Electronic Supplementary Material (ESI) for Green Chemistry
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Replacing dichloroethane as a solvent for rhodium-catalysed intermolecular alkyne hydroacylation reactions: The utility of propylene carbonate

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Supporting Information

General considerations

Reactions were performed under an inert atmosphere of nitrogen with technical grade solvent unless otherwise stated. All glassware was oven dried at >100 °C and allowed to cool to room temperature under a positive nitrogen pressure. Cooling of reactions to 0 °C was achieved using an ice-water bath and cooling to -40 °C was achieved using a dry ice-acetone bath. Reactions were monitored by TLC until deemed complete using aluminium backed silica plates. Plates were visualised under ultraviolet light and/or by staining with $KMnO_4$. Reagents were purchased from Sigma-Aldrich Chemical Co. Ltd., Acros Organics Ltd., Lancaster Synthesis Ltd, or Strem Chemicals Inc. and were used as supplied unless otherwise stated. Dry tetrahydrofuran was obtained by passing through anhydrous alumina columns using an Innovative Technology Inc. PS-400-7 solvent purification system. 3-(methylthio)propionaldehyde and 2-(methylthio)benzaldehyde were distilled under reduced pressure prior to use. Petroleum ether refers to the fraction obtained between 30 and 40 °C. Flash chromatography was carried out using matrix 60 silica, pressure was applied at the column head *via* hand bellows. Infra-red spectra were recorded using NaCl discs on a Perkin-Elmer Spectrum One FT-IR or a Bruker Tensor 27 FT-IR spectrometer. ^{1}H NMR spectra were obtained on a Bruker DPX-200 (200 MHz), Bruker DQX-400 (400 MHz) or Bruker AVC-500 (500 MHz) spectrometer using the residual solvent as an internal standard. ^{13}C NMR spectra were obtained on a Bruker DQX-400 (100 MHz) or Bruker AVC-500 (125 MHz) spectrometer using the residual solvent as an internal standard. Chemical shifts were reported in parts per million (ppm) with the multiplicities of the spectra reported as following: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m) and broad (br). Mass spectrometry measurements were recorded by the internal service at the Department of Organic Chemistry, University of Oxford.

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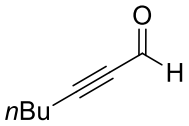
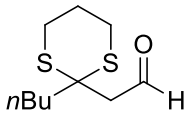
Hept-2-ynal



Prepared according to a procedure described by Journet *et al.*[1] *n*BuLi (2.5M in hexanes, 8.0 mL, 20 mmol) added over 2 minutes to a solution of 1-hexyne (3.3 mL, 20 mmol) in dry THF (50 mL) at -40 °C. DMF (3.1 mL, 40 mmol) added in one portion and the reaction mixture was allowed to warm to room temperature over 30 minutes, before being added to a rapidly stirred mixture of Et_2O (140 mL) and NaH_2PO_4 (14.2 g, 80 mmol) in water (140 mL) at 0 °C. The organic phase was separated and washed with water (2 x 40 mL), and the combined aqueous washes back-extracted into Et_2O. The combined organic extracts were dried over $MgSO_4$, concentrated *in vacuo* and purified by column chromatography (20% Et_2O in petrol) to furnish the aldehyde as a light yellow oil (1.68 g, 76%). ν_{max} (film)/cm^{-1} 2961, 2937, 2873, 2281, 2203, 1671; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 9.17 (s, 1H), 2.41 (t, J = 7.1 Hz, 2H), 1.58 (tt, J = 7.6, 6.8 Hz, 2H), 1.44 (sxt, J = 7.58 Hz, 2H) and 0.93 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 177.2, 99.3, 81.7, 29.5, 21.9, 18.8 and 13.4; HRMS (FI) found *m/z* 111.0810 $[M+H]^+$, $C_7H_{11}O$ requires 111.0810.

2-(2-butyl-1,3-dithian-2-yl)acetaldehyde



A solution of hept-2-ynal (3.10 g, 28 mmol) in THF (280 mL) was cooled to 0 °C, followed by the addition of 1,3-propanedithiol (3.11 mL, 31 mmol) then NaOMe (1.97 g, 36 mmol). The reaction mixture was allowed to warm to room temperature overnight, then quenched with saturated aqueous NH_4Cl. The phases were separated and the aqueous phase extracted into Et_2O (2 x 100 mL). The combined organic extracts were washed with water then brine, dried over $MgSO_4$ and concentrated *in vacuo*. The crude product was washed with saturated aqueous $CuSO_4$ then purified by column chromatography (20% Et_2O in petrol) to provide the aldehyde as a bright yellow oil (2.92 g, 48%). ν_{max} (film)/cm^{-1} 2955, 2934, 1718; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H 9.78 (t, J = 2.7 Hz, 1H), 2.94-2.80 (m, 6H), 2.09-1.88 (m, 4H), 1.55-1.46 (m, 2H), 1.40-1.30 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 199.8, 50.1, 49.2, 40.2, 26.2, 26.1, 24.7, 22.8, 13.9; HRMS (ESI) found *m/z* 273.0953 $[M+Na]^+$, $C_{11}H_{22}NaO_2S_2$ requires 273.0953.

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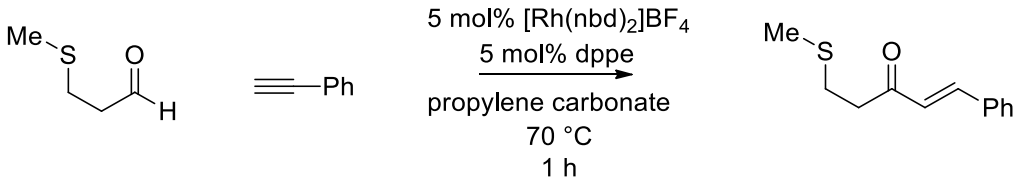
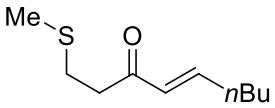
General procedure, as exemplified by the preparation of (*E*)-5-(methylthio)-1-phenylpent-1-en-3-one



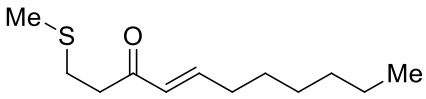
$[Rh(nbd)_2]BF_4$ (14 mg, 0.0375 mmol) and dppe (15 mg, 0.0375 mmol) were dissolved in propylene carbonate (2.5 mL) and stirred at room temperature for 10 minutes. 3-(methylthio)propionaldehyde (75 μL, 0.75 mmol) then phenylacetylene (90 μL, 0.83 mmol) were added and the reaction heated at 70 °C for 1 hour. The reaction mixture was loaded directly onto silica and eluted with 30% Et_2O/petrol to furnish the pure product as a yellow oil (139 mg, 90%). ν_{max} (film)/cm^{-1} 2917, 1688, 1661, 1612; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.69-7.49 (m, 3H [including d, J = 16.3 Hz, 1H]), 7.47-7.35 (m, 3H) 6.75 (d, J = 16.3 Hz, 1H), 3.05-2.92 (m, 2H), 2.91-2.77 (m, 2H), 2.15 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 198.4, 143.1, 134.3, 130.7, 129.0, 128.4, 125.9, 40.6, 28.4, 15.9; HRMS (ESI) found *m/z* 229.0658 $[M+Na]^+$, $C_{12}H_{14}NaOS$ requires 229.0660.

(*E*)-1-(methylthio)non-4-en-3-one



Prepared according to general procedure A to furnish the product as a yellow oil (127 mg, 91%). ^{1}H NMR (200 MHz, $CDCl_3$) δ_H 6.85 (dt, J = 15.9, 6.9 Hz, 1H), 6.08 (dt, J = 15.9, 1.5 Hz, 1H), 2.92-2.66 (m, 4H), 2.29-2.13 (m, 2H), 2.10 (s, 3H), 1.56-1.19 (m, 4H), 0.91 (t, J = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 198.6, 148.2, 130.1, 39.7, 32.2, 30.1, 28.3, 22.2, 15.8, 13.8; *m/z* (ESI) 187 (25%, $[M+H]^+$). Data consistent with the literature.[2]

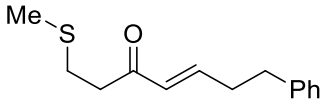
(*E*)-1-(methylthio)undec-4-en-3-one



Prepared according to general procedure A to furnish the product as a yellow oil (146 mg, 91%) as a mixture of linear and branched isomers (84:16 l:b). ν_{max} (film)/cm^{-1} 2956, 2927, 2857, 1696, 1673, 1629; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 6.81 (dt, J = 15.9, 6.9 Hz, 1H), 6.05 (d, J = 15.9 Hz, 1H), 2.82-2.78 (m, 2H), 2.73-2.69 (m, 2H), 2.21-2.14 (m, 2H), 2.07 (s, 3H), 1.45-1.38 (m, 2H), 1.31-1.28 (m, 6H), 0.85-0.81 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 198.5, 148.2, 130.0, 124.0, 39.7, 32.5, 31.6,

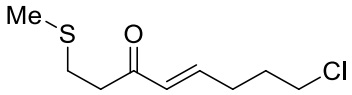
28.3, 28.0, 22.5, 15.8, 14.0; HRMS (ESI) found *m/z* 237.1281 $[M+Na]^+$, $C_{12}H_{22}NaOS$ requires 237.1284.

(*E*)-1-(methylthio)-7-phenylhept-4-en-3-one



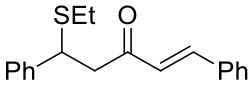
Prepared according to general procedure A to furnish the product as a pale yellow oil (146 mg, 83%) as a mixture of linear and branched isomers (80:20 l:b). ν_{max} (film)/cm^{-1} 2917, 1671, 1628; ^1H NMR (400 MHz, $CDCl_3$) δ_H 7.32-7.17 (m, 5H), 6.88 (dt, *J* = 15.9, 6.8 Hz, 1H), 6.13 (d, *J* = 15.9 Hz, 1H), 2.85-2.72 (m, 6H), 2.55 (q, *J* = 7.4 Hz, 2H), 2.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 198.4, 146.7, 140.6, 130.6, 128.6, 128.4, 126.3, 39.9, 34.4, 34.2, 28.3, 15.8; HRMS (ESI) found *m/z* 257.0972 $[M+Na]^+$, $C_{14}H_{18}NaOS$ requires 257.0971.

(*E*)-8-chloro-1-(methylthio)oct-4-en-3-one



Prepared according to general procedure A to furnish the product as a pale yellow oil (85 mg, 66%). ^1H NMR (400 MHz, $CDCl_3$) δ_H 6.80 (dt, *J* = 15.8, 6.9 Hz, 1H), 6.13 (d, *J* = 15.8 Hz, 1H), 3.52 (t, *J* = 6.4 Hz, 2H), 2.83-2.80 (m, 2H), 2.74-2.71 (m, 2H), 2.40-2.34 (m, 2H), 2.08 (s, 3H) , 1.91 (quin, *J* = 7.1 Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 198.2, 145.6, 130.8, 44.0, 40.0, 30.7, 29.5, 28.2, 15.8; *m/z* (FI) 206 (100%, $[M]^+$). Data consistent with the literature.[2]

(*E*)-5-(Ethylthio)-1,5-diphenylpent-1-en-3-one



Prepared according to general procedure A to furnish the product as a white solid (196 mg, 86%). m.p. 100-102 ^oC; ν_{max} (film)/cm^{-1} 2970, 1648, 699; ^1H NMR (400 MHz, $CDCl_3$) δ_H 7.54-7.50 (m, 3H), 7.43-7.39 (m, 5H), 7.34-7.30 (m, 2H), 7.25-7.21 (m, 1H), 6.68 (d, *J* = 16.1 Hz, 1H), 4.50 (t, *J* = 7.2 Hz, 1H), 3.23 (d, *J* = 7.2 Hz, 2H), 2.43-2.29 (m, 2H), 1.18 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 196.9, 143.2, 142.1, 134.3, 130.6 (2×CH), 128.9 (2×CH), 128.5, 128.3 (2×CH), 127.8 (2×CH), 127.2, 126.1, 47.4, 44.2, 25.4 and 14.3. HRMS (FI) found *m/z* 296.1231 $[M]^+$, $C_{19}H_{20}OS$ requires 296.1235. Data consistent with the literature.[3]

(*E*)-1-Cyclopropyl-5-(ethylthio)-5-phenylpent-1-en-3-one

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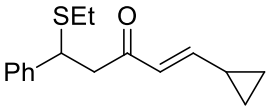
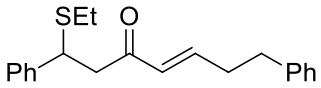


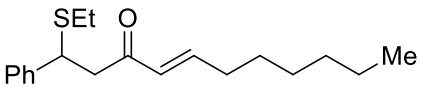
Prepared according to general procedure A to furnish the product as a colourless oil (174 mg, 90%). ν_{max} (film)/cm^{-1} 3027, 3006, 2926, 2870, 1949, 1876, 1805, 1713, 1667, 1616, 950, 937, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.36-7.09 (m, 5H), 6.19 (dd, *J* = 15.6, 9.6 Hz, 1H), 6.08 (d, *J* = 15.6 Hz, 1H), 4.34 (t, *J* = 7.2, 1H) , 2.96 (d, *J* = 7.2 Hz, 2H), 2.33-2.17 (m, 2H), 1.51-1.39 (m, 1H), 1.08 (t, *J* = 7.4 Hz, 3H), 0.95-0.84 (m, 2H), 0.59-0.51 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 196.1, 153.5, 142.2, 128.4 (2×CH_2), 127.7 (2×CH_2), 127.2, 127.1, 46.8, 44.1, 25.4, 14.8, 14.3, 9.1 (2×CH_2). HRMS (FI) found *m/z* 260.1242 $[M]^+$, $C_{16}H_{20}OS$ requires 260.1235. Data consistent with the literature.[3]

(*E*)-1-(Ethylthio)-1,7-diphenylhept-4-en-3-one



Prepared according to general procedure A to furnish the product as a colourless oil (210 mg, 87%). ν_{max}(film)/cm^{-1} 3061, 3027, 2967, 2926, 2869, 1693, 1677, 1453, 748; ^{1}H NMR (400 MHz, $CDCl_3$), δ_H 7.37-6.96 (m, 10H), 6.74 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.98 (d, *J* = 15.8Hz, 1H), 4.33 (t, *J* = 7.2, 1H), 2.99 (d, *J* = 7.2 Hz, 2H), 2.67 (t, *J* = 7.7 Hz, 2H), 2.42 (q, *J* = 7.4 Hz, 2H), 2.30-2.18 (m, 2H), 1.08 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$), δ_C 196.9, 146.89, 142.1, 140.6, 130.8, 128.5 (2×CH), 128.4 (2×CH), 128.3 (2×CH), 127.7 (2×CH), 127.1, 126.2, 46.6, 44.1, 34.3, 34.1, 25.4, 14.3; HRMS (ESI) found *m/z* 325.1621 $[M+H]^+$, $C_{21}H_{25}OS$ requires 325.1621.

(*E*)-1-(ethylthio)-1-phenylundec-4-en-3-one



Prepared according to general procedure A to furnish the product as a colourless oil (207 mg, 95%). ν_{max}(film)/cm^{-1} 2928, 2870, 1674, 1627, 1409, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.40-7.35 (m, 2H), 7.30 (t, *J* = 7.7, 2H), 7.25-7.19 (m, 1H), 6.80 (dt, *J* = 15.8, 6.9 Hz, 1H), 6.04 (d, *J* = 15.8 Hz, 1H), 4.43 (t, *J* = 7.2 Hz, 1H), 3.09 (d, *J* = 7.2 Hz, 2H), 2.33 (t, *J* = 7.3, 2H), 2.24-2.11 (m, 2H), 1.48-1.36 (m, 2H), 1.34-1.22 (m, 6H), 1.16 (t, *J* = 7.3 Hz, 3H), 0.95-0.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 197.1, 148.5, 142.2, 130.3, 128.43 (2×CH), 127.7 (2×CH), 127.1, 46.5, 44.1, 32.5, 31.5, 28.8, 27.9, 25.4, 22.5, 14.3, 14.0; *m/z* (ESI) 327 (100%, $[M+Na]^+$), 305 (45%, $[M+H]^+$); HRMS (ESI) found *m/z* 305.1934 $[M+H]^+$, $C_{19}H_{29}OS$ requires 305.1934.

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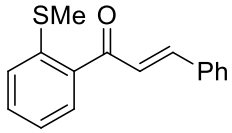
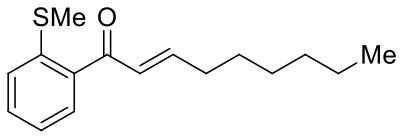
(*E*)-1-(2-(methylthio)phenyl)-3-phenylprop-2-en-1-ol



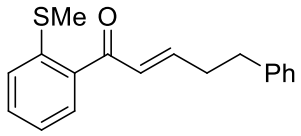
Prepared according to general procedure A to furnish the product as a yellow oil (160 mg, 84%) as a mixture of linear and branched isomers (11:1, l:b). ν_{max}(film)/cm^{-1} 3059, 2912, 1655, 1598, 1208, 750; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.70 (dd, *J* = 1.3 and 7.7 Hz, 1H), 7.64 (d, *J* = 15.9, 1H), 7.60-7.58 (m, 2H), 7.49-7.45 (m, 1H), 7.40-7.37 (m, 4H), 7.32 (d, *J* = 15.9, 1H), 7.26-7.22 (m, 1H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 192.9, 145.2, 140.5, 137.2, 134.8, 131.6, 130.6, 129.5, 129.0, 128.5, 126.3, 124.8, 124.2, 16.5; HRMS (ESI) found *m/z* 277.0657 $[M+Na]^+$, $C_{16}H_{14}NaOS$ requires 277.0658.

(*E*)-1-(2-(methylthio)phenyl)non-2-en-1-one



Prepared according to general procedure A to furnish the product as a yellow oil (169 mg, 86%). ν_{max}(film)/cm^{-1} 2955, 2926, 2856, 1661, 1615; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.56 (d, *J* = 7.7 Hz, 1H), 7.43-7.39 (m, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.19-7.15 (m, 1H), 6.90-6.83 (m, 1H), 6.63 (d, *J* = 15.6 Hz, 1H), 2.41 (s, 3H), 2.26 (app. q, *J* = 7.2 Hz, 2H), 1.51-1.44 (m, 2H), 1.36-1.28 (m, 6H), 0.87 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 193.4, 151.1, 140.2, 137.1, 131.2, 129.3, 128.5, 126.1, 124.0, 32.8, 31.6, 28.9, 28.0, 22.5, 16.4, 14.1; HRMS (ESI) found *m/z* 285.1280 $[M+Na]^+$, $C_{16}H_{22}NaOS$ requires 285.1284.

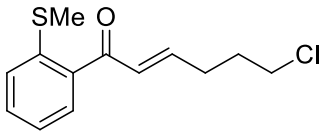
(*E*)-1-(2-(methylthio)phenyl)-5-phenylpent-2-en-1-one



Prepared according to general procedure A to furnish the product as a yellow oil (149 mg, 70%). ν_{max}(film)/cm^{-1} 3060, 3026, 2920, 2856, 1661, 1615, 1300, 748; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.52 (d, *J* = 7.7 Hz, 1H), 7.46-7.41 (m, 1H), 7.36-7.29 (m, 3H), 7.24-7.16 (m, 4H), 6.91 (dt, *J* = 15.5, 6.8 Hz, 1H), 6.67 (dd, *J* = 15.5, 1.0 Hz, 1H), 2.83 (t, *J* = 7.7 Hz, 2H), 2.61 (m, 2H), 3.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 193.2, 149.4, 140.8, 140.5, 136.8, 131.4, 129.5, 129.2, 128.5, 128.4,

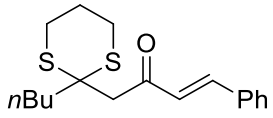
126.2, 126.1, 124.0, 34.5, 34.4, 16.4; HRMS (ESI) found *m/z* 305.0972 $[M+Na]^+$, $C_{18}H_{18}NaOS$ requires 305.0971.

(*E*)-6-chloro-1-(2-(methylthio)phenyl)hex-2-en-1-one



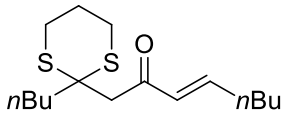
Prepared according to general procedure A to furnish the product as a yellow oil (126 mg, 66%). ν_{max}(film)/cm^{-1} 3059, 2957, 2020, 1662, 1616, 1298, 755, 740; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.60 (d, *J* = 7.7 Hz, 1H), 7.46-7.43 (m, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.22-7.18 (m, 1H), 6.85 (dt, *J* = 15.5, 6.8 Hz, 1H), 6.72 (d, *J* = 15.6 Hz, 1H), 3.57 (t, *J* = 6.4 Hz, 2H), 2.50-2.44 (m, 2H), 2.44 (s, 3H), 2.01-1.94 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 192.8, 148.1, 140.6, 136.7, 131.5, 129.5, 129.4, 126.1, 124.0, 44.1, 30.7, 29.7 and 16.4; HRMS (ESI) found *m/z* 277.0424 $[M+Na]^+$, $C_{13}H_{15}ClNaOS$ requires 277.0424.

(*E*)-1-(2-butyl-1,3-dithian-2-yl)-4-phenylbut-3-en-2-one



Prepared according to general procedure A to furnish the product as a bright yellow oil (175 mg, 73%). ν_{max}(film)/cm^{-1} 2954, 1717, 1605; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.59-7.54 (m, 3H [incl. 7.57, d, *J* = 15.9 Hz, 1H]), 7.39-7.38 (m, 3H), 6.85 (d, *J* = 15.9 Hz, 1H), 3.25 (s, 2H), 2.95-2.82 (m, 2H), 2.16-2.09 (m, 2H), 2.08-1.90 (m, 2H), 1.57-1.50 (m, 2H), 1.41-1.31 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 195.8, 142.6, 134.5, 130.5, 129.0, 128.4, 126.7, 51.1, 48.6, 38.6, 26.5, 26.4, 25.0, 22.8, 14.0; HRMS (ESI) found *m/z* 343.1159 $[M+Na]^+$, $C_{18}H_{24}NaOS_2$ requires 343.1161.

(*E*)-1-(2-butyl-1,3-dithian-2-yl)oct-3-en-2-one



Prepared according to general procedure A to furnish the product as a bright yellow oil (175 mg, 73%). ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 6.80 (1H, dt, *J* = 15.6, 6.9 Hz), 6.12 (1H, dt, *J* = 15.6, 1.5 Hz), 3.06 (2H, s), 2.82-2.77 (4H, m), 2.15 (2H, tdd, *J* = 7.0, 6.9, 1.4 Hz), 2.05-1.85 (4H, m), 1.47-1.19 (8H, m), 0.86 (3H, t, *J* = 7.2 Hz), 0.84 (3H, t, *J* = 7.1 Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 196.4, 148.3, 131.4, 51.6, 47.8, 38.9, 32.6, 30.6, 26.9 (2 x CH_2), 26.8, 25.5, 23.3, 22.7, 14.4, 14.2; HRMS (EI) found *m/z* 301.1654 $[M+H]^+$, $C_{16}H_{29}OS_2$ requires 301.1651. Data consistent with the literature.[4]

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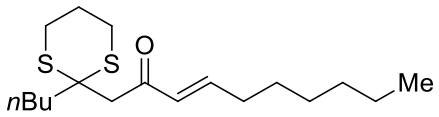
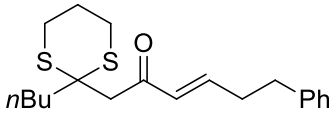
(*E*)-1-(2-butyl-1,3-dithian-2-yl)dec-3-en-2-one



Prepared according to general procedure A to furnish the product as a yellow oil (204 mg, 83%). ν_{max} (film)/cm^{-1} 2956, 2929, 2858, 1618, 1555; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 6.87 (dt, J = 15.7, 6.9 Hz, 1H), 6.20 (d, J = 16.9 Hz, 1H), 3.14 (s, 2H), 2.89-2.85 (m, 4H), 2.25-2.20 (m, 2H), 2.11-2.07 (m, 2H), 2.01-1.95 (m, 2H), 1.52-1.46 (m, 4H), 1.38-1.26 (m, 8H), 0.96-0.87 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 196.0, 148.0, 130.9, 50.9, 47.3, 38.4, 32.4, 31.6, 28.8, 28.0, 26.5, 26.4, 25.0, 22.8, 22.5, 14.02, 13.97; HRMS (ESI) found m/z 351.1785 $[M+Na]^+$, $C_{18}H_{32}NaOS_2$ requires 351.1787.

(*E*)-1-(2-butyl-1,3-dithian-2-yl)-6-phenylhex-3-en-2-one



Prepared according to general procedure A to furnish the product as a yellow oil (196 mg, 75%). ν_{max} (film)/cm^{-1} 2954, 2931, 2870, 1686, 1663, 1622; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.32-7.28 (m, 2H), 7.23-7.18 (m, 3H), 6.89 (dt, J = 15.7, 7.1 Hz, 1H), 6.21 (d, J = 15.7 Hz, 1H), 3.12 (s, 2H), 2.87-2.78 (m, 6H), 2.59-2.52 (m, 2H), 2.09-2.05 (m, 2H), 2.01-1.92 (m, 2H), 1.52-1.45 (m, 2H), 1.38-1.29 (m, 2H), 0.93 (t, J = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 195.8, 146.3, 140.7, 131.4, 128.5, 128.4, 126.2, 50.9, 47.4, 38.5, 34.3, 34.1, 26.4, 26.1, 25.0, 22.8, 14.0; HRMS (ESI) found m/z 371.1469 $[M+Na]^+$, $C_{20}H_{28}NaOS_2$ requires 371.1474.

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1. M. Journet, D. Cai, L. M. DiMichele and R. D. Larsen, *Tetrahedron Lett.*, 1998, **39**, 6427-6428.

2. M. C. Willis, H. E. Randell-Sly, R. L. Woodward, S. J. McNally and G. S. Currie, *J. Org. Chem.*, 2006, **71**, 5291-5297.

3. C. González-Rodríguez, S. R. Parsons, A. L. Thompson and M. C. Willis, *Chem. Eur. J.*, 2010, **16**, 10950-10954.

4. M. C. Willis, H. E. Randell-Sly, R. L. Woodward and G. S. Currie, *Org. Lett.*, 2005, **7**, 2249-2251.

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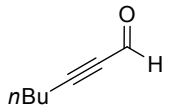

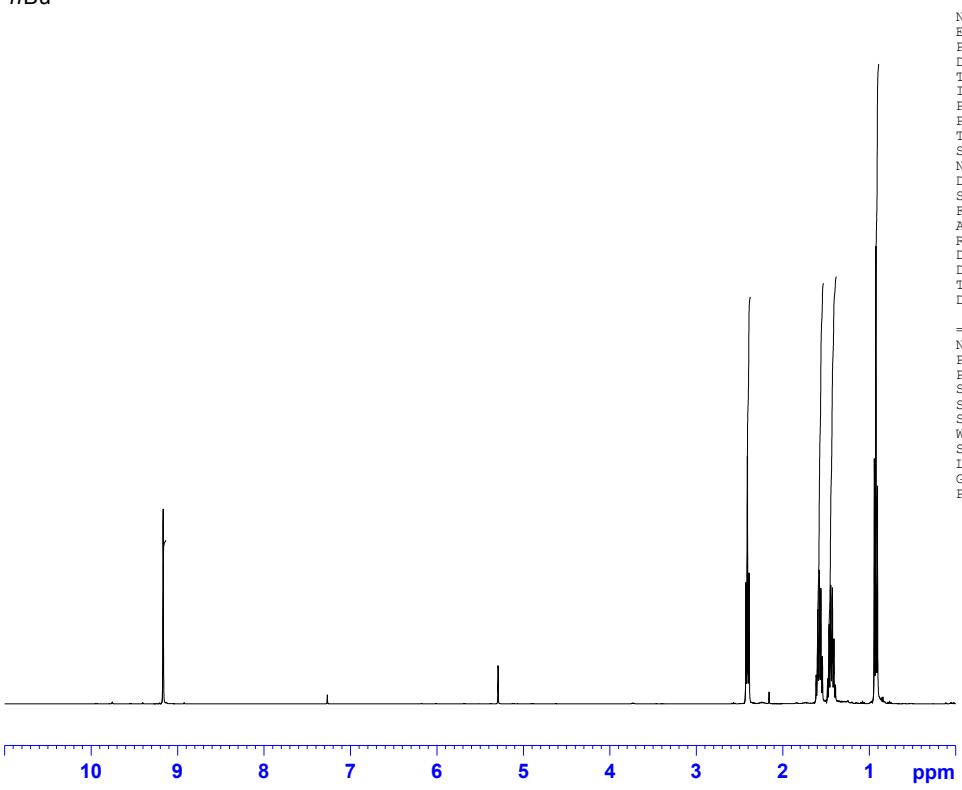


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TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 40.3
DW               60.400 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1                0.00 dB
SFO1        400.2024714 MHz
SI                32768
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WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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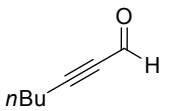

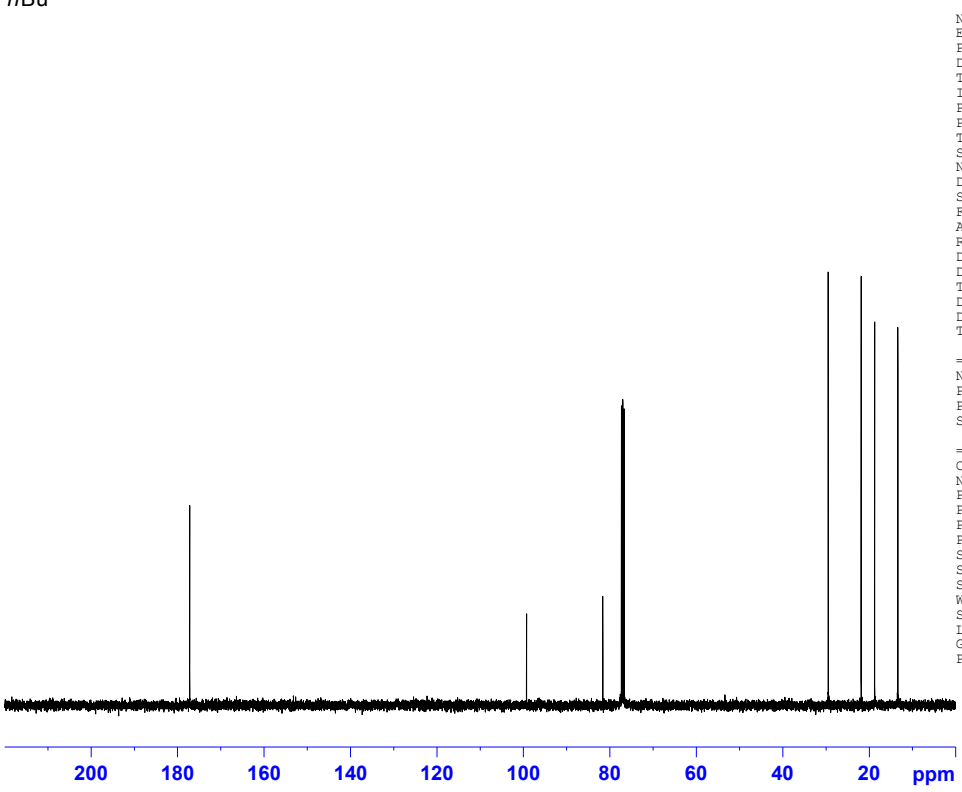


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PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           26178.010 Hz
FIDRES        0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW               19.100 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.50 usec
PL1                0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2        400.2016008 MHz
SI                32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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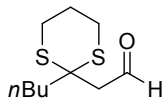

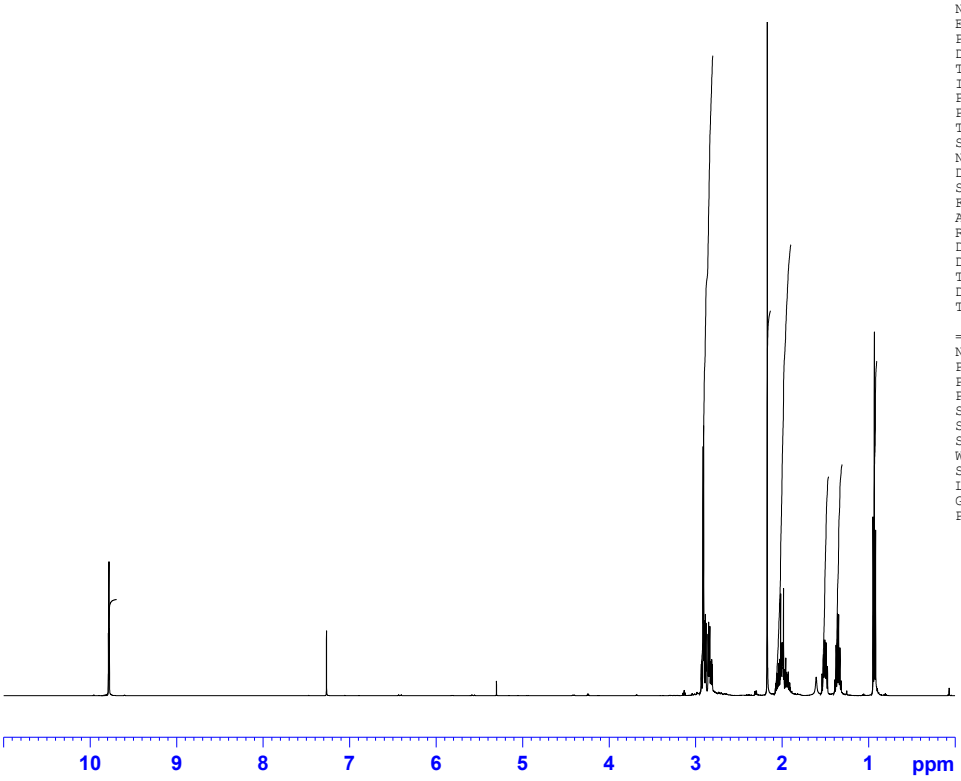


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PROCNO                 1
Date_           20101109
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PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                    4
DW               48.400 usec
DE                6.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
PL1W        15.19999981 W
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SI                 32768
SF          500.3000240 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
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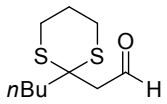

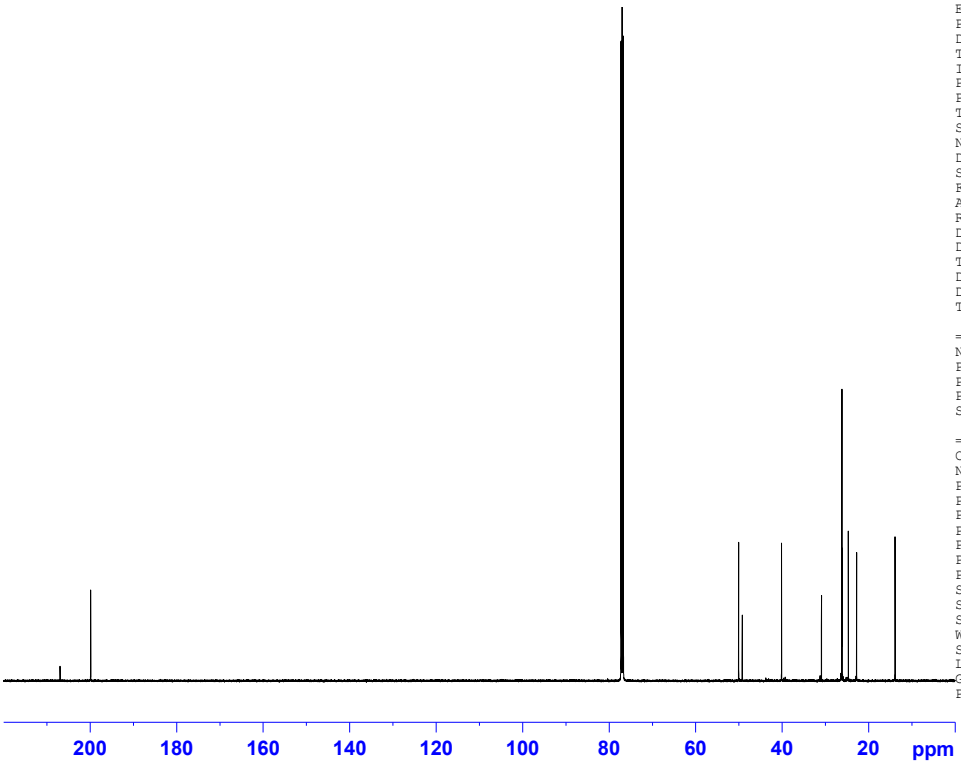


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PROCNO                 1
Date_           20101109
Time               8.00
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ             1.0486259 sec
RG                 1820
DW               16.000 usec
DE                20.00 usec
TE               298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -4.40 dB
PL1W        28.15752029 W
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
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NUC2                  1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               12.42 dB
PL13               18.42 dB
PL2W        15.19999981 W
PL12W        0.21869738 W
PL13W        0.05493430 W
SFO2        500.3020012 MHz
SI                 32768
SF          125.8005438 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
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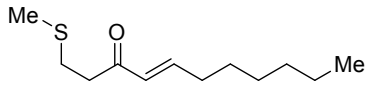

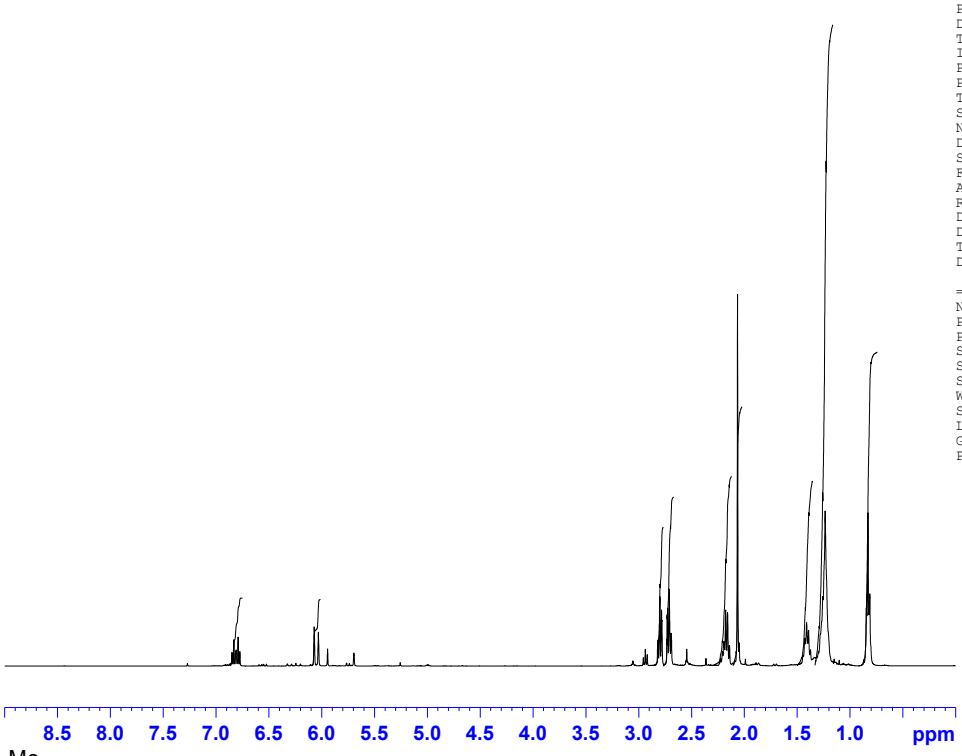


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PROCNO                1
Date_          20091214
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PULPROG            zg60
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   18
DW               60.400 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.00 usec
PL1               0.00 dB
SFO1        400.2024714 MHz
SI                32768
SF          400.2000028 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
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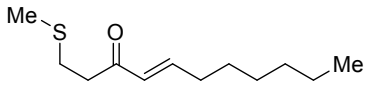

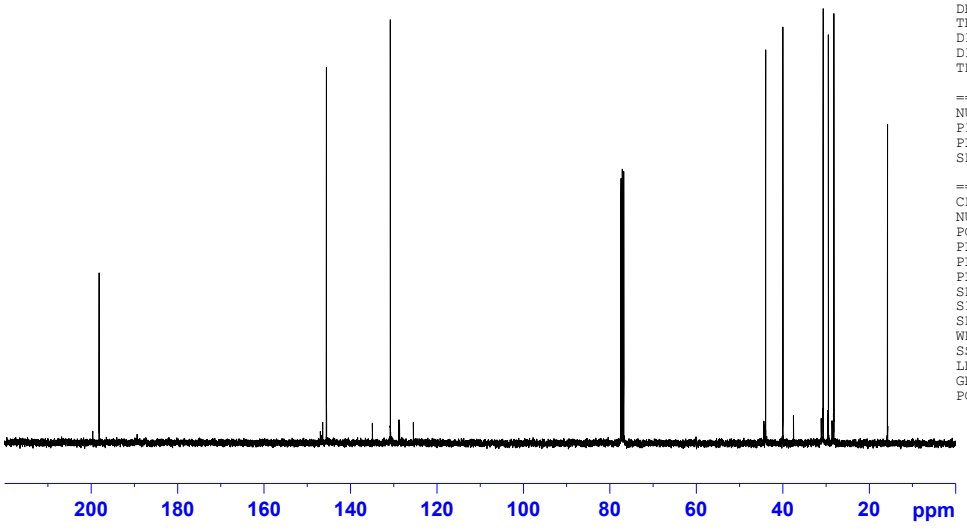


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NAME       Dec14-2009-33
EXPNO                 3
PROCNO                1
Date_          20091214
Time              15.52
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           26178.010 Hz
FIDRES         0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW               19.100 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1               0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2        400.2016008 MHz
SI                32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
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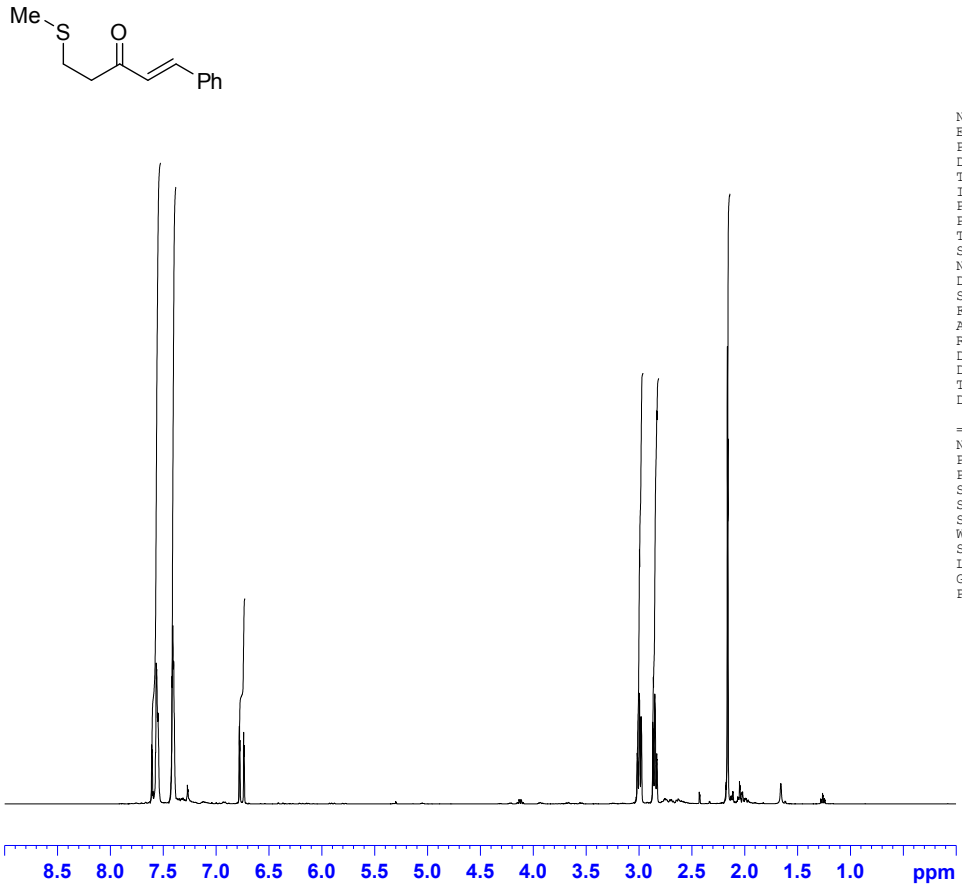


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EXPNO                  1
PROCNO                 1
Date_           20101108
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INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG            zg60
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                90.5
DW              60.400 usec
DE               7.50 usec
TE              300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.00 usec
PL1                0.00 dB
SFO1        400.2024714 MHz
SI                 32768
SF          400.2000028 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
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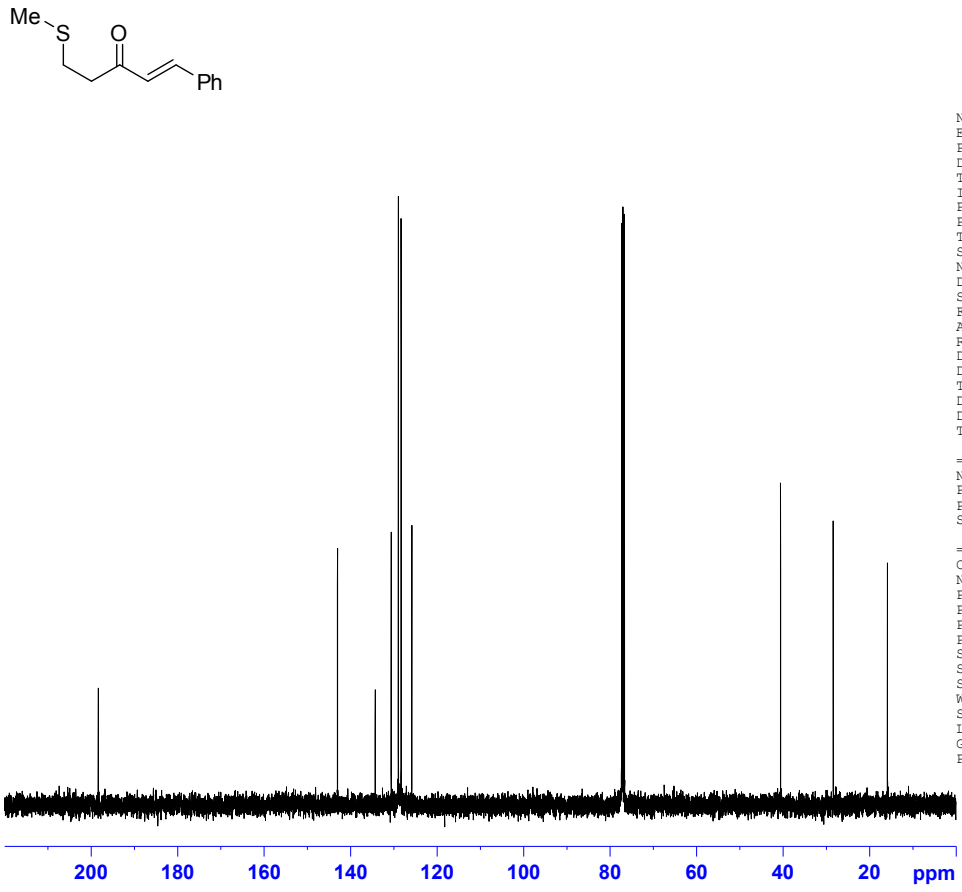


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TD                32768
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           26178.010 Hz
FIDRES         0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW              19.100 usec
DE               7.50 usec
TE              300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1                0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                0.00 dB
PL12               19.00 dB
PL13               25.00 dB
SFO2        400.2016008 MHz
SI                 32768
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WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
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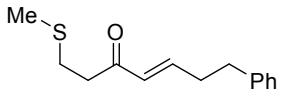

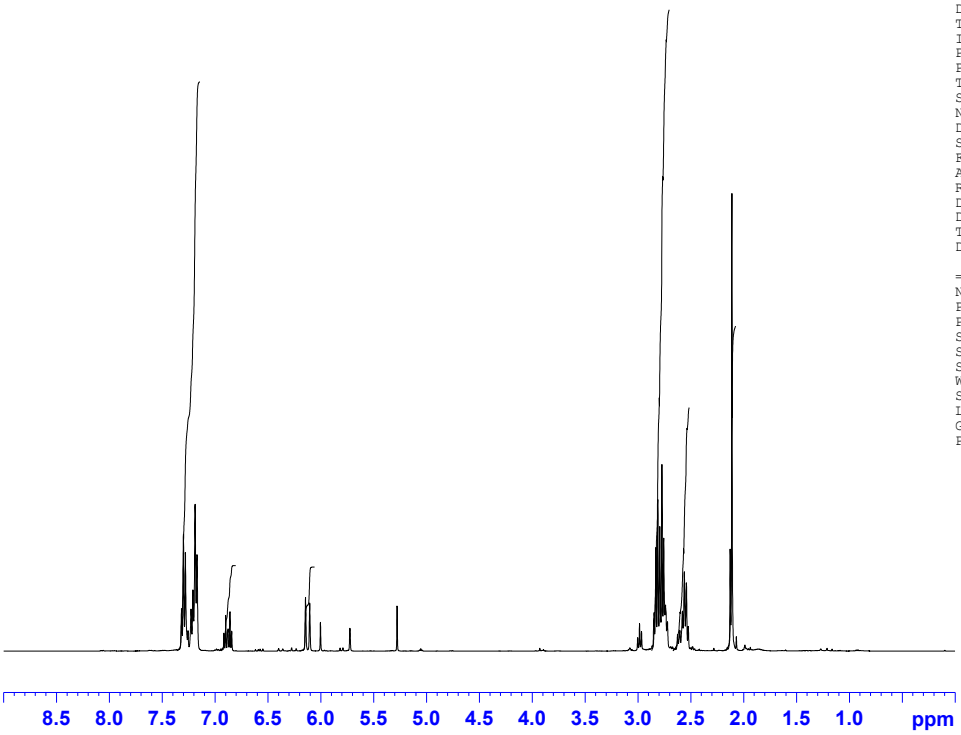


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TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 25.4
DW               60.400 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1                0.00 dB
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SI                 32768
SF          400.2000028 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
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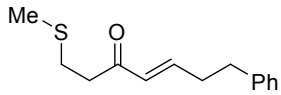

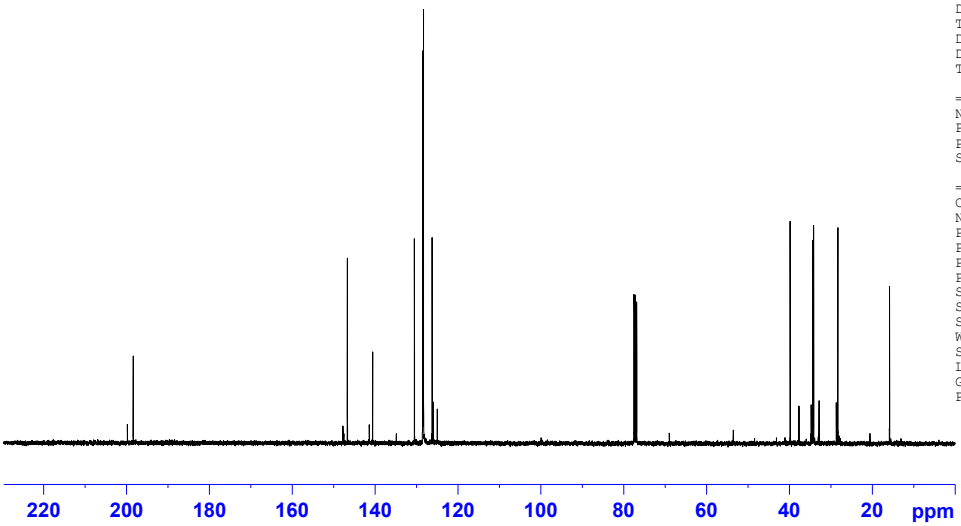


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PROCNO                1
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Time              23.21
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TD                32768
SOLVENT           CDCl3
NS                  256
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SWH           26178.010 Hz
FIDRES        0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW               19.100 usec
DE                7.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.50 usec
PL1                0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2        400.2016008 MHz
SI                 32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

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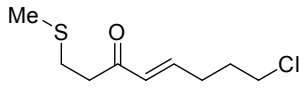
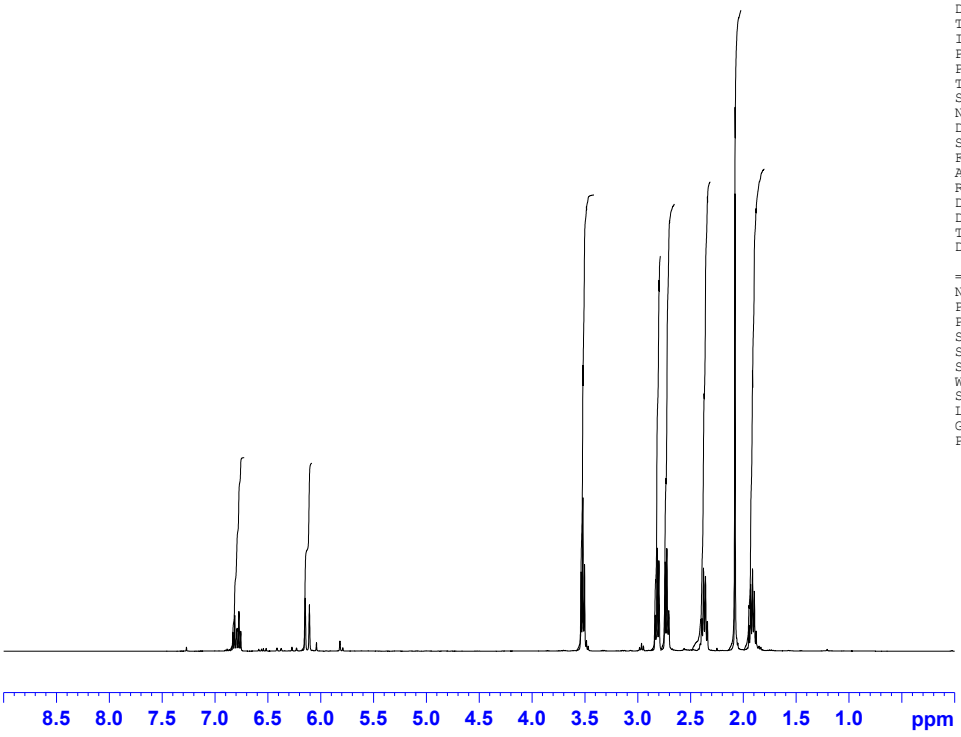
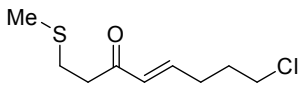
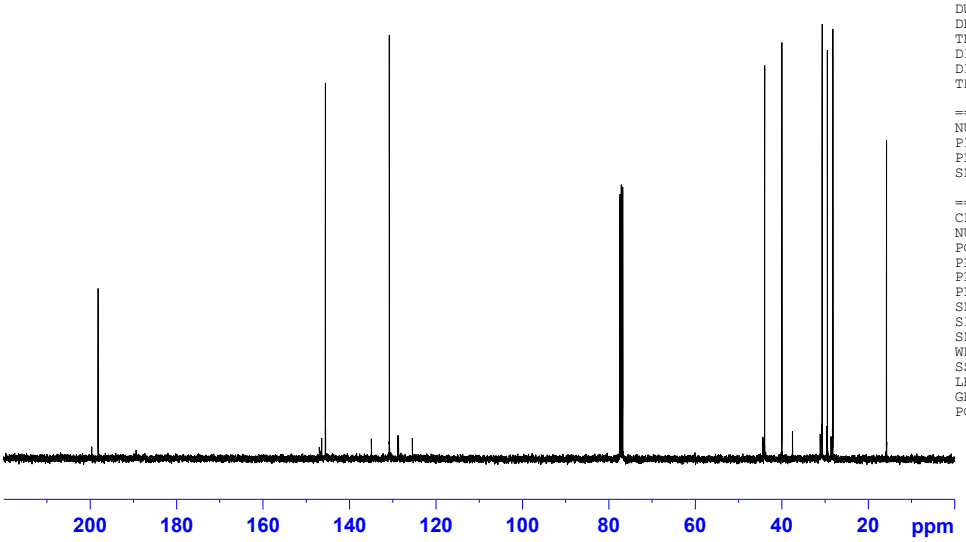





```
NAME       Dec14-2009-33
EXPNO                 1
PROCNO                1
Date_          20091214
Time              15.44
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG           zg60
TD               65536
SOLVENT          CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   32
DW               60.400 usec
DE                7.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.00 usec
PL1               0.00 dB
SFO1        400.2024714 MHz
SI                32768
SF          400.2000028 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```






```
NAME       Dec14-2009-33
EXPNO                 3
PROCNO                1
Date_          20091214
Time              15.52
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                  256
DS                    4
SWH           26178.010 Hz
FIDRES        0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW               19.100 usec
DE                7.50 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1               0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             19.00 dB
PL13             25.00 dB
SFO2        400.2016008 MHz
SI                32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

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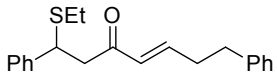
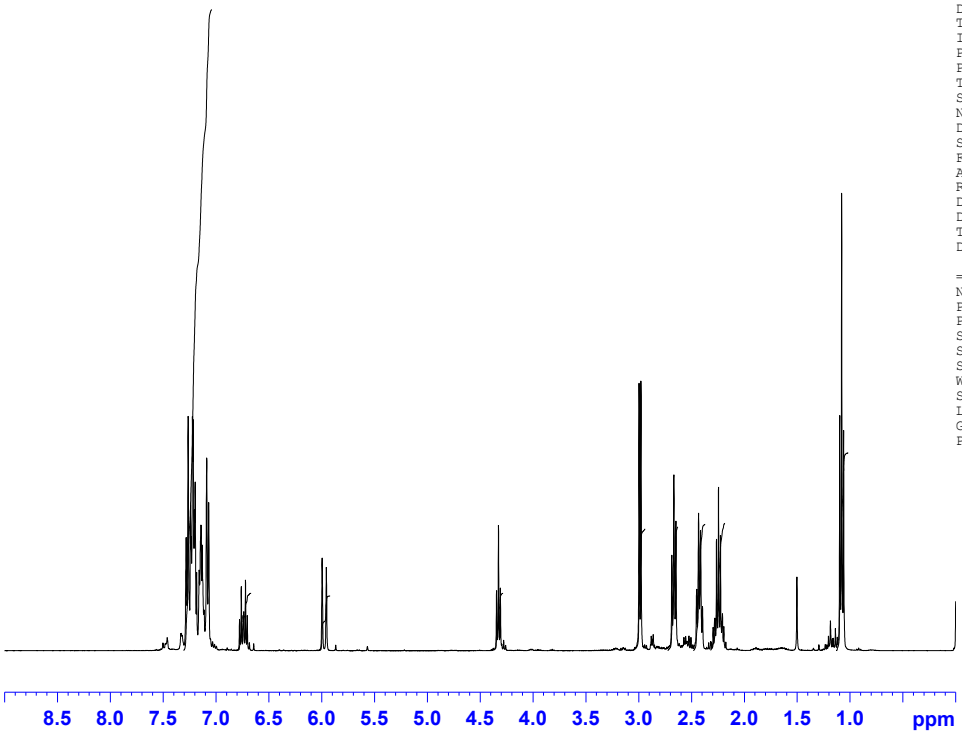
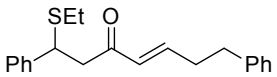
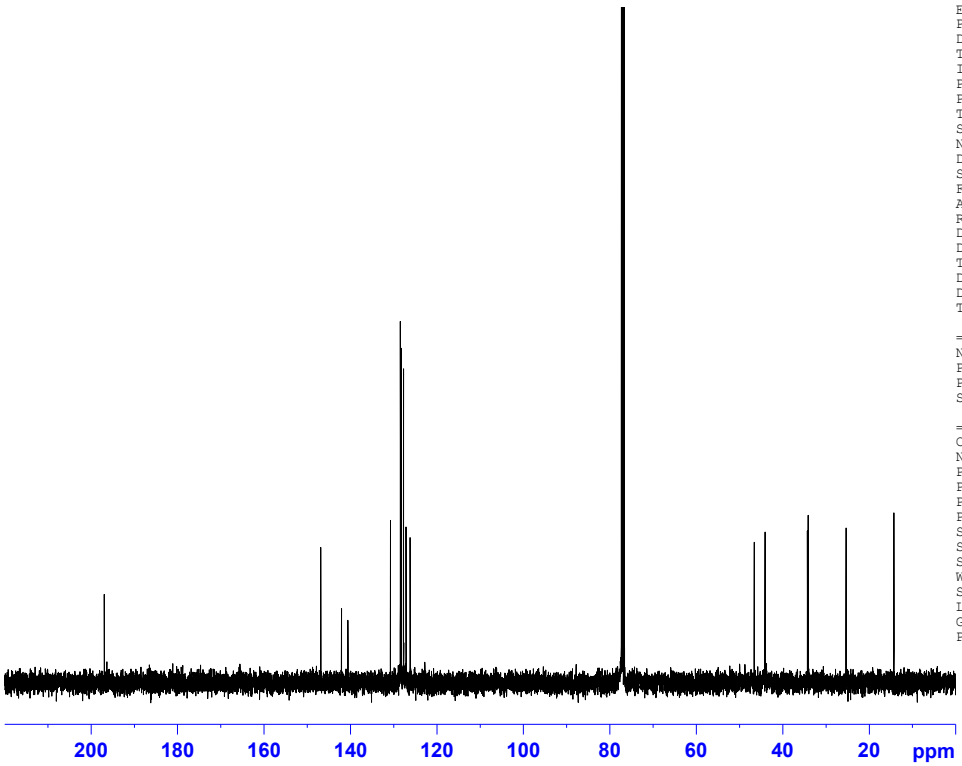





```
NAME                  1c
EXPNO                  1
PROCNO                 1
Date_           20100417
Time                9.16
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG             zg60
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 7.50 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.00 usec
PL1                 0.00 dB
SFO1         400.2024714 MHz
SI                 32768
SF           400.2000378 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```






```
NAME                  1c
EXPNO                  2
PROCNO                 1
Date_           20100417
Time                9.24
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            26178.010 Hz
FIDRES          0.798889 Hz
AQ              0.6259188 sec
RG                 32768
DW                19.100 usec
DE                 7.50 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                 0.00 dB
SFO1         100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               19.00 dB
PL13               25.00 dB
SFO2         400.2016008 MHz
SI                 32768
SF           100.6303718 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

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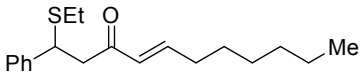
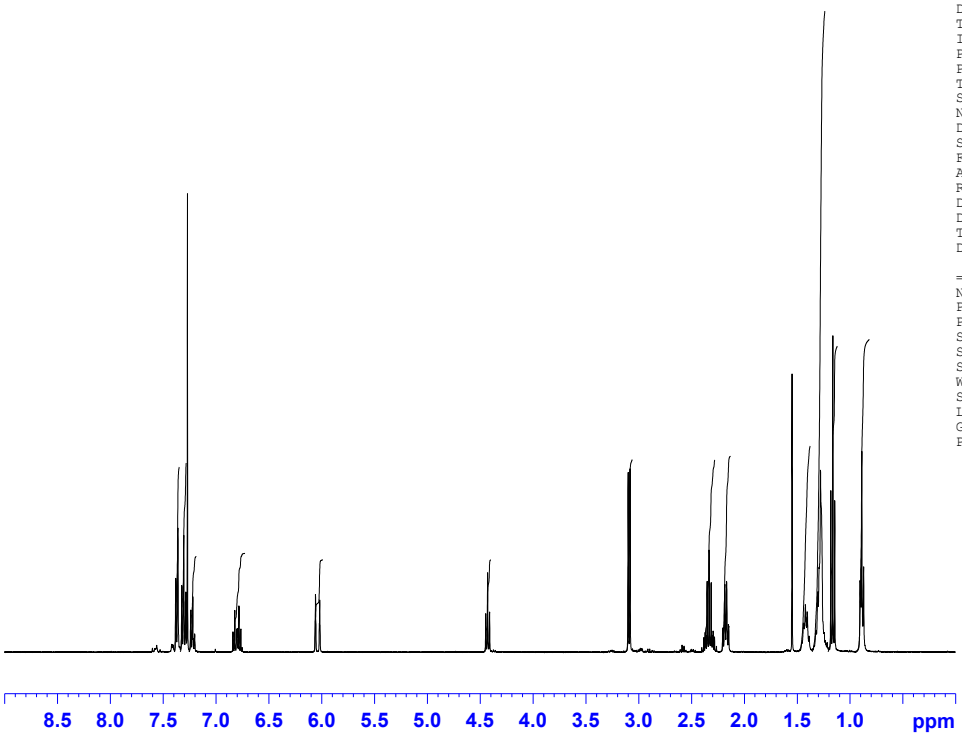
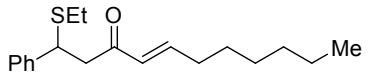
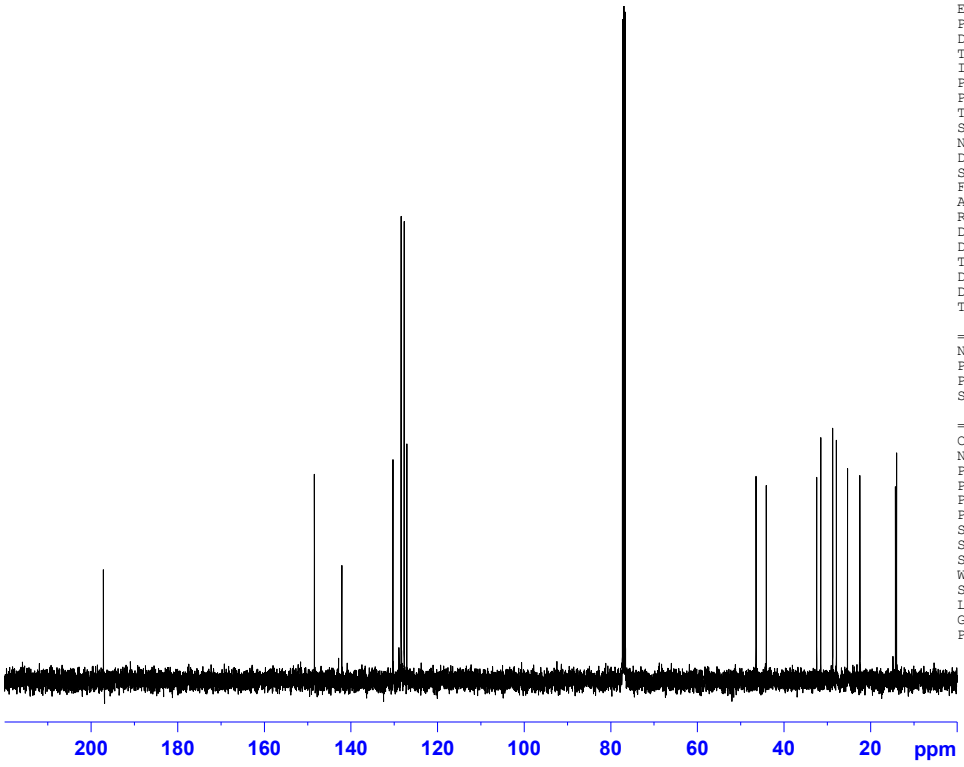





```
NAME          Sep18-2010-5
EXPNO                    1
PROCNO                   1
Date_             20100918
Time                 19.03
INSTRUM              av400
PROBHD   5 mm QNP 1H/13
PULPROG               zg60
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                   812.7
DW                  60.400 usec
DE                   7.50 usec
TE                  300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.00 usec
PL1                   0.00 dB
SFO1          400.2024714 MHz
SI                   32768
SF            400.2000028 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```






```
NAME                    1d
EXPNO                    2
PROCNO                   1
Date_             20100918
Time                 19.18
INSTRUM              av400
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     256
DS                       4
SWH              26178.010 Hz
FIDRES            0.798889 Hz
AQ                0.6259188 sec
RG                   32768
DW                  19.100 usec
DE                   7.50 usec
TE                  300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.50 usec
PL1                   0.00 dB
SFO1          100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 19.00 dB
PL13                 25.00 dB
SFO2          400.2016008 MHz
SI                   32768
SF            100.6303718 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

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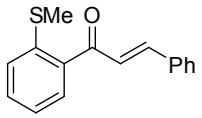
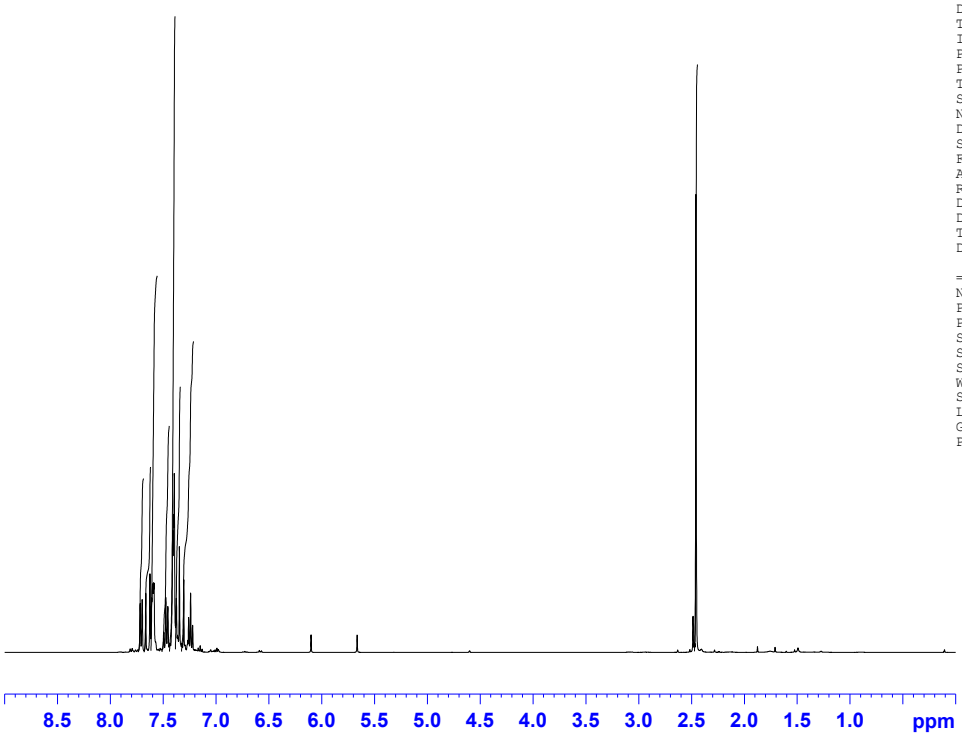
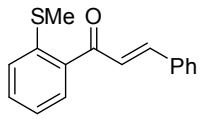
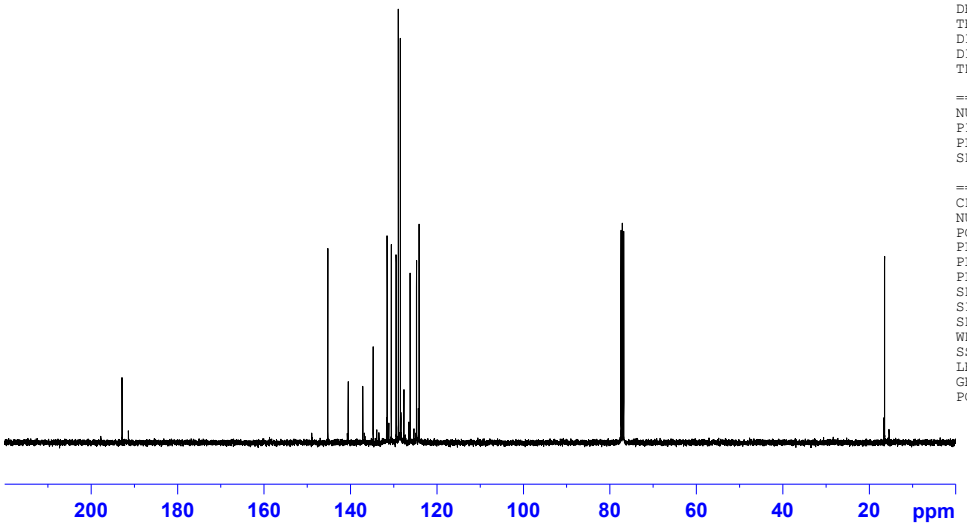





```
NAME      PY063021 DQX400
EXPNO                1
PROCNO               1
Date_         20100104
Time             21.03
INSTRUM          av400
PROBHD   5 mm QNP 1H/13
PULPROG          zg60
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                40.3
DW              60.400 usec
DE               7.50 usec
TE              300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1               0.00 dB
SFO1        400.2024714 MHz
SI                32768
SF          400.2000028 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```






```
NAME      PY063021 DQX400
EXPNO                2
PROCNO               1
Date_         20100104
Time             21.11
INSTRUM          av400
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          26178.010 Hz
FIDRES        0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW              19.100 usec
DE               7.50 usec
TE              300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.50 usec
PL1               0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             19.00 dB
PL13             25.00 dB
SFO2        400.2016008 MHz
SI                32768
SF          100.6303718 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

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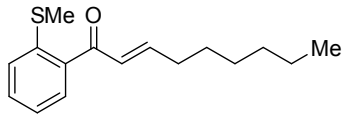
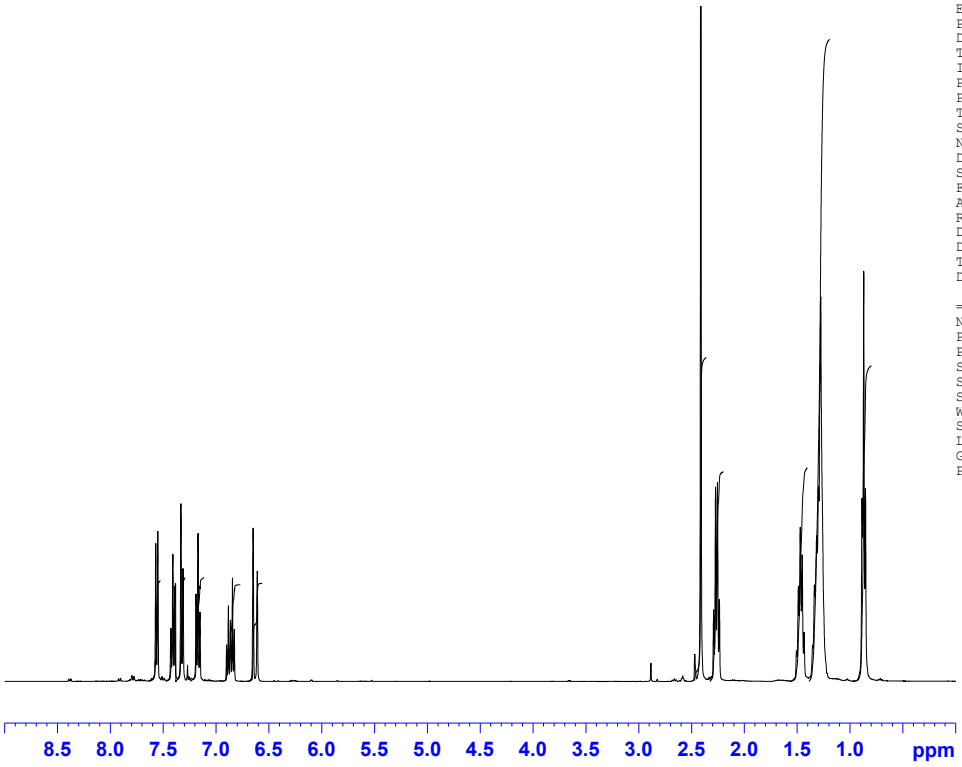
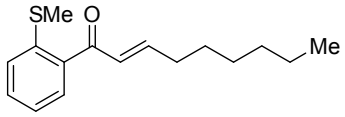
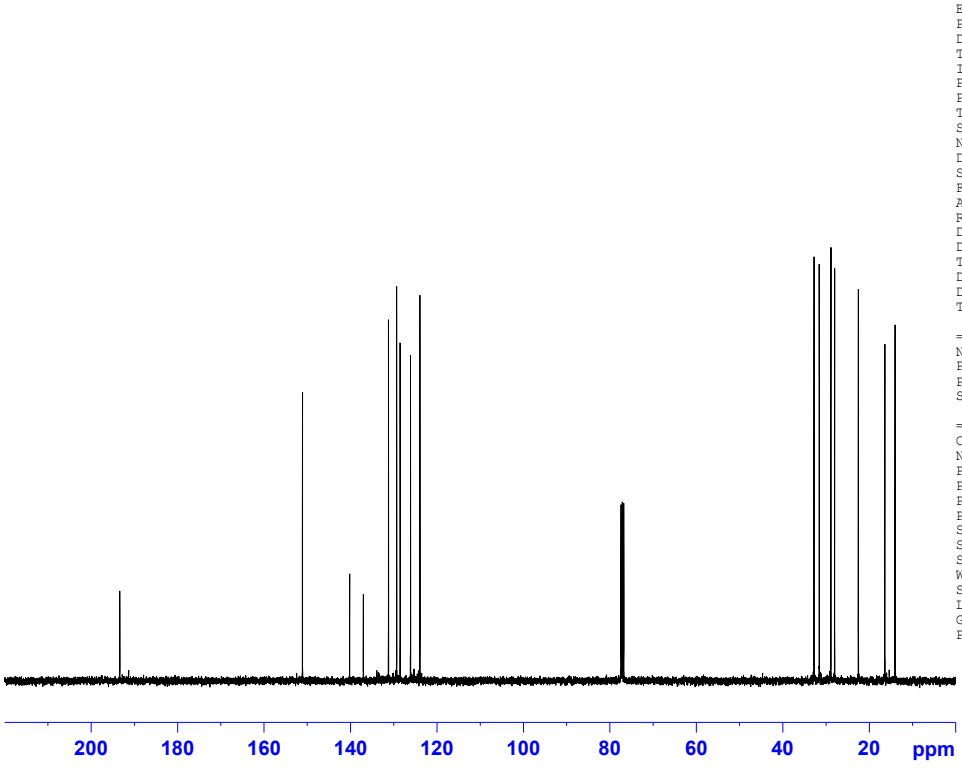





```
NAME                 Hex
EXPNO                  1
PROCNO                 1
Date_           20100104
Time               21.16
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG             zg60
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  28.5
DW                60.400 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1                0.00 dB
SFO1        400.2024714 MHz
SI                 32768
SF          400.2000028 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```






```
NAME                 Hex
EXPNO                  2
PROCNO                 1
Date_           20100104
Time               21.24
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            26178.010 Hz
FIDRES          0.798889 Hz
AQ              0.6259188 sec
RG                 32768
DW                19.100 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.50 usec
PL1                0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2        400.2016008 MHz
SI                 32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

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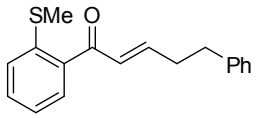
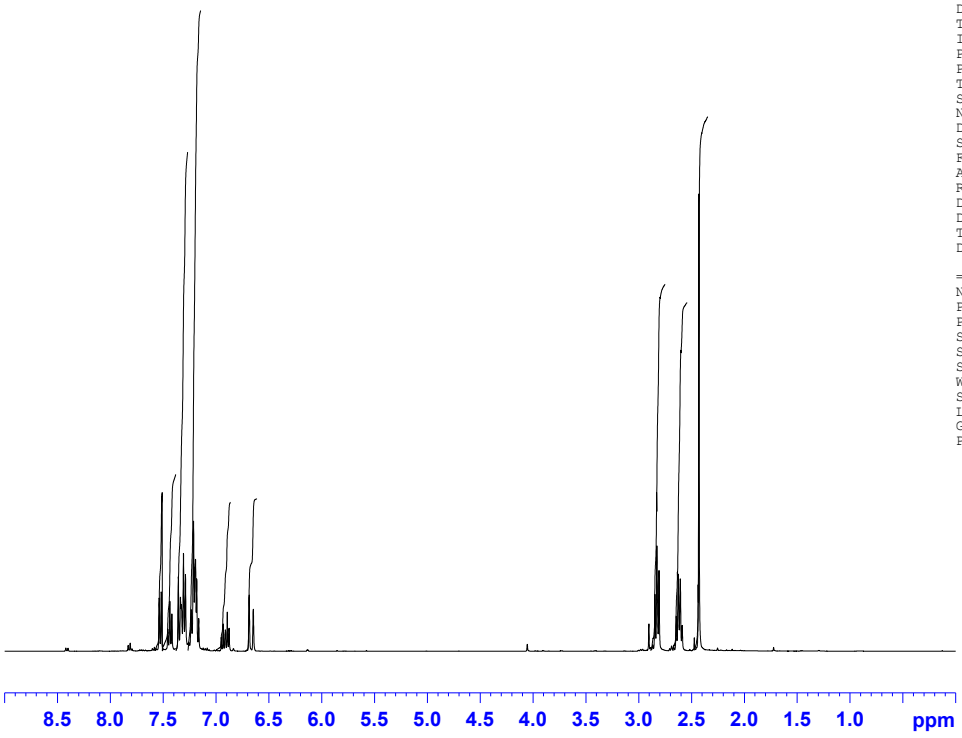
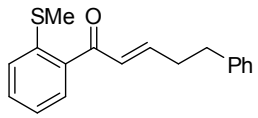
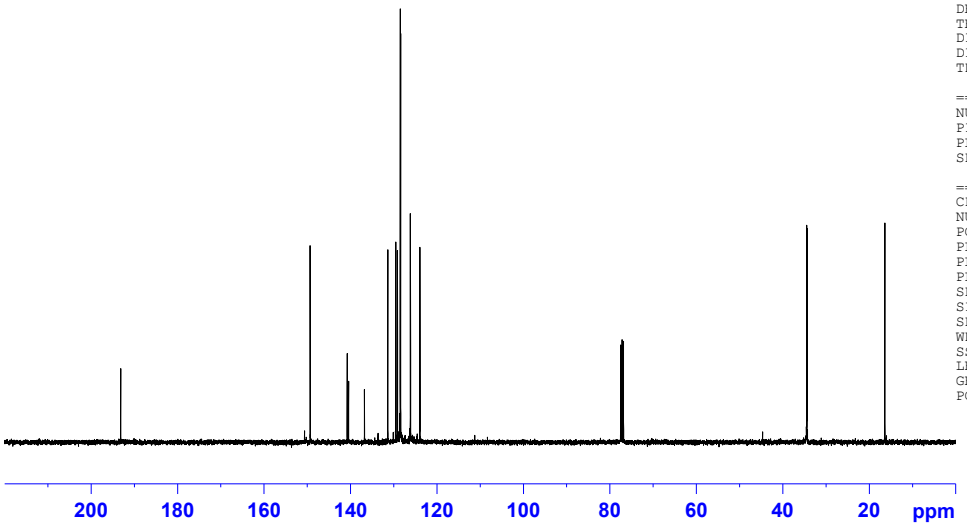





```
NAME            CH2CH2Ph
EXPNO                  1
PROCNO                 1
Date_           20100812
Time               12.15
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG             zg60
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  28.5
DW                60.400 usec
DE                 7.50 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.00 usec
PL1                0.00 dB
SFO1         400.2024714 MHz
SI                 32768
SF           400.2000028 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```






```
NAME            CH2CH2Ph
EXPNO                  2
PROCNO                 1
Date_           20100812
Time               12.23
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            26178.010 Hz
FIDRES          0.798889 Hz
AQ              0.6259188 sec
RG                 32768
DW                19.100 usec
DE                 7.50 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1                0.00 dB
SFO1         100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               19.00 dB
PL13               25.00 dB
SFO2         400.2016008 MHz
SI                 32768
SF           100.6303718 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

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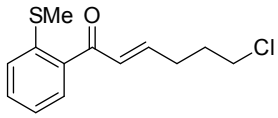
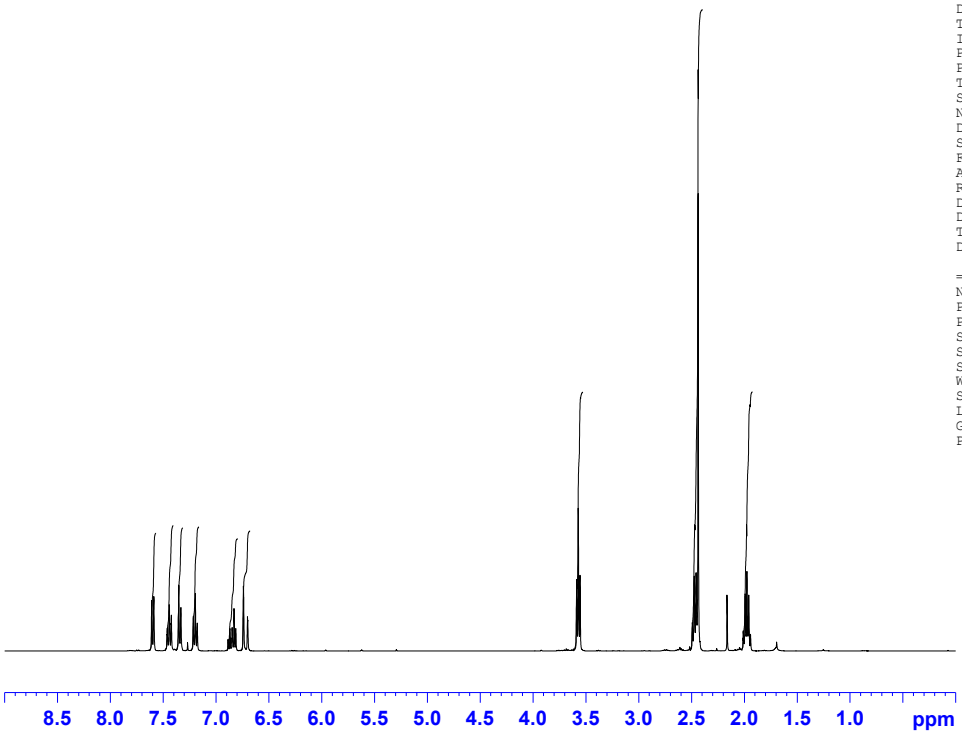
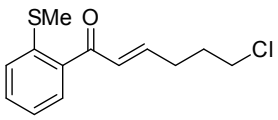
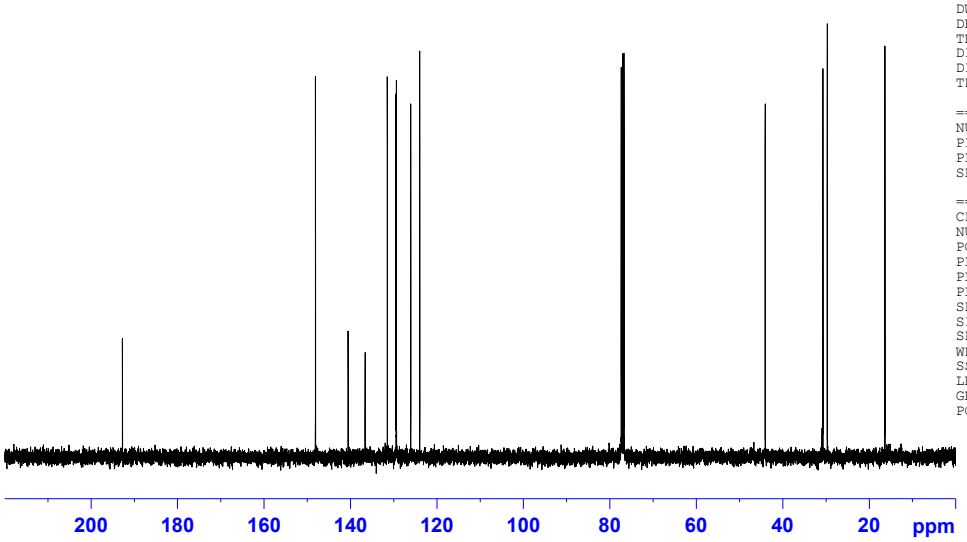





```
NAME                 C12
EXPNO                  1
PROCNO                 1
Date_           20091110
Time               10.49
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG             zg60
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.00 usec
PL1                0.00 dB
SFO1         400.2024714 MHz
SI                 32768
SF           400.2000028 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```






```
NAME                 C12
EXPNO                  3
PROCNO                 1
Date_           20091110
Time               11.01
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            26178.010 Hz
FIDRES          0.798889 Hz
AQ              0.6259188 sec
RG                 32768
DW                19.100 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1                0.00 dB
SFO1         100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2         400.2016008 MHz
SI                 32768
SF           100.6303718 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

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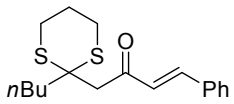
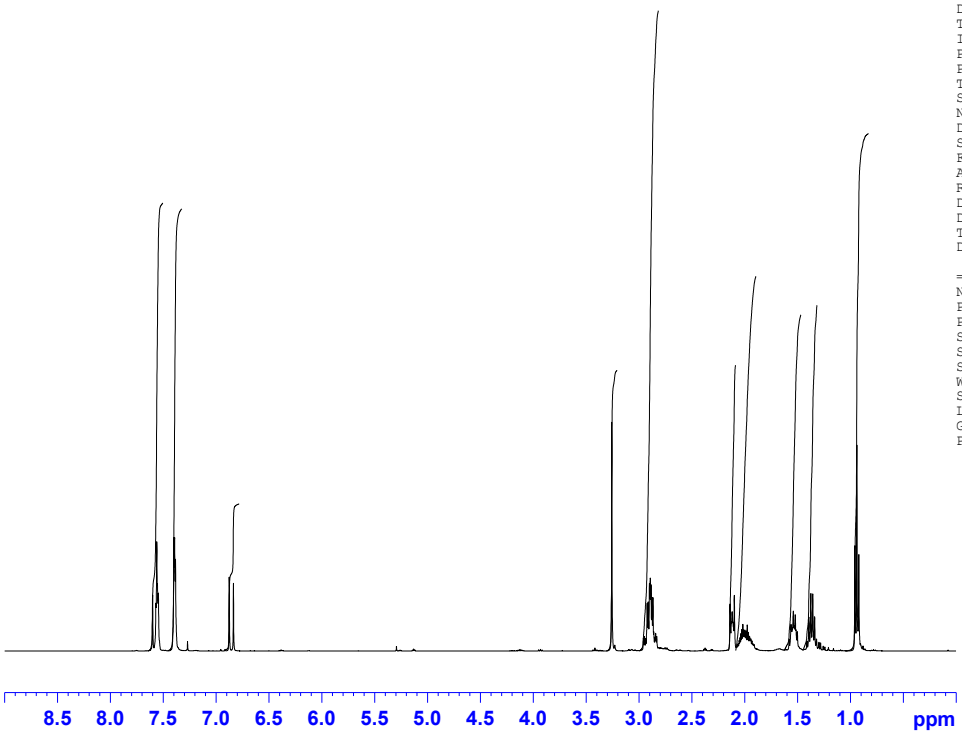
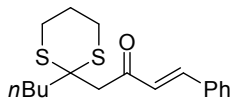
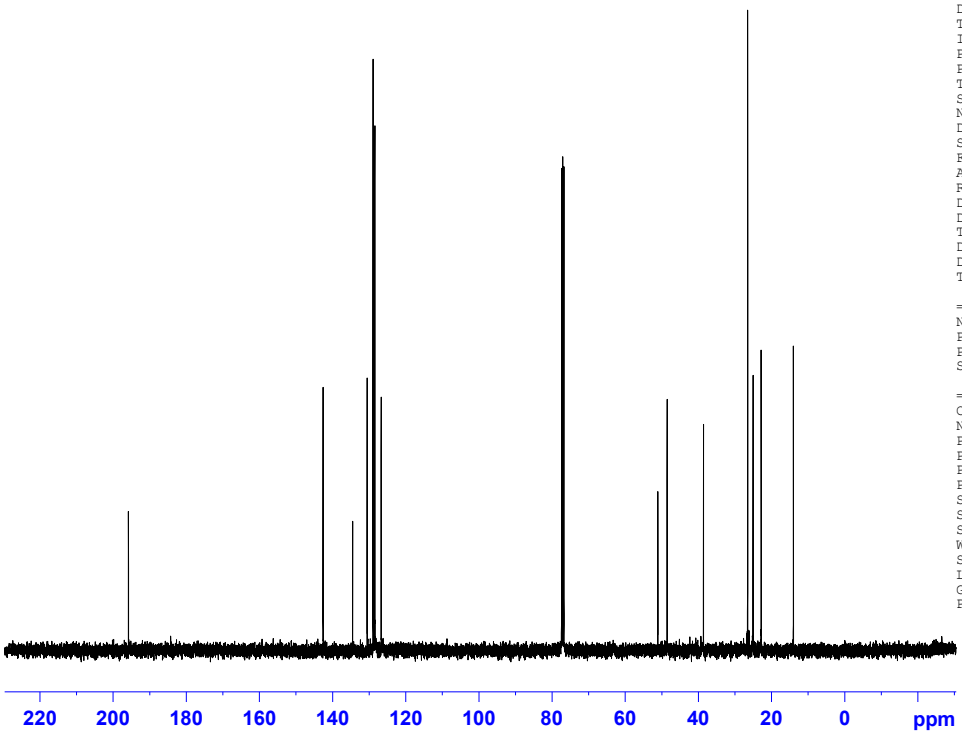





```
NAME       Aug09-2010-11
EXPNO                  1
PROCNO                 1
Date_           20100809
Time               13.19
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG             zg60
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  35.9
DW                60.400 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.00 usec
PL1                0.00 dB
SFO1         400.2024714 MHz
SI                 32768
SF           400.2000028 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```






```
NAME       Aug09-2010-11
EXPNO                  2
PROCNO                 1
Date_           20100809
Time               13.26
INSTRUM            av400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            26178.010 Hz
FIDRES          0.798889 Hz
AQ              0.6259188 sec
RG                 32768
DW                19.100 usec
DE                 7.50 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1                0.00 dB
SFO1         100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              19.00 dB
PL13              25.00 dB
SFO2         400.2016008 MHz
SI                 32768
SF           100.6303718 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

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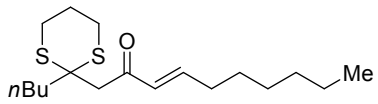
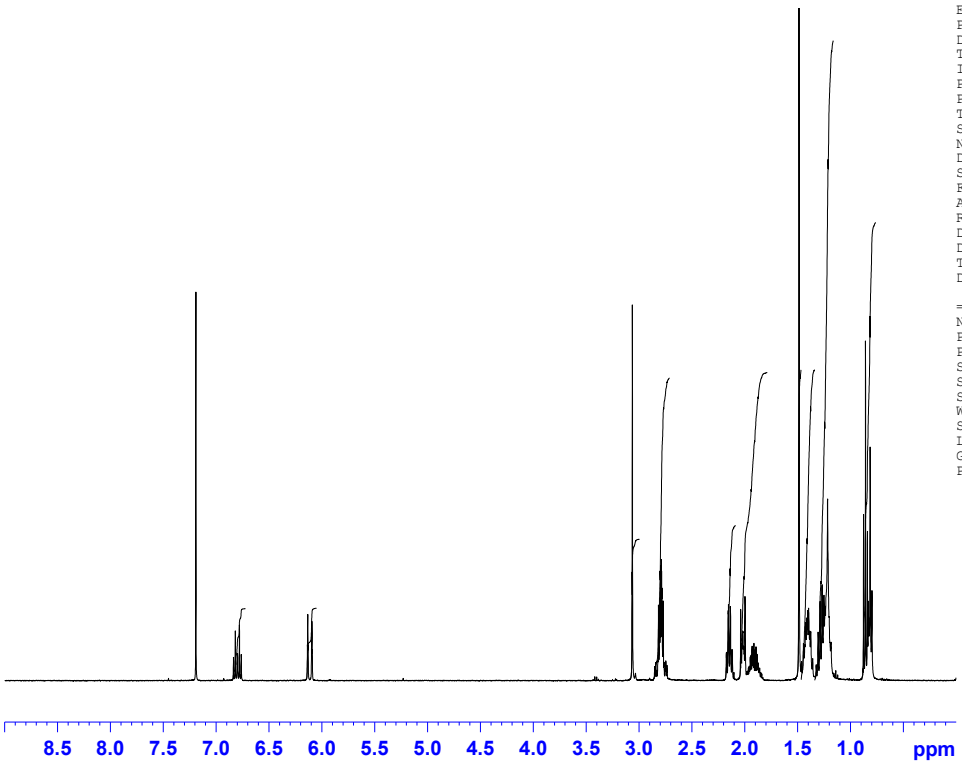
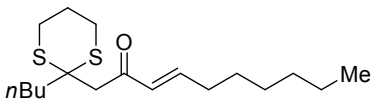
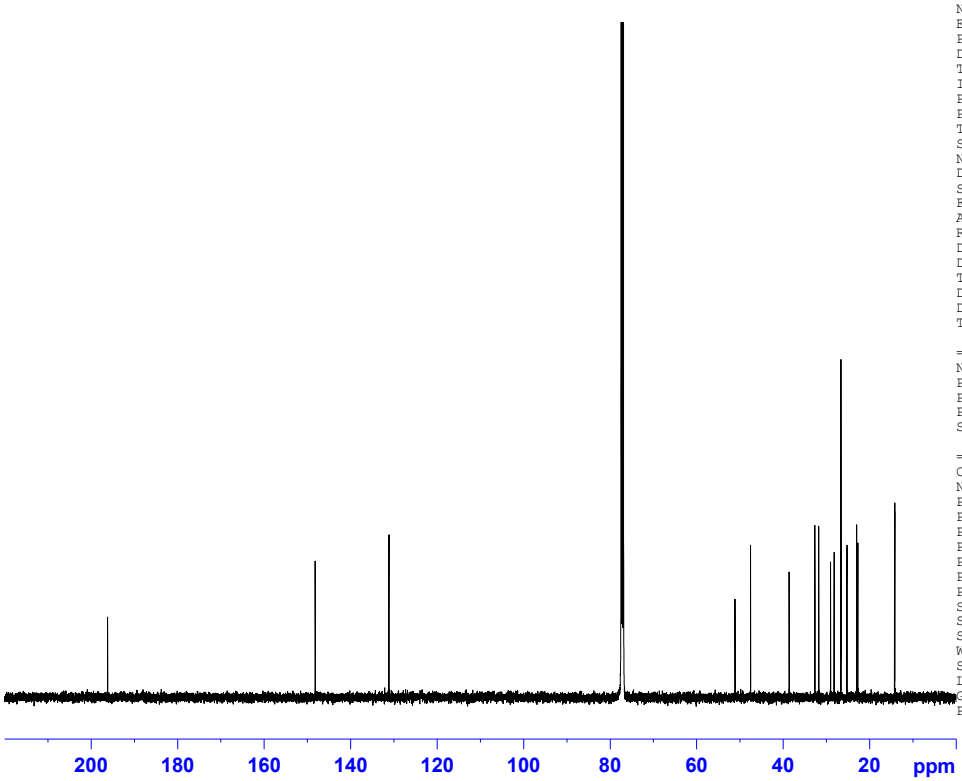





```
NAME       Oct12-2010-55
EXPNO                 5
PROCNO                1
Date_          20101012
Time_             21.21
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG            zg60
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                574.7
DW               60.400 usec
DE                7.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1               0.00 dB
SFO1        400.2024714 MHz
SI                32768
SF          400.2000337 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00
```






```
NAME          p11840311
EXPNO                 1
PROCNO                1
Date_          20101104
Time               3.54
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           31250.000 Hz
FIDRES        0.476837 Hz
AQ            1.0486259 sec
RG                1820
DW               16.000 usec
DE                20.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1              -4.40 dB
PL1W        28.15752029 W
SFO1       125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               -6.00 dB
PL12              12.42 dB
PL13              18.42 dB
PL2W        15.19999981 W
PL12W        0.21869738 W
PL13W        0.05493430 W
SFO2       500.3020012 MHz
SI                32768
SF         125.8005176 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

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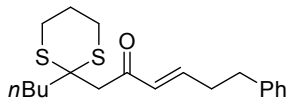
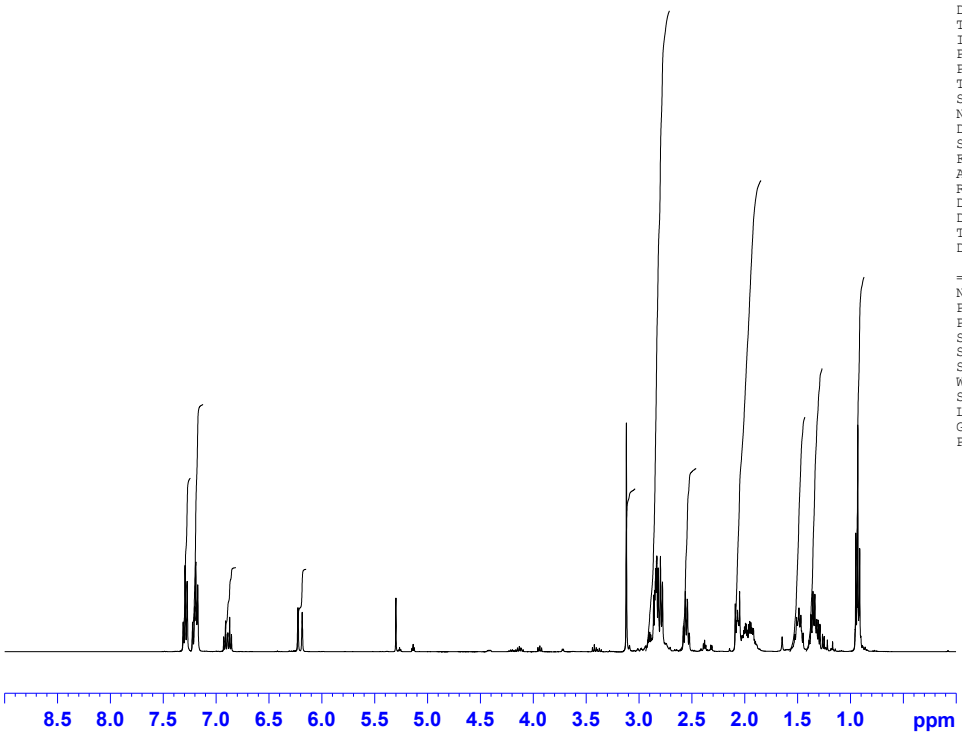
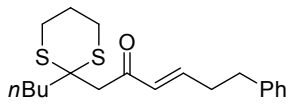
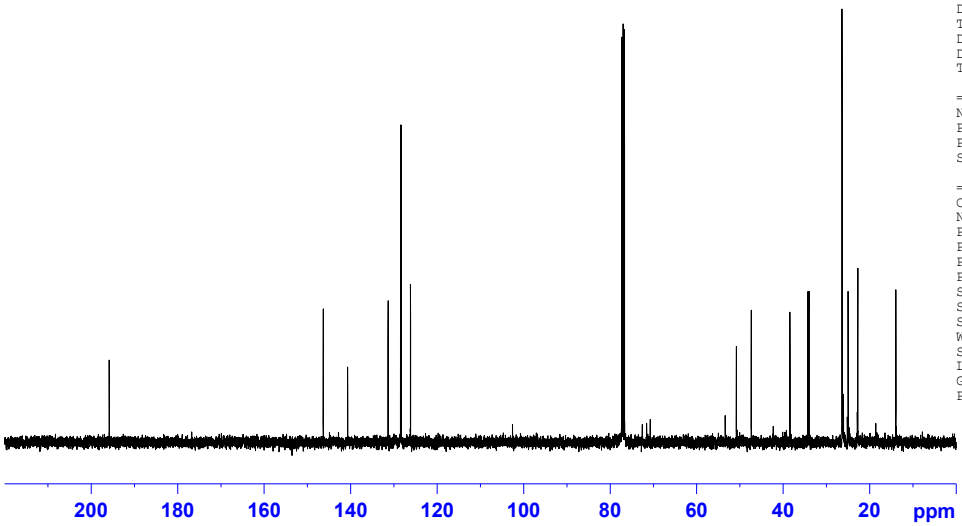





```
NAME       Aug04-2010-38
EXPNO                 1
PROCNO                1
Date_          20100804
Time              15.59
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG            zg60
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 35.9
DW               60.400 usec
DE                7.50 usec
TE               300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.00 usec
PL1               0.00 dB
SFO1        400.2024714 MHz
SI                32768
SF          400.2000028 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```






```
NAME       Aug09-2010-33
EXPNO                 2
PROCNO                1
Date_          20100809
Time              20.57
INSTRUM           av400
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           26178.010 Hz
FIDRES        0.798889 Hz
AQ            0.6259188 sec
RG                32768
DW               19.100 usec
DE                7.50 usec
TE               300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1               13C
P1                9.50 usec
PL1               0.00 dB
SFO1        100.6403931 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             19.00 dB
PL13             25.00 dB
SFO2        400.2016008 MHz
SI                32768
SF          100.6303718 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```