Electronic Supplementary Material (ESI) for Chemical Communications

Synthesis of Chromans via Pd-Catalyzed Alkene Carboetherification Reactions.

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Supporting Information

Experimental procedures and characterization data for new compounds.

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General. All reactions were carried out under a nitrogen atmosphere in oven or flame dried glassware. Tris(dibenzylideneacetone)dipalladium (0) and all phosphine ligands were purchased from Strem Chemical Co. and used without further purification. 2-Allylphenol and all aryl bromides were obtained from commercial sources (Aldrich Chemical Co. or Acros Chemical Co.) and were used as obtained. 2-(But-3-en-1-yl)phenol (**7**),[1] 2-(3-methylbut-3-en-1-yl)phenol (**10**),[1] (*E*)-3-(2-methoxyphenyl)-1-phenylpropan-1-one,[2] 2-cyclopentylidene-1,1-dimethylhydrazine,[3] 1-(bromomethyl)-2-methoxybenzene,[4] 2-cyclohexylidene-1,1-

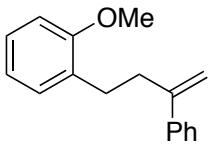
dimethylhydrazine,[3] and [2-(bromomethyl)phenoxy](*tert*-butyl)dimethylsilane,[5] were prepared according to literature procedures. Toluene and THF were purified using a GlassContour solvent purification system. Yields refer to isolated yields of compounds estimated to be ≥95% pure as determined by ^{1}H NMR. The yields reported in the supporting information describe the result of a single experiment, whereas the yields reported in Tables 2–3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Tables 2–3.

Synthesis of Substrates

General Procedure 1: Alkylation of hydrazones.[6] An oven-dried flask equipped with a magnetic stirbar was cooled under a stream of nitrogen and charged with the appropriate hydrazone (1 equiv) and THF (1 M). The resulting solution was cooled to 0 °C and a solution of *n*-BuLi (1 equiv, 1.6 M in hexanes) was added dropwise. The reaction mixture was stirred at 0 °C for 1 hr, then the alkyl halide (1 equiv) was added dropwise as a 1 M solution in THF, and the reaction mixture was warmed to rt. The mixture was stirred at rt until GC analysis indicated that the starting materials were fully consumed, then 1 M HCl was added (10 mL), and the reaction was stirred for 4 hr at rt. Brine (5 mL) and EtOAc (5 mL) were added, and the mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel.

General Procedure 2: Methylenation of ketones.[7] A flame-dried round-bottomed flask equipped with a magnetic stirbar and a rubber septum was cooled under a stream of nitrogen and charged with methyltriphenylphosphonium bromide (1 equiv) and THF (1 M). The reaction mixture was cooled to –78 ^{o}C and a solution of NaHMDS was added dropwise (1 equiv, 2 M in THF). The resulting mixture was stirred at –78 ^{o}C for 2 h then the ketone substrate (1 equiv) was added dropwise as a 1 M solution in THF and the reaction mixture was heated to 40 ^{o}C until the starting material had been completely consumed as judged by TLC analysis. The mixture was cooled to rt, brine (5 mL) and EtOAc (5 mL) were added, and the mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel.

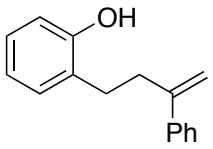
General Procedure 3: Deprotection of aryl(methyl)ethers. A flame-dried round-bottomed flask equipped with a magnetic stirbar, reflux condenser and a rubber septum was cooled under a stream of nitrogen and charged with NaH (4 equiv) and DMF (2 M). The reaction mixture was cooled to 0 ^{o}C and a 2 M solution of ethanethiol (2.6 equiv) in DMF was added dropwise. The resulting mixture was stirred at rt for 30 min, then the methyl ether substrate (1 equiv) was added and the reaction mixture was heated to 160 ^{o}C until the starting material had been completely consumed as judged by TLC analysis. The reaction mixture was then cooled to rt and 1 M HCl (5 mL) and EtOAc (5 mL) were added. The mixture was transferred to a separatory funnel and the layers were separated. The aqueous layer was extracted with EtOAc (2 x 20 mL), and the combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel.



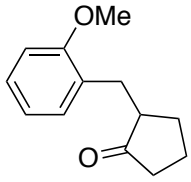
1-Methoxy-2-(3-phenylbut-3-en-1-yl)benzene (S2). A flame-dried round-bottomed flask equipped with a magnetic stirbar and a rubber septum was cooled under a stream of nitrogen and charged with Cp_2TiCl_2 (0.10 g 0.43 mmol), activated Zn powder (1.42 g, 21.7 mmol), NEt_3HCl (5.98 g, 44 mmol) and CH_2Cl_2 (80 mL). The resulting mixture was stirred at rt for 10 min then a solution of (*E*)-3-(2-methoxyphenyl)-1-phenylprop-2-en-1-one (2.07 g, 8.8 mmol) in CH_2Cl_2 (40 mL) was added dropwise. The resulting mixture was stirred at rt until the starting material had been completely consumed as judged by TLC analysis. The mixture was filtered through a plug of celite, transferred to a separatory funnel, and washed with saturated aqueous NH_4Cl (20 mL). The layers were separated, the aqueous layer was extracted with EtOAc (2 x 20 mL), and the organic phases were then combined, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 1.56 g (75%) of 3-(2-methoxyphenyl)-1-phenylpropan-1-one (**S1**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02–8.00 (m, 2 H), 7.58–7.52 (m, 1 H), 7.4 (t, *J* = 7.6 Hz, 2 H), 7.26–7.20 (m, 2 H), 6.93 (t, *J* = 7.4 Hz, 1 H), 6.88 (d, *J* = 8.6 Hz, 1 H), 3.83 (s, 3 H), 3.32–3.26 (m, 2 H), 3.10 (t, *J* = 8.2 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 157.5, 137.0, 132.9, 130.2, 129.5, 128.6, 128.1, 127.6, 120.6, 110.3, 55.2, 38.9, 25.8;

General Procedure 2 was used for the conversion of 3-(2-methoxyphenyl)-1-phenylpropan-1-one (1.77 g, 7.36 mmol) to the title compound. This procedure afforded 0.98 g (56%) of the title compound as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.0 Hz, 2 H), 7.33 (t, *J* = 7.0 Hz, 2 H), 7.26 (d, *J* = 7.0 Hz, 1 H), 7.20–7.14 (m, 1 H), 7.09 (dd, *J* = 1.6, 7.2 Hz, 1 H), 6.89–6.81 (m, 1 H), 5.30 (s, 1 H), 5.06 (s, 1 H), 3.81 (s, 3 H), 2.76 (s, 4 H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 157.5, 148.3, 141.2, 130.4, 129.9, 128.3, 127.3, 127.1, 126.1, 120.3, 112.2, 110.2, 55.2, 35.5, 29.7; IR (film, cm^{-1}) 2929, 1495, 1243; MS(ESI): 241.1228 (241.1223 calcd for $C_{17}H_{18}O$, $M + H^+$).

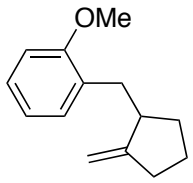


2-(3-Phenylbut-3-en-1-yl)phenol (10). General Procedure 3 was used for the conversion of 1-methoxy-2-(3-phenylbut-3-en-1-yl)benzene (1.1 g, 4.6 mmol) to the title compound. This procedure afforded 0.66 g (63%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.8 Hz, 2 H), 7.43 (t, *J* = 7.3 Hz, 2 H), 7.40–7.34 (m, 1 H), 7.20–7.14 (m, 2 H), 6.96 (t, *J* = 7.6 Hz, 1 H), 6.79 (d, *J* = 7.8 Hz, 1 H), 5.42 (s, 1 H), 5.18 (s, 1 H), 4.86 (s, 1 H), 2.94–2.84 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5, 148.0, 141.0, 130.4, 128.5, 128.0, 127.6, 127.4, 126.2, 120.9, 115.4, 112.8, 35.5, 29.3; IR (film, cm^{-1}) 3411, 3030, 1454; MS(EI): 224.1208 (224.1201 calcd for $C_{16}H_{16}O$, M^+).

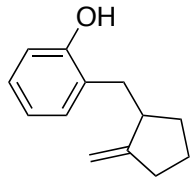


2-(2-Methoxybenzyl)cyclopentanone (S3). General Procedure 1 was used for the conversion of 2-cyclopentylidene-1,1-dimethylhydrazine (1.0 g, 7.9 mmol) and 1-(bromomethyl)-2-methoxybenzene (1.24 g, 7.9 mmol) to the title compound. This procedure afforded 1.0 g (62%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (dd, *J* = 1.7, 7.8 Hz, 1 H), 7.12 (dd, *J* = 1.7, 7.3 Hz, 1 H), 6.88 (t, *J* = 7.6 Hz, 1 H), 6.85 (d, *J* = 8.0 Hz, 1 H), 3.82 (s, 3 H), 3.28–3.20 (m, 1 H), 2.51–2.42 (m, 2 H), 2.37–2.29 (m, 1 H), 2.19–2.10 (m, 1 H), 2.04–1.92

(m, 2 H), 1.79–1.66 (m, 1 H), 1.60–1.50 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 220.7, 157.6, 130.5, 128.5, 127.4, 120.3, 110.2, 55.2, 49.6, 38.1, 30.2, 29.3, 20.6; IR (film, cm^{-1}) 2928, 1699, 1456; MS(EI): 204.1148 (204.1150 calcd for $C_{13}H_{16}O_2$, M^+).

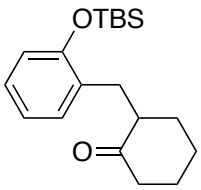


1-Methoxy-2-[(2-methylenecyclopentyl)methyl]benzene (S4). General Procedure 2 was used for the conversion of 2-(2-methoxybenzyl)cyclopentanone (0.7 g, 3.5 mmol) to the title compound. This procedure afforded 0.64 g (93%) of the title compound as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.23 (m, 2 H), 6.99 (t, J = 7.3 Hz, 1 H), 6.94 (d, J = 8.3 Hz, 1 H), 5.05 (s, 1 H), 4.97 (s, 1 H), 3.91 (s, 3 H), 3.12 (dd, J = 5.1, 13.2 Hz, 1 H), 2.87–2.80 (m, 1 H), 2.60 (dd, J = 9.8, 13.2 Hz, 1 H), 2.53–2.47 (m, 2 H), 1.88–1.76 (m, 2 H), 1.68–1.56 (m, 1 H), 1.51–1.43 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 156.7, 130.7, 130.0, 127.1, 120.3, 110.2, 104.6, 55.2, 44.2, 35.3, 33.3, 32.8, 24.1; IR (film, cm^{-1}) 2928, 1490, 1261; MS(EI): 302.0274 (302.0266 calcd for $C_{14}H_{18}O$, M^+).



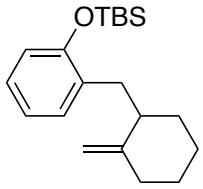
2-[(2-Methylenecyclopentyl)methyl]phenol (11). An oven-dried flask equipped with a magnetic stirbar and a reflux condenser was cooled under a stream of nitrogen and charged with 1-methoxy-2-[(2-methylenecyclopentyl)methyl]benzene (0.1 g, 0.46 mmol). L-selectride (3

equiv, 1 M in THF) was added, and the reaction mixture was stirred at rt for 3 days. After the starting material had been completely consumed, 1 M HCl (5 mL) and EtOAc (5 mL) were added, and the mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 0.75 g (75%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15–7.07 (m, 2 H), 6.88 (t, J = 7.4 Hz, 1 H), 6.78 (d, J = 7.8 Hz, 1 H), 4.97 (s, 1 H), 4.91 (s, 1 H), 2.95 (dd, J = 5.4, 13.6 Hz, 1 H), 2.78–2.70 (m, 1 H), 2.57 (dd, J = 8.9, 13.6 Hz, 1 H), 2.41–2.34 (m, 2 H), 1.79–1.65 (m, 3 H), 1.57–1.49 (m, 1 H), 1.45–1.30 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 153.7, 131.2, 127.3, 120.6, 115.5, 105.0, 44.0, 35.1, 33.3, 32.4, 24.1, one peak is missing due to incidental equivalence; IR (film, cm^{-1}) 3435, 2928, 1456; MS(EI): 188.1201 (188.1197 calcd for $C_{13}H_{16}O$, M^+).

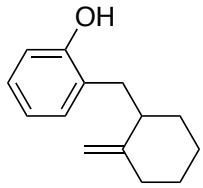


2-[2-(*tert*-Butyldimethylsiloxy)benzyl]cyclohexanone (S5). General Procedure 1 was used for the conversion of 2-cyclohexylidene-1,1-dimethylhydrazine (0.23 g, 1.66 mmol) and 2-(bromomethylphenoxy)(*tert*-butyl)dimethylsilane (0.5 g, 1.66 mmol) to the title compound. This procedure afforded 0.36 g (68%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13–7.04 (m, 2 H), 6.86 (dt, J = 1.2, 7.4 Hz, 1 H), 6.77 (dd, J = 0.2, 8.0 Hz, 1 H), 3.21 (dd, J = 4.5, 9.2 Hz, 1 H), 2.71–2.61 (m, 1 H), 2.45–2.23 (m, 3 H), 2.10–1.94 (m, 2 H), 1.85–1.77 (m, 1 H), 1.73–1.49 (m, 3 H), 1.41–1.21 (m, 2 H), 0.98 (s, 9 H), 0.24 (s, 3 H), 0.21 (s, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.9, 153.8, 131.6, 130.8, 127.0, 120.8, 118.3, 50.7, 42.2, 33.6, 30.7, 28.2, 25.7, 25.2, 18.2, 0.2, 0.0; IR (film, cm^{-1}) 2931, 1711, 1253; MS(ESI): 319.2088 (319.2088 calcd for $C_{19}H_{30}O_2Si$, $M + H^+$).

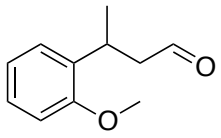


***tert*-Butyldimethyl{2-[(2-methylenecyclohexyl)methyl]phenoxy}silane (S6).** General Procedure 2 was used for the conversion of 2-[2-(*tert*-butyldimethylsiloxy)benzyl]cyclohexanone (1.4 g, 4.41 mmol) to the title compound. This procedure afforded 0.86 g (64%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.90–6.84 (m, 2 H), 6.68–6.62 (m, 1 H), 6.59 (d, J = 7.8 Hz, 1 H), 4.48 (s, 1 H), 4.41 (s, 1 H), 2.78 (dd, J = 5.4, 13.4 Hz, 1 H), 2.32 (dd, J = 6.2, 13.2 Hz, 1 H), 2.21–2.10 (m, 2 H), 1.86–1.78 (m, 1 H), 1.55–1.41 (m, 3 H), 1.30–1.22 (m, 1 H), 1.17–1.05 (m, 1 H), 1.00–0.92 (m, 1 H), 0.81 (s, 9 H), 0.05 (s, 3 H), 0.02 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 157.0, 135.6, 135.1, 130.6, 124.6, 122.3, 109.2, 46.7, 39.6, 37.4, 35.5, 29.7, 29.6, 28.9, 22.1, 0.2, 0.0; IR (film, cm^{-1}) 2930, 1598, 1252; MS(EI): 316.2225 (316.2222 calcd for $C_{20}H_{32}OSi$, M^+).



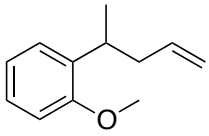
2-[(2-Methylenecyclohexyl)methyl]phenol (12). An oven-dried flask equipped with a magnetic stirbar was cooled under a stream of nitrogen and charged with *tert*-butyldimethyl{2-[(2-

methylenecyclohexyl)methyl]phenoxy}silane (0.57 g, 1.8 mmol). The flask was cooled to 0 °C and TBAF (3 equiv, 1 M in THF) was added. The resulting mixture was warmed to rt and was stirred for 2 h until the starting material had been completely consumed as judged by tlc analysis. A solution of 1 M HCl (5 mL) and EtOAc (5 mL) were added, and the resulting mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel 0.37 g (73%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10–7.05 (m, 2 H), 6.85 (dt, *J* = 1.0, 8.2 Hz, 1 H), 6.79–6.76 (m, 1 H), 5.51 (s, 1 H), 4.70 (s, 1 H), 4.64 (s, 1 H), 2.97 (dd, *J* = 5.3, 13.4 Hz, 1 H), 2.58 (dd, *J* = 9.3, 13.7 Hz, 1 H), 2.42–2.31 (m, 2 H), 2.10–2.05 (m, 1 H), 1.75–1.61 (m, 3 H), 1.54–1.34 (m, 2 H), 1.28–1.17 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 153.1, 131.2, 127.2, 127.1, 120.4, 115.3, 105.6, 43.0, 35.4, 33.1, 33.0, 28.7, 24.7; IR (film, cm^{-1}) 3435, 1507, 1229; MS(ESI): 203.1427 (203.1430 calcd for $C_{14}H_{18}O$, M + H^+).

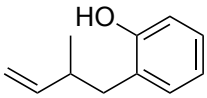


3-(2-Methoxyphenyl)butanal (S7). An oven-dried flask equipped with a magnetic stirbar was cooled under a stream of nitrogen and charged with sodium bicarbonate (0.89 g, 10.7 mmol), palladium acetate (19.2 mg, 0.09 mmol) and tetrabutylammonium chloride (1.19 g, 4.3 mmol). DMF (10 mL), 2-iodoanisole (1.0 g, 4.27 mmol), and 2-methylprop-2-en-1-ol (0.46 mL, 6.4 mmol) were added, and the reaction mixture was heated to 85 °C with stirring for 12 h. TLC analysis indicated the reaction had not proceeded to completion, so a second portion of palladium acetate (19.2 mg, 0.09 mmol) was added, and the reaction mixture was heated to 85 °C

with stirring for an additional 12 h. At this time TLC analysis indicated the starting material had been completely consumed. The mixture was cooled to rt, saturated aqueous NH_4Cl (5 mL) and ether (5 mL) were added, and the resulting mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with ether (2 x 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel 0.61 g (85%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.68 (s, 1 H), 7.20 (dt, *J* = 1.8, 8.9 Hz, 1 H), 7.10 (dd, *J* = 1.4, 7.4 Hz, 1 H), 6.88 (dt, *J* = 1.0, 7.4 Hz, 1 H), 6.84 (d, *J* = 6.8 Hz, 1 H), 3.78 (s, 3 H), 3.07 (dd, *J* = 6.4, 13.1 Hz, 1 H), 2.71 (m, 1 H), 2.63 (dd, *J* = 7.4, 13.1 Hz, 1 H), 1.04 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 157.4, 130.9, 127.8, 127.0, 120.4, 110.3, 55.1, 46.4, 31.7, 13.3; IR (film, cm^{-1}) 2963, 1718, 1245; MS(EI): 178.0994 (178.0994 calcd for $C_{11}H_{14}O_2$, M^+).



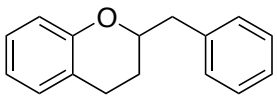
1-Methoxy-2-(pent-4-en-2-yl)benzene (S8). General Procedure 2 was used for the conversion of 3-(2-methoxyphenyl)butanal (0.47 g, 2.62 mmol) to the title compound. This procedure afforded 0.43 g (93%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.41 (m, 1 H), 7.20 (dt, *J* = 1.8, 8.0 Hz, 1 H), 7.19 (dd, *J* = 1.6, 7.2 Hz, 1 H), 7.00–6.91 (m, 1 H), 5.98–5.88 (m, 1 H), 5.07–4.98 (m, 2 H), 3.88 (s, 3 H), 2.84–2.76 (m, 1 H), 2.70–2.58 (m, 2 H), 1.10 (d, *J* = 6.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 144.6, 131.0, 129.3, 128.6, 127.1, 120.2, 112.2, 55.2, 37.8, 37.5, 19.6; IR (film, cm^{-1}) 2962, 1495, 1243; MS(EI): 176.1201 (176.1201 calcd for $C_{12}H_{16}O$, M^+).



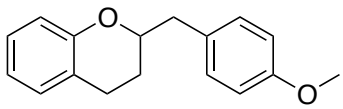
2-(Pent-4-en-2-yl)phenol (13). General Procedure 3 was used for the conversion of 1-methoxy-2-(pent-4-en-2-yl)benzene (0.16 g, 0.91 mmol) to the title compound. This procedure afforded 0.10 g (75%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10–7.04 (m, 2 H), 6.87–6.82 (m, 1 H), 6.74 (d, J = 8.7 Hz, 1 H), 5.89–5.75 (m, 1 H), 5.00–4.90 (m, 2 H), 4.78 (s, 1 H), 2.69–2.60 (m, 1 H), 2.59–2.48 (m, 2 H), 1.02 (d, J = 6.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.8, 144.2, 131.5, 127.3, 127.1, 120.6, 115.6, 113.0, 38.0, 37.4, 19.6; IR (film, cm^{-1}) 3367, 2974, 1456; MS(EI): 162.1042 (162.1045 calcd for $C_{11}H_{14}O$, M^+).

Synthesis of Benzopyrans via Pd-Catalyzed Alkene Carboetherification

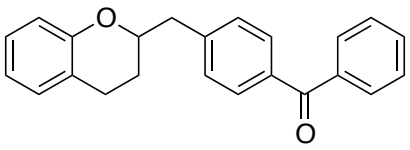
General Procedure 4: Palladium-Catalyzed Carboetherification Reactions. An oven or flame-dried Schlenk tube was cooled under a stream of nitrogen and charged with $Pd_2(dba)_3$ (2 mol % complex, 4 mol % Pd), S-Phos (4 mol %), NaO^tBu (2.0 equiv), and the aryl bromide (2.0 equiv). The tube was purged with nitrogen and the phenol substrate (1.0 equiv), and toluene (0.25 M substrate concentration) were added. The mixture was heated to 110 °C with stirring until the starting material had been consumed as judged by GC or ^{1}H NMR analysis. The mixture was cooled to room temperature, quenched with saturated aqueous NH_4Cl (2 mL), and diluted with ethyl acetate (10 mL). The layers were separated and the aqueous layer was extracted with ethyl acetate (3 X 10 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel.



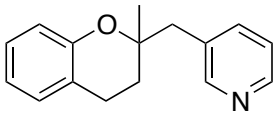
(±)-2-Benzylchroman (**8**). The coupling of 2-(but-3-en-1-yl)phenol (30 mg, 0.20 mmol) with bromobenzene (0.43 μL, 0.40 mmol) was conducted following General Procedure 4. This procedure afforded 37 mg (83%) of the title compound as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.19 (m, 5 H), 7.09–6.97 (m, 2 H), 6.83–6.75 (m, 2 H), 4.23–4.15 (m, 1 H), 3.12 (dd, *J* = 7.6, 13.6 Hz, 1 H), 2.85 (dd, *J* = 7.0, 13.6 Hz, 1 H), 2.78–2.68 (m, 2 H), 2.00–1.91 (m, 1 H), 1.74–1.62 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 137.8, 129.6, 129.5, 128.3, 127.1, 126.4, 121.9, 120.0, 116.7, 76.5, 41.8, 26.5, 24.5; IR (film, cm^{-1}) 2924, 1456, 1236; MS(EI): 224.1207 (224.1201 calcd for $C_{16}H_{16}O$, M^+).



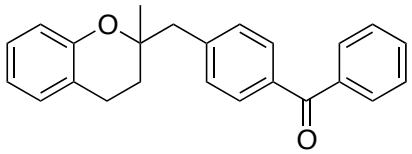
(±)-2-(4-Methoxybenzyl)chroman (**14**). The coupling of 2-(but-3-en-1-yl)phenol (25 mg, 0.17 mmol) with 4-bromoanisole (40 μL, 0.34 mmol) was conducted following General Procedure 4. This procedure afforded 28 mg (66%) of the title compound as an orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.15 (m, 2 H), 7.06 (t, *J* = 7.4 Hz, 1 H), 7.01 (d, *J* = 7.0 Hz, 1 H), 6.86–6.75 (m, 4 H), 4.19–4.11 (m, 1 H), 3.78 (s, 3 H), 3.07 (dd, *J* = 6.1, 13.9 Hz, 1 H), 2.83–2.70 (m, 3 H), 2.00–1.92 (m, 1 H), 1.73–1.63 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.2, 130.5, 129.9, 129.5, 127.2, 120.0, 116.7, 115.7, 113.8, 76.7, 55.4, 55.2, 40.9, 26.4, 24.6; IR (film, cm^{-1}) 2929, 1488, 1247; MS(EI): 254.1307 (254.1313 calcd for $C_{16}H_{16}O$, M^+).



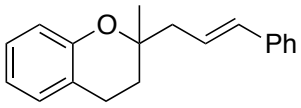
(±)-[4-(Chroman-2-ylmethyl)phenyl](phenyl)methanone (**15**). The coupling of 2-(but-3-en-1-yl)phenol (20 mg, 0.13 mmol) with 4-bromobenzophenone (70 mg, 0.26 mmol) was conducted following General Procedure 4. This procedure afforded 25 mg (56%) of the title compound as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84–7.78 (m, 4 H), 7.63–7.58 (m, 1 H), 7.51 (t, *J* = 7.8 Hz, 2 H), 7.42 (d, *J* = 8.0 Hz, 2 H), 7.11 (t, *J* = 7.3 Hz, 1 H), 7.06 (d, *J* = 7.6 Hz, 1 H), 6.88–6.81 (m, 2 H), 4.33–4.27 (m, 1 H), 3.21 (dd, *J* = 6.6, 13.9 Hz, 1 H), 3.01 (dd, *J* = 6.1, 13.7 Hz, 1 H), 2.90–2.75 (m, 2 H), 2.07–2.00 (m, 1 H), 1.83–1.73 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 154.6, 143.0, 137.8, 135.8, 132.3, 130.2, 130.0, 129.5, 128.3, 127.3, 121.8, 120.2, 116.8, 76.0, 41.8, 26.8, 24.5, 18.5; IR (film, cm^{-1}) 2918, 1616, 1457; MS(ESI): 329.1537 (329.1536 calcd for $C_{23}H_{20}O_2$, M + H^+).



(±)-3-[(2-Methylchroman-2-yl)methyl]pyridine (16). The coupling of 2-(3-methylbut-3-en-1-yl)phenol (20 mg, 0.12 mmol) with 3-bromopyridine (23.7 μL, 0.24 mmol) was conducted following General Procedure 4. This procedure afforded 24 mg (81%) of the title compound as an amber oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16–7.07 (m, 4 H), 6.88–6.84 (m, 2 H), 6.80–6.77 (m, 2 H), 2.94 (d, *J* = 13.6 Hz, 1 H), 2.89–2.78 (m, 3 H), 1.87–1.74 (m, 2 H), 1.24 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.5, 131.5, 129.9, 129.5, 127.3, 119.7, 119.5, 117.4, 76.3, 45.1, 30.8, 25.7, 24.3, 22.1, 18.4, 18.2; IR (film, cm^{-1}) 2928, 1581, 1455, 1243; MS(ESI): 240.1386 (240.1383 calcd for $C_{16}H_{17}NO$, M + H^+).

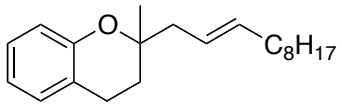


(±)-{4-[(2-Methylchroman-2-yl)methyl]phenyl}(phenyl)methanone (**17**). The coupling of 2-(3-methylbut-3-en-1-yl)phenol (20.0 mg, 0.12 mmol) with 4-bromobenzophenone (70.0 mg, 0.24 mmol) was conducted following General Procedure 4. This procedure afforded 35 mg (83%) of the title compound as an orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81–7.77 (m, 2 H), 7.75–7.71 (m, 2 H), 7.60–7.53 (m, 1 H), 7.49–7.44 (m, 2 H), 7.36–7.32 (m, 2 H), 7.13–7.04 (m, 2 H), 6.86-6.81 (m, 2 H), 3.07 (d, *J* = 13.3 Hz, 1 H), 2.90 (d, *J* = 13.5 Hz, 1 H), 2.82 (t, *J* = 6.6 Hz, 2 H), 1.83 (t, *J* = 6.8 Hz, 2 H), 1.26 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 153.6, 142.4, 137.8, 135.7, 132.3, 130.6, 130.0, 129.9, 129.6, 128.2, 127.4, 120.8, 119.9, 117.3, 75.9, 45.7, 31.2, 24.5, 22.1; IR (film, cm^{-1}) 2927, 1653, 1278; MS(EI): 343.1697 (343.1693 calcd for $C_{24}H_{22}O_2$, M + H^+).

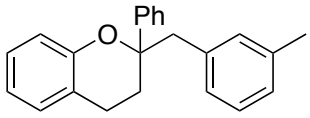


(±)-2-Cinnamyl-2-methylchroman (**18**). The coupling of 2-(3-methylbut-3-en-1-yl)phenol (20 mg, 0.12 mmol) with (*E*)-β-bromostyrene (30 μL, 0.24 mmol) was conducted following General Procedure 4. This procedure afforded 28 mg (88%) of the title compound as an orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.27 (m, 4 H), 7.26–7.18 (m, 2 H), 7.13–7.02 (m, 2 H), 6.86–6.79 (m, 1 H), 6.45 (d, *J* = 15.8 Hz, 1 H), 6.33–6.24 (m, 1 H), 2.79 (t, *J* = 6.8 Hz, 2 H), 2.56–2.50 (m, 2 H), 1.94–1.85 (m, 1 H), 1.83–1.74 (m, 1 H), 1.33 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

153.8, 137.4, 133.2, 129.4, 128.5, 127.3, 127.2, 126.1, 125.2, 121.0, 119.8, 117.3, 76.1, 43.4, 30.8, 24.6, 22.1; IR (film, cm^{-1}) 2929, 1581, 1453; MS(EI): 264.1512 (264.1514 calcd for $C_{19}H_{20}O$, M^+).

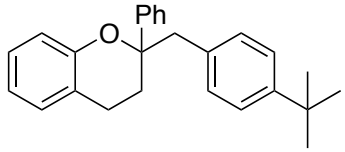


(±)-(*E*)-2-Methyl-2-(undec-2-en-1-yl)chroman (**19**). The coupling of 2-(3-methylbut-3-en-1-yl)phenol (20 mg, 0.12 mmol) with (*E*)-1-bromodec-1-ene (30 μL, 0.54 mmol) was conducted following General Procedure 4. This procedure afforded 37 mg (80%) of the title compound as an amber oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09–6.99 (m, 2 H), 6.81–6.73 (m, 2 H), 5.56–5.36 (m, 2 H), 2.76–2.68 (m, 2 H), 2.37–2.25 (m, 2 H), 1.99 (pent, J = 6.9 Hz, 2 H), 1.87–1.64 (m, 2 H), 1.35–1.17 (m, 16 H), 0.85 (t, J = 6.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 134.5, 133.0, 129.4, 127.2, 123.7, 121.1, 117.2, 76.0, 42.8, 37.3, 32.6, 31.8, 30.6, 30.4, 29.4, 29.3, 27.4, 24.4, 22.6, 14.1; IR (film, cm^{-1}) 2926, 1653, 1456; MS(EI): 300.2453 (300.2453 calcd for $C_{21}H_{32}O$, M^+).

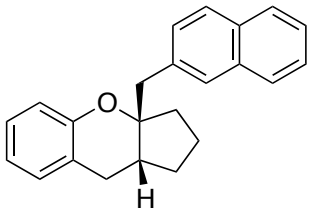


(±)-2-(3-Methylbenzyl)-2-phenylchroman (**20**). The coupling of 2-(3-phenylbut-3-en-1-yl)phenol (25 mg, 0.11 mmol) with *m*-bromotoluene (24 μL, 0.22 mmol) was conducted following General Procedure 4. This procedure afforded 35 mg (57%) of the title compound as an amber oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.23 (m, 4 H), 7.17–7.08 (m, 3 H), 7.04–6.98 (m, 2 H), 6.92–6.88 (m, 3 H), 6.79 (dt, J = 1.2, 7.3 Hz, 1 H), 3.22 (d, J = 13.4 Hz, 1 H), 3.10 (d,

J = 13.4 Hz, 1 H), 2.63–2.56 (m, 1 H), 2.49–2.38 (m, 2 H), 2.28 (s, 3 H), 2.08-2.00 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.1, 143.8, 137.0, 136.3, 131.8, 129.3, 128.3, 128.1, 128.0, 127.5, 127.3, 127.0, 126.7, 125.8, 121.9, 120.0, 80.7, 50.0, 29.6, 22.3, 21.4; IR (film, cm^{-1}) 3058, 2927, 1237; MS(ESI): 315.1747 (315.1743 calcd for $C_{23}H_{22}O$, $M + H^+$).

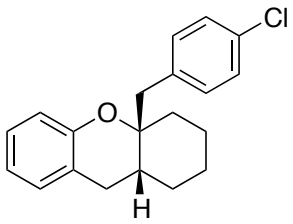


(±)-2-[4-(*tert*-Butyl)benzyl]-2-phenylchroman (**21**). The coupling of 2-(3-phenylbut-3-en-1-yl)phenol (25 mg, 0.11 mmol) with 4-bromo-*tert*-butylbenzene (35 μL, 0.22 mmol) was conducted following General Procedure 4. This procedure afforded 24.3 mg (61%) of the title compound as an amber oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.17 (m, 7 H), 7.15–7.09 (m, 1 H), 7.07–7.01 (m, 3 H), 6.89–6.85 (m, 1 H), 6.76 (dt, *J* = 1.2, 7.4 Hz, 1 H), 3.22 (d, *J* = 13.7 Hz, 1 H), 3.06 (d, *J* = 13.7 Hz, 1 H), 2.60–2.51 (m, 1 H), 2.46–2.35 (m, 2 H), 2.04–1.95 (m, 1 H), 1.29 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.1, 149.0, 144.0, 133.4, 130.5, 129.3, 128.1, 127.2, 126.7, 125.7, 124.6, 121.8, 119.8, 117.0, 80.8, 49.6, 34.3, 31.4, 29.3, 22.3; IR (film, cm^{-1}) 2962, 1490, 1237; MS(ESI): 209.0963 (209.0966 calcd for $C_{26}H_{28}O$, $[M - C_{11}H_{15}]^+$).

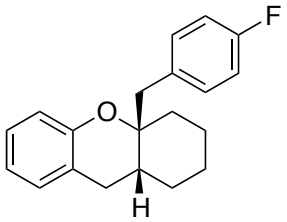


(±)-(3a*S,9a*R**)-3a-(Naphthalen-2-ylmethyl)-1,2,3,3a,9,9a-hexahydrocyclopenta[*b*]chromene** (**22**). The coupling of 2-[(2-methylenecyclopentyl)methyl]phenol (25 mg, 0.13 mmol) with 2-bromonaphthalene (55 mg,

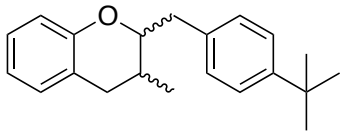
0.26 mmol) was conducted following General Procedure 4. This procedure afforded 25.6 mg (61%) of the title compound as an orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81–7.71 (m, 3 H), 7.61 (s, 1 H), 7.46–7.39 (m, 2 H), 7.36–7.32 (m, 1 H), 7.15 (t, *J* = 7.6 Hz, 1 H), 7.10 (d, *J* = 7.0 Hz, 1 H), 6.90–6.83 (m, 2 H), 3.18 (d, *J* = 13.7 Hz, 1 H), 3.04 (dd, *J* = 6.5, 17.0 Hz, 1 H), 2.83 (d, *J* = 13.7 Hz, 1 H), 2.68 (d, *J* = 16.4 Hz, 1 H), 2.21–2.12 (m, 1 H),1.88–1.64 (m, 4 H), 1.62–1.42 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.3, 135.6, 133.3, 132.2, 130.0, 128.9, 128.6, 127.6, 127.4, 127.3, 125.8, 125.3, 120.2, 120.0, 117.5, 86.1, 69.0, 42.1, 39.9, 36.8, 29.0, 25.4, 20.4; IR (film, cm^{-1}) 2916, 1456, 1231; MS(EI): 314.1666 (314.1671 calcd for $C_{23}H_{22}O$, M^+).



(±)-(4a*S,9a*R**)-4a-(4-Chlorobenzyl)-2,3,4,4a,9,9a-hexahydro-1*H*-xanthene** (**23**). The coupling of 2-[(2-methylenecyclohexyl)methyl]phenol (20 mg, 0.10 mmol) with 4-bromochlorobenzene (38 mg, 0.20 mmol) was conducted following General Procedure 4. This procedure afforded 23 mg (76%) of the title compound as an orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22–7.19 (m, 2 H), 7.14 (t, *J* = 7.4 Hz, 1 H), 7.07 (d, *J* = 7.6 Hz, 1 H), 7.04–6.99 (m, 2 H), 6.89–6.82 (m, 2 H), 3.23 (dd, *J* = 6.3, 16.8 Hz, 1 H), 3.08 (d, *J* = 13.8 Hz, 1 H), 2.47 (d, *J* = 13.7 Hz, 1 H), 2.41 (d, *J* = 17.0 Hz, 1 H), 1.81–1.72 (m, 1 H), 1.68–1.59 (m, 3 H), 1.51–1.41 (m, 2 H), 1.39–1.16 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 132.1, 131.9, 131.8, 130.1, 127.9, 127.4, 120.0, 117.3, 76.4, 42.6, 42.1, 36.0, 34.6, 29.2, 28.6, 25.2, 21.2; IR (film, cm^{-1}) 2920, 1456, 1247; MS(EI): 312.1281 (312.1278 calcd for $C_{20}H_{21}ClO$, M^+).

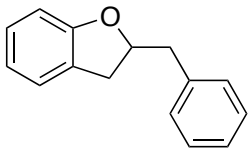


(±)-(4a*S,9a*R**)-4a-(4-Fluorobenzyl)-2,3,4,4a,9,9a-hexahydro-1*H*-xanthene** (**24**). The coupling of 2-[(2-methylenecyclohexyl)methyl]phenol (20 mg, 0.10 mmol) with 4-bromofluorobenzene (22 μL, 0.20 mmol) was conducted following General Procedure 4. This procedure afforded 21 mg (72%) of the title compound as an amber oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (t, *J* = 7.6 Hz, 1 H), 7.10–7.01 (m, 3 H), 6.96–6.90 (m, 2 H), 6.89–6.83 (m, 2 H), 3.24 (dd, *J* = 6.4, 16.8 Hz, 1 H), 3.08 (d, *J* = 13.9 Hz, 1 H), 2.48 (d, *J* = 13.9 Hz, 1 H), 2.41 (d, *J* = 16.8 Hz, 1 H), 1.81–1.73 (m, 1 H), 1.68–1.60 (m, 2 H), 1.51–1.42 (m, 3 H), 1.39–1.30 (m, 1 H), 1.30–1.19 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 152.8, 132.7, 131.9, 131.8 (q, *J* = 254.8 Hz), 129.7, 120.1, 119.9, 117.3, 114.7 (q, *J* = 21.0 Hz), 76.5, 42.4, 36.0, 34.6, 28.7, 25.8, 25.2, 21.2; IR (film, cm^{-1}) 2952, 1736, 1249; MS(EI): 296.1579 (296.1576 calcd for $C_{20}H_{21}FO$, M^{+}).



(±)-2-[4-(*tert*-Butyl)benzyl]-3-methylchroman (**25**). The coupling of 2-(2-methylbut-3-en-1-yl)phenol (30 mg, 0.19 mmol) with 4-bromo-*tert*-butyl benzene (60 μL, 0.37 mmol) was conducted following General Procedure 4. This procedure afforded 0.036 mg (65%) of the title compound as an amber oil. This material was obtained as a ca. 2:1 mixture of diastereomers that contained ca 25% of an unidentified low molecular weight impurity. Data are for the mixture. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.31 (m, 6.60 H), 7.29–7.24 (m, 3.78 H), 7.23–7.19 (m, 1.43

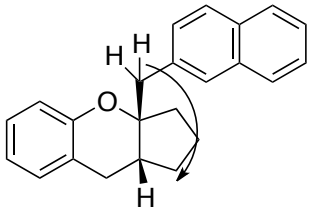
H), 7.13–7.07 (m, 1.90 H), 7.06–7.01 (m, 2.33 H), 6.88–6.76 (m, 4.10 H), 4.71 (d, J = 10.9 Hz, 0.10 H), 4.47 (t, J = 6.4 Hz, 0.55 H), 4.25 (dt, J = 2.2, 8.2 Hz, 0.49 H), 4.18–4.14 (m, 0.63 H), 4.01 (dt, J = 4.3, 7.4 Hz, 1 H), 3.07–2.98 (m, 1.75 H), 2.96–2.76 (m, 3.56 H), 2.53–2.41 (m, 2.43 H), 2.16–2.08 (m, 0.57 H), 1.98–1.86 (m, 1.73 H), 1.36–1.27 (m, 27.84 H), 1.12–1.08 (m, 4.72 H), 1.06 (d, J = 6.8 Hz, 1.52 H), 1.01–0.98 (m, 0.36 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.0, 153.5, 149.1, 148.9, 139.3, 137.9, 135.2, 135.1, 130.1, 129.6, 129.4, 129.3, 129.1, 128.9, 127.1, 127.0, 125.3, 125.2, 125.1, 121.5, 121.4, 121.1, 120.2, 120.0, 119.9, 116.9, 116.6, 82.7, 81.2, 79.4, 51.7, 38.4, 37.1, 34.4, 34.3, 32.9, 31.9, 31.4, 31.3, 31.1, 29.6, 28.2, 27.7, 18.3, 18.1, 13.1; IR (film, cm^{-1}) 2962, 1249; MS(EI): 294.1991 (294.1984 calcd for $C_{21}H_{26}O$, M^+).



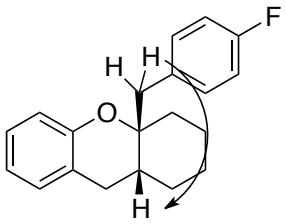
(±)-2-Benzyl-2,3-dihydrobenzofuran (**26**). The coupling of 2-allylphenol (20 mg, 0.15 mmol) with bromobenzene (0.31 μL, 0.30 mmol) was conducted following General Procedure 4. This procedure afforded 14 mg (43%) of the title compound as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.20 (m, 5 H), 7.14–7.07 (m, 2 H), 6.84–6.75 (m, 2 H), 5.04–4.96 (m, 1 H), 3.24–3.14 (m, 2 H), 2.98–2.89 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 129.4, 128.5, 128.0, 126.5, 125.0, 120.2, 109.4, 83.5, 41.9, 34.9, 2 signals are missing due to incidental equivalence; IR (film, cm^{-1}) 2920, 1653, 1456; MS(EI): 210.1049 (210.1045 calcd for $C_{15}H_{14}O$, M^+).

Assignment of Stereochemistry

The relative stereochemistry of **22** was assigned on the basis of signals observed in ^{1}H NMR nOe experiments. Relevant nOe data is shown below.



The relative stereochemistry of **24** was assigned on the basis of signals observed in ^{1}H NMR nOe experiments. Relevant nOe data is shown below. The stereochemistry of compound **23** was assigned based on analogy to **24**.



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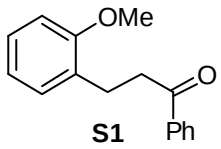

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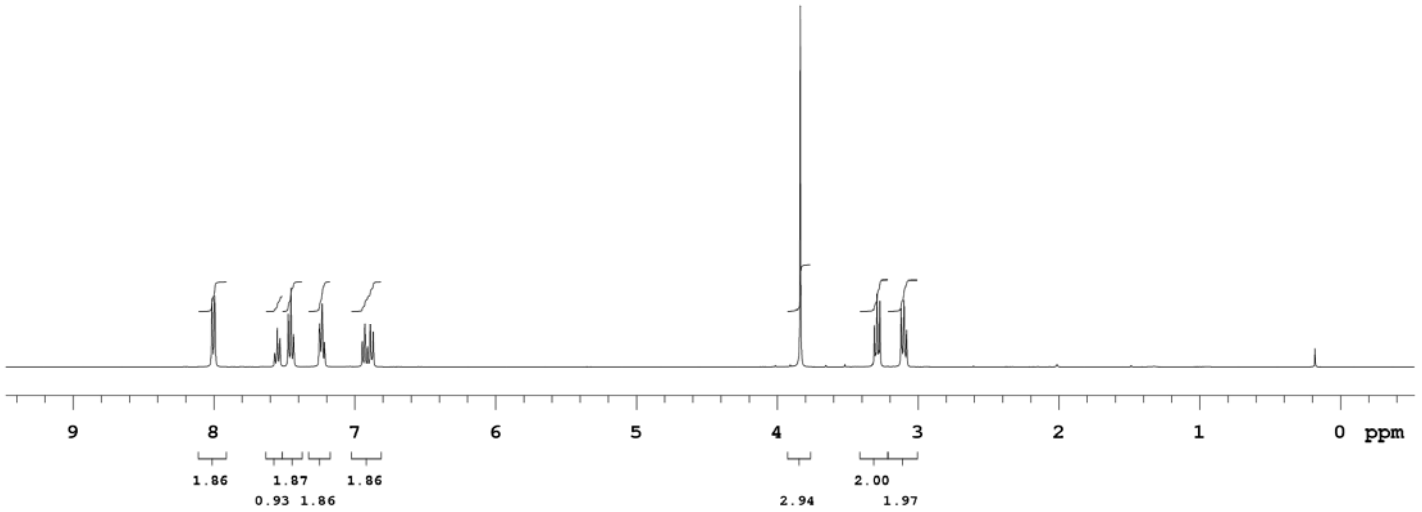

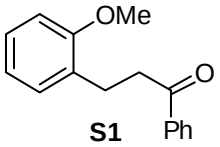

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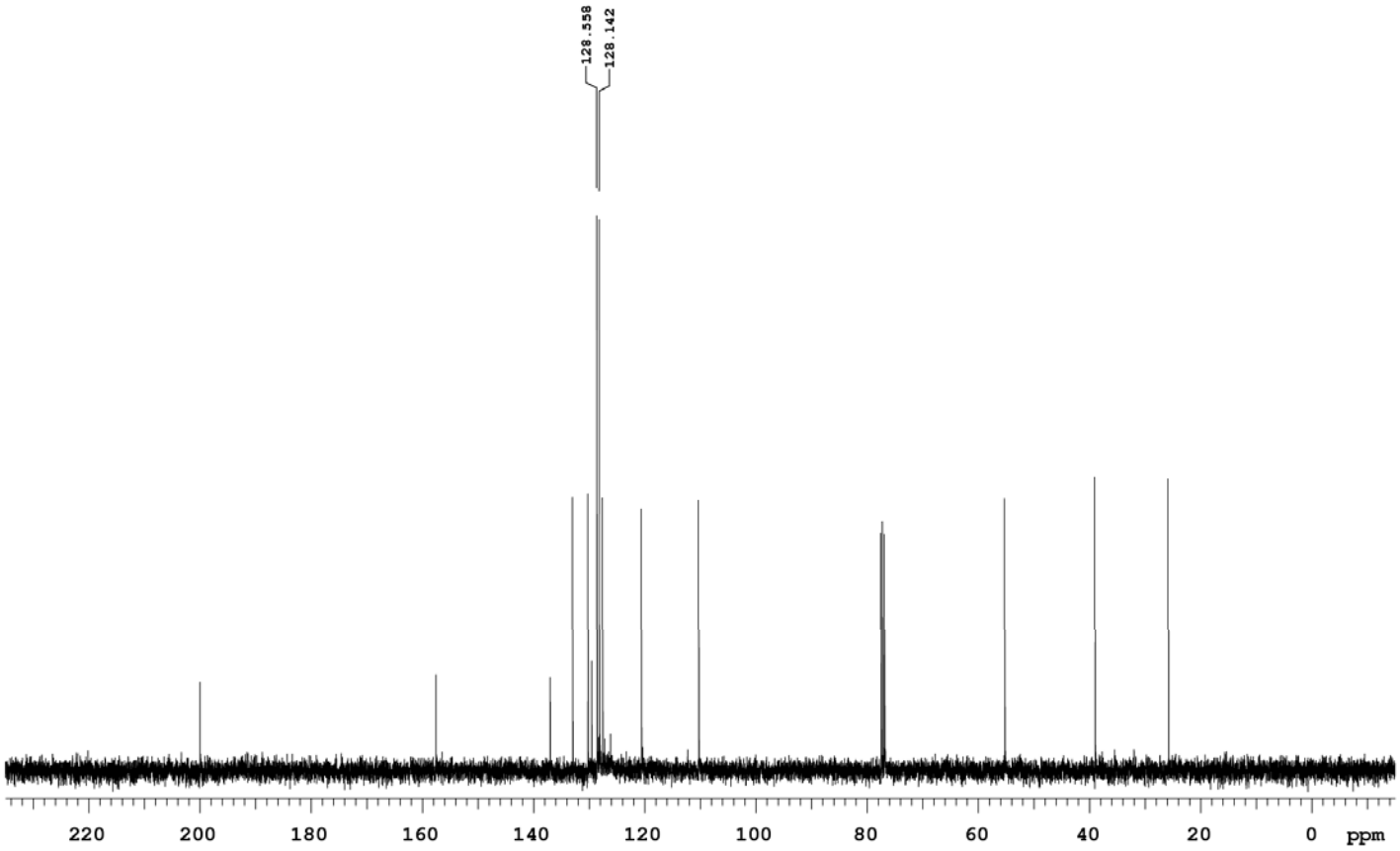

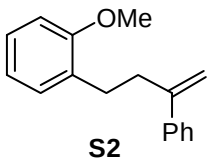

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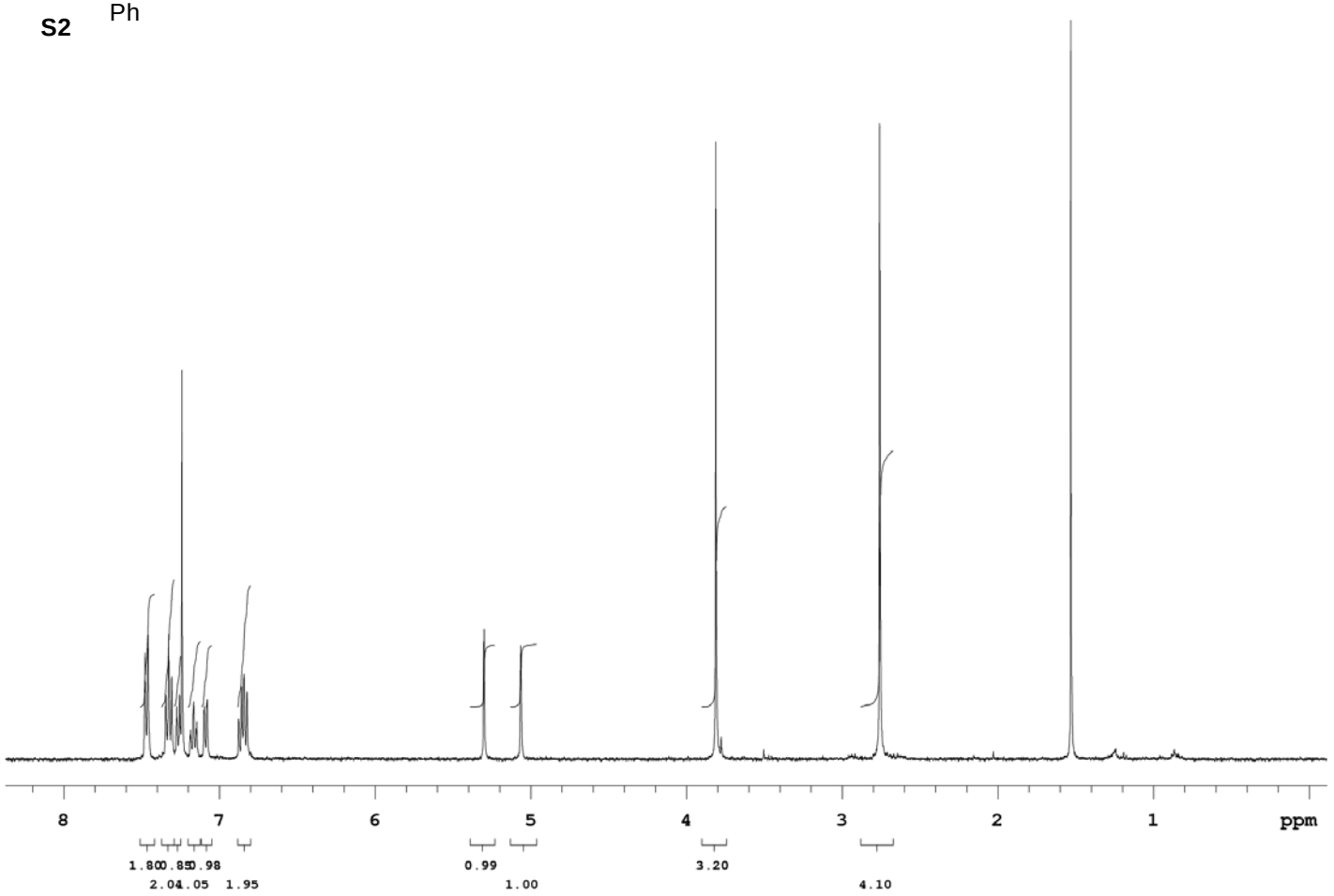

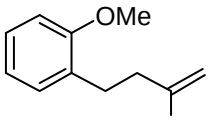

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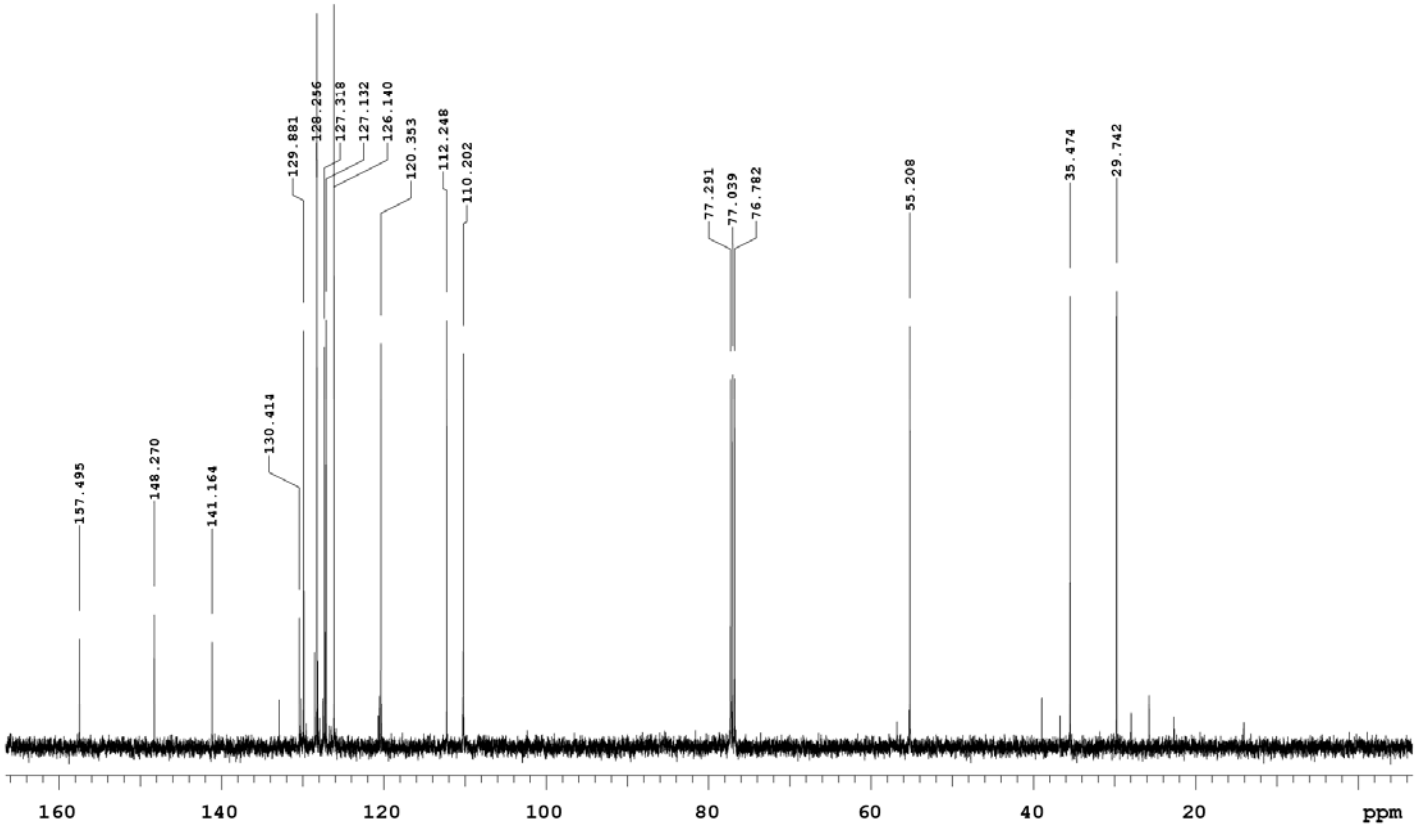

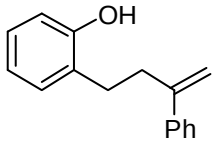

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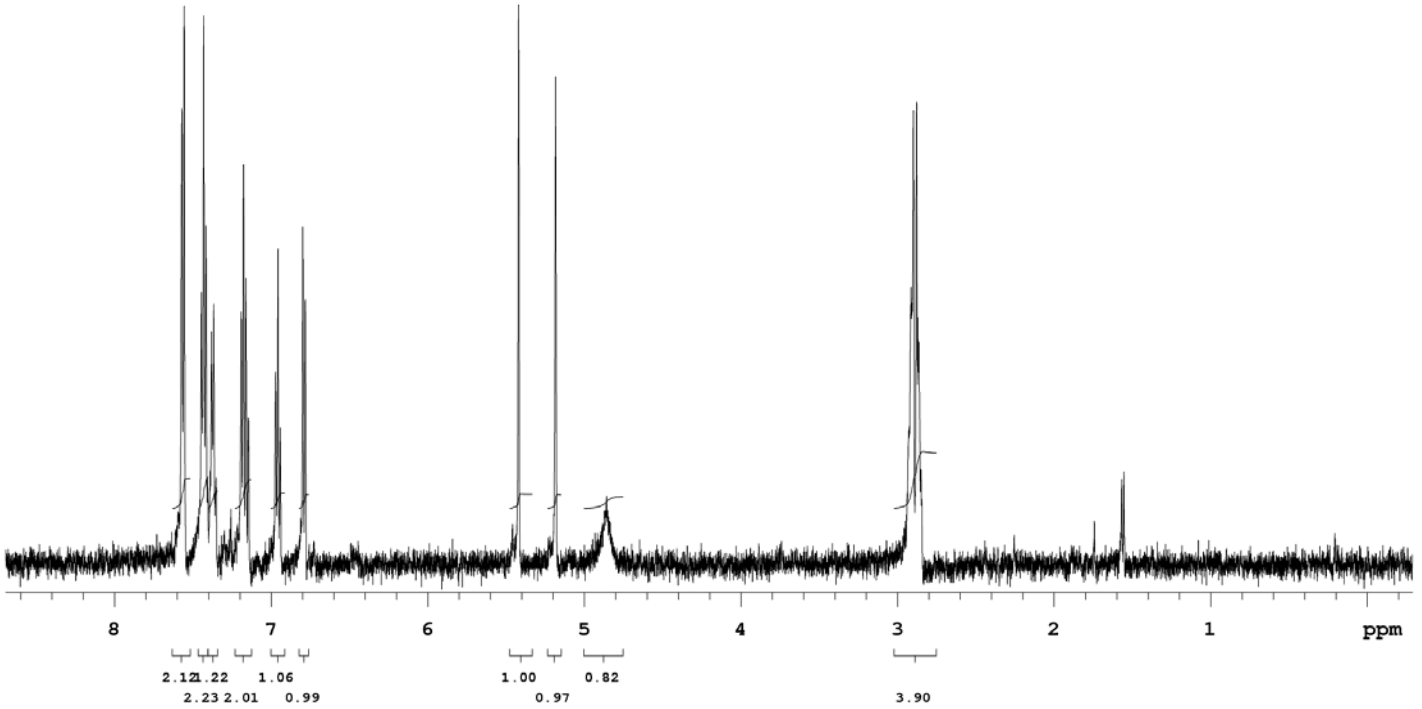

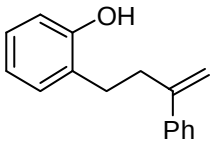

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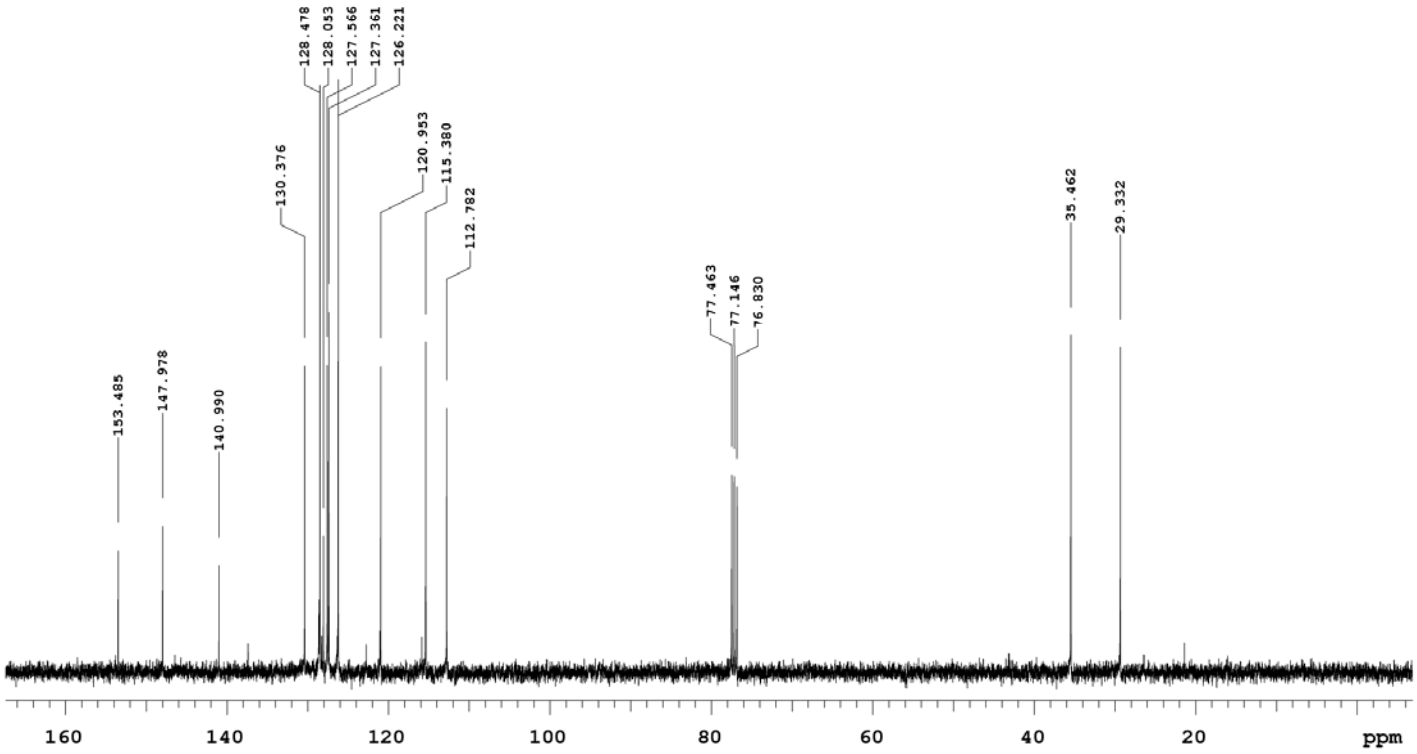

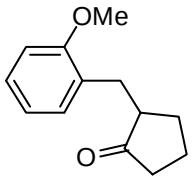

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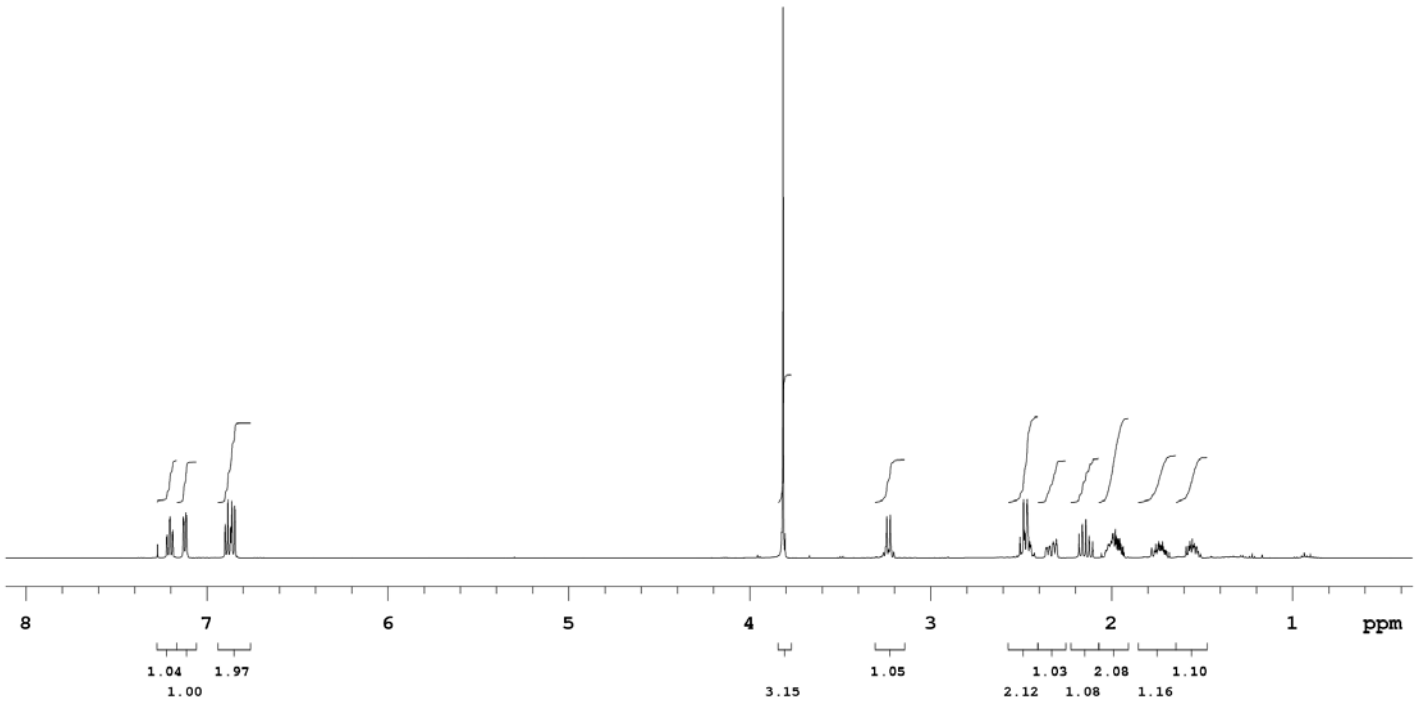

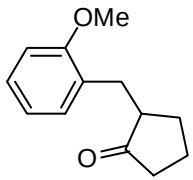

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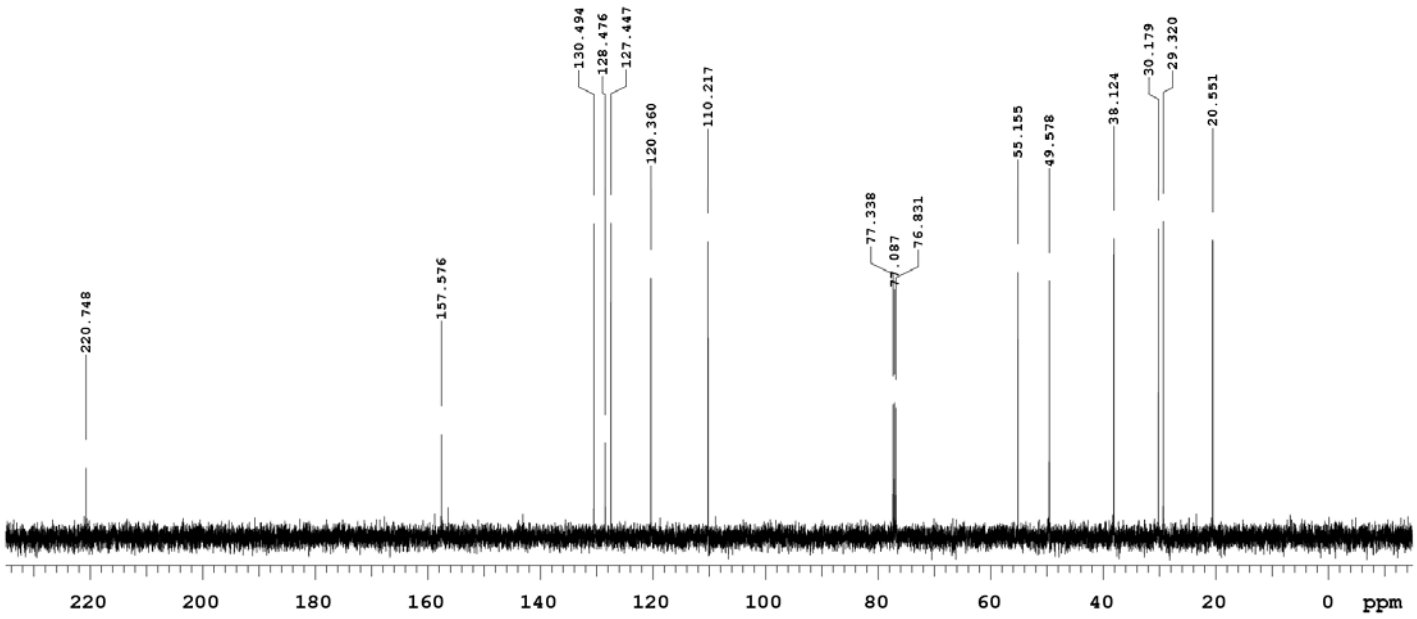

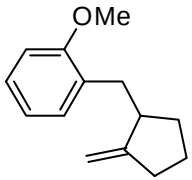

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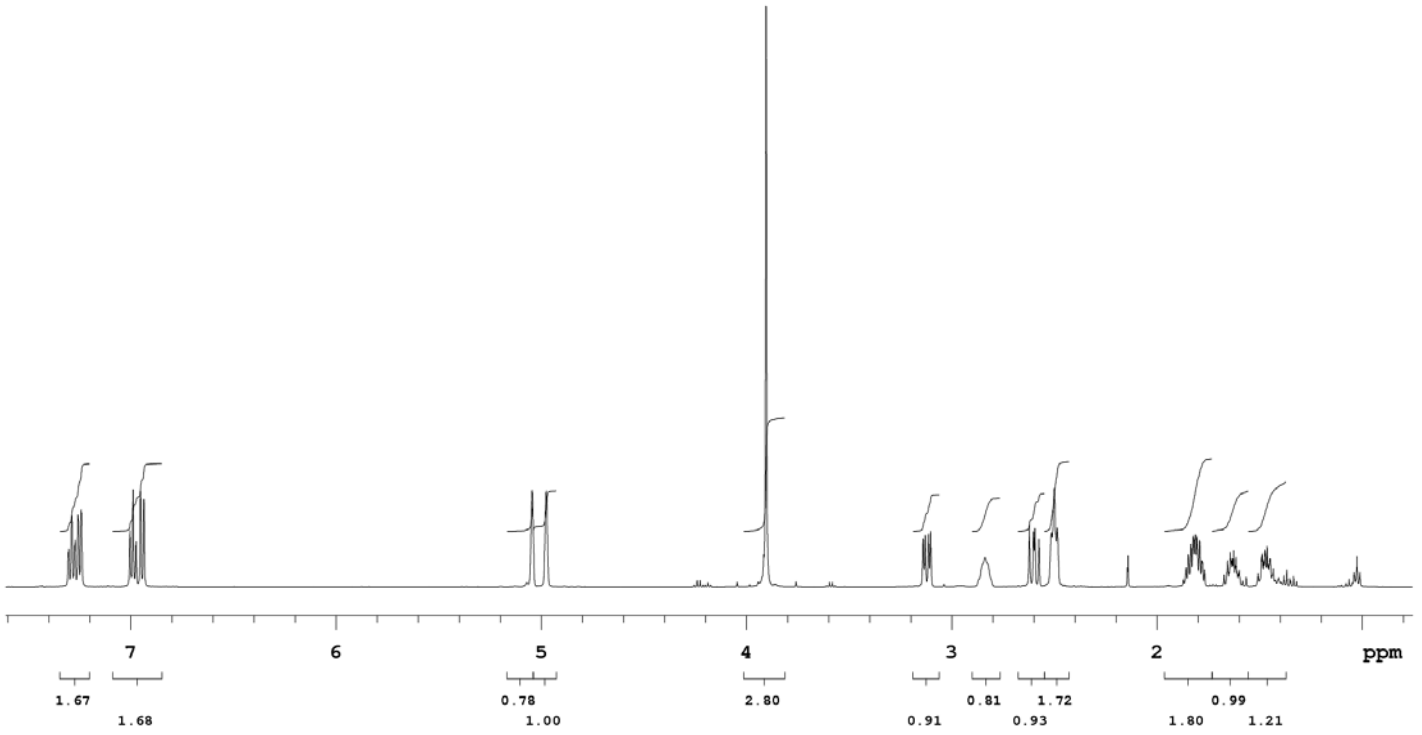

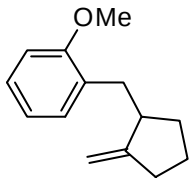

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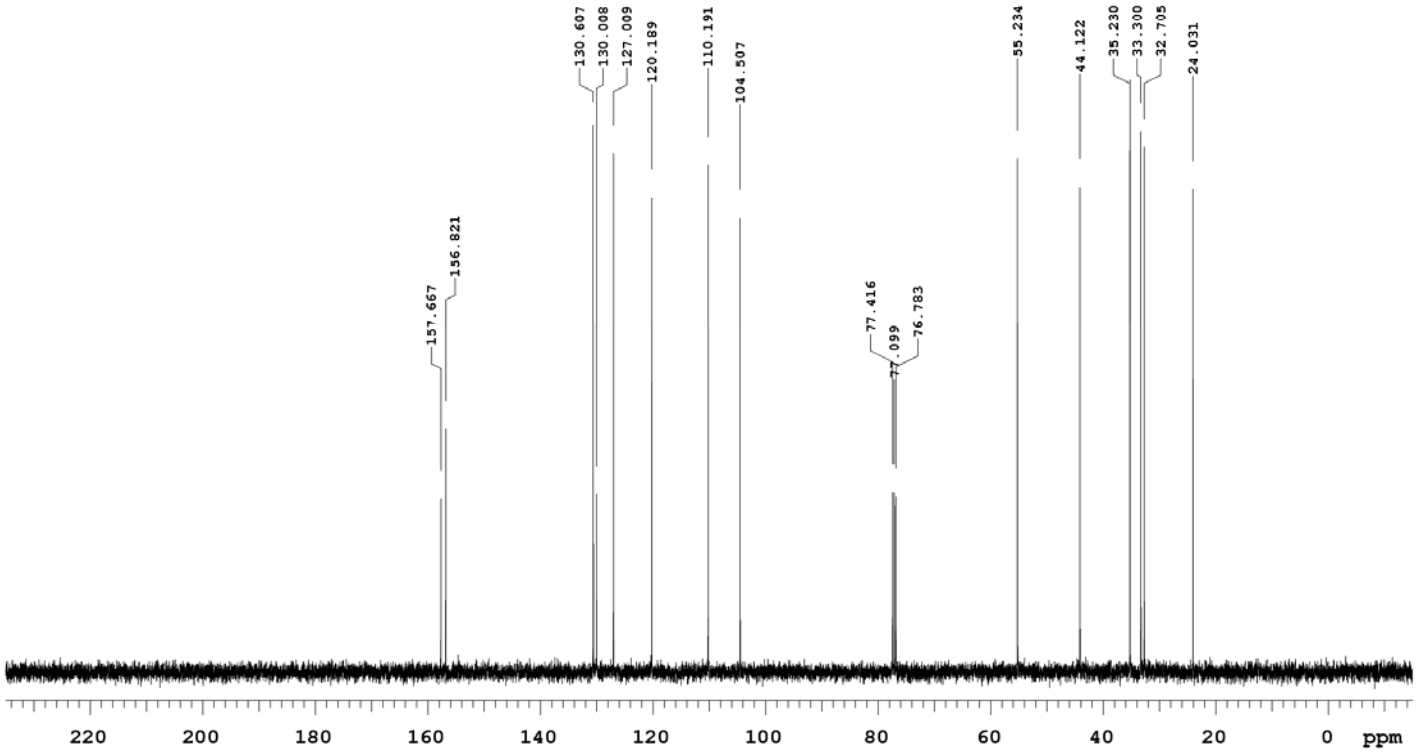

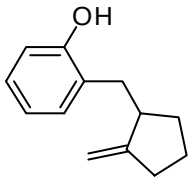

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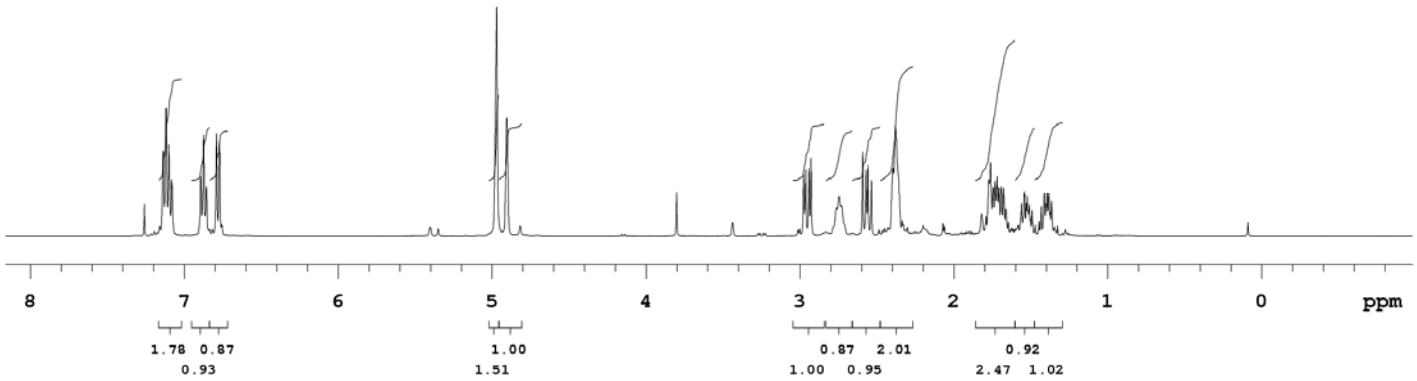

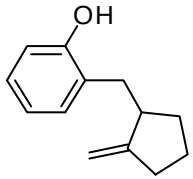

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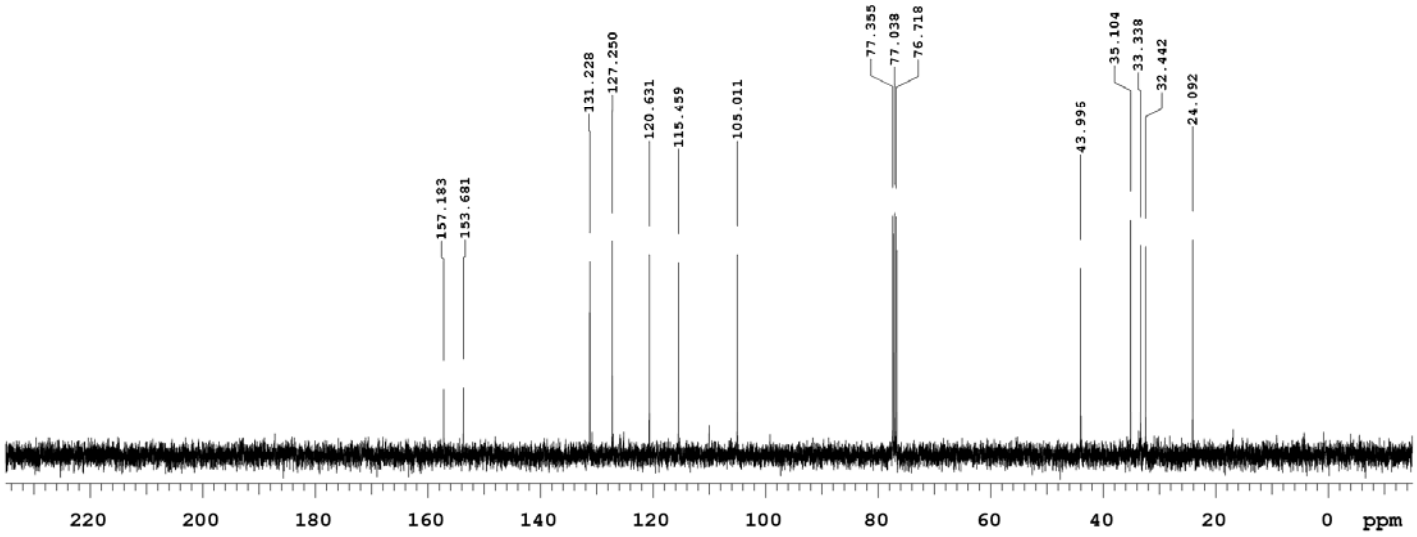

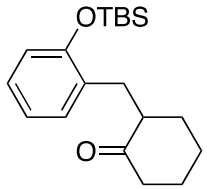

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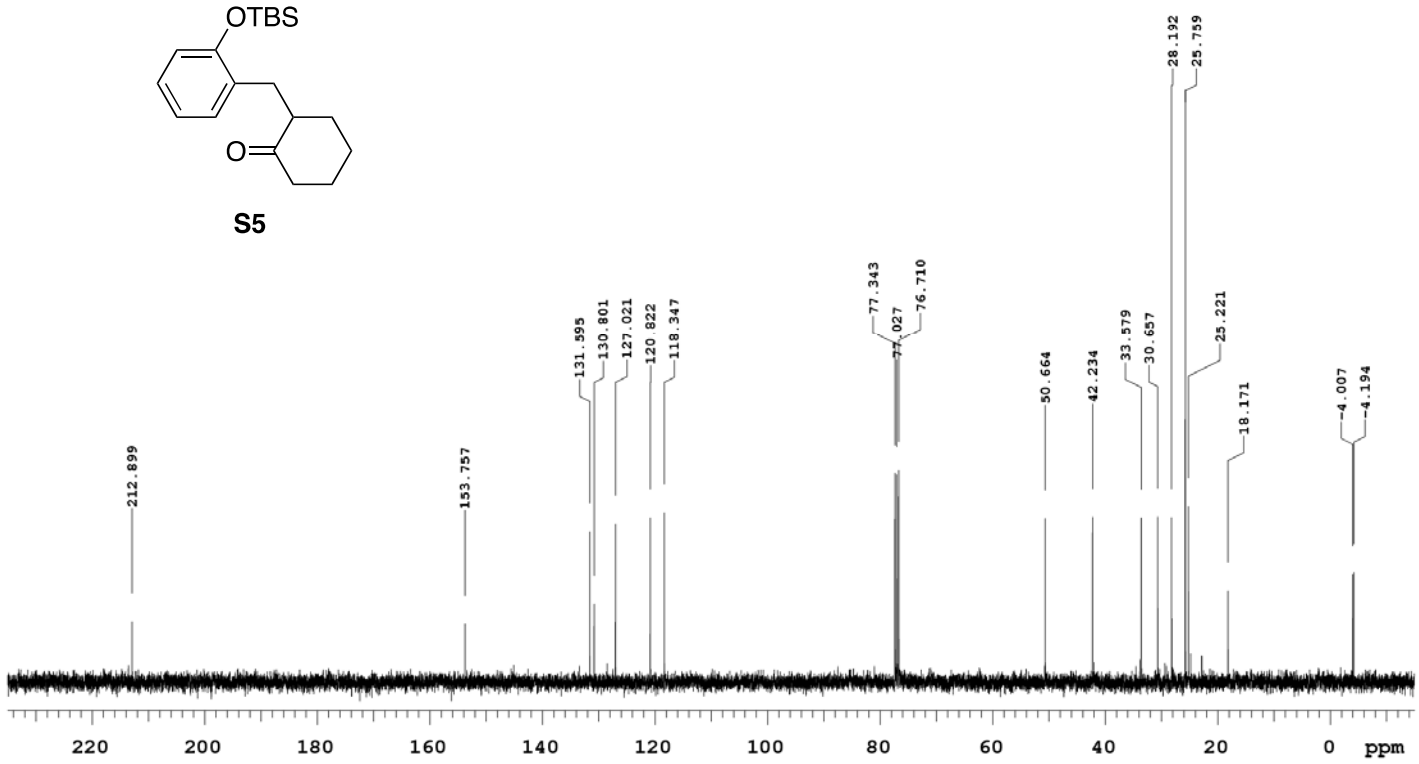

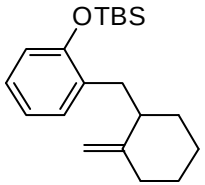

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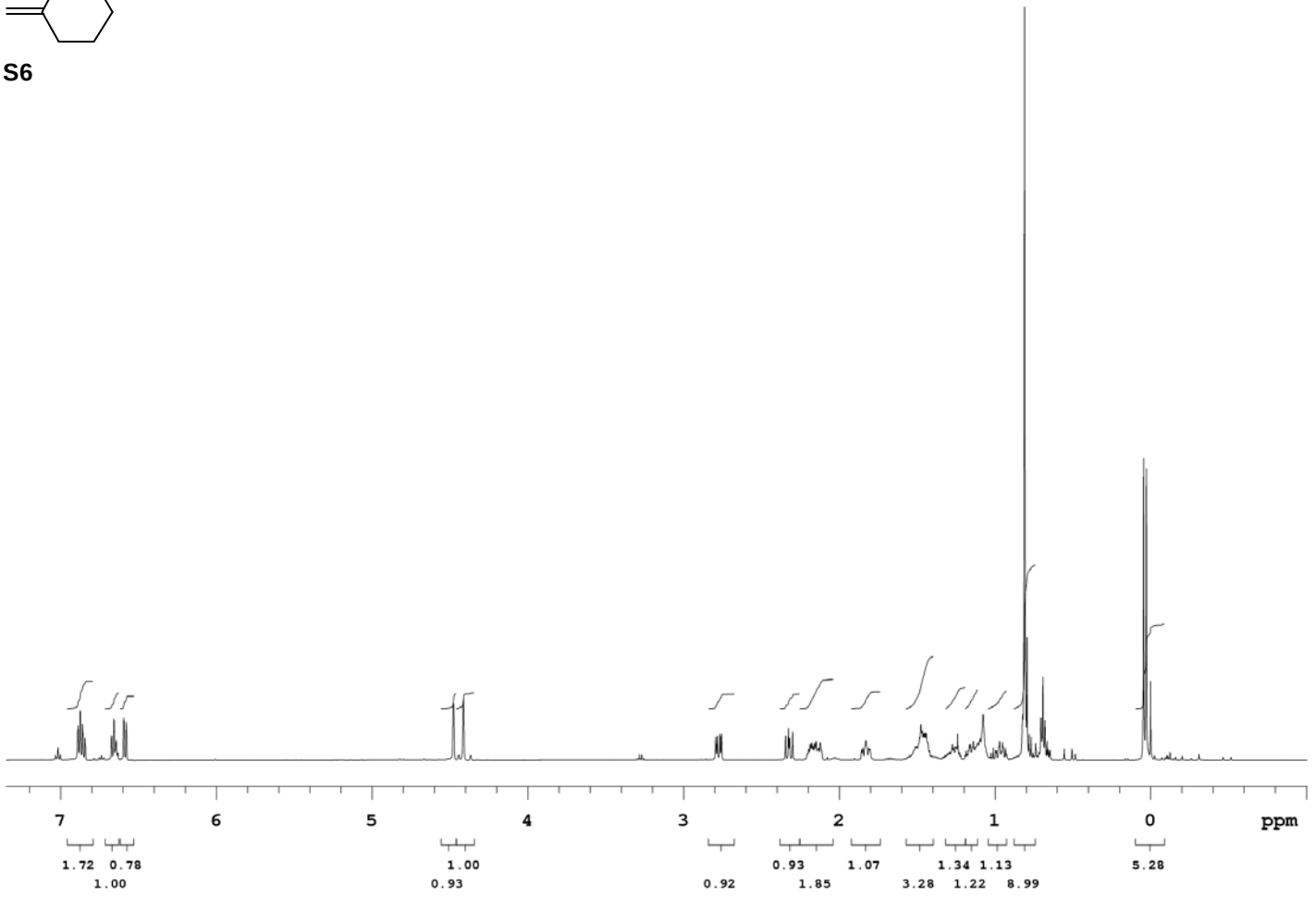

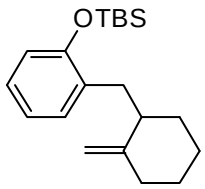

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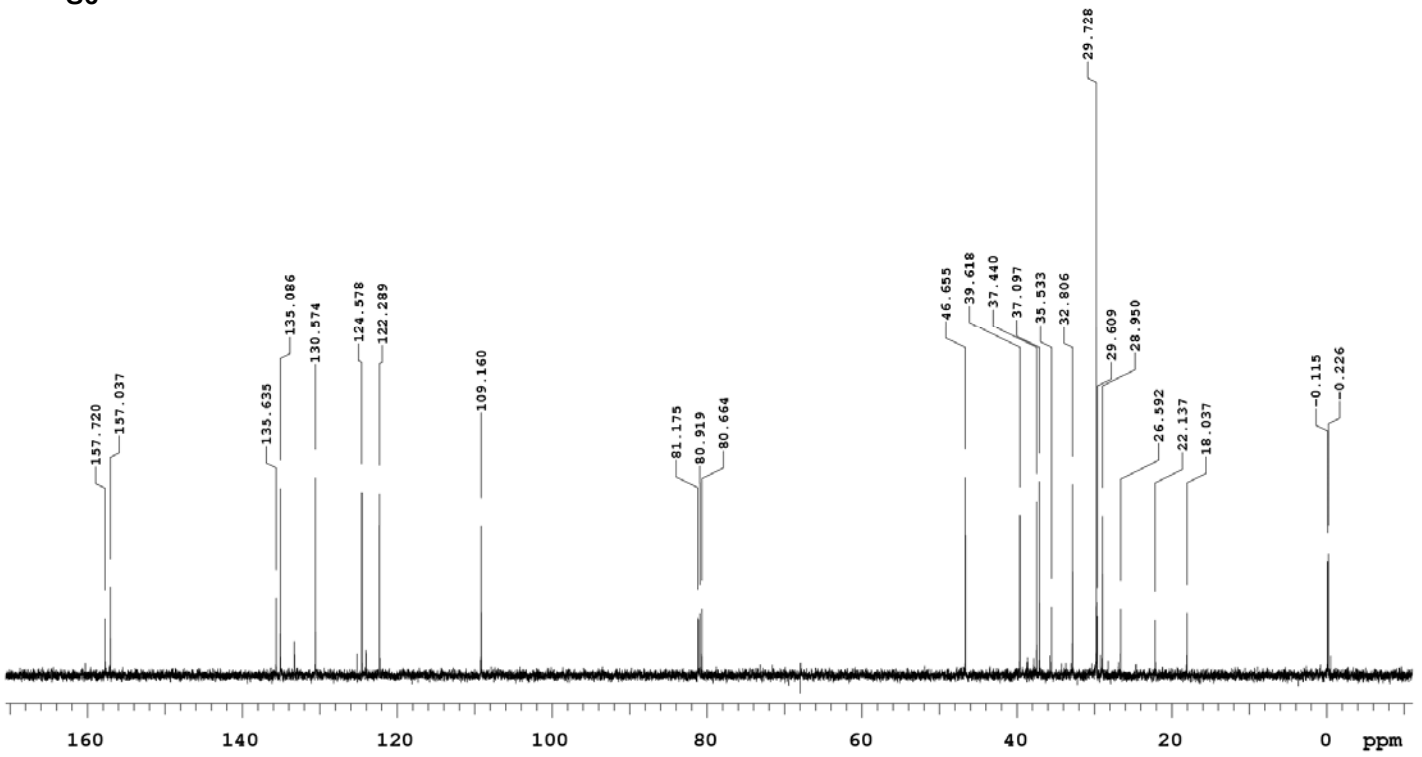

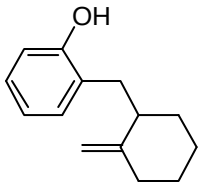

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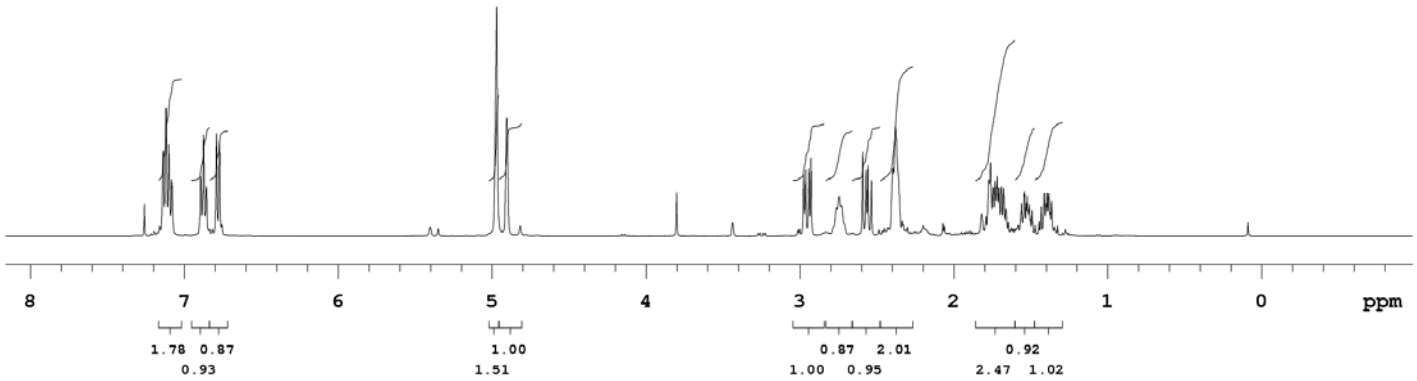

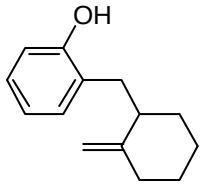

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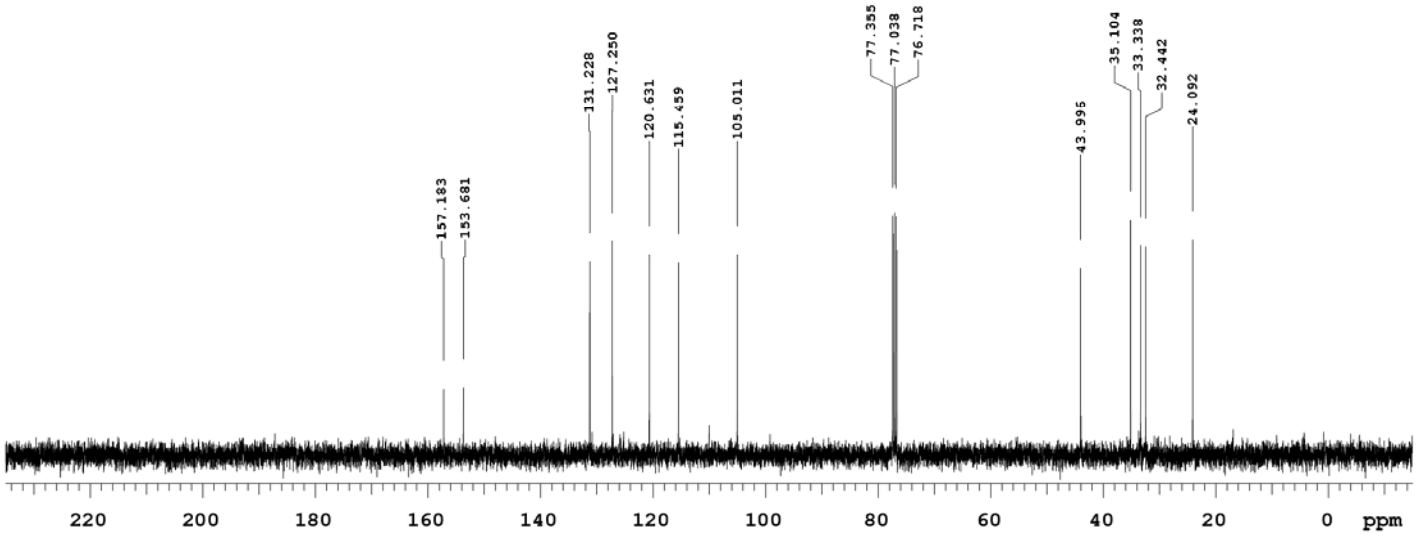

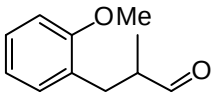

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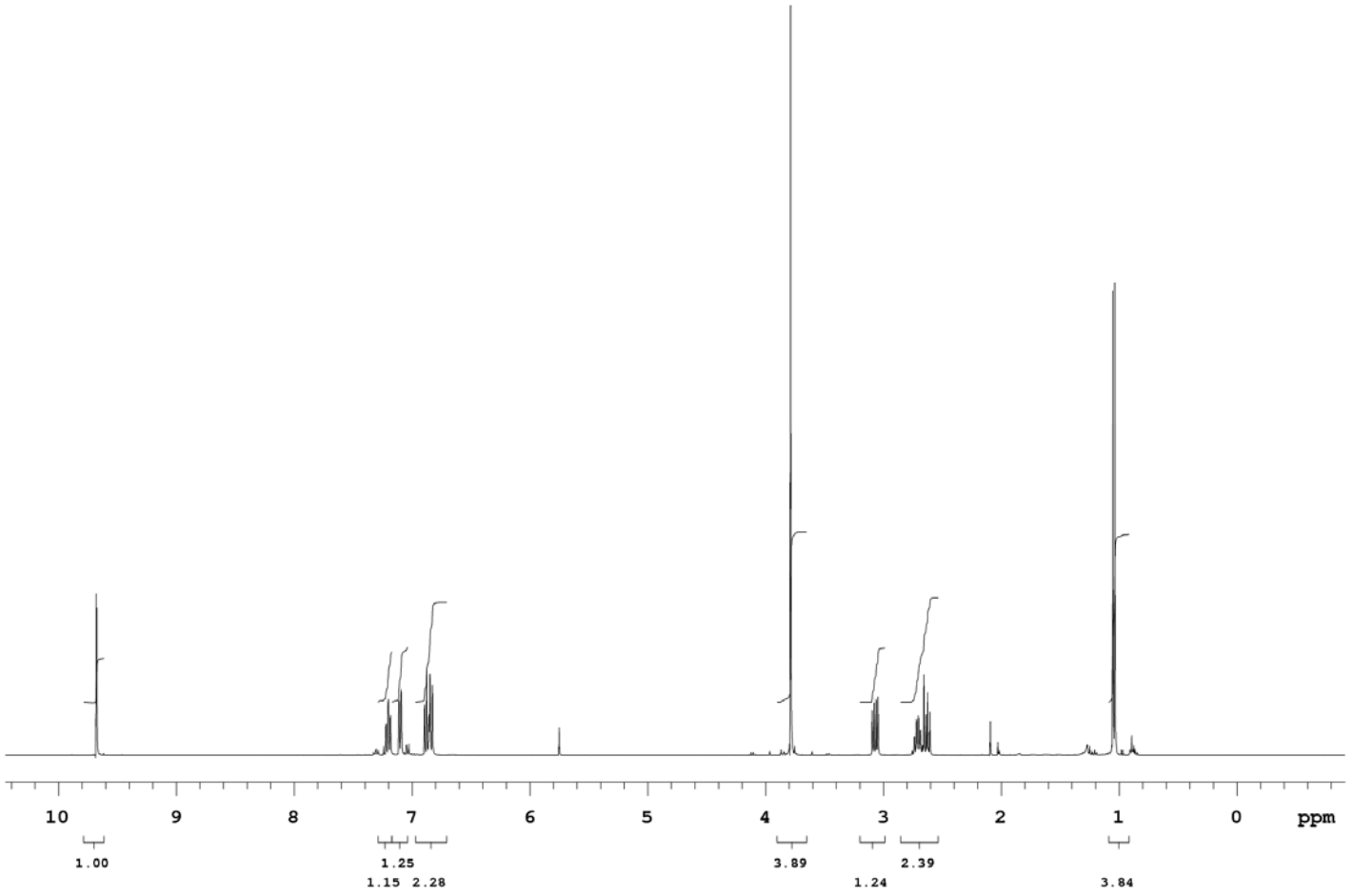

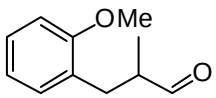

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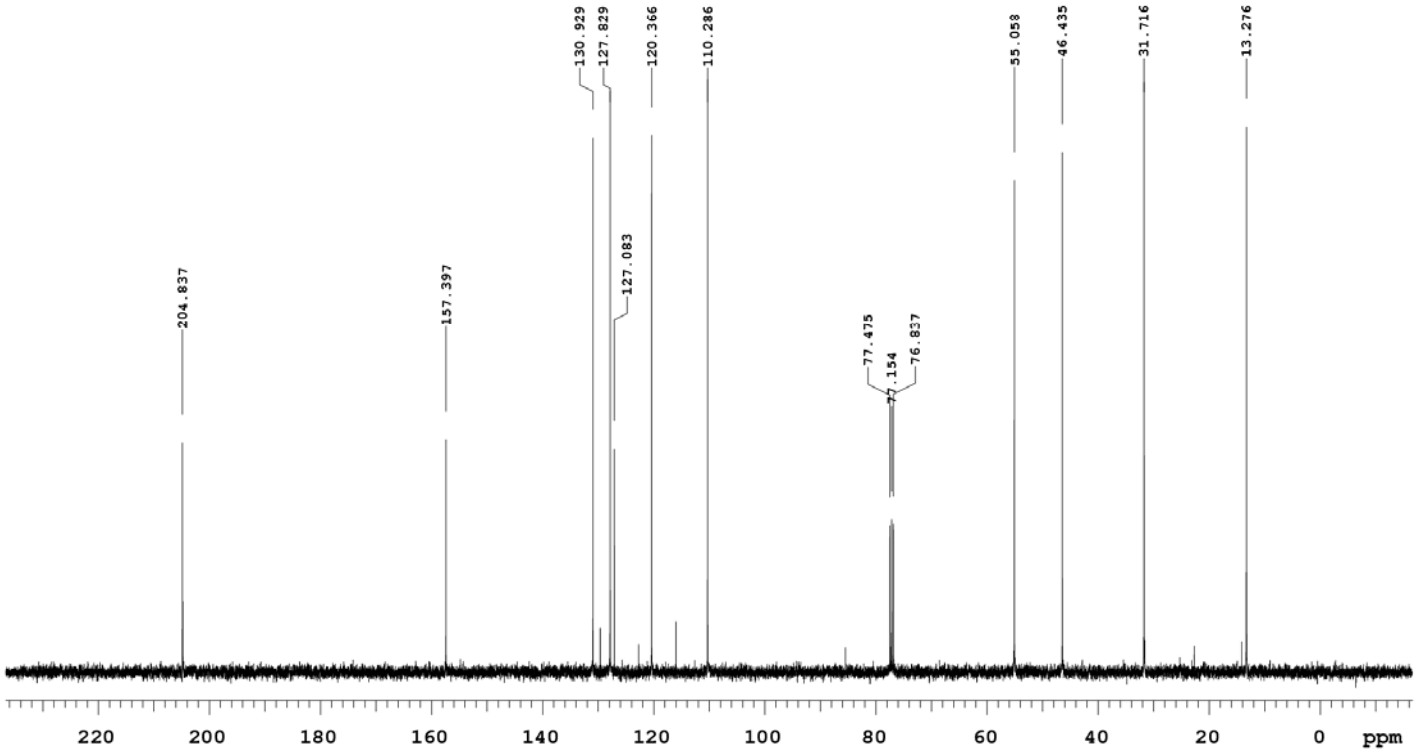

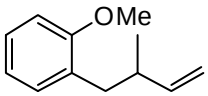

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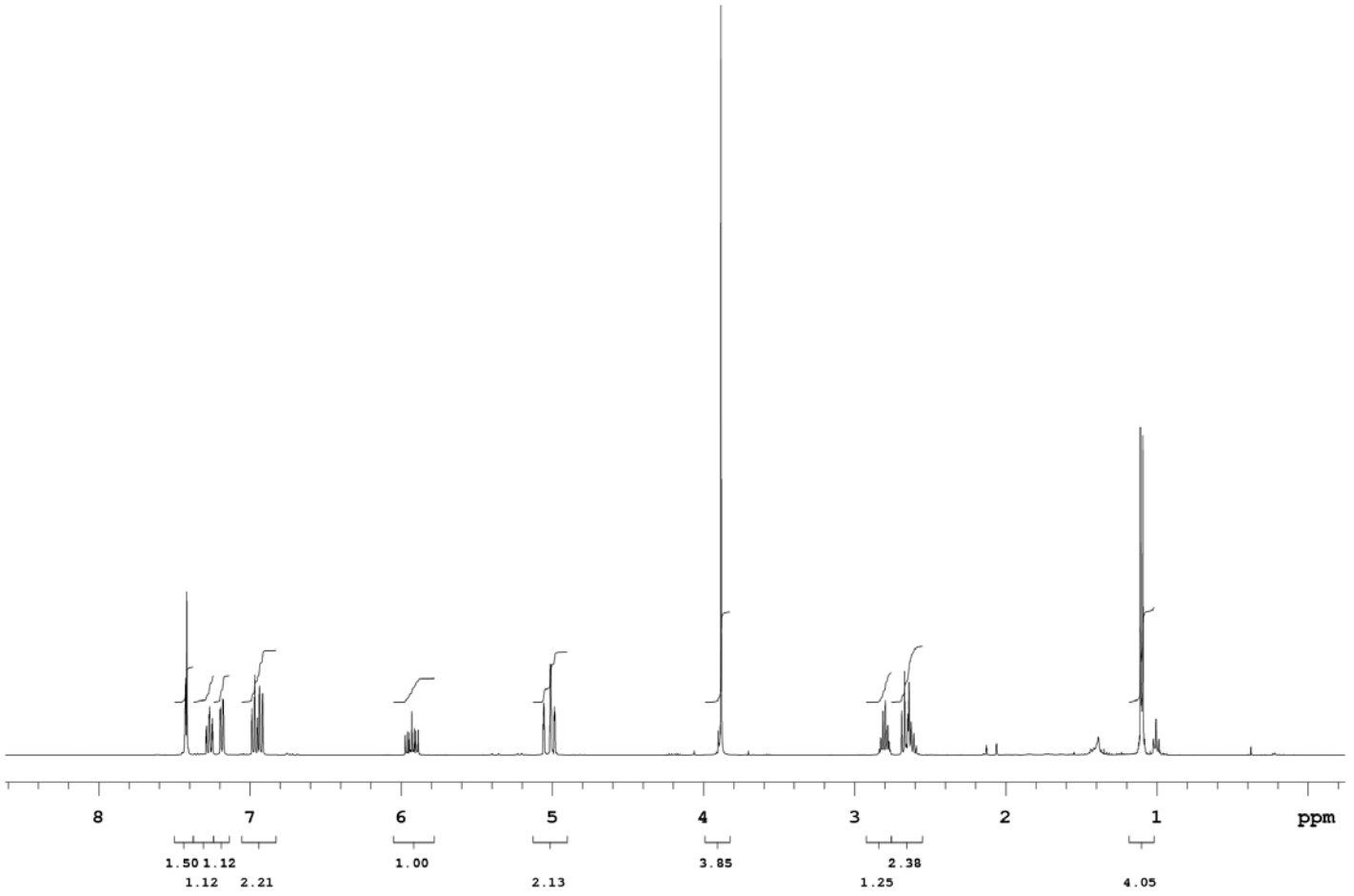

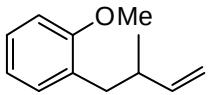

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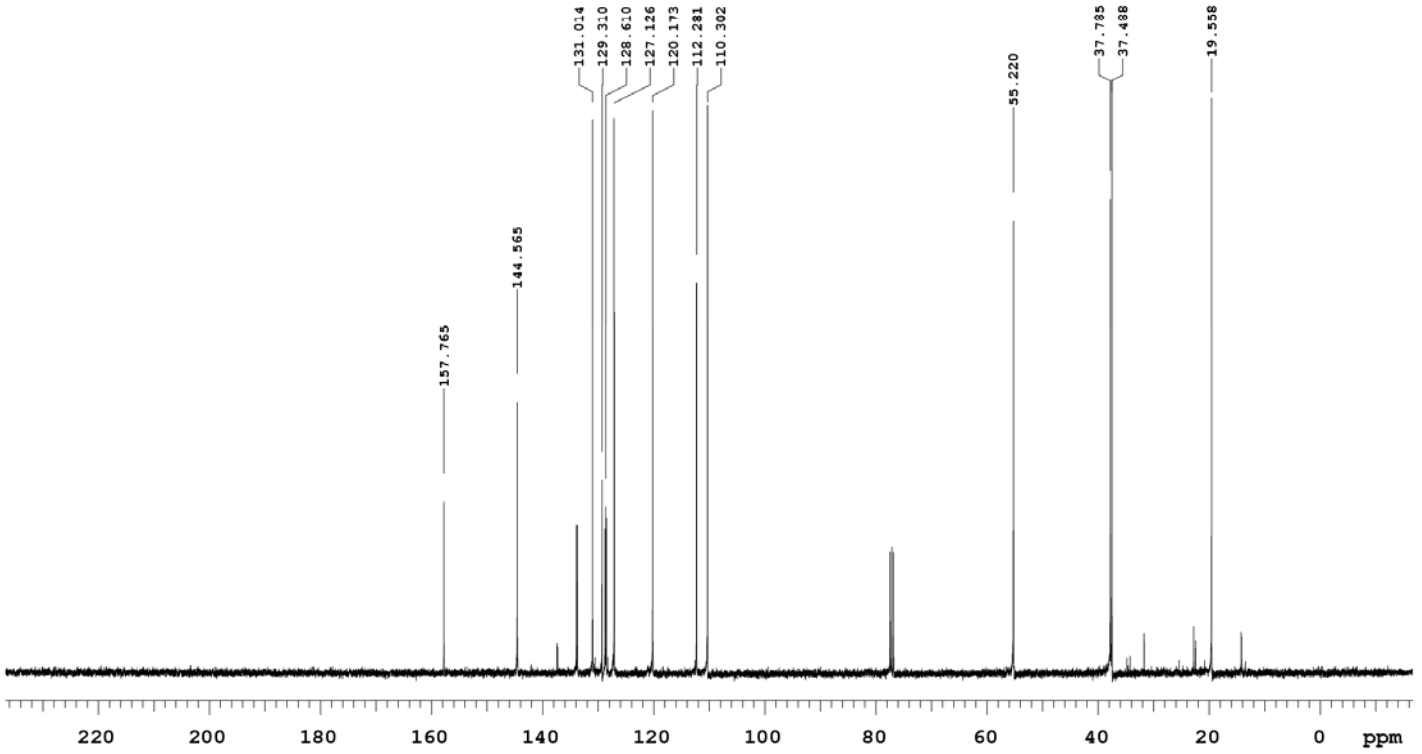

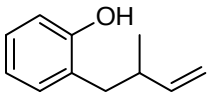

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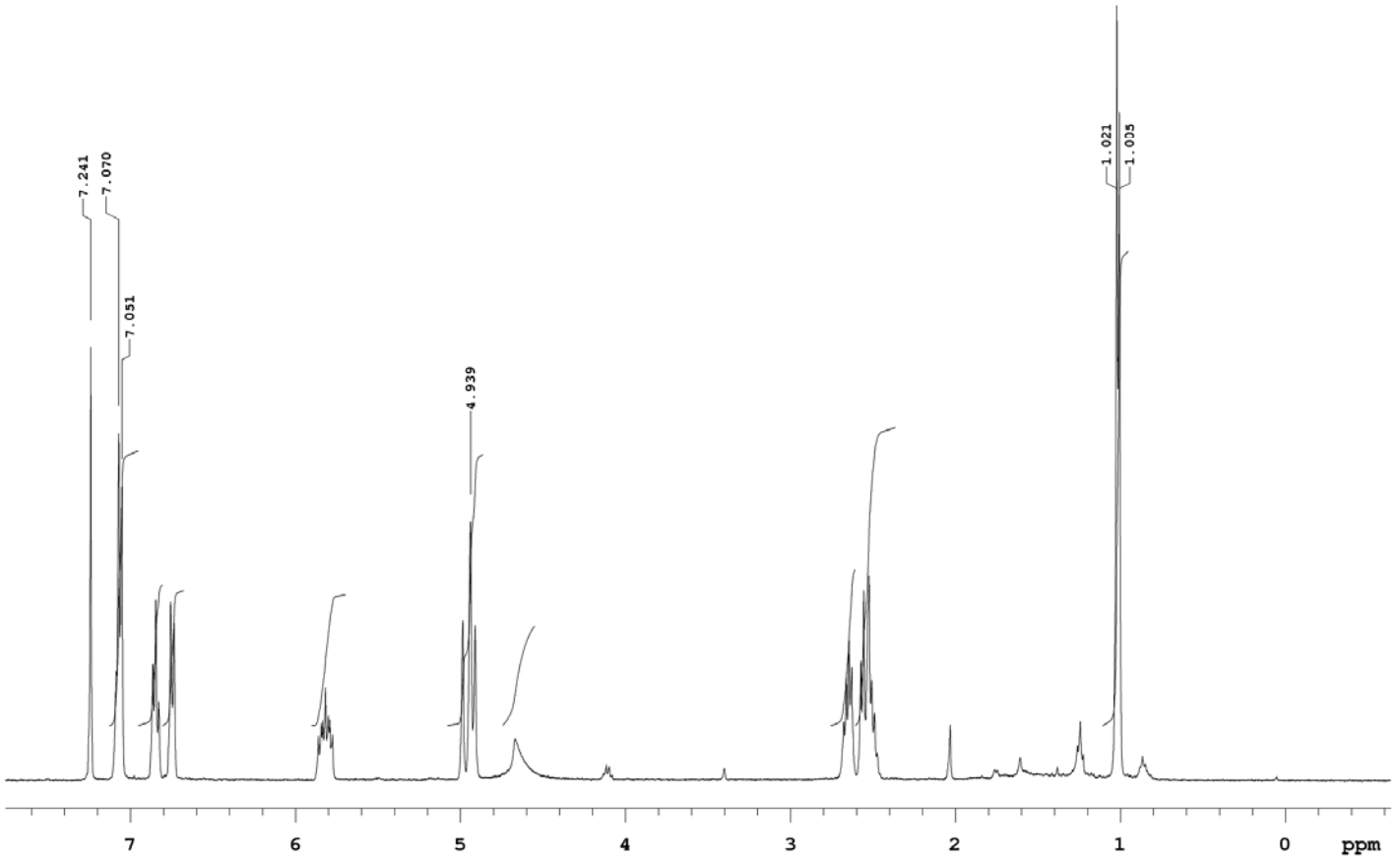

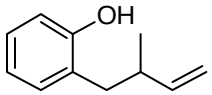

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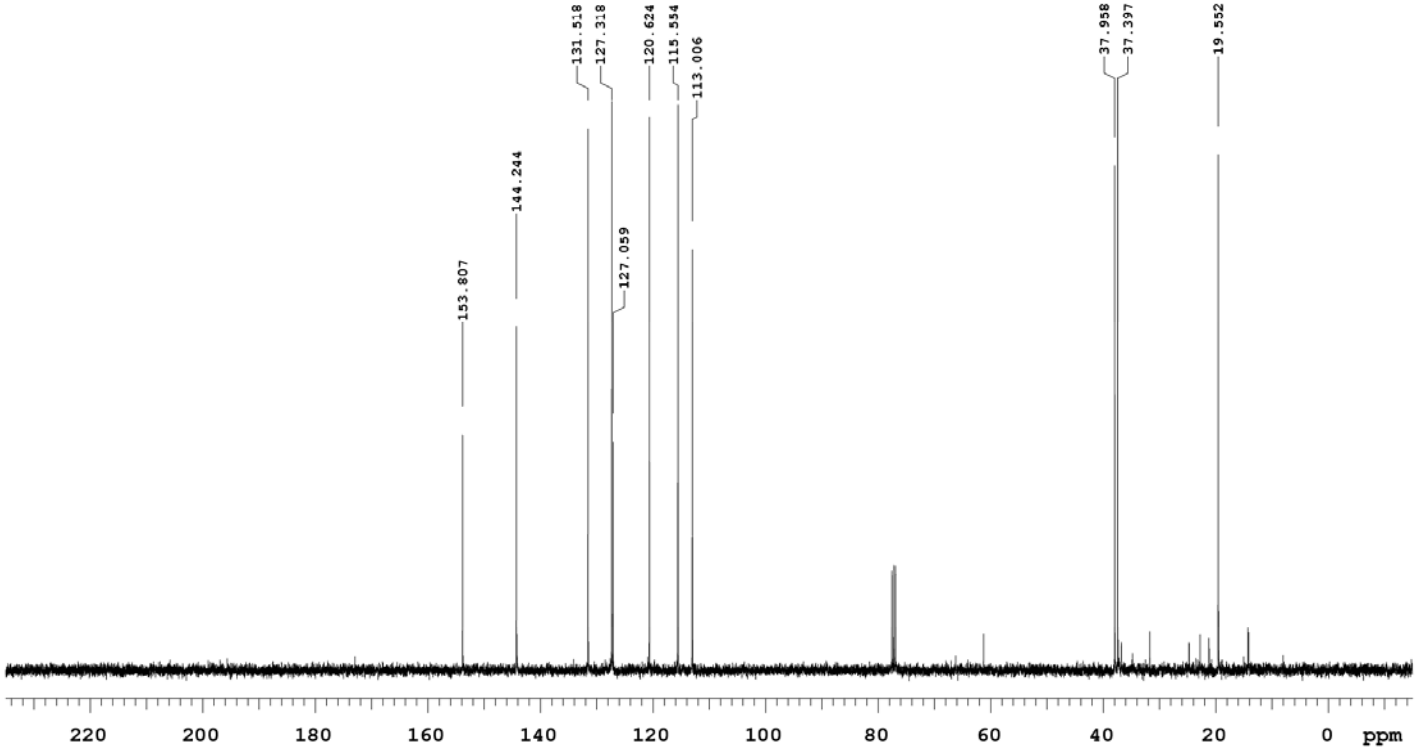

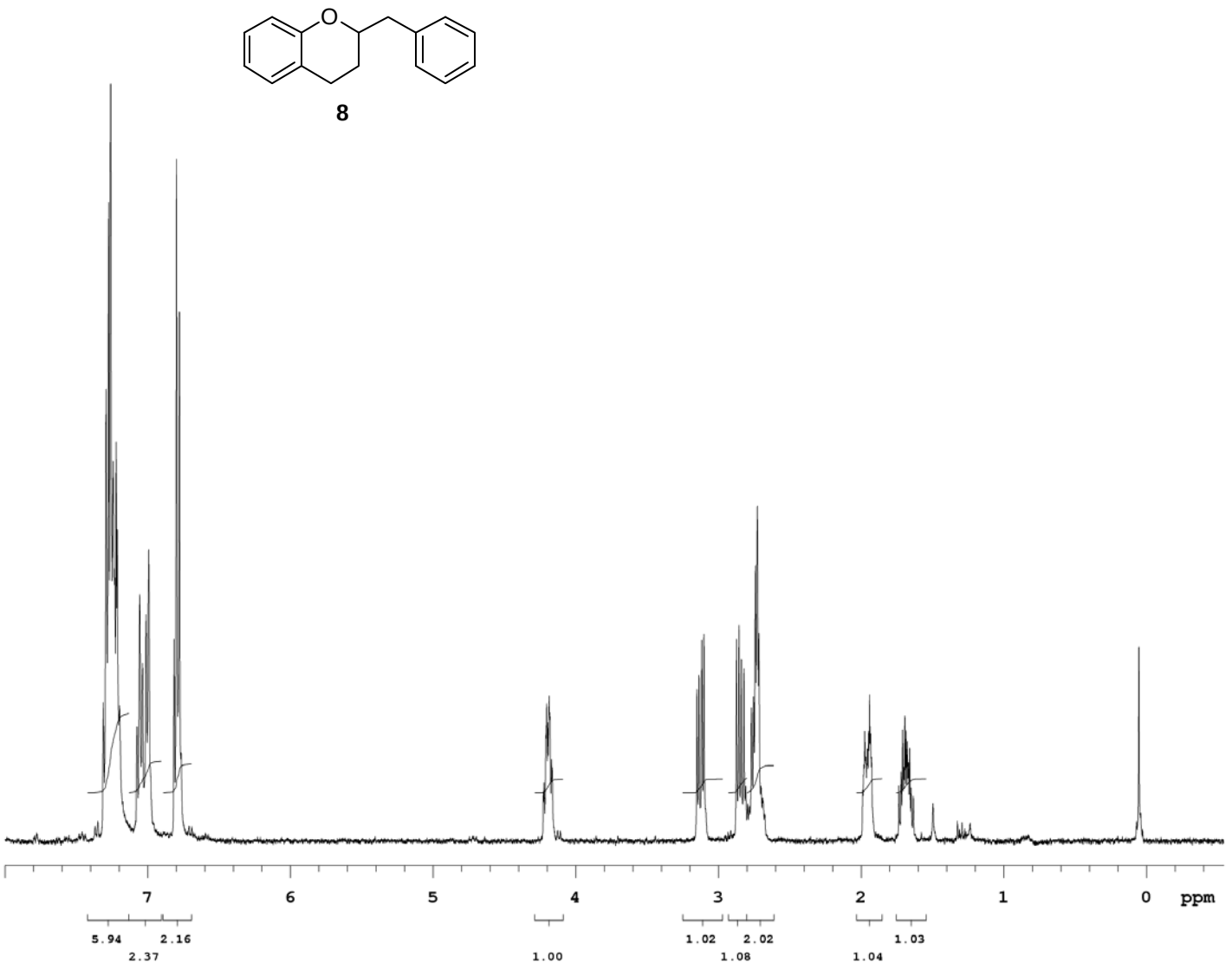

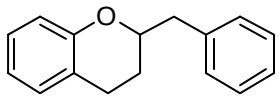

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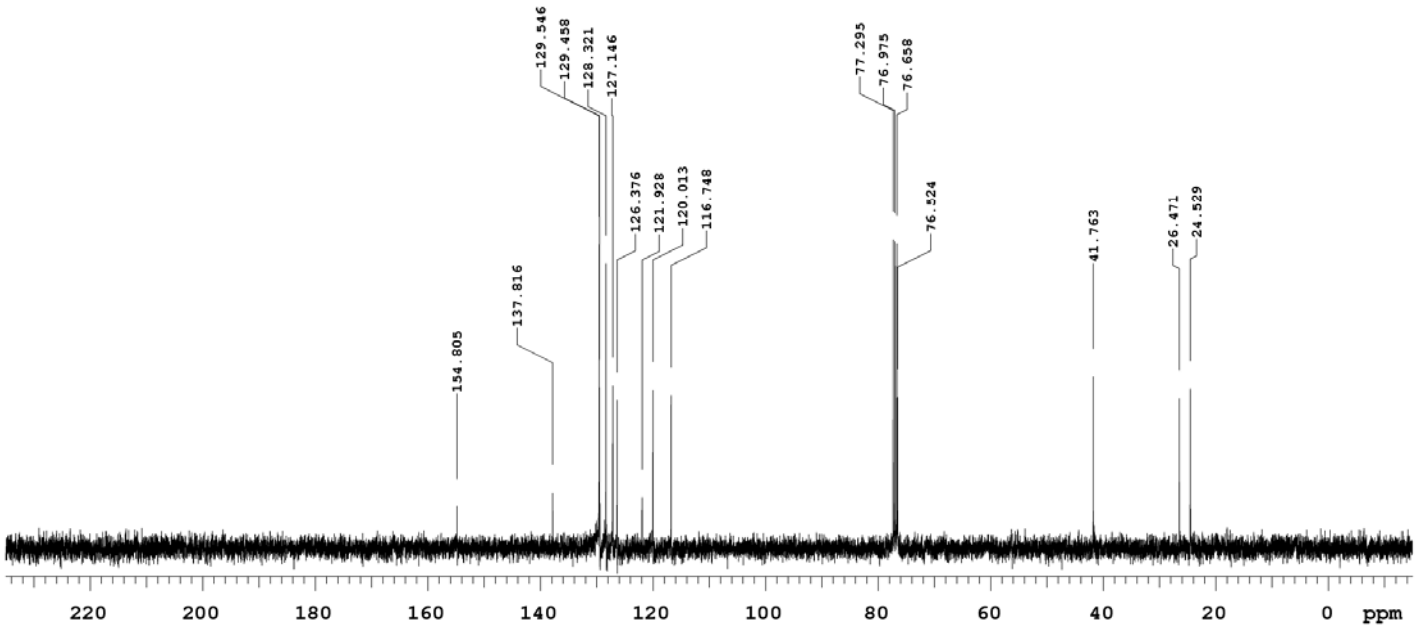
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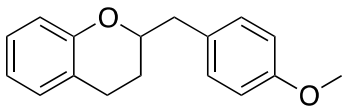
40
20
0
ppm



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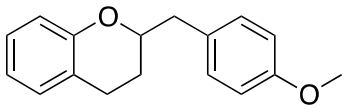

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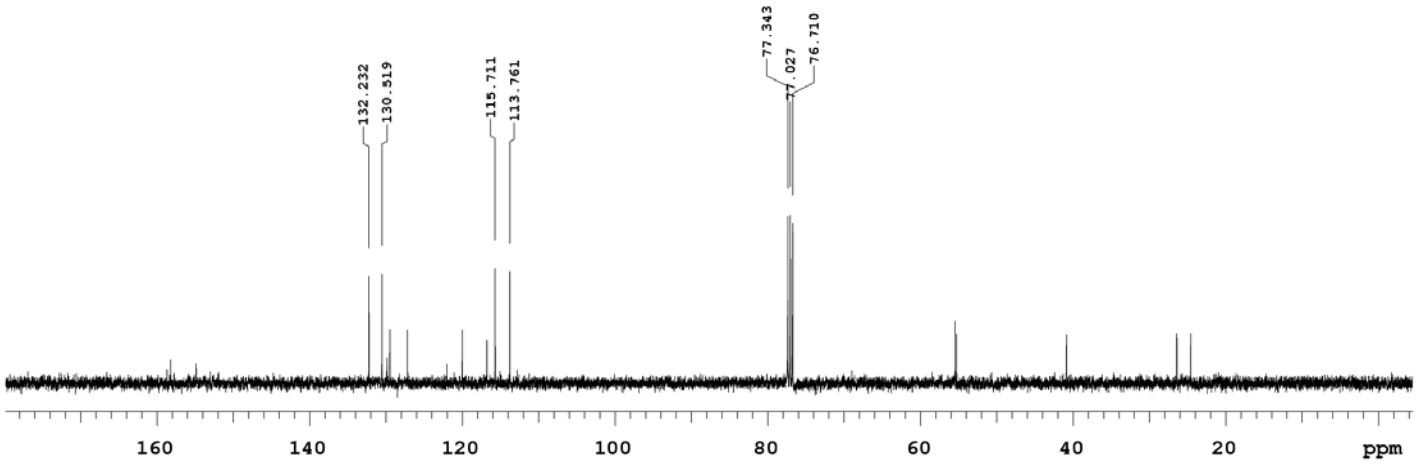

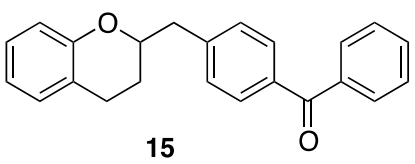

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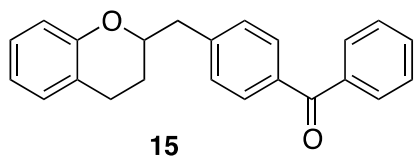

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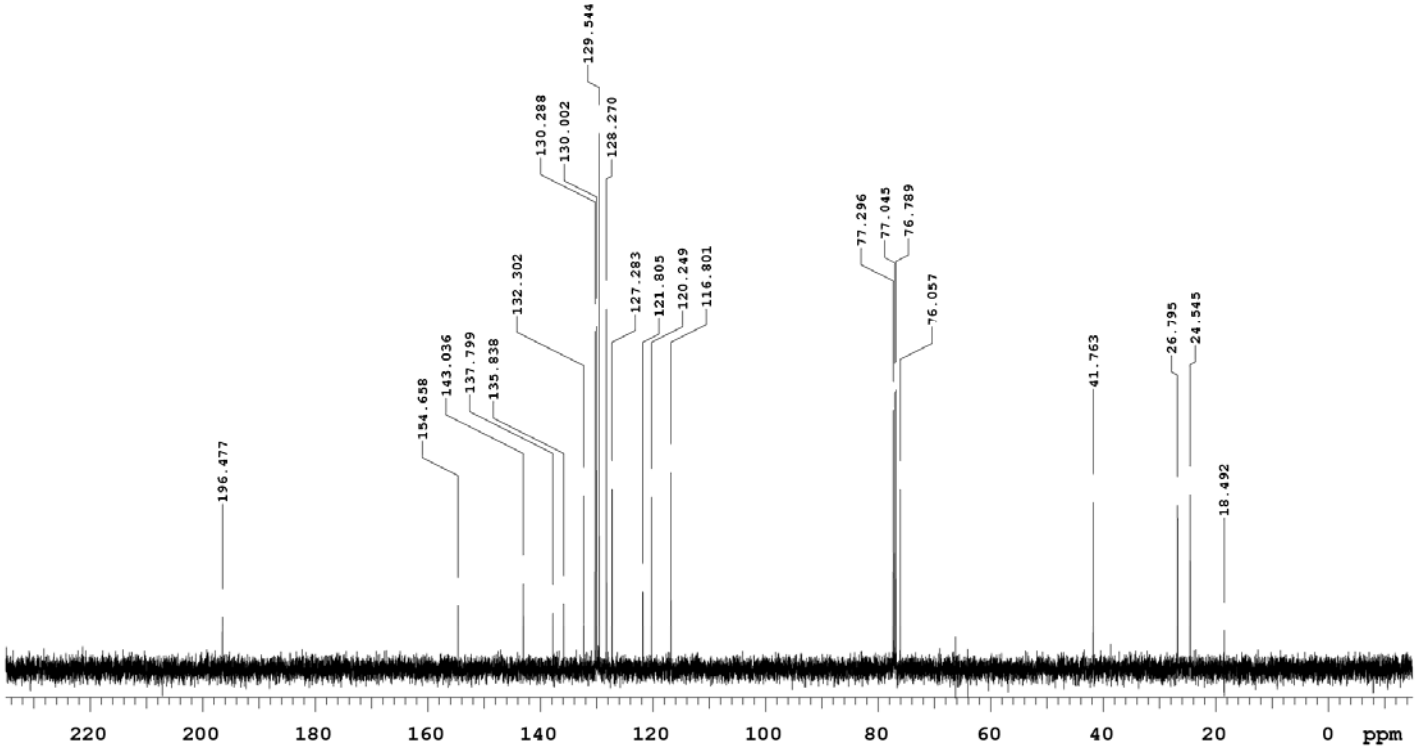

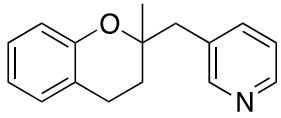

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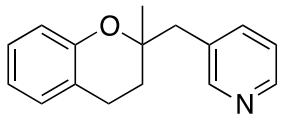

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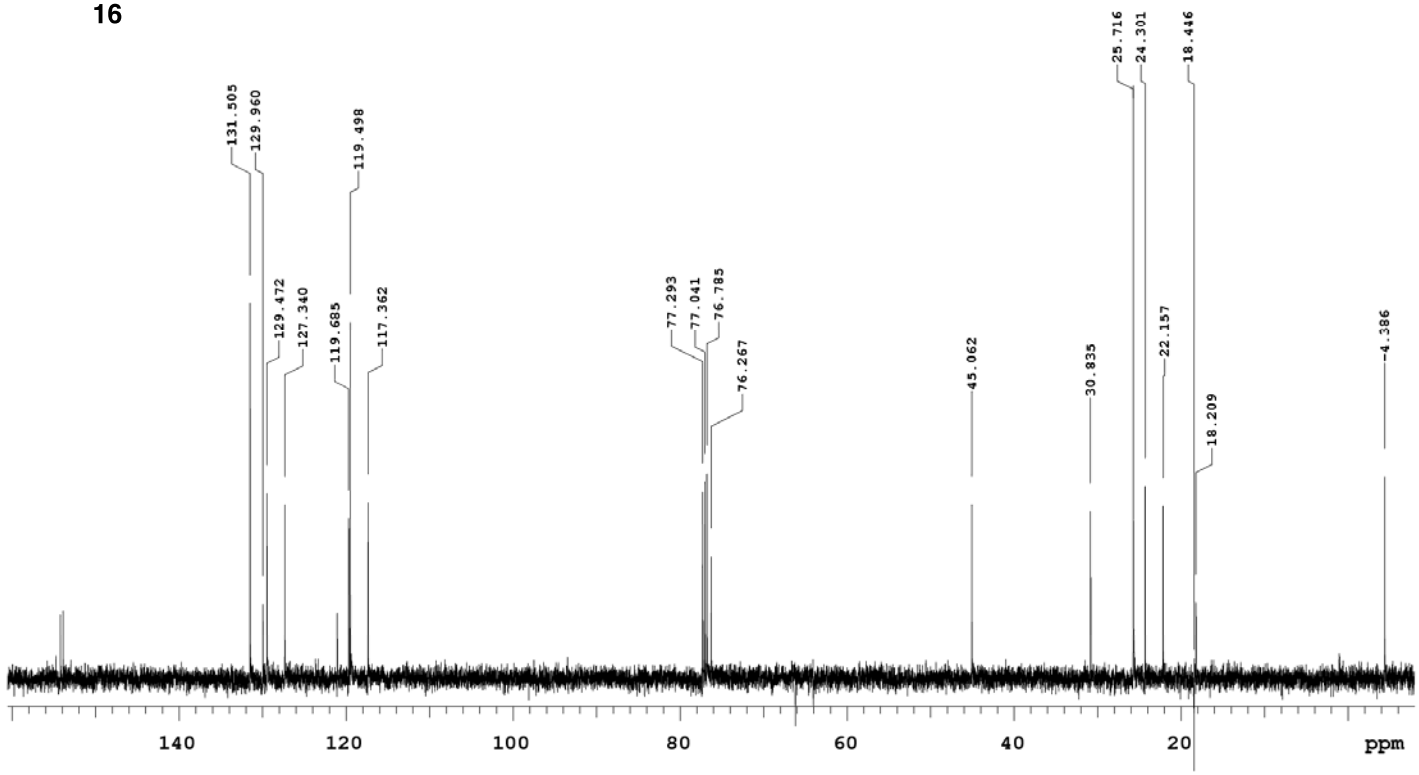

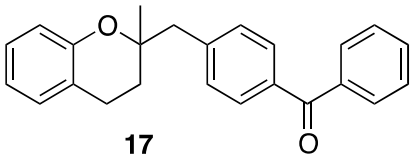

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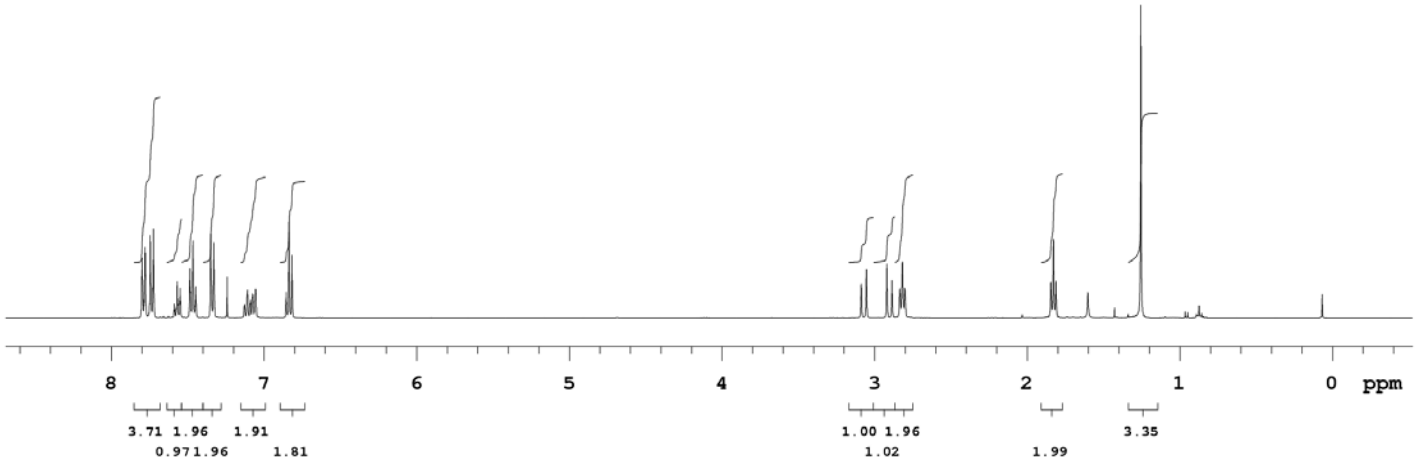

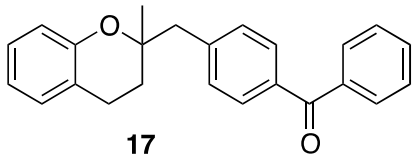

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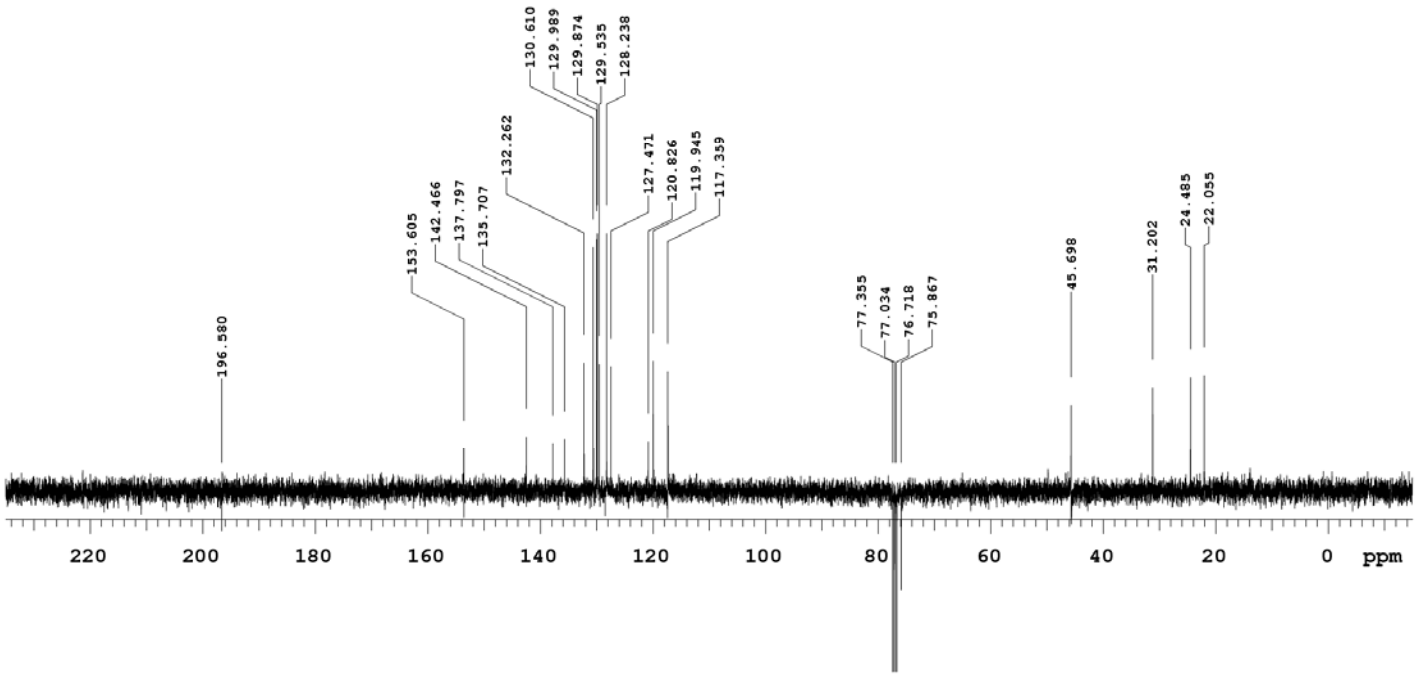

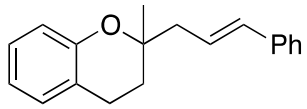

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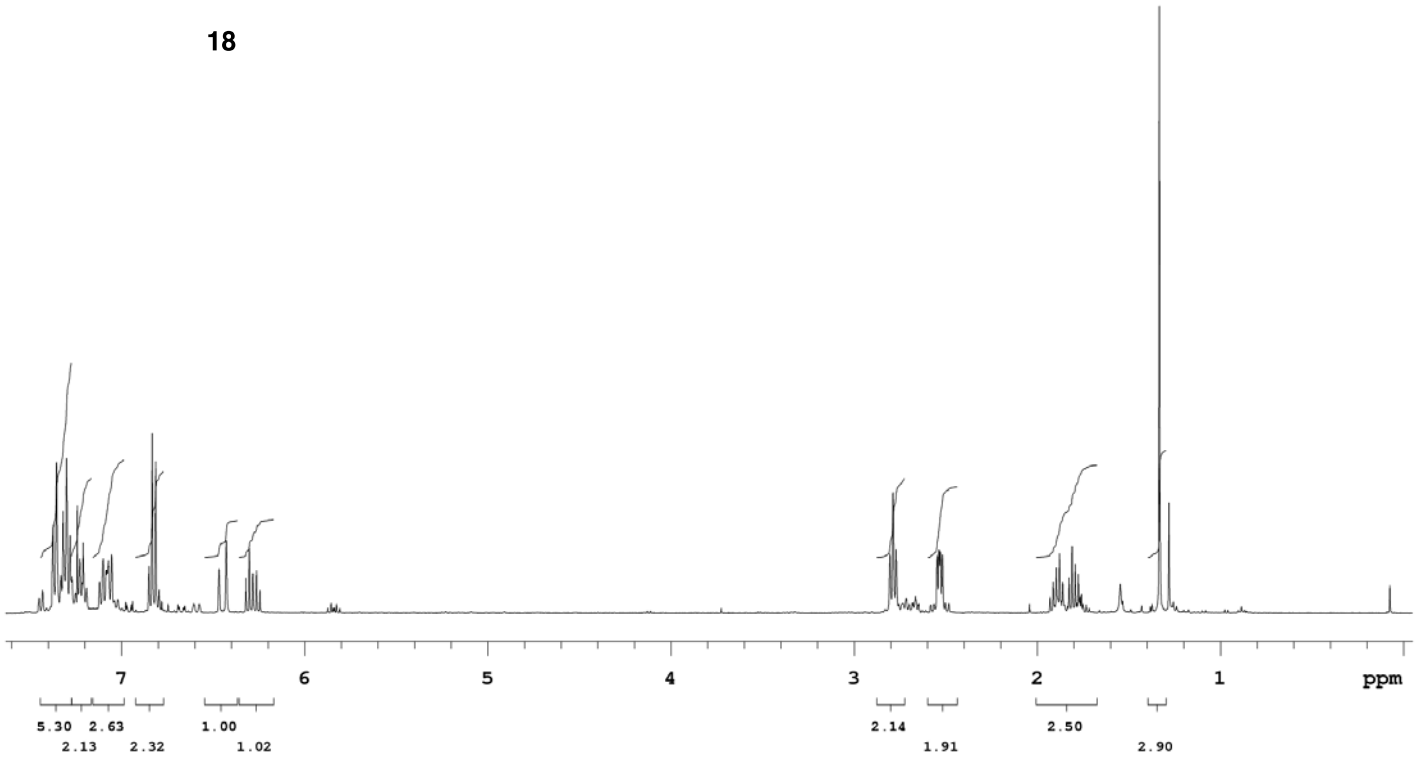

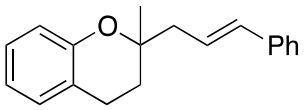

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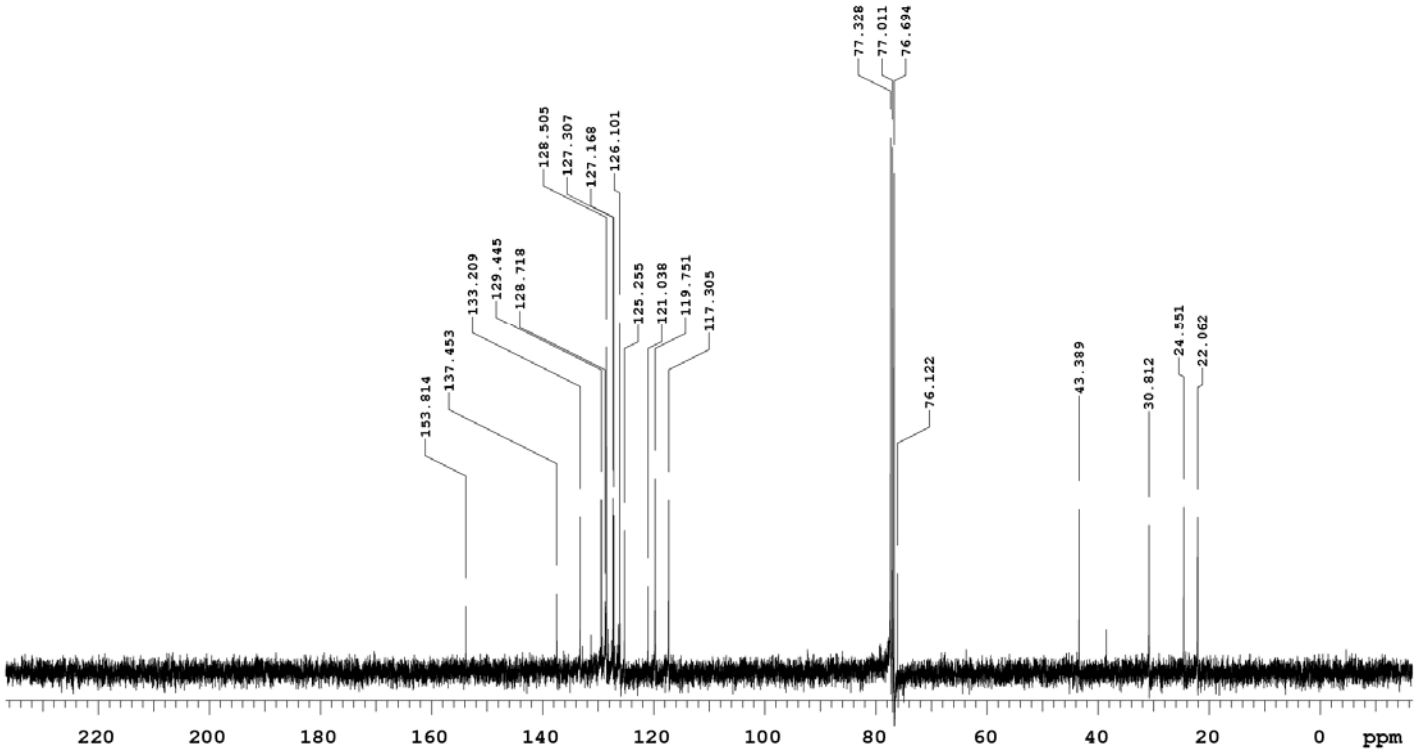

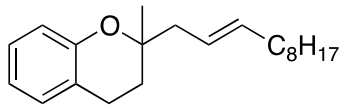

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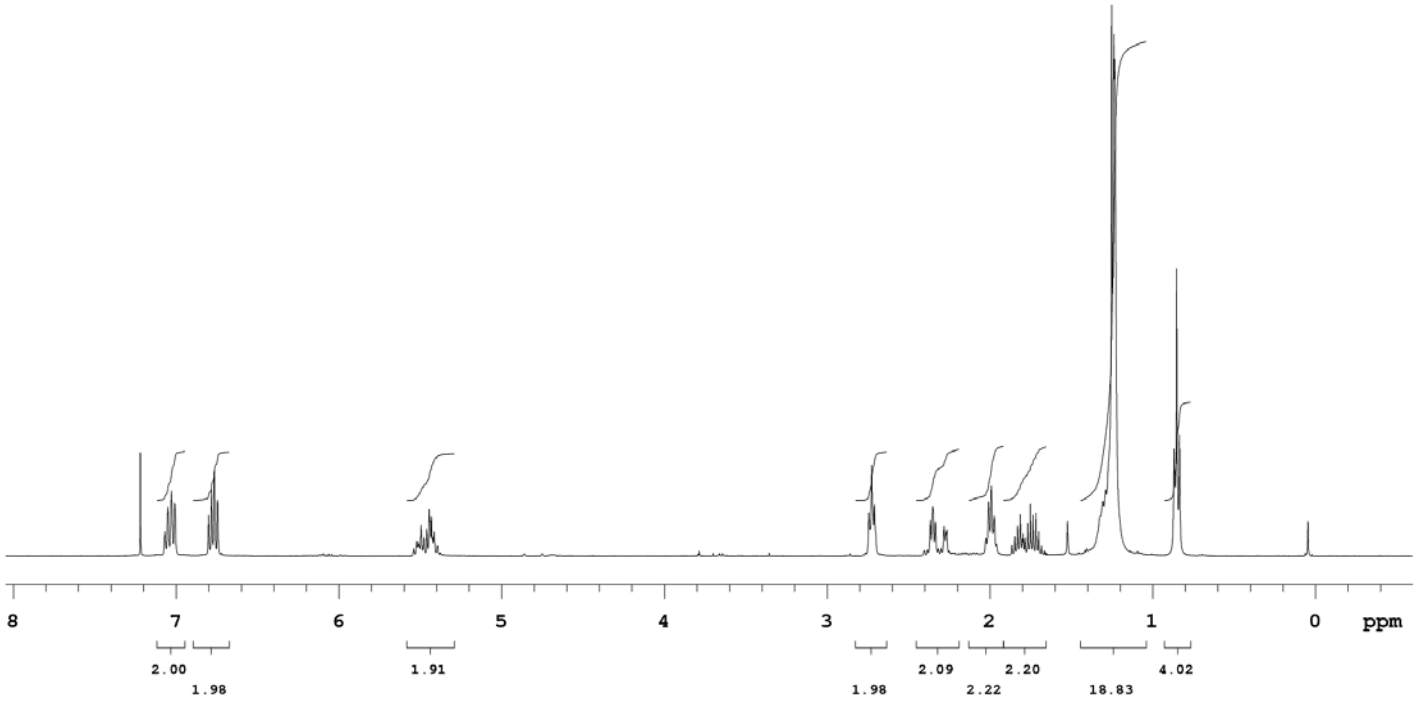

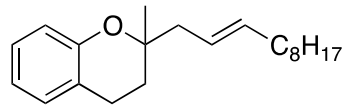

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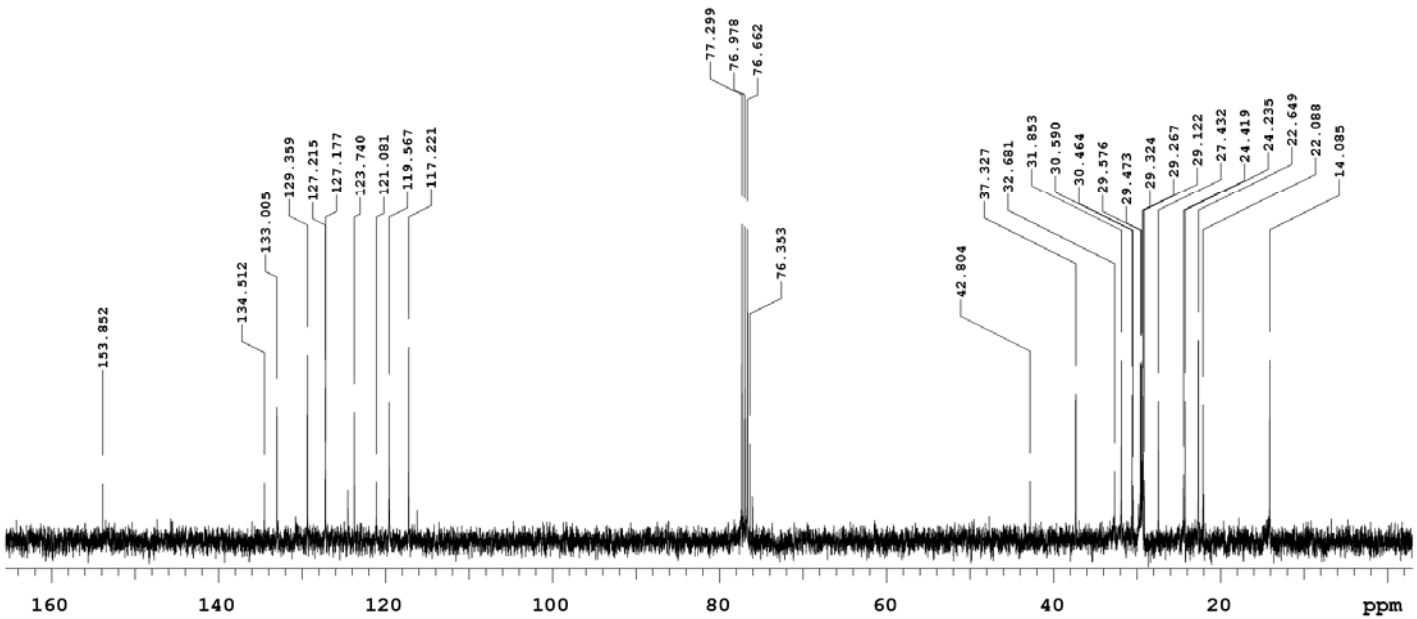

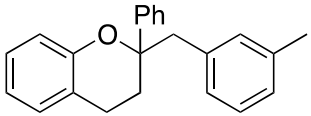

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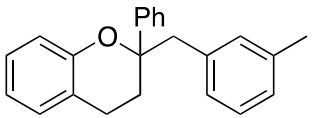
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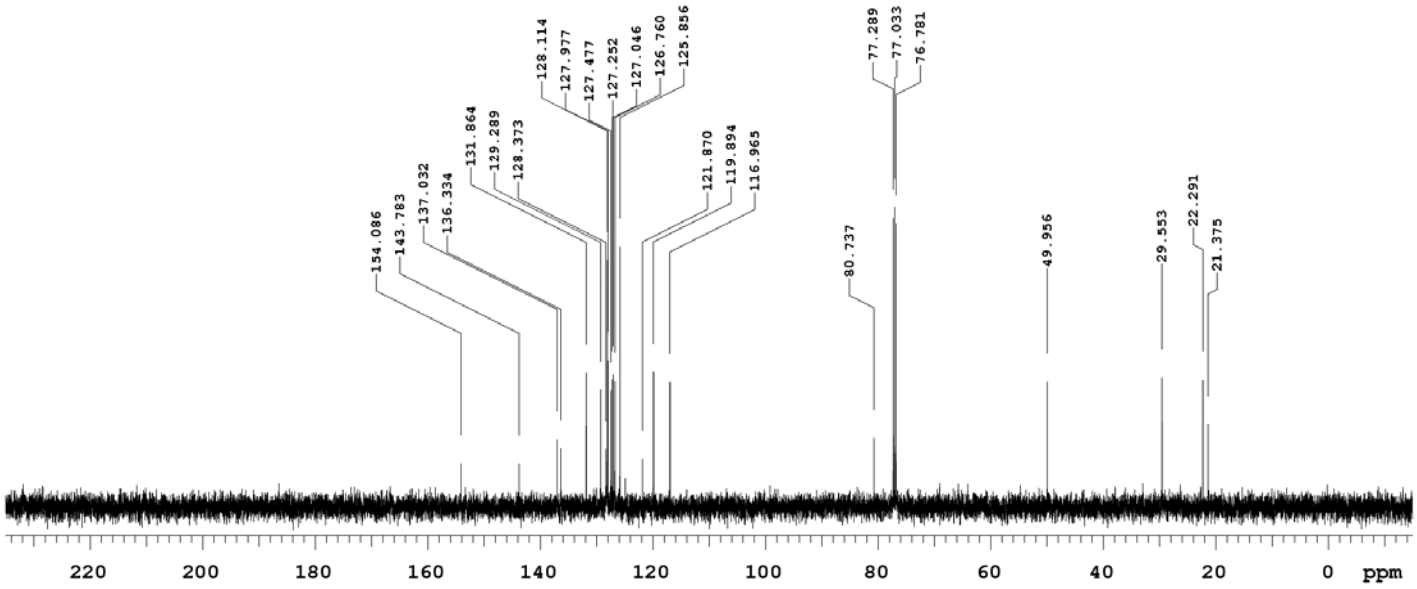
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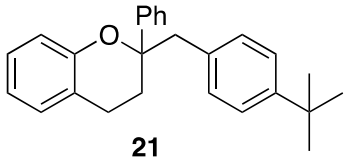
121.870
119.894
116.965
80.737
77.289
77.033
76.781
49.956
29.553
22.291
21.375
220
200
180
160
140
120
100
80
60
40
20
0
ppm



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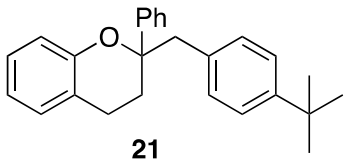

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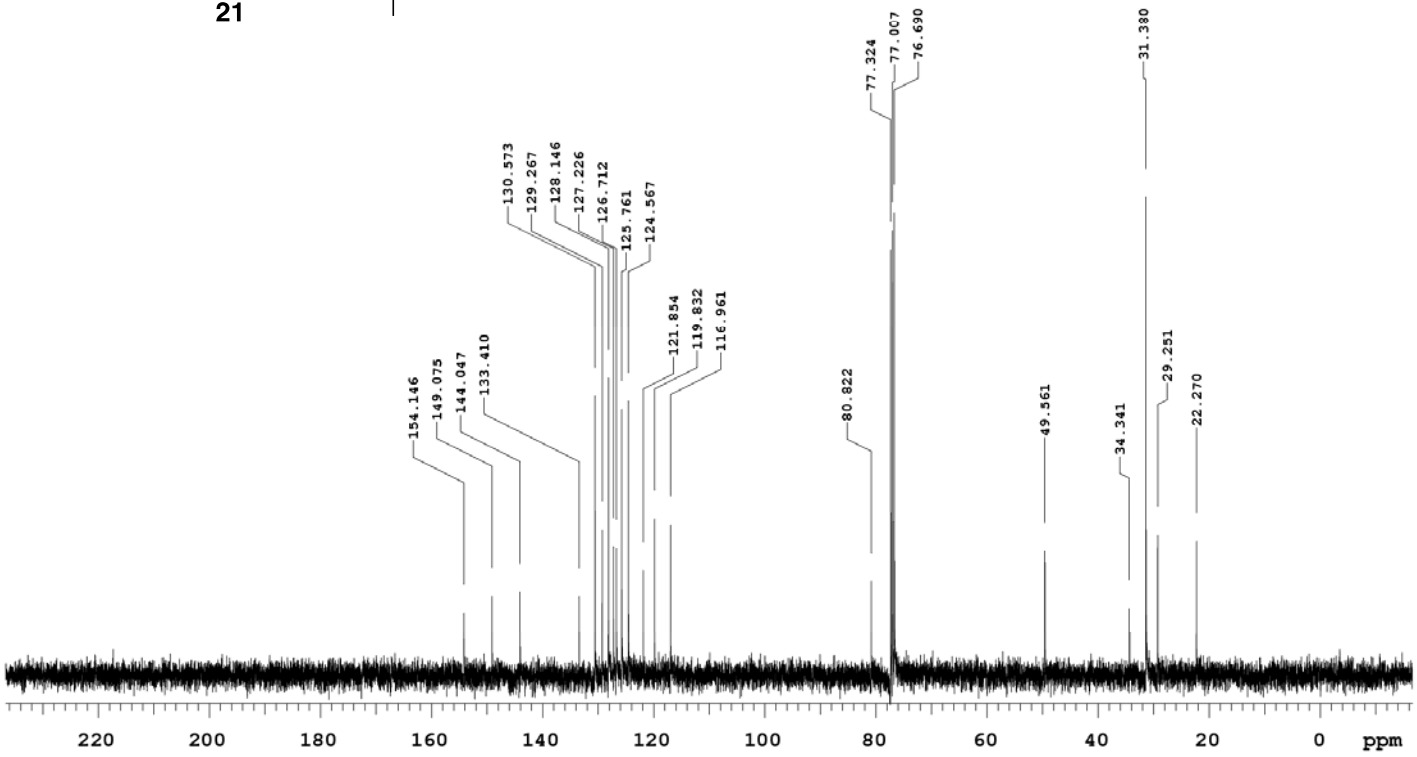

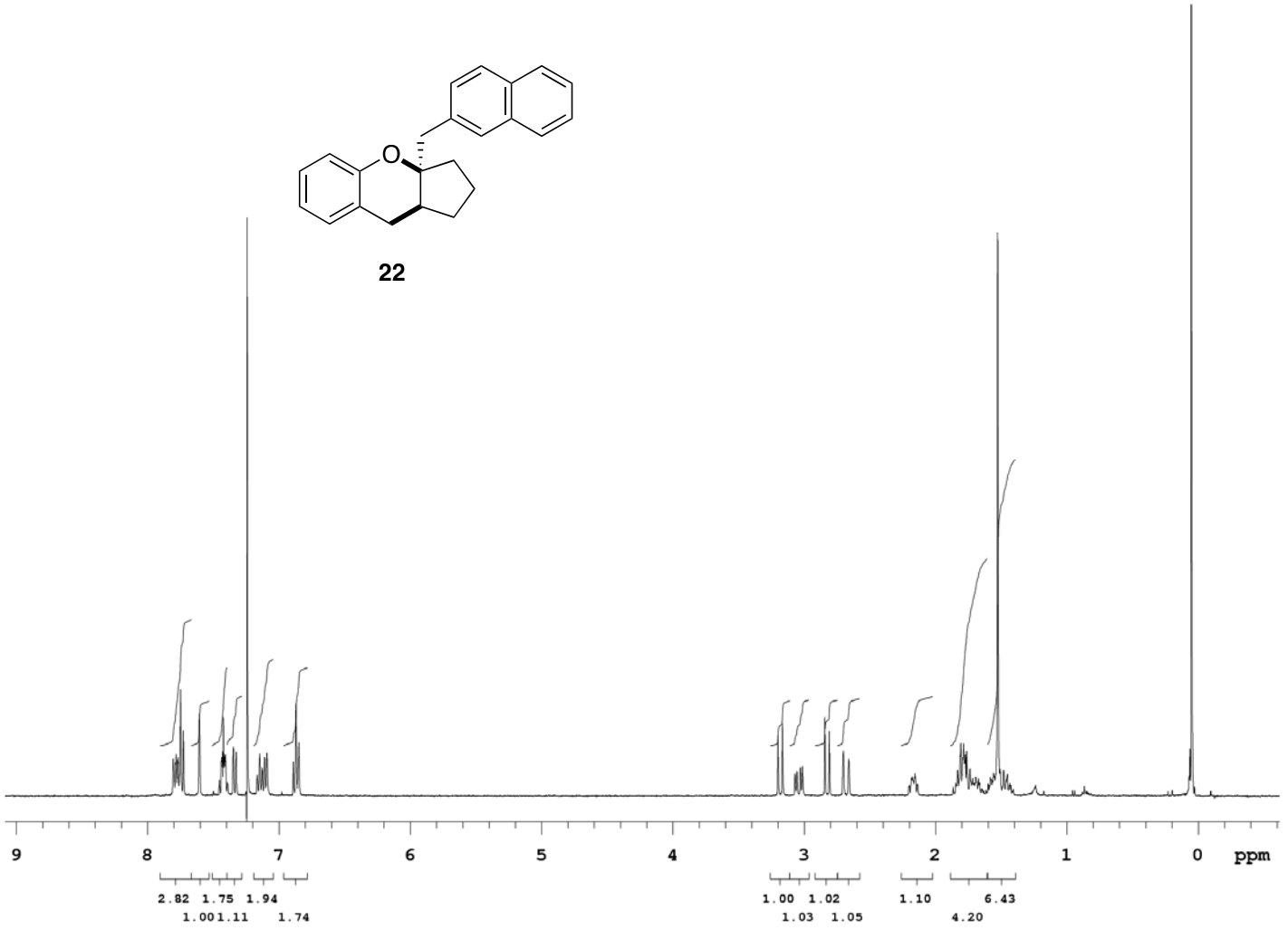

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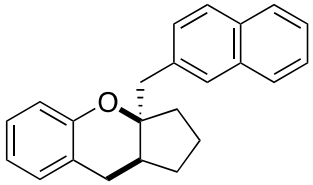

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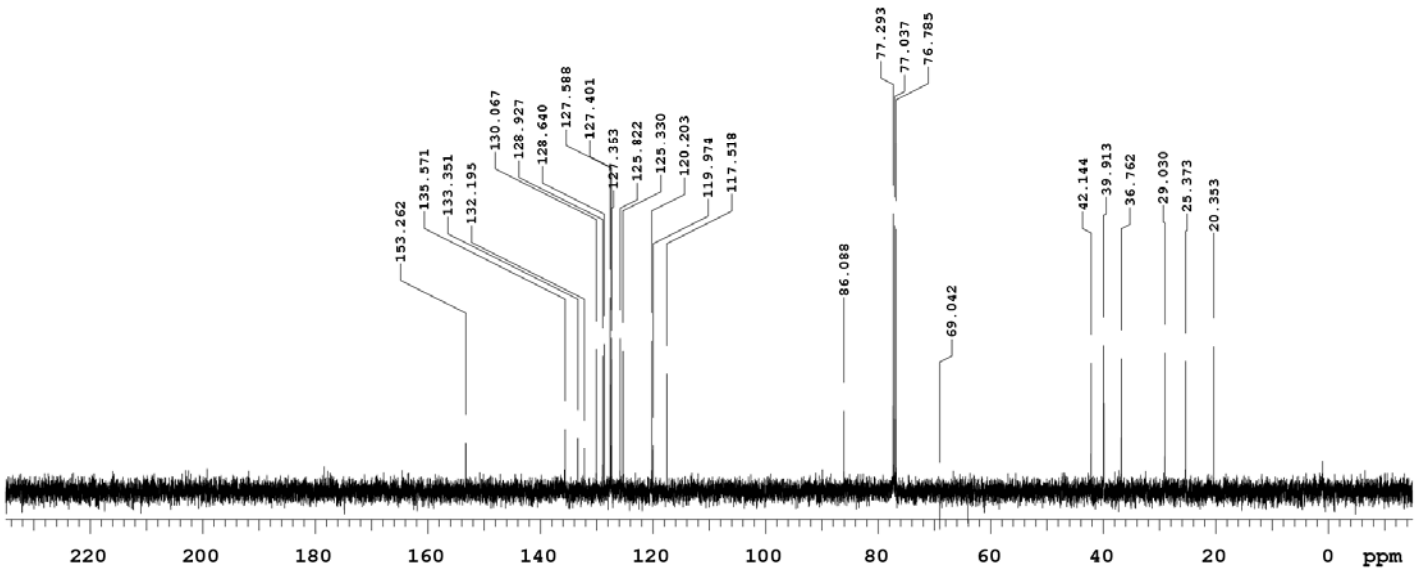
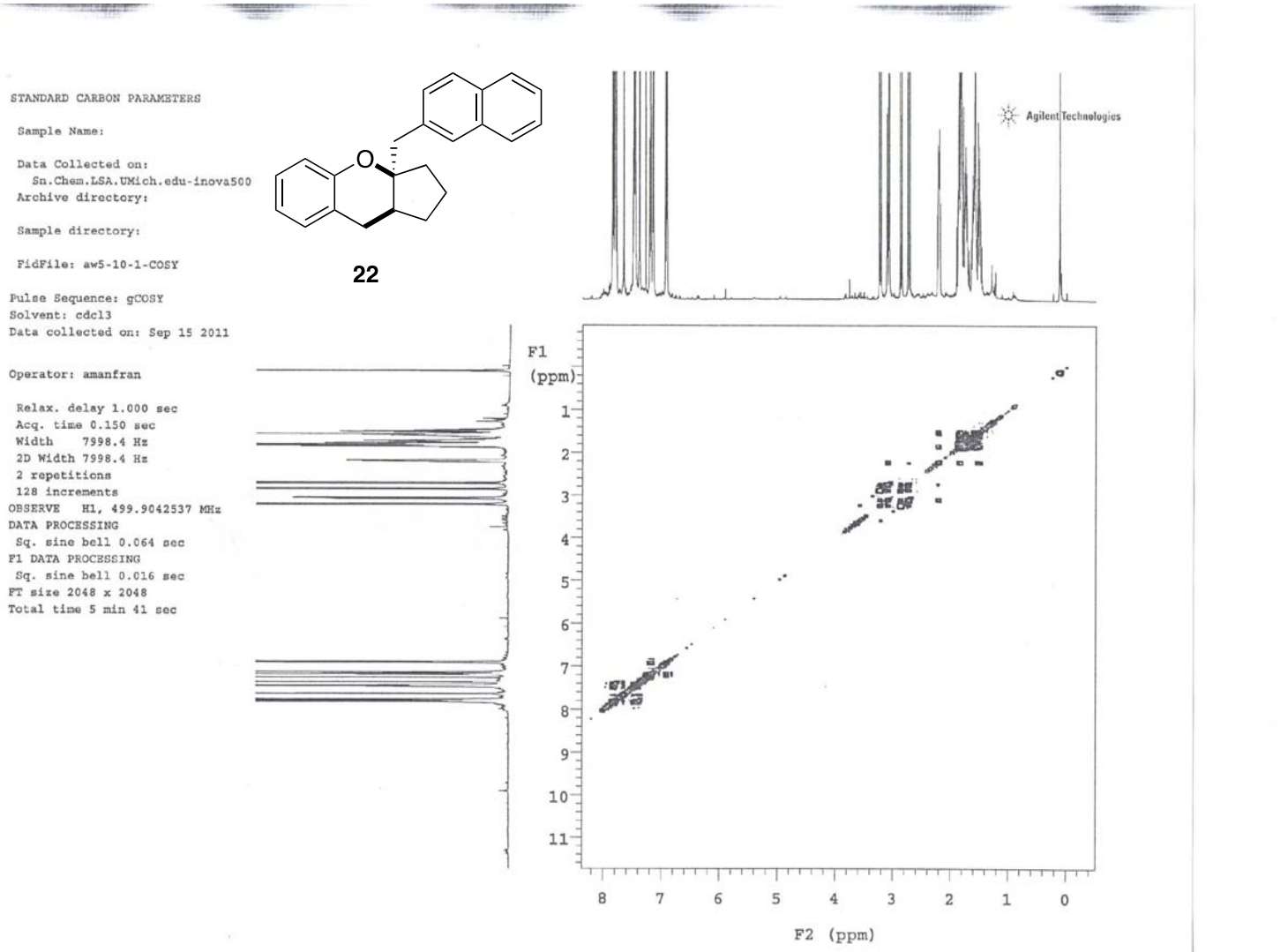
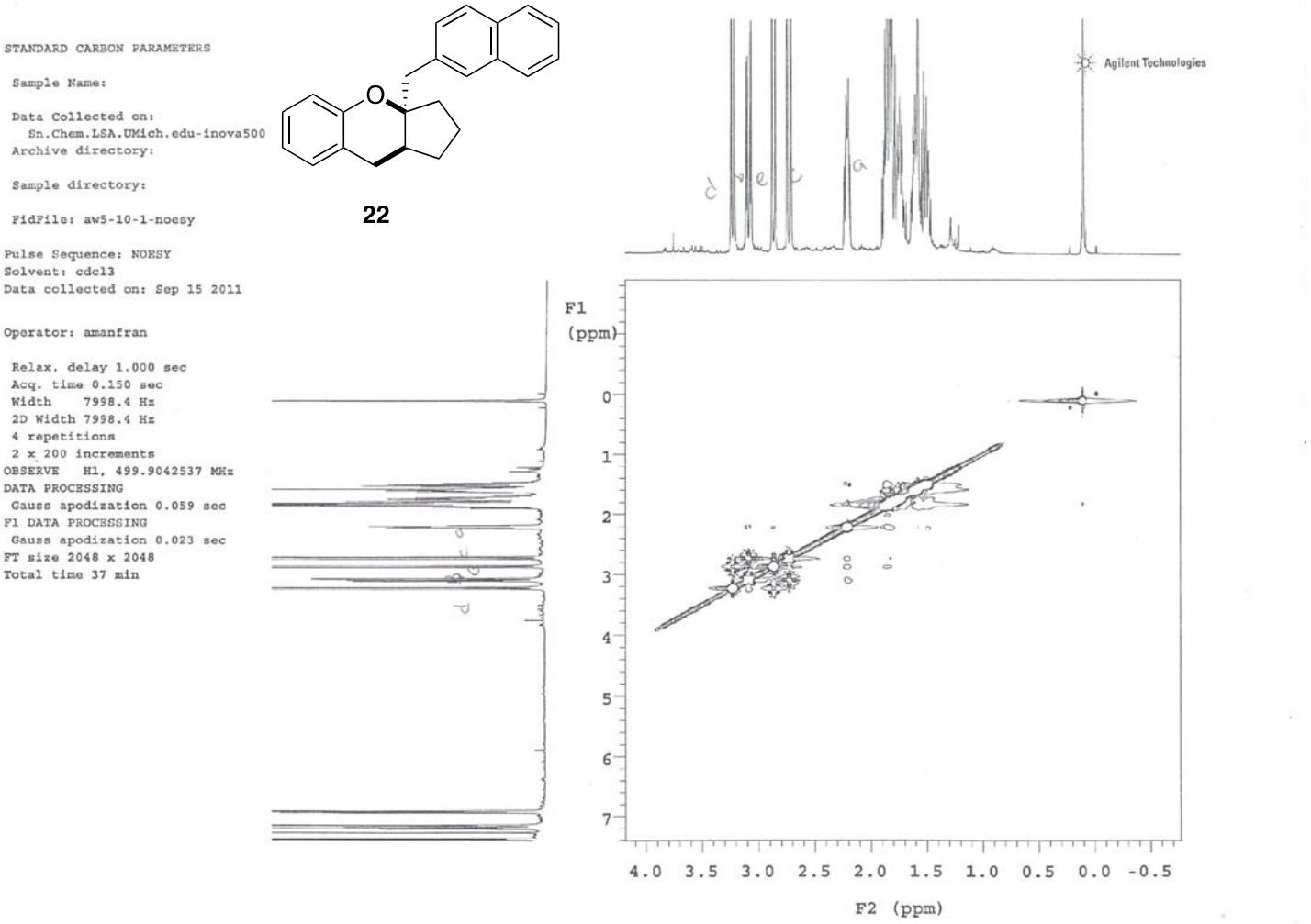
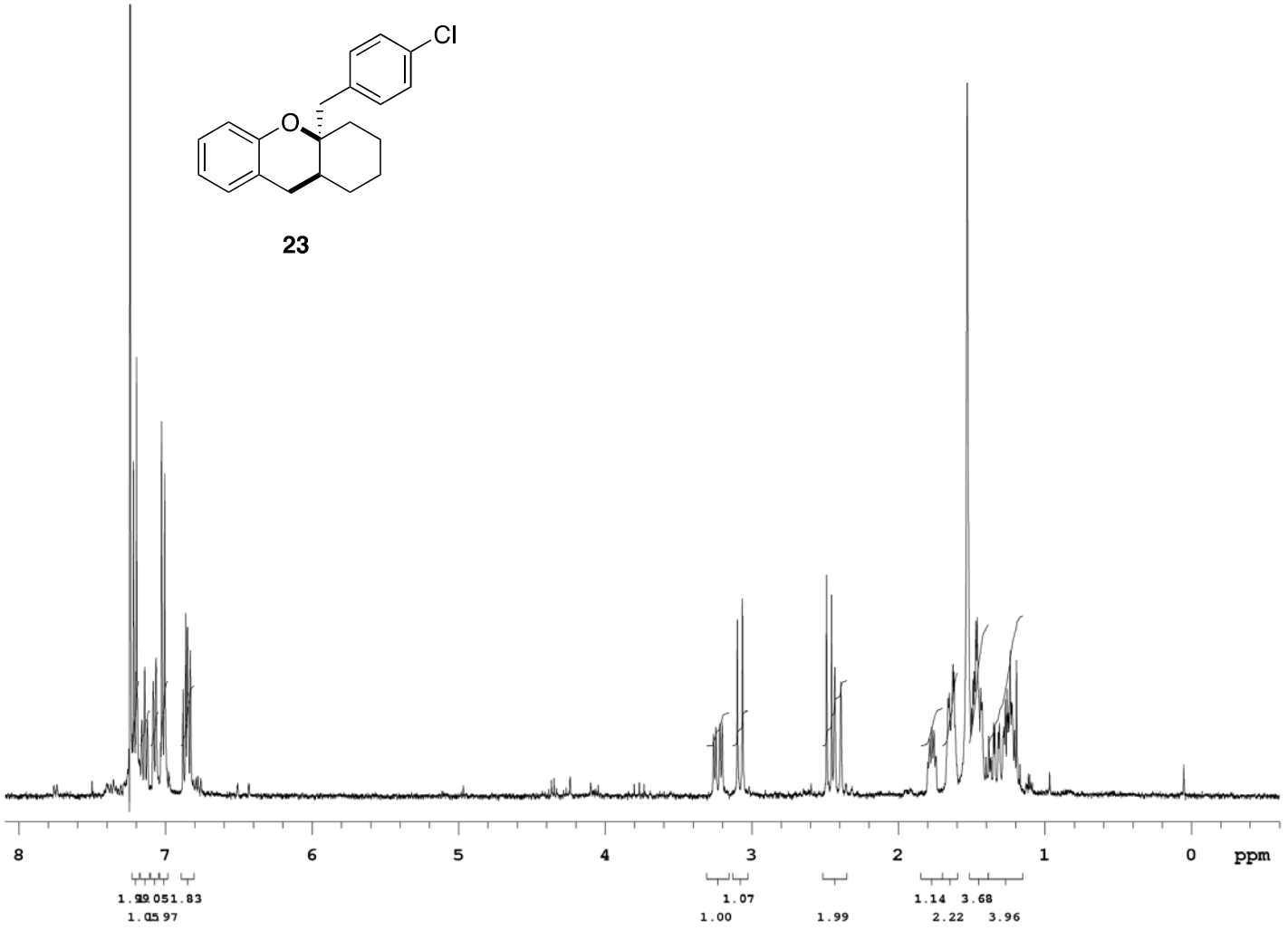
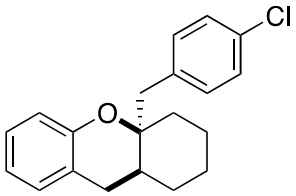


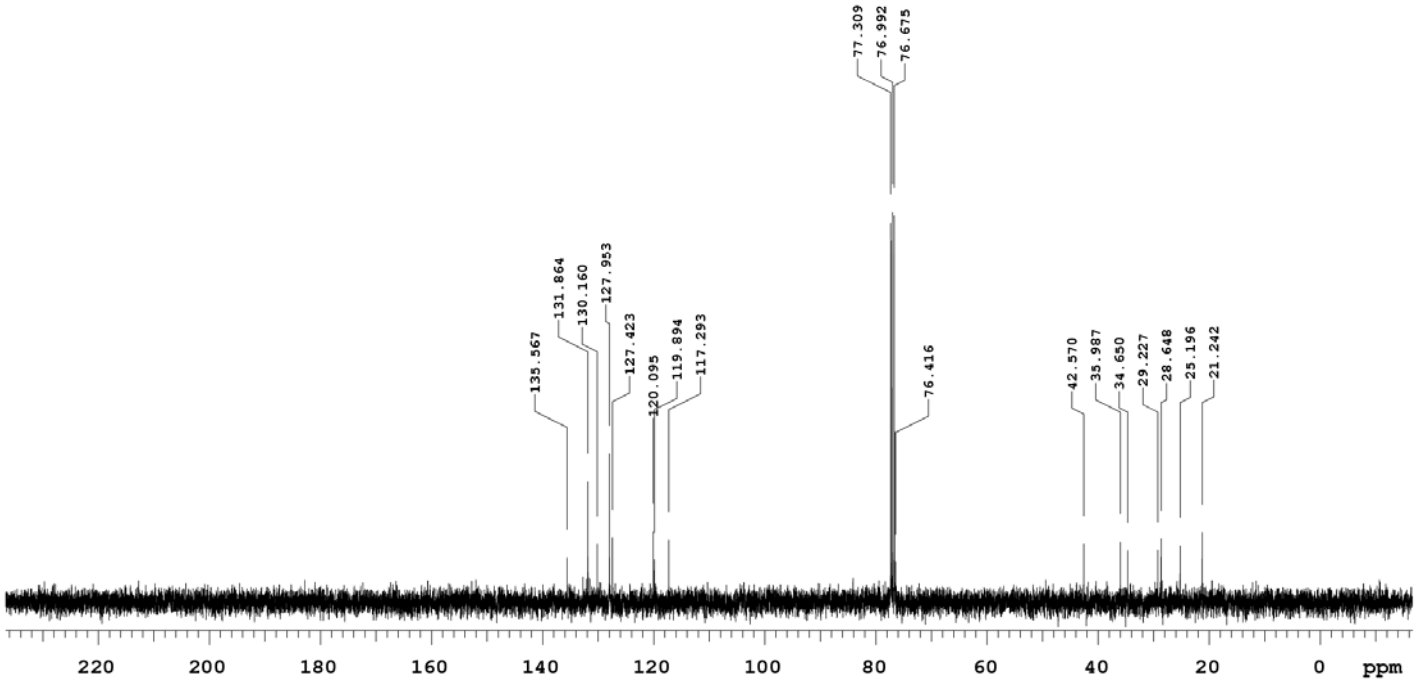

STANDARD CARBON PARAMETERS
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: aw5-10-1-COSY
Pulse Sequence: gCOSY
Solvent: cdcl3
Data collected on: Sep 15 2011
Operator: amanfran
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 7998.4 Hz
2D Width 7998.4 Hz
2 repetitions
128 increments
OBSERVE H1, 499.9042537 MHz
DATA PROCESSING
Sq. sine bell 0.064 sec
F1 DATA PROCESSING
Sq. sine bell 0.016 sec
FT size 2048 x 2048
Total time 5 min 41 sec
O
22
Agilent Technologies
F1 (ppm)
F2 (ppm)

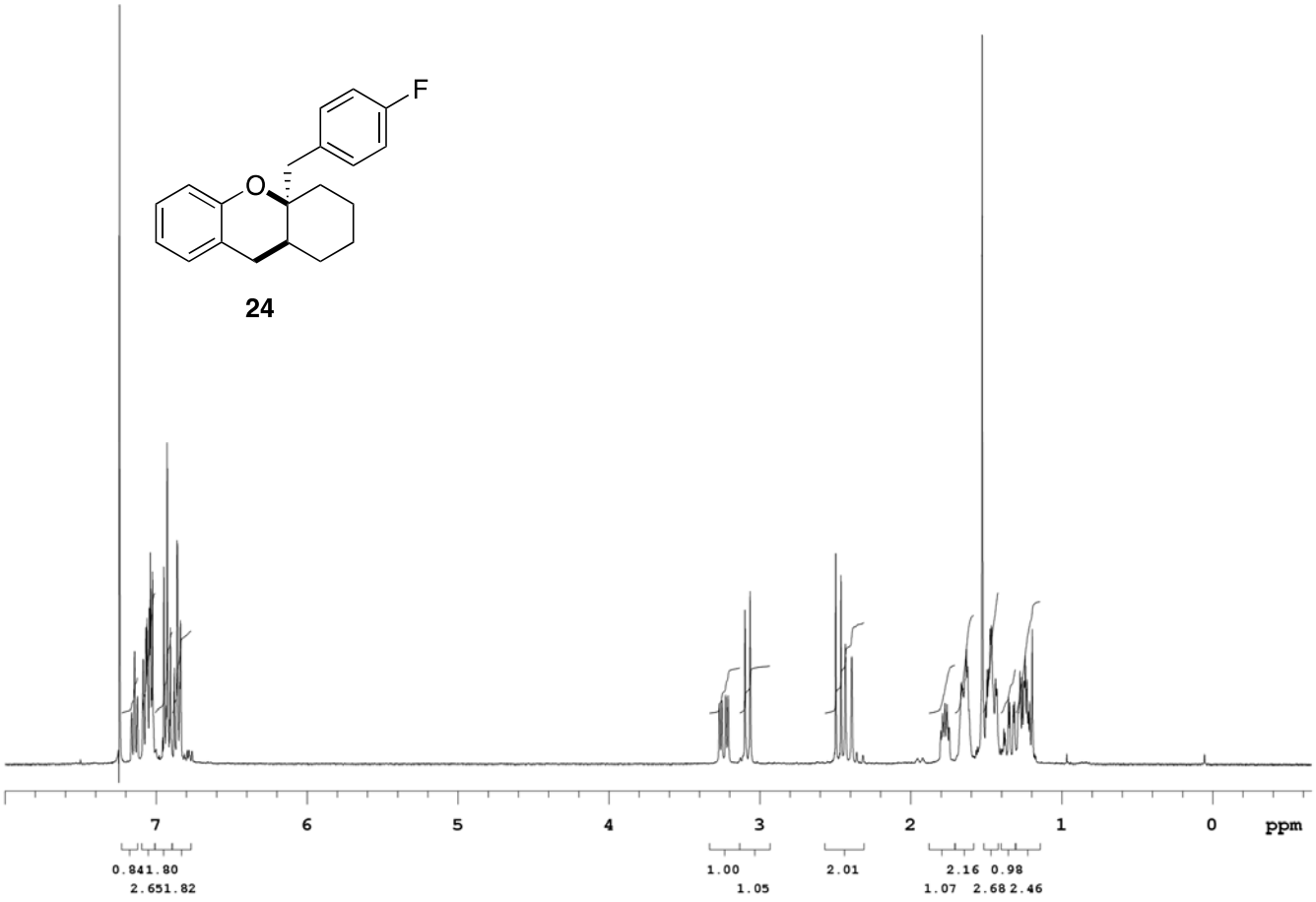

STANDARD CARBON PARAMETERS
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: aw5-10-1-noesy
Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Sep 15 2011
Operator: amanfran
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 7998.4 Hz
2D Width 7998.4 Hz
4 repetitions
2 x 200 increments
OBSERVE H1, 499.9042537 MHz
DATA PROCESSING
Gauss apodization 0.059 sec
F1 DATA PROCESSING
Gauss apodization 0.023 sec
FT size 2048 x 2048
Total time 37 min
O
22
Agilent Technologies
F1
(ppm)
0
1
2
3
4
5
6
7
4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
F2 (ppm)

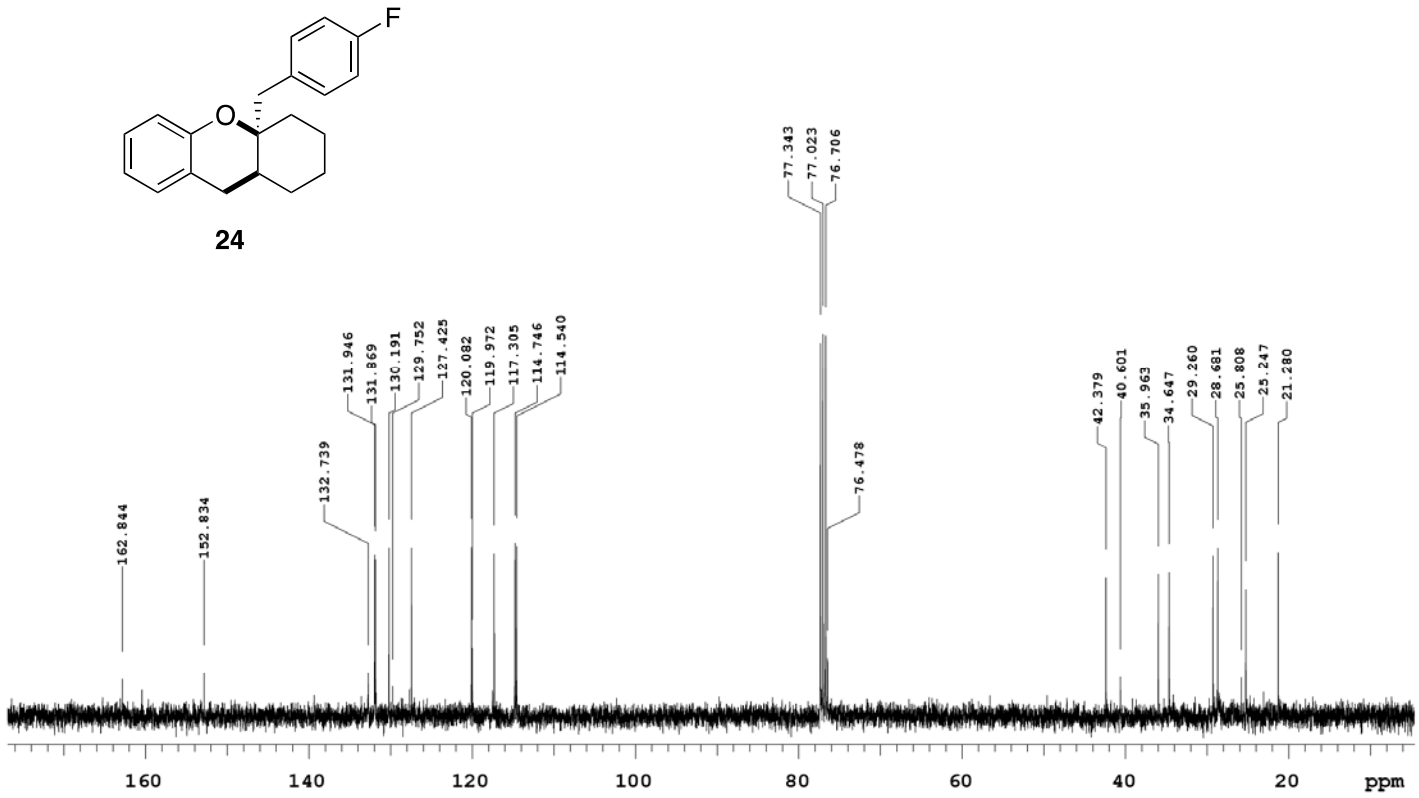


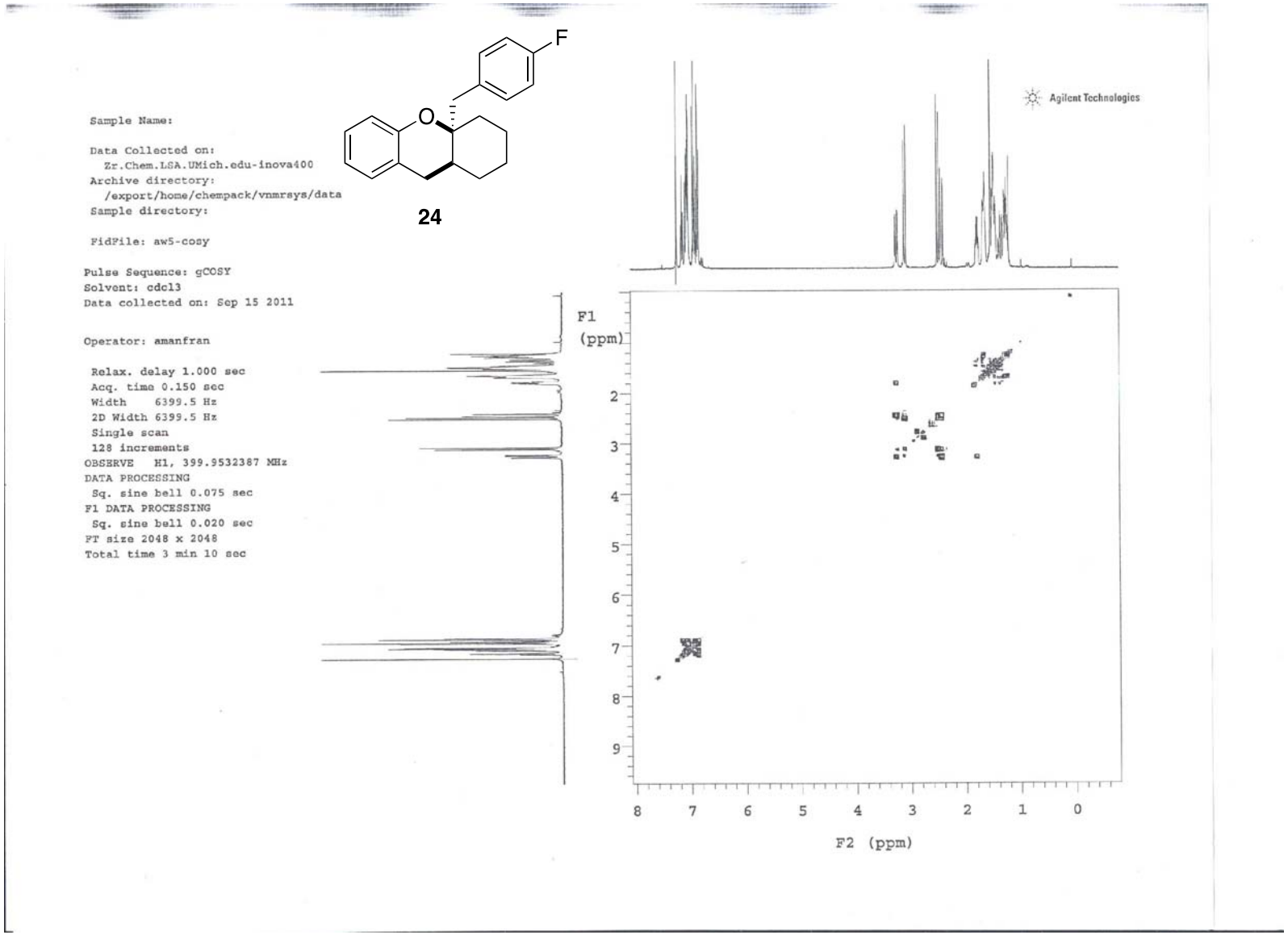


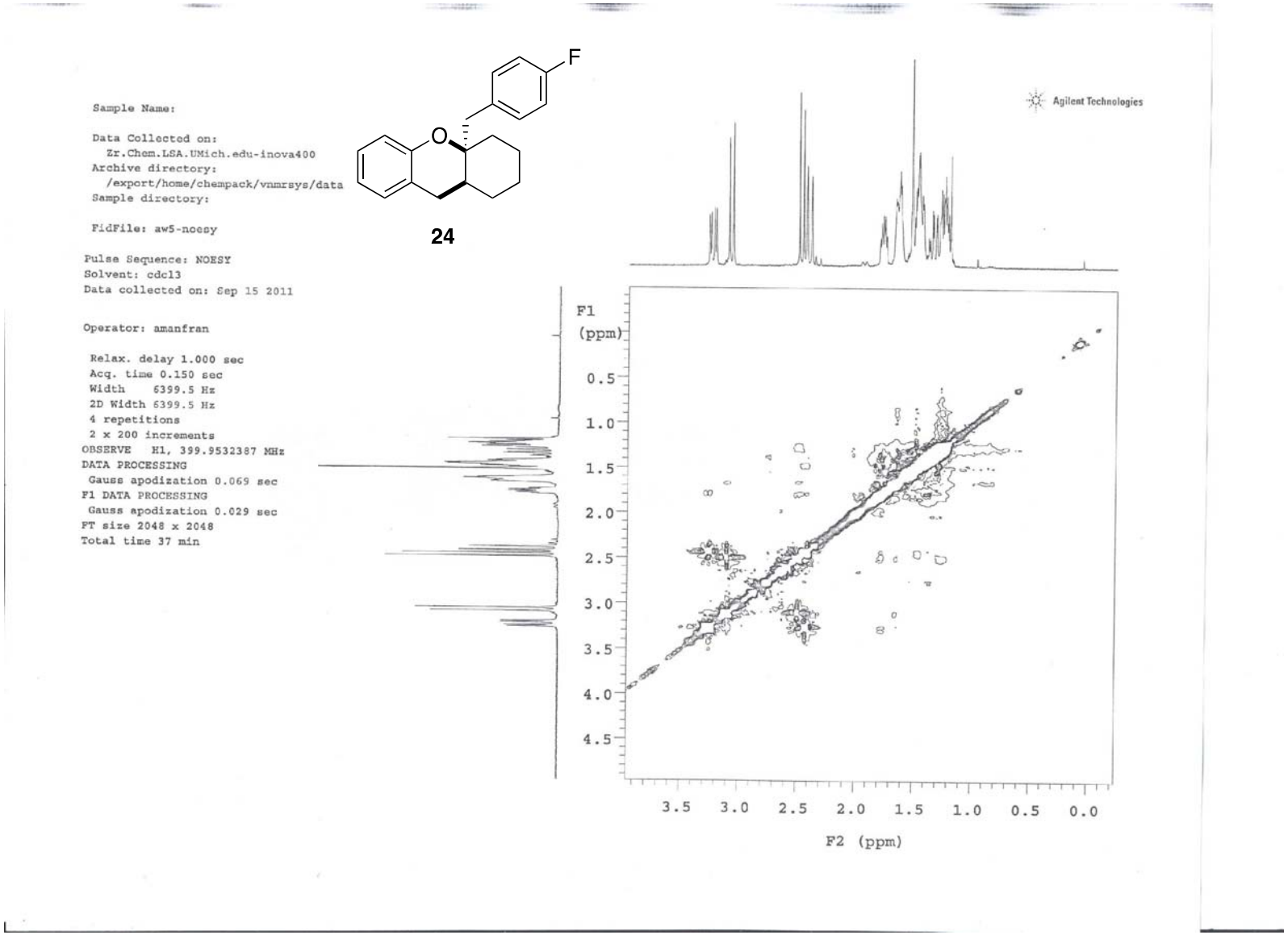
23

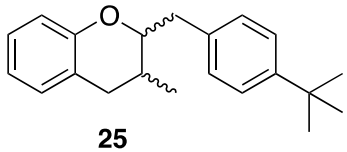




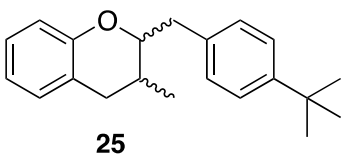



F
O
24
Agilent Technologies
Sample Name:
Data Collected on:
Zr.Chem.LSA.UMich.edu-inova400
Archive directory:
/export/home/chempack/vnmrsys/data
Sample directory:
FidFile: aw5-cosy
Pulse Sequence: gCOSY
Solvent: cdcl3
Data collected on: Sep 15 2011
Operator: amanfran
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6399.5 Hz
2D Width 6399.5 Hz
Single scan
128 increments
OBSERVE H1, 399.9532387 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.020 sec
FT size 2048 x 2048
Total time 3 min 10 sec
F1 (ppm)
F2 (ppm)


Sample Name:
Data Collected on:
Zr.Chem.LSA.UMich.edu-inova400
Archive directory:
/export/home/chempack/vnmrsys/data
Sample directory:
FidFile: aw5-noesy
Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Sep 15 2011
Operator: amanfran
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6399.5 Hz
2D Width 6399.5 Hz
4 repetitions
2 x 200 increments
OBSERVE H1, 399.9532387 MHz
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.029 sec
FT size 2048 x 2048
Total time 37 min
F
O
24
Agilent Technologies
F1
(ppm)
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
F2 (ppm)



25



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