Electronic Supplementary Material (ESI) for RSC Advances.

Cs_2CO_3-promoted cross-dehydrogenative coupling of thiophenols with active methylene compounds

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SUPPORTING INFORMATION

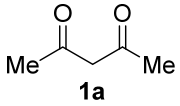
Content:

1. General Information

All reactions were carried out under an atmosphere of air using oven-dried glassware and standard syringe/septa techniques. Petroleum ether refers to the petroleum fraction bp 40~60 °C. Commercial reagents were used without purification unless otherwise noted. Flash chromatography was performed using the indicated solvent system on silica gel standard grade 60 (230–400 mesh). ^{1}H NMR spectra were recorded on a 400 MHz spectrometer. ^{13}C NMR spectra were recorded on a 100 MHz spectrometer. Chemical shifts are reported relative to $CDCl_3$ (δ 7.26 ppm) for ^{1}H NMR and $CDCl_3$ (δ 77.00 ppm) for ^{13}C NMR. High-resolution mass spectra (HRMS) were recorded on ESI-TOF. Melting points (mp) were uncorrected and measured on micro melting point apparatus.

2. Overview of Substrates Numbering

Active methylene compounds:



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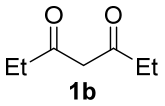

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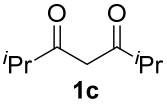

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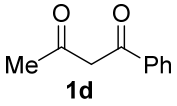

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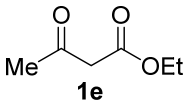

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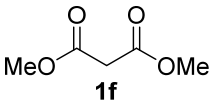

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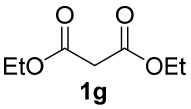

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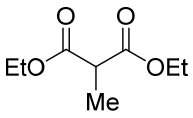

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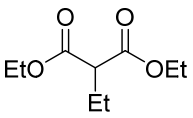
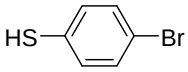


1i

Thiols:



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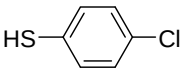

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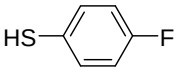

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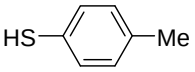

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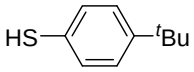

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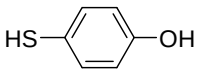

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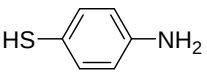

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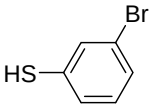

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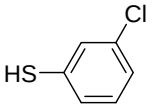

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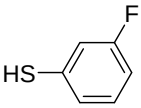

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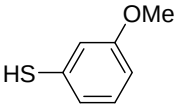

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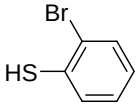

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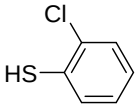

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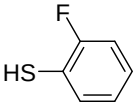

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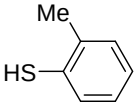

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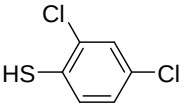

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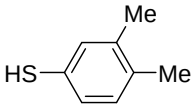

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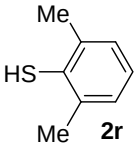

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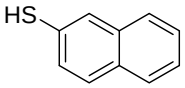

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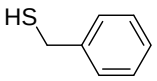

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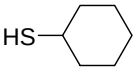
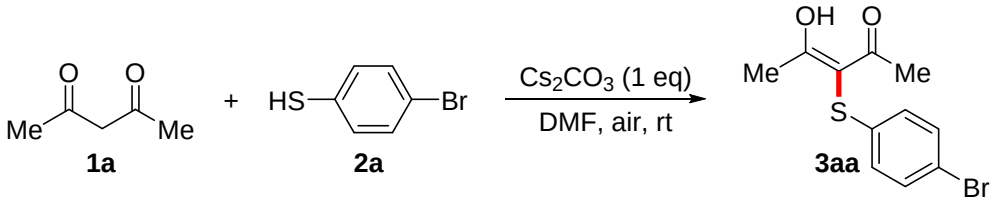


2u

3. General Procedure for the CDC Reactions

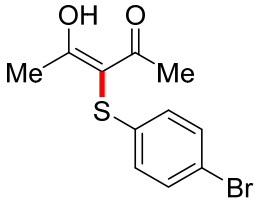


To a solution of acetylacetone **1a** (40 mg, 0.4 mmol) and 4-bromo-thiophenol **2a** (151 mg, 0.8 mmol) in DMF (2 mL) was added Cs_2CO_3 (130 mg, 0.4 mmol). The mixture was stirred at room temperature under air atmosphere for 6 h. The mixture was then added to water (5 mL). The resulting mixture was extracted with EtOAc (10 mL) for three times. The combined organic layers were washed with water and brine, dried over Na_2SO_4 and concentrated under reduced pressure. After removal of the solvent, the residue was then purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (99:1) to give the desired **3aa** (107 mg, 93%) as a white solid.

4. Characterizations of Compounds 3

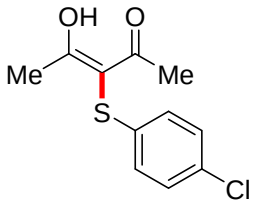
The known compounds **3aa**,[1] **3ab**,[1] **3ac**,[1] **3ad**,[1] **3ah**,[1] **3ak**,[1] **3al**,[1] **3ao**,[1] **3ba**,[2] **3ca**,[2] and **3fa**[3] showed characterization data in full agreement with previously reported data.

3-(4-Bromophenylthio)-4-hydroxypent-3-en-2-one (3aa)[1]



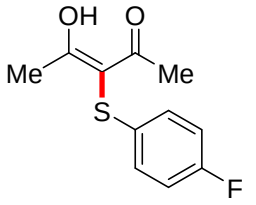
White solid (107 mg, 93%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.28 (s, 1H), 7.38 (d, J = 8.4 Hz, 2H), 6.95 (d, J = 8.4 Hz, 2H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 137.0, 132.2, 126.2, 118.8, 101.1, 24.3.

3-(4-Chlorophenylthio)-4-hydroxypent-3-en-2-one (3ab)[1]



White solid (60 mg, 62%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.27 (s, 1H), 7.24 (d, J = 8.4 Hz, 2H), 7.01 (d, J = 8.4 Hz, 2H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 136.3, 131.1, 129.3, 125.9, 101.3, 24.3.

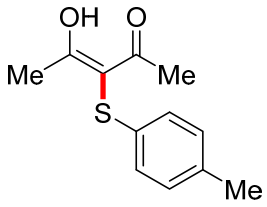
3-(4-Fluorophenylthio)-4-hydroxypent-3-en-2-one (3ac)[1]



Colorless oil (68 mg, 75%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.24 (s, 1H), 7.07–7.04 (m, 2H), 7.01–6.97 (m, 2H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 161.0

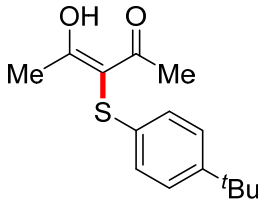
(d, J = 243 Hz), 132.8 (d, J = 3.2 Hz), 126.5 (d, J = 7.7 Hz), 116.3 (d, J = 22.0 Hz), 102.1 (d, J = 1.1 Hz), 24.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.5.

4-Hydroxy-3-(*p*-tolylthio)pent-3-en-2-one (3ad)[1]



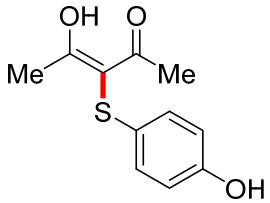
Colorless oil (77 mg, 86%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.26 (s, 1H), 7.10 (d, J = 8.4 Hz, 2H), 7.00 (d, J = 8.2 Hz, 2H), 2.35 (s, 6H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 135.0, 134.1, 129.9, 124.8, 102.0, 24.3, 20.8.

3-((4-(*tert*-Butyl)phenyl)thio)-4-hydroxypent-3-en-2-one (3ae)



White solid (92 mg, 87%): mp 104−106 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.26 (s, 1H), 7.31 (d, J = 8.5 Hz, 2H), 7.03 (d, J = 8.4 Hz, 2H), 2.35 (s, 6H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 148.4, 134.2, 126.2, 124.6, 102.0, 34.3, 31.3, 24.4; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{20}O_2SNa$ 287.1076; Found 287.1078.

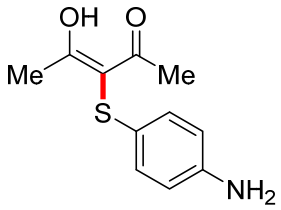
4-Hydroxy-3-((4-hydroxyphenyl)thio)pent-3-en-2-one (3af)



White solid (45 mg, 50%): mp 105−106 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.11 (s, 1H), 6.99 (d, J = 8.8 Hz, 2H), 6.78 (d, J = 8.8 Hz, 2H), 5.51 (brs, 1H), 2.36 (s, 6H);

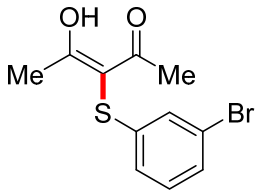
^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 154.0, 128.3, 127.1, 116.4, 103.2, 24.5; HRMS (ESI-TOF) *m*/*z*: $[M + Na]^+$ Calcd for $C_{11}H_{12}O_3SNa$ 247.0399; Found 247.0404.

3-((4-Aminophenyl)thio)-4-hydroxypent-3-en-2-one (3ag)



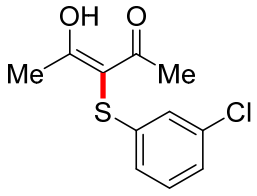
Brown oil (47 mg, 53%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.12 (brs, 1H), 6.94 (d, J = 8.8 Hz, 2H), 6.62 (d, J = 8.4 Hz, 2H), 3.43 (brs, 2H), 2.36 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.8, 144.7, 127.4, 125.4, 116.0, 103.7, 24.4; HRMS (ESI-TOF) *m*/*z*: $[M + Na]^+$ Calcd for $C_{11}H_{13}NO_2SNa$ 246.0559; Found 246.0561.

3-((3-Bromophenyl)thio)-4-hydroxypent-3-en-2-one (3ah)[1]



Colorless oil (70 mg, 61%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.30 (s, 1H), 7.24−7.19 (m, 2H), 7.12 (dd, J = 8.0, 7.2 Hz, 1H), 7.00−6.98 (m, 1H), 2.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 140.2, 130.4, 128.2, 127.0, 123.3, 123.0, 100.7, 24.2.

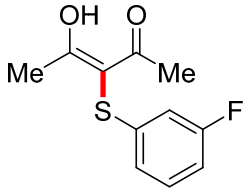
3-((3-Chlorophenyl)thio)-4-hydroxypent-3-en-2-one (3ai)



Colorless oil (78 mg, 80%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.31 (s, 1H), 7.20 (dd, J = 8.0, 8.0 Hz, 1H), 7.13−7.03 (m, 2H), 6.99−6.94 (m, 1H), 2.33 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 140.0, 135.2, 130.2, 125.4, 124.2, 122.6, 100.8, 24.3;

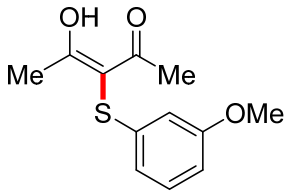
HRMS (ESI-TOF) *m*/*z*: [M + Na]$^+$ Calcd for $C_{11}H_{11}ClO_2SNa$ 265.0060; Found 265.0064.

3-((3-Fluorophenyl)thio)-4-hydroxypent-3-en-2-one (3aj)



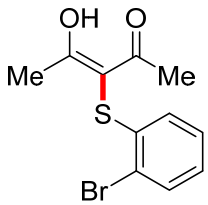
Colorless oil (81 mg, 89%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.36 (s, 1H), 7.31−7.26 (m, 1H), 6.93−6.91 (m, 1H), 6.89−6.82 (m, 2H), 2.39 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 163.4 (d, J = 247 Hz), 140.5 (d, J = 7.2 Hz), 130.5 (d, J = 8.5 Hz), 120.2 (d, J = 2.9 Hz), 112.2 (d, J = 21.3 Hz), 111.5 (d, J = 24.1 Hz), 100.9 (d, J = 1.0 Hz), 24.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.7; HRMS (ESI-TOF) *m*/*z*: [M + Na]$^+$ Calcd for $C_{11}H_{11}FO_2SNa$ 249.0356; Found 249.0359.

4-Hydroxy-3-((3-methoxyphenyl)thio)pent-3-en-2-one (3ak)



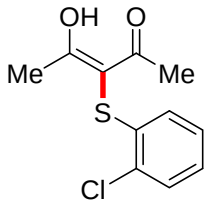
Colorless oil (73 mg, 76%):[12c] ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.27 (s, 1H), 7.19 (dd, J = 8.0, 8.0 Hz, 1H), 6.68−6.66 (m, 2H), 6.64−6.63 (m, 1H), 3.78 (s, 3H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 160.3, 139.3, 130.0, 116.9, 110.5 (2C), 101.4, 55.2, 24.4.

3-((2-Bromophenyl)thio)-4-hydroxypent-3-en-2-one (3al)[1]



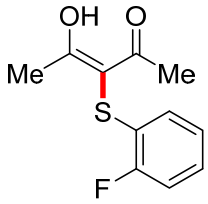
White solid (108 mg, 94%): mp 84−85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.37 (s, 1H), 7.52 (dd, J = 7.9, 1.1 Hz, 1H), 7.26−7.21 (m, 1H), 7.03−6.98 (m, 1H), 6.84 (dd, J = 7.9, 1.3 Hz, 1H), 2.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 138.5, 133.2, 128.0, 126.2, 124.3, 120.1, 100.9, 24.2.

3-((2-Chlorophenyl)thio)-4-hydroxypent-3-en-2-one (3am)



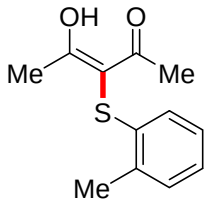
Colorless oil (70 mg, 72%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.38 (s, 1H), 7.35 (dd, J = 7.9, 1.3 Hz, 1H), 7.21−7.16 (m, 1H), 7.10−7.05 (m, 1H), 6.86 (dd, J = 7.9, 1.5 Hz, 1H), 2.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 136.6, 130.5, 129.9, 127.4, 125.9, 124.2, 100.1, 24.2; HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{11}H_{11}ClO_2SNa$ 265.0060; Found 265.0064.

3-((2-Fluorophenyl)thio)-4-hydroxypent-3-en-2-one (3an)



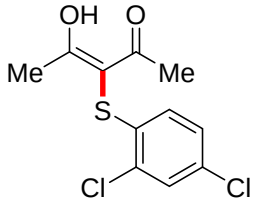
Colorless oil (72 mg, 80%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.32 (s, 1H), 7.17−7.11 (m, 1H), 7.09−7.02 (m, 2H), 6.96−6.91 (m, 1H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 159.3 (d, J = 243 Hz), 126.8 (d, J = 7.4 Hz), 126.1 (d, J = 2.6 Hz), 124.9 (d, J = 16.6 Hz), 124.8 (d, J = 3.5 Hz), 115.7 (d, J = 20.9 Hz), 99.7, 24.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.6; HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{11}H_{11}FO_2SNa$ 249.0356; Found 249.0359.

4-Hydroxy-3-(*o*-tolylthio)pent-3-en-2-one (3ao)[1]



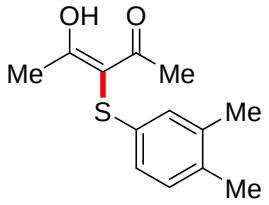
Colorless oil (68 mg, 76%):[12c] ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.33 (s, 1H), 7.17−7.11 (m, 2H), 7.06 (dd, J = 7.2, 6.9 Hz, 1H), 6.86−6.83 (m, 1H), 2.39 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 136.5, 134.3, 130.3, 126.7, 124.7, 122.7, 100.7, 24.2, 19.6.

3-((2,4-Dichlorophenyl)thio)-4-hydroxypent-3-en-2-one (3ap)



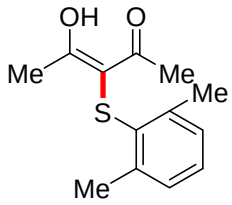
Colorless oil (71 mg, 64%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.37 (s, 1H), 7.37 (d, J = 2.1 Hz, 1H), 7.17 (dd, J = 8.5, 2.2 Hz, 1H), 6.78 (d, J = 8.5 Hz, 1H), 2.30 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 135.5, 131.1, 131.0, 129.7, 127.7, 125.1, 99.8, 24.2; HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{11}H_{10}Cl_2O_2SNa$ 298.9671; Found 298.9670.

3-((3,4-Dimethylphenyl)thio)-4-hydroxypent-3-en-2-one (3aq)



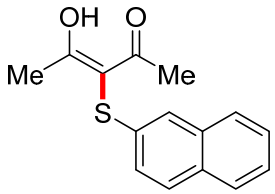
Colorless oil (69 mg, 73%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.26 (s, 1H), 7.05 (d, J = 7.9 Hz, 1H), 6.89 (s, 1H), 6.83 (dd, J = 7.9, 1.8 Hz, 1H), 2.36 (s, 6H), 2.24 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 137.6, 134.3, 133.8, 130.4, 126.0, 122.2, 102.0, 24.4, 19.8, 19.1; HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{13}H_{16}O_2SNa$ 259.0763; Found 259.0766.

3-((2,6-Dimethylphenyl)thio)-4-hydroxypent-3-en-2-one (3ar)



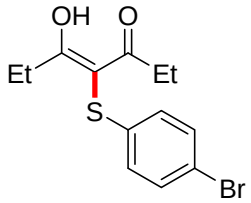
Yellow oil (55 mg, 58%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 16.87 (s, 1H), 7.08−7.01 (m, 3H), 2.39 (s, 6H), 2.24 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 139.9, 133.8, 129.0, 127.2, 105.3, 24.5, 21.6; HRMS (ESI-TOF) *m*/*z*: $[M + Na]^+$ Calcd for $C_{13}H_{16}O_2SNa$ 259.0763; Found 259.0766.

4-Hydroxy-3-(naphthalen-2-ylthio)pent-3-en-2-one (3as)



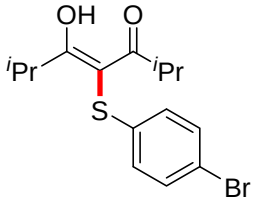
White solid (98 mg, 95%): mp 88−89 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.35 (s, 1H), 7.77 (dd, *J* = 8.4, 8.0 Hz, 2H), 7.71−7.69 (m, 1H), 7.50−7.39 (m, 3H), 7.26 (dd, *J* = 8.6, 1.9 Hz, 1H), 2.38 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 135.3, 133.9, 131.5, 128.9, 127.8, 126.8, 126,7, 125.4, 123.6, 121.8, 101.5, 24.4; HRMS (ESI-TOF) *m*/*z*: $[M + Na]^+$ Calcd for $C_{15}H_{14}O_2SNa$ 281.0607; Found 281.0605.

4-((4-Bromophenyl)thio)-5-hydroxyhept-4-en-3-one (3ba)[2]



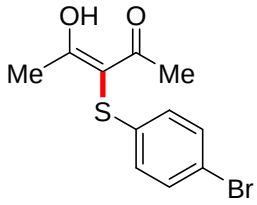
Yellow oil (107 mg, 85%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.47 (s, 1H), 7.37 (d, *J* = 8.6 Hz, 2H), 6.94 (d, *J* = 8.6 Hz, 2H), 2.69 (brs, 4H), 1.09 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.3, 137.6, 132.1, 126.0, 118.6, 99.6, 29.9, 9.5.

4-((4-Bromophenyl)thio)-5-hydroxy-2,6-dimethylhept-4-en-3-one (3ca)[2]



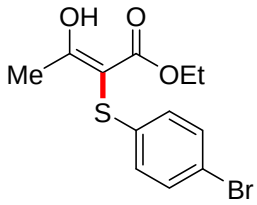
Yellow oil (114 mg, 83%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 18.05 (s, 1H), 7.38 (d, J = 8.6 Hz, 2H), 6.95 (d, J = 8.6 Hz, 2H), 3.46–3.36 (m, 2H), 1.08 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 138.4, 132.0, 125.9, 118.5, 97.7, 33.8, 19.5 (br).

2-((4-Bromophenyl)thio)-3-hydroxy-1-phenylbut-2-en-1-one (3da)



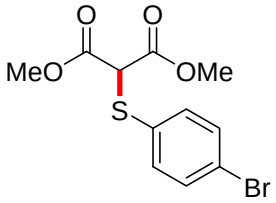
Yellow solid (95 mg, 68%): mp 87–88 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 17.75 (s, 1H), 7.62 (d, J = 7.4 Hz, 2H), 7.46–7.30 (m, 5H), 6.98 (d, J = 8.5 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.9, 191.0, 137.9, 135.4, 132.2, 131.3, 128.3, 127.8, 126.3, 118.8, 100.3, 25.6; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{13}BrO_2SNa$ 370.9712; Found 370.9715.

Ethyl 2-((4-bromophenyl)thio)-3-hydroxybut-2-enoate (3ea)



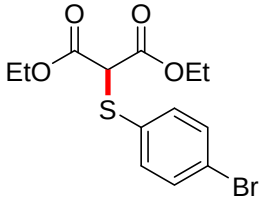
Colorless oil (104 mg, 82%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.85 (s, 1H), 7.35 (d, J = 8.6 Hz, 2H), 6.99 (d, J = 8.6 Hz, 2H), 4.21 (q, J = 7.1 Hz, 2H), 2.32 (s, 3H), 1.19 (q, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.8, 172.7, 137.4, 131.8, 127.1, 118.6, 91.8, 61.7, 20.9, 14.0; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{12}H_{13}BrO_3SNa$ 338.9661; Found 338.9667.

Dimethyl 2-((4-bromophenyl)thio)malonate (3fa)[3]



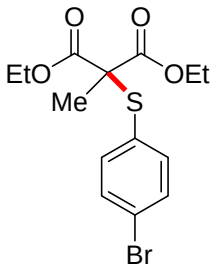
Colorless oil (102 mg, 80%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.5 Hz, 2H), 7.38 (d, J = 8.5 Hz, 2H), 4.49 (s, 1H), 3.73 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 134.9, 132.2, 130.9, 123.3, 54.9, 53.2.

Diethyl 2-((4-bromophenyl)thio)malonate (3ga)



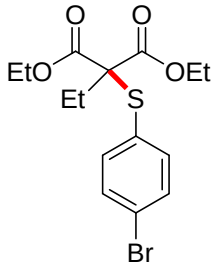
Colorless oil (110 mg, 79%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.7 Hz, 2H), 7.38 (d, J = 8.7 Hz, 2H), 4.47 (s, 1H), 4.18 (q, J = 7.1 Hz, 4H), 1.21 (t, J = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 134.7, 132.1, 131.2, 123.0, 62.4, 55.2, 13.8; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{13}H_{15}BrO_4SNa$ 368.9767; Found 368.9766.

Diethyl 2-((4-bromophenyl)thio)-2-methylmalonate (3ha)



Colorless oil (121 mg, 84%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 8.3 Hz, 2H), 7.39 (d, J = 8.5 Hz, 2H), 4.24–4.12 (m, 4H), 1.61 (s, 3H), 1.22 (t, J = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 138.5, 131.9, 129.1, 124.7, 62.2, 60.0, 22.2, 13.9; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{17}BrO_4SNa$ 382.9923; Found 382.9925.

Diethyl 2-((4-bromophenyl)thio)-2-ethylmalonate (3ia)

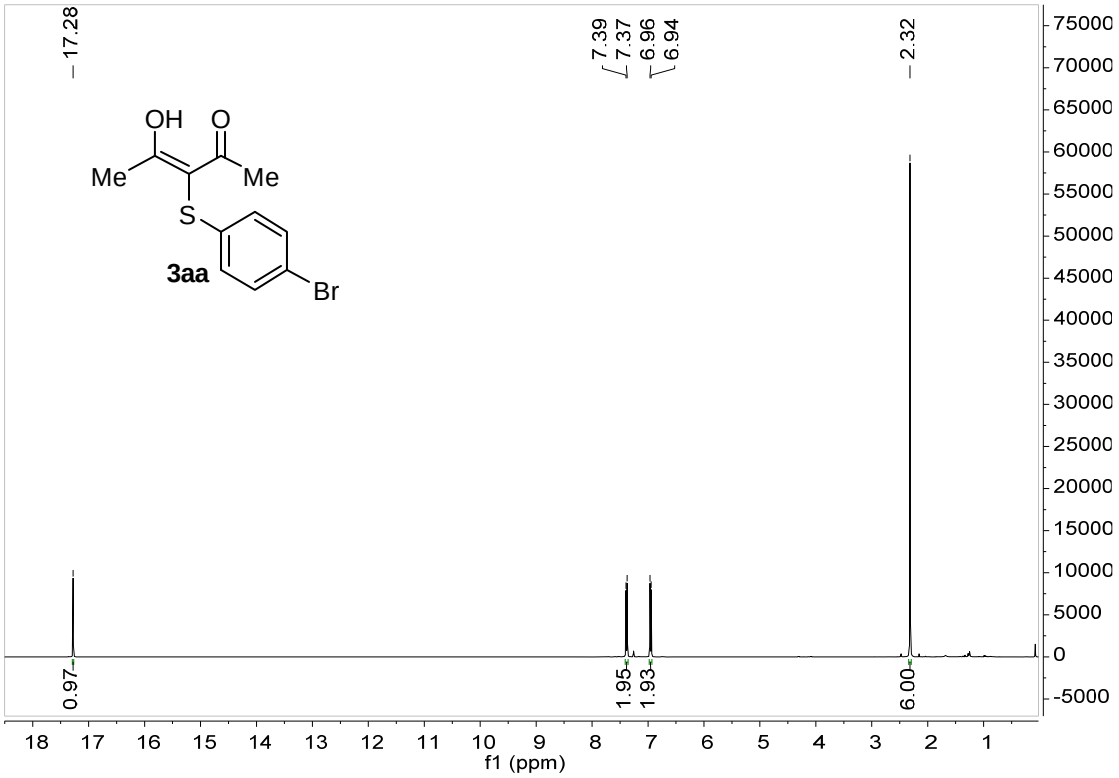


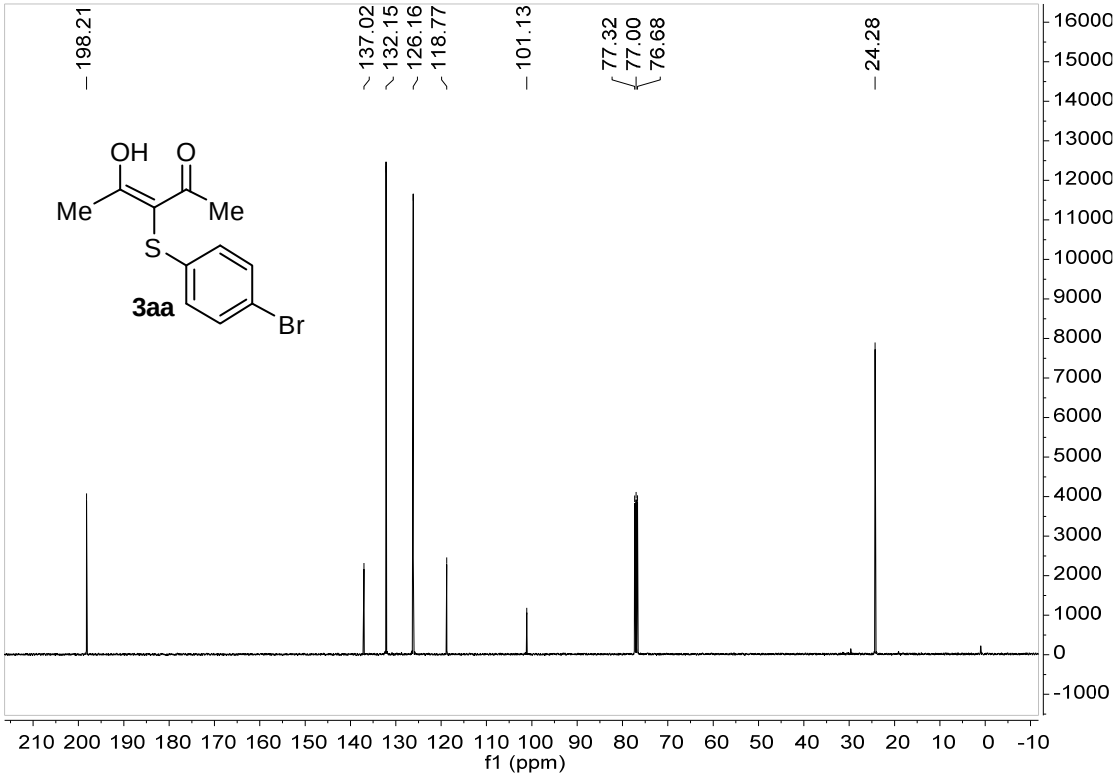
Colorless oil (134 mg, 89%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.5 Hz, 2H), 7.33 (d, J = 8.5 Hz, 2H), 4.25–4.11 (m, 4H), 1.97 (q, J = 7.4 Hz, 2H), 1.22 (t, J = 7.1 Hz, 6H), 1.00 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 138.3, 131.9, 129.0, 124.5, 66.0, 62.0, 26.8, 13.9, 8.5; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{19}BrO_4SNa$ 397.0080; Found 397.0084.

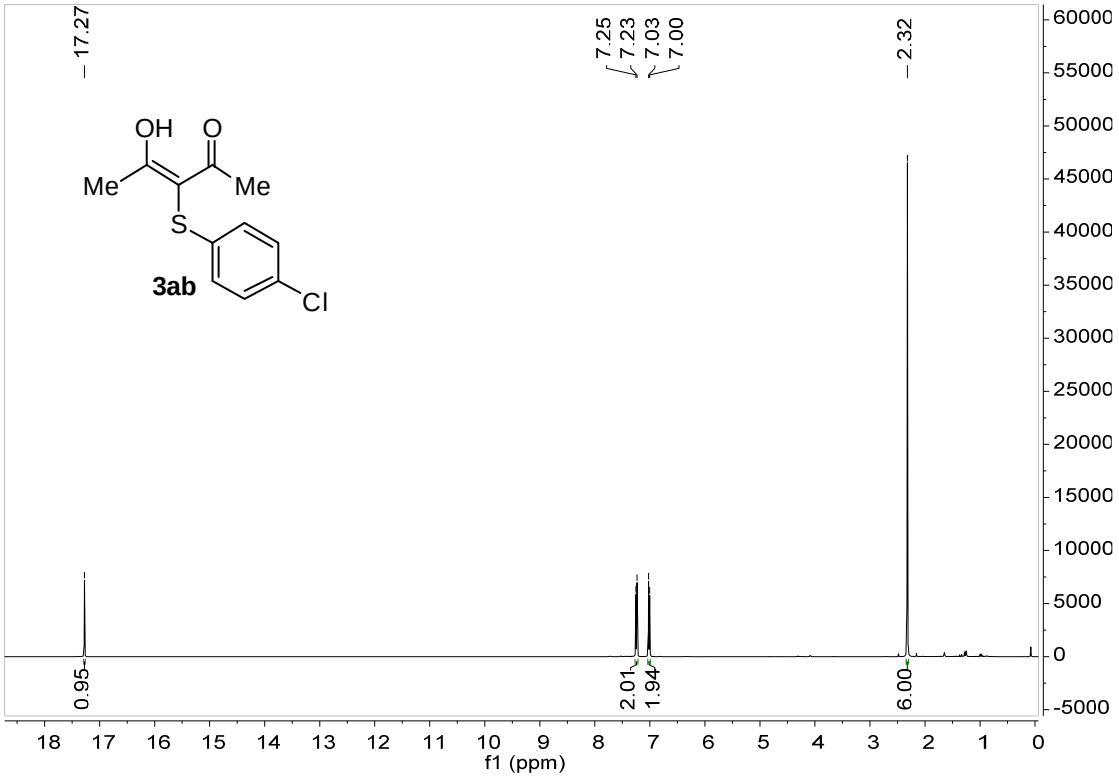
References:

1. H. Cao, J. Yuan, C. Liu, X. Hu and A. Lei, *RSC Adv.*, 2015, **5**, 41493.
2. Y.-W. Liu, S. S. Badsara, Y.-C. Liu and C.-F. Lee, *RSC Adv.*, 2015, **5**, 44299.
3. R. Rahaman, N. Devi and P. Barman, *Tetrahedron Lett.*, 2015, **56**, 4224.

5. Copies of NMR Spectra for 3



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7.25
7.23
7.03
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O
Me
Me
S
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Cl
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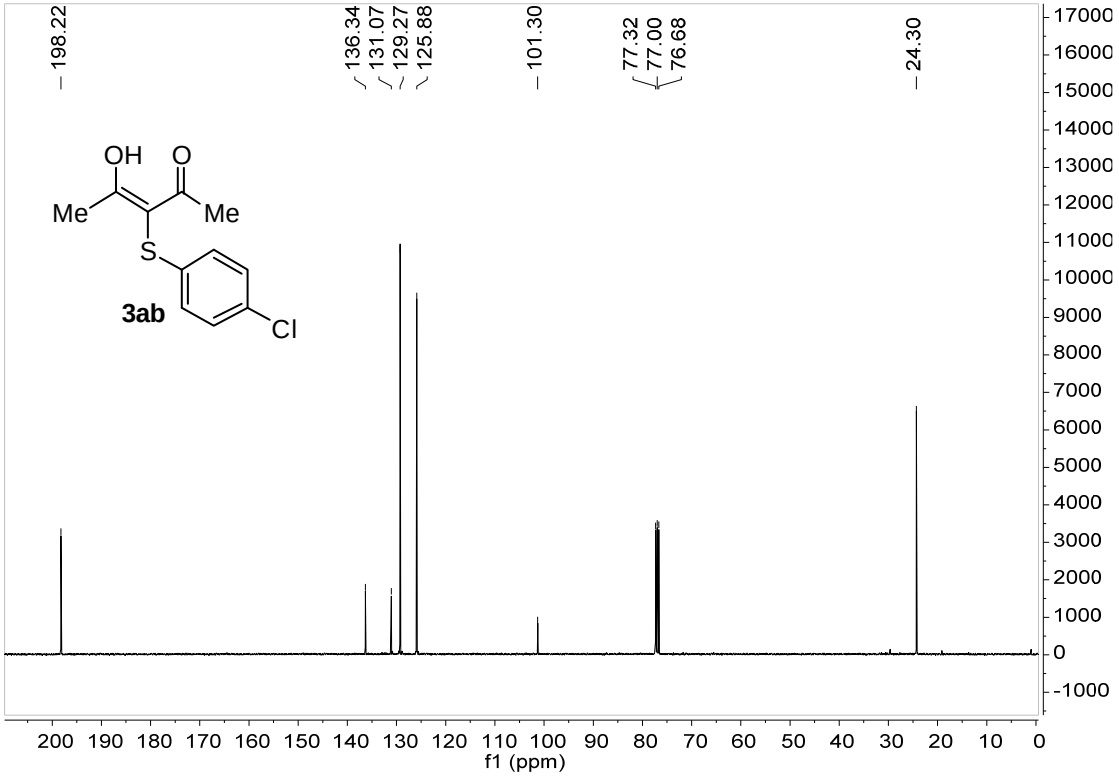
6.00
f1 (ppm)


198.22
136.34
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129.27
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77.00
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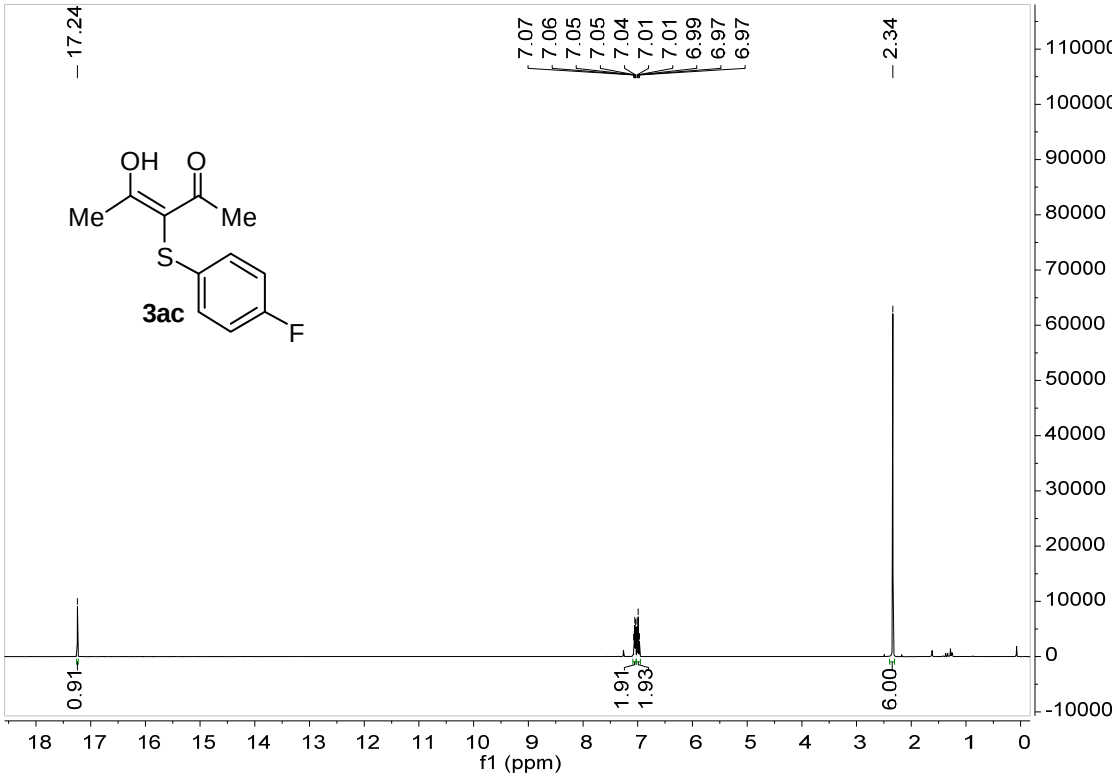
O
Me
Me
S
3ab
Cl
f1 (ppm)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7.07
7.06
7.05
7.05
7.04
7.01
7.01
6.99
6.97
6.97
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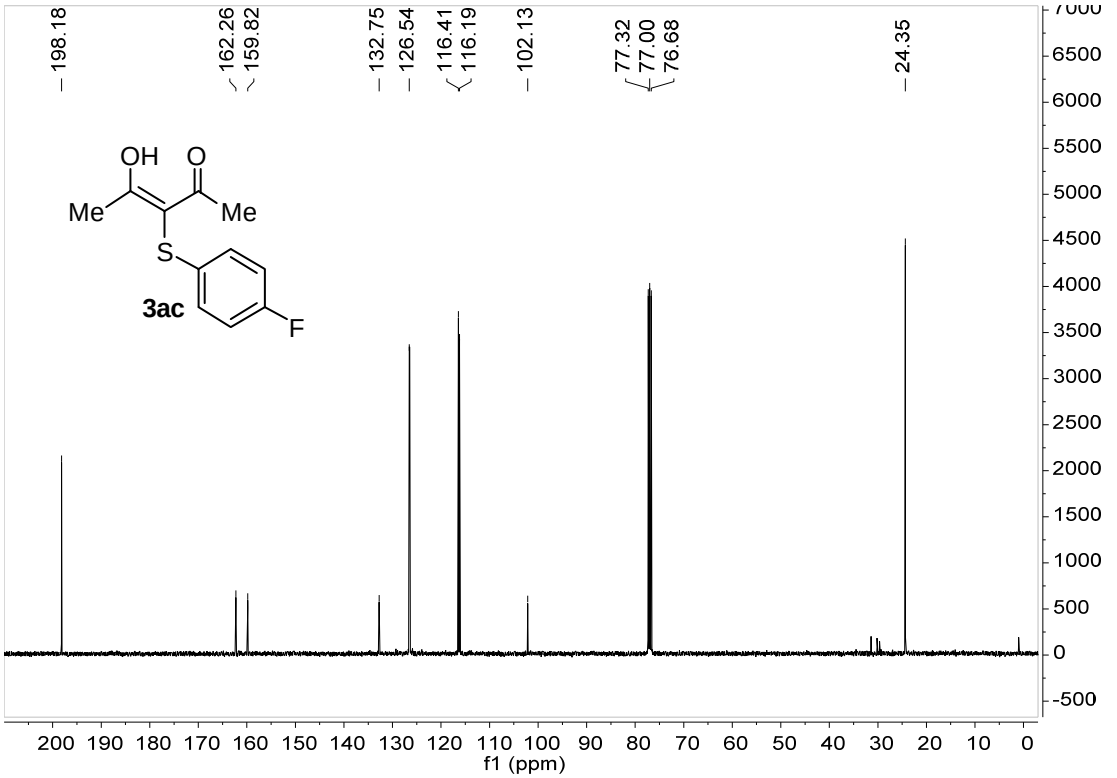
OH
O
Me
Me
S
3ac
F
0.91
1.91
1.93
6.00
f1 (ppm)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O
Me
Me
S
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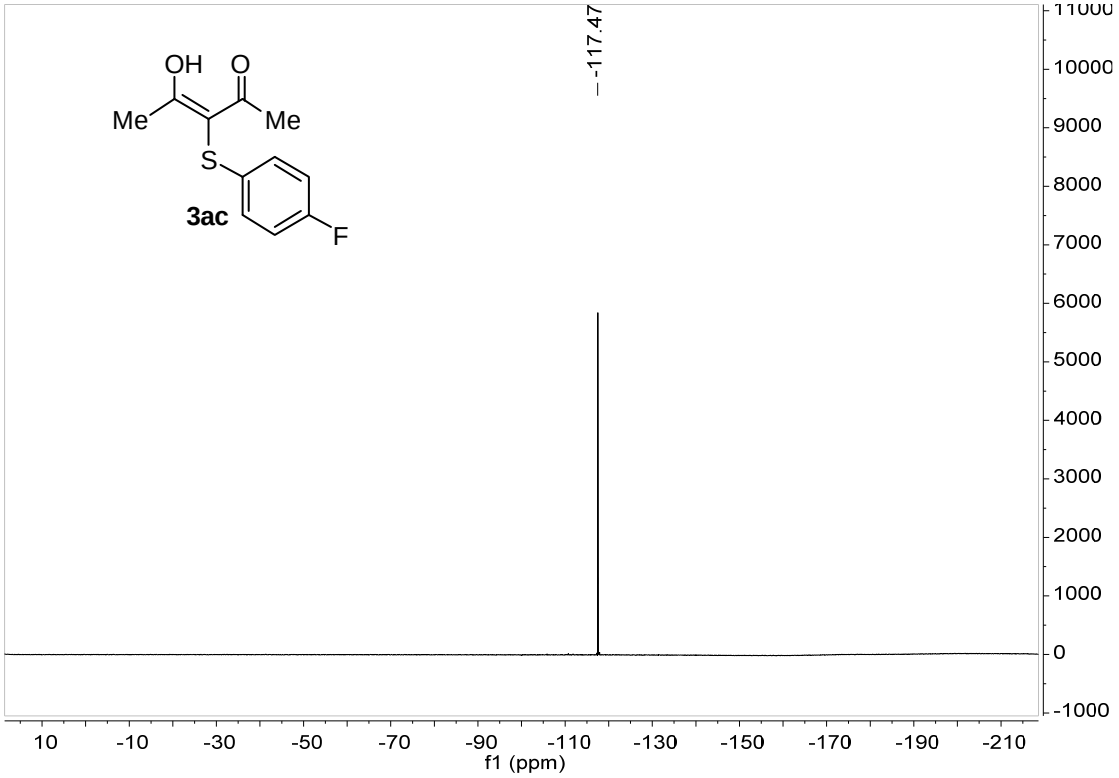
F
f1 (ppm)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Me
S
F
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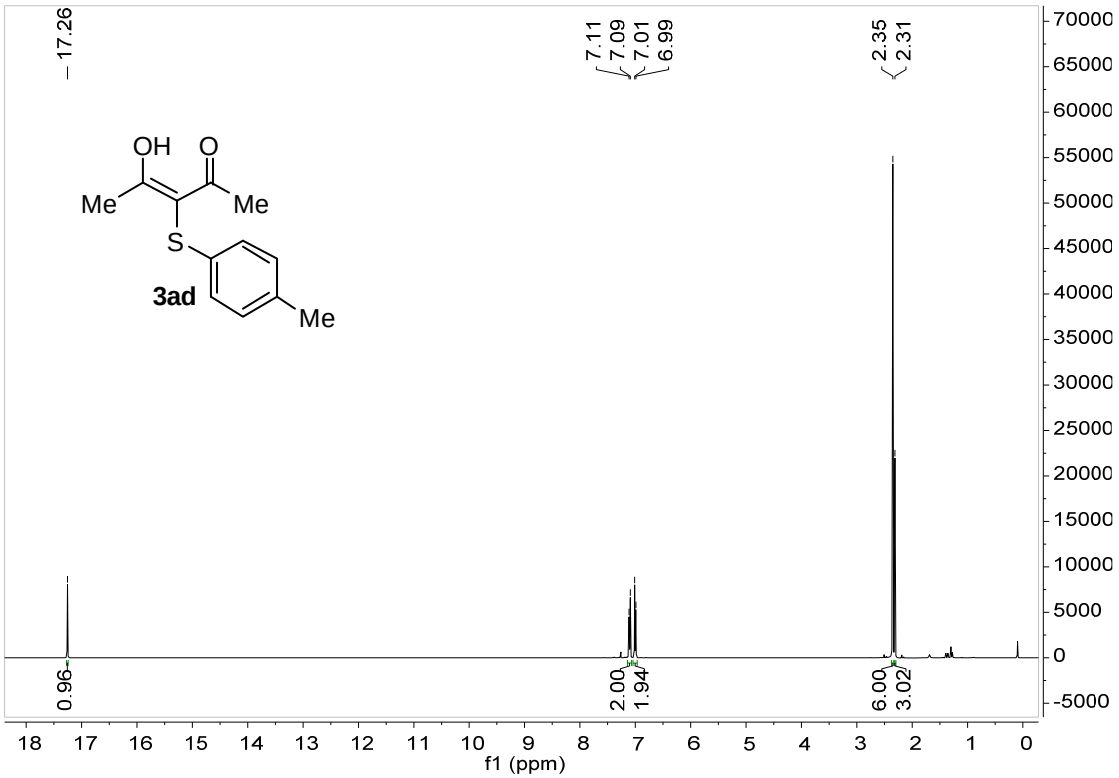
f1 (ppm)


17.26
7.11
7.09
7.01
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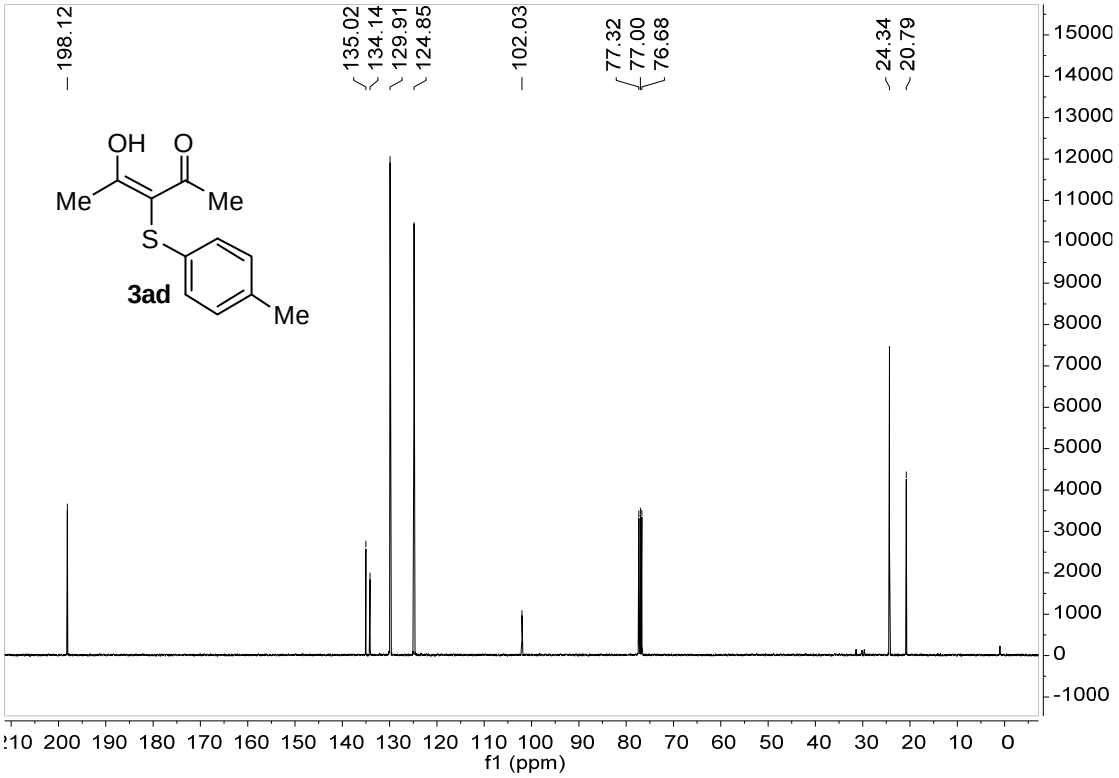
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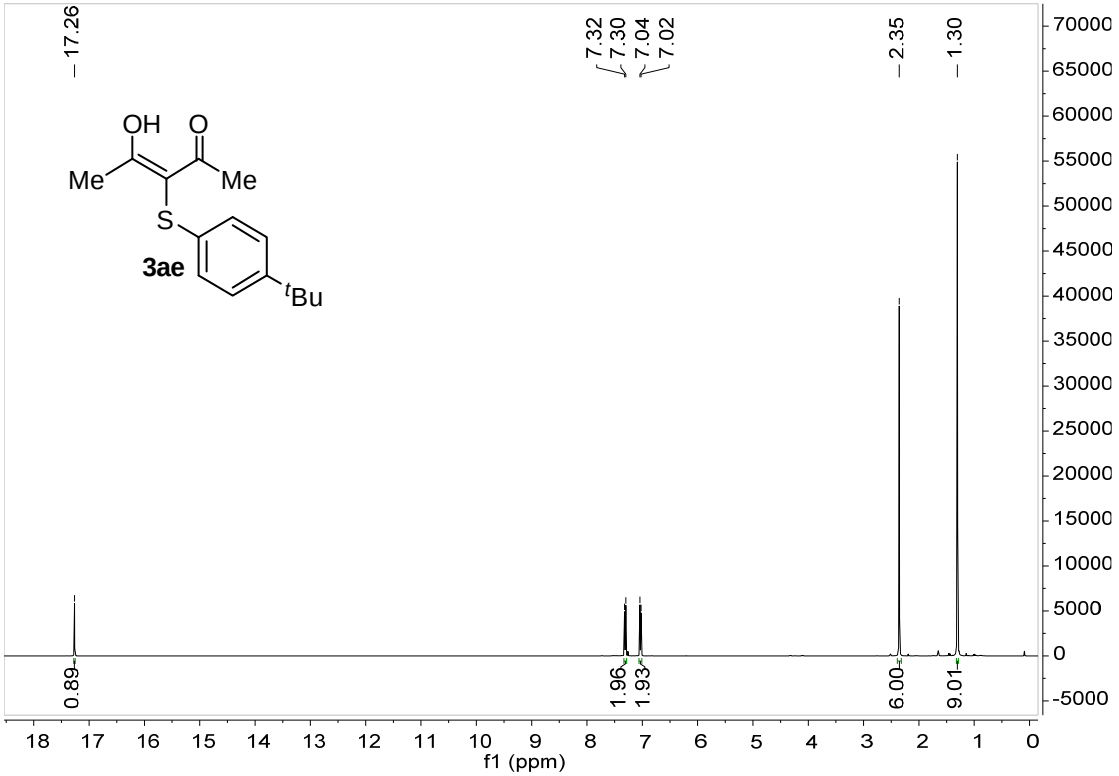
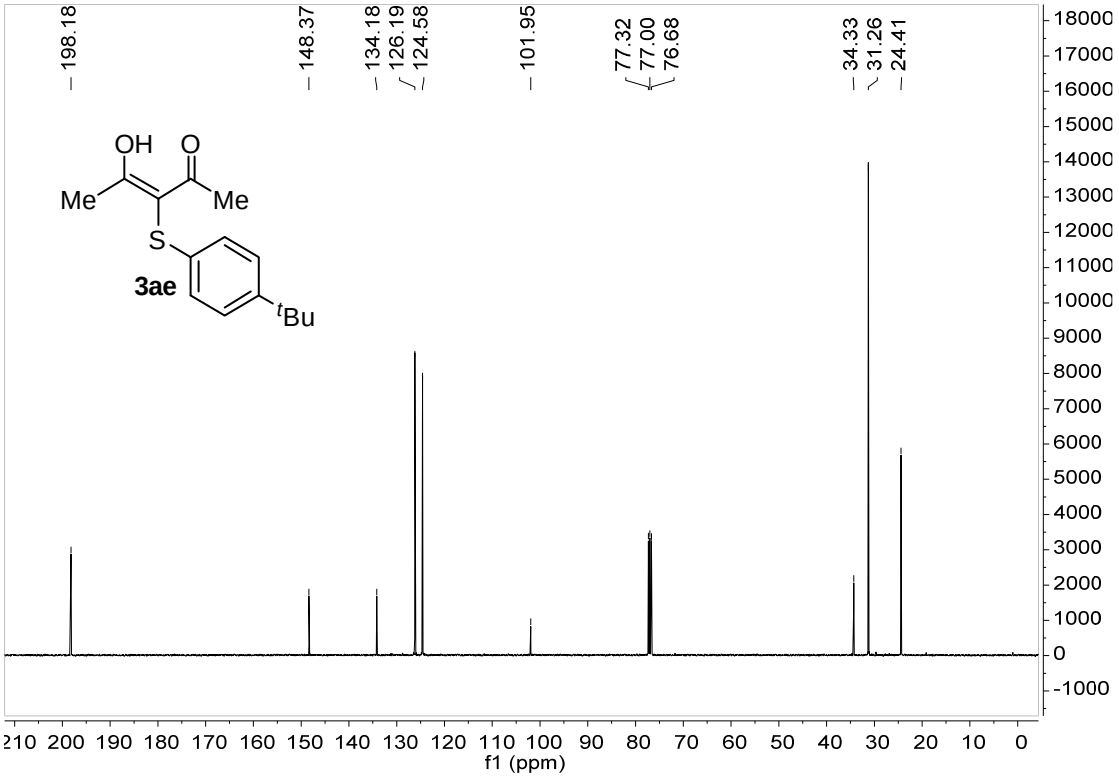
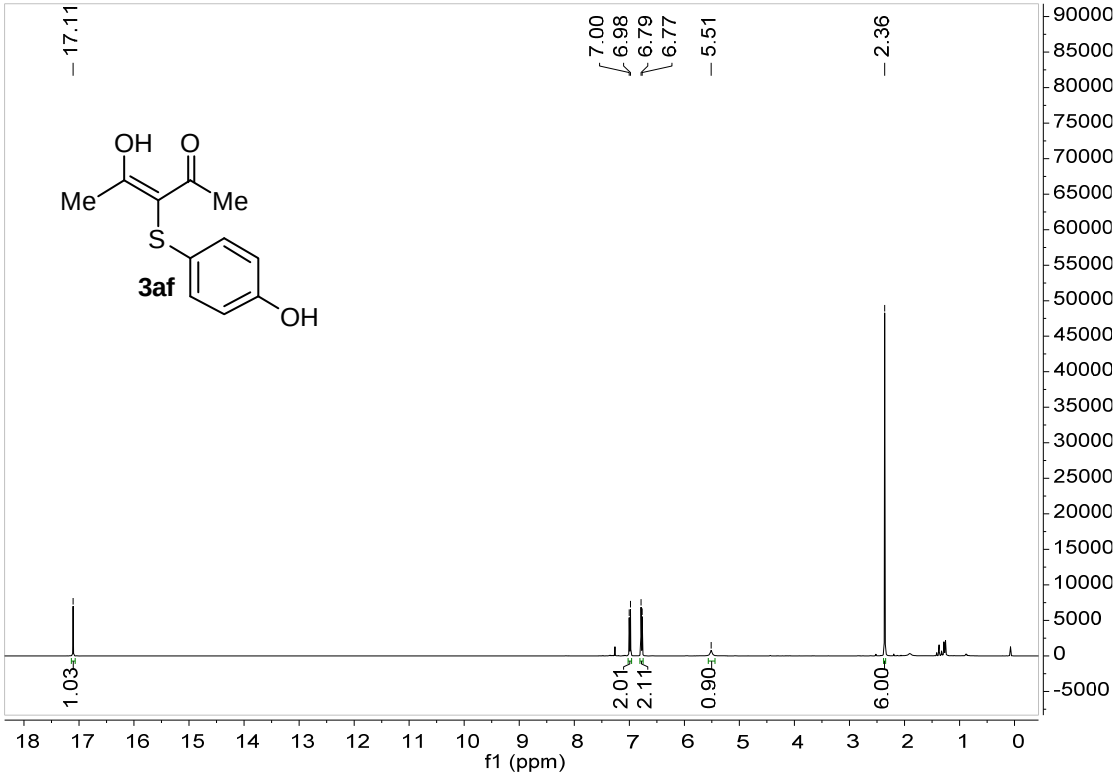
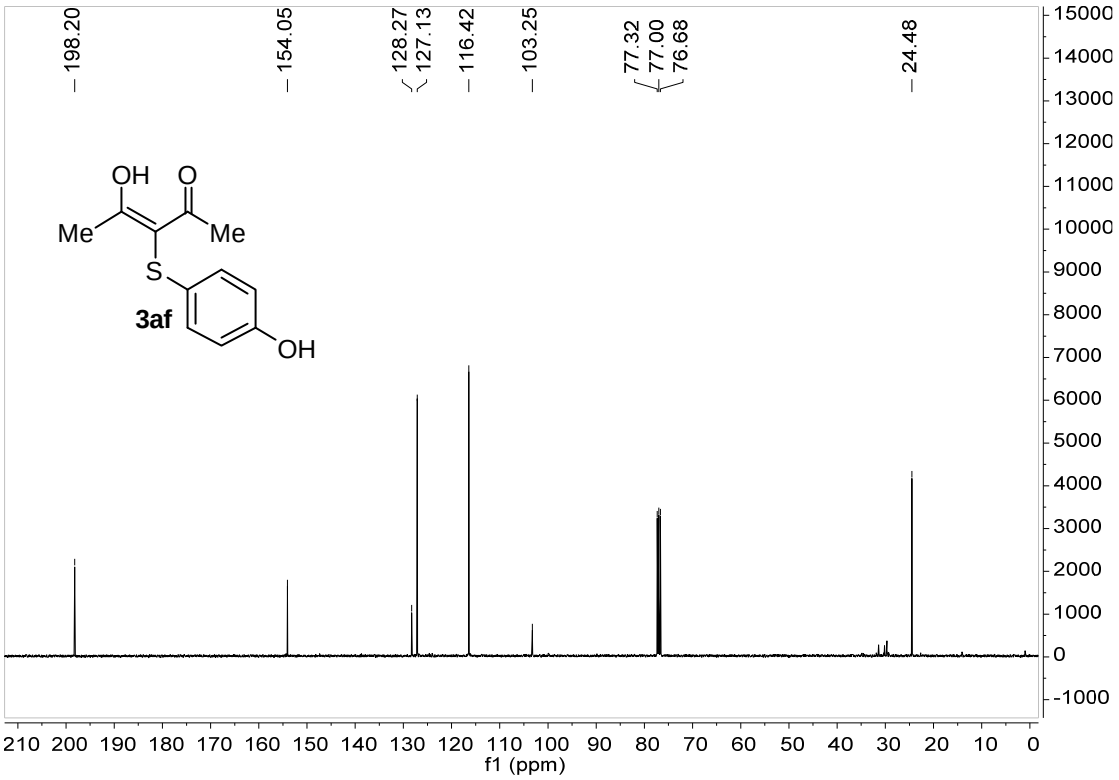
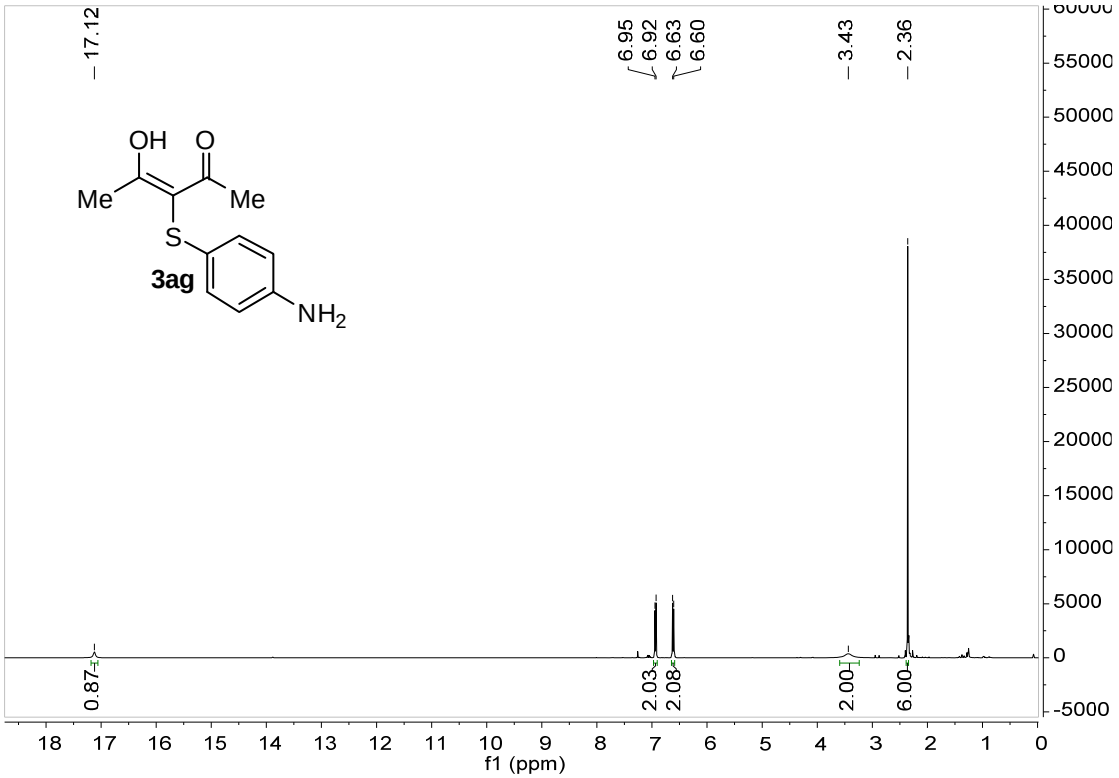
S
3ad
Me
f1 (ppm)

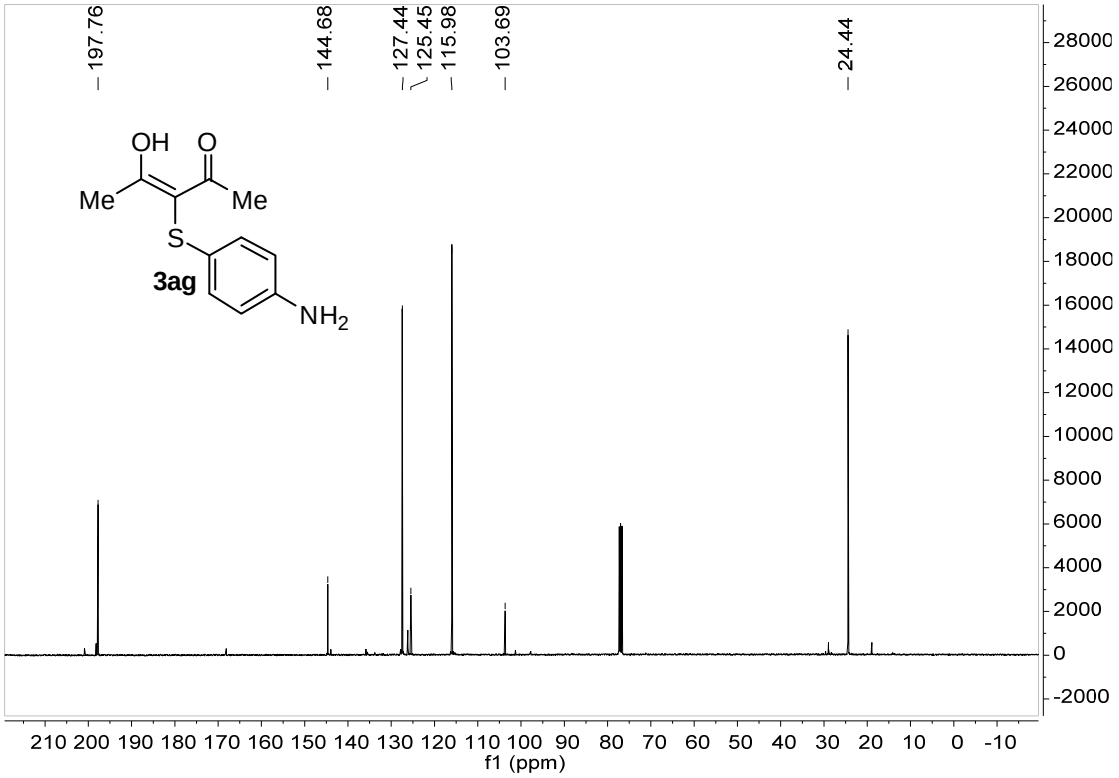

17.26
7.32
7.30
7.04
7.02
2.35
1.30
OH
O
Me
Me
S
3ae
tBu
0.89
1.96
1.93
6.00
9.01
f1 (ppm)


198.18
148.37
134.18
126.19
124.58
101.95
77.32
77.00
76.68
34.33
31.26
24.41
OH
O
Me
Me
S
3ae
tBu
f1 (ppm)


17.11
7.00
6.98
6.79
6.77
5.51
2.36
OH
O
Me
Me
S
3af
OH
1.03
2.01
2.11
0.90
6.00
f1 (ppm)


198.20
154.05
128.27
127.13
116.42
103.25
77.32
77.00
76.68
24.48
OH
O
Me
Me
S
3af
OH
f1 (ppm)


17.12
6.95
6.92
6.63
6.60
3.43
2.36
OH
O
Me
Me
S
3ag
NH2
0.87
2.03
2.08
2.00
6.00
f1 (ppm)


197.76
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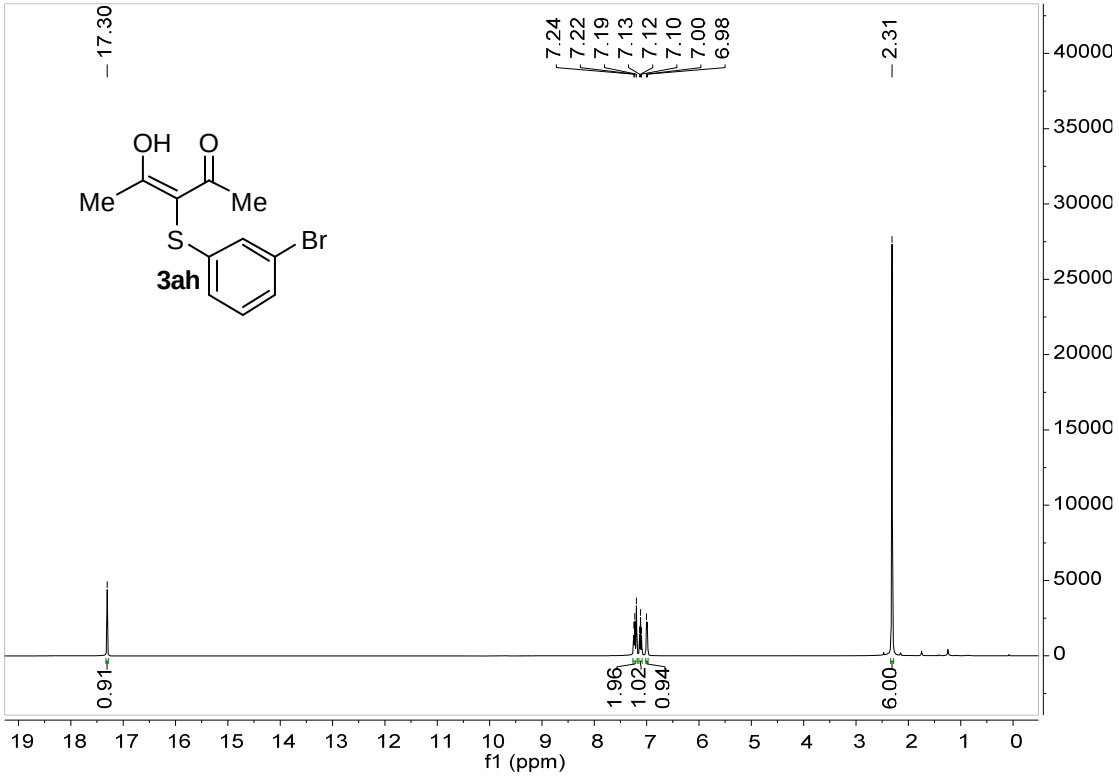
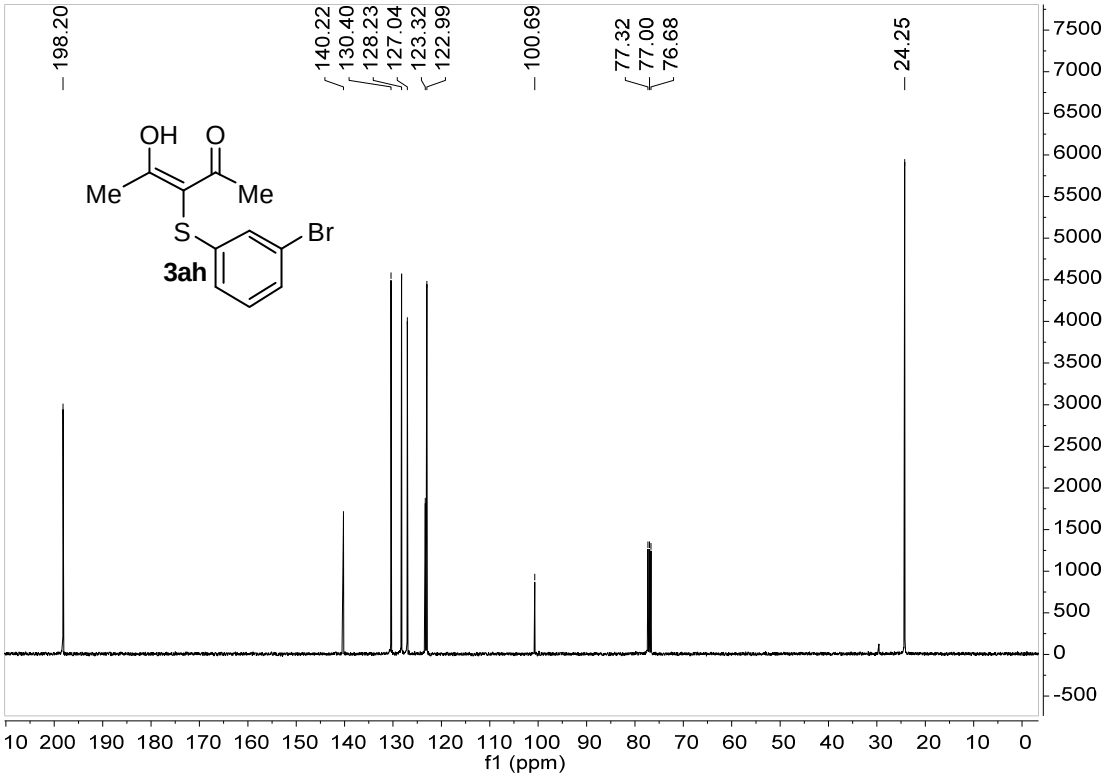
125.45
115.98
103.69
24.44
OH
O
Me
Me
S
3ag
NH2
f1 (ppm)


17.30
7.24
7.22
7.19
7.13
7.12
7.10
7.00
6.98
2.31
OH
O
Me
Me
S
Br
3ah
0.91
1.96
1.02
0.94
6.00
f1 (ppm)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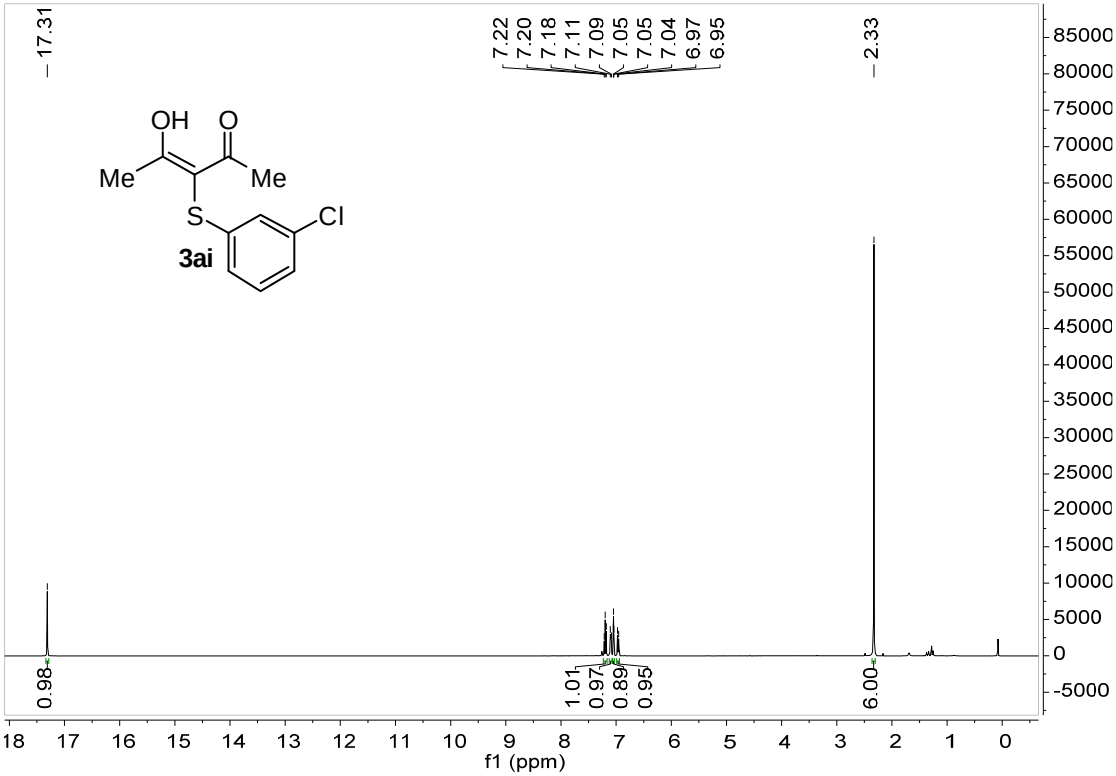
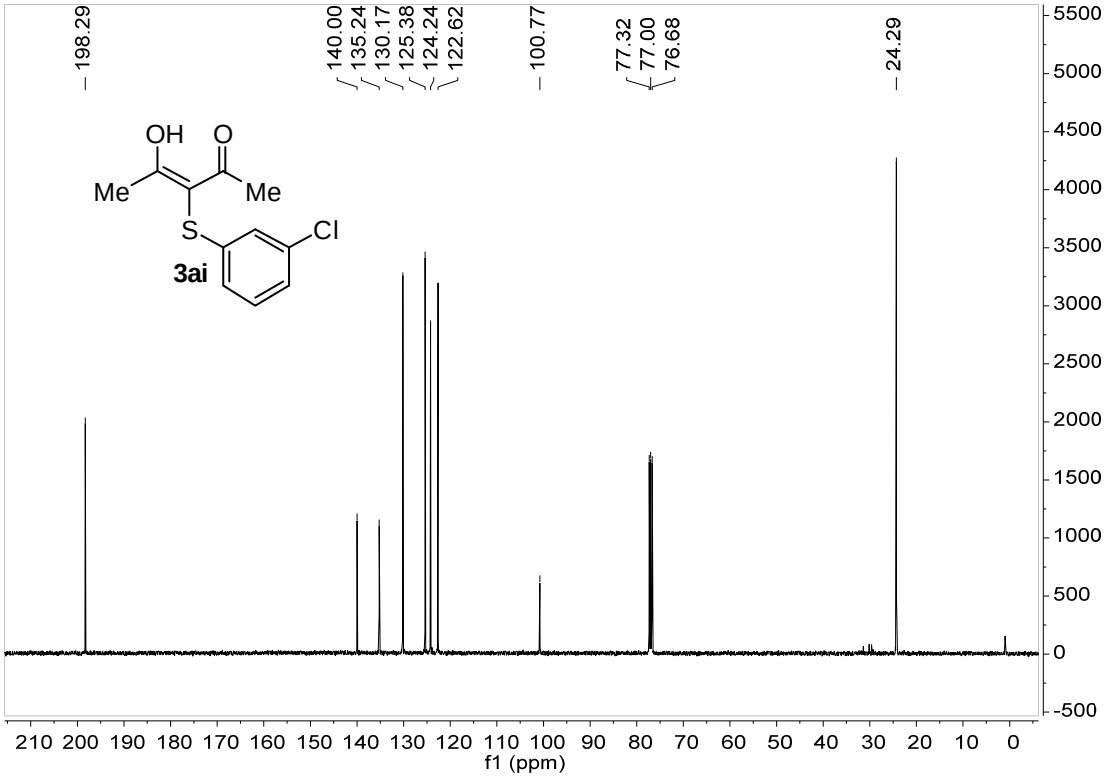
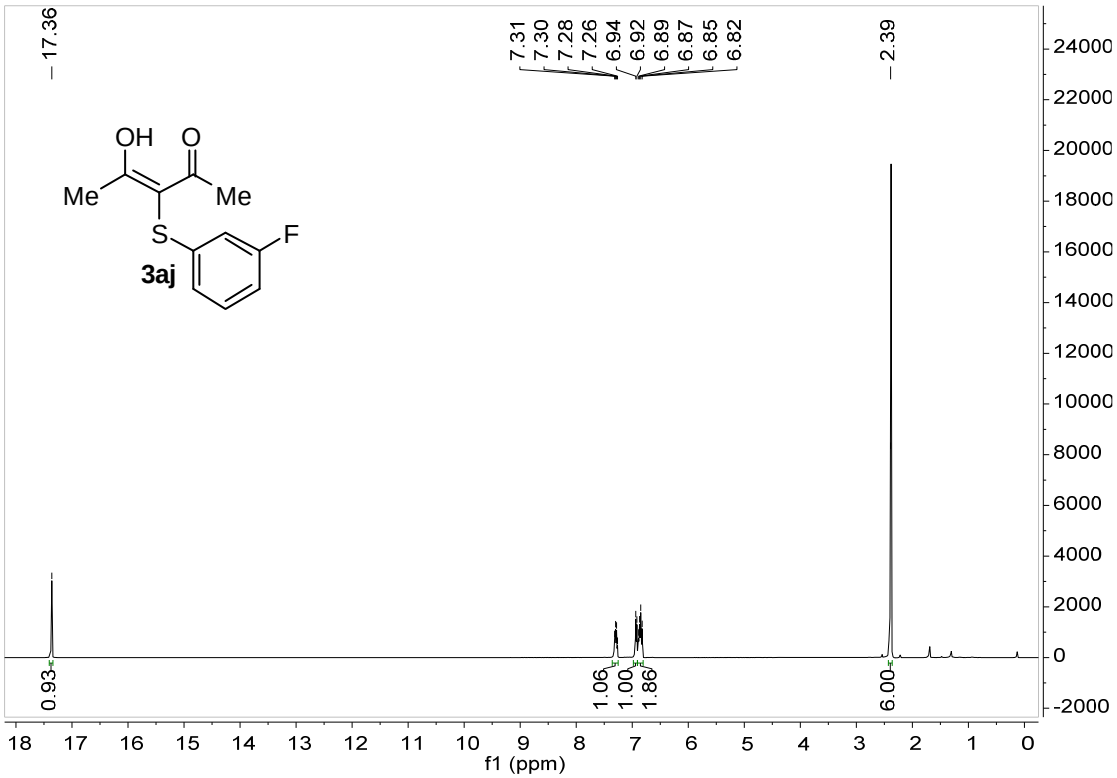
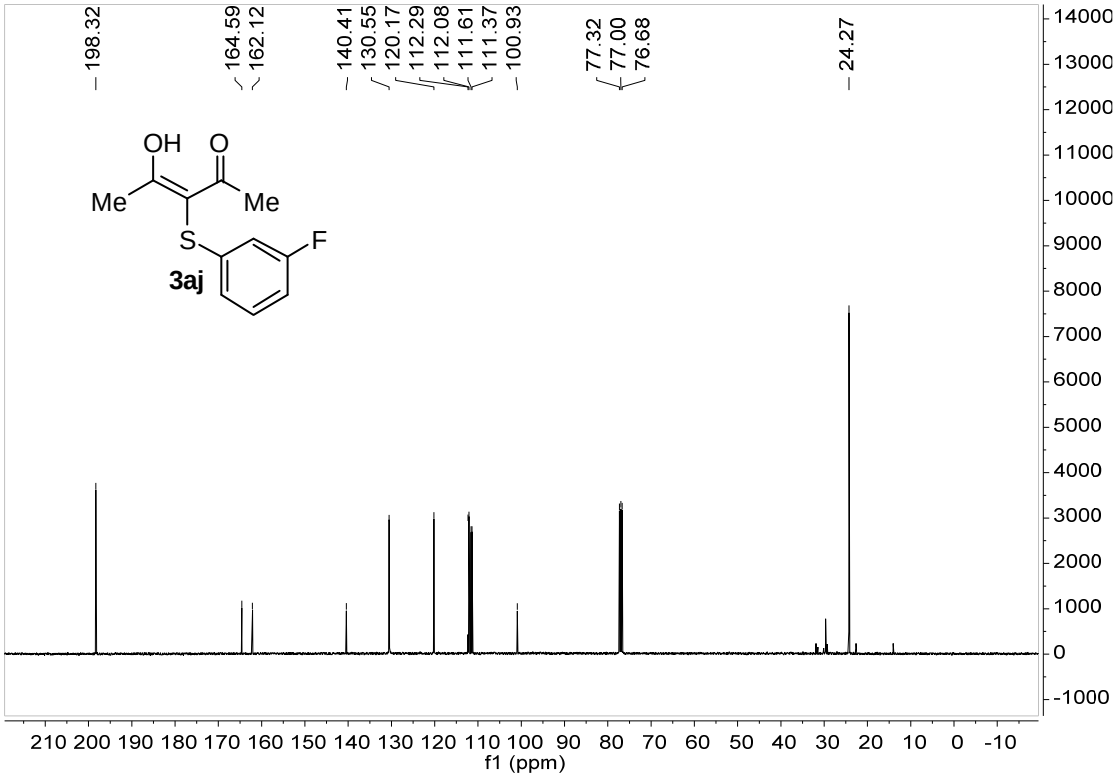
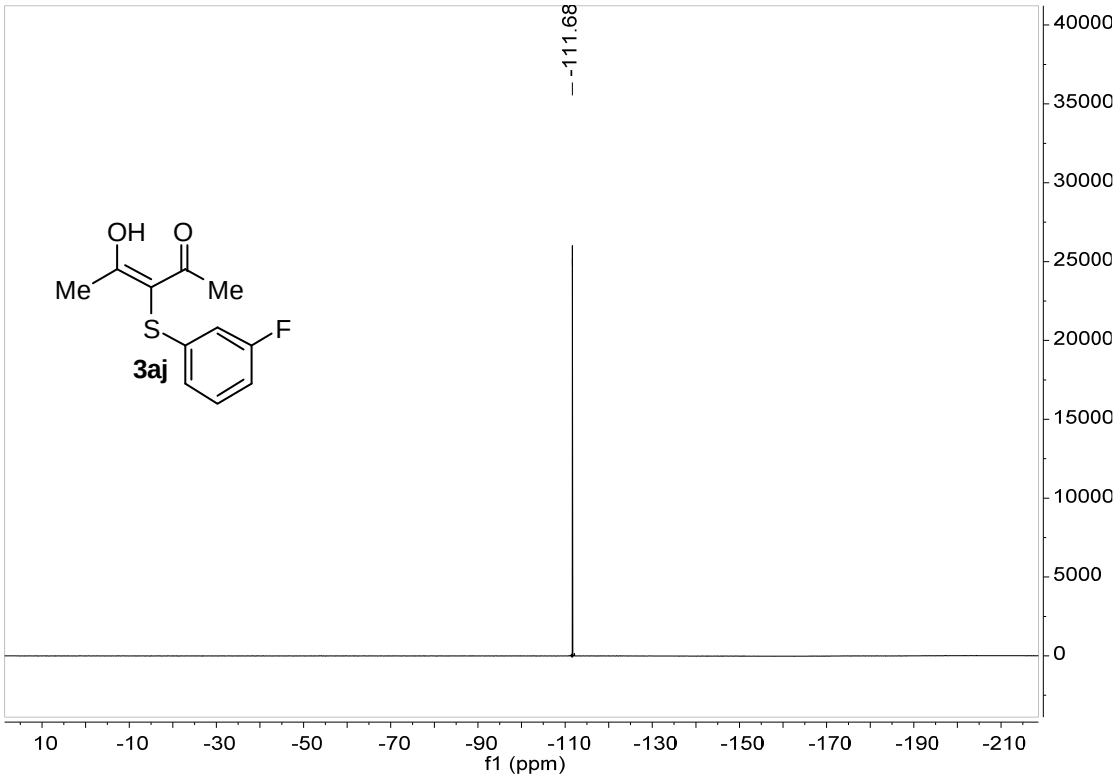
140.22
130.40
128.23
127.04
123.32
122.99
100.69
77.32
77.00
76.68
24.25
OH
O
Me
Me
S
Br
3ah
f1 (ppm)


17.31
7.22
7.20
7.18
7.11
7.09
7.05
7.05
7.04
6.97
6.95
2.33
OH
O
Me
Me
S
Cl
3ai
0.98
1.01
0.97
0.89
0.95
6.00
f1 (ppm)


198.29
140.00
135.24
130.17
125.38
124.24
122.62
100.77
77.32
77.00
76.68
24.29
OH
O
Me
Me
S
Cl
3ai
f1 (ppm)


17.36
7.31
7.30
7.28
7.26
6.94
6.92
6.89
6.87
6.85
6.82
2.39
OH
O
Me
Me
S
F
3aj
0.93
1.06
1.00
1.86
6.00
f1 (ppm)


198.32
164.59
162.12
140.41
130.55
120.17
112.29
112.08
111.61
111.37
100.93
77.32
77.00
76.68
24.27
OH
O
Me
Me
S
F
3aj
f1 (ppm)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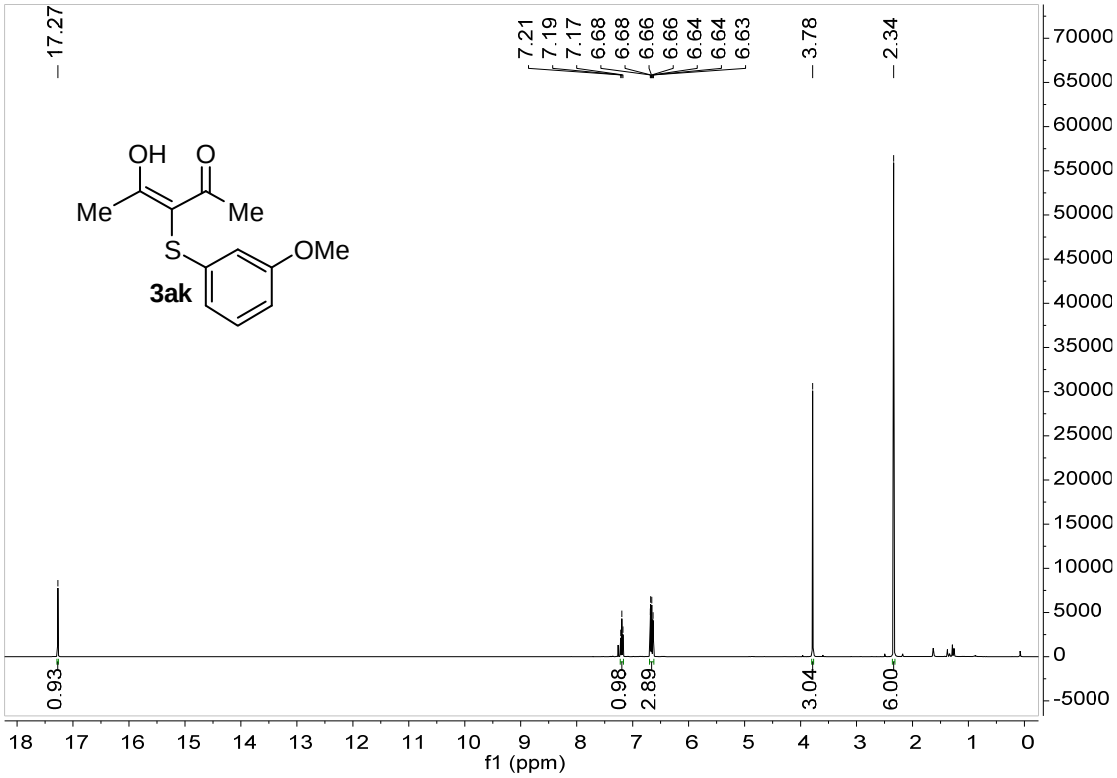
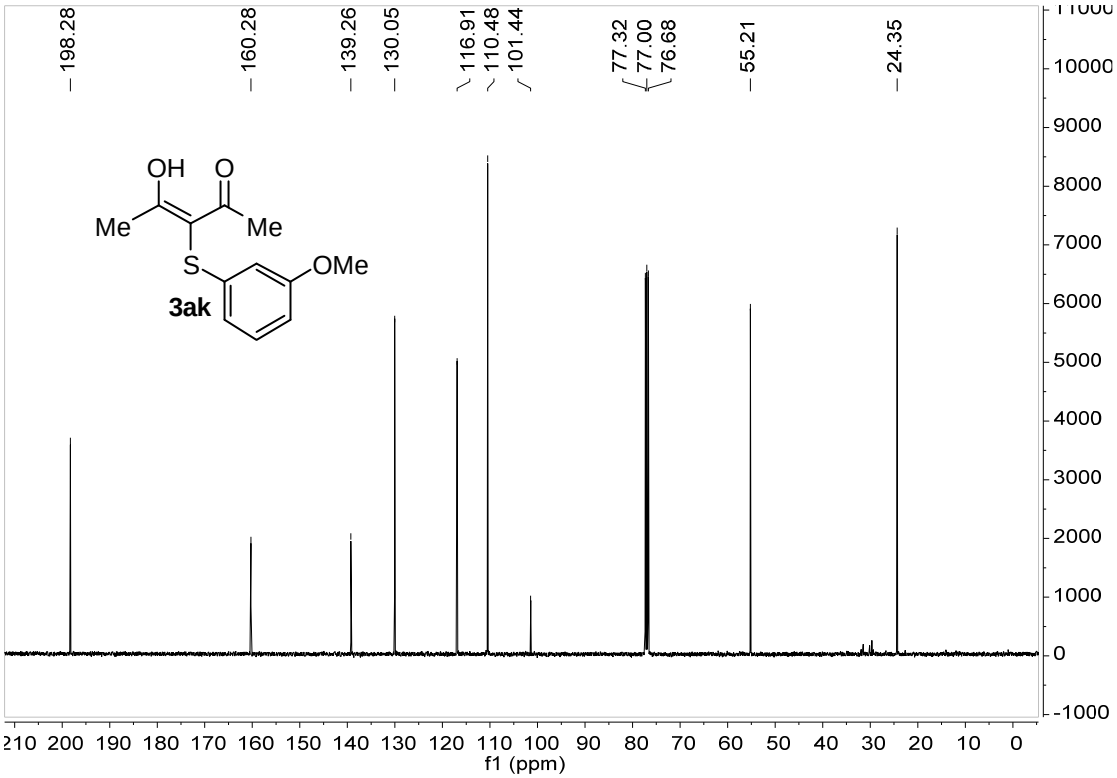
OH
O
Me
Me
S
F
3aj
f1 (ppm)


17.27
7.21
7.19
7.17
6.68
6.68
6.66
6.66
6.64
6.64
6.63
3.78
2.34
OH
O
Me
Me
S
OMe
3ak
0.93
0.98
2.89
3.04
6.00
f1 (ppm)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160.28
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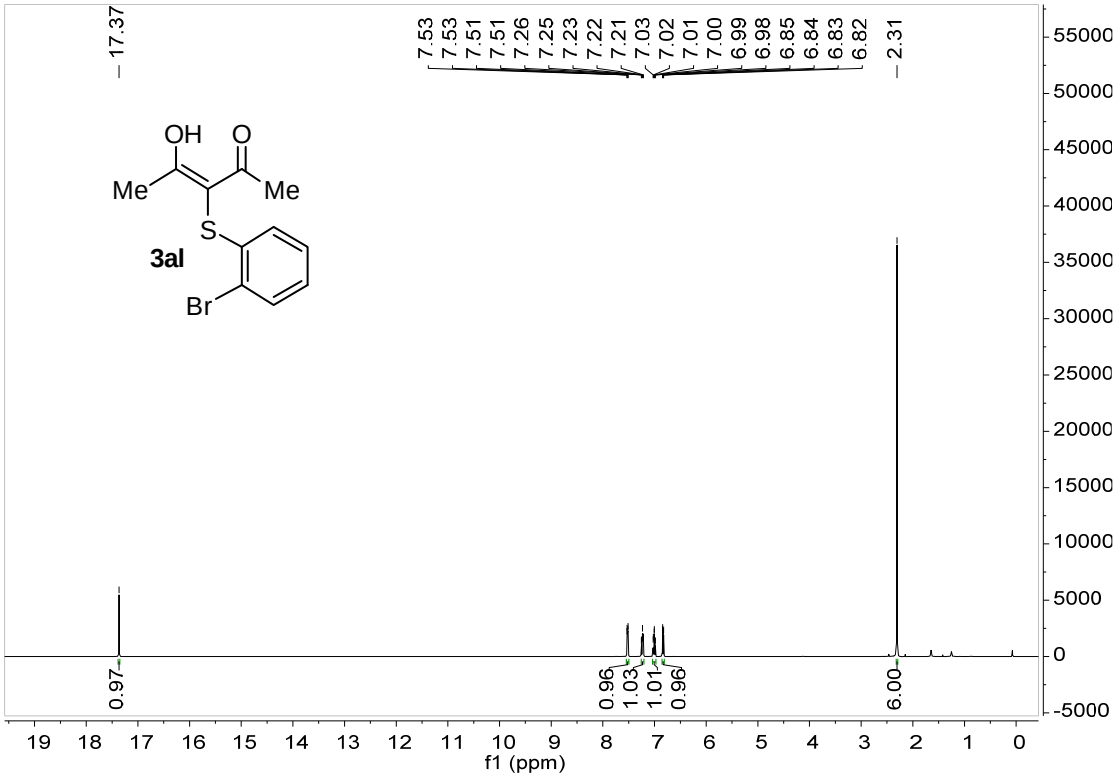
116.91
110.48
101.44
77.32
77.00
76.68
55.21
24.35
OH
O
Me
Me
S
OMe
3ak
f1 (ppm)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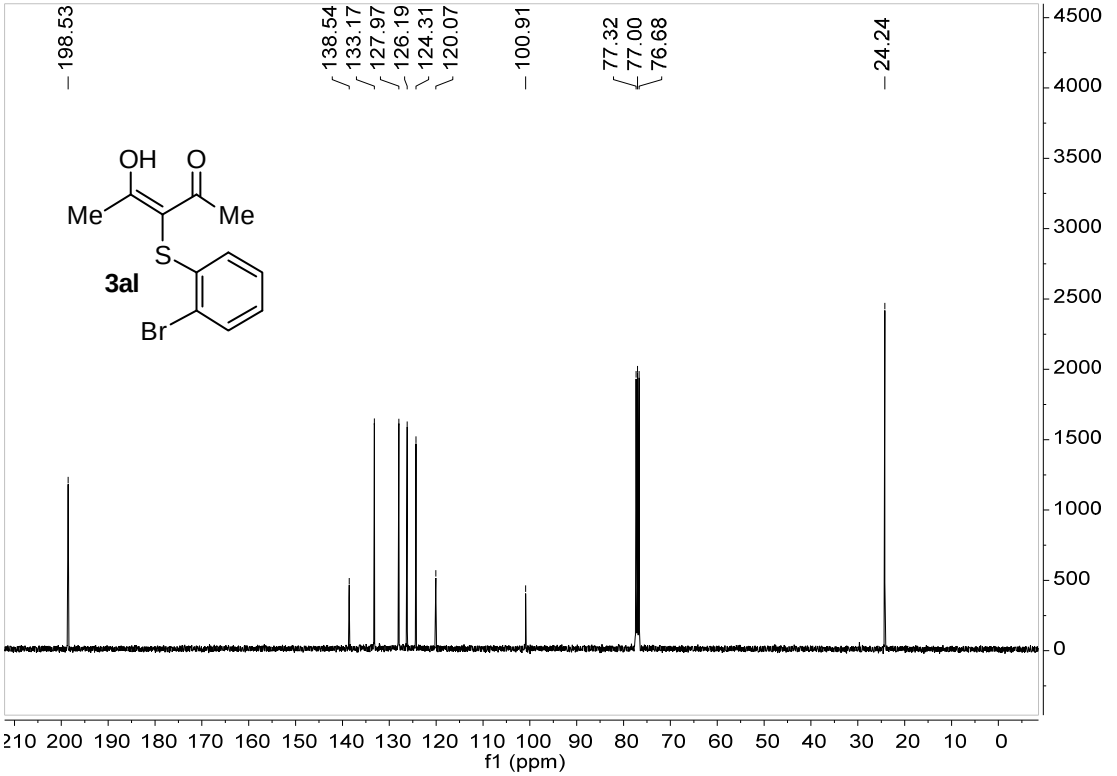
7.53
7.53
7.51
7.51
7.26
7.25
7.23
7.22
7.21
7.03
7.02
7.01
7.00
6.99
6.98
6.85
6.84
6.83
6.82
2.31
OH
O
Me
Me
S
3al
Br
0.97
0.96
1.03
1.01
0.96
6.00
f1 (ppm)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124.31
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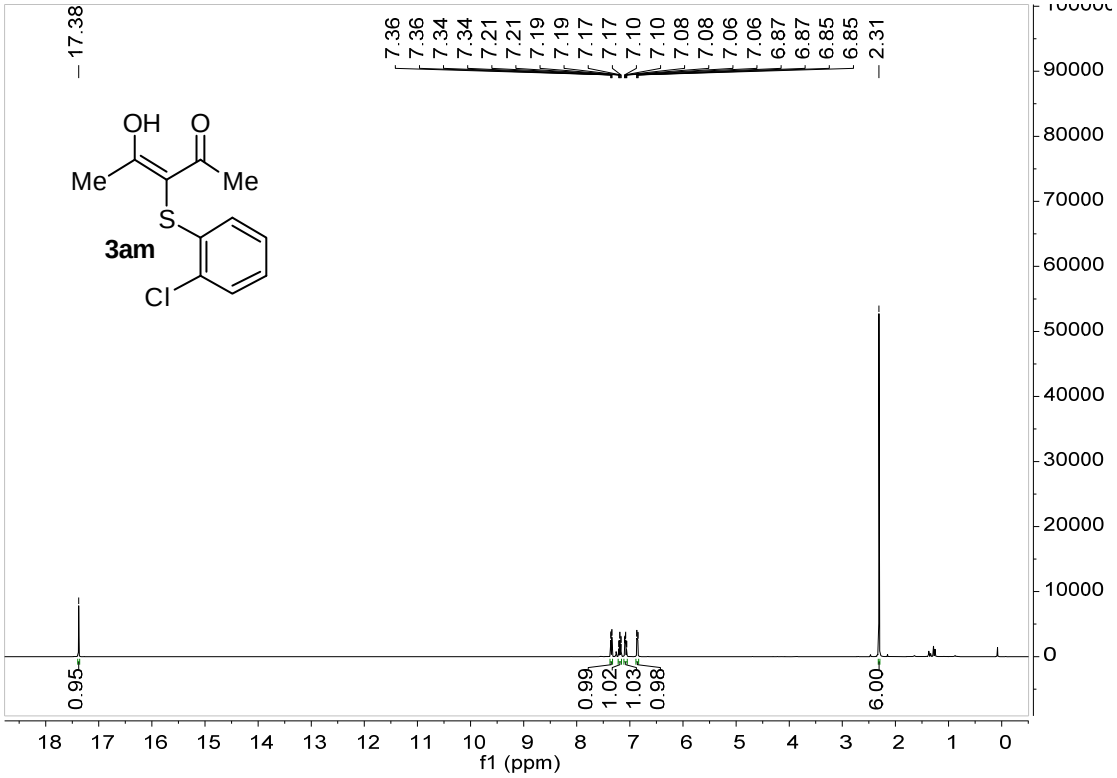
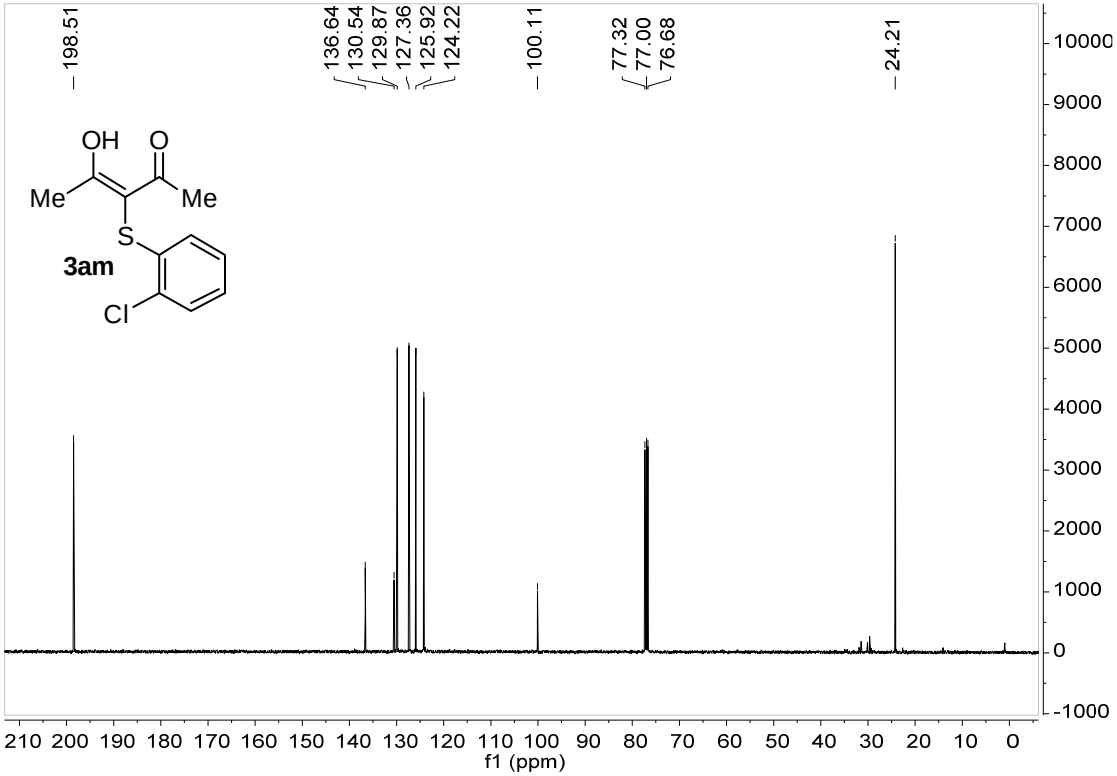
77.32
77.00
76.68
24.24
OH
O
Me
Me
S
3al
Br
f1 (ppm)


17.38
7.36
7.36
7.34
7.34
7.21
7.21
7.19
7.19
7.17
7.17
7.10
7.10
7.08
7.08
7.06
7.06
6.87
6.87
6.85
6.85
2.31
OH
O
Me
Me
S
3am
Cl
0.95
0.99
1.02
1.03
0.98
6.00
f1 (ppm)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129.87
127.36
125.92
124.22
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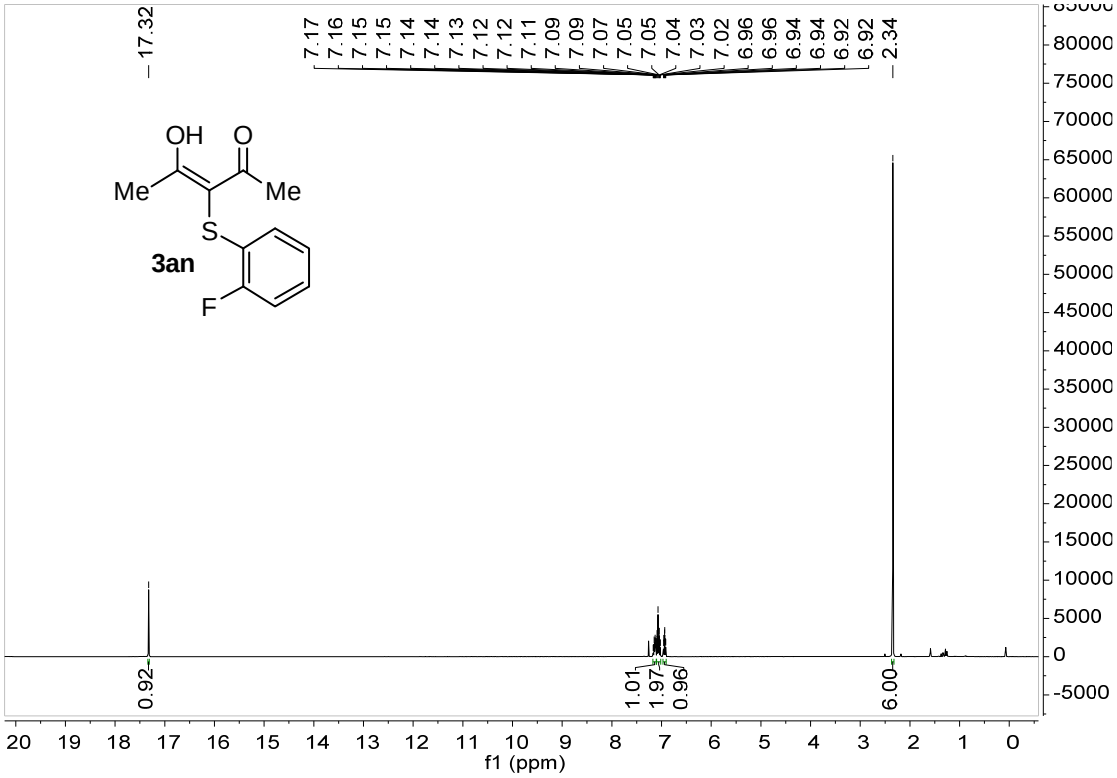
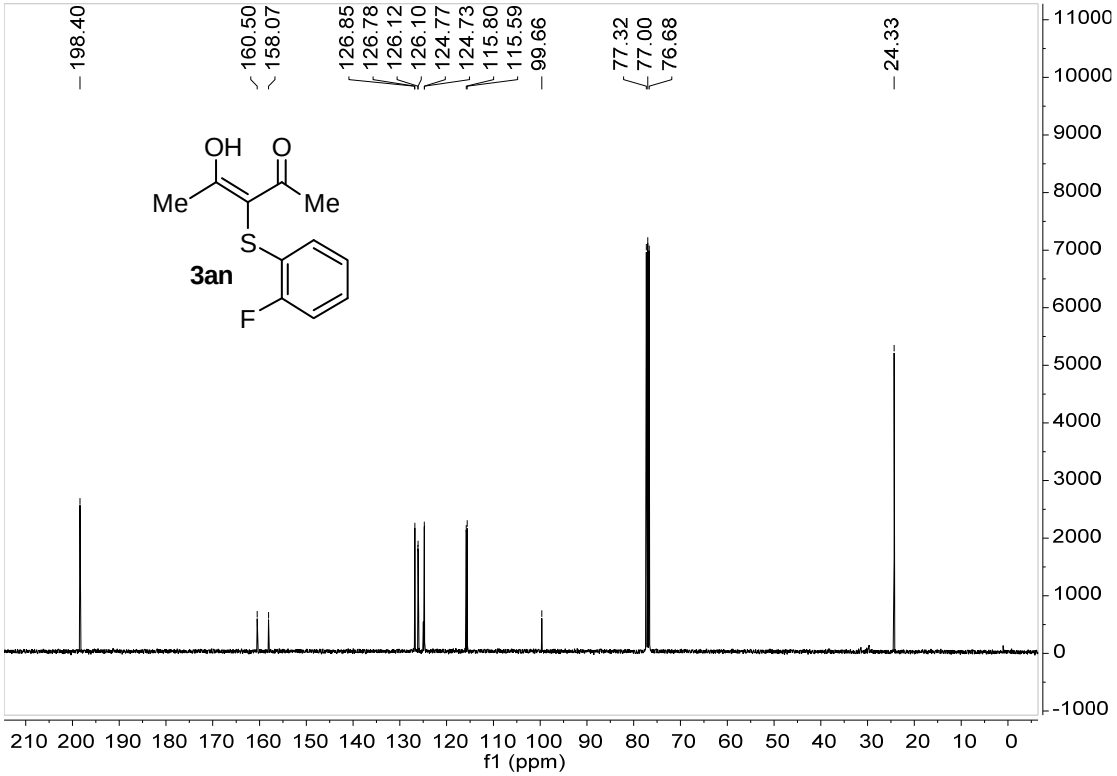
77.32
77.00
76.68
24.21
OH
O
Me
Me
S
3am
Cl
f1 (ppm)


17.32
7.17
7.16
7.15
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.09
7.09
7.07
7.05
7.05
7.04
7.03
7.02
6.96
6.96
6.94
6.94
6.92
6.92
2.34
OH
O
Me
Me
S
3an
F
0.92
1.01
1.97
0.96
6.00
f1 (ppm)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158.07
126.85
126.78
126.12
126.10
124.77
124.73
115.80
115.59
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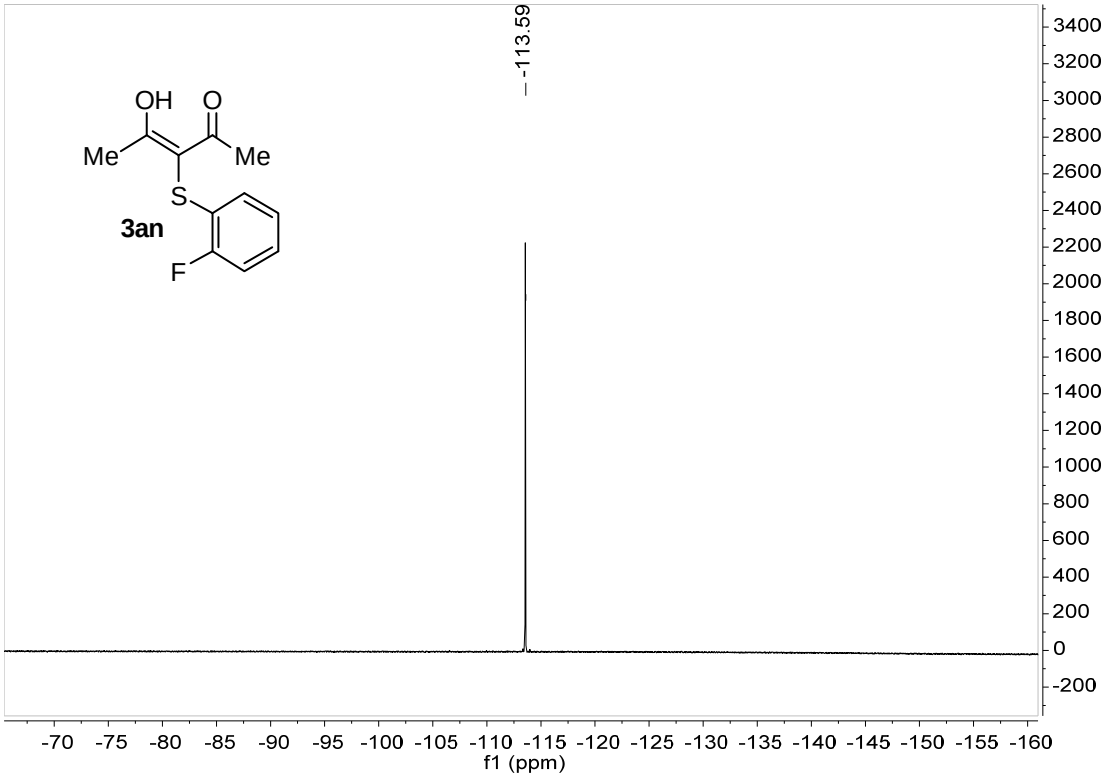
77.32
77.00
76.68
24.33
OH
O
Me
Me
S
3an
F
f1 (ppm)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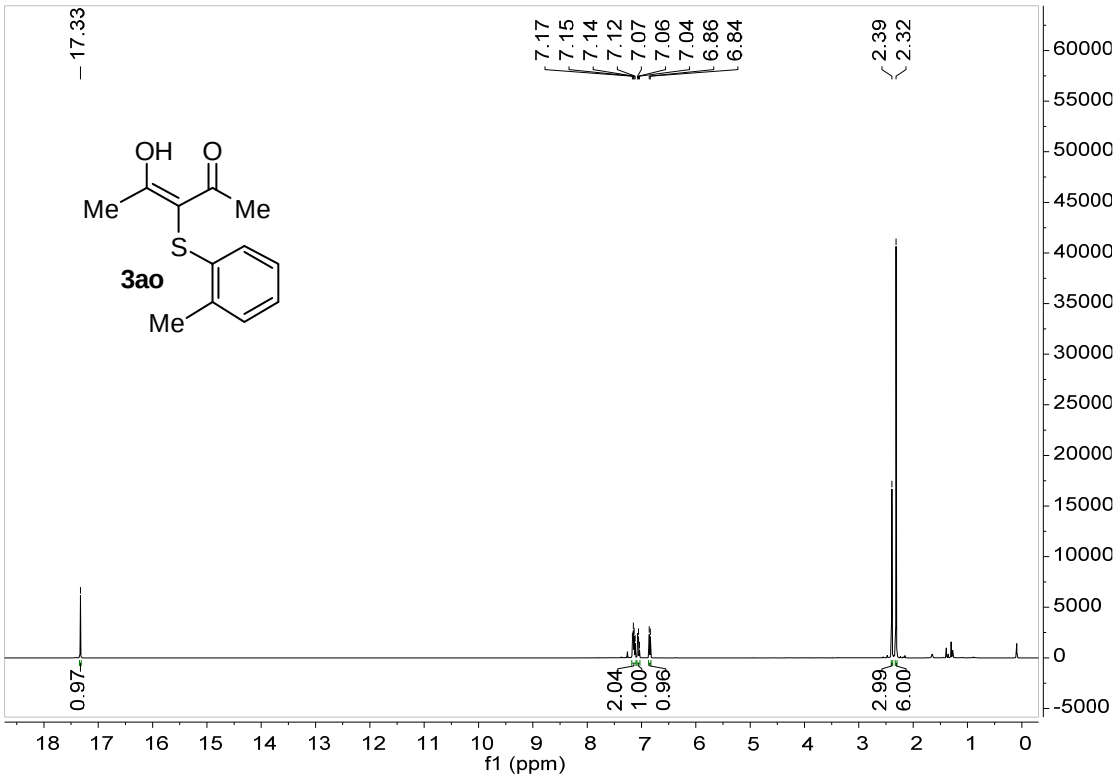
O
Me
Me
S
3an
F
f1 (ppm)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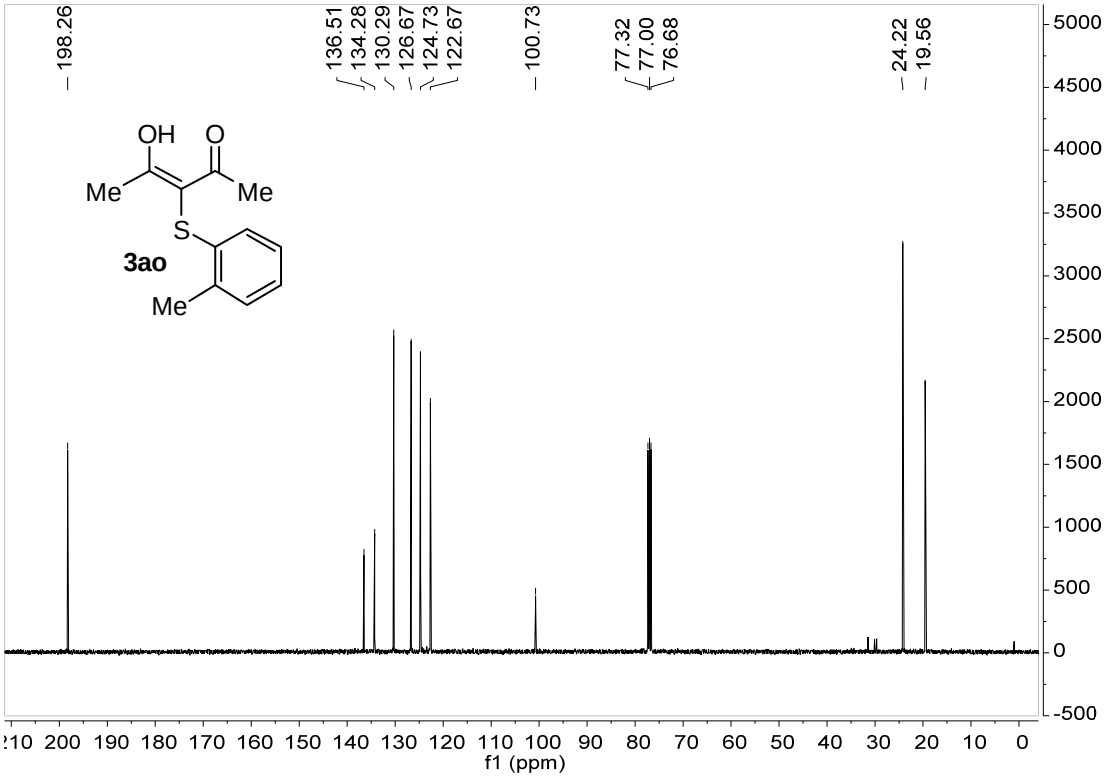
7.17
7.15
7.14
7.12
7.07
7.06
7.04
6.86
6.84
2.39
2.32
OH
O
Me
Me
S
3ao
Me
0.97
2.04
1.00
0.96
2.99
6.00
f1 (ppm)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130.29
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124.73
122.67
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77.00
76.68
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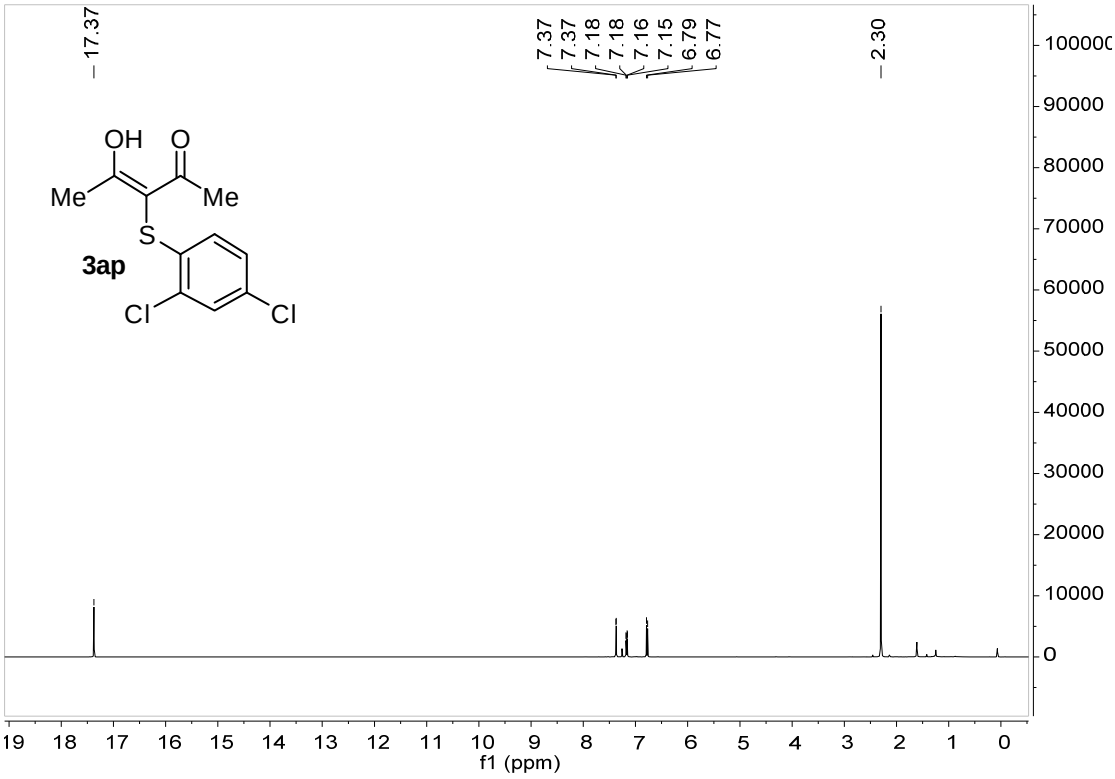
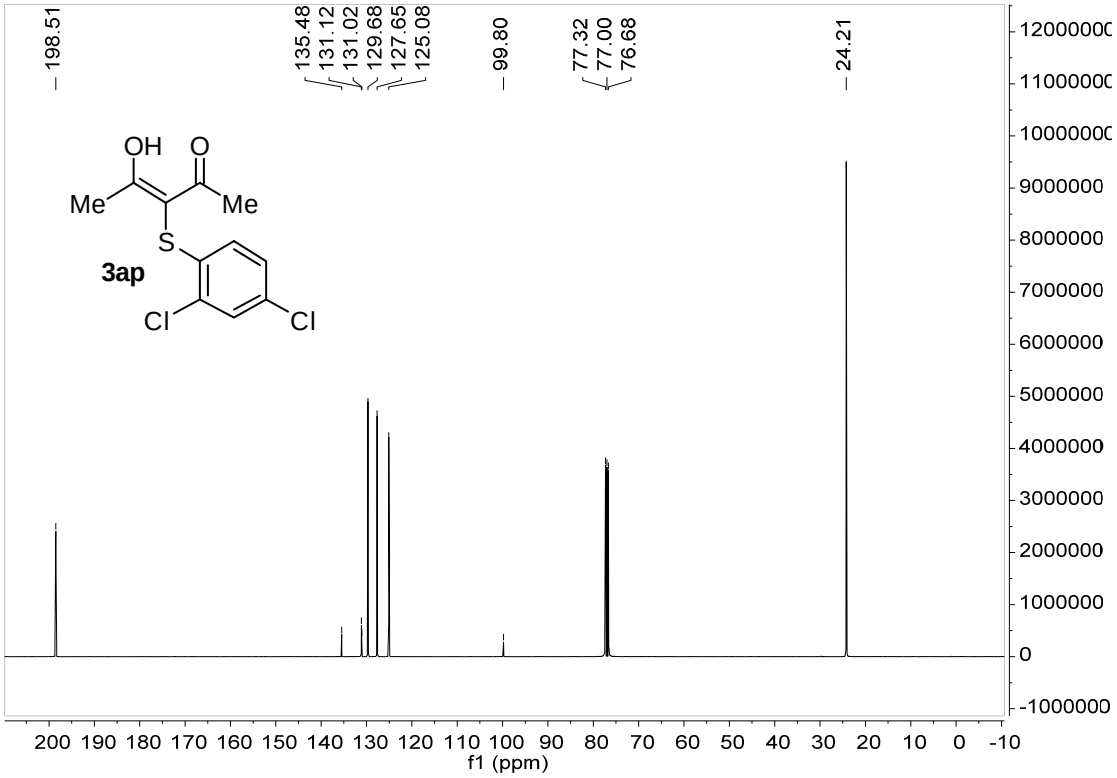
O
Me
Me
S
3ao
Me
f1 (ppm)


17.37
7.37
7.37
7.18
7.18
7.16
7.15
6.79
6.77
2.30
OH
O
Me
Me
S
3ap
Cl
Cl
f1 (ppm)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131.02
129.68
127.65
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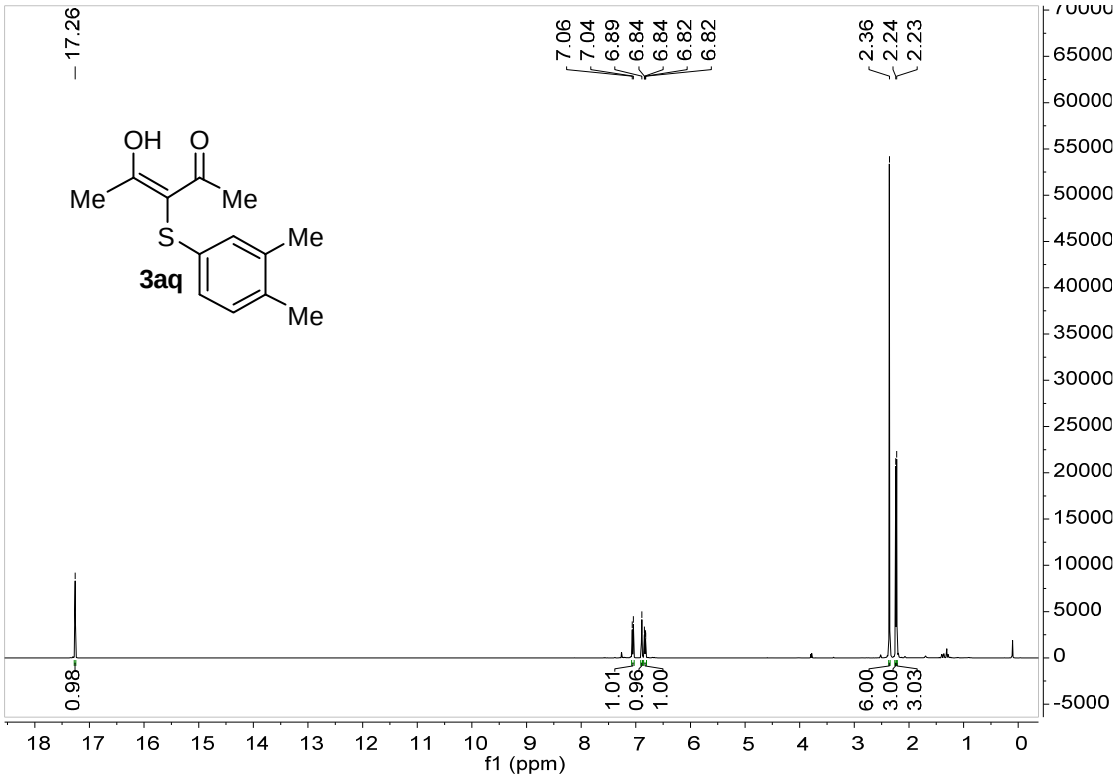
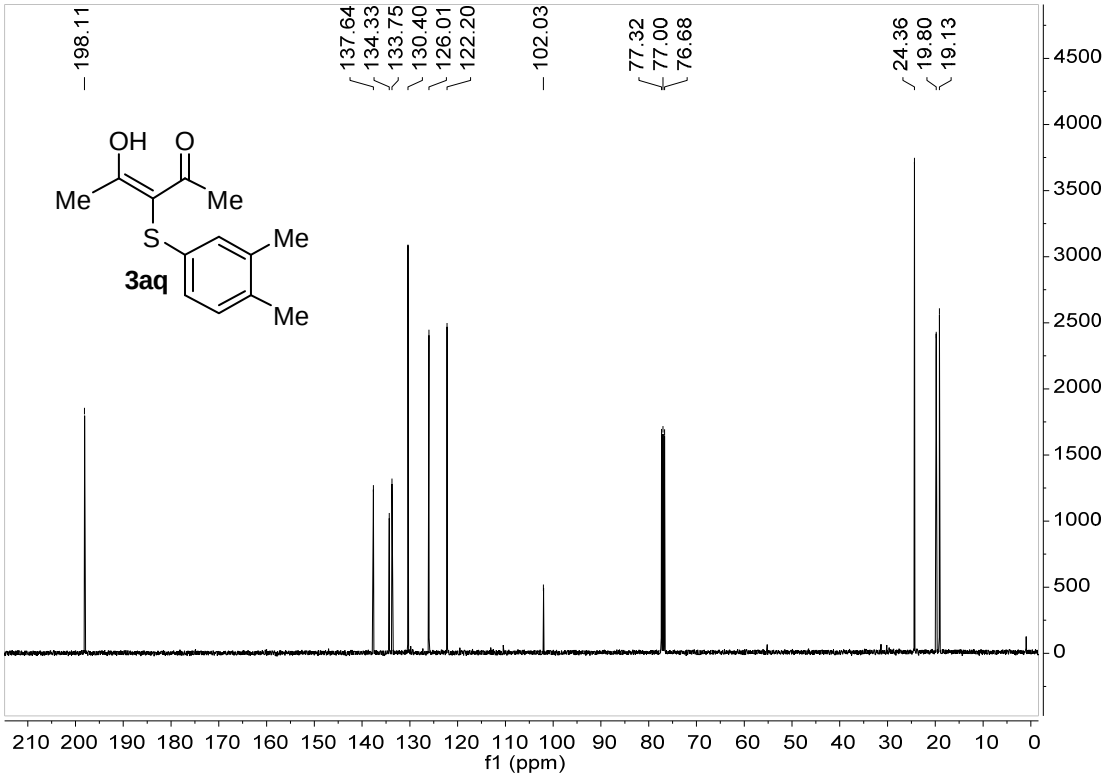
99.80
77.32
77.00
76.68
24.21
OH
O
Me
Me
S
3ap
Cl
Cl
f1 (ppm)


17.26
7.06
7.04
6.89
6.84
6.84
6.82
6.82
2.36
2.24
2.23
OH
O
Me
Me
S
Me
3aq
Me
0.98
1.01
0.96
1.00
6.00
3.00
3.03
f1 (ppm)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122.20
102.03
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77.00
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24.36
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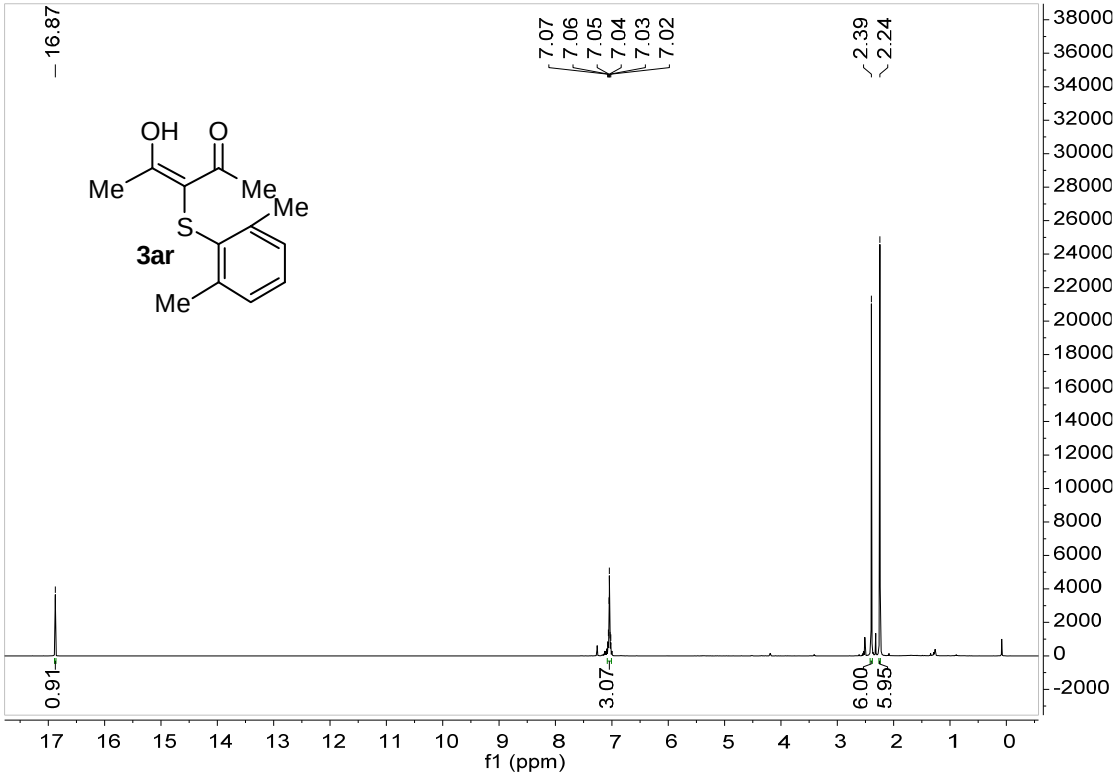
O
Me
Me
S
Me
Me
3aq
f1 (ppm)


16.87
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7.06
7.05
7.04
7.03
7.02
2.39
2.24
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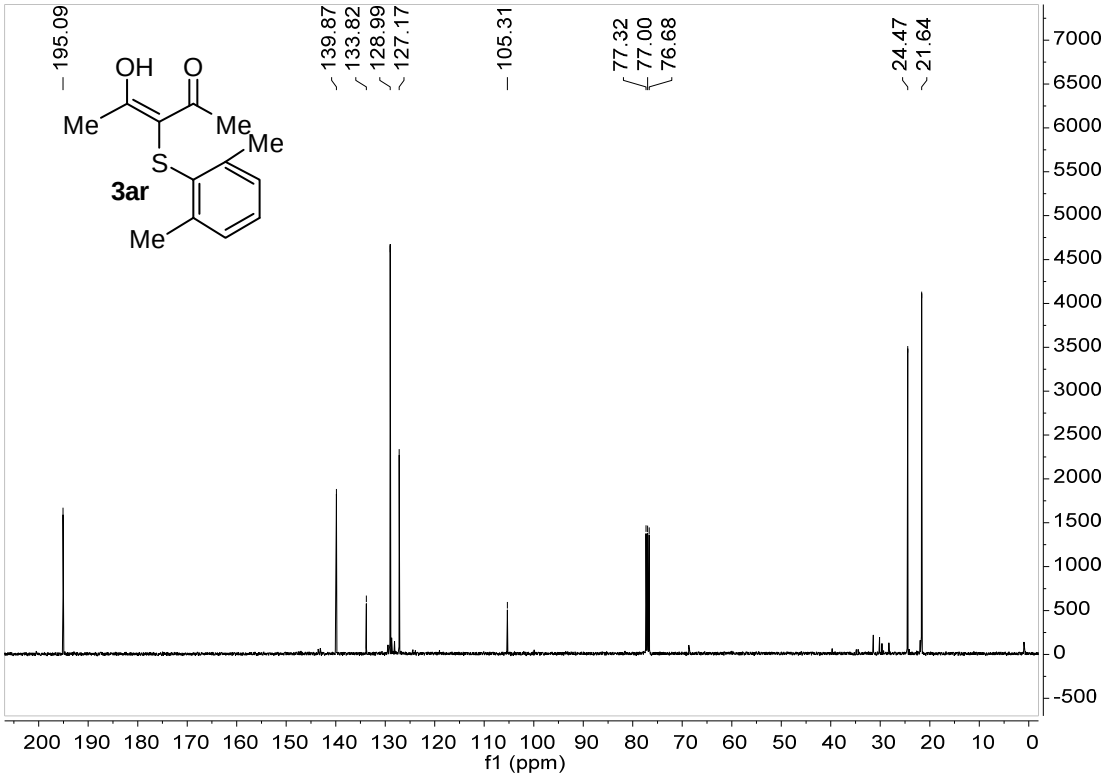
O
Me
Me
Me
S
3ar
Me
0.91
3.07
6.00
5.95
f1 (ppm)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76.68
24.47
21.64
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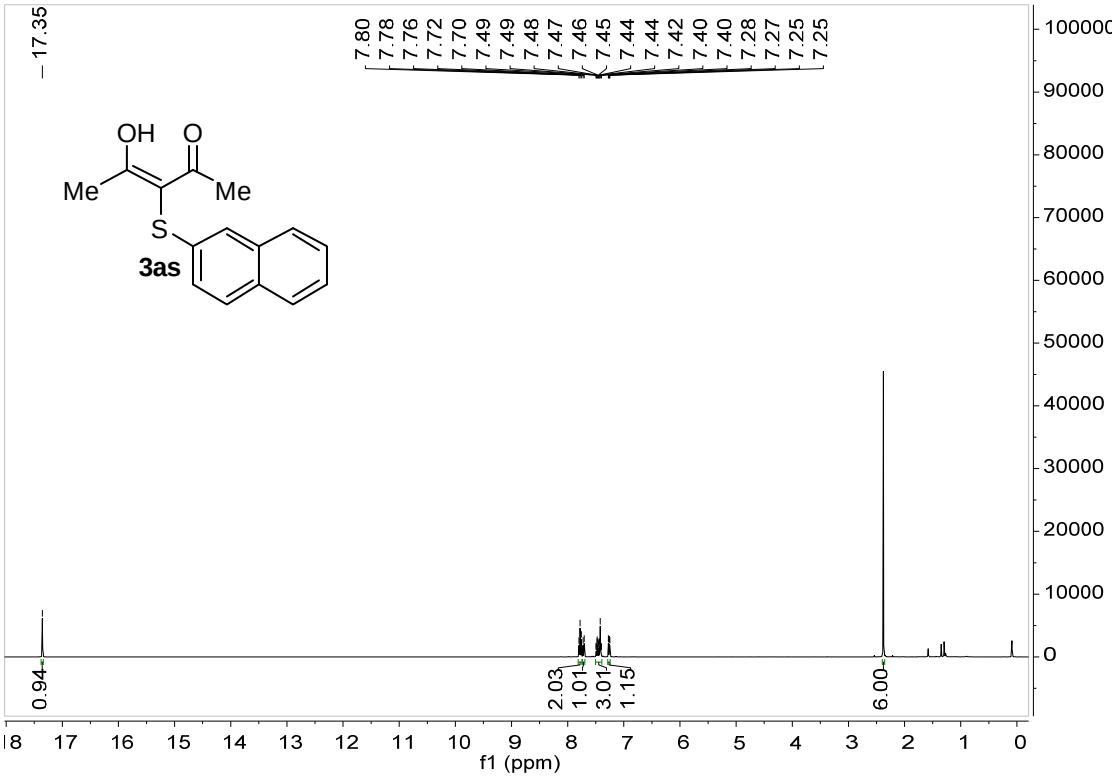
O
Me
Me
Me
S
3ar
Me
f1 (ppm)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7.49
7.49
7.48
7.47
7.46
7.45
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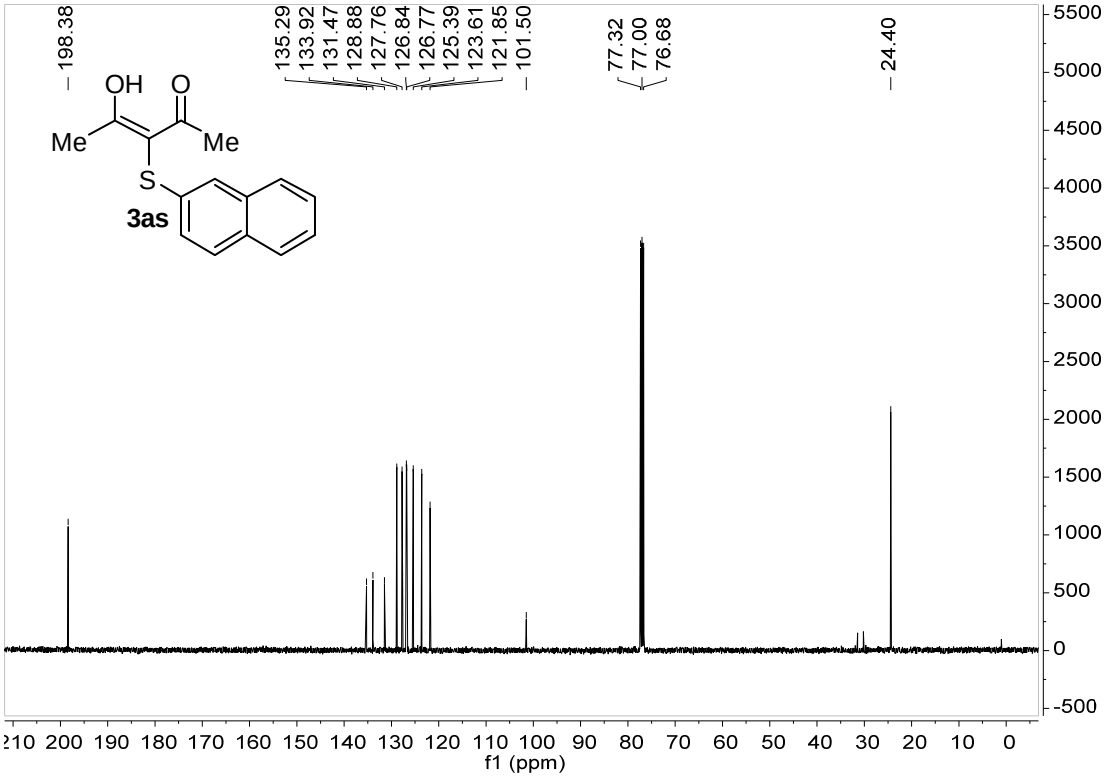
7.44
7.42
7.40
7.40
7.28
7.27
7.25
7.25
OH
O
Me
Me
S
3as
0.94
2.03
1.01
3.01
1.15
6.00
f1 (ppm)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127.76
126.84
126.77
125.39
123.61
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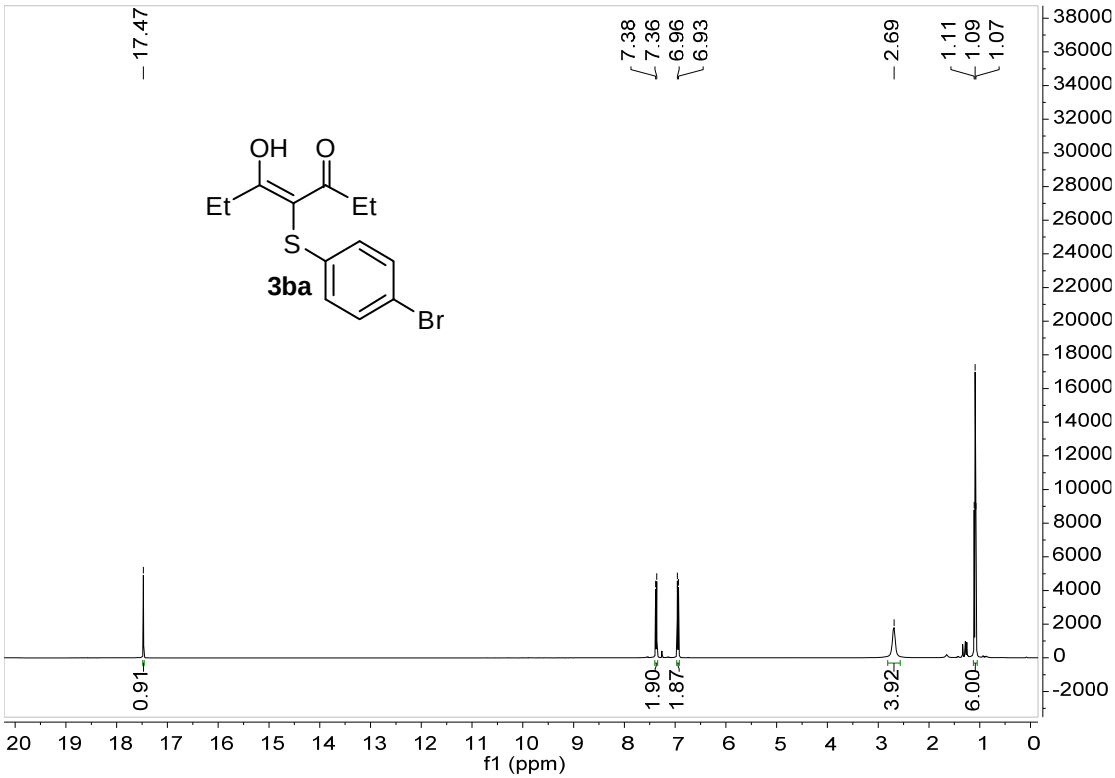
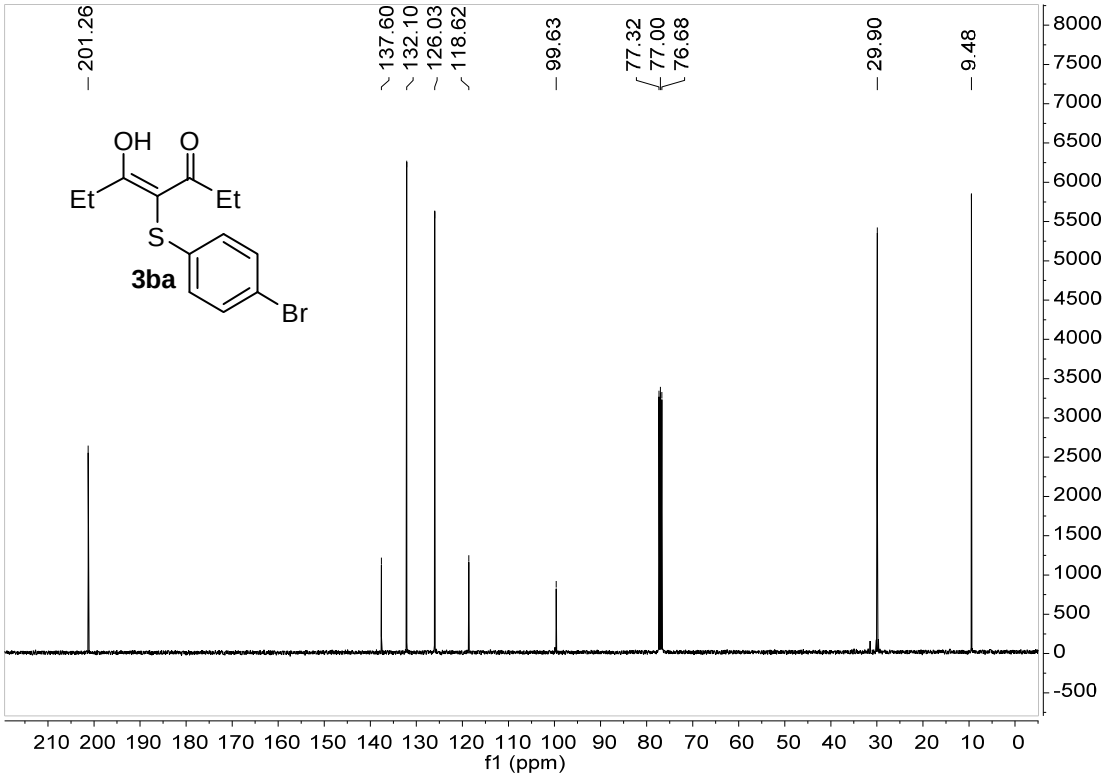
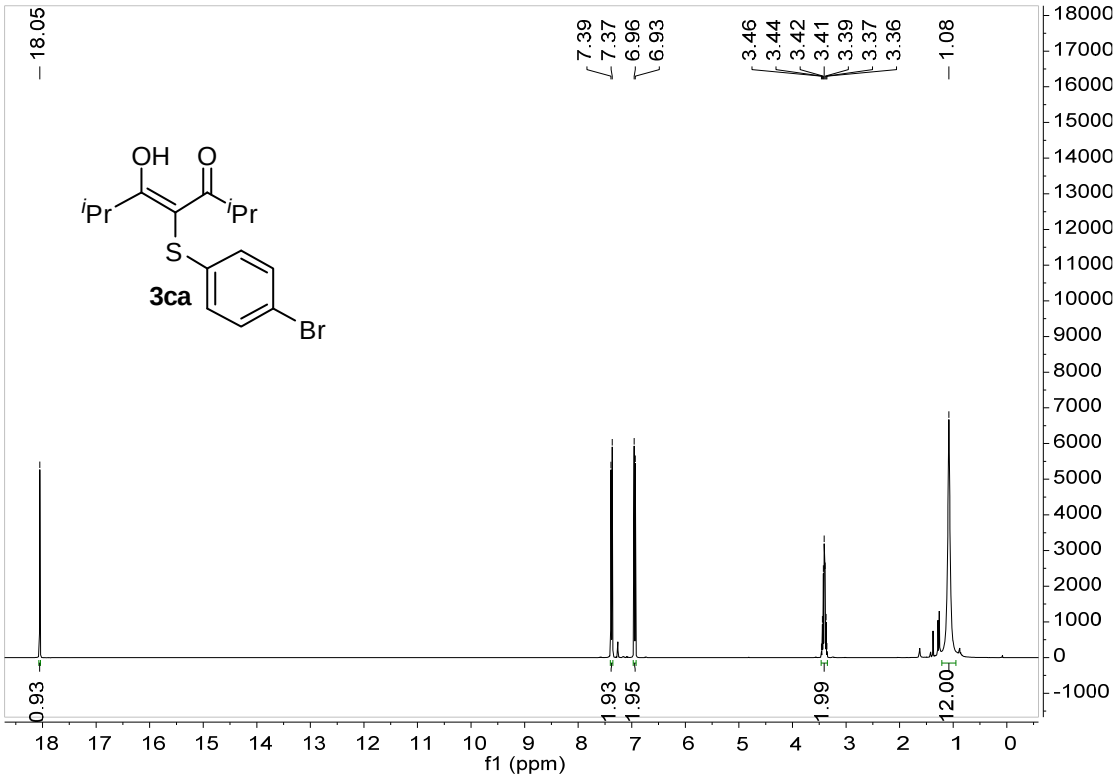
77.32
77.00
76.68
24.40
OH
O
Me
Me
S
3as
f1 (ppm)


17.47
7.38
7.36
6.96
6.93
2.69
1.11
1.09
1.07
OH
O
Et
Et
S
3ba
Br
0.91
1.90
1.87
3.92
6.00
f1 (ppm)


201.26
137.60
132.10
126.03
118.62
99.63
77.32
77.00
76.68
29.90
9.48
OH
O
Et
Et
S
3ba
Br
f1 (ppm)


18.05
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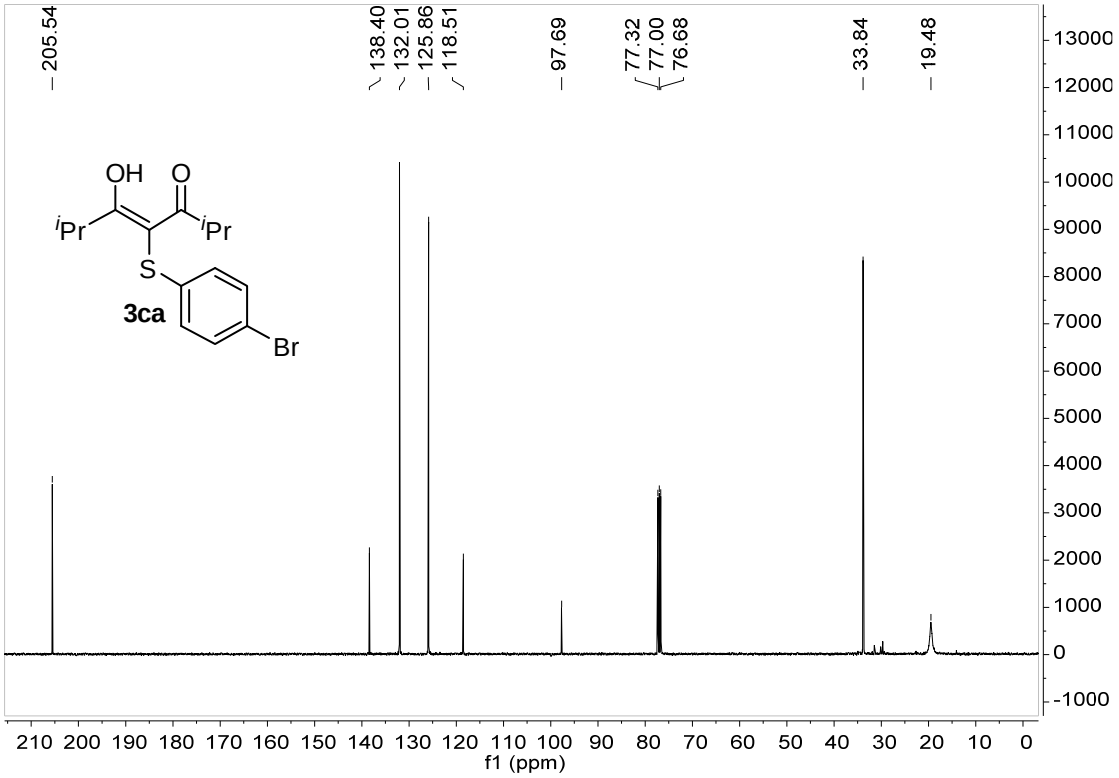
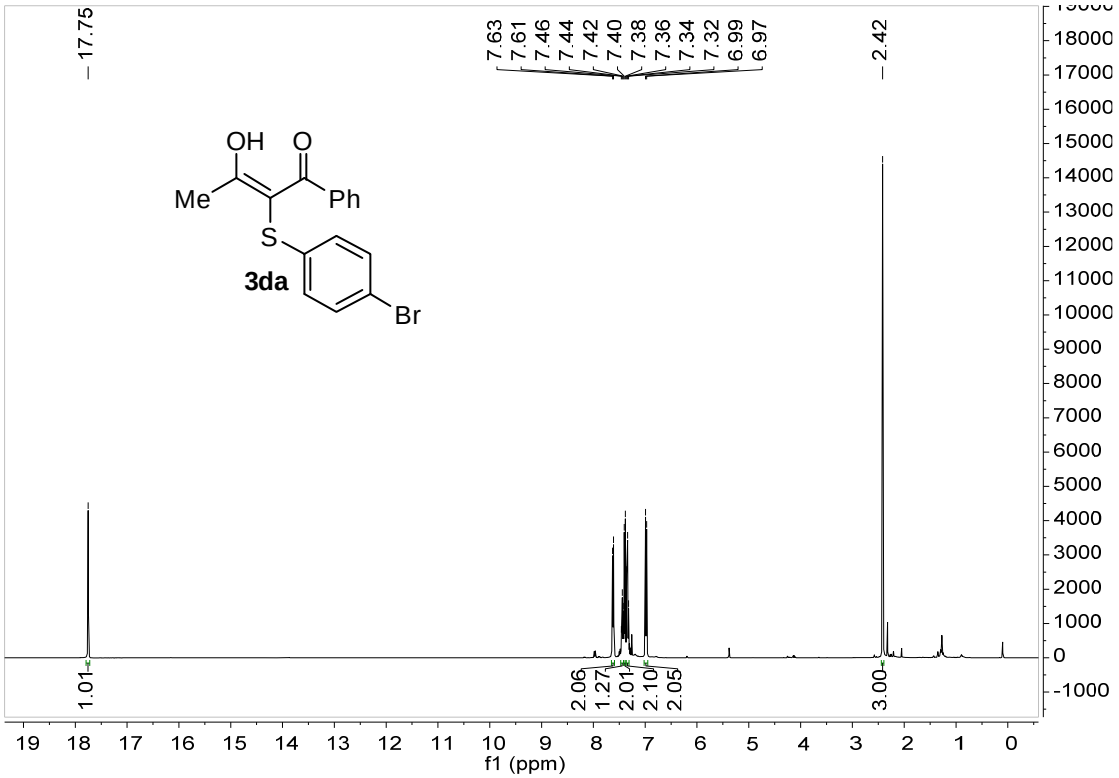
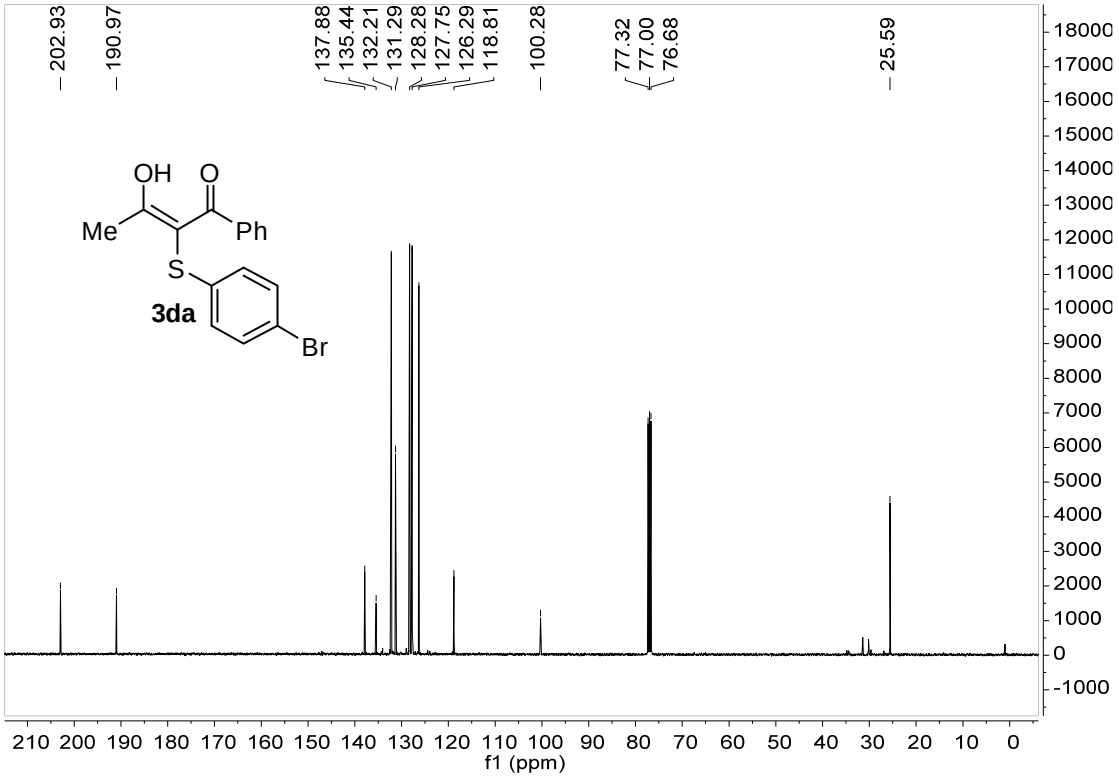
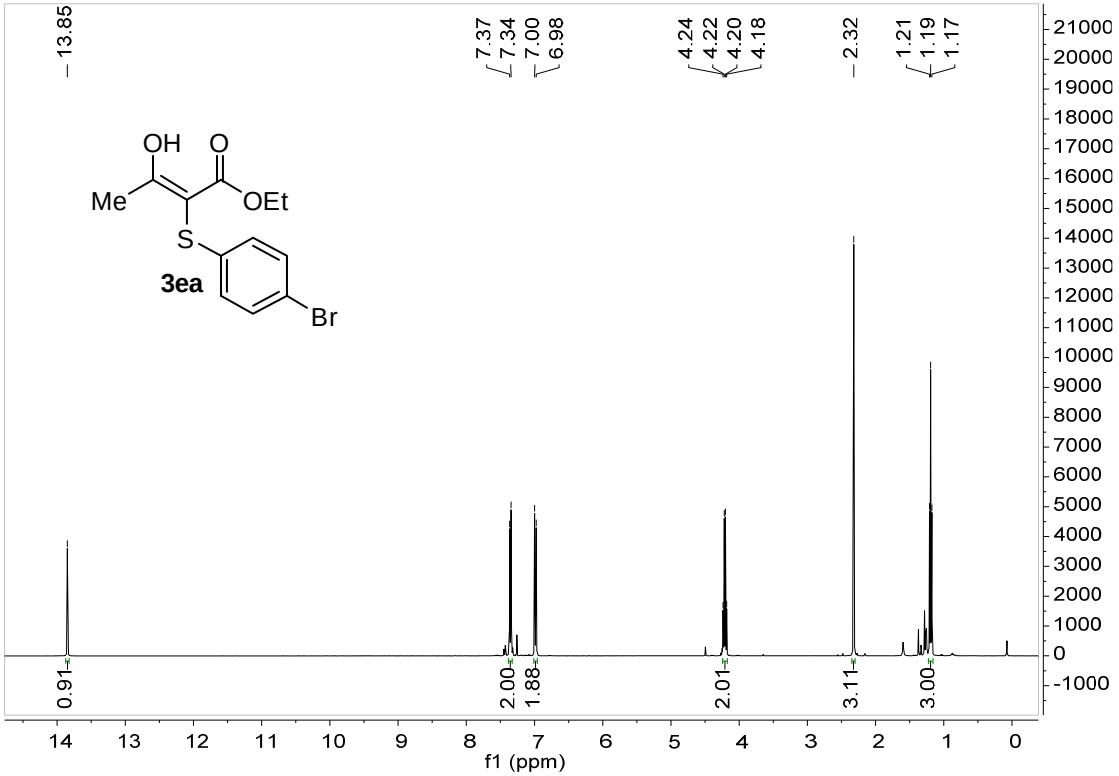
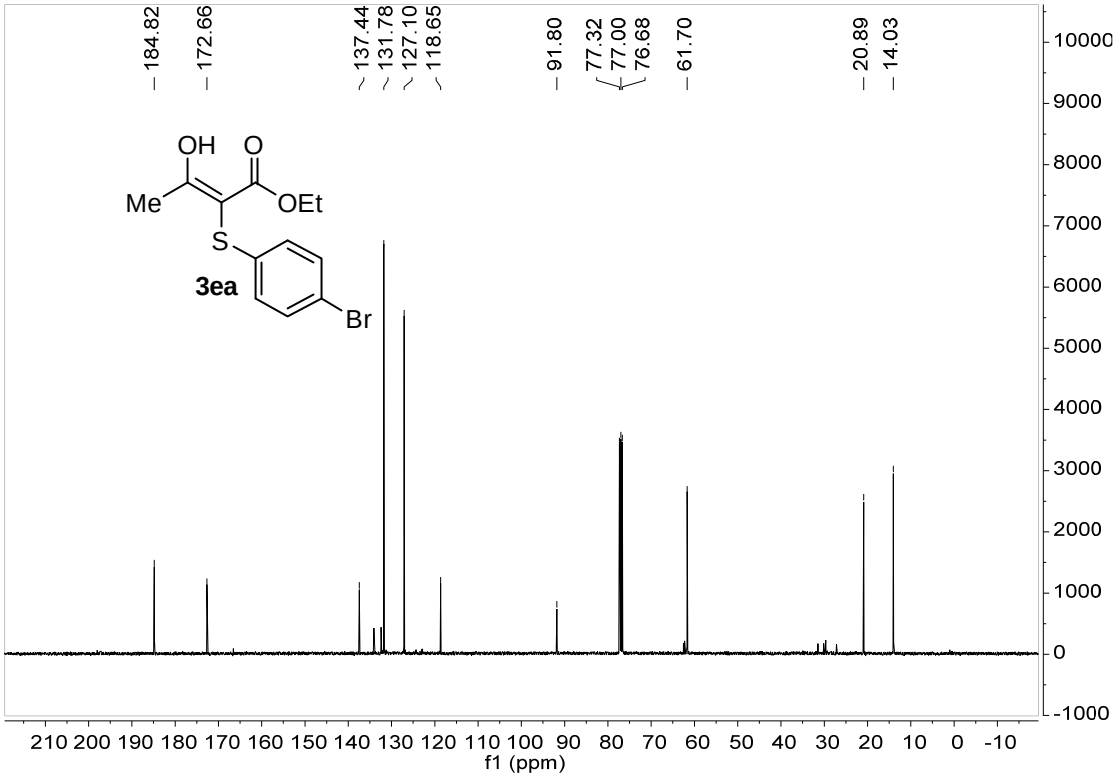
6.96
6.93
3.46
3.44
3.42
3.41
3.39
3.37
3.36
1.08
OH
O
iPr
iPr
S
3ca
Br
0.93
1.93
1.95
1.99
12.00
f1 (ppm)

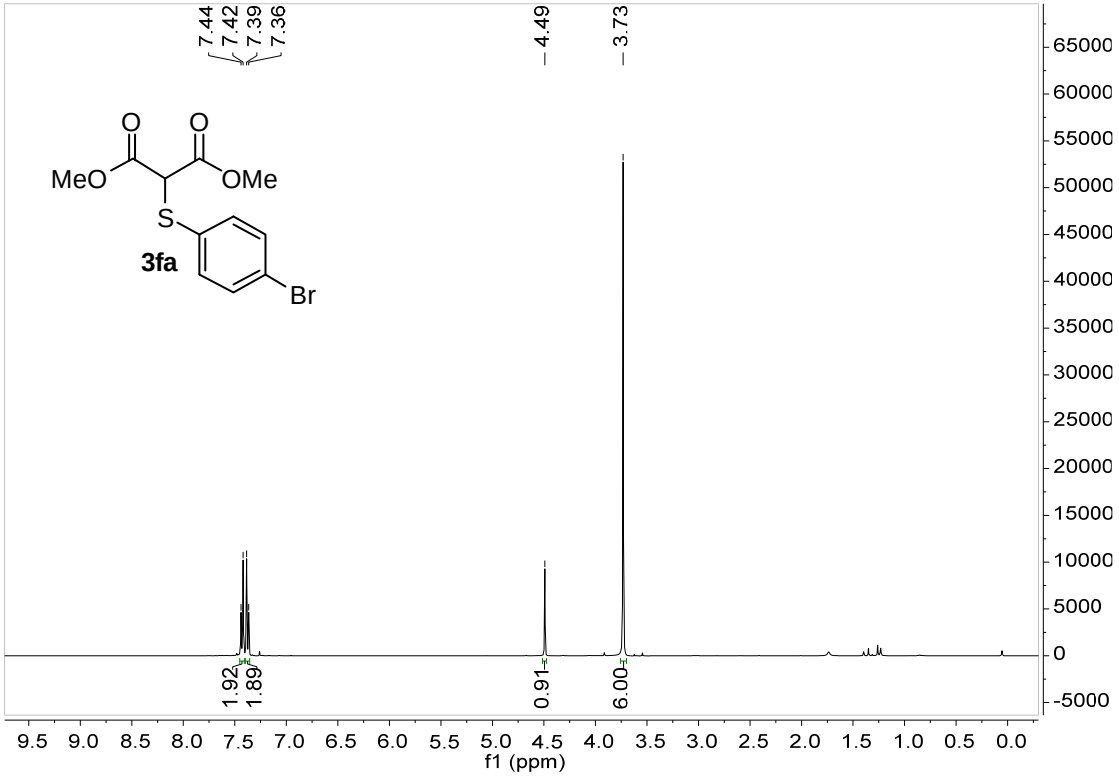

205.54
138.40
132.01
125.86
118.51
97.69
77.32
77.00
76.68
33.84
19.48
OH
O
iPr
iPr
S
3ca
Br
f1 (ppm)

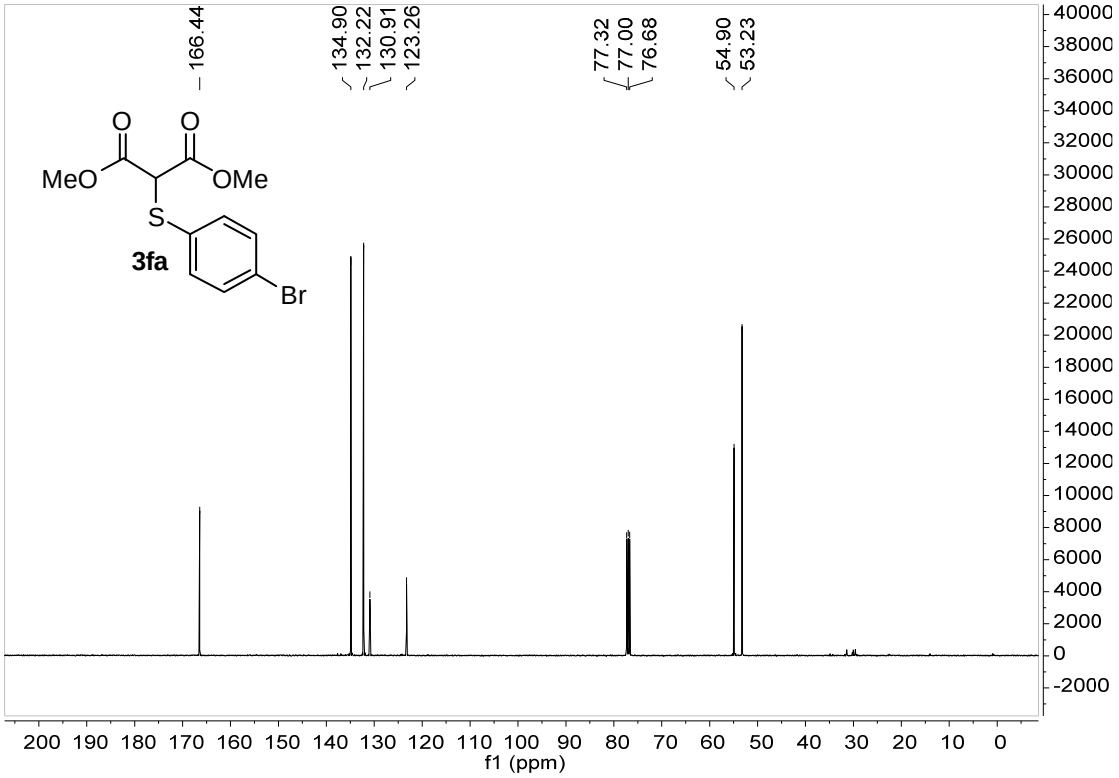

17.75
7.63
7.61
7.46
7.44
7.42
7.40
7.38
7.36
7.34
7.32
6.99
6.97
2.42
OH
O
Me
Ph
S
3da
Br
1.01
2.06
1.27
2.01
2.10
2.05
3.00
f1 (ppm)

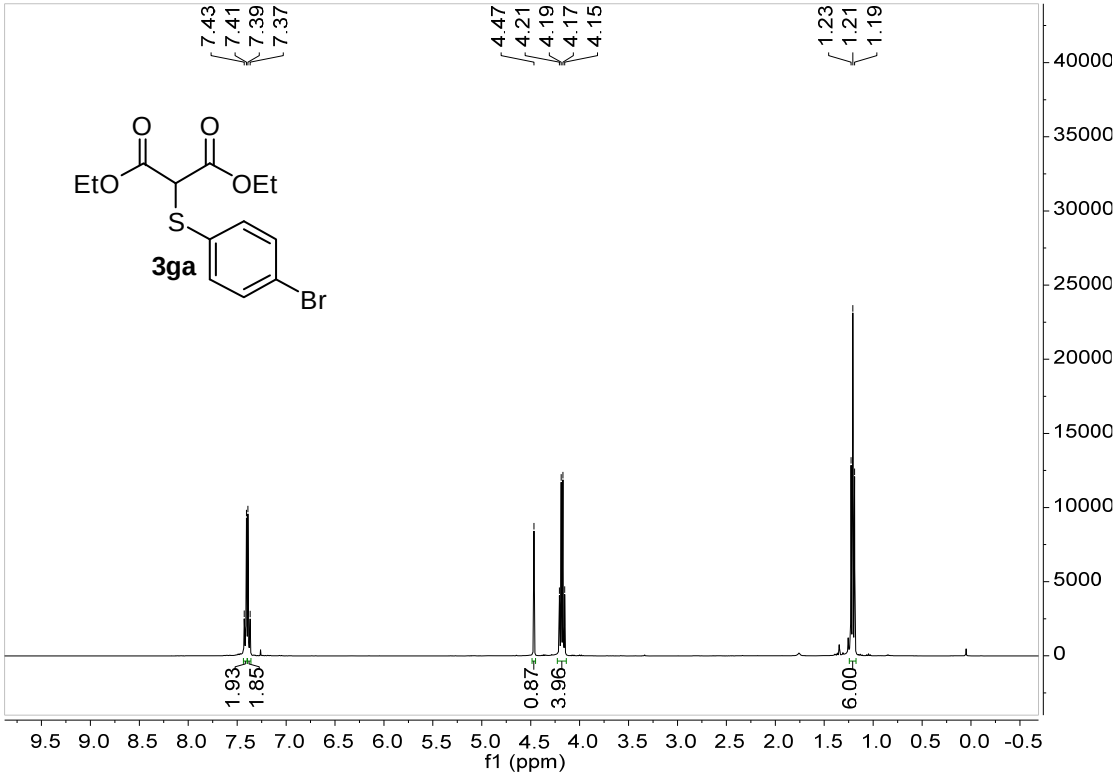

202.93
190.97
137.88
135.44
132.21
131.29
128.28
127.75
126.29
118.81
100.28
77.32
77.00
76.68
25.59
OH
O
Me
Ph
S
3da
Br
f1 (ppm)

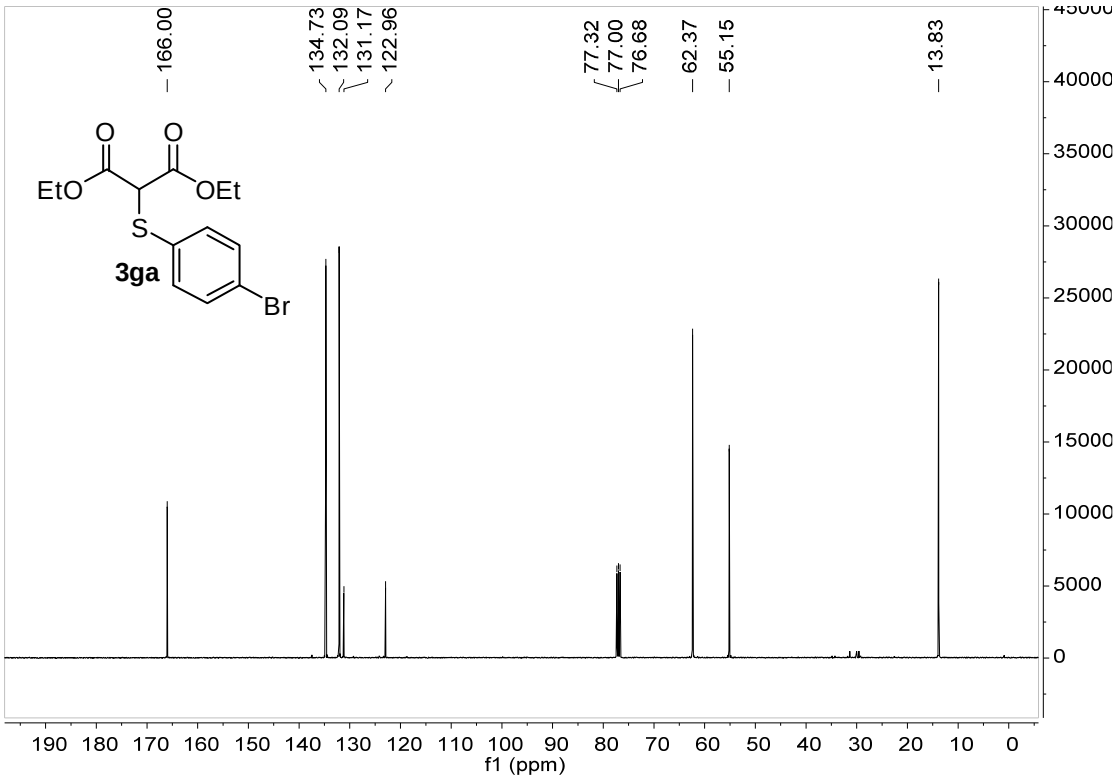

13.85
7.37
7.34
7.00
6.98
4.24
4.22
4.20
4.18
2.32
1.21
1.19
1.17
OH
O
Me
OEt
S
3ea
Br
0.91
2.00
1.88
2.01
3.11
3.00
f1 (ppm)

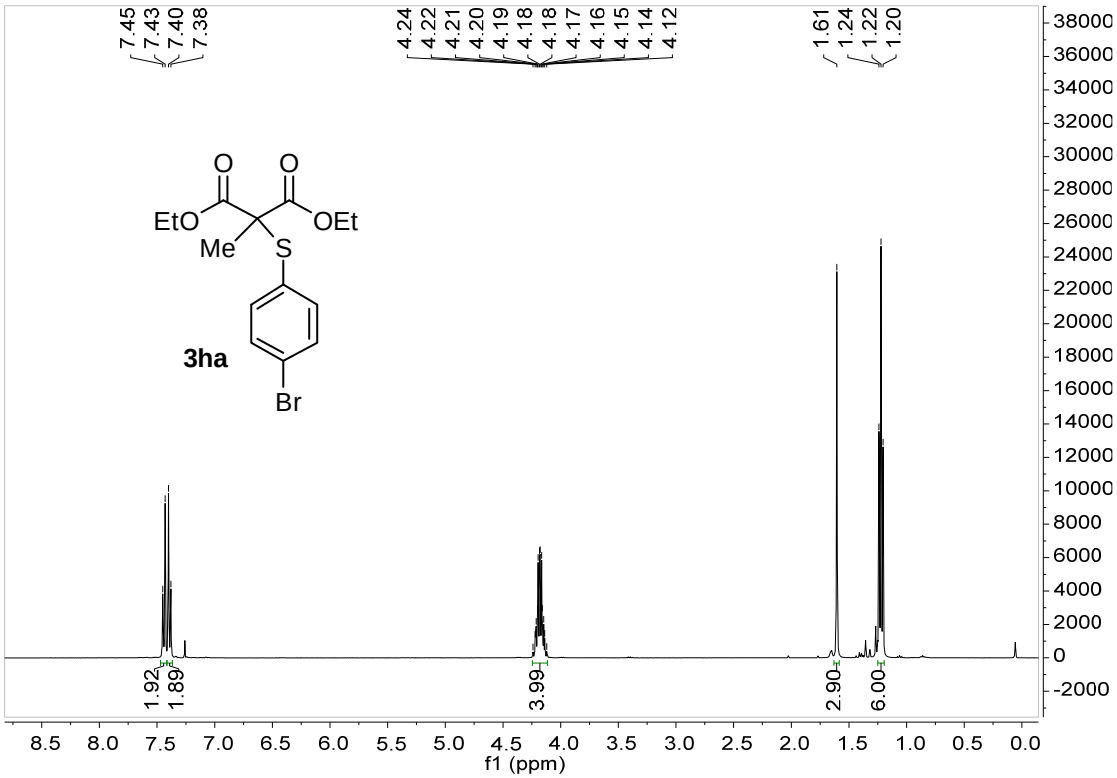

184.82
172.66
137.44
131.78
127.10
118.65
91.80
77.32
77.00
76.68
61.70
20.89
14.03
OH
O
Me
OEt
S
3ea
Br
f1 (ppm)

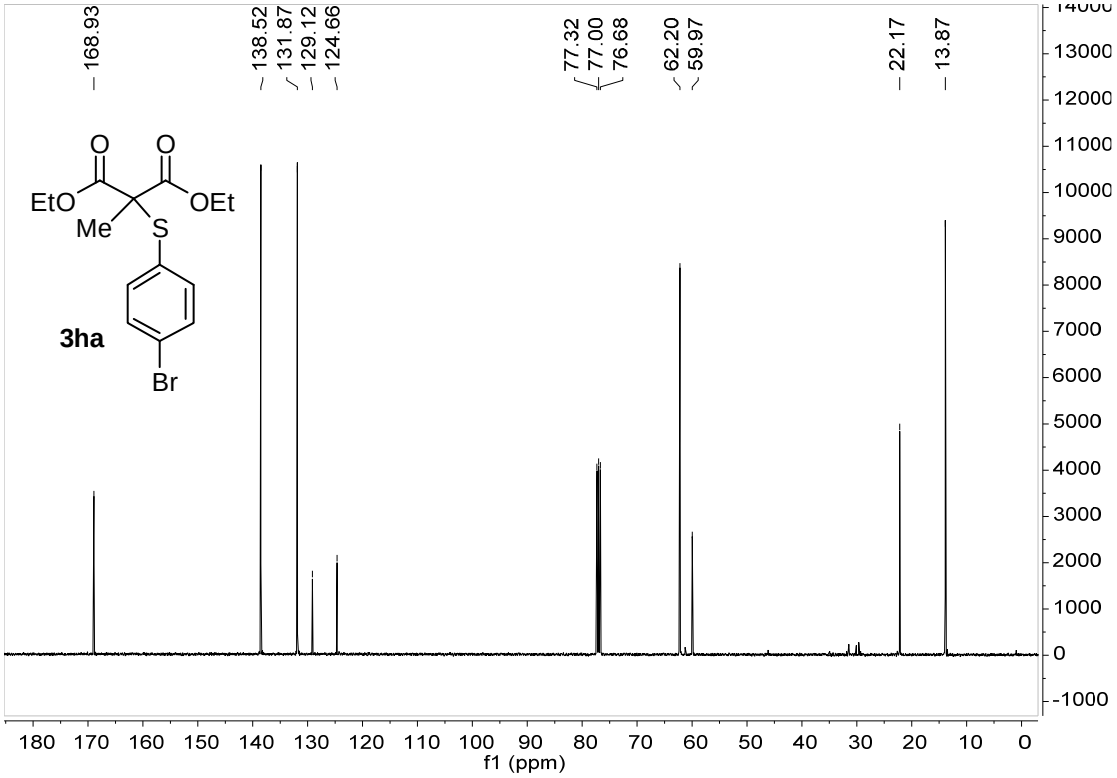

7.44
7.42
7.39
7.36
4.49
3.73
O
O
MeO
OMe
S
3fa
Br
1.92
1.89
0.91
6.00
f1 (ppm)

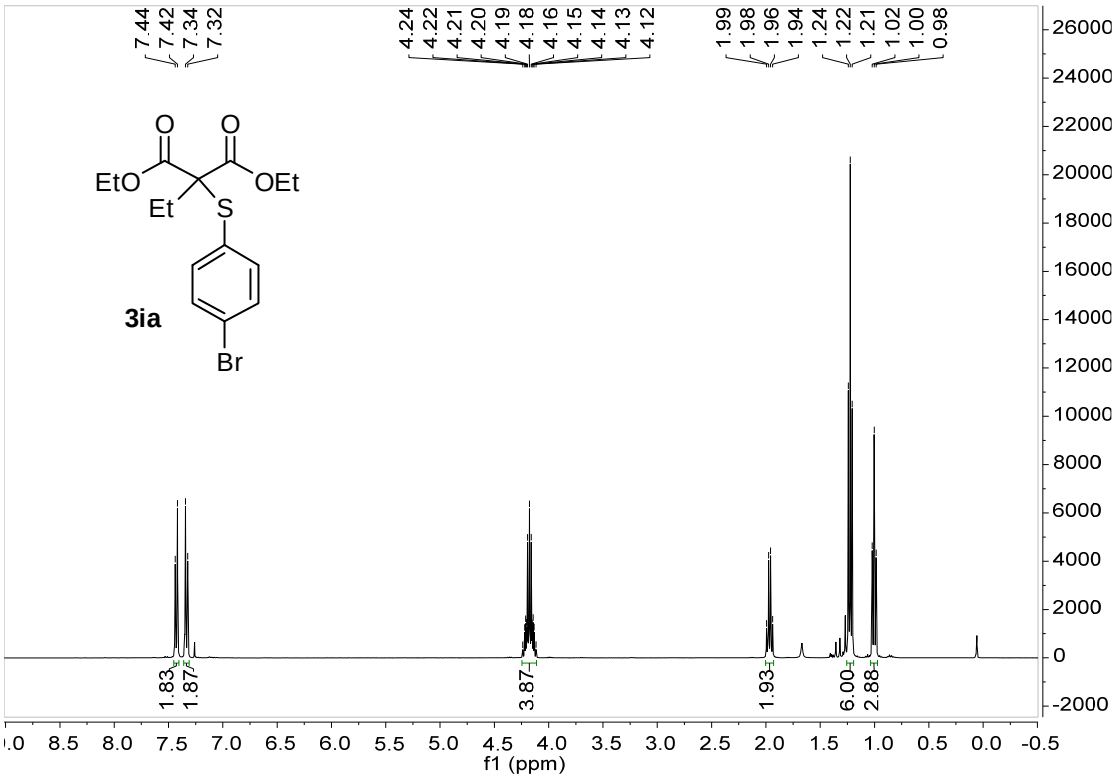

166.44
134.90
132.22
130.91
123.26
77.32
77.00
76.68
54.90
53.23
MeO
OMe
O
O
S
3fa
Br
f1 (ppm)

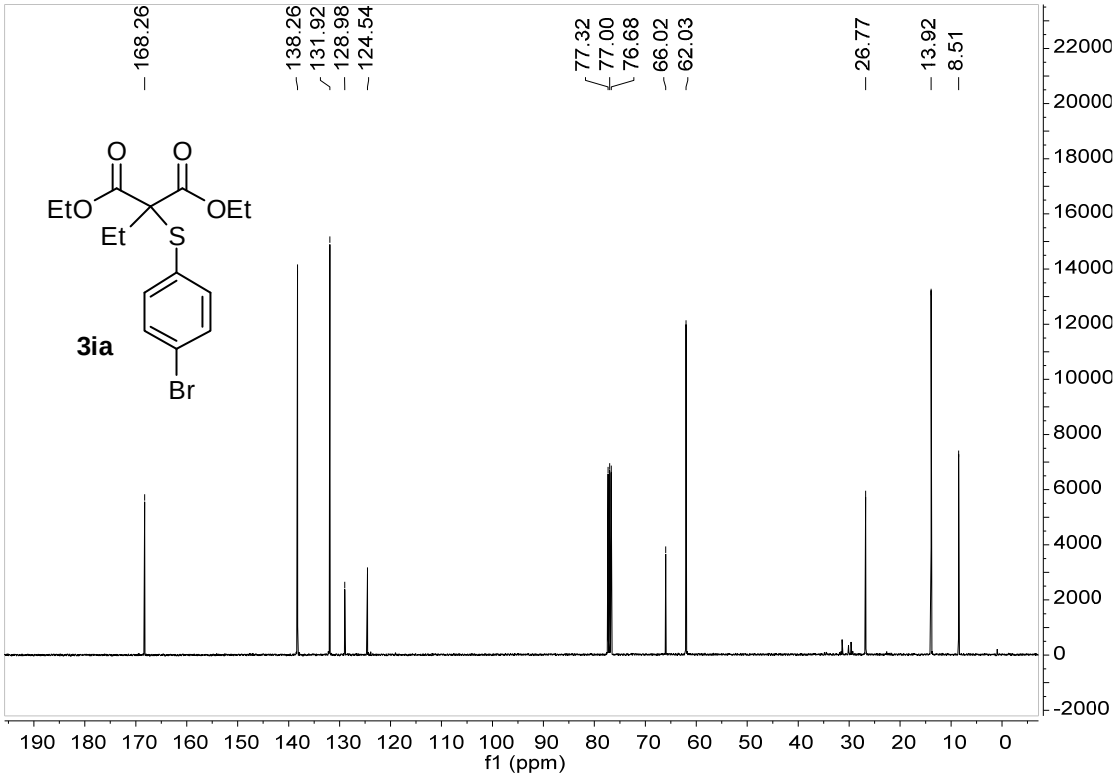

7.43
7.41
7.39
7.37
4.47
4.21
4.19
4.17
4.15
1.23
1.21
1.19
EtO
OEt
O
O
S
3ga
Br
1.93
1.85
0.87
3.96
6.00
f1 (ppm)


166.00
134.73
132.09
131.17
122.96
77.32
77.00
76.68
62.37
55.15
13.83
O
O
EtO
OEt
S
3ga
Br
f1 (ppm)


7.45
7.43
7.40
7.38
4.24
4.22
4.21
4.20
4.19
4.18
4.18
4.17
4.16
4.15
4.14
4.12
1.61
1.24
1.22
1.20
O
O
EtO
OEt
Me
S
3ha
Br
1.92
1.89
3.99
2.90
6.00
f1 (ppm)


168.93
138.52
131.87
129.12
124.66
77.32
77.00
76.68
62.20
59.97
22.17
13.87
O
O
EtO
OEt
Me
S
3ha
Br
f1 (ppm)


7.44
7.42
7.34
7.32
4.24
4.22
4.21
4.20
4.19
4.18
4.16
4.15
4.14
4.13
4.12
1.99
1.98
1.96
1.94
1.24
1.22
1.21
1.02
1.00
0.98
O
O
EtO
OEt
Et
S
3ia
Br
1.83
1.87
3.87
1.93
6.00
2.88
f1 (ppm)


168.26
138.26
131.92
128.98
124.54
77.32
77.00
76.68
66.02
62.03
26.77
13.92
8.51
O
O
EtO
OEt
Et
S
Br
3ia
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)