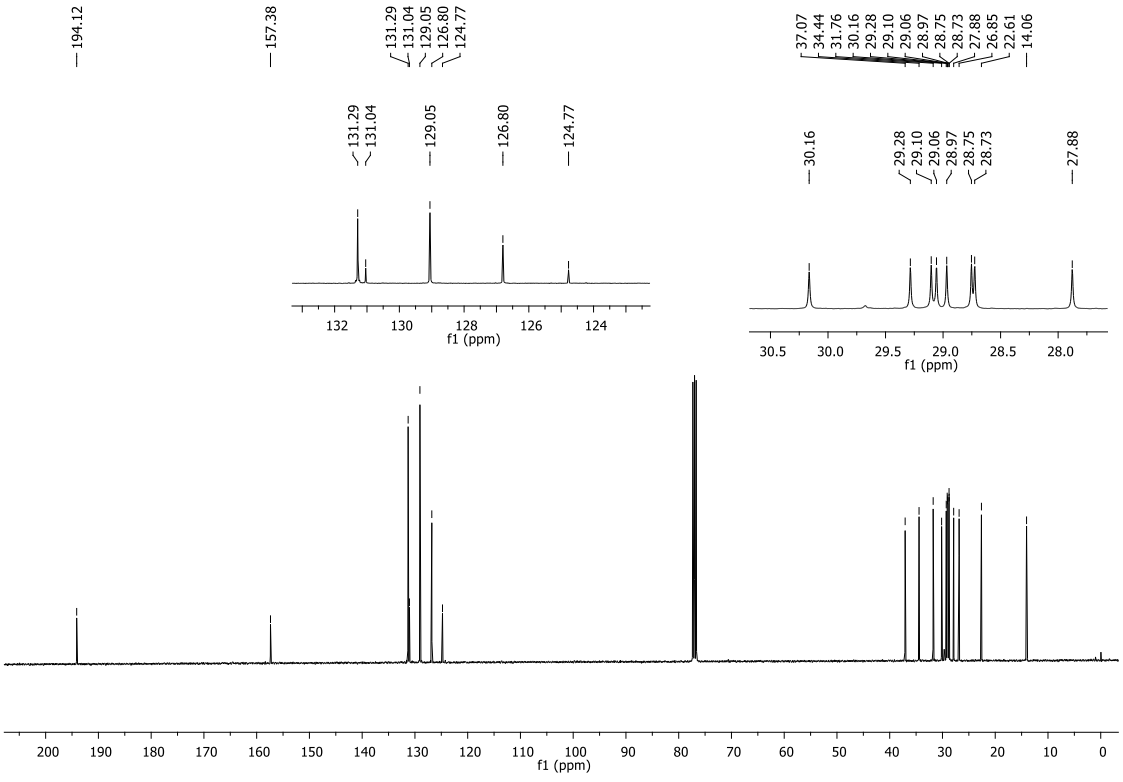
^{1}H NMR spectrum for compound **18** ($CDCl_3$, 400 MHz)



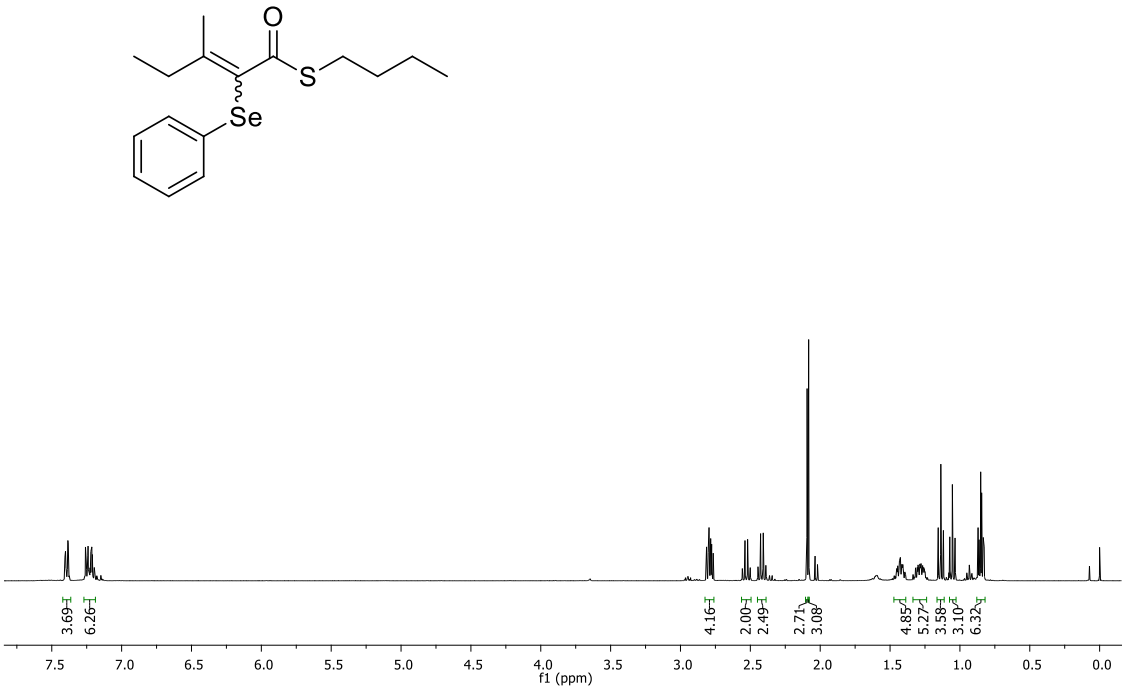
^{13}C NMR spectrum for compound **18** ($CDCl_3$, 100 MHz)



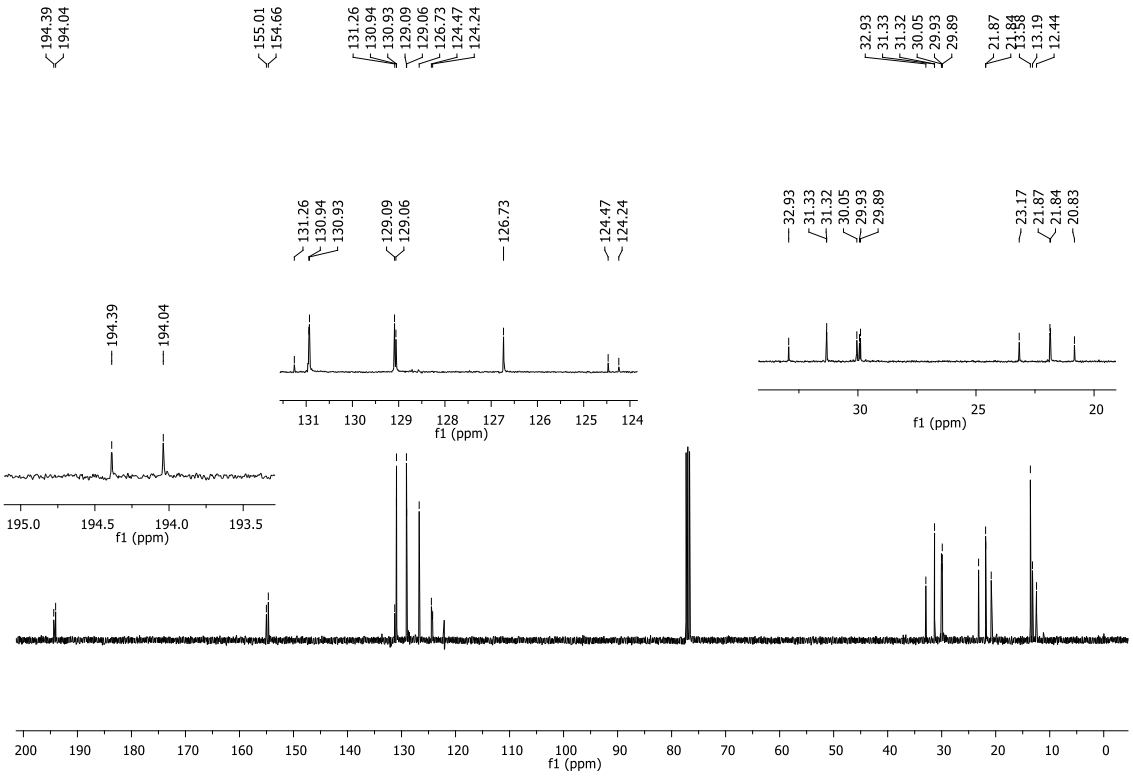
^{1}H NMR spectrum for compound **19** ($CDCl_3$, 400 MHz)



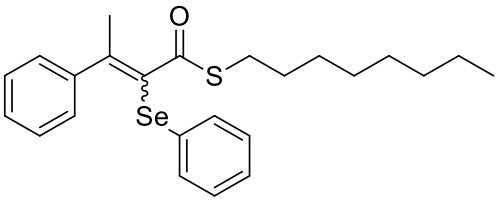
^{13}C NMR spectrum for compound **19** ($CDCl_3$, 100 MHz)

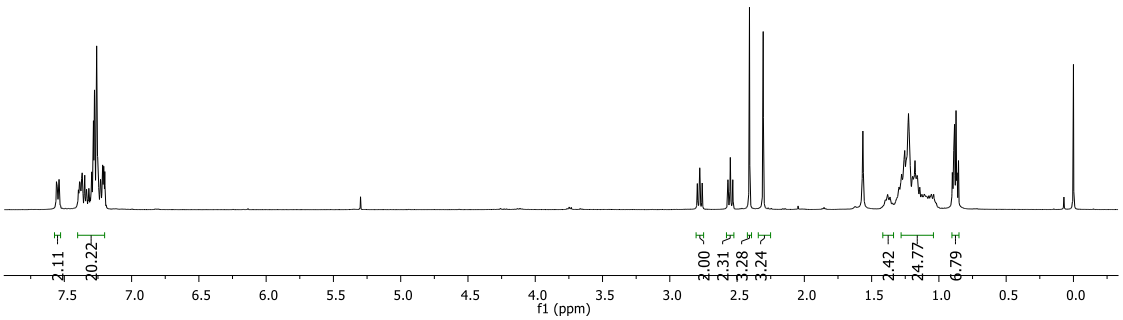


^{1}H NMR spectrum for compound **21** ($CDCl_3$, 400 MHz)

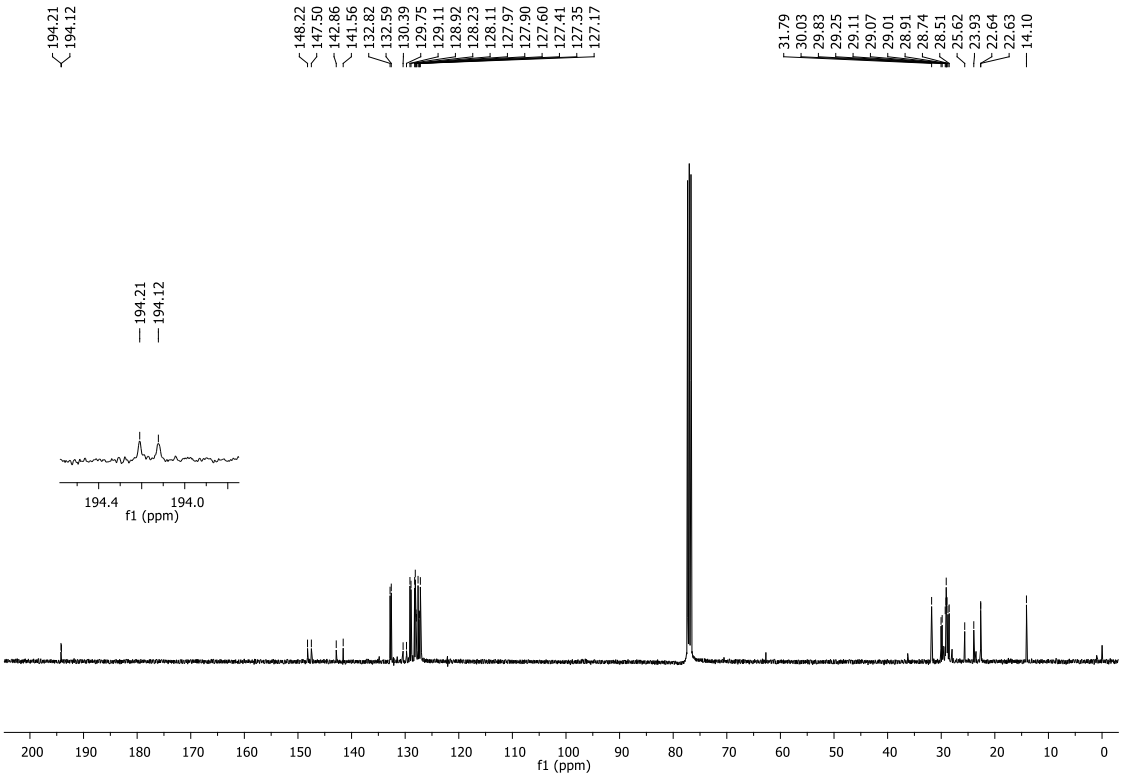


^{13}C NMR spectrum for compound **21** ($CDCl_3$, 100 MHz)

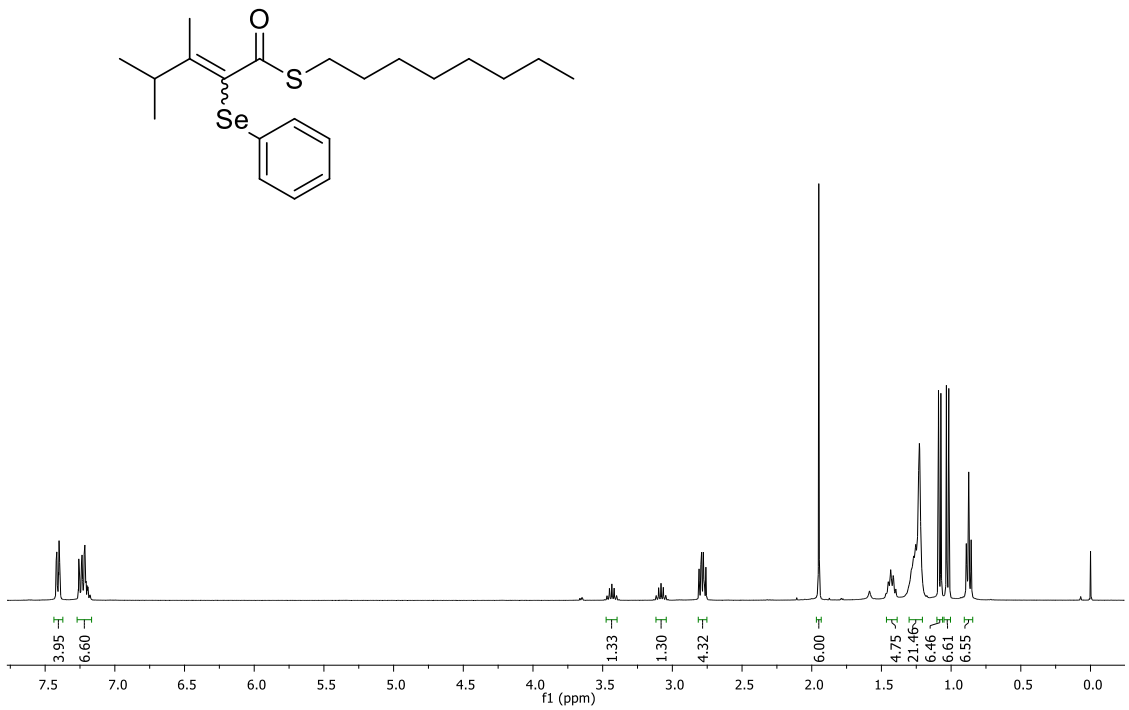




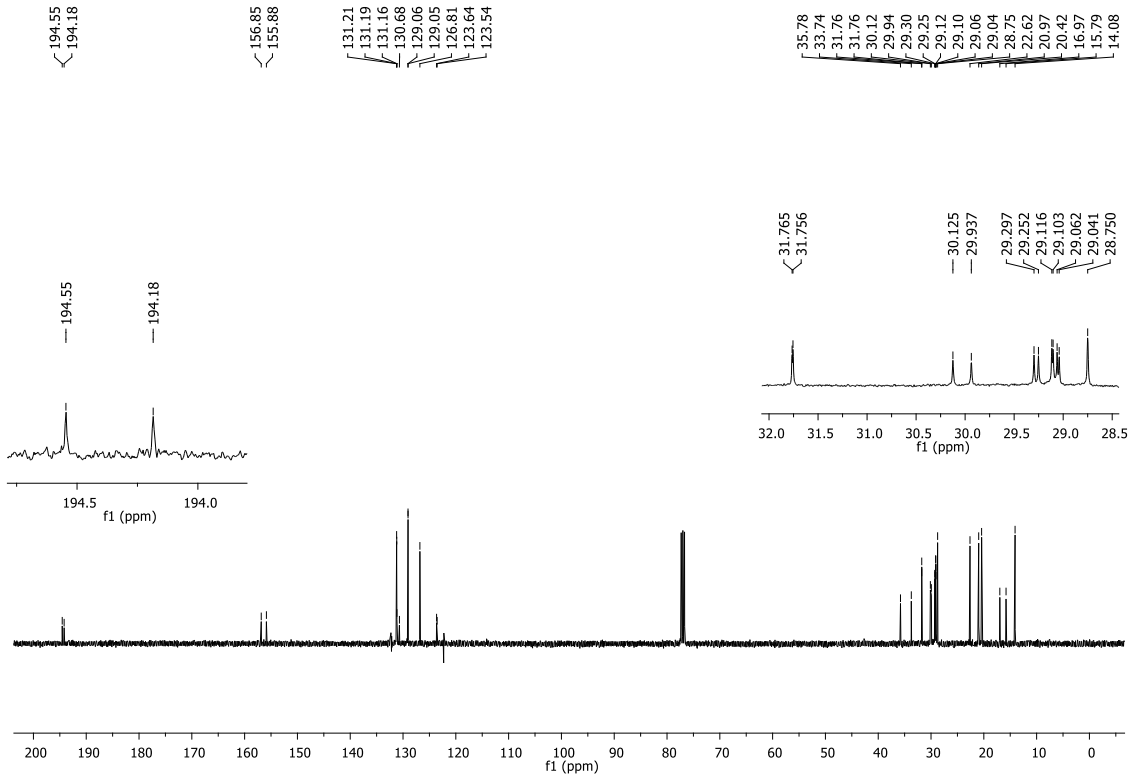
^{1}H NMR spectrum for compound **22** ($CDCl_3$, 400 MHz)



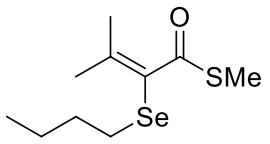
^{13}C NMR spectrum for compound **22** ($CDCl_3$, 100 MHz)

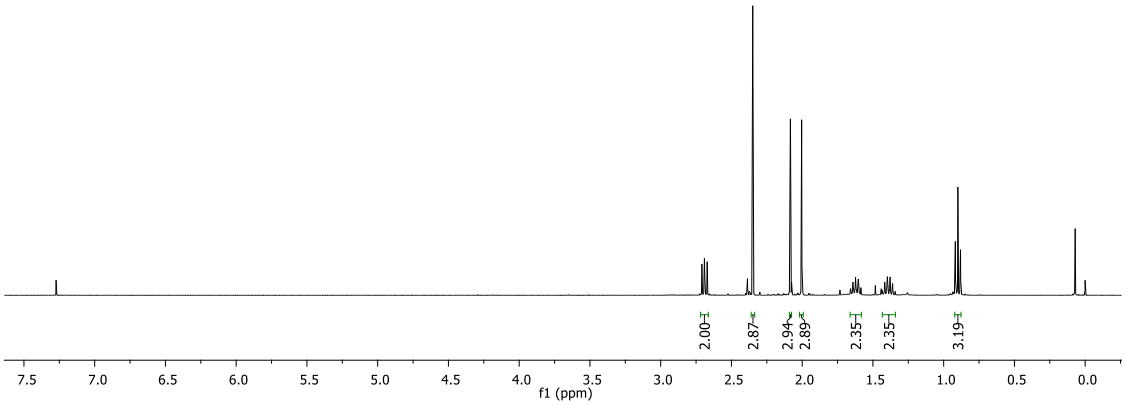


^{1}H NMR spectrum for compound **23** ($CDCl_3$, 400 MHz)

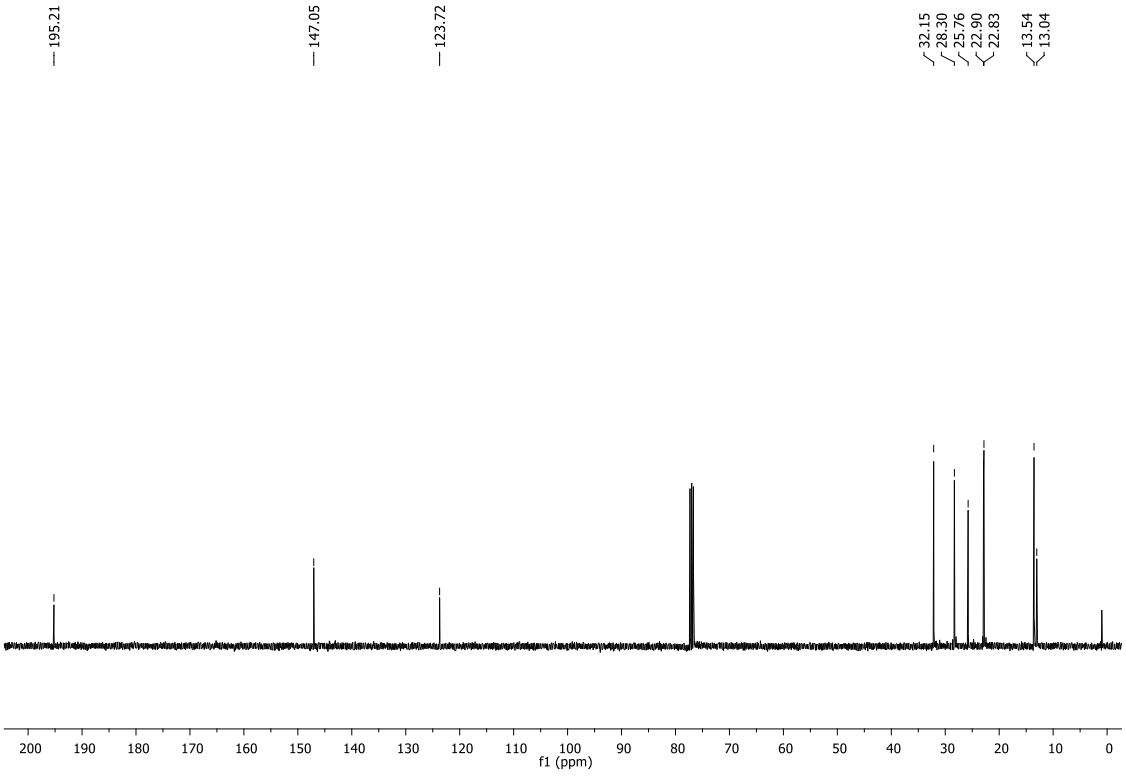


^{13}C NMR spectrum for compound **23** ($CDCl_3$, 100 MHz)

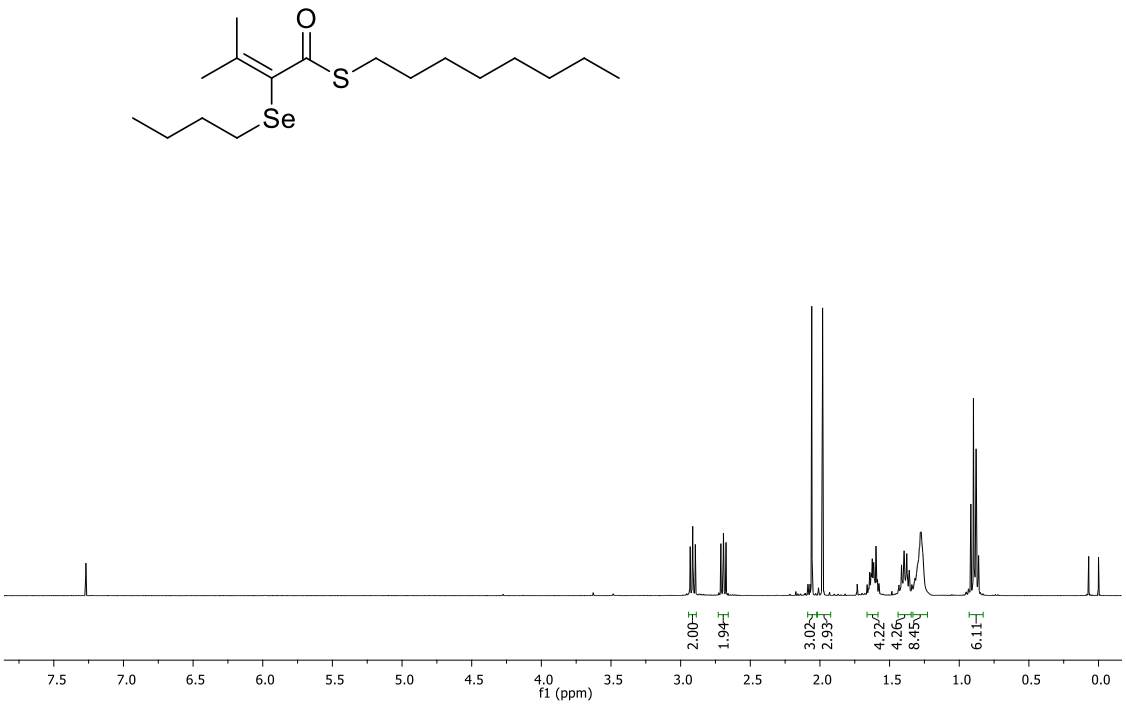




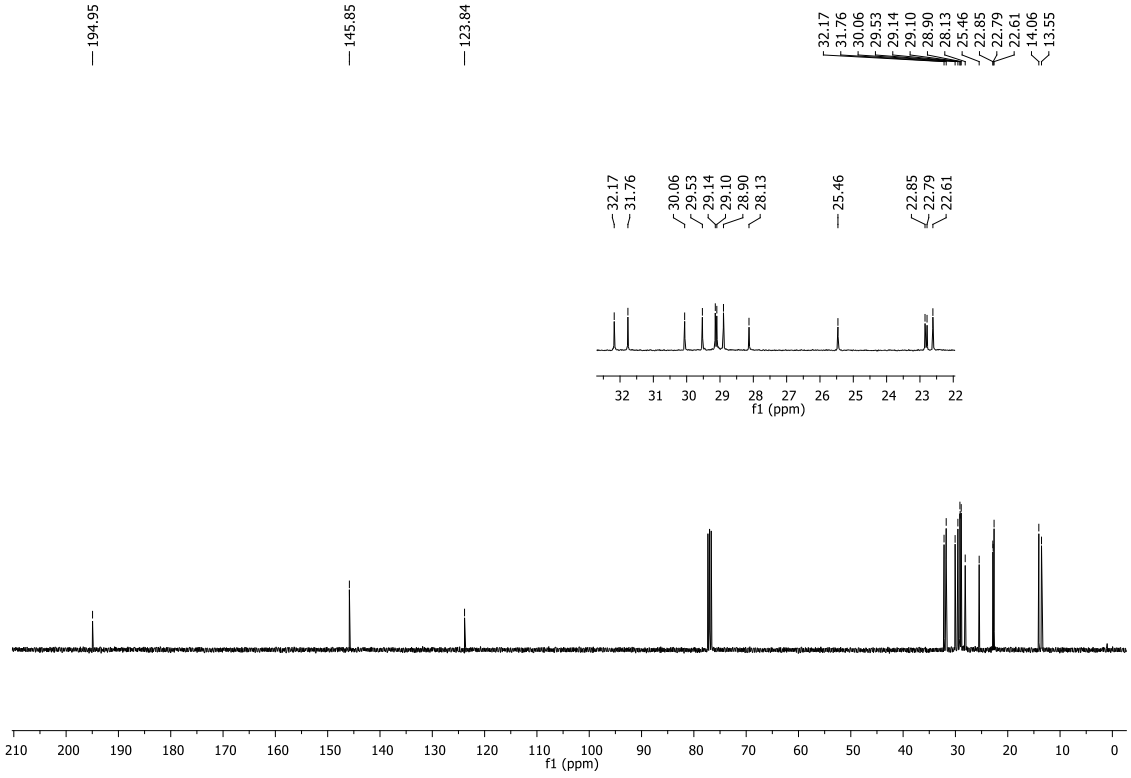
^{1}H NMR spectrum for compound **24** ($CDCl_3$, 400 MHz)



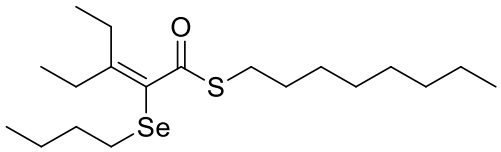
^{13}C NMR spectrum for compound **24** ($CDCl_3$, 100 MHz)

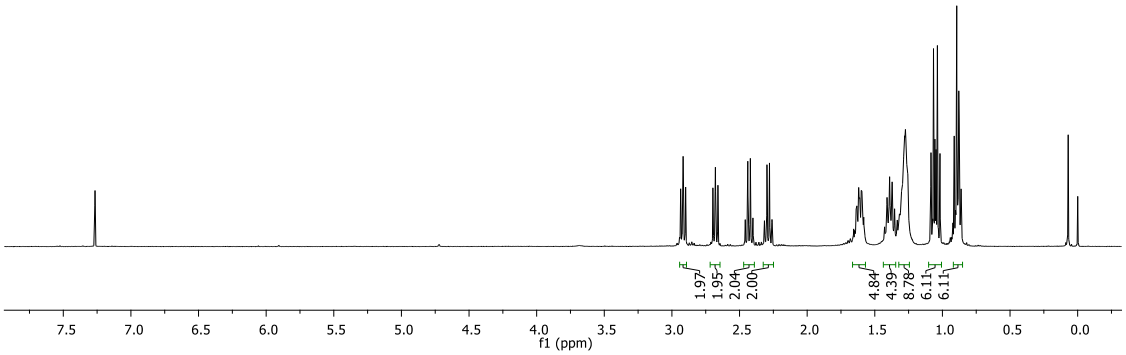


^{1}H NMR spectrum for compound **25** ($CDCl_3$, 400 MHz)

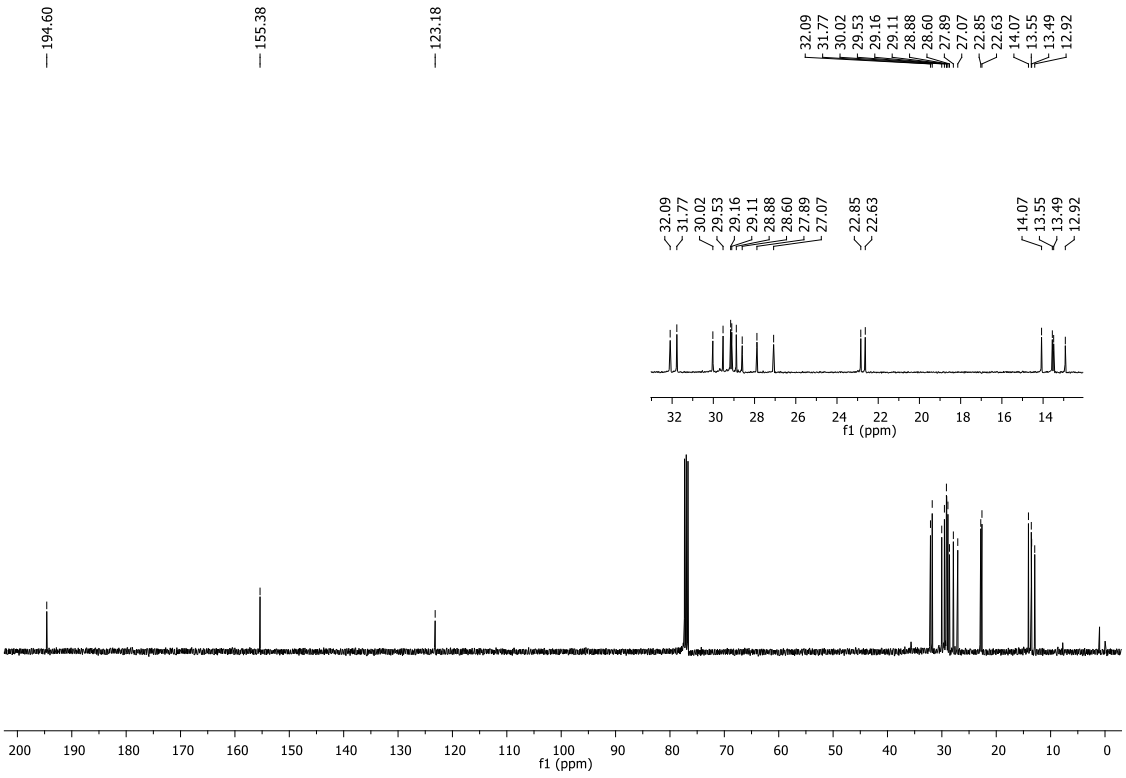


^{13}C NMR spectrum for compound **25** ($CDCl_3$, 100 MHz)

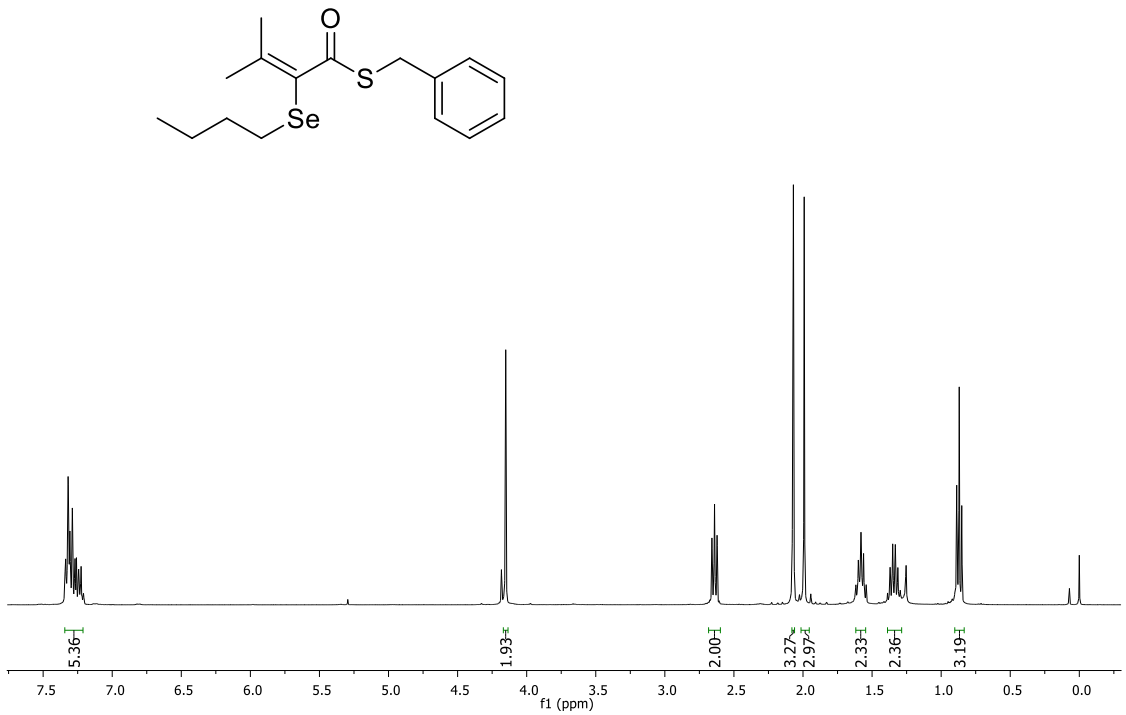




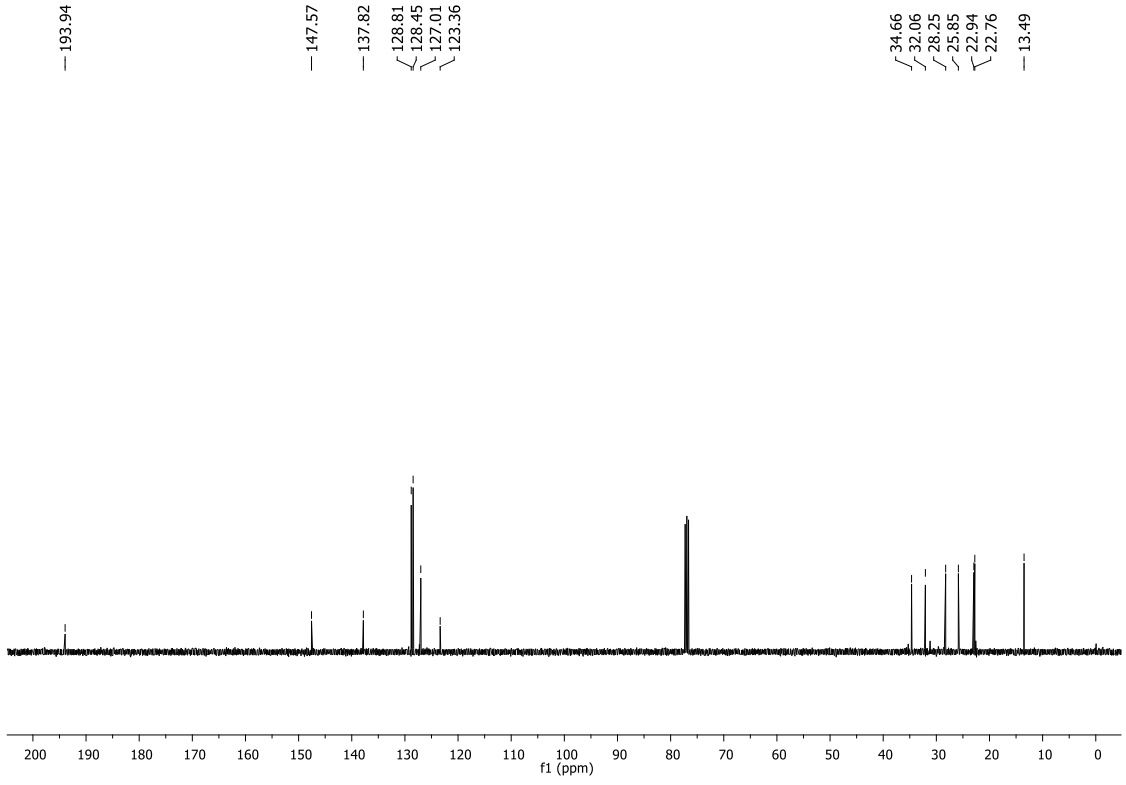
^{1}H NMR spectrum for compound **26** ($CDCl_3$, 400 MHz)



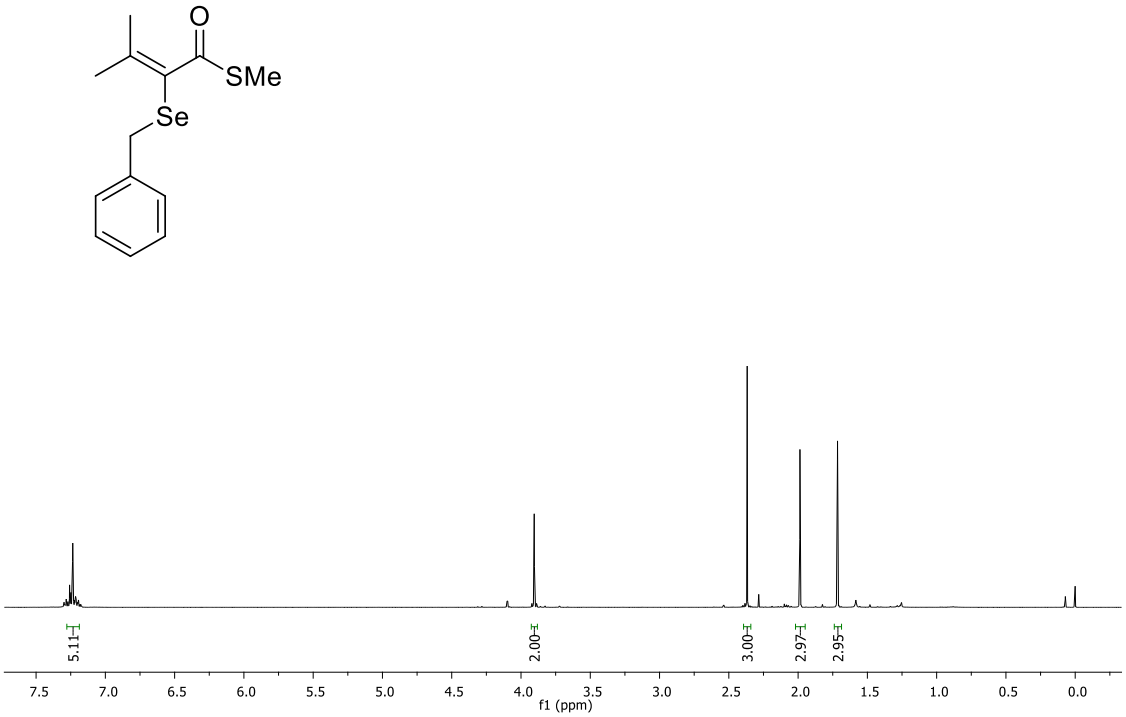
^{13}C NMR spectrum for compound **26** ($CDCl_3$, 100 MHz)



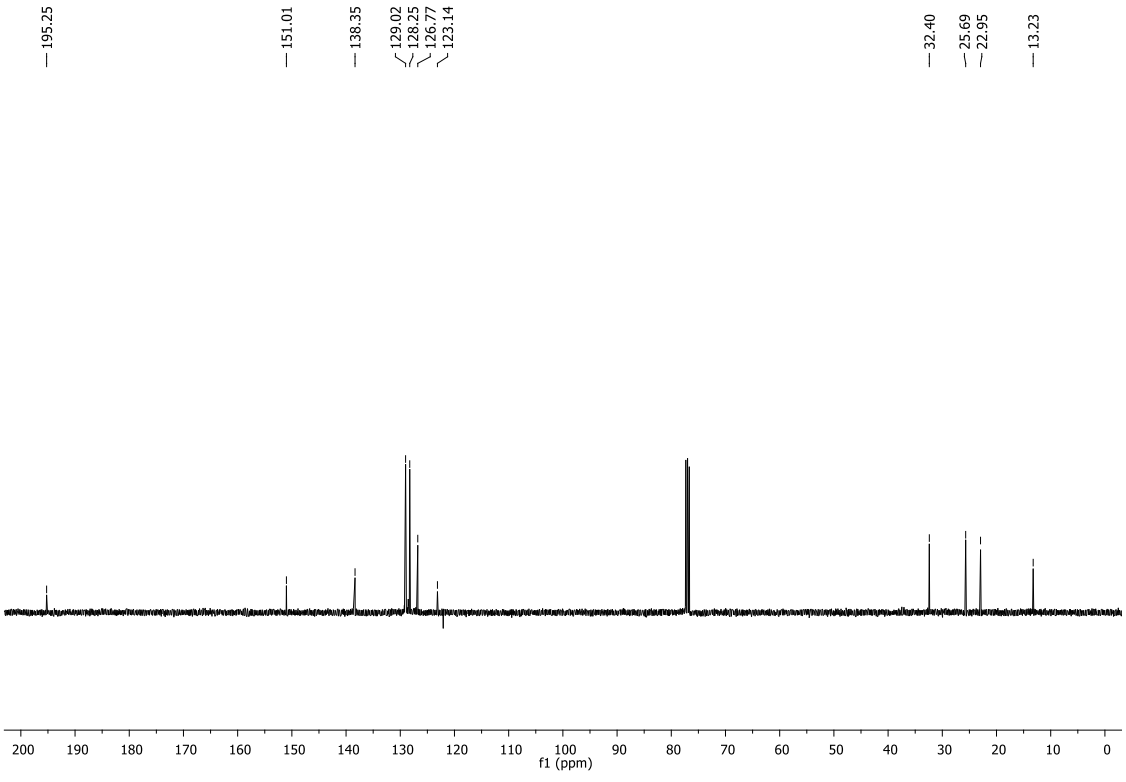
^{1}H NMR spectrum for compound **27** ($CDCl_3$, 400 MHz)



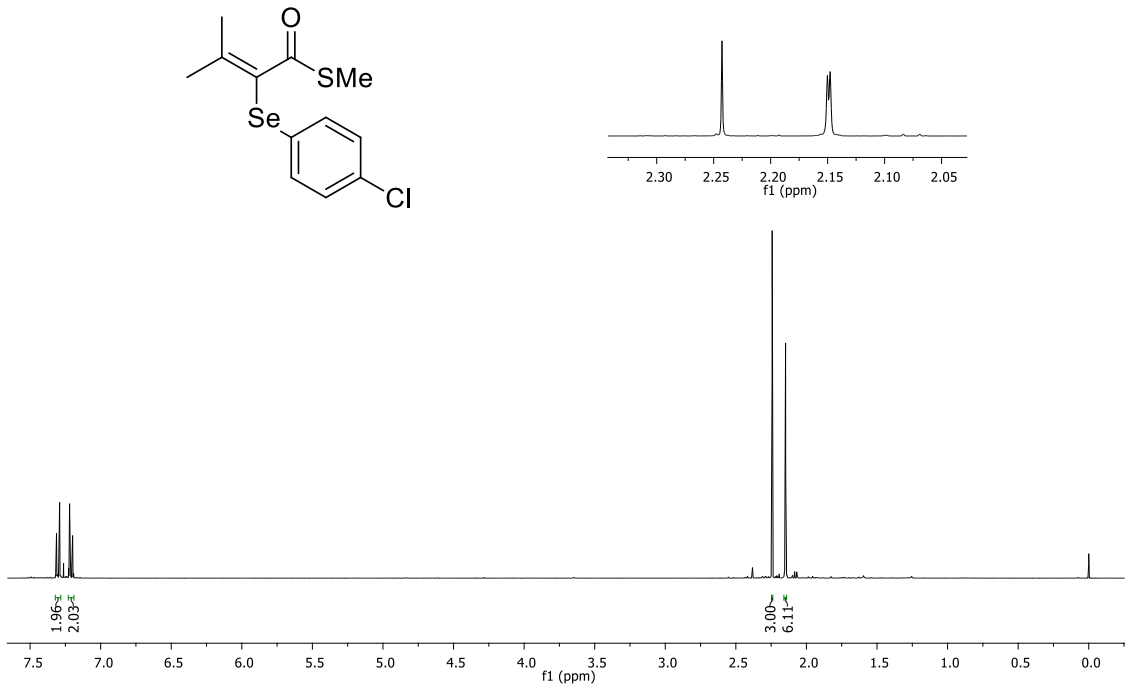
^{13}C NMR spectrum for compound **27** ($CDCl_3$, 100 MHz)



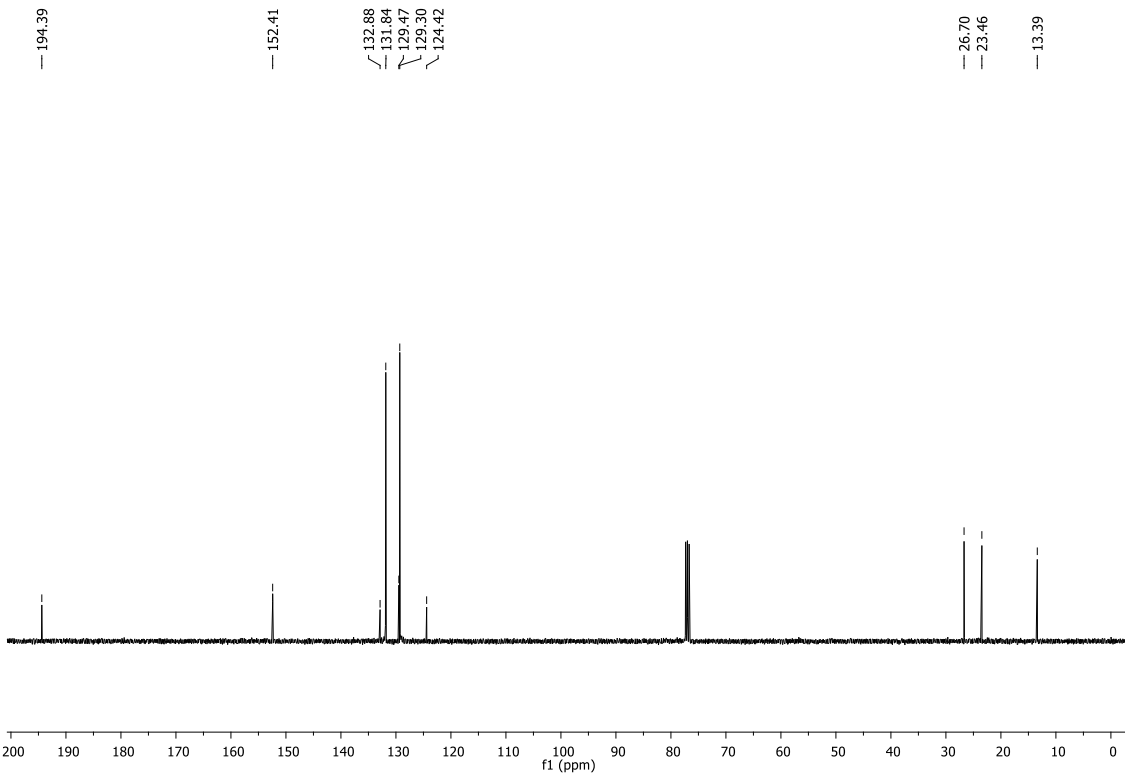
^{1}H NMR spectrum for compound **28** ($CDCl_3$, 400 MHz)



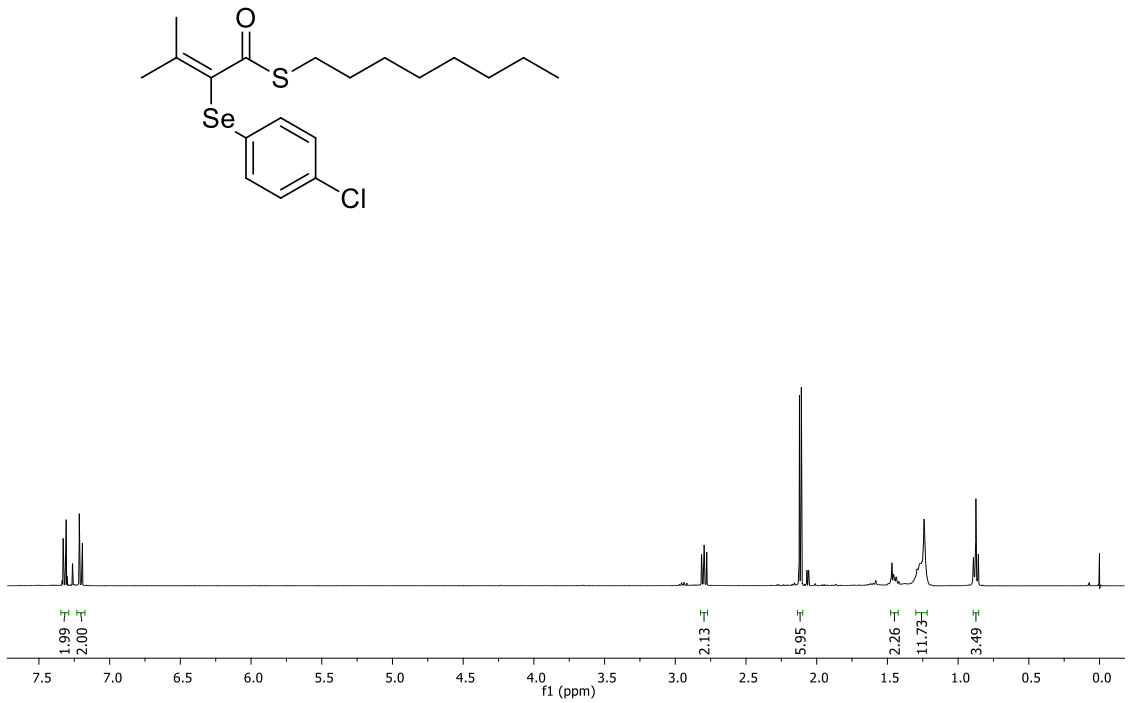
^{13}C NMR spectrum for compound **28** ($CDCl_3$, 100 MHz)



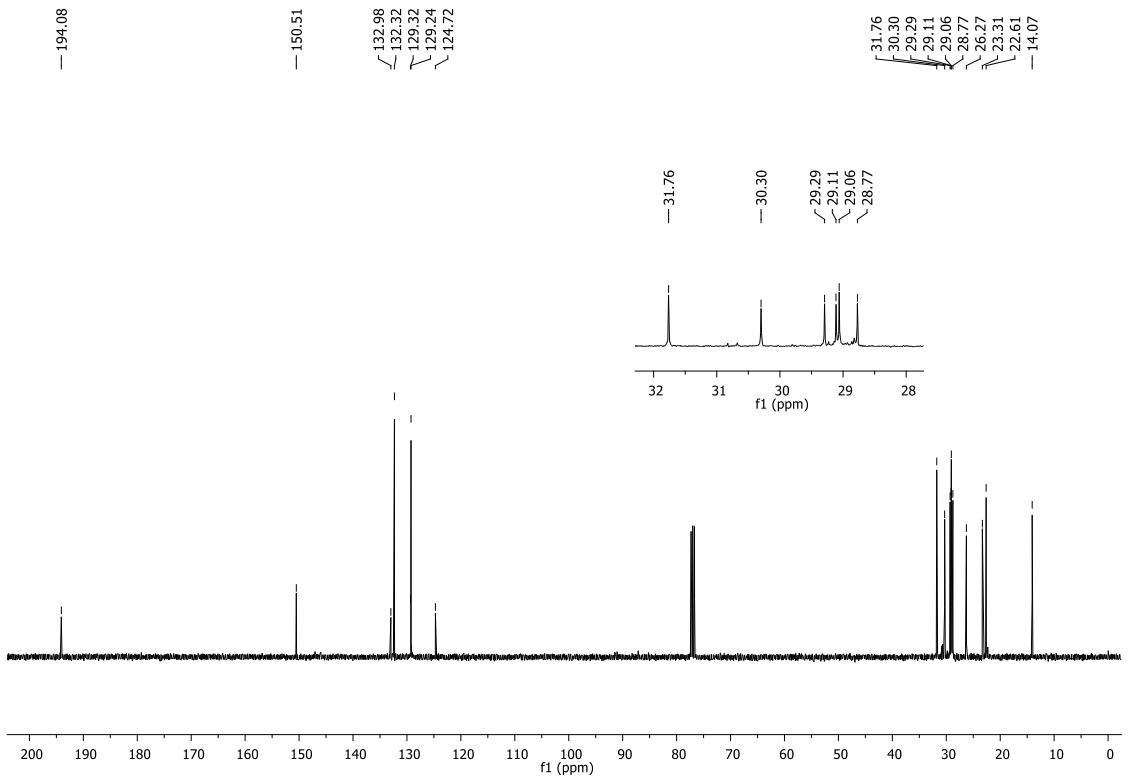
^{1}H NMR spectrum for compound **29** ($CDCl_3$, 400 MHz)



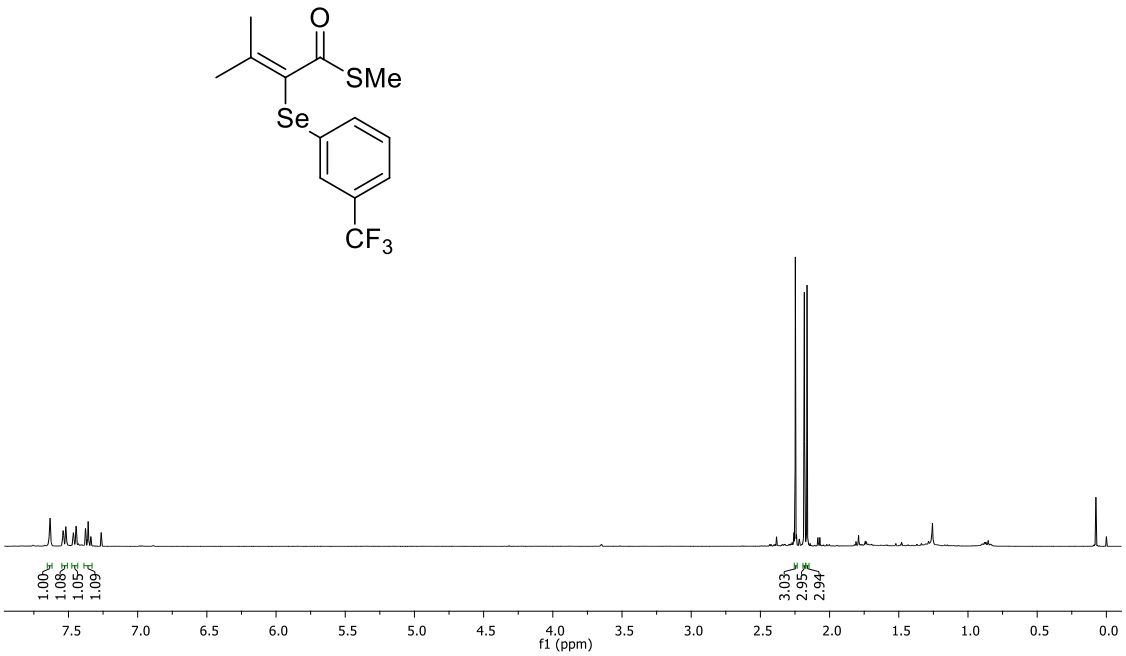
^{13}C NMR spectrum for compound **29** ($CDCl_3$, 100 MHz)



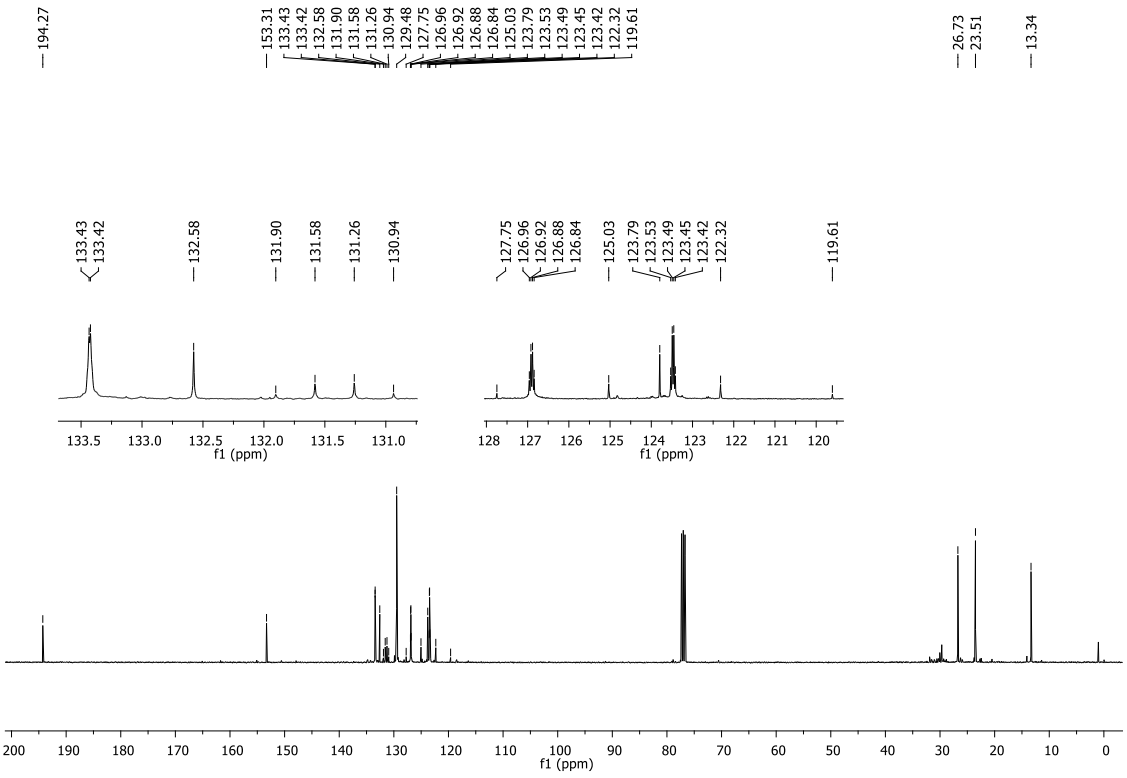
^{1}H NMR spectrum for compound **30** ($CDCl_3$, 400 MHz)



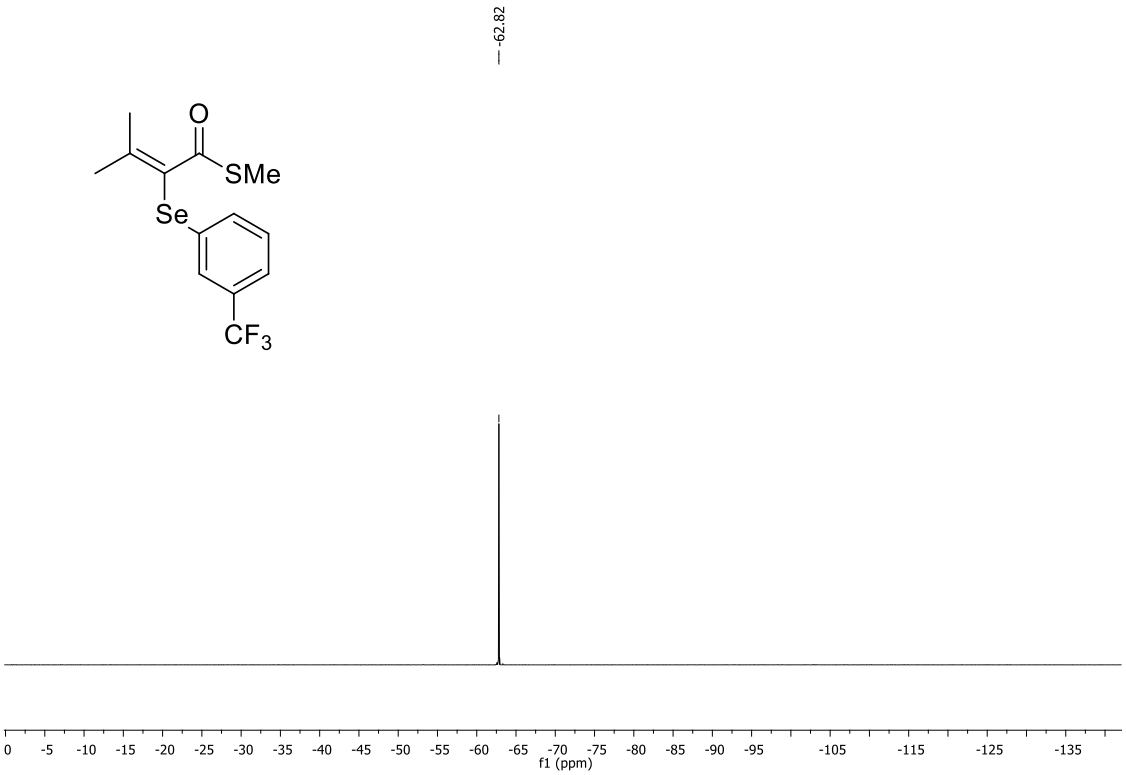
^{13}C NMR spectrum for compound **30** ($CDCl_3$, 100 MHz)



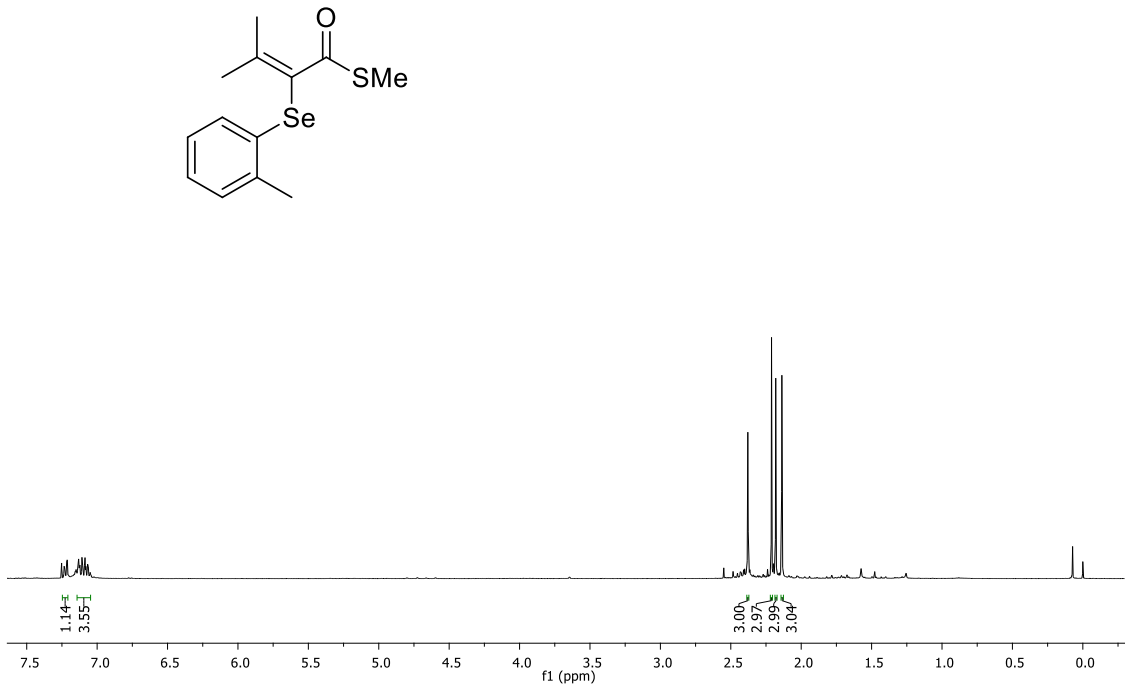
^{1}H NMR spectrum for compound **31** ($CDCl_3$, 400 MHz)



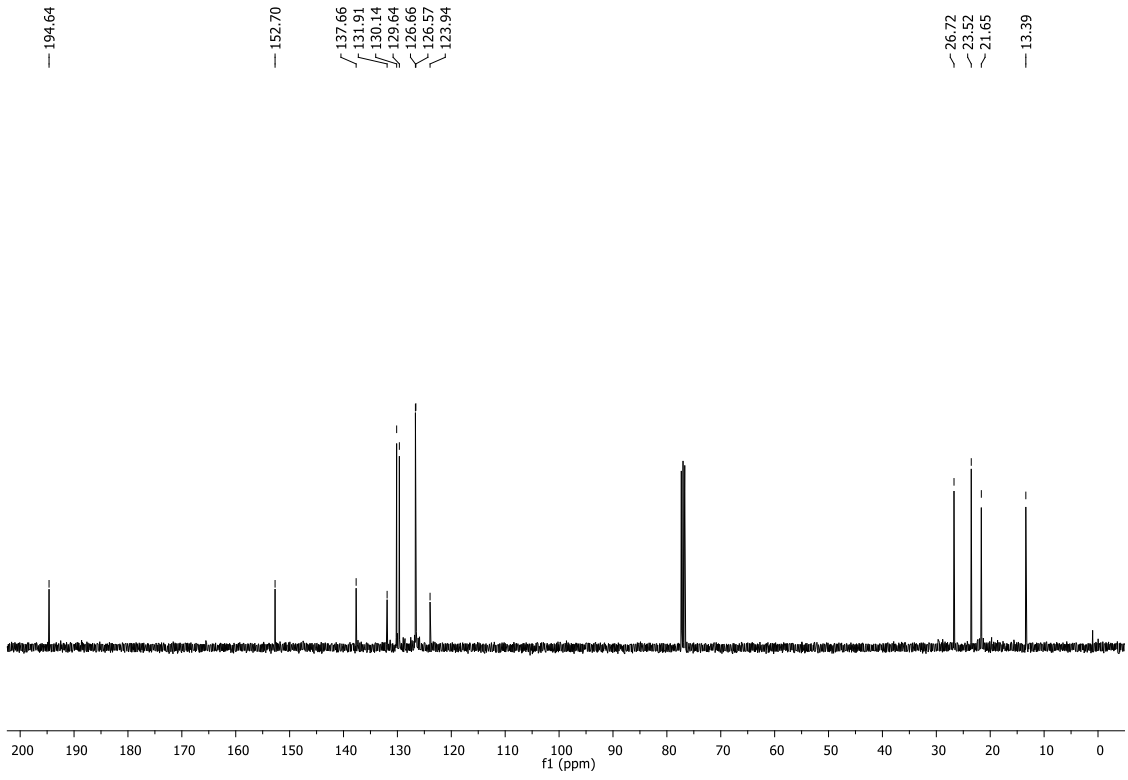
^{13}C NMR spectrum for compound **31** ($CDCl_3$, 100 MHz)



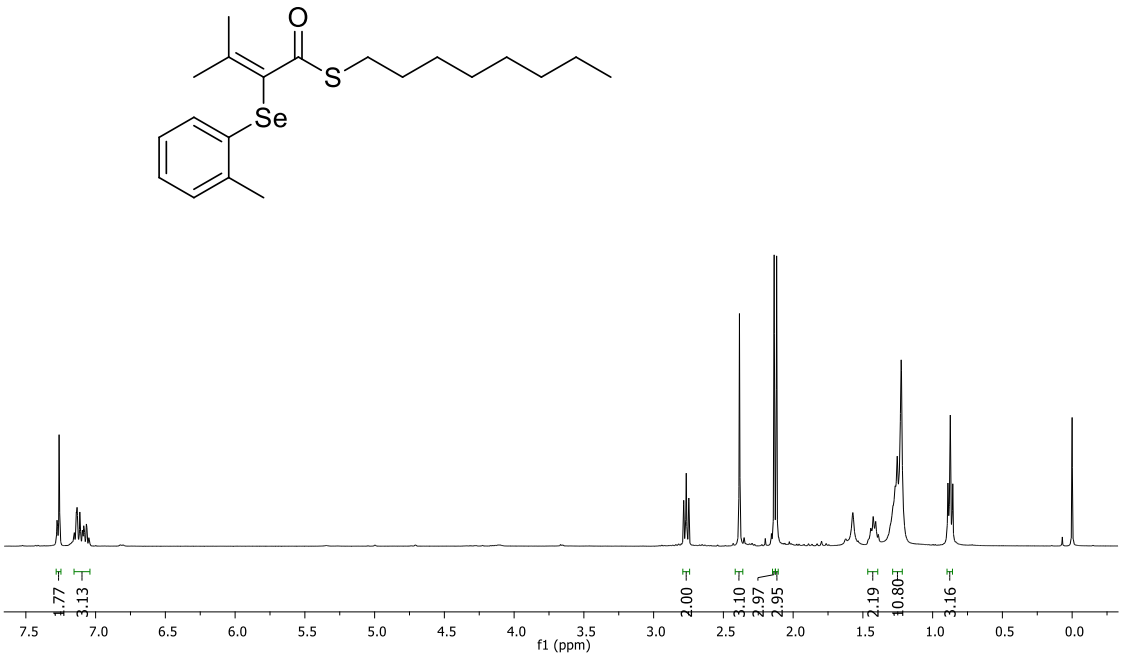
^{19}F NMR spectrum for compound **31** ($CDCl_3$, 376 MHz)



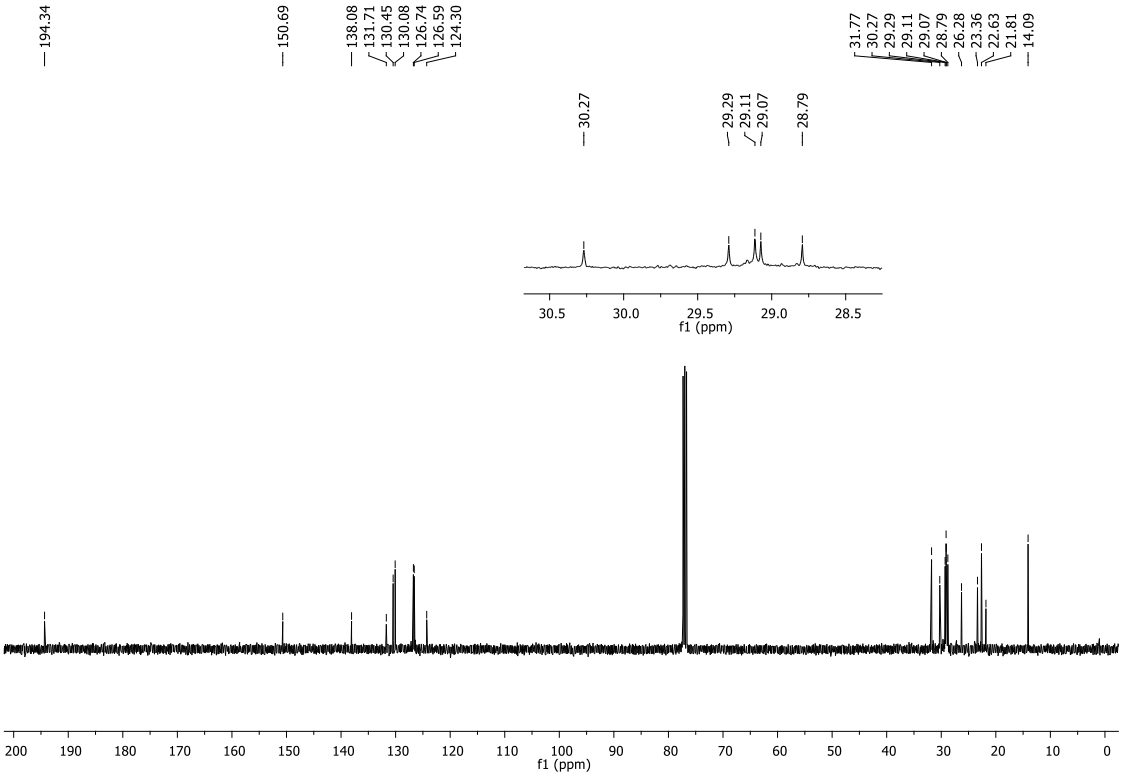
^{1}H NMR spectrum for compound **32** ($CDCl_3$, 400 MHz)



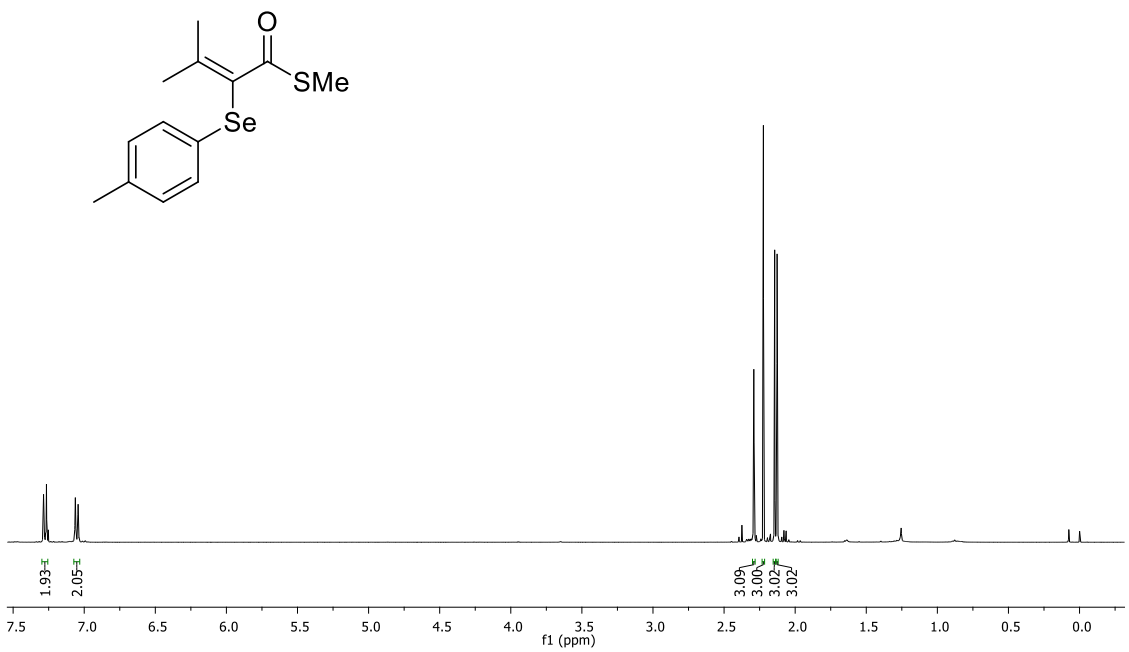
^{13}C NMR spectrum for compound **32** ($CDCl_3$, 100 MHz)



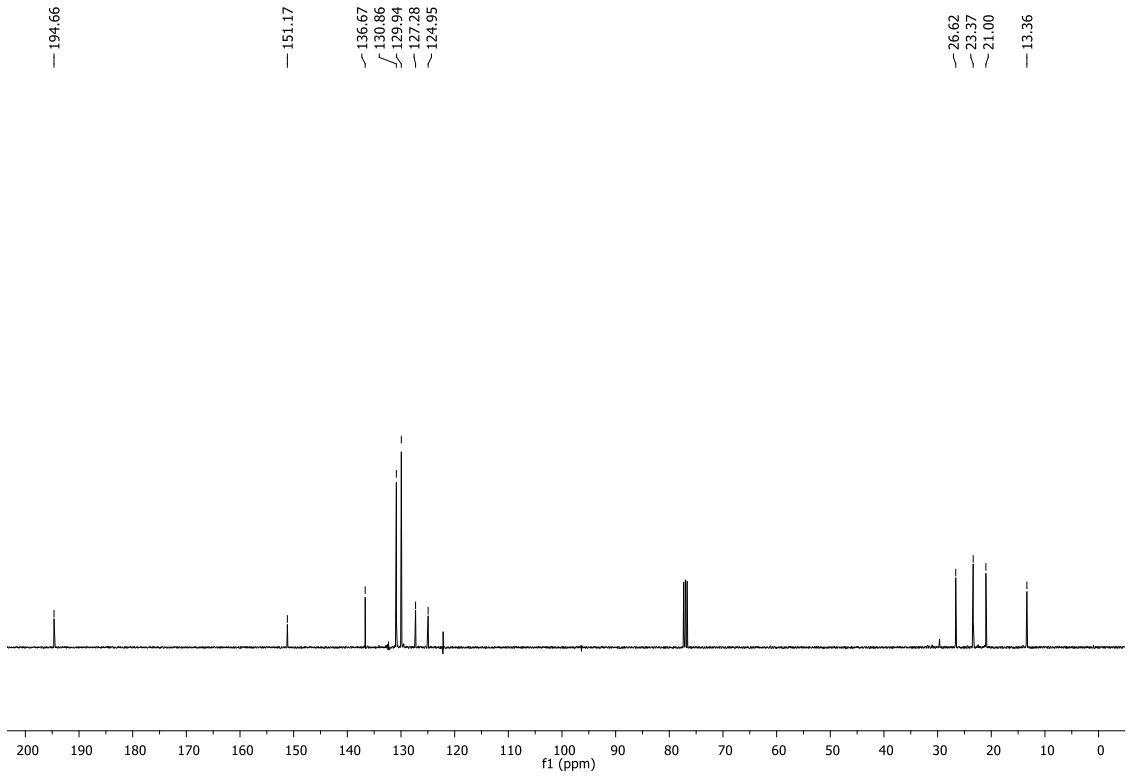
^{1}H NMR spectrum for compound **33** ($CDCl_3$, 400 MHz)



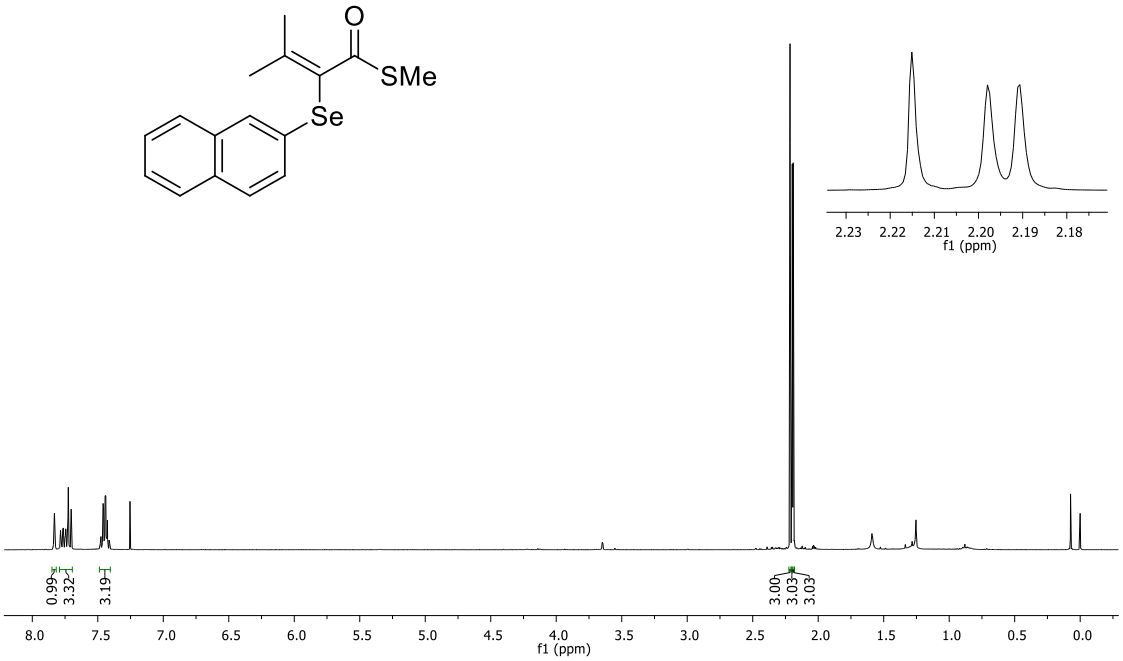
^{13}C NMR spectrum for compound **33** ($CDCl_3$, 100 MHz)



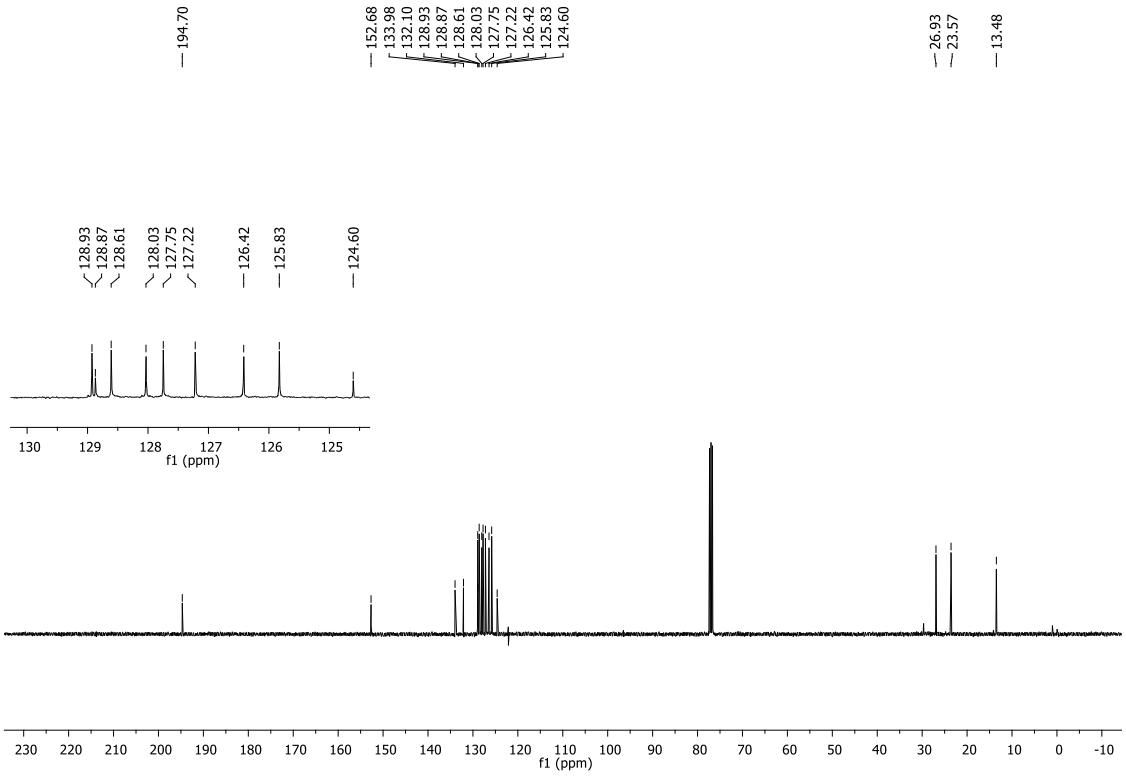
^{1}H NMR spectrum for compound **34** ($CDCl_3$, 400 MHz)



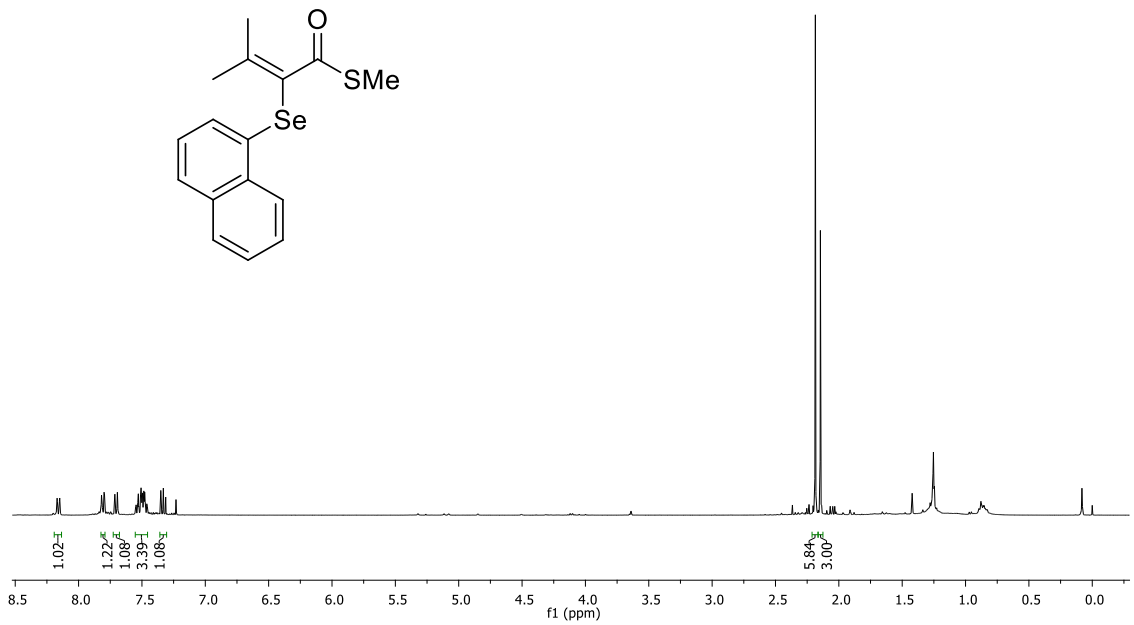
^{13}C NMR spectrum for compound **34** ($CDCl_3$, 100 MHz)



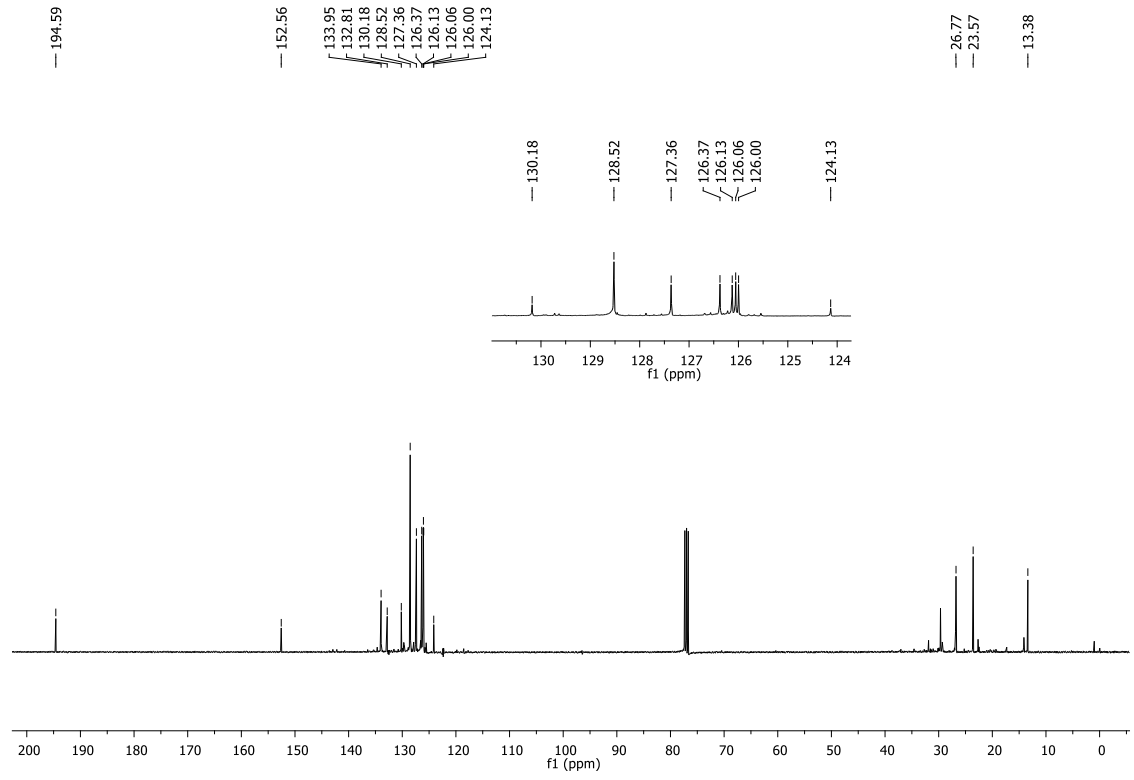
^{1}H NMR spectrum for compound **35** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **35** ($CDCl_3$, 100 MHz)

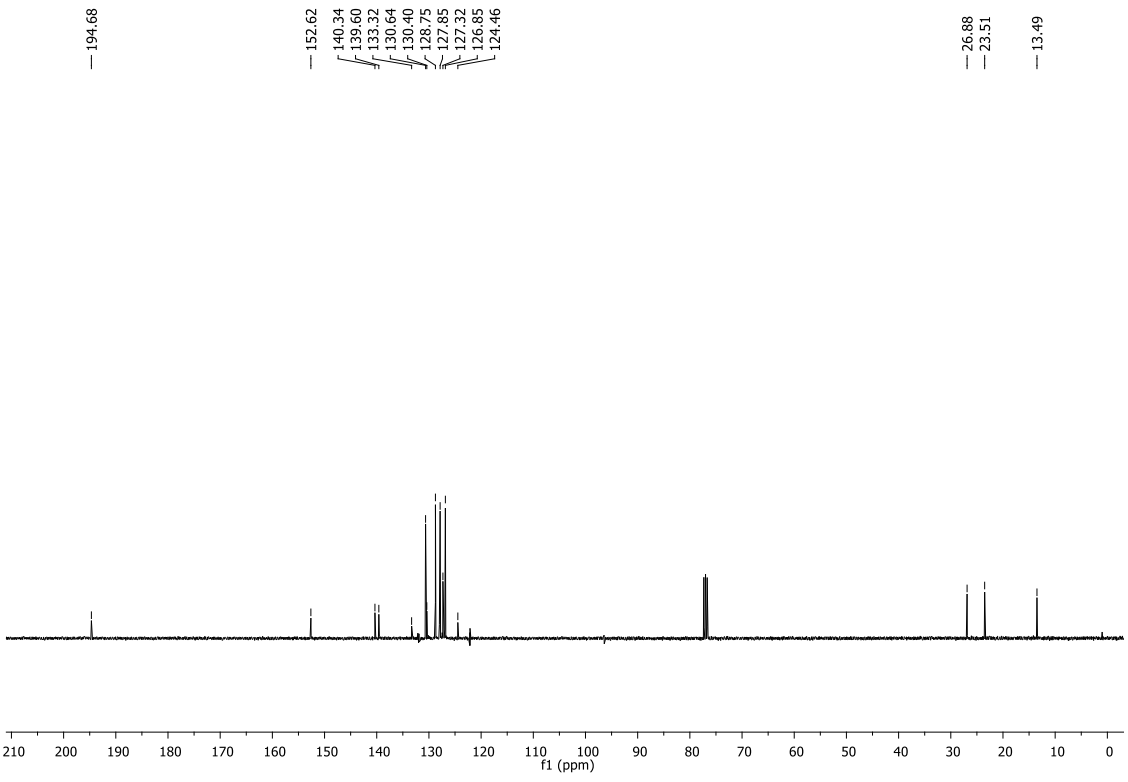


^{1}H NMR spectrum for compound **36** ($CDCl_3$, 400 MHz)

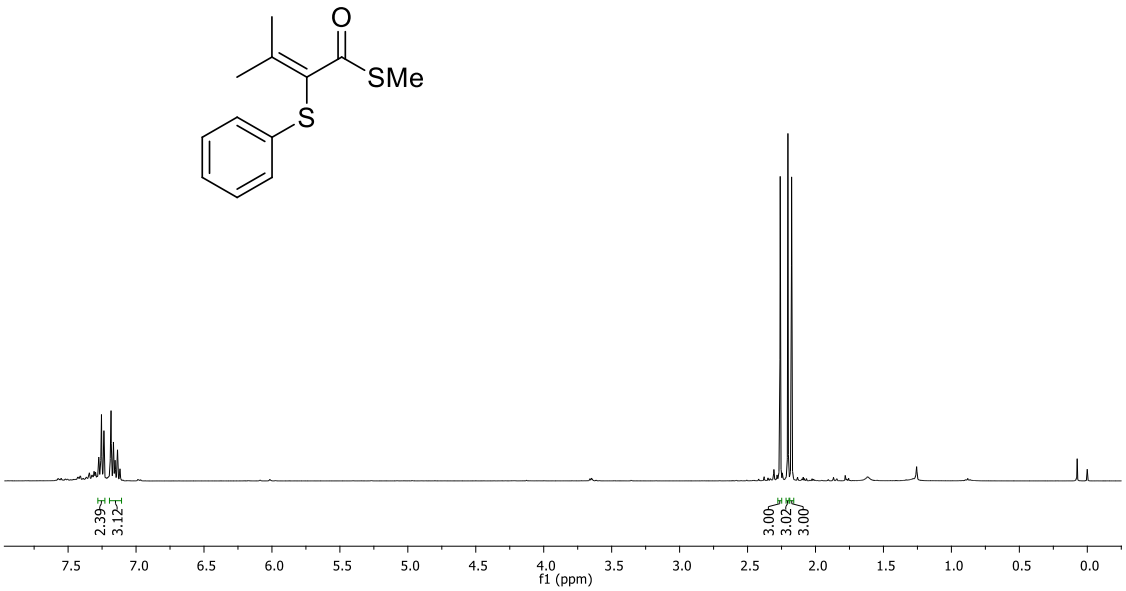


^{13}C NMR spectrum for compound **36** ($CDCl_3$, 100 MHz)

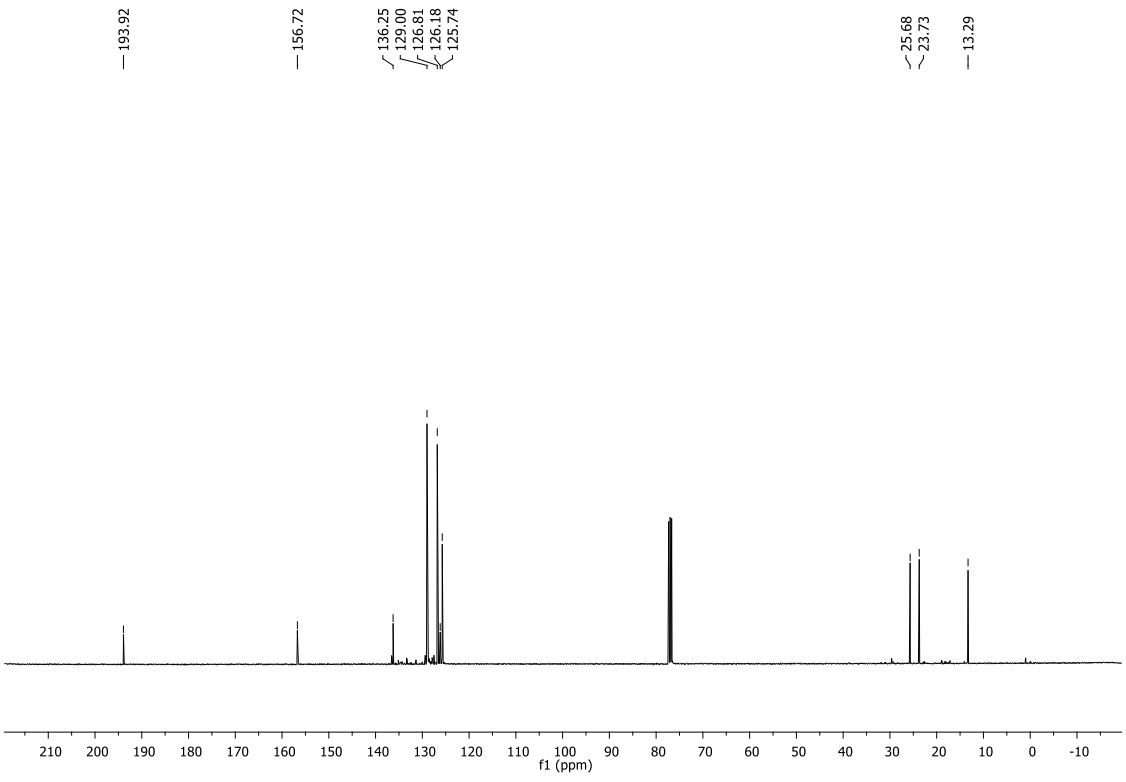
^{1}H NMR spectrum for compound **37** ($CDCl_3$, 400 MHz)



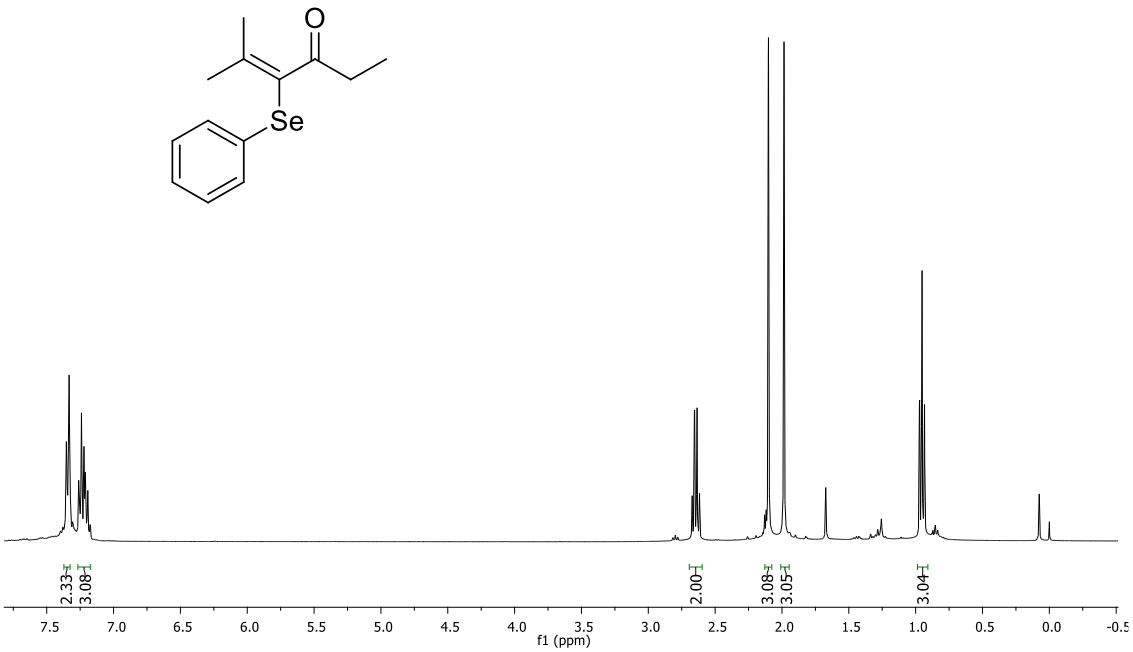
^{13}C NMR spectrum for compound **37** ($CDCl_3$, 100 MHz)



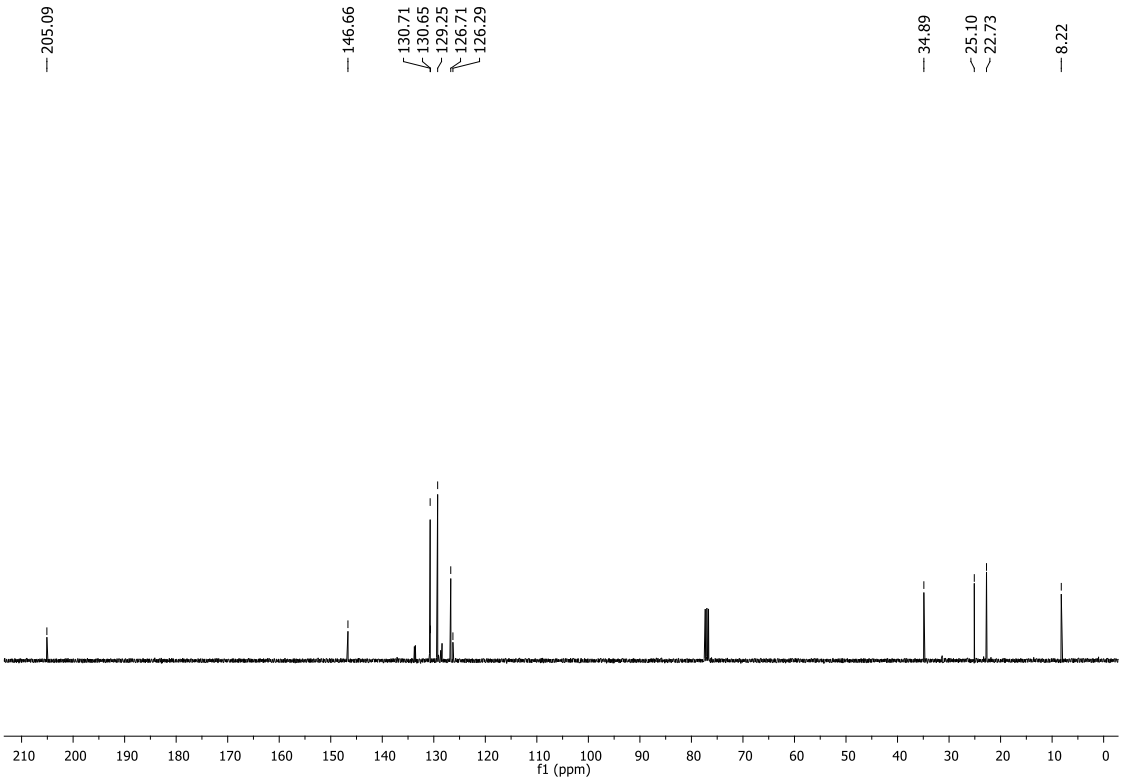
^{1}H NMR spectrum for compound **38** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **38** ($CDCl_3$, 100 MHz)



^{1}H NMR spectrum for compound **39** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **39** ($CDCl_3$, 100 MHz)