

Supporting Information

Exploration of One-Pot, Tandem Sulfamoylation and *Aza*-Michael Cyclization Reactions for the Syntheses of Oxathiazinane Dioxide Heterocycles

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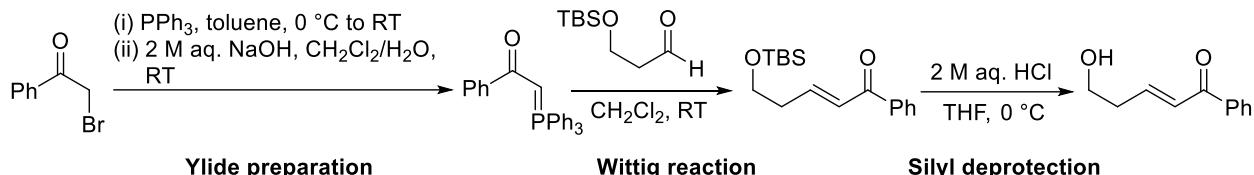
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I. General Considerations

All reagents were obtained commercially unless otherwise noted. Solvents were purified by passage under 10 psi N₂ through activated alumina columns. Infrared (IR) spectra were recorded on a Thermo Scientific™ Nicolet™ iS™5 FT-IR Spectrometer; data are reported in frequency of absorption (cm⁻¹). ¹H NMR spectra were recorded at 400, 500, or 600 MHz. Data are recorded as: chemical shift in ppm referenced internally using residual solvent peaks, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances, qdd = quartet of doublet of doublets, tdt = triplet of doublet of triplets, dtq = doublet of triplet of quartets, qd = quartet of doublets, tdq = triplet of doublet of quartets), integration, coupling constant (Hz). ¹³C NMR spectra were recorded at 101 or 126 MHz. Exact mass spectra were recorded using an electrospray ion source (ESI) either in positive mode or negative mode and with a time-of-flight (TOF) analyzer on a Waters LCT PremierTM mass spectrometer and are given in m/z. Thin Layer Chromatography (TLC) was performed on pre-coated glass plates (Merck) and visualized either with a UV lamp (254 nm) or by dipping into a solution of KMnO₄–K₂CO₃ in water followed by heating. Flash chromatography was performed on silica gel (230-400 mesh) or Florisil (60-100 mesh).

II. Representative Procedures for Substrate Syntheses and General Procedures for One-Pot Cascade Reactions (Schemes 2-4)

Representative Sequence for Starting Material Preparation (Type I):



Ylide preparation: A solution of triphenylphosphine (5.25 g, 20.0 mmol, 1.0 equivalent) in toluene (15 mL) was added dropwise to a magnetically-stirred solution of α -bromoacetophenone (3.98 g, 20.0 mmol, 1.0 equivalent) in toluene (15 mL) at 0 °C (maintained using an ice-water bath). The reaction mixture was allowed to warm to room temperature with stirring over a period of 18 h. The resulting phosphonium salt was filtered, washed with diethyl ether (3 x 100 mL), and dried. The phosphonium salt was obtained in quantitative yield and was used without further purification.

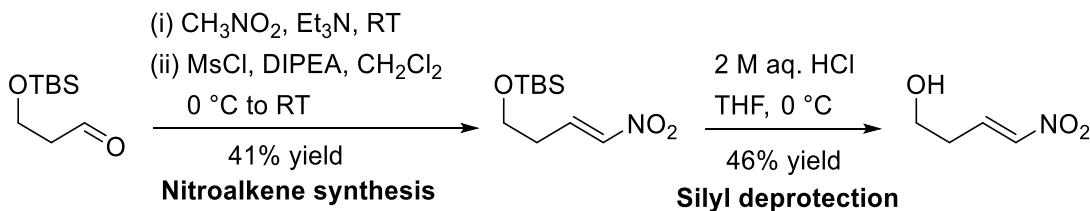
The phosphonium salt was then dissolved in a mixture of H₂O/CH₂Cl₂ (1.5:1, 150 mL total volume) and 2.5 M aqueous NaOH solution (100 mL) was added. The mixture was stirred for 3 h at ambient temperature and then transferred to a separatory funnel. The organic phase was separated, and the aqueous phase was extracted with CH₂Cl₂ (3×100 mL). The combined organic phases were washed with brine, dried with MgSO₄, and concentrated *in vacuo* to afford the desired ylide as a white solid (7.02 g, 18.45 mmol, 92% yield), which was used without further purification.

Wittig Reaction: To a magnetically-stirred solution of 3-((tert-butyldimethylsilyl)oxy)propanal (2.0 g, 10.62 mmol, 1.0 equivalent) in CH₂Cl₂ (40.0 mL, ≈0.27 M, relative to the aldehyde) was added the ylide reagent (4.4 g, 11.6 mmol, 1.1 equiv.) at room temperature. The reaction mixture was stirred until TLC indicated complete consumption of the starting aldehyde (24 hours). Next, the reaction mixture was carefully transferred to a separatory funnel using CH₂Cl₂ and washed with water. The organic layer was separated, and the aqueous layer was extracted three times with CH₂Cl₂. The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting residue was purified using a gradient of 0-4% EtOAc/hexanes on silica gel to yield the desired product (1.9 g, 6.54 mmol, 62% yield).

Silyl deprotection: A round-bottom flask was charged with a stir-bar, (*E*)-5-((tert-butyldimethylsilyl)oxy)-1-phenypent-2-en-1-one (1.7 g, 5.85 mmol, 1.0 equiv.), and THF (20.0 mL, ≈0.3 M). The resulting solution was cooled to 0 °C using an ice-water bath and stirred for 10 minutes at this temperature. Then, 2 M aqueous HCl solution (30.0 mL, 60 mmol, ~10 equivalents) was added. The reaction mixture was stirred for 30 minutes at the same temperature. Following this time, the reaction mixture was carefully transferred to a separatory funnel using EtOAc. The aqueous layer was removed, and the organic layer was washed with water. The organic layer was collected, dried over anhydrous Na₂SO₄, and concentrated under reduced pressure. The resulting residue was purified using a gradient of 5-40% EtOAc/hexanes on silica gel to yield the desired product (833 mg, 4.73 mmol, 81% yield).

Note: The reaction requires a longer time (24 hours) to complete for secondary silyl ether substrates. For secondary silyl ether substrates, the ice-bath was removed after the addition of 2 M aqueous HCl solution and the reaction was allowed to stir at room temperature for 24 hours.

Representative Sequence for Starting Material Preparation (Type 2):



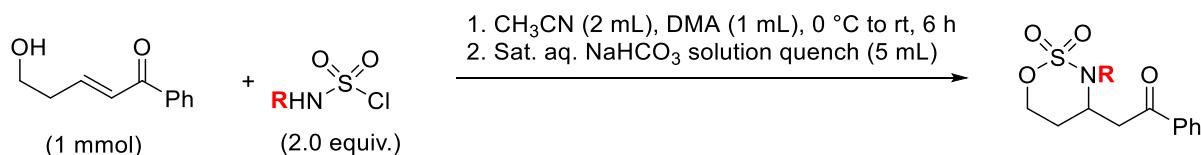
Nitroalkene synthesis: An oven-dried round-bottom flask cooled under nitrogen gas was charged with a stir bar, 3-((tert-butyldimethylsilyl)oxy)propanal (4.7 g, 25.0 mmol, 1 equiv.) and nitromethane (6.8 mL, 7.75 g, 127 mmol, ~5 equivalents). The mixture was stirred at room temperature, and triethylamine (1.05 mL, 0.762 g, 7.5 mmol, 0.3 equiv.) was added dropwise. After stirring for 8 h at the same temperature, the reaction mixture was filtered through a pad of silica gel using 20% EtOAc in hexane. The filtrate was concentrated to yield the crude nitro-alcohol, which was used in the next step without further purification.

The crude nitro-alcohol was dissolved in CH₂Cl₂ (100 mL) and added to a 250 mL round-bottom flask equipped with a magnetic stir bar. The flask was cooled to 0 °C using an ice-water bath. Methanesulfonyl chloride (3.87 mL, 5.73 g, 50.0 mmol, 2 equiv. [relative to starting aldehyde]) and diisopropylethylamine (15.0 mL, 11.13 g, 86.1 mmol, 3.4 equiv. [relative to the starting aldehyde]) were added successively. The mixture was stirred at 0 °C for 30 min. The ice-water bath was removed, and the mixture was allowed to warm to room temperature over a period of 15 minutes with stirring. Following this time, the reaction mixture was transferred to a separatory funnel. Brine (60 mL) was added, and the organic phase was separated. The aqueous layer was extracted with CH₂Cl₂ (60 mL x 3). The combined organic layers were dried over MgSO₄, filtered, and concentrated under reduced pressure. The resulting residue was purified using a gradient of 0-5% EtOAc/hexanes on silica gel to yield the desired nitroalkene ((E)-tert-butyldimethyl((4-nitrobut-3-en-1-yl)oxy)silane) as a yellow oil (2.37 g, 10.24 mmol, 41% yield over two steps).

Silyl Deprotection

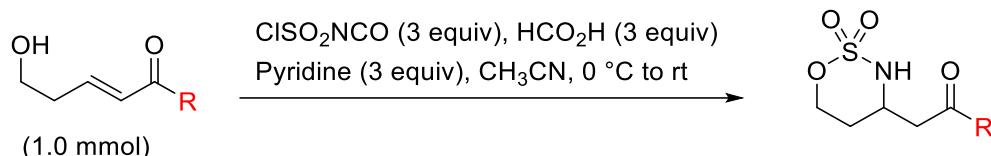
Note: For TBS-deprotection of (E)-tert-butyldimethyl((4-nitrobut-3-en-1-yl)oxy)silane, please refer to the silyl deprotection procedure given in the **representative sequence for starting material preparation (type 1)**.

General Procedure A: One-Pot Cascade Reactions (Type 1)



An oven-dried glass vial was fitted with a nitrogen balloon and charged with a stir bar, δ -hydroxy enone (1.0 mmol, 1 equivalent), and DMA (1 mL). The reaction mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. Subsequently, a solution of appropriate sulfamoyl chloride (2.0 mmol, 2.0 equivalent, prepared from the procedure of Kloek, J.A; Leschinsky, K.L. *J. Org. Chem.* **1976**, *41*, 4028-4029) in CH₃CN (2 mL) was added to it in a dropwise manner (final reaction concentration 0.33 M, relative to δ -hydroxy enone). The ice-bath was removed, and the reaction mixture was stirred for 6 h. Following this time, the reaction was quenched by the addition of saturated aqueous NaHCO₃ solution (5 mL) and vigorously stirred for an additional 10 min. Following this time, the mixture was carefully transferred to a separatory funnel using ethyl acetate (15 mL) and further diluted with 5 mL of water. The biphasic mixture was shaken well, and the organic layer was separated. The aqueous layer was extracted with ethyl acetate (15 mL x 3). The combined organic layers were concentrated under reduced pressure until a volume of 15-20 mL and then transferred to a separatory funnel. The organic layer was washed with an ice-cold aqueous solution of ~10% w/w LiCl (15 mL x 2). The organic layer was separated, dried over anhydrous Na₂SO₄, and fully concentrated *in vacuo*. The resulting residue was purified on silica gel to yield the desired cyclization product (specific purification conditions are given with each product).

General Procedure B: One-Pot Cascade Reactions (Type 2)

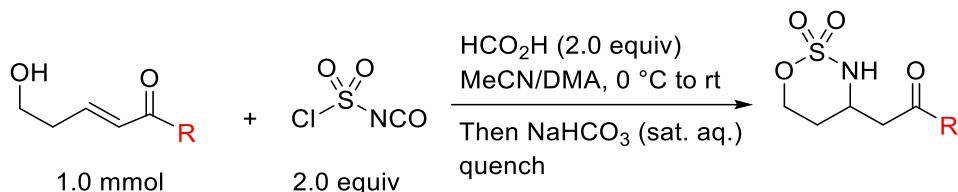


Preparation of CISO₂NH₂: An oven-dried 5 mL glass vial fitted with a balloon of N₂ gas was charged with a stir bar and CISO₂NCO (261.0 μ L, 0.425 g, 3.0 mmol, 3.0 equiv). The reaction mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. HCO₂H (113.0 μ L, 0.138 g, 3.0 mmol, 3.0 equiv) was added dropwise (**Caution: vigorous gas evolution upon addition**). The mixture solidified into a white solid within 5-10 minutes of addition. CH₃CN (2 ml) was added to it, and the reaction mixture was warmed to room temperature over a period of 16 h.

Cyclization: A separate oven-dried round bottom flask fitted with a nitrogen balloon was charged with a stir bar, appropriate alcohol (1.0 mmol, 1.0 equiv), and CH₃CN (2 ml). The flask was cooled to 0 °C using an ice-water bath. Next, pyridine (242.0 μ L, 0.237 g, 3.0 mmol, 3.0 equiv) was added to it, and the reaction mixture was stirred at same temperature for 5 minutes. Then freshly prepared CISO₂NH₂ (in CH₃CN) was added dropwise, and the vial was rinsed by adding another 1 mL of CH₃CN (Final reaction concentration = 0.2 M relative to δ -hydroxy enone). With stirring, the reaction mixture was warmed to room temperature over a period of 16 h. Following this time, the reaction was quenched by the addition of 1 M aqueous HCl solution (5 mL). The mixture was transferred to a separatory funnel with EtOAc and further diluted with water. The organic layer was collected. The aqueous layer was extracted with EtOAc (3 x 5 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄, and concentrated *in vacuo*.

The resulting residue was purified on silica gel to yield the desired cyclization product (specific conditions are associated with each product).

General Procedure C: One-Pot Cascade Reactions (Type 3)

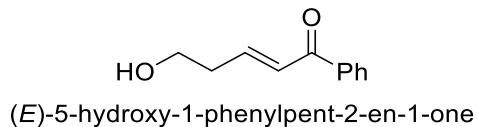


Preparation of ClSO_2NH_2 : An oven-dried glass vial equipped with a stir bar was fitted with a balloon of N_2 gas. ClSO_2NCO (174.0 μL , 0.283 g, 2.0 mmol, 2.0 equiv) was added to it, and the vial was cooled to 0°C using an ice-water bath. HCO_2H (76.0 μL , 0.092 g, 2.0 mmol, 2.0 equiv) was added dropwise with stirring (**Caution: vigorous gas evolution upon addition**). The mixture solidified into a white solid within 5-10 minutes of addition. CH_3CN (1.7 mL) was added, and the reaction mixture was slowly warmed to room temperature over a period of 16 h with constant stirring.

Cyclization: A separate oven-dried glass vial fitted with a N_2 gas balloon was charged with a stir bar, δ -hydroxy enone (1.0 mmol, 1 equiv), and DMA (1.1 mL). The reaction mixture was cooled to 0°C using an ice-water bath and stirred at this temperature for 10 minutes. Then, freshly prepared ClSO_2NH_2 (in CH_3CN) was added dropwise (final reaction concentration = 0.36 M, relative to δ -hydroxy enone). The ice-water bath was removed, and the reaction mixture was stirred for 6 h. Following this time, the reaction was quenched by the addition of saturated aqueous NaHCO_3 solution (5 mL) and vigorously stirred for another 10 minutes. The mixture was carefully transferred to a separatory funnel using EtOAc (15 mL) and further diluted with 10 mL of water. The biphasic mixture was shaken well, and the organic layer was separated. The aqueous layer was extracted with ethyl acetate (15 mL x 3 times). The combined organic layers were concentrated under reduced pressure to a volume of ~15 mL. After transfer to a separatory funnel, the organic layer was washed with cold water (30 mL). The organic layer was collected, dried over anhydrous Na_2SO_4 , and concentrated *in vacuo*. The resulting residue was purified on silica gel (specific conditions are associated with each product).

Note: Equivalents are varied appropriately for scales different from the representative procedures.

III. Characterization of Substrates and Products (Schemes 2-4)



Compound 1: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

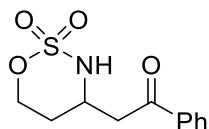
¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.88 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.41 (m, 2H), 7.13 – 6.90 (m, 2H), 3.83 (t, *J* = 6.2 Hz, 2H), 2.59 (q, *J* = 6.2 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.7, 145.8, 137.8, 132.9, 128.7, 128.6, 128.1, 61.1, 36.1.

IR ν 1667, 1618, 1446, 1349, 1285, 1036, 970, 734, 694 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₁H₁₂O₂Na⁺ 199.0735. Found 199.0764 (2.9 mmu error).

Previously characterized in *J. Am. Chem. Soc.* **2008**, *130*, 46-48. Our characterization data matches the literature.



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

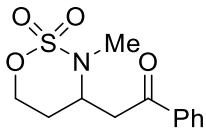
Compound 2: Synthesized using **General Procedure A** on a 200 mg (1.13 mmol) scale; Purified using a gradient of 10 to 40% EtOAc/hexanes on silica gel; (white solid, 220 mg, 0.86 mmol, 76% yield).

¹H NMR (400 MHz, CD₂Cl₂) δ 7.98 – 7.92 (m, 2H), 7.67 – 7.60 (m, 1H), 7.51 (dd, *J* = 8.5, 7.1 Hz, 2H), 5.25 (d, *J* = 10.0 Hz, 1H), 4.74 (ddd, *J* = 12.6, 11.6, 2.5 Hz, 1H), 4.57 (ddd, *J* = 11.6, 4.9, 2.0 Hz, 1H), 4.29 – 4.18 (m, 1H), 3.40 (dd, *J* = 18.0, 4.7 Hz, 1H), 3.26 (dd, *J* = 18.0, 5.9 Hz, 1H), 2.08 (dtd, *J* = 14.4, 12.2, 5.0 Hz, 1H), 1.85 – 1.75 (m, 1H).

¹³C{¹H} NMR (101 MHz, CD₂Cl₂) δ 198.2, 136.7, 134.4, 129.2, 128.4, 72.6, 53.3, 41.9, 28.8.

IR ν 3243, 1670, 1596, 1369, 1174, 996, 754, 686 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₁H₁₃NO₄SnA⁺ 278.0463. Found 278.0485 (2.2 mmu error).



2-(3-methyl-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

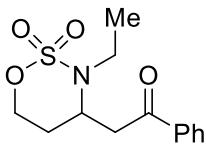
Compound 3: Synthesized using **General Procedure A** on a 177 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 206 mg, 0.76 mmol, 76% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.00 – 7.93 (m, 2H), 7.64 – 7.57 (m, 1H), 7.49 (dd, *J* = 8.4, 7.1 Hz, 2H), 4.71 – 4.52 (m, 3H), 3.53 (dd, *J* = 17.4, 4.2 Hz, 1H), 3.30 (dd, *J* = 17.3, 9.3 Hz, 1H), 2.90 (s, 3H), 2.09 – 1.99 (m, 1H), 1.91 – 1.78 (m, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 196.3, 136.3, 133.9, 128.9, 128.2, 71.1, 56.4, 41.5, 34.2, 23.3.

IR ν 3034, 1670, 1598, 1420, 1354, 1260, 1168, 1016, 990, 772 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₆NO₄S⁺ 270.0800. Found 270.0805 (1.8 ppm error).



2-(3-ethyl-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

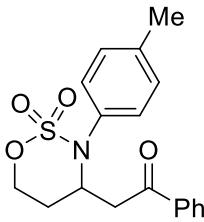
Compound 4: Synthesized using **General Procedure A** on a 177 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 189 mg, 0.66 mmol, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.04 – 7.96 (m, 2H), 7.64 – 7.57 (m, 1H), 7.49 (dd, *J* = 8.3, 7.0 Hz, 2H), 4.76 (ddd, *J* = 11.8, 9.7, 3.2 Hz, 1H), 4.61 – 4.50 (m, 1H), 4.45 (dq, *J* = 9.4, 4.8 Hz, 1H), 3.71 (dd, *J* = 17.8, 4.5 Hz, 1H), 3.45 (dd, *J* = 17.9, 8.9 Hz, 1H), 3.34 (q, *J* = 7.1 Hz, 2H), 2.25 (ddt, *J* = 14.6, 9.6, 4.8 Hz, 1H), 1.70 (dtd, *J* = 14.8, 5.1, 3.3 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 197.1, 136.4, 133.8, 128.9, 128.2, 70.1, 55.1, 45.3, 41.6, 25.2, 14.9.

IR ν 3269, 1676, 1596, 1372, 1217, 1179, 987, 769, 686 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₈NO₄S⁺ 284.0957. Found 284.0966 (3.2 ppm error).



2-(2,2-dioxido-3-(*p*-tolyl)-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

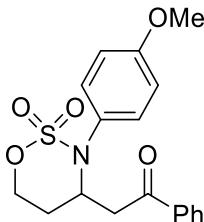
Compound 5: Synthesized using **General Procedure A** on a 177 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 182 mg, 0.52 mmol, 52% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.85 – 7.76 (m, 2H), 7.60 – 7.53 (m, 1H), 7.47 – 7.39 (m, 2H), 7.36 – 7.27 (m, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 4.93 – 4.70 (m, 3H), 3.28 (dd, *J* = 17.6, 4.1 Hz, 1H), 3.03 (dd, *J* = 17.6, 9.3 Hz, 1H), 2.34 (s, 3H), 2.30 – 2.22 (m, 1H), 2.11 – 2.01 (m, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 196.4, 139.0, 136.3, 135.8, 133.7, 130.3, 128.8, 128.7, 128.0, 71.6, 58.2, 41.8, 28.3, 21.2.

IR ν 1687, 1596, 1377, 1240, 1174, 1010, 978, 766, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₈H₂₀NO₄S⁺ 346.1113. Found 346.1107 (1.7 ppm error).



2-(3-(4-methoxyphenyl)-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

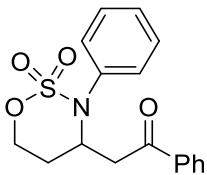
Compound 6: Synthesized using **General Procedure A** on a 177 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 187 mg, 0.51 mmol, 51% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.83 – 7.75 (m, 2H), 7.59 – 7.53 (m, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.32 (m, 2H), 6.93 – 6.83 (m, 2H), 4.89 (td, *J* = 11.1, 3.2 Hz, 1H), 4.79 (ddd, *J* = 9.3, 5.4, 3.9 Hz, 1H), 4.71 (dt, *J* = 11.6, 4.3 Hz, 1H), 3.79 (s, 3H), 3.25 (dd, *J* = 17.5, 4.2 Hz, 1H), 2.97 (ddd, *J* = 17.5, 9.1, 1.4 Hz, 1H), 2.29 – 2.18 (m, 1H), 2.14 – 2.00 (m, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 196.4, 159.8, 136.3, 133.8, 130.7, 130.3, 128.8, 128.0, 114.8, 71.7, 58.2, 55.6, 41.9, 28.5.

IR ν 1687, 1507, 1377, 1240, 1182, 1010, 978, 763, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₈H₂₀NO₅S⁺ 362.1062. Found 362.1071 (2.4 ppm error).



2-(2,2-dioxido-3-phenyl-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

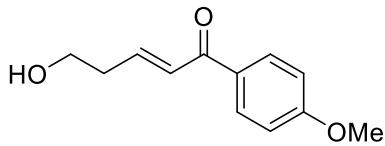
Compound 7: Synthesized using **General Procedure A** on a 177 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 181 mg, 0.54 mmol, 54% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.85 – 7.76 (m, 2H), 7.59 – 7.54 (m, 1H), 7.48 – 7.32 (m, 7H), 4.98 – 4.71 (m, 3H), 3.30 (dd, *J* = 17.6, 4.1 Hz, 1H), 3.07 (dd, *J* = 17.6, 9.2 Hz, 1H), 2.36 – 2.25 (m, 1H), 2.15 – 1.99 (m, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 196.4, 138.7, 136.3, 133.8, 129.7, 128.94, 128.91, 128.8, 128.0, 71.6, 58.4, 41.7, 28.1.

IR v 3276, 1687, 1492, 1377, 1179, 967, 772, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₇H₁₈NO₄S⁺ 332.0957. Found 332.0953 (1.2 ppm error).



(E)-5-hydroxy-1-(4-methoxyphenyl)pent-2-en-1-one

Compound 8: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

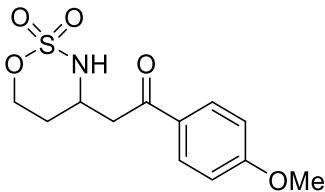
¹H NMR (400 MHz, CDCl₃) δ 7.94 (dd, *J* = 8.9, 2.4 Hz, 2H), 7.07 – 6.95 (m, 2H), 6.93 (dd, *J* = 9.0, 2.3 Hz, 2H), 3.86 (s, 3H), 3.82 (dd, *J* = 7.6, 5.5 Hz, 2H), 2.73 – 2.36 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 188.9, 163.5, 144.7, 131.0, 130.6, 127.8, 113.9, 61.2, 55.5, 36.1.

IR v 3441, 1656, 1593, 1564, 1418, 1245, 1036, 970, 826, 680 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₅O₃⁺ 207.1021. Found 207.1042 (2.1 mmu error).

Previously characterized in *J. Am. Chem. Soc.* **2008**, *130*, 46-48. Our characterization data matches the literature.



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-(4-methoxyphenyl)ethan-1-one

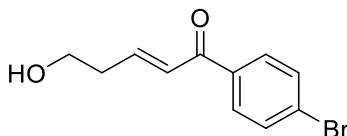
Compound 9: Synthesized using **General Procedure B** on a 207 mg (1.00 mmol) scale; Purified using a gradient of 5 to 35% EtOAc/hexanes on silica gel; (white solid, 197 mg, 0.69 mmol, 69% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, *J* = 8.8 Hz, 2H), 6.95 (dd, *J* = 8.8, 1.9 Hz, 2H), 5.59 – 5.33 (m, 1H), 4.75 (ddd, *J* = 13.8, 11.9, 2.4 Hz, 1H), 4.56 (ddd, *J* = 11.6, 5.0, 1.8 Hz, 1H), 4.26 – 4.17 (m, 1H), 3.88 (s, 3H), 3.34 (ddd, *J* = 17.6, 4.4, 1.5 Hz, 1H), 3.19 (dd, *J* = 17.6, 6.0 Hz, 1H), 2.19 – 2.02 (m, 1H), 1.77 (dt, *J* = 14.3, 2.5 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 196.3, 164.3, 130.6, 129.3, 114.1, 72.0, 55.7, 53.0, 40.9, 28.4.

IR ν 1670, 1598, 1420, 1354, 1168, 1016, 990, 866, 772 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₆NO₅S⁺ 286.0749. Found 286.0750 (0.3 ppm error).



(*E*)-1-(4-bromophenyl)-5-hydroxypent-2-en-1-one

Compound 10: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

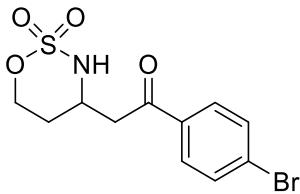
¹H NMR (400 MHz, CDCl₃) δ 7.84 – 7.74 (m, 2H), 7.59 (dd, *J* = 8.5, 1.7 Hz, 2H), 7.06 (dt, *J* = 15.2, 6.9 Hz, 1H), 6.93 (dd, *J* = 15.4, 1.3 Hz, 1H), 3.83 (t, *J* = 6.2 Hz, 2H), 2.58 (q, *J* = 6.3 Hz, 2H), 1.95 (broad s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 189.5, 146.5, 136.5, 132.0, 130.2, 128.0, 127.5, 61.1, 36.1.

IR ν 3295, 1667, 1618, 1581, 1340, 1257, 1021, 964, 818, 726, 665 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₁H₁₁BrO₂Na⁺ 276.9840. Found 276.9870 (3.0 mmu error).

Previously characterized in *Chem. Commun.*, **2015**, *51*, 11693 – 11696. Our data matches the literature.



1-(4-bromophenyl)-2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)ethan-1-one

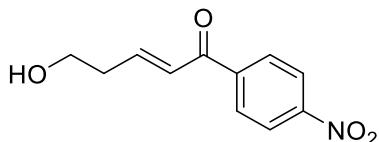
Compound 11: Synthesized using **General Procedure B** on a 256 mg (1.00 mmol) scale; Purified using a gradient of 10 to 40% EtOAc/hexanes on silica gel; (white solid, 251 mg, 0.75 mmol, 75% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.92 – 7.82 (m, 2H), 7.74 – 7.65 (m, 2H), 5.31 (d, *J* = 10.2 Hz, 1H), 4.73 – 4.62 (m, 1H), 4.58 (ddd, *J* = 11.6, 4.8, 2.5 Hz, 1H), 4.21 (ttd, *J* = 10.4, 6.5, 4.0 Hz, 1H), 3.33 (dd, *J* = 17.6, 6.5 Hz, 1H), 3.18 (dd, *J* = 17.7, 6.3 Hz, 1H), 1.91 – 1.76 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 196.9, 136.6, 132.9, 130.7, 128.9, 73.3, 53.4, 43.2, 29.6.

IR v 3263, 1687, 1584, 1349, 1240, 1176, 1064, 987, 772, 628 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₁H₁₂BrNO₄SNa⁺ 355.9568. Found 355.9594 (2.6 mmu error).



(*E*)-5-hydroxy-1-(4-nitrophenyl)pent-2-en-1-one

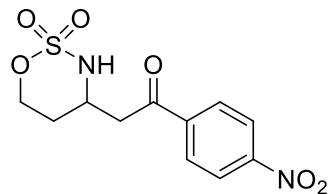
Compound 12: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 8.31 (d, *J* = 8.8 Hz, 2H), 8.06 (d, *J* = 8.8 Hz, 2H), 7.12 (dt, *J* = 15.5, 7.0 Hz, 1H), 6.97 (dt, *J* = 15.5, 1.3 Hz, 1H), 3.87 (t, *J* = 6.1 Hz, 2H), 2.62 (qd, *J* = 6.2, 1.4 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 189.1, 150.2, 148.3, 142.6, 129.6, 127.5, 123.9, 61.0, 36.1.

IR v 3519, 1667, 1601, 1521, 1346, 1217, 1033, 964, 846, 709 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₁H₁₂NO₄⁺ 222.0766. Found 222.0786 (2.0 mmu error).



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-(4-nitrophenyl)ethan-1-one

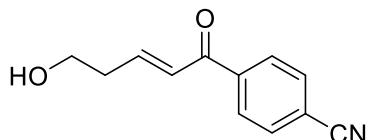
Compound 13: Synthesized using **General Procedure B** on a 222 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 214 mg, 0.71 mmol, 71% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.39 – 8.32 (m, 2H), 8.21 – 8.13 (m, 2H), 5.37 (d, *J* = 10.3 Hz, 1H), 4.80 – 4.69 (m, 1H), 4.64 (ddd, *J* = 11.7, 4.8, 2.4 Hz, 1H), 4.27 (ddt, *J* = 13.1, 6.6, 3.4 Hz, 1H), 3.46 (dd, *J* = 17.9, 6.6 Hz, 1H), 3.32 (dd, *J* = 17.9, 6.3 Hz, 1H), 2.00 – 1.83 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 196.7, 151.5, 142.0, 130.1, 124.7, 73.3, 53.2, 43.8, 29.6.

IR ν 3232, 1690, 1515, 1317, 1288, 1182, 1067, 921, 789, 686 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₁H₁₂N₂O₆SnA⁺ 323.0314. Found 323.0336 (2.2 mmu error).



(*E*)-4-(5-hydroxypent-2-enoyl)benzonitrile

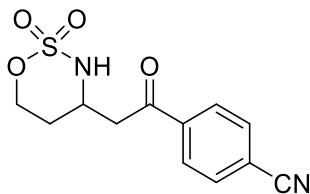
Compound 14: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 8.02 – 7.96 (m, 2H), 7.80 – 7.73 (m, 2H), 7.10 (dt, *J* = 15.4, 6.9 Hz, 1H), 7.01 – 6.88 (m, 1H), 3.85 (t, *J* = 6.1 Hz, 2H), 2.67 – 2.52 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 189.3, 148.0, 141.0, 132.5, 129.0, 127.3, 118.1, 116.0, 60.9, 36.1.

IR ν 2230, 1667, 1616, 1507, 1346, 1240, 1013, 970, 800, 663 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₂NO₂⁺ 202.0868. Found 202.0877 (4.4 ppm error).



4-(2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)acetyl)benzonitrile

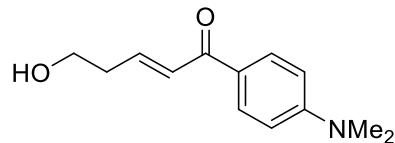
Compound 15: Synthesized using **General Procedure B** on a 202 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 217 mg, 0.77 mmol, 77% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.10 – 8.01 (m, 2H), 7.91 – 7.83 (m, 2H), 5.31 (d, *J* = 10.2 Hz, 1H), 4.68 (td, *J* = 11.6, 3.4 Hz, 1H), 4.58 (ddd, *J* = 11.7, 4.8, 2.4 Hz, 1H), 4.22 (ddt, *J* = 10.3, 6.5, 3.9 Hz, 1H), 3.38 (dd, *J* = 17.8, 6.6 Hz, 1H), 3.24 (dd, *J* = 17.8, 6.3 Hz, 1H), 1.92 – 1.77 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 197.0, 140.6, 133.7, 129.4, 118.9, 117.2, 73.3, 53.3, 43.6, 29.6.

IR ν 3275, 1684, 1418, 1352, 1285, 1182, 1064, 990, 864, 798, 694 cm⁻¹.

HRMS (ESI) m/z = [M - H]⁻ Calcd C₁₂H₁₁N₂O₄S⁻ 279.0440. Found 279.0432 (2.9 ppm error).



(*E*)-1-(4-(dimethylamino)phenyl)-5-hydroxypent-2-en-1-one

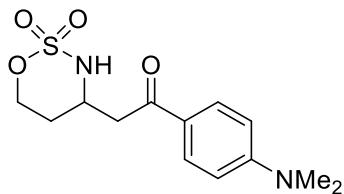
Compound 16: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, *J* = 9.0 Hz, 2H), 7.07 – 6.92 (m, 2H), 6.66 (dd, *J* = 9.0, 1.4 Hz, 2H), 3.81 (t, *J* = 6.3 Hz, 2H), 3.05 (s, 6H), 2.56 (q, *J* = 6.3 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 188.1, 153.4, 142.9, 131.0, 127.9, 125.5, 110.9, 61.3, 40.1, 36.2.

IR ν 3456, 1647, 1607, 1590, 1346, 1234, 1039, 964, 812, 800 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₈NO₂⁺ 220.1338. Found 220.1359 (2.1 mmu error).



1-(4-(dimethylamino)phenyl)-2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)ethan-1-one

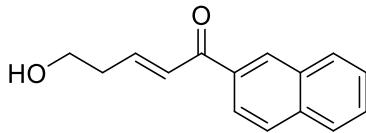
Compound 17: Synthesized using **General Procedure B** on a 220 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 53 mg, 0.17 mmol, 17% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.86 – 7.80 (m, 2H), 6.76 – 6.68 (m, 2H), 5.43 (d, *J* = 10.1 Hz, 1H), 4.72 – 4.61 (m, 1H), 4.61 – 4.52 (m, 1H), 4.16 (ddq, *J* = 10.1, 8.8, 6.2 Hz, 1H), 3.24 (dd, *J* = 16.9, 6.3 Hz, 1H), 3.09 – 2.99 (m, 7H), 1.90 – 1.80 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 195.4, 154.9, 131.1, 125.2, 111.6, 73.4, 54.1, 42.0, 40.2, 29.7.

IR ν 3212, 1650, 1590, 1438, 1363, 1182, 1059, 918, 775, 634 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₉N₂O₄S⁺ 299.1066. Found 299.1083 (1.7 mmu error).



(*E*)-5-hydroxy-1-(naphthalen-2-yl)pent-2-en-1-one

Compound 18: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

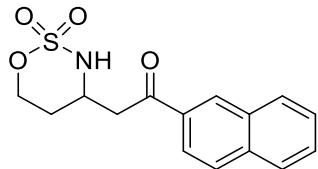
¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 1.6 Hz, 1H), 8.06 – 7.84 (m, 4H), 7.57 (dd, *J* = 19.7, 8.2, 6.9, 1.4 Hz, 2H), 7.22 – 7.05 (m, 2H), 3.88 (t, *J* = 6.3 Hz, 2H), 2.64 (q, *J* = 6.1 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.4, 145.6, 135.6, 135.1, 132.6, 130.2, 129.6, 128.6, 128.5, 128.0, 127.9, 126.8, 124.5, 61.2, 36.2.

IR ν 3326, 1664, 1613, 1458, 1369, 1285, 1122, 1041, 967, 818, 746 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₅O₂⁺ 227.1072. Found 227.1096 (2.4 mmu error).

Previously characterized in *Chem. Commun.* **2015**, 51, 11693 – 11696. Our data matches the literature.



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-(naphthalen-2-yl)ethan-1-one

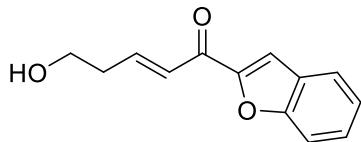
Compound 19: Synthesized using **General Procedure B** on a 227 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 158 mg, 0.51 mmol, 51% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.57 (d, *J* = 1.6 Hz, 1H), 8.07 (dd, *J* = 8.1, 1.4 Hz, 1H), 8.05 – 7.91 (m, 3H), 7.64 (dddd, *J* = 20.2, 8.1, 6.8, 1.4 Hz, 2H), 5.40 (d, *J* = 10.2 Hz, 1H), 4.76 – 4.65 (m, 1H), 4.65 – 4.55 (m, 1H), 4.35 – 4.22 (m, 1H), 3.50 (dd, *J* = 17.4, 6.6 Hz, 1H), 3.33 (dd, *J* = 17.3, 6.3 Hz, 1H), 1.93 – 1.83 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 197.8, 136.5, 135.0, 133.4, 131.0, 130.5, 129.7, 129.4, 128.6, 128.0, 124.3, 73.4, 53.7, 43.2, 29.7.

IR v 3246, 1682, 1595, 1418, 1352, 1285, 1185, 1062, 915, 798 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₆NO₄S⁺ 306.0800. Found 306.0826 (2.6 mmu error).



(*E*)-1-(benzofuran-2-yl)-5-hydroxypent-2-en-1-one

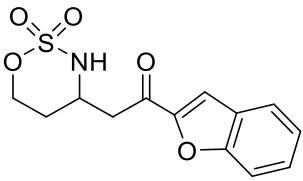
Compound 20: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.74 – 7.65 (m, 1H), 7.61 – 7.52 (m, 2H), 7.51 – 7.42 (m, 1H), 7.33 – 7.18 (m, 2H), 7.09 – 7.00 (m, 1H), 3.92 – 3.83 (m, 2H), 2.67 – 2.56 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 179.7, 155.9, 153.2, 146.0, 128.4, 127.2, 127.1, 124.0, 123.3, 113.7, 112.5, 61.0, 36.1.

IR v 3461, 1656, 1613, 1363, 1288, 1165, 1041, 967, 829, 752 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₃O₃⁺ 217.0865. Found 217.0860 (2.3 ppm error).



1-(benzofuran-2-yl)-2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)ethan-1-one

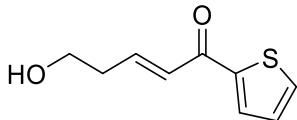
Compound 21: Synthesized using **General Procedure B** on a 217 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 196 mg, 0.66 mmol, 66% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.81 (dt, *J* = 8.0, 1.1 Hz, 1H), 7.69 (d, *J* = 1.0 Hz, 1H), 7.67 – 7.60 (m, 1H), 7.55 (ddd, *J* = 8.4, 7.1, 1.3 Hz, 1H), 7.37 (ddd, *J* = 8.0, 7.2, 1.0 Hz, 1H), 5.37 (d, *J* = 10.3 Hz, 1H), 4.72 – 4.63 (m, 1H), 4.63 – 4.55 (m, 1H), 4.33 – 4.16 (m, 1H), 3.33 (dd, *J* = 16.9, 7.1 Hz, 1H), 3.14 (dd, *J* = 16.9, 6.2 Hz, 1H), 1.93 – 1.81 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 188.2, 156.5, 153.2, 129.6, 127.9, 125.0, 124.6, 115.0, 113.1, 73.3, 53.4, 43.4, 29.6.

IR v 3243, 1673, 1555, 1357, 1159, 1024, 998, 754, 628 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₄NO₅S⁺ 296.0593. Found 296.0606 (4.4 ppm error).



(*E*)-5-hydroxy-1-(thiophen-2-yl)pent-2-en-1-one

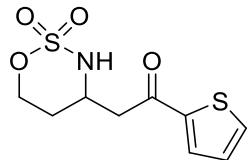
Compound 22: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.76 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.65 (dd, *J* = 4.8, 1.1 Hz, 1H), 7.18 – 7.03 (m, 2H), 6.95 – 6.85 (m, 1H), 3.83 (t, *J* = 6.3 Hz, 2H), 2.58 (td, *J* = 6.3, 5.0 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 182.2, 145.1, 145.0, 134.1, 132.2, 128.3, 127.5, 61.1, 36.0.

IR v 3438, 1650, 1596, 1409, 1357, 1285, 1059, 944, 746, 694 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₉H₁₁O₂S⁺ 183.0480. Found 183.0492 (1.2 mmu error).



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-(thiophen-2-yl)ethan-1-one

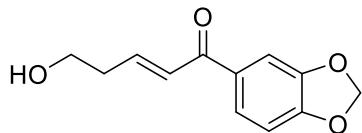
Compound 23: Synthesized using **General Procedure B** on a 188 mg (1.03 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 190 mg, 0.73 mmol, 71% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.83 (dd, *J* = 4.3, 3.2 Hz, 2H), 7.21 (dd, *J* = 4.8, 3.9 Hz, 1H), 5.35 (d, *J* = 10.2 Hz, 1H), 4.73 – 4.62 (m, 1H), 4.63 – 4.53 (m, 1H), 4.26 – 4.13 (m, 1H), 3.31 (dd, *J* = 16.8, 7.1 Hz, 1H), 3.12 (dd, *J* = 16.8, 6.1 Hz, 1H), 1.85 (ddd, *J* = 10.4, 7.6, 4.1 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 190.8, 144.8, 135.8, 134.2, 129.6, 73.3, 53.7, 43.5, 29.6.

IR v 3226, 1644, 1409, 1349, 1291, 1185, 1059, 912, 769, 691 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₉H₁₁NO₄S₂Na⁺ 284.0027. Found 284.0034 (2.5 ppm error).



(*E*)-1-(benzo[d][1,3]dioxol-5-yl)-5-hydroxypent-2-en-1-one

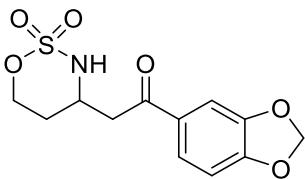
Compound 24: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.54 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.43 (d, *J* = 1.6 Hz, 1H), 7.10 – 6.90 (m, 2H), 6.84 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.03 (s, 2H), 3.88 – 3.77 (m, 2H), 2.61 – 2.53 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 188.5, 151.8, 148.3, 145.0, 132.5, 127.6, 124.9, 108.5, 107.9, 101.9, 61.1, 36.1.

IR v 3280, 1664, 1601, 1438, 1242, 1110, 1019, 924, 800, 757 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₃O₄⁺ 221.0814. Found 221.0831 (1.7 mmu error).



1-(benzo[*d*][1,3]dioxol-5-yl)-2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)ethan-1-one

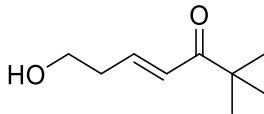
Compound 25: Synthesized using **General Procedure B** on a 221 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 183 mg, 0.61 mmol, 61% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.61 – 7.56 (m, 1H), 7.41 (d, *J* = 1.8 Hz, 1H), 6.93 (d, *J* = 8.2 Hz, 1H), 6.06 (s, 2H), 5.32 (d, *J* = 10.2 Hz, 1H), 4.74 – 4.61 (m, 1H), 4.62 – 4.53 (m, 1H), 4.27 – 4.12 (m, 1H), 3.27 (dd, *J* = 17.3, 6.6 Hz, 1H), 3.10 (dd, *J* = 17.3, 6.3 Hz, 1H), 1.91 – 1.74 (m, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 195.8, 153.2, 149.3, 132.4, 125.5, 108.7, 108.2, 103.4, 73.3, 53.7, 42.9, 29.7.

IR ν 3111, 1653, 1604, 1435, 1360, 1240, 1182, 1010, 938, 783, 642 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₂H₁₄NO₆S⁺ 300.0542. Found 300.0562 (2.0 mmu error).



(*E*)-7-hydroxy-2,2-dimethylhept-4-en-3-one

Compound 26: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

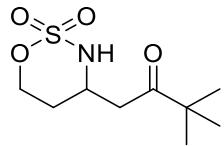
¹H NMR (400 MHz, CDCl₃) δ 6.89 (dt, *J* = 15.1, 7.2 Hz, 1H), 6.59 (dt, *J* = 15.4, 1.5 Hz, 1H), 3.75 (td, *J* = 6.4, 1.0 Hz, 2H), 2.53 – 2.41 (m, 2H), 1.14 (s, 9H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 204.4, 143.5, 126.5, 61.1, 42.9, 35.8, 26.2.

IR ν 3111, 1656, 1604, 1435, 1363, 1240, 1182, 1039, 938, 783 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₉H₁₆O₂Na⁺ 179.1048. Found 179.1050 (1.1 ppm error).

Previously characterized in *J. Am. Chem. Soc.* **2008**, *130*, 46–48. Our characterization data matches the literature.



1-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-3,3-dimethylbutan-2-one

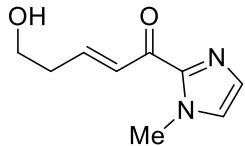
Compound 27: Synthesized using **General Procedure B** on a 157 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 147 mg, 0.62 mmol, 62% yield).

¹H NMR (400 MHz, CD₃CN) δ 5.22 (d, *J* = 10.3 Hz, 1H), 4.69 – 4.59 (m, 1H), 4.57 – 4.50 (m, 1H), 4.10 – 3.99 (m, 1H), 2.89 (dd, *J* = 17.8, 6.5 Hz, 1H), 2.68 (dd, *J* = 17.9, 6.1 Hz, 1H), 1.78 – 1.69 (m, 2H), 1.11 (s, 9H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 213.4, 73.3, 53.3, 44.7, 41.4, 29.4, 26.1.

IR ν 3249, 1713, 1679, 1354, 1188, 1079, 998, 769, 640 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₉H₁₇NO₄SNa⁺ 258.0776. Found 258.0803 (2.7 mmu error).



(*E*)-5-hydroxy-1-(1-methyl-1*H*-imidazol-2-yl)pent-2-en-1-one

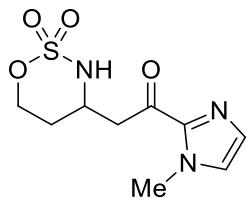
Compound 28: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, Acetone-*d*₆) δ 7.45 (dt, *J* = 15.8, 1.5 Hz, 1H), 7.34 (d, *J* = 0.9 Hz, 1H), 7.18 – 7.00 (m, 2H), 4.03 (s, 3H), 3.95 (broad s, 1H), 3.74 (t, *J* = 6.4 Hz, 2H), 2.52 (qd, *J* = 6.4, 1.6 Hz, 2H).

¹³C{¹H} NMR (101 MHz, Acetone-*d*₆) δ 180.7, 145.4, 144.4, 129.6, 128.6, 128.5, 61.2, 36.8, 36.3.

IR ν 3269, 1664, 1616, 1403, 1283, 1165, 1093, 1039, 973, 915, 803, 777 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ calculated for C₉H₁₃N₂O₂⁺ 181.0977. Found 181.0963 (1.4 mmu error).



2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-(1-methyl-1*H*-imidazol-2-yl)ethan-1-one

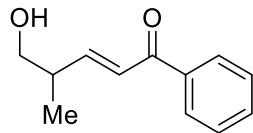
Compound 29: See Supporting Information, Scale-up Section for full procedure; Purified using a gradient of 1 to 6% methanol in ethyl acetate on silica gel; (white solid, 769 mg, 2.97 mmol, 78% yield).

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.52 (d, *J* = 0.8 Hz, 1H), 7.28 (broad s, 1H), 7.13 (d, *J* = 1.0 Hz, 1H), 4.62 – 4.43 (m, 2H), 4.11 (ddt, *J* = 14.1, 7.0, 3.4 Hz, 1H), 3.92 (s, 3H), 3.32 (dd, *J* = 16.3, 7.3 Hz, 1H), 3.21 (dd, *J* = 16.4, 6.7 Hz, 1H), 1.81 – 1.58 (m, 2H).

¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 188.3, 142.2, 128.8, 128.5, 71.9, 52.1, 43.1, 35.7, 28.6.

IR ν 1676, 1478, 1420, 1349, 1176, 1076, 990, 924, 826, 769 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ calculated for C₉H₁₄N₃O₄S⁺ 260.0705. Found 260.0723 (1.8 mmu error).



(*E*)-5-hydroxy-4-methyl-1-phenylpent-2-en-1-one

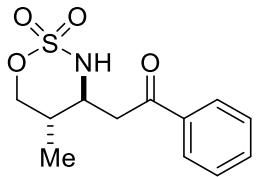
Compound 30: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.88 (m, 2H), 7.60 – 7.53 (m, 1H), 7.51 – 7.41 (m, 2H), 7.02 – 6.91 (m, 2H), 3.73 – 3.58 (m, 2H), 2.66 (dddd, *J* = 11.1, 6.8, 5.5, 1.2 Hz, 1H), 1.15 (d, *J* = 6.8 Hz, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.9, 151.3, 137.8, 132.9, 128.7, 128.6, 126.1, 66.7, 39.9, 15.8.

IR ν 3240, 1653, 1598, 1389, 1214, 1182, 1013, 775, 694 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₂H₁₄O₂Na⁺ 213.0892. Found 213.0913 (2.1 mmu error).



2-((4S*,5S*)-5-methyl-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

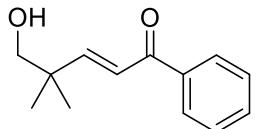
Compound 31: Synthesized using **General Procedure B** on a 100 mg (0.53 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; single diastereomer; (White solid, 69 mg, 0.25 mmol, 48% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.02 – 7.95 (m, 2H), 7.69 – 7.62 (m, 1H), 7.58 – 7.49 (m, 2H), 5.41 (d, *J* = 9.9 Hz, 1H), 4.45 (dd, *J* = 11.6, 4.7 Hz, 1H), 4.31 (t, *J* = 11.4 Hz, 1H), 3.94 (tdd, *J* = 10.1, 7.6, 4.7 Hz, 1H), 3.41 – 3.22 (m, 2H), 2.06 (tdd, *J* = 11.1, 6.8, 4.6 Hz, 1H), 0.91 (d, *J* = 6.7 Hz, 3H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 198.3, 137.7, 134.4, 129.7, 129.0, 77.3, 58.6, 41.0, 33.3, 12.3.

IR ν 3200, 1662, 1596, 1446, 1360, 1182, 967, 780, 688 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₂H₁₅NO₄SNa⁺ 292.0620. Found 292.0638 (1.8 mmu error).



(*E*)-5-hydroxy-4,4-dimethyl-1-phenylpent-2-en-1-one

Compound 32: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

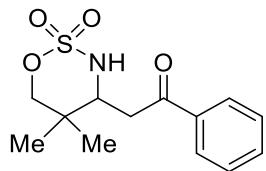
¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.88 (m, 2H), 7.58 – 7.52 (m, 1H), 7.46 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.02 (d, *J* = 15.8 Hz, 1H), 6.88 (d, *J* = 15.8 Hz, 1H), 3.49 (s, 2H), 1.15 (s, 6H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 191.3, 155.6, 138.0, 132.9, 128.7, 128.6, 124.0, 71.1, 39.8, 23.3.

IR ν 3200, 2958, 1664, 1596, 1360, 1294, 1182, 1041, 973, 777, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₃H₁₇O₂⁺ 205.1229. Found 205.1243 (1.4 mmu error).

Previously characterized in *Org. Lett.* **2019**, *21*, 2688-2692. Our characterization data matches the literature.



2-(5,5-dimethyl-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

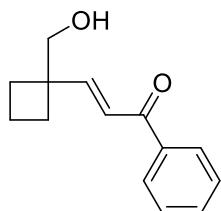
Compound 33: Synthesized using **General Procedure B** on a 100 mg (0.48 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 81 mg, 0.28 mmol, 58% yield).

¹H NMR (400 MHz, CD₃OD) δ 8.03 – 7.97 (m, 2H), 7.65 – 7.59 (m, 1H), 7.55 – 7.48 (m, 2H), 4.41 (dd, *J* = 11.6, 0.9 Hz, 1H), 4.18 (d, *J* = 3.0 Hz, 1H), 4.08 (d, *J* = 11.6 Hz, 1H), 3.24 (dd, *J* = 16.8, 9.7 Hz, 1H), 3.10 (dd, *J* = 16.8, 3.1 Hz, 1H), 1.17 (s, 3H), 0.96 (s, 3H).

¹³C{¹H} NMR (101 MHz, CD₃OD) δ 198.8, 138.3, 134.5, 129.8, 129.2, 82.6, 60.7, 38.8, 32.8, 21.5, 17.7.

IR ν 3220, 1670, 1596, 1446, 1360, 1185, 1001, 961, 786, 683 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₃H₁₇NO₄SNa⁺ 306.0776. Found 306.0790 (4.6 ppm error).



(*E*)-3-(1-(hydroxymethyl)cyclobutyl)-1-phenylprop-2-en-1-one

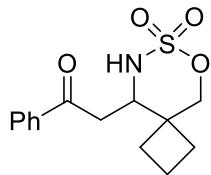
Compound 34: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.95 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.59 – 7.53 (m, 1H), 7.52 – 7.43 (m, 2H), 7.13 (d, *J* = 15.6 Hz, 1H), 6.93 (d, *J* = 15.5 Hz, 1H), 3.75 (s, 2H), 2.17 – 1.92 (m, 6H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 191.0, 153.6, 138.0, 132.9, 128.7, 128.7, 124.3, 68.2, 46.7, 28.5, 15.5.

IR ν 2933, 1664, 1610, 1331, 1214, 1174, 1016, 981, 772, 697 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₄H₁₆O₂Na⁺ 239.1048. Found 239.1074 (2.6 mmu error).



2-(7,7-dioxido-6-oxa-7-thia-8-azaspiro[3.5]nonan-9-yl)-1-phenylethan-1-one

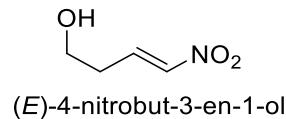
Compound 35: Synthesized using **General Procedure B** on a 100 mg (0.46 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 85 mg, 0.28 mmol, 60% yield).

¹H NMR (400 MHz, Acetone-d₆) δ 8.09 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.72 – 7.63 (m, 1H), 7.61 – 7.50 (m, 2H), 6.46 (d, *J* = 8.9 Hz, 1H), 4.74 (d, *J* = 11.6 Hz, 1H), 4.59 (dd, *J* = 11.7, 1.1 Hz, 1H), 4.33 (td, *J* = 9.3, 3.0 Hz, 1H), 3.84 (dd, *J* = 17.2, 9.7 Hz, 1H), 3.35 (dd, *J* = 17.2, 3.0 Hz, 1H), 2.39 – 2.26 (m, 1H), 2.19 – 2.07 (m, 2H), 2.03 – 1.90 (m, 3H).

¹³C{¹H} NMR (101 MHz, Acetone-d₆) δ 197.5, 138.3, 134.1, 129.5, 129.0, 79.0, 58.8, 39.4, 38.3, 26.5, 24.6, 14.9.

IR ν 3255, 1684, 1452, 1383, 1179, 947, 783, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₄H₁₈NO₄S⁺ 296.0957. Found 296.0969 (4.1 ppm error).



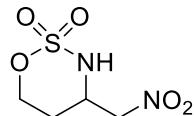
Compound 36: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.30 (dt, *J* = 13.4, 7.4 Hz, 1H), 7.08 (dt, *J* = 13.5, 1.5 Hz, 1H), 3.83 (t, *J* = 6.0 Hz, 2H), 2.52 (dtd, *J* = 7.4, 6.0, 1.5 Hz, 2H), 1.83 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 141.0, 139.3, 60.3, 31.5.

IR ν 3392, 3105, 2953, 2887, 1650, 1521, 1354, 1277, 1047, 958, 841, 823, 749 cm⁻¹.

HRMS (APCI) m/z = [M]⁺ Calcd C₄H₇NO₃⁺ 117.0426. Found 117.0413 (1.3 mmu error).



4-(nitromethyl)-1,2,3-oxathiazinane 2,2-dioxide

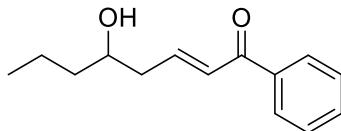
Compound 37: Synthesized using **General Procedure C** on a 60 mg (0.51 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (Colorless oil, 61 mg, 0.31 mmol, 60% yield).

¹H NMR (400 MHz, Acetone-*d*₆) δ 6.50 (d, *J* = 10.4 Hz, 1H), 4.93 (dd, *J* = 13.5, 4.4 Hz, 1H), 4.84 – 4.68 (m, 3H), 4.56 (dddd, *J* = 13.6, 9.8, 6.9, 3.7 Hz, 1H), 2.10 – 1.93 (m, 2H).

¹³C{¹H} NMR (101 MHz, Acetone-*d*₆) δ 77.8, 72.3, 54.9, 26.7.

IR ν 3252, 1552, 1420, 1368, 1356, 1182, 1010, 950, 864, 780 cm⁻¹.

HRMS (ESI) m/z = [M - H]⁻ Calcd C₄H₇N₂O₅S⁻ 195.0076. Found 195.0087 (5.6 ppm error).



(*E*)-5-hydroxy-1-phenyloct-2-en-1-one

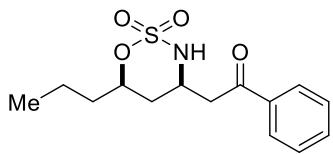
Compound 38: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.93 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.60 – 7.52 (m, 1H), 7.46 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.08 (dt, *J* = 15.4, 7.2 Hz, 1H), 6.96 (dt, *J* = 15.4, 1.2 Hz, 1H), 3.91 – 3.80 (m, 1H), 2.57 – 2.38 (m, 2H), 1.54 – 1.46 (m, 3H), 1.43 – 1.35 (m, 1H), 0.97 – 0.91 (m, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.7, 145.9, 137.8, 132.9, 128.7, 128.6, 128.3, 70.5, 40.9, 39.5, 18.9, 14.1.

IR ν 3255, 1684, 1596, 1343, 1179, 1013, 947, 783, 688 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₄H₁₈O₂Na⁺ 241.1205. Found 241.1234 (2.9 mmu error).



2-((4*R*^{*},6*R*^{*})-2,2-dioxido-6-propyl-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

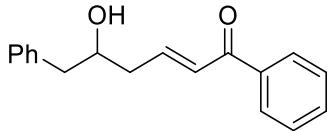
Compound 39: Synthesized using **General Procedure B** on a 100 mg (0.45 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; single diastereomer; (Colorless oil, 81 mg, 0.27 mmol, 60% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.89 (m, 2H), 7.66 – 7.58 (m, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 5.22 (d, *J* = 9.6 Hz, 1H), 4.79 (dd, *J* = 11.1, 7.8, 4.6, 3.0 Hz, 1H), 4.19 (s, 1H), 3.36 (dd, *J* = 17.9, 4.3 Hz, 1H), 3.25 (dd, *J* = 17.9, 6.1 Hz, 1H), 1.86 – 1.71 (m, 3H), 1.64 – 1.36 (m, 3H), 0.93 (t, *J* = 7.3 Hz, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 197.9, 136.2, 134.2, 129.0, 128.1, 84.3, 52.2, 41.5, 37.2, 34.4, 17.9, 13.7.

IR v 2962, 1679, 1596, 1415, 1354, 1179, 927, 864, 754, 688 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₄H₁₉NO₄SnA⁺ 320.0933. Found 320.0943 (3.1 ppm error).



(*E*)-5-hydroxy-1,6-diphenylhex-2-en-1-one

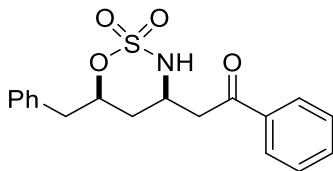
Compound 40: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.98 – 7.90 (m, 2H), 7.59 – 7.53 (m, 1H), 7.47 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.33 (dd, *J* = 8.0, 6.5 Hz, 2H), 7.28 – 7.21 (m, 3H), 7.11 (dt, *J* = 15.5, 7.2 Hz, 1H), 6.98 (d, *J* = 15.5 Hz, 1H), 4.05 (ddd, *J* = 7.8, 4.6, 2.9 Hz, 1H), 2.88 (dd, *J* = 13.6, 4.8 Hz, 1H), 2.77 (dd, *J* = 13.7, 8.1 Hz, 1H), 2.62 – 2.47 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.6, 145.5, 137.9, 137.8, 132.9, 129.5, 128.8, 128.7, 128.6, 128.4, 126.8, 71.5, 43.8, 40.1.

IR v 3222, 1667, 1618, 1449, 1225, 1179, 1019, 973, 749, 694 cm⁻¹.

HRMS (APCI) m/z = [M + H]⁺ Calcd C₁₈H₁₉O₂⁺ 267.1385. Found 267.1372 (4.9 ppm error).



2-((4*R*^{*},6*R*^{*})-6-benzyl-2,2-dioxido-1,2,3-oxathiazinan-4-yl)-1-phenylethan-1-one

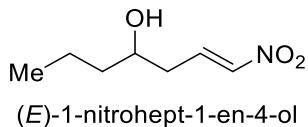
Compound 41: Synthesized using **General Procedure B** on a 100 mg (0.37 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; single diastereomer; (Colorless oil, 73 mg, 0.21 mmol, 56% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.98 – 7.86 (m, 2H), 7.65 – 7.59 (m, 1H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.23 (m, 3H), 7.22 – 7.15 (m, 2H), 5.31 (d, *J* = 10.2 Hz, 1H), 5.06 – 4.89 (m, 1H), 4.13 (tt, *J* = 10.3, 5.1 Hz, 1H), 3.31 (dd, *J* = 18.1, 4.3 Hz, 1H), 3.22 (dd, *J* = 18.0, 5.7 Hz, 1H), 3.14 (dd, *J* = 13.9, 6.2 Hz, 1H), 2.90 (dd, *J* = 14.0, 7.1 Hz, 1H), 1.90 – 1.71 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 197.9, 136.2, 135.1, 134.2, 129.5, 129.0, 128.8, 128.1, 127.3, 84.3, 52.1, 41.6, 41.2, 33.5.

IR v 2950, 1679, 1596, 1449, 1357, 1179, 924, 864, 752, 688 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₈H₁₉NO₄SNa⁺ 368.0933. Found 368.0930 (0.8 ppm error).



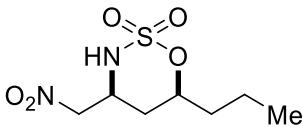
Compound 42: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.32 (dt, *J* = 13.4, 7.7 Hz, 1H), 7.10 – 7.01 (m, 1H), 3.88 – 3.78 (m, 1H), 2.48 – 2.30 (m, 2H), 1.48 (td, *J* = 4.5, 2.2 Hz, 3H), 1.43 – 1.31 (m, 1H), 0.94 (t, *J* = 6.9 Hz, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 141.0, 139.3, 70.0, 39.6, 36.1, 18.8, 14.0.

IR v 2959, 1651, 1519, 1466, 1359, 1123, 960, 864, 734 cm⁻¹.

HRMS (APCI) m/z = [M]⁺ Calcd C₇H₁₃NO₃⁺ 159.0895. Found 159.0918 (2.3 mmu error).



(*4S*^{*},*6S*^{*})-4-(nitromethyl)-6-propyl-1,2,3-oxathiazinane 2,2-dioxide

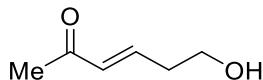
Compound 43: Synthesized using **General Procedure C** on a 0.62 mmol scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; single diastereomer; (Colorless oil, 112 mg, 0.47 mmol, 75% yield).

¹H NMR (500 MHz, CDCl₃) δ 4.94 (d, *J* = 10.8 Hz, 1H), 4.83 – 4.76 (m, 1H), 4.67 – 4.53 (m, 2H), 4.31 – 4.22 (m, 1H), 1.87 (dq, *J* = 14.1, 1.9 Hz, 1H), 1.82 – 1.73 (m, 1H), 1.66 – 1.60 (m, 2H), 1.55 – 1.40 (m, 2H), 0.95 (t, *J* = 7.6 Hz, 3H).

¹³C{¹H} NMR (126 MHz, CDCl₃) δ 83.6, 77.4, 52.5, 37.1, 31.9, 17.8, 13.6.

IR ν 2962, 1578, 1423, 1352, 1191, 1178, 948, 849, 548 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₇H₁₅N₂O₅S⁺ 239.0702. Found 239.0719 (1.7 mmu error).



(*E*)-6-hydroxyhex-3-en-2-one

Compound 44: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

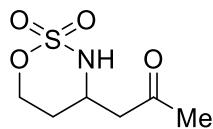
¹H NMR (400 MHz, CDCl₃) δ 6.82 (dt, *J* = 16.1, 7.0 Hz, 1H), 6.14 (dd, *J* = 15.8, 1.6 Hz, 1H), 3.77 (td, *J* = 6.2, 1.8 Hz, 2H), 2.48 (td, *J* = 7.0, 5.4 Hz, 2H), 2.24 (s, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 198.8, 144.8, 133.1, 61.0, 35.7, 27.0.

IR ν 2922, 1667, 1626, 1423, 1362, 1255, 1044, 974, 543 cm⁻¹.

HRMS (APCI) m/z = [M + H]⁺ Calcd C₆H₁₁O₂⁺ 115.0759. Found 115.0751 (0.8 mmu error).

Previously characterized in *J. Org. Chem.* **2021**, *86*, 7537–7551. Our data matches the literature.



1-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)propan-2-one

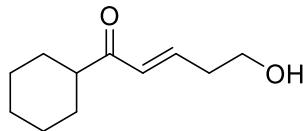
Compound 45: Synthesized using **General Procedure B** on a 0.87 mmol scale; Purified using a gradient of 20 to 70% EtOAc/hexanes on silica gel; (Colorless oil, 105 mg, 0.54 mmol, 62% yield).

¹H NMR (400 MHz, CDCl₃) δ 5.11 (d, *J* = 10.0 Hz, 1H), 4.71 (ddd, *J* = 12.5, 11.6, 2.5 Hz, 1H), 4.54 (ddd, *J* = 11.6, 5.0, 2.0 Hz, 1H), 4.10 – 3.98 (m, 1H), 2.85 – 2.72 (m, 2H), 2.20 (s, 3H), 2.04 – 1.94 (m, 1H), 1.71 (dq, *J* = 14.3, 2.5 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 206.7, 71.9, 52.5, 46.2, 30.8, 28.3.

IR ν 3237, 1712, 1445, 1352, 1181, 1063, 957, 865, 548 cm⁻¹.

HRMS (APCI) m/z = [M + H]⁺ Calcd C₆H₁₂NO₄S⁺ 194.0487. Found 194.0482 (2.6 ppm error).



(*E*)-1-cyclohexyl-5-hydroxypent-2-en-1-one

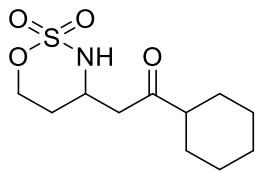
Compound 46: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 6.85 (dt, *J* = 15.7, 7.0 Hz, 1H), 6.26 (dt, *J* = 15.8, 1.5 Hz, 1H), 3.78 (t, *J* = 6.3 Hz, 2H), 2.61 – 2.51 (m, 1H), 2.48 (qd, *J* = 6.3, 1.5 Hz, 2H), 1.80 (dddt, *J* = 10.9, 9.2, 5.6, 2.7 Hz, 4H), 1.70 – 1.63 (m, 2H), 1.36 – 1.23 (m, 4H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 203.2, 142.8, 130.7, 61.2, 48.8, 35.8, 28.7, 26.0, 25.8.

IR ν 2927, 1662, 1623, 1449, 1146, 1046, 975, 513 cm⁻¹.

HRMS (APCI) m/z = [M + H]⁺ Calcd C₁₁H₁₉O₂⁺ 183.1385. Found 183.1396 (1.1 mmu error).



1-cyclohexyl-2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)ethan-1-one

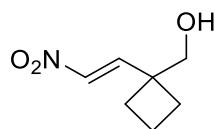
Compound 47: Synthesized using **General Procedure B** on a 100 mg (0.54 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (Colorless oil, 94 mg, 0.35 mmol, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ 5.24 (d, *J* = 9.9 Hz, 1H), 4.70 (ddd, *J* = 12.5, 11.6, 2.5 Hz, 1H), 4.53 (ddd, *J* = 11.6, 5.0, 2.0 Hz, 1H), 4.04 (t, *J* = 10.0 Hz, 1H), 2.78 (qd, *J* = 18.0, 5.1 Hz, 2H), 2.33 (ddd, *J* = 11.2, 7.7, 3.4 Hz, 1H), 1.97 (dtd, *J* = 14.2, 12.1, 4.9 Hz, 1H), 1.80 (dp, *J* = 7.8, 3.3 Hz, 4H), 1.71 – 1.63 (m, 2H), 1.37 – 1.18 (m, 5H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 212.5, 71.8, 52.7, 51.4, 43.1, 28.33, 28.30, 28.28, 25.7, 25.6, 25.5.

IR ν 2926, 1705, 1340, 1174, 1067, 996, 777, 508 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₁H₂₀NO₄S⁺ 262.1113. Found 262.1131 (1.8 mmu error).



(*E*)-(1-(2-nitrovinyl)cyclobutyl)methanol

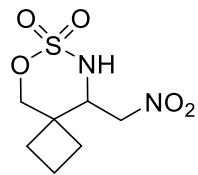
Compound 48: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; Colorless oil

¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, *J* = 13.5 Hz, 1H), 7.04 (d, *J* = 13.6 Hz, 1H), 3.77 (s, 2H), 2.13 – 2.07 (m, 4H), 2.07 – 1.96 (m, 2H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 146.6, 139.3, 67.6, 44.1, 28.6, 15.6.

IR ν 3219, 1564, 1442, 1350, 1172, 1095, 909, 561 cm⁻¹.

HRMS (APCI) m/z = [M + H]⁺ Calcd C₇H₁₂NO₃⁺ 158.0817. Found 158.0807 (1.0 mmu error).



9-(nitromethyl)-6-oxa-7-thia-8-azaspiro[3.5]nonane 7,7-dioxide

Compound 49: Synthesized using **General Procedure C** on a 20 mg (0.13 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (White solid, 25 mg, 0.10 mmol, 77% yield).

¹H NMR (400 MHz, Acetone-*d*₆) δ 6.90 (d, *J* = 8.7 Hz, 1H), 5.12 – 4.97 (m, 2H), 4.71 (d, *J* = 11.8 Hz, 1H), 4.62 (d, *J* = 11.8 Hz, 1H), 4.38 (ddd, *J* = 10.6, 8.7, 3.7 Hz, 1H), 2.23 (td, *J* = 8.0, 3.7 Hz, 2H), 2.14 – 2.00 (m, 2H), 1.95 (td, *J* = 8.7, 4.5 Hz, 2H).

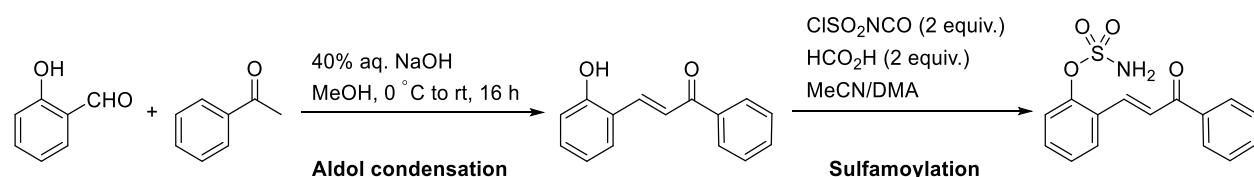
¹³C{¹H} NMR (101 MHz, Acetone-*d*₆) δ 78.4, 74.9, 60.8, 38.1, 27.2, 24.6, 14.9.

IR ν 3219, 1562, 1352, 1188, 1050, 940, 805, 561 cm⁻¹.

HRMS (ESI) m/z = [M - H]⁻ Calcd C₇H₁₁N₂O₅S⁻ 235.0389. Found 235.0400 (4.7 ppm error).

IV. Representative Procedures for Sulfamate Substrate Syntheses, General Procedure for Cyclization, and Characterization (Scheme 5)

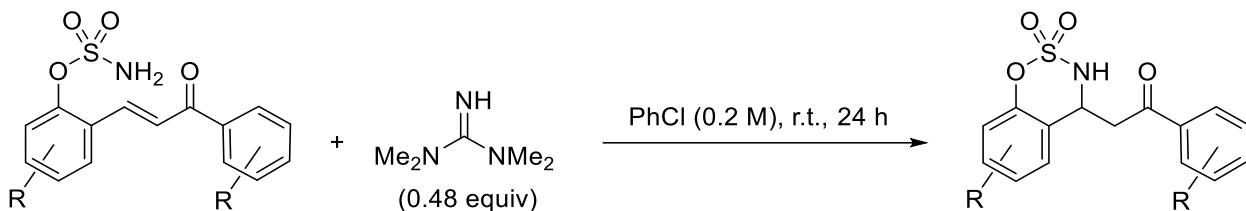
Representative Sequence for Starting Material Preparation:



Aldol condensation (representative example): Salicylaldehyde (2.0 g, 16.4 mmol, 1 equiv.) and acetophenone (1.97 g, 16.4 mmol, 1 equiv.) were dissolved in MeOH (30.0 mL) and added to a 100 mL round-bottom flask equipped with a magnetic stir-bar. The reaction mixture was cooled to 0 °C using an ice-water bath. Next, 40% w/w aqueous NaOH solution (8.0 mL) was added dropwise to the reaction mixture. After completion of addition, the reaction mixture was allowed to warm to room temperature and stirred for 16 hours. MeOH was evaporated under reduced pressure, and 1 M aqueous HCl (150 mL) was added to quench the reaction. The mixture was transferred to a separatory funnel. The aqueous layer was extracted with EtOAc (3 x 100 mL). The combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. The resulting residue was purified using a gradient of 5–30% EtOAc/hexane on silica gel to yield the desired product (2.7 g, 12.04 mmol, 73% yield).

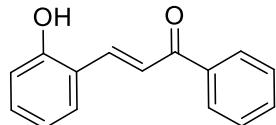
Sulfamoylation (representative example): An oven-dried round-bottom flask was fitted with a balloon of N₂ gas and charged with a stir bar. CISO₂NCO (388 μL, 0.632 g, 4.46 mmol, 2.0 equiv) was added to it, and the flask was cooled to 0 °C using an ice-water bath. HCO₂H (168 μL, 0.205 g, 4.46 mmol, 2.0 equiv) was added dropwise (**Caution: vigorous gas evolution upon addition**). The mixture solidified into a white solid within 5 minutes of addition. CH₃CN (4.5 mL) was added, and the reaction mixture was warmed to room temperature over a period of 12 h. A separate oven-dried round-bottom flask was fitted with a nitrogen balloon and charged with a stir bar, phenol substrate (500.0 mg, 2.23 mmol, 1 equiv.), and DMA (1.5 mL, 1.5 M, relative to phenol). The reaction flask was cooled to 0 °C using an ice-water bath, and freshly prepared CISO₂NH₂ (in CH₃CN) was added dropwise. With stirring, the reaction mixture was warmed to room temperature over a period of 16 h. Following this time, the reaction was quenched with water (15 mL). The mixture was transferred to a separatory funnel. The aqueous layer was extracted with EtOAc (3 x 25 mL). The organic layers were combined and concentrated under reduced pressure to ~30 mL. After transferring to a separatory funnel, they were washed with ice-cold 10% w/w LiCl (aq.) solution (30 mL x 2). The organic layer was separated, dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified using a gradient of 10–60% EtOAc/hexanes on silica gel to yield the desired product (407.0 mg, 1.34 mmol, 60% yield).

General Procedure D: Aza-Michael Cyclization



An oven dried 5 mL glass vial was charged with a stir bar, sulfamate substrate (1.0 equiv.), and chlorobenzene (reaction concentration = 0.2 M). The resulting mixture was stirred for 2 minutes, and then 1,1,3,3-tetramethyl guanidine (0.48 equiv.) was added. The reaction was stirred for 24 h at room temperature. Following this time, the reaction mixture was carefully transferred to a separatory funnel using CH₂Cl₂ and washed with water. The organic layer was separated, and the aqueous layer was extracted with CH₂Cl₂ (x 3). The combined organic layers were dried with Na₂SO₄, filtered, and concentrated *in vacuo*. The crude product was purified on silica gel to yield the cyclization product (specific conditions are associated with each product).

Note on compound numbering: Compound SX' refers to the phenol and Compound SX refers to the sulfamate. For example, Compound S1' would refer to the phenol precursor, and Compound S1 would refer to the sulfamate substrate for product 1.



(*E*)-3-(2-hydroxyphenyl)-1-phenylprop-2-en-1-one

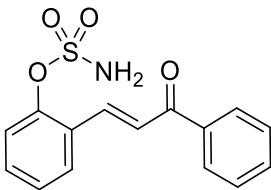
Compound S50': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

¹H NMR (400 MHz, CD₃CN) δ 8.04 (dd, *J* = 7.0, 1.7 Hz, 2H), 8.00 (s, 1H), 7.78 – 7.68 (m, 2H), 7.66 – 7.59 (m, 2H), 7.59 – 7.50 (m, 2H), 7.29 (td, *J* = 7.8, 1.7 Hz, 1H), 6.95 (t, *J* = 7.6 Hz, 2H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 191.3, 157.4, 140.3, 139.3, 133.6, 132.8, 129.9, 129.6, 129.2, 123.1, 122.8, 121.3, 117.1.

IR ν 3272, 1639, 1581, 1558, 1455, 1228, 1182, 1021, 990, 749, 680 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₃O₂⁺ 225.0916. Found 225.0928 (1.2 mmu error).



(*E*)-2-(3-oxo-3-phenylprop-1-en-1-yl)phenyl sulfamate

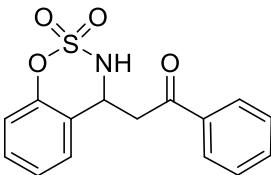
Compound S50: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

$^1\text{H NMR}$ (400 MHz, CD₃CN) δ 8.12 – 8.05 (m, 2H), 8.05 – 7.96 (m, 2H), 7.76 (d, J = 15.9 Hz, 1H), 7.69 – 7.62 (m, 1H), 7.61 – 7.48 (m, 4H), 7.43 (t, J = 7.4 Hz, 1H), 6.27 (s, 2H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CD₃CN) δ 190.8, 150.1, 138.8, 138.1, 134.0, 132.6, 129.7, 129.6, 129.4, 129.2, 128.2, 125.4, 123.9.

IR ν 3332, 1653, 1598, 1573, 1452, 1363, 1219, 1162, 1019, 944, 875, 714 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₄NO₄S⁺ 304.0644. Found 304.0656 (4 ppm error).



2-(2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylethan-1-one

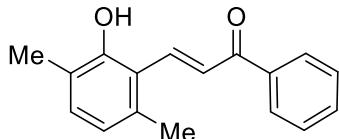
Compound 50: Synthesized using **General Procedure D** on a 304 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 228 mg, 0.75 mmol, 75% yield).

$^1\text{H NMR}$ (400 MHz, CD₃CN) δ 8.01 (dd, J = 8.4, 1.4 Hz, 2H), 7.69 – 7.61 (m, 1H), 7.58 – 7.49 (m, 2H), 7.41 – 7.33 (m, 2H), 7.24 (ddd, J = 8.3, 7.1, 1.3 Hz, 1H), 7.17 – 7.00 (m, 1H), 6.38 (broad s, 1H), 5.42 (dd, J = 8.9, 3.5 Hz, 1H), 4.12 (dd, J = 18.1, 9.0 Hz, 1H), 3.55 (dd, J = 18.1, 3.6 Hz, 1H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CD₃CN) δ 197.6, 152.0, 137.5, 134.6, 130.4, 129.7, 129.0, 128.0, 126.4, 123.3, 119.4, 54.1, 43.6.

IR ν 3229, 1676, 1598, 1486, 1354, 1208, 1185, 915, 757, 688 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₄NO₄S⁺ 304.0644. Found 304.0669 (2.5 mmu error).



(*E*)-3-(2-hydroxy-3,6-dimethylphenyl)-1-phenylprop-2-en-1-one

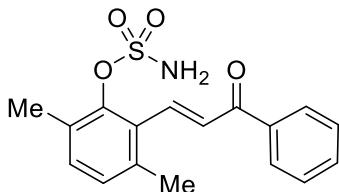
Compound S51': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.09 – 7.96 (m, 3H), 7.75 (dd, $J = 16.2, 1.1$ Hz, 1H), 7.61 – 7.54 (m, 1H), 7.49 (td, $J = 7.6, 1.6$ Hz, 2H), 7.04 (d, $J = 7.5$ Hz, 1H), 6.74 (d, $J = 7.6$ Hz, 1H), 2.39 (s, 3H), 2.28 (s, 3H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CDCl_3) δ 191.1, 153.4, 139.9, 138.2, 137.6, 132.9, 131.8, 128.7, 126.4, 122.6, 121.7, 121.2, 20.7, 15.9.

IR v 3272, 1641, 1596, 1584, 1329, 1219, 1205, 1016, 970, 686 cm^{-1} .

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₇H₁₆O₂Na⁺ 275.1048. Found 275.1059 (4.0 ppm error).



(*E*)-3,6-dimethyl-2-(3-oxo-3-phenylprop-1-en-1-yl)phenyl sulfamate

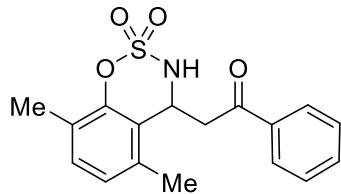
Compound S51: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

$^1\text{H NMR}$ (400 MHz, CD_3CN) δ 8.05 (dd, $J = 8.4, 1.4$ Hz, 2H), 7.88 (d, $J = 16.1$ Hz, 1H), 7.67 – 7.62 (m, 1H), 7.58 – 7.46 (m, 3H), 7.23 (d, $J = 7.9$ Hz, 1H), 7.15 (d, $J = 7.8$ Hz, 1H), 6.10 (broad s, 2H), 2.43 (s, 3H), 2.37 (s, 3H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CD_3CN) δ 191.1, 148.5, 139.7, 138.7, 137.7, 134.0, 132.8, 131.3, 130.6, 129.9, 129.7, 129.5, 20.7, 17.2.

IR v 3240, 1653, 1601, 1392, 1211, 1182, 1013, 792, 694 cm^{-1} .

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₇H₁₈NO₄S⁺ 332.0957. Found 332.0968 (3.3 ppm error).



2-(5,8-dimethyl-2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylethan-1-one

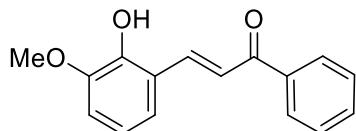
Compound 51: Synthesized using **General Procedure D** on a 200 mg (0.6 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (yellow solid, 137 mg, 0.41 mmol, 68% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.04 – 7.96 (m, 2H), 7.67 – 7.60 (m, 1H), 7.52 (dd, *J* = 8.4, 7.0 Hz, 2H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.00 (d, *J* = 7.6 Hz, 1H), 5.45 (dd, *J* = 10.5, 2.6 Hz, 1H), 4.32 (dd, *J* = 18.2, 10.5 Hz, 1H), 3.14 (dd, *J* = 18.2, 2.7 Hz, 1H), 2.26 (s, 3H), 2.23 (s, 3H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 197.5, 150.9, 137.6, 134.5, 134.3, 131.4, 129.7, 129.0, 127.9, 126.1, 122.6, 52.4, 41.6, 18.7, 15.2.

IR ν 3315, 1673, 1596, 1406, 1352, 1199, 1013, 829, 683 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₇H₁₈NO₄S⁺ 332.0957. Found 332.0940 (5.1 ppm error).



(E)-3-(2-hydroxy-3-methoxyphenyl)-1-phenylprop-2-en-1-one

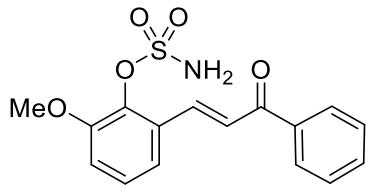
Compound S52': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

¹H NMR (400 MHz, CD₃CN) δ 8.08 – 7.96 (m, 3H), 7.76 – 7.68 (m, 1H), 7.66 – 7.59 (m, 1H), 7.53 (ddd, *J* = 8.9, 6.9, 2.1 Hz, 2H), 7.31 (dd, *J* = 7.8, 2.4 Hz, 1H), 7.17 (dd, *J* = 4.1, 1.9 Hz, 1H), 7.04 – 6.96 (m, 1H), 6.88 (td, *J* = 8.0, 2.4 Hz, 1H), 3.87 (s, 3H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 191.0, 148.2, 146.8, 139.6, 139.1, 133.4, 129.4, 129.0, 123.1, 121.9, 120.9, 120.4, 113.7, 56.7.

IR ν 3065, 1650, 1598, 1570, 1285, 1211, 1073, 996, 760, 686 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₆H₁₄O₃Na⁺ 277.0841. Found 277.0865 (2.4 mmu error).



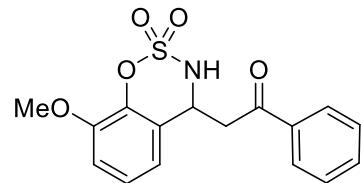
Compound S52: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

¹H NMR (400 MHz, Acetone-d₆) δ 8.22 – 8.06 (m, 3H), 7.87 – 7.79 (m, 1H), 7.68 – 7.59 (m, 2H), 7.55 (td, *J* = 7.7, 1.8 Hz, 2H), 7.38 – 7.30 (m, 1H), 7.23 (dd, *J* = 8.3, 1.9 Hz, 1H), 7.08 (s, 1H), 3.93 (s, 3H).

¹³C{¹H} NMR (101 MHz, Acetone-d₆) δ 190.1, 154.0, 139.9, 138.9 (2C), 133.7, 131.5, 129.5, 129.4, 128.1, 125.2, 120.0, 115.4, 56.5.

IR ν 3206, 1656, 1598, 1573, 1374, 1274, 1156, 1064, 858, 754, 691 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₆H₁₆NO₅S⁺ 334.0749. Found 334.0771 (2.2 mmu error).



2-(8-methoxy-2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylethan-1-one

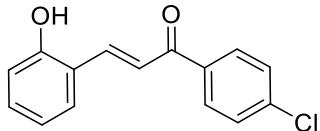
Compound 52: Synthesized using **General Procedure D** on a 200 mg (0.59 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (yellow solid, 110 mg, 0.32 mmol, 54% yield).

¹H NMR (400 MHz, CD₃CN) δ 8.01 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.69 – 7.62 (m, 1H), 7.53 (dd, *J* = 8.4, 7.1 Hz, 2H), 7.17 (t, *J* = 8.1 Hz, 1H), 7.03 (dd, *J* = 8.2, 1.4 Hz, 1H), 6.89 (dt, *J* = 8.0, 1.2 Hz, 1H), 6.36 (broad s, 1H), 5.44 – 5.36 (m, 1H), 4.11 (dd, *J* = 18.1, 9.0 Hz, 1H), 3.87 (s, 3H), 3.52 (dd, *J* = 18.1, 3.6 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 197.6, 149.5, 141.3, 137.5, 134.6, 129.7, 129.0, 126.1, 124.2, 118.7, 112.8, 56.8, 54.2, 43.6.

IR ν 3335, 1673, 1578, 1475, 1268, 1156, 1085, 864, 780, 686 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₆H₁₅NO₅SNa⁺ 356.0569. Found 356.0567 (0.6 ppm error).



(*E*)-1-(4-chlorophenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one

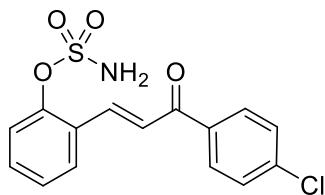
Compound S53': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

^1H NMR (400 MHz, CD₃CN) δ 8.10 – 7.97 (m, 3H), 7.76 – 7.65 (m, 3H), 7.53 (d, *J* = 8.6 Hz, 2H), 7.33 – 7.25 (m, 1H), 7.00 – 6.90 (m, 2H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CD₃CN) δ 190.0, 157.5, 140.8, 139.3, 137.9, 132.9, 131.0, 130.0, 129.7, 122.7, 122.6, 121.3, 117.1.

IR ν 3312, 1650, 1604, 1578, 1458, 1340, 1234, 1090, 987, 835, 740 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₂ClO₂⁺ 259.0526. Found 259.0545 (1.9 mmu error).



(*E*)-2-(3-(4-chlorophenyl)-3-oxoprop-1-en-1-yl)phenyl sulfamate

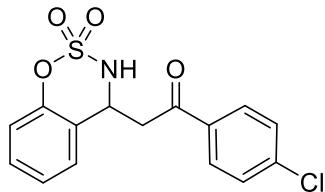
Compound S53: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

^1H NMR (400 MHz, CD₃CN) δ 8.11 – 7.94 (m, 4H), 7.72 (d, *J* = 15.7 Hz, 1H), 7.61 – 7.45 (m, 4H), 7.42 (td, *J* = 7.4, 1.6 Hz, 1H), 6.25 (broad s, 2H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CD₃CN) δ 189.6, 150.1, 139.7, 138.6, 137.3, 132.7, 131.2, 129.8, 129.6, 129.2, 128.2, 125.0, 124.0.

IR ν 3220, 2958, 1653, 1598, 1398, 1168, 1085, 1007, 868, 769 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₃ClNO₄S⁺ 338.0254. Found 338.0267 (3.8 ppm error).



1-(4-chlorophenyl)-2-(2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)ethan-1-one

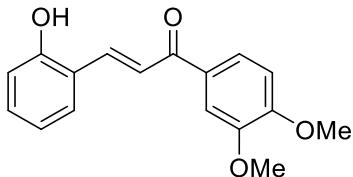
Compound 53: Synthesized using **General Procedure D** on a 200 mg (0.59 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (yellow solid, 175 mg, 0.51 mmol, 86% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.98 (d, *J* = 8.7 Hz, 2H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.41 – 7.33 (m, 2H), 7.24 (ddd, *J* = 8.2, 7.2, 1.3 Hz, 1H), 7.07 (dd, *J* = 8.2, 1.3 Hz, 1H), 6.35 (broad s, 1H), 5.41 (dd, *J* = 9.1, 3.5 Hz, 1H), 4.09 (dd, *J* = 18.1, 9.1 Hz, 1H), 3.52 (dd, *J* = 18.1, 3.6 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 196.6, 151.9, 140.3, 136.1, 130.8, 130.5, 129.9, 128.0, 126.4, 123.1, 119.4, 54.0, 43.7.

IR ν 3197, 1673, 1590, 1441, 1377, 1211, 1168, 1090, 760, 677 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₅H₁₃ClNO₄S⁺ 338.0254. Found 338.0273 (1.9 mmu error).



(*E*)-1-(3,4-dimethoxyphenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one

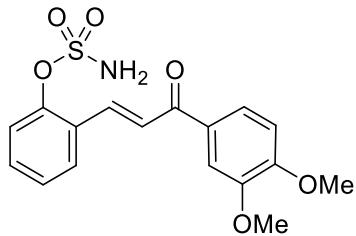
Compound S54': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

¹H NMR (400 MHz, CDCl₃) δ 8.27 – 8.19 (m, 1H), 7.77 – 7.68 (m, 2H), 7.66 – 7.57 (m, 2H), 7.30 – 7.22 (m, 1H), 6.99 – 6.89 (m, 3H), 3.95 (s, 6H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 190.5, 156.3, 153.4, 149.3, 140.7, 131.8, 131.4, 129.3, 123.5, 122.5, 122.2, 120.8, 116.9, 111.1, 110.2, 56.2, 56.1.

IR ν 3160, 1636, 1596, 1558, 1461, 1323, 1260, 1145, 1019, 826, 760 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₇H₁₇O₄⁺ 285.1127. Found 285.1145 (1.8 mmu error).



(*E*)-2-(3-(3,4-dimethoxyphenyl)-3-oxoprop-1-en-1-yl)phenyl sulfamate

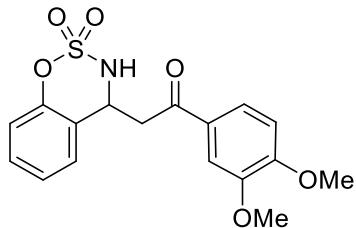
Compound S54: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

¹H NMR (400 MHz, Acetone-d₆) δ 8.13 – 7.98 (m, 2H), 7.92 (d, *J* = 15.7 Hz, 1H), 7.86 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.67 (d, *J* = 2.1 Hz, 1H), 7.57 – 7.43 (m, 2H), 7.40 (td, *J* = 7.4, 1.6 Hz, 1H), 7.09 (d, *J* = 8.5 Hz, 1H), 3.92 (s, 3H), 3.90 (s, 3H).

¹³C{¹H} NMR (101 MHz, Acetone-d₆) δ 188.1, 154.7, 150.4, 150.3, 137.4, 132.0, 131.9, 129.8, 129.2, 127.7, 124.9, 124.0, 123.7, 111.9, 111.5, 56.2, 56.1.

IR ν 3249, 1693, 1650, 1596, 1512, 1420, 1380, 1260, 1165, 1019, 878, 754 cm⁻¹.

HRMS (ESI) m/z = [M + Na]⁺ Calcd C₁₇H₁₇NO₆SNa⁺ 386.0674. Found 386.0685 (2.8 ppm error).



1-(3,4-dimethoxyphenyl)-2-(2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)ethan-1-one

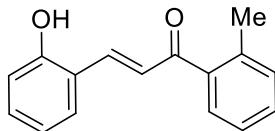
Compound 54: Synthesized using **General Procedure D** on a 100 mg (0.27 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (colorless oil, 80 mg, 0.22 mmol, 81% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.67 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.52 (d, *J* = 2.2 Hz, 1H), 7.39 – 7.33 (m, 2H), 7.24 (td, *J* = 7.5, 1.2 Hz, 1H), 7.10 – 7.03 (m, 1H), 7.00 (dd, *J* = 8.3, 1.2 Hz, 1H), 6.40 (broad s, 1H), 5.39 (dd, *J* = 8.9, 3.6 Hz, 1H), 4.10 (dd, *J* = 17.8, 8.9 Hz, 1H), 3.88 (s, 3H), 3.85 (s, 3H), 3.46 (dd, *J* = 17.8, 3.6 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CD₃CN) δ 196.1, 154.9, 152.0, 150.1, 130.5, 130.4, 128.0, 126.4, 124.0, 123.4, 119.4, 111.6, 111.1, 56.5, 56.3, 54.4, 42.9.

IR ν 3212, 1656, 1587, 1420, 1372, 1265, 1153, 763, 628 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₇H₁₈NO₆S⁺ 364.0855. Found 364.0852 (0.8 ppm error).



(*E*)-3-(2-hydroxyphenyl)-1-(*o*-tolyl)prop-2-en-1-one

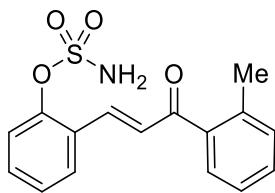
Compound S55': Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.96 – 7.91 (m, 1H), 7.75 (d, $J = 16.2$ Hz, 1H), 7.58 (d, $J = 16.2$ Hz, 1H), 7.51 (dd, $J = 7.5, 1.6$ Hz, 1H), 7.45 – 7.37 (m, 2H), 7.31 – 7.23 (m, 3H), 6.97 (dd, $J = 8.2, 1.1$ Hz, 1H), 6.91 (td, $J = 7.5, 1.2$ Hz, 1H), 2.46 (s, 3H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CDCl_3) δ 199.9, 156.7, 144.4, 138.9, 136.9, 132.2, 131.4, 130.7, 130.6, 128.4, 127.6, 125.6, 121.8, 120.6, 116.9, 20.3.

IR v 3171, 1641, 1590, 1558, 1446, 1334, 1274, 1188, 1021, 996, 740 cm^{-1} .

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₆H₁₅O₂⁺ 239.1072. Found 239.1090 (1.8 mmu error).



(*E*)-2-(3-oxo-3-(*o*-tolyl)prop-1-en-1-yl)phenyl sulfamate

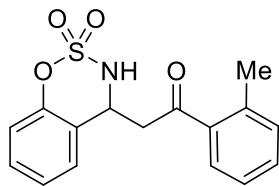
Compound S55: Purified using a gradient of 0 to 100% EtOAc/hexanes on silica gel; white solid

$^1\text{H NMR}$ (400 MHz, CD_3CN) δ 7.91 (d, $J = 7.8$ Hz, 1H), 7.77 (d, $J = 16.1$ Hz, 1H), 7.63 – 7.59 (m, 1H), 7.56 – 7.49 (m, 1H), 7.49 – 7.38 (m, 3H), 7.37 – 7.28 (m, 3H), 6.20 (s, 2H), 2.43 (s, 3H).

$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CD_3CN) δ 196.2, 149.9, 139.7, 139.0, 138.0, 132.6, 132.2, 131.7, 129.5, 129.4, 129.3, 129.0, 128.3, 126.5, 123.9, 20.4.

IR v 3171, 1641, 1590, 1558, 1360, 1294, 1208, 1188, 1021, 996, 740, 597 cm^{-1} .

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₆H₁₆NO₄S⁺ 318.0800. Found 318.0818 (1.8 mmu error).



2-(2,2-dioxido-3,4-dihydrobenzo[e][1,2,3]oxathiazin-4-yl)-1-(*o*-tolyl)ethan-1-one

Compound 55: Synthesized using **General Procedure D** on a 318 mg (1.00 mmol) scale; Purified using a gradient of 10 to 50% EtOAc/hexanes on silica gel; (white solid, 228 mg, 0.71 mmol, 71% yield).

¹H NMR (400 MHz, CD₃CN) δ 7.80 – 7.73 (m, 1H), 7.44 (td, *J* = 7.6, 1.3 Hz, 1H), 7.40 – 7.29 (m, 4H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 6.38 (broad s, 1H), 5.37 (dd, *J* = 9.4, 3.7 Hz, 1H), 3.95 (dd, *J* = 17.8, 9.3 Hz, 1H), 3.49 (dd, *J* = 17.8, 3.6 Hz, 1H), 2.47 (s, 3H).

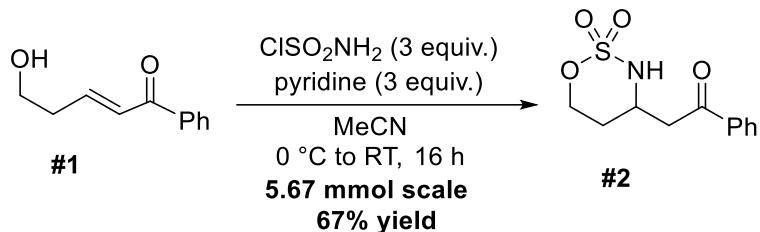
¹³C{¹H} NMR (101 MHz, CD₃CN) δ 201.4, 152.0, 138.9, 138.4, 132.8, 132.7, 130.4, 129.6, 128.0, 126.8, 126.4, 123.2, 119.4, 54.3, 46.3, 21.1.

IR ν 3266, 1679, 1598, 1452, 1374, 1205, 1165, 1001, 849, 752 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₁₆H₁₆NO₄S⁺ 318.0800. Found 318.0826 (2.6 mmu error).

V. **Scale-up, Product Diversification, and Characterization (Scheme 6)**

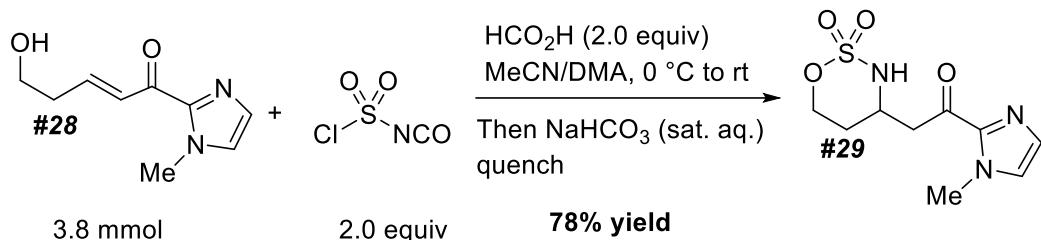
Scale-up Reaction:



Preparation of CISO_2NH_2 : An oven-dried 50 mL round bottom flask fitted with a balloon of N_2 gas was charged with a stir bar and CISO_2NCO (1.47 mL, 2.40 g, 17 mmol, 3.0 equiv). The reaction mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. HCO_2H (0.64 mL, 0.78 g, 17 mmol, 3.0 equiv) was added dropwise (**Caution: vigorous gas evolution upon addition**). The mixture solidified into a white solid within 5-10 minutes of addition. CH_3CN (10 mL) was added to it, and the reaction mixture was warmed to room temperature over a period of 16 h.

Cyclization: A separate oven-dried 100 mL round bottom flask fitted with a nitrogen balloon was charged with a stir bar, **1** (1 g, 5.67 mmol, 1.0 equiv), and CH_3CN (10 mL). The flask was cooled to 0 °C using an ice-water bath. Next, pyridine (1.36 mL, 1.34 g, 16.94 mmol, 3.0 equiv) was added to it, and the reaction mixture was stirred at same temperature for 5 minutes. Then, freshly prepared CISO_2NH_2 (in CH_3CN) was added dropwise, and the vial was rinsed by adding another 5 mL of CH_3CN (Final reaction concentration = ~0.2 M relative to **1**). With stirring, the reaction mixture was warmed to room temperature over a period of 16 h. Following this time, the reaction was quenched by the addition of 1 M aqueous HCl solution (25 mL). The mixture was transferred to a separatory funnel with EtOAc and further diluted with water. The organic layer was collected. The aqueous layer was extracted with EtOAc (3 x 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated *in vacuo*. The resulting residue was purified on silica gel using a gradient of 5 to 50% $\text{EtOAc}/\text{hexanes}$ to give **2** as a white solid (0.980 g, 3.83 mmol, 67% yield).

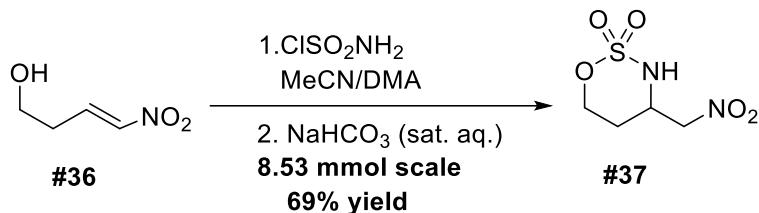
Scale-up reaction:



Preparation of ClSO₂NH₂: An oven-dried round bottom flask equipped with a stir bar was fitted with a balloon of N₂ gas. ClSO₂NCO (662 μ L, 1.08 g, 7.6 mmol, 2.0 equiv.) was added to it, and the vial was cooled to 0 °C using an ice-water bath. HCO₂H (287.0 μ L, 0.350 g, 7.6 mmol, 2.0 equiv.) was added dropwise with vigorous stirring (**Caution: gas evolution upon addition**). The mixture solidified into a white solid within 10 minutes of addition. CH₃CN (6.5 mL) was added, and the reaction mixture was slowly warmed to room temperature over a period of 12 h with constant stirring.

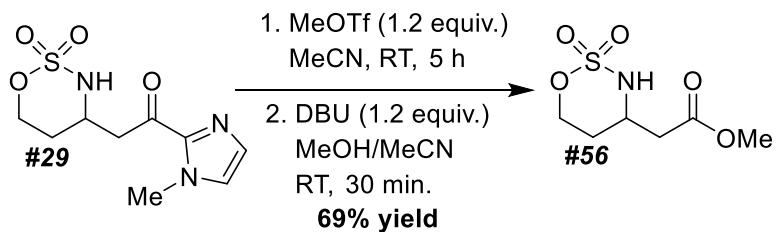
Cyclization: A separate oven-dried round bottom flask fitted with a N₂ gas balloon was charged with a stir bar, **28** (685.0 mg, 3.8 mmol, 1 equiv.), and DMA (4.0 mL, \approx 1 M). The reaction mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. Then, freshly prepared ClSO₂NH₂ (in CH₃CN) was added dropwise. The ice-bath was removed, and the reaction mixture was stirred for 3 h. Following this time, the reaction was quenched by the addition of saturated aqueous NaHCO₃ solution (25 mL) and stirred for another 15 minutes. The mixture was carefully transferred to a separatory funnel using EtOAc and further diluted with 30 mL water. The biphasic mixture was shaken well, and the organic layer (with white precipitate of product) was separated. The aqueous layer was extracted with ethyl acetate (75 mL x 3 times). The combined organic layers were concentrated until the volume halved and then transferred to a separatory funnel. The organic layer was washed with cold water (100 mL). The organic layer was separated, dried over MgSO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified using a gradient of 1-6% MeOH in EtOAc on silica gel (short column) to yield **29** as a white solid (769 mg, 2.96 mmol, 78% yield).

Scale-up reaction:

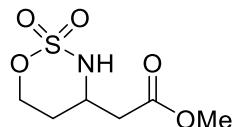


Preparation of CISO_2NH_2 : An oven-dried 50 mL round bottom flask with a stir bar was fitted with a balloon of N_2 gas. CISO_2NCO (1.47 mL, 2.4 g, 17 mmol, 2.0 equiv) was added to it, and the vial was cooled to 0 °C using an ice-water bath. HCO_2H (0.64 mL, 0.781 g, 17 mmol, 2.0 equiv) was added dropwise with stirring (**Caution: vigorous gas evolution upon addition**). The mixture solidified into a white solid within 5-10 minutes of addition. CH_3CN (16 mL) was added, and the reaction mixture was slowly warmed to room temperature over a period of 16 h with constant stirring.

Cyclization: A separate oven-dried 50 mL round bottom flask fitted with a N_2 gas balloon was charged with a stir bar, **36** (1.0 g, 8.53 mmol, 1 equiv), and DMA (8 mL). The reaction mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. Then, freshly prepared CISO_2NH_2 (in CH_3CN) was added dropwise (final reaction concentration = 0.35 M, relative to **36**). The ice-water bath was removed, and the reaction mixture was stirred for 6 h. Following this time, the reaction was quenched by the addition of saturated aqueous NaHCO_3 solution (50 mL) and vigorously stirred for another 20 minutes. The mixture was carefully transferred to a separatory funnel using EtOAc (50 mL) and further diluted with 40 mL water. The biphasic mixture was shaken well, and the organic layer was separated. The aqueous layer was extracted with ethyl acetate (50 mL x 3). The combined organic layer was washed with LiCl solution (aqueous, ~10% w/w) (70 mL x 2). The organic layer was separated, dried over anhydrous Na_2SO_4 , filtered, and fully concentrated *in vacuo*. The resulting residue was purified on silica gel gradient of 10 to 70% $\text{EtOAc}/\text{hexanes}$ to give **37** as a white solid (1.17 g, 5.96 mmol, 69% yield).



An oven-dried 10 mL round bottom flask was charged with a stir bar, **29** (0.070 g, 0.26 mmol, 1 equiv), anhydrous CH₃CN (1.5 ml), and 4Å molecular sieves (150 mg). MeOTf (0.035 mL, 0.053 g, 0.32 mmol, 1.2 equiv) was added, and the reaction mixture was stirred for 5 hours at room temperature under a N₂ atmosphere. Following this time, MeOH (0.5 mL) and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) (0.048 mL, 0.049 g, 0.32 mmol, 1.2 equiv) were added. The resulting mixture was allowed to stir for an additional 30 min at room temperature. Following this time, the reaction mixture was diluted with EtOAc (2 mL) and transferred to a separatory funnel. The organic layer was sequentially washed with saturated aqueous NaHCO₃ solution (1 mL) and brine (1 mL). The organic layer was collected, dried over MgSO₄, and filtered. After concentrating under reduced pressure, the resulting residue was purified by chromatography on silica gel (gradient of 10 to 70% EtOAc/hexanes) to give **56** as a colorless oil (0.039 g, 0.18 mmol, 69% yield).



methyl 2-(2,2-dioxido-1,2,3-oxathiazinan-4-yl)acetate

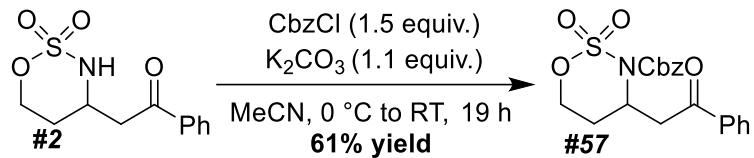
Compound **56**:

¹H NMR (400 MHz, CDCl₃) δ 5.14 (d, *J* = 10.3 Hz, 1H), 4.72 (ddd, *J* = 12.6, 11.6, 2.5 Hz, 1H), 4.55 (ddd, *J* = 11.6, 5.0, 1.8 Hz, 1H), 4.06 (dddd, *J* = 9.9, 6.5, 5.1, 2.9 Hz, 1H), 3.73 (s, 3H), 2.66 (qd, *J* = 16.7, 5.3 Hz, 2H), 2.05 – 1.89 (m, 1H), 1.79 – 1.68 (m, 1H).

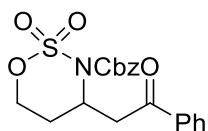
¹³C{¹H} NMR (101 MHz, CDCl₃) δ 171.1, 71.6, 52.5, 52.4, 37.8, 28.3.

IR ν 3258, 1723, 1427, 1360, 1293, 1179, 1064, 862, 542 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₆H₁₂NO₅S⁺ 210.0436. Found 210.0461 (2.5 mmu error).



A 10 mL round-bottom flask was charged with a stir bar, **2** (0.200 g, 0.78 mmol, 1 equiv.), and anhydrous CH_3CN (5 mL). The reaction flask was cooled to 0 °C using an ice-water bath. Subsequently, K_2CO_3 (0.119 g, 0.86 mmol, 1.1 equiv.) followed by CbzCl (0.168 mL, 0.202 g, 1.18 mmol, 1.5 equiv.) were added. The reaction mixture was warmed to room temperature over a period of 19 hours with stirring. Following this time, the reaction was quenched with a saturated aqueous solution of NH_4Cl (7 mL). The mixture was transferred to a separatory funnel. The aqueous layer was extracted with EtOAc (3 x 5 mL). The organic layers were collected, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. The resulting residue was purified using a gradient of 5 to 30% EtOAc/hexanes on silica gel to give **57** (colorless oil, 0.189 g, 0.48 mmol, 61% yield).



benzyl 4-(2-oxo-2-phenylethyl)-1,2,3-oxathiazinane-3-carboxylate 2,2-dioxide

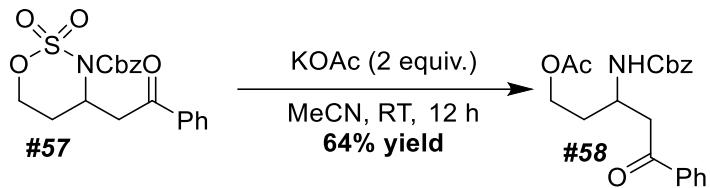
Compound **57**:

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 (dd, $J = 8.4, 1.3$ Hz, 2H), 7.64 – 7.56 (m, 1H), 7.48 (dd, $J = 8.4, 7.1$ Hz, 2H), 7.43 – 7.38 (m, 2H), 7.38 – 7.29 (m, 3H), 5.38 – 5.24 (m, 3H), 4.80 (dt, $J = 11.3, 5.6$ Hz, 1H), 4.68 (ddd, $J = 11.4, 6.7, 2.8$ Hz, 1H), 3.63 (dd, $J = 17.4, 10.6$ Hz, 1H), 3.47 (ddd, $J = 17.5, 3.6, 1.2$ Hz, 1H), 2.66 – 2.53 (m, 1H), 2.25 – 2.17 (m, 1H).

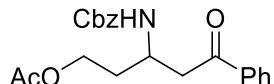
$^{13}\text{C}\{\text{H}\} \text{NMR}$ (101 MHz, CDCl_3) δ 196.8, 151.8, 136.1, 134.7, 133.9, 128.9, 128.7, 128.6, 128.2, 128.0, 70.2, 69.7, 54.2, 40.7, 25.9.

IR ν 1730, 1682, 1382, 1274, 1174, 1057, 977, 753 cm^{-1} .

HRMS (ESI) m/z = [M + H]⁺ Calcd $\text{C}_{19}\text{H}_{20}\text{NO}_6\text{S}^+$ 390.1011. Found 390.1017 (1.5 ppm error).



A 10 mL round-bottom flask was charged with a stir bar, **57** (40 mg, 0.10 mmol, 1 equiv.), anhydrous CH₃CN (2 mL), and KOAc (20 mg, 0.20 mmol, 2 equiv.). The reaction mixture was stirred at room temperature under a balloon of N₂ (~1 atm) for 12 hours. Following this time, the reaction was diluted with water (5 mL). The mixture was transferred to a separatory funnel. The aqueous layer was extracted with EtOAc (3 x 5 mL). The organic layers were combined, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The resulting residue was purified using a gradient of 5 to 60% EtOAc/hexanes on silica gel to yield **58** (colorless oil, 24 mg, 0.064 mmol, 64% yield).



3-((benzyloxy)carbonyl)amino-5-oxo-5-phenylpentyl acetate

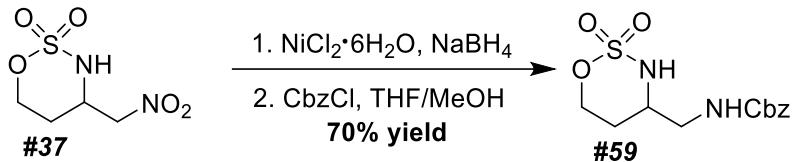
Compound **58**:

¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 7.7 Hz, 2H), 7.61 – 7.55 (m, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.42 – 7.29 (m, 5H), 5.52 (d, *J* = 9.0 Hz, 1H), 5.07 (s, 2H), 4.29 – 4.09 (m, 3H), 3.43 (dd, *J* = 17.5, 4.7 Hz, 1H), 3.19 (dd, *J* = 17.4, 5.6 Hz, 1H), 2.15 – 2.07 (m, 1H), 2.04 – 1.90 (m, 4H).

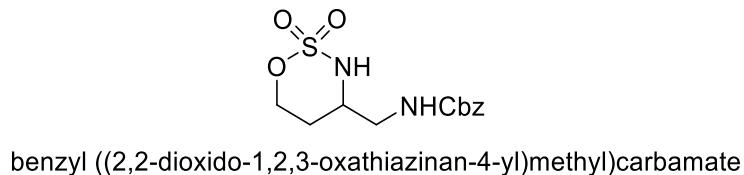
¹³C{¹H} NMR (101 MHz, CDCl₃) δ 198.6, 171.0, 155.8, 136.7, 136.4, 133.5, 128.7, 128.5, 128.1, 128.0, 66.6, 61.5, 45.7, 41.9, 32.7, 20.8.

IR ν 2929, 1716, 1687, 1525, 1449, 1228, 1044, 733 cm⁻¹.

HRMS (ESI) m/z = [M + H]⁺ Calcd C₂₁H₂₄NO₅⁺ 370.1654. Found 370.1656 (0.5 ppm error).



A 10 mL round-bottom flask was charged with a stir bar, **37** (74 mg, 0.37 mmol, 1 equiv.), THF (1.5 mL), MeOH (1.5 mL), and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (179 mg, 0.75 mmol, 2 equiv.). The reaction mixture was stirred at room temperature for 10 min and then cooled to 0 °C using an ice-water bath. After stirring for 10 min., NaBH_4 (85 mg, 2.25 mmol, 6 equiv.) was added in two portions (~43 mg each) under a flow of nitrogen. The ice-water bath was removed, and the reaction was allowed to warm to room temperature over a period of 30 minutes. Following this time, CbzCl (0.324 mL, 0.389 g, 2.28 mmol, 6 equiv.) was added, and the reaction was stirred for an additional 3 hours. Following this time, the reaction was diluted with water (10 mL) and transferred to a separatory funnel. The aqueous layer was extracted with diethyl ether (3 x 20 mL), ethyl acetate (3 x 20 mL), and CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. The resulting residue was purified using a gradient of 10 to 80% EtOAc/hexanes on silica gel to give **59** (white solid, 80 mg, 0.26 mmol, 70% yield).



Compound 59:

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48 – 7.24 (m, 5H), 5.28 – 5.17 (m, 1H), 5.10 (s, 2H), 4.94 – 4.80 (m, 1H), 4.69 (td, $J = 12.1, 2.7$ Hz, 1H), 4.54 (dd, $J = 11.9, 4.7$ Hz, 1H), 3.91 – 3.74 (m, 1H), 3.48 – 3.35 (m, 1H), 3.31 (dt, $J = 14.4, 6.9$ Hz, 1H), 1.79 (td, $J = 12.9, 4.5$ Hz, 1H), 1.74 – 1.62 (m, 1H).

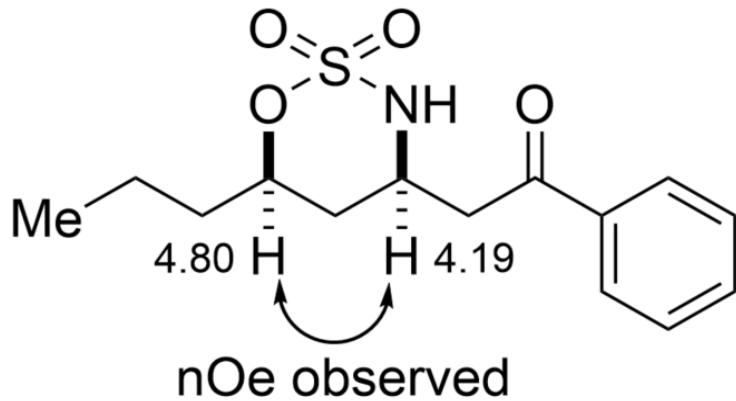
$^{13}\text{C}\{\text{H}\} \text{NMR}$ δ 157.3, 136.0, 128.7, 128.5, 128.2, 71.6, 67.5, 56.3, 44.2, 26.9.

IR ν 3229, 1720, 1670, 1540, 1454, 1352, 1270, 1149, 1017, 774 cm^{-1} .

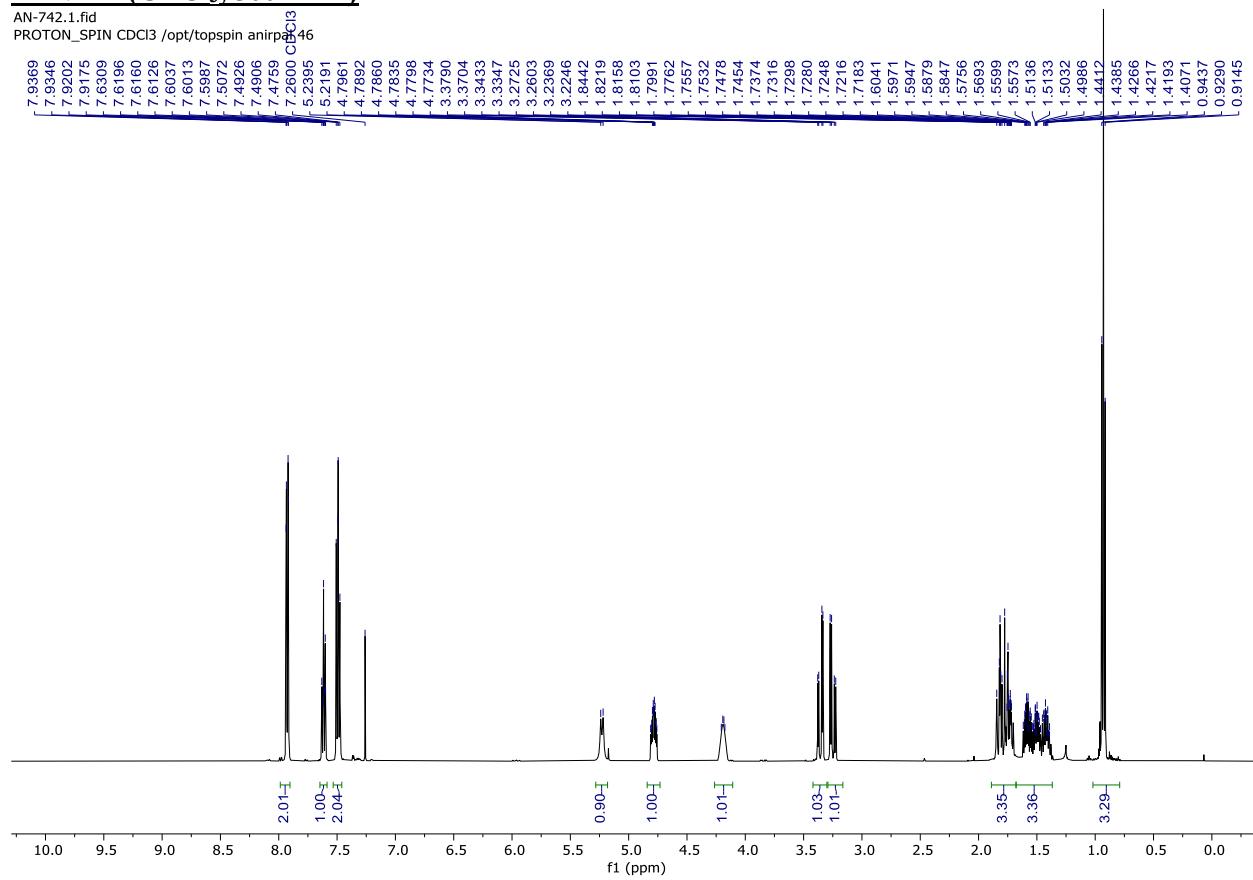
HRMS (ESI) m/z = [M + Na]⁺ Calcd $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_5\text{SNa}^+$ 323.0678. Found 323.0694 (5.0 ppm error).

VI. Structural Reasoning

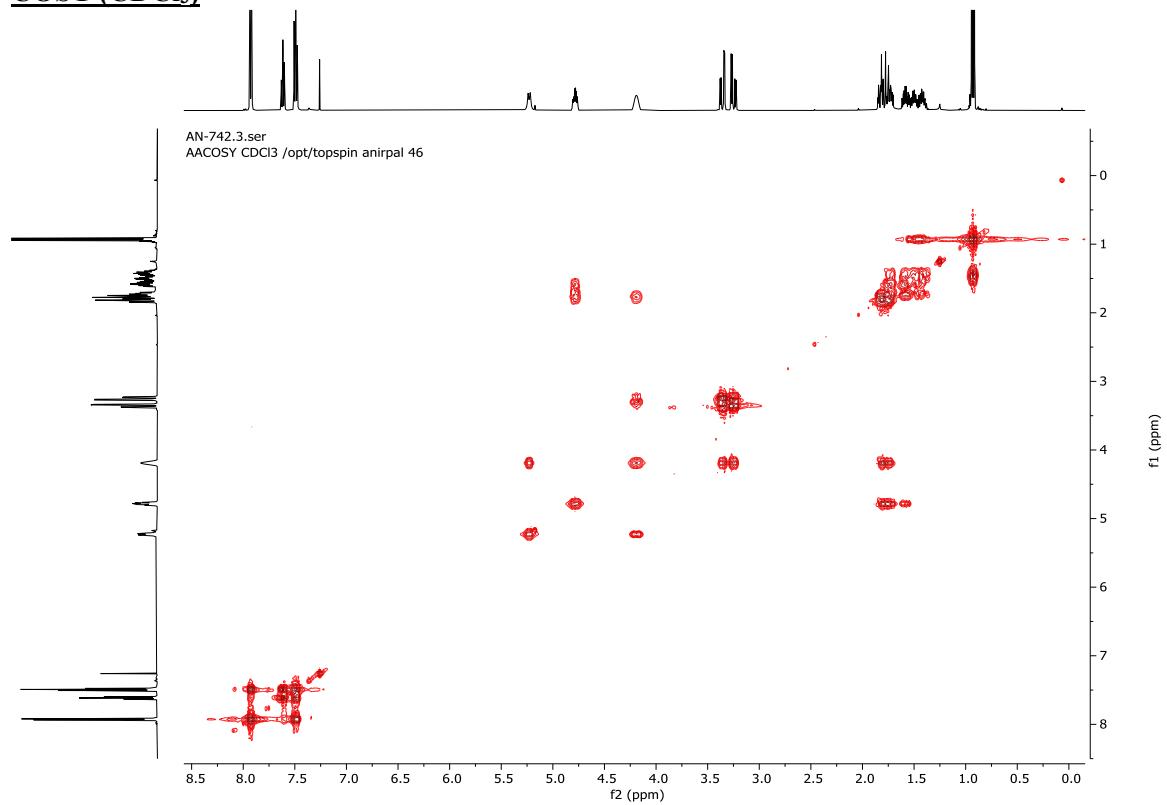
Compound 39



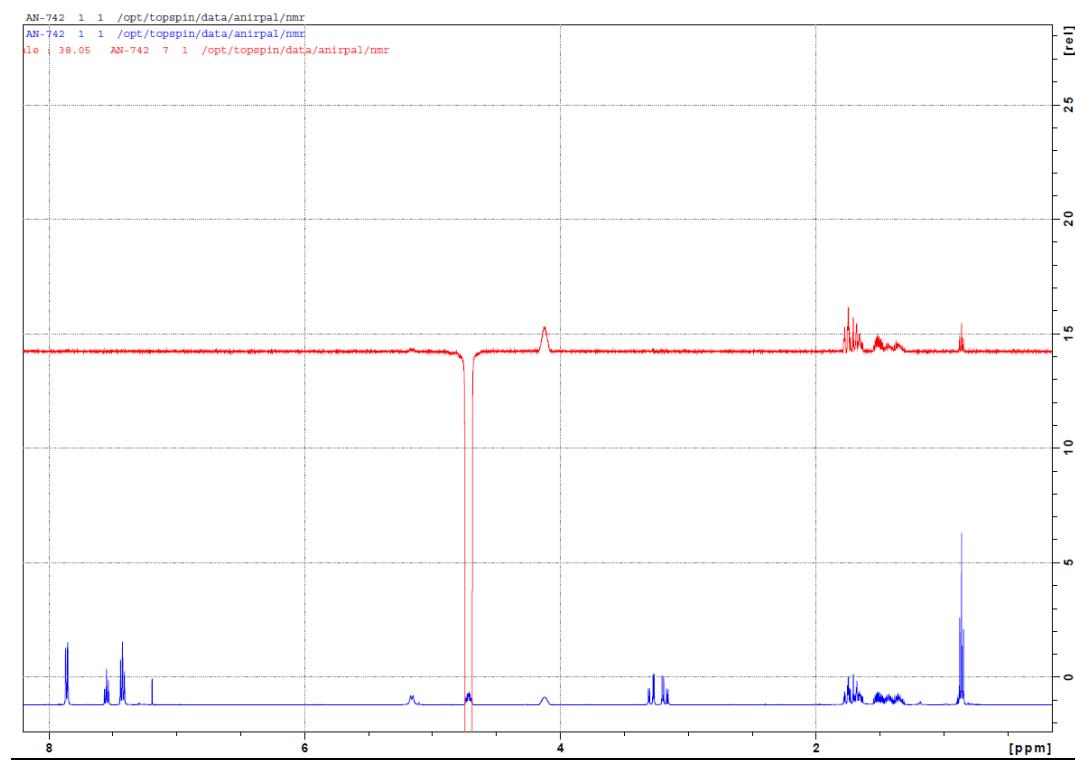
¹H NMR (CDCl₃, 500 MHz)



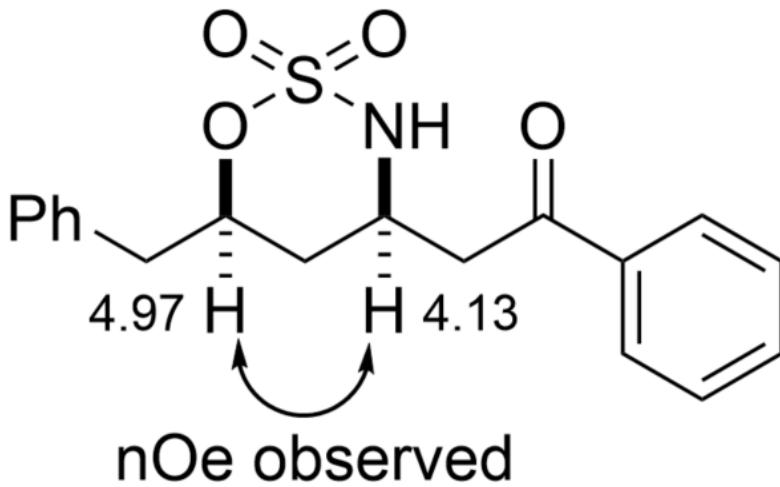
COSY (CDCl₃)



1-D nOe

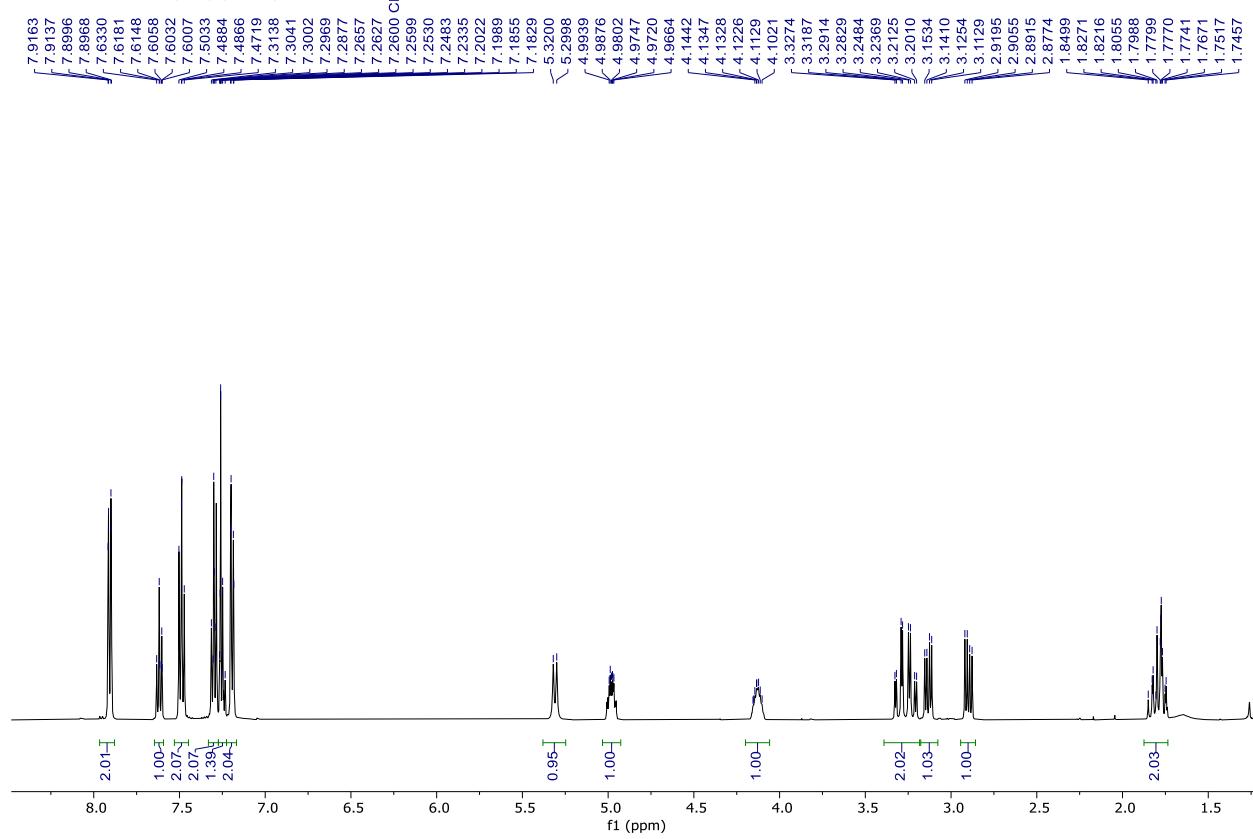


Compound 41

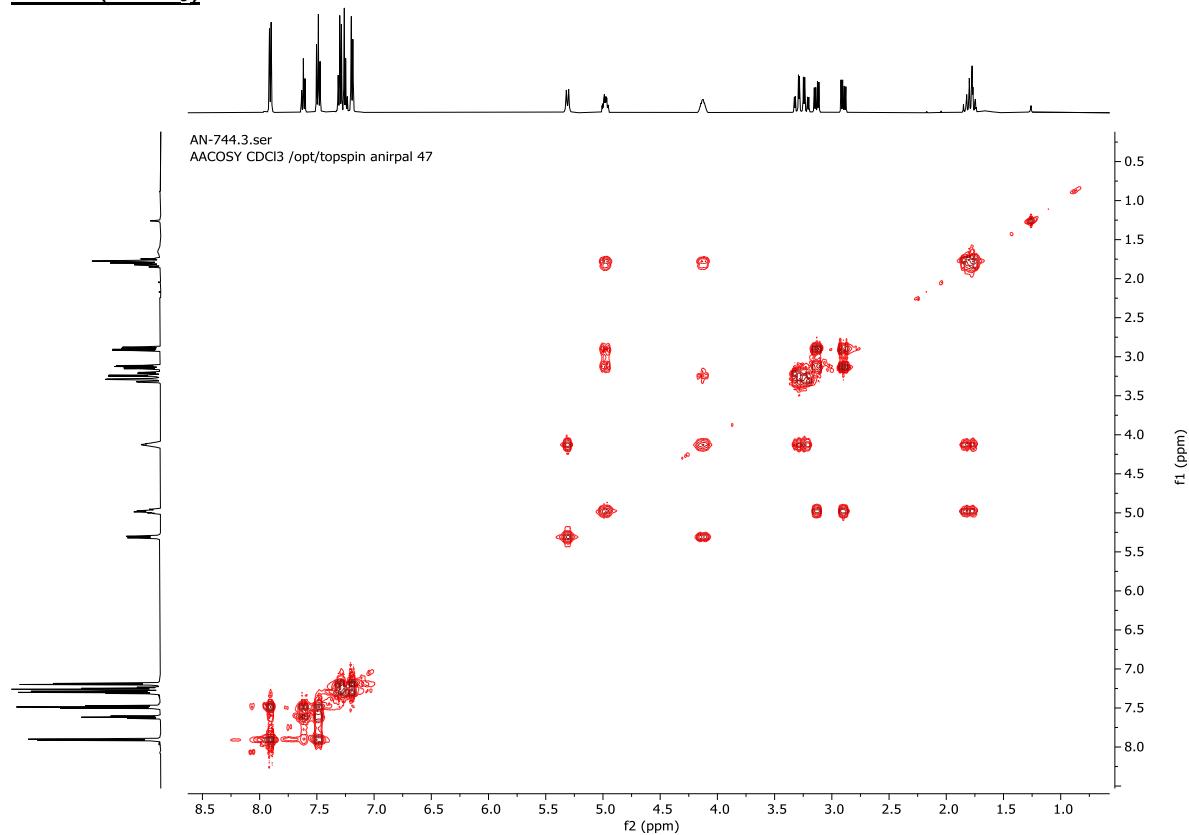


^1H NMR (CDCl₃, 500 MHz)

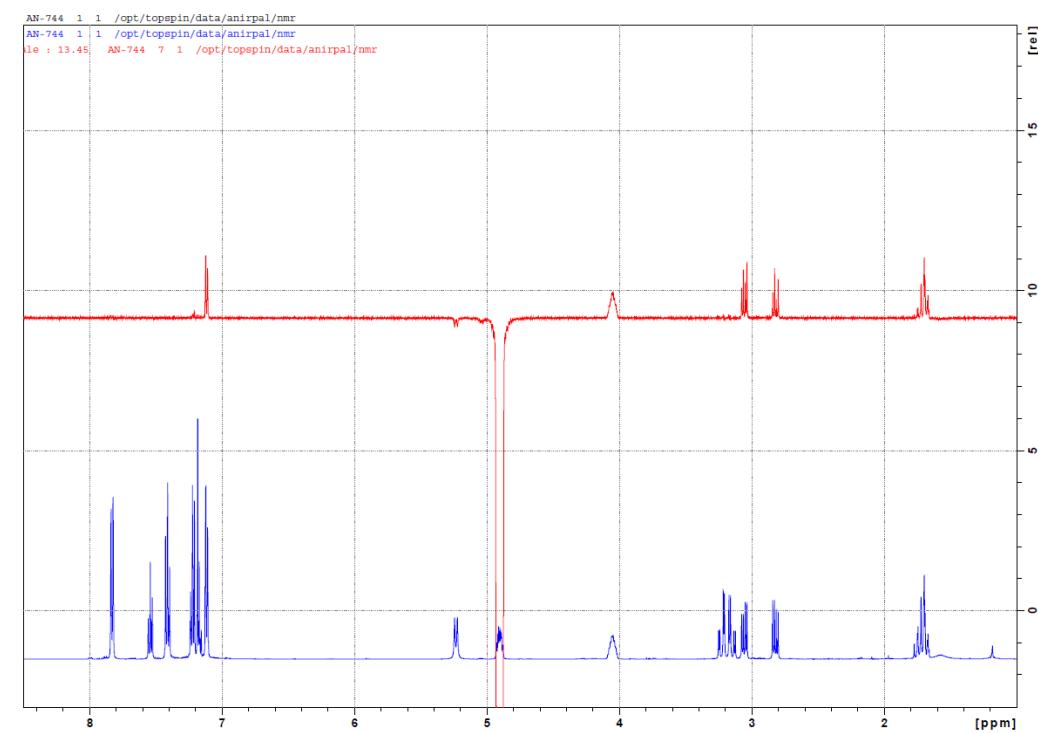
AN-744.1.fid
PROTON_SPIN CDCl₃ /opt/topspin anirpal 47



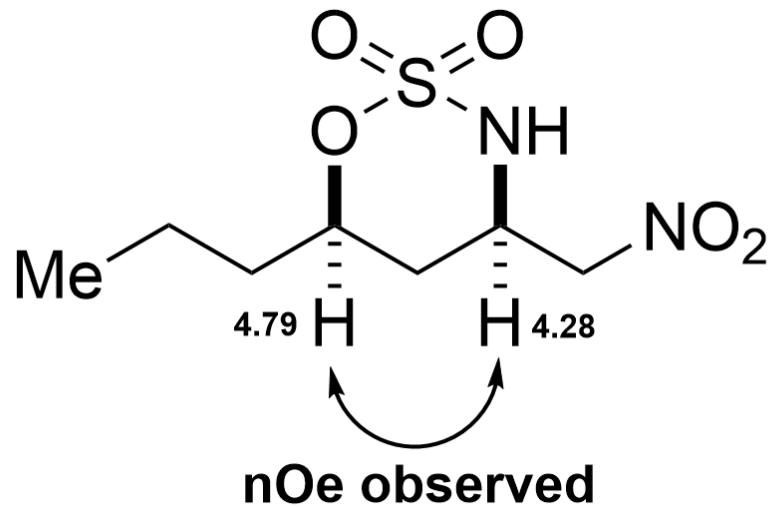
COSY (CDCl₃)



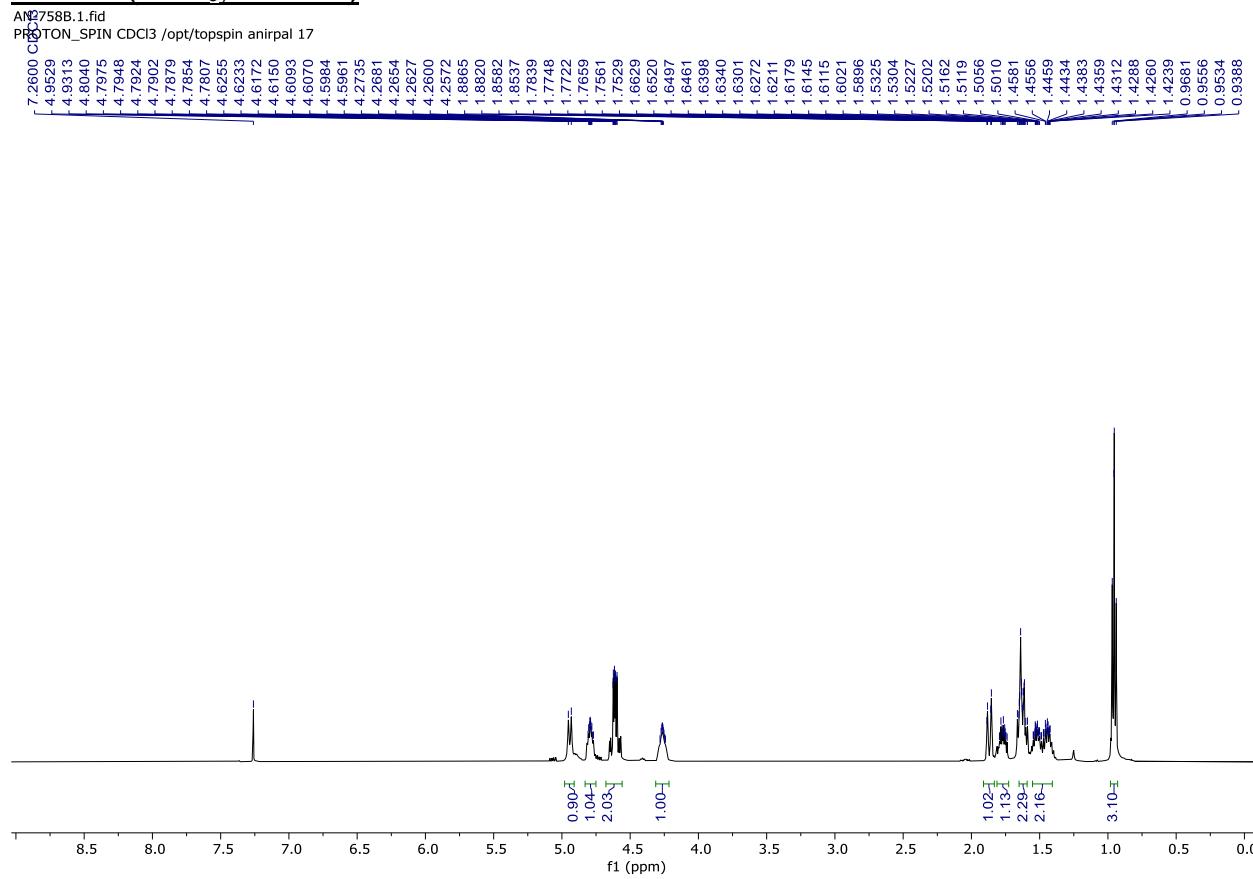
1-D nOe



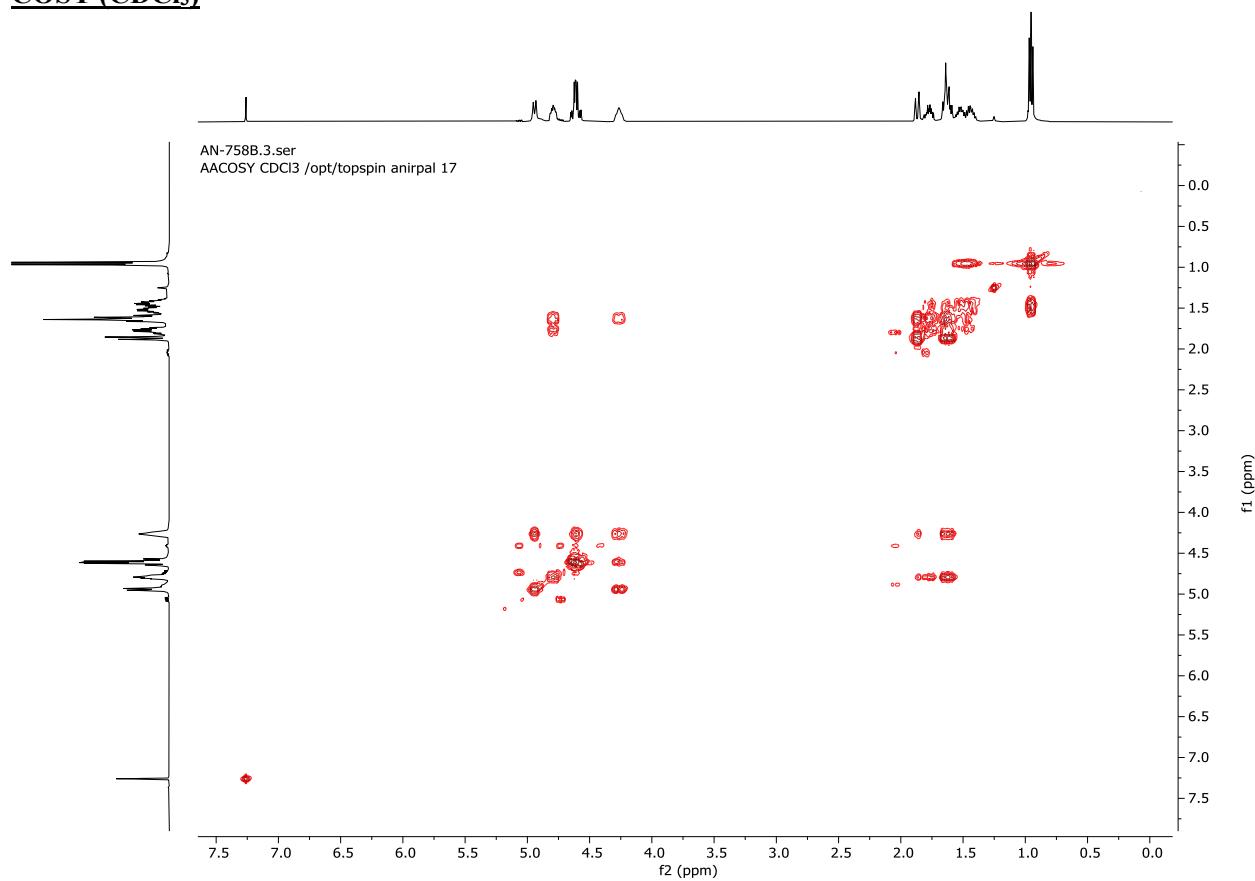
Compound 43



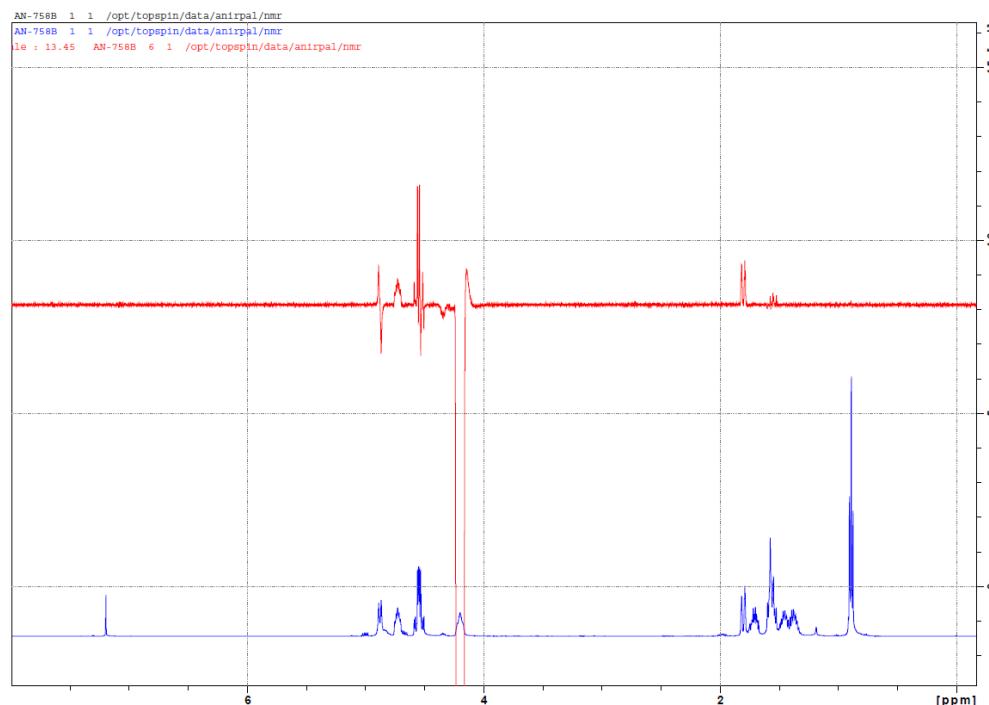
^1H NMR (CDCl_3 , 500 MHz)



COSY (CDCl₃)



1-D nOe

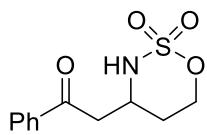


VII. X-ray Crystallographic Data

Single crystal X-ray diffraction data for **2** and **31** were collected on a Bruker D8 Venture diffractometer equipped with a Photon II CMOS area detector using Mo-K α radiation from a microfocus source (Bruker AXS, Madison, WI, USA). Crystals were cooled to the collection temperatures under streams of N₂ gas using a Cryostream 800 cryostat (Oxford Cryosystems, Oxford, UK). Hemispheres of data were collected using strategies of scans about the omega and phi axes. Data collection, unit cell determination, data reduction, absorption correction and scaling, and space group determination were performed using the Bruker Apex3 software suite.¹

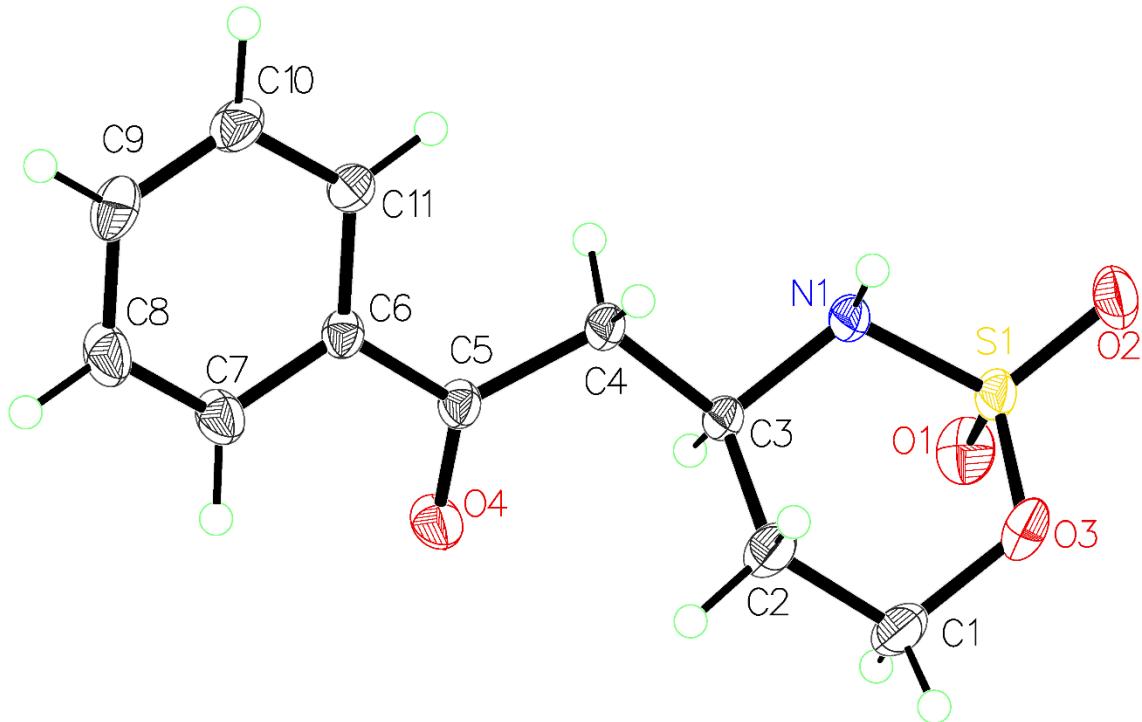
Both structures were solved by direct methods as implemented in SHELXS v.2013-1² and refined by full matrix least squares refinement against F² using SHELXL v. 2019/3.³ Olex2 was used as a graphical interface for model building and data visualization.⁴ For both structures all non-hydrogen atoms could be located from the difference map and refined anisotropically. Hydrogen atoms bonded to strong hydrogen bond donors were located from the difference map, and their coordinates were refined. In **31** the thermal parameters and difference map indicated possible disorder which was found to be consistent with the presence of two mostly overlapping diastereomers in the structure. The occupancies of the major and minor parts were fixed at 80% and 20%, respectively, and both parts could be refined anisotropically. Hydrogen atoms bonded to carbon were placed in calculated positions for both structures. In **2** all hydrogen atom coordinates could be refined while in **31** they were constrained to ride on the carrier atoms. All hydrogen atom thermal parameters were constrained to ride on the carrier atoms.

- 1.Apex4, AXScale, and SAINT, version 2022.1, Bruker AXS, Inc., Madison, WI, 2022.
- 2.Sheldrick, G. M. SHELXS, v.2013-1, 2013.
- 3.Sheldrick, G. M. Crystal structure refinement with *SHELXL*. *Acta Cryst. Sect. C. Struct. Chem.* **2015**, *71*, 3-8.
- 4.Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H. *OLEX2: A complete structure solution, refinement, and analysis program*. *J. Appl. Cryst.* **2009**, *42*, 339-341.



Compound 2 (CCDC 2353012)

Crystals grown from EtOAc/hexanes.



Labeled 50% probability ellipsoid plot of asymmetric formula unit of AN-541.

Table S1. Crystal data and structure refinement for AN-541.

Identification code	s1	
Empirical formula	C11 H13 N O4 S	
Formula weight	255.28	
Temperature	173.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 1 2/c 1	
Unit cell dimensions	a = 21.1502(10) Å b = 5.6688(3) Å c = 20.5070(10) Å	a = 90°. b = 110.8228(15)°. g = 90°.
Volume	2298.1(2) Å ³	
Z	8	
Density (calculated)	1.476 Mg/m ³	
Absorption coefficient	0.284 mm ⁻¹	
F(000)	1072	
Crystal size	0.23 x 0.19 x 0.06 mm ³	
Theta range for data collection	2.060 to 33.210°.	
Index ranges	-32<=h<=32, -8<=k<=8, -31<=l<=31	
Reflections collected	32480	
Independent reflections	4397 [R(int) = 0.0389]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7465 and 0.6831	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4397 / 0 / 193	
Goodness-of-fit on F ²	1.051	
Final R indices [I>2sigma(I)]	R1 = 0.0422, wR2 = 0.1026	
R indices (all data)	R1 = 0.0515, wR2 = 0.1103	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.361 and -0.496 e.Å ⁻³	

Table S2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for AN-541. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	-1211(1)	8610(1)	-228(1)	21(1)
O(1)	-1388(1)	6906(2)	-772(1)	31(1)
O(2)	-1227(1)	11053(2)	-389(1)	37(1)
O(3)	-1712(1)	8295(2)	179(1)	28(1)
O(4)	100(1)	1551(2)	1433(1)	30(1)
N(1)	-472(1)	7974(2)	330(1)	21(1)
C(1)	-1671(1)	5938(3)	495(1)	32(1)
C(2)	-972(1)	5558(3)	1033(1)	28(1)
C(3)	-422(1)	5710(2)	714(1)	18(1)
C(4)	288(1)	5619(2)	1263(1)	20(1)
C(5)	469(1)	3269(2)	1628(1)	19(1)
C(6)	1110(1)	3081(2)	2243(1)	18(1)
C(7)	1213(1)	1081(2)	2664(1)	26(1)
C(8)	1811(1)	806(3)	3229(1)	30(1)
C(9)	2313(1)	2519(3)	3376(1)	27(1)
C(10)	2213(1)	4518(3)	2964(1)	26(1)
C(11)	1612(1)	4807(2)	2398(1)	22(1)

Table S3. Bond lengths [\AA] and angles [$^\circ$] for AN-541.

S(1)-O(1)	1.4223(11)
S(1)-O(2)	1.4218(11)
S(1)-O(3)	1.5746(11)
S(1)-N(1)	1.6149(10)
O(3)-C(1)	1.4738(19)
O(4)-C(5)	1.2227(15)
N(1)-C(3)	1.4895(15)
C(1)-C(2)	1.5141(19)
C(2)-C(3)	1.5258(17)
C(3)-C(4)	1.5240(16)
C(4)-C(5)	1.5095(16)
C(5)-C(6)	1.4905(16)
C(6)-C(7)	1.3950(17)
C(6)-C(11)	1.3952(17)
C(7)-C(8)	1.3873(18)
C(8)-C(9)	1.391(2)
C(9)-C(10)	1.384(2)
C(10)-C(11)	1.3933(17)
O(1)-S(1)-O(3)	107.45(6)
O(1)-S(1)-N(1)	108.88(6)
O(2)-S(1)-O(1)	120.08(7)
O(2)-S(1)-O(3)	105.56(7)
O(2)-S(1)-N(1)	108.66(6)
O(3)-S(1)-N(1)	105.22(6)
C(1)-O(3)-S(1)	112.98(8)
C(3)-N(1)-S(1)	115.85(8)
O(3)-C(1)-C(2)	109.80(12)
C(1)-C(2)-C(3)	111.98(11)
N(1)-C(3)-C(2)	110.71(10)
N(1)-C(3)-C(4)	106.58(9)
C(4)-C(3)-C(2)	112.53(10)
C(5)-C(4)-C(3)	113.92(10)
O(4)-C(5)-C(4)	121.42(11)
O(4)-C(5)-C(6)	120.12(11)

C(6)-C(5)-C(4)	118.46(10)
C(7)-C(6)-C(5)	118.54(11)
C(7)-C(6)-C(11)	119.27(11)
C(11)-C(6)-C(5)	122.18(11)
C(8)-C(7)-C(6)	120.31(12)
C(7)-C(8)-C(9)	120.11(13)
C(10)-C(9)-C(8)	120.01(12)
C(9)-C(10)-C(11)	120.06(12)
C(10)-C(11)-C(6)	120.23(12)

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for AN-541. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2}U^{11} + \dots + 2hk a^{*}b^{*}U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	17(1)	20(1)	21(1)	2(1)	1(1)	2(1)
O(1)	32(1)	34(1)	20(1)	-3(1)	2(1)	2(1)
O(2)	30(1)	23(1)	46(1)	11(1)	-2(1)	3(1)
O(3)	19(1)	35(1)	29(1)	2(1)	6(1)	9(1)
O(4)	28(1)	19(1)	31(1)	0(1)	-4(1)	-5(1)
N(1)	16(1)	18(1)	23(1)	3(1)	0(1)	0(1)
C(1)	17(1)	45(1)	33(1)	11(1)	8(1)	2(1)
C(2)	18(1)	42(1)	24(1)	10(1)	8(1)	4(1)
C(3)	15(1)	18(1)	18(1)	1(1)	3(1)	1(1)
C(4)	15(1)	18(1)	23(1)	2(1)	1(1)	1(1)
C(5)	18(1)	17(1)	19(1)	-1(1)	2(1)	1(1)
C(6)	17(1)	18(1)	17(1)	-1(1)	3(1)	2(1)
C(7)	27(1)	21(1)	23(1)	3(1)	1(1)	-1(1)
C(8)	32(1)	26(1)	24(1)	6(1)	-1(1)	3(1)
C(9)	22(1)	33(1)	20(1)	-1(1)	-1(1)	5(1)
C(10)	18(1)	30(1)	25(1)	-2(1)	2(1)	-2(1)
C(11)	18(1)	22(1)	22(1)	1(1)	2(1)	-1(1)

Table S5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for AN-541.

	x	y	z	U(eq)
H(1A)	-2010(11)	5990(40)	702(11)	38
H(1B)	-1778(10)	4770(40)	127(11)	38
H(1)	-342(11)	9100(40)	578(11)	38
H(2A)	-893(10)	6700(30)	1432(10)	34
H(2B)	-973(10)	4010(40)	1244(10)	34
H(3)	-484(9)	4470(30)	375(9)	21
H(4A)	326(9)	6800(30)	1604(9)	24
H(4B)	608(9)	5930(30)	1044(9)	24
H(7)	859(10)	-90(30)	2560(10)	31
H(8)	1871(10)	-540(40)	3514(10)	36
H(9)	2713(10)	2360(40)	3758(10)	33
H(10)	2558(10)	5720(30)	3057(10)	31
H(11)	1560(9)	6140(30)	2109(10)	26

Table S6. Torsion angles [°] for AN-541.

S(1)-O(3)-C(1)-C(2)	62.79(14)
S(1)-N(1)-C(3)-C(2)	-51.01(13)
S(1)-N(1)-C(3)-C(4)	-173.68(8)
O(1)-S(1)-O(3)-C(1)	60.69(10)
O(1)-S(1)-N(1)-C(3)	-64.85(11)
O(2)-S(1)-O(3)-C(1)	-170.05(10)
O(2)-S(1)-N(1)-C(3)	162.73(10)
O(3)-S(1)-N(1)-C(3)	50.07(10)
O(3)-C(1)-C(2)-C(3)	-60.31(17)
O(4)-C(5)-C(6)-C(7)	-12.07(18)
O(4)-C(5)-C(6)-C(11)	166.45(13)
N(1)-S(1)-O(3)-C(1)	-55.22(10)
N(1)-C(3)-C(4)-C(5)	-170.79(10)
C(1)-C(2)-C(3)-N(1)	54.14(16)
C(1)-C(2)-C(3)-C(4)	173.29(12)
C(2)-C(3)-C(4)-C(5)	67.69(14)
C(3)-C(4)-C(5)-O(4)	8.03(18)
C(3)-C(4)-C(5)-C(6)	-171.29(10)
C(4)-C(5)-C(6)-C(7)	167.25(12)
C(4)-C(5)-C(6)-C(11)	-14.23(17)
C(5)-C(6)-C(7)-C(8)	178.08(13)
C(5)-C(6)-C(11)-C(10)	-177.67(12)
C(6)-C(7)-C(8)-C(9)	-0.4(2)
C(7)-C(6)-C(11)-C(10)	0.85(19)
C(7)-C(8)-C(9)-C(10)	0.9(2)
C(8)-C(9)-C(10)-C(11)	-0.6(2)
C(9)-C(10)-C(11)-C(6)	-0.3(2)
C(11)-C(6)-C(7)-C(8)	-0.5(2)

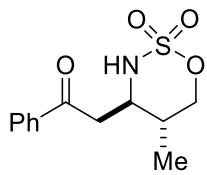
Symmetry transformations used to generate equivalent atoms:

Table S7. Hydrogen bonds for AN-541 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	∠(DHA)
N(1)-H(1)...O(4)#1	0.80(2)	2.17(2)	2.9572(15)	167(2)

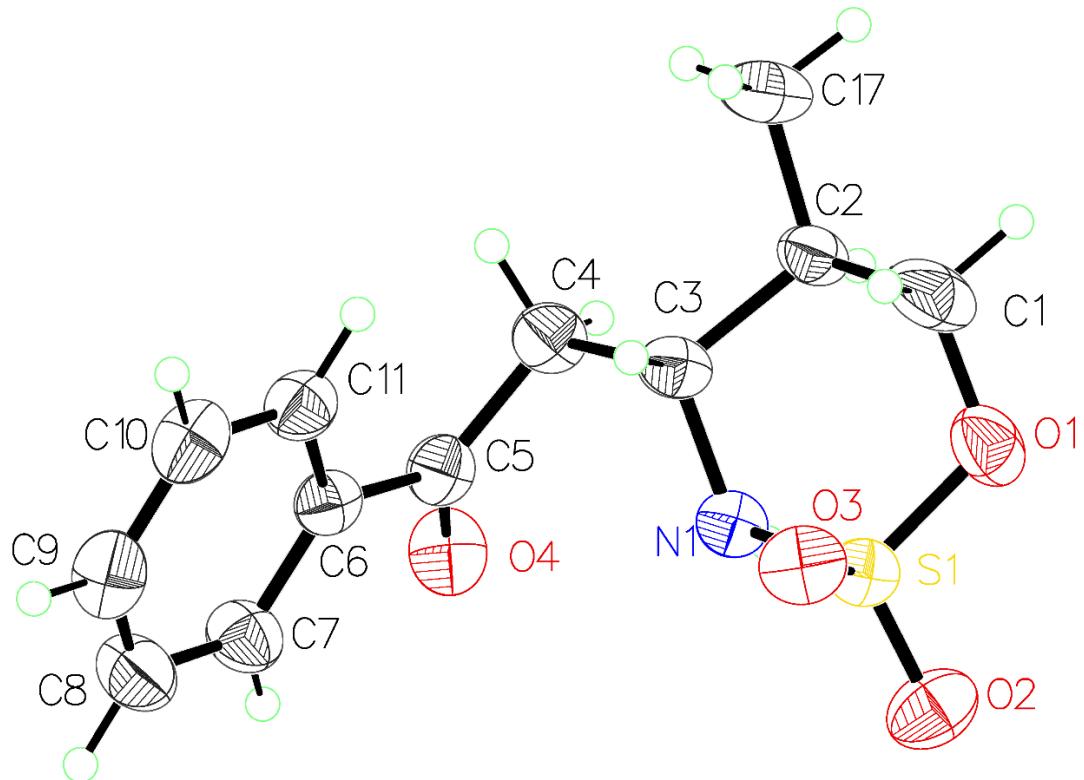
Symmetry transformations used to generate equivalent atoms:

#1 x,y+1,z



Compound 31 (CCDC 2353013)

Crystals grown from EtOAc/hexanes.



Labelled 50% probability ellipsoid plot for the major isomer of SBC-2045.

Table S8. Crystal data and structure refinement for SBC-2045.

Identification code	s1_pl	
Empirical formula	C12 H15 N O4 S	
Formula weight	269.31	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.7167(2) Å b = 9.6879(2) Å c = 10.0793(3) Å	a= 109.5650(10)°. b= 112.506(2)°. g = 96.2920(10)°.
Volume	630.99(3) Å ³	
Z	2	
Density (calculated)	1.417 Mg/m ³	
Absorption coefficient	0.263 mm ⁻¹	
F(000)	284	
Crystal size	0.25 x 0.05 x 0.05 mm ³	
Theta range for data collection	2.319 to 29.592°.	
Index ranges	-10<=h<=10, -13<=k<=13, -13<=l<=13	
Reflections collected	27629	
Independent reflections	3535 [R(int) = 0.0412]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	None	
Max. and min. transmission	0.7459 and 0.6960	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3535 / 0 / 192	
Goodness-of-fit on F ²	1.051	
Final R indices [I>2sigma(I)]	R1 = 0.0583, wR2 = 0.1183	
R indices (all data)	R1 = 0.0743, wR2 = 0.1287	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.562 and -0.479 e.Å ⁻³	

Table S9. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for SBC-2045. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
S(1)	4674(1)	3272(1)	7032(1)	37(1)
O(1)	3907(2)	4486(2)	6426(2)	52(1)
O(2)	5347(3)	2424(2)	5975(2)	55(1)
O(3)	5982(2)	4044(2)	8656(2)	48(1)
O(4)	-8(3)	-727(2)	6480(2)	48(1)
N(1)	2765(3)	2182(2)	6828(2)	35(1)
C(1)	2854(4)	5273(3)	7181(5)	62(1)
C(2)	1041(6)	4185(5)	6886(5)	38(1)
C(3)	1681(3)	3010(2)	7608(3)	37(1)
C(4)	-50(3)	1846(3)	7358(3)	41(1)
C(5)	506(3)	477(2)	7617(3)	38(1)
C(6)	1697(3)	597(2)	9235(2)	34(1)
C(7)	2366(4)	-644(3)	9382(3)	45(1)
C(8)	3436(4)	-631(3)	10832(3)	53(1)
C(9)	3857(4)	633(3)	12178(3)	50(1)
C(10)	3213(4)	1879(3)	12059(3)	48(1)
C(11)	2144(3)	1869(3)	10595(3)	42(1)
C(17)	-11(5)	5134(3)	7673(5)	54(1)
C(2')	1180(30)	4360(30)	7496(19)	52(5)
C(17')	-490(30)	4070(20)	5994(18)	69(4)

Table S10. Bond lengths [\AA] and angles [$^\circ$] for SBC-2045.

S(1)-O(3)	1.4168(17)
S(1)-O(2)	1.4181(18)
S(1)-O(1)	1.5698(18)
S(1)-N(1)	1.6157(18)
O(1)-C(1)	1.429(4)
O(4)-C(5)	1.219(3)
N(1)-C(3)	1.480(3)
N(1)-H(1)	0.91(3)
C(1)-C(2)	1.518(5)
C(1)-C(2')	1.68(2)
C(1)-H(1AA)	0.9900
C(1)-H(1AB)	0.9900
C(1)-H(1BC)	1.19(17)
C(1)-H(1BD)	0.94(17)
C(2)-C(17)	1.517(5)
C(2)-C(3)	1.573(5)
C(2)-H(2)	1.0000
C(3)-C(2')	1.43(2)
C(3)-C(4)	1.530(3)
C(3)-H(3A)	1.0000
C(3)-H(3B)	1.0000
C(4)-C(5)	1.510(3)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.490(3)
C(6)-C(11)	1.389(3)
C(6)-C(7)	1.390(3)
C(7)-C(8)	1.371(4)
C(7)-H(7)	0.9500
C(8)-C(9)	1.381(4)
C(8)-H(8)	0.9500
C(9)-C(10)	1.378(4)
C(9)-H(9)	0.9500
C(10)-C(11)	1.384(3)

C(10)-H(10)	0.9500
C(11)-H(11)	0.9500
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(2')-C(17')	1.47(2)
C(2')-H(2')	1.0000
C(17')-H(17D)	0.9800
C(17')-H(17E)	0.9800
C(17')-H(17F)	0.9800

O(3)-S(1)-O(2)	119.31(11)
O(3)-S(1)-O(1)	107.94(11)
O(2)-S(1)-O(1)	105.93(12)
O(3)-S(1)-N(1)	108.91(10)
O(2)-S(1)-N(1)	108.93(10)
O(1)-S(1)-N(1)	104.87(9)
C(1)-O(1)-S(1)	113.81(18)
C(3)-N(1)-S(1)	113.84(14)
C(3)-N(1)-H(1)	113.6(16)
S(1)-N(1)-H(1)	108.0(16)
O(1)-C(1)-C(2)	111.3(3)
O(1)-C(1)-C(2')	122.0(9)
O(1)-C(1)-H(1AA)	109.4
C(2)-C(1)-H(1AA)	109.4
O(1)-C(1)-H(1AB)	109.4
C(2)-C(1)-H(1AB)	109.4
H(1AA)-C(1)-H(1AB)	108.0
O(1)-C(1)-H(1BC)	88(8)
C(2')-C(1)-H(1BC)	125(8)
O(1)-C(1)-H(1BD)	110(10)
C(2')-C(1)-H(1BD)	91(10)
H(1BC)-C(1)-H(1BD)	125(10)
C(17)-C(2)-C(1)	107.2(3)
C(17)-C(2)-C(3)	111.3(3)
C(1)-C(2)-C(3)	108.7(3)

C(17)-C(2)-H(2)	109.9
C(1)-C(2)-H(2)	109.9
C(3)-C(2)-H(2)	109.9
C(2')-C(3)-N(1)	124.7(8)
C(2')-C(3)-C(4)	111.8(10)
N(1)-C(3)-C(4)	108.40(17)
N(1)-C(3)-C(2)	107.9(2)
C(4)-C(3)-C(2)	112.9(2)
N(1)-C(3)-H(3A)	109.2
C(4)-C(3)-H(3A)	109.2
C(2)-C(3)-H(3A)	109.2
C(2')-C(3)-H(3B)	103.1
N(1)-C(3)-H(3B)	103.1
C(4)-C(3)-H(3B)	103.1
C(5)-C(4)-C(3)	112.30(17)
C(5)-C(4)-H(4A)	109.1
C(3)-C(4)-H(4A)	109.1
C(5)-C(4)-H(4B)	109.1
C(3)-C(4)-H(4B)	109.1
H(4A)-C(4)-H(4B)	107.9
O(4)-C(5)-C(6)	120.2(2)
O(4)-C(5)-C(4)	119.1(2)
C(6)-C(5)-C(4)	120.67(19)
C(11)-C(6)-C(7)	118.6(2)
C(11)-C(6)-C(5)	123.5(2)
C(7)-C(6)-C(5)	117.85(19)
C(8)-C(7)-C(6)	121.2(2)
C(8)-C(7)-H(7)	119.4
C(6)-C(7)-H(7)	119.4
C(7)-C(8)-C(9)	119.7(2)
C(7)-C(8)-H(8)	120.1
C(9)-C(8)-H(8)	120.1
C(10)-C(9)-C(8)	120.0(2)
C(10)-C(9)-H(9)	120.0
C(8)-C(9)-H(9)	120.0
C(9)-C(10)-C(11)	120.3(2)

C(9)-C(10)-H(10)	119.9
C(11)-C(10)-H(10)	119.9
C(10)-C(11)-C(6)	120.2(2)
C(10)-C(11)-H(11)	119.9
C(6)-C(11)-H(11)	119.9
C(2)-C(17)-H(17A)	109.5
C(2)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(2)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(3)-C(2')-C(17')	113.7(17)
C(3)-C(2')-C(1)	107.6(15)
C(17')-C(2')-C(1)	95.9(12)
C(3)-C(2')-H(2')	112.8
C(17')-C(2')-H(2')	112.8
C(1)-C(2')-H(2')	112.8
C(2')-C(17')-H(17D)	109.5
C(2')-C(17')-H(17E)	109.5
H(17D)-C(17')-H(17E)	109.5
C(2')-C(17')-H(17F)	109.5
H(17D)-C(17')-H(17F)	109.5
H(17E)-C(17')-H(17F)	109.5

Symmetry transformations used to generate equivalent atoms:

Table S11. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for SBC-2045. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2}U^{11} + \dots + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	29(1)	34(1)	40(1)	11(1)	11(1)	5(1)
O(1)	43(1)	47(1)	74(1)	36(1)	23(1)	11(1)
O(2)	51(1)	52(1)	51(1)	6(1)	26(1)	7(1)
O(3)	38(1)	47(1)	40(1)	8(1)	9(1)	5(1)
O(4)	50(1)	39(1)	37(1)	8(1)	12(1)	-1(1)
N(1)	32(1)	29(1)	34(1)	8(1)	10(1)	4(1)
C(1)	47(2)	39(1)	111(3)	38(2)	37(2)	16(1)
C(2)	33(2)	35(2)	50(3)	21(2)	17(2)	14(1)
C(3)	31(1)	31(1)	43(1)	12(1)	14(1)	9(1)
C(4)	28(1)	42(1)	49(1)	20(1)	13(1)	7(1)
C(5)	30(1)	37(1)	42(1)	15(1)	15(1)	3(1)
C(6)	29(1)	30(1)	38(1)	10(1)	15(1)	3(1)
C(7)	52(1)	34(1)	42(1)	12(1)	19(1)	12(1)
C(8)	60(2)	50(1)	54(2)	26(1)	23(1)	22(1)
C(9)	39(1)	62(2)	42(1)	22(1)	12(1)	5(1)
C(10)	49(1)	42(1)	38(1)	4(1)	20(1)	-2(1)
C(11)	46(1)	33(1)	43(1)	9(1)	23(1)	7(1)
C(17)	39(2)	36(2)	83(2)	19(2)	28(2)	15(1)
C(2')	53(9)	70(11)	31(9)	27(9)	13(8)	10(7)
C(17')	76(11)	88(12)	59(9)	46(9)	26(8)	41(9)

Table S12. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for SBC-2045.

	x	y	z	U(eq)
H(1)	2020(40)	1590(30)	5780(30)	42
H(1AA)	2472	6051	6779	75
H(1AB)	3708	5804	8328	75
H(1BC)	2500(200)	5860(180)	6280(190)	75
H(1BD)	3700(300)	5800(200)	8300(200)	75
H(2)	179	3635	5728	46
H(3A)	2559	3572	8762	44
H(3B)	2579	3365	8753	44
H(4A)	-576	2339	8095	50
H(4B)	-1093	1504	6272	50
H(7)	2076	-1518	8460	54
H(8)	3887	-1488	10912	64
H(9)	4590	644	13185	60
H(10)	3505	2747	12986	57
H(11)	1714	2734	10520	50
H(17A)	-1216	4471	7477	81
H(17B)	-327	5892	7240	81
H(17C)	831	5654	8809	81
H(2')	1040	5019	8435	63
H(17D)	-219	3519	5117	104
H(17E)	-694	5044	5964	104
H(17F)	-1664	3463	5901	104

Table S13. Torsion angles [°] for SBC-2045.

O(3)-S(1)-O(1)-C(1)	-61.6(2)
O(2)-S(1)-O(1)-C(1)	169.49(18)
N(1)-S(1)-O(1)-C(1)	54.4(2)
O(3)-S(1)-N(1)-C(3)	60.96(17)
O(2)-S(1)-N(1)-C(3)	-167.40(16)
O(1)-S(1)-N(1)-C(3)	-54.37(17)
S(1)-O(1)-C(1)-C(2)	-61.7(3)
S(1)-O(1)-C(1)-C(2')	-44.4(8)
O(1)-C(1)-C(2)-C(17)	-177.7(3)
O(1)-C(1)-C(2)-C(3)	61.9(4)
S(1)-N(1)-C(3)-C(2')	46.5(11)
S(1)-N(1)-C(3)-C(4)	-178.64(15)
S(1)-N(1)-C(3)-C(2)	58.9(3)
C(17)-C(2)-C(3)-N(1)	-177.6(3)
C(1)-C(2)-C(3)-N(1)	-59.7(3)
C(17)-C(2)-C(3)-C(4)	62.7(4)
C(1)-C(2)-C(3)-C(4)	-179.4(3)
C(2')-C(3)-C(4)-C(5)	-174.9(7)
N(1)-C(3)-C(4)-C(5)	44.0(3)
C(2)-C(3)-C(4)-C(5)	163.4(2)
C(3)-C(4)-C(5)-O(4)	-106.0(2)
C(3)-C(4)-C(5)-C(6)	73.0(3)
O(4)-C(5)-C(6)-C(11)	-172.2(2)
C(4)-C(5)-C(6)-C(11)	8.9(3)
O(4)-C(5)-C(6)-C(7)	6.8(3)
C(4)-C(5)-C(6)-C(7)	-172.1(2)
C(11)-C(6)-C(7)-C(8)	0.3(4)
C(5)-C(6)-C(7)-C(8)	-178.7(2)
C(6)-C(7)-C(8)-C(9)	0.3(4)
C(7)-C(8)-C(9)-C(10)	-0.5(4)
C(8)-C(9)-C(10)-C(11)	0.0(4)
C(9)-C(10)-C(11)-C(6)	0.6(4)
C(7)-C(6)-C(11)-C(10)	-0.8(3)
C(5)-C(6)-C(11)-C(10)	178.2(2)

N(1)-C(3)-C(2')-C(17')	77.1(19)
C(4)-C(3)-C(2')-C(17')	-56.5(16)
N(1)-C(3)-C(2')-C(1)	-27.8(14)
C(4)-C(3)-C(2')-C(1)	-161.4(7)
O(1)-C(1)-C(2')-C(3)	26.9(13)
O(1)-C(1)-C(2')-C(17')	-90.3(14)

Symmetry transformations used to generate equivalent atoms:

Table S14. Hydrogen bonds for SBC-2045 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	∠(DHA)
N(1)-H(1)...O(4)#1	0.91(3)	2.01(3)	2.898(2)	165(2)
C(3)-H(3A^a)...O(3)#2	1.00	2.56	3.471(3)	152.1

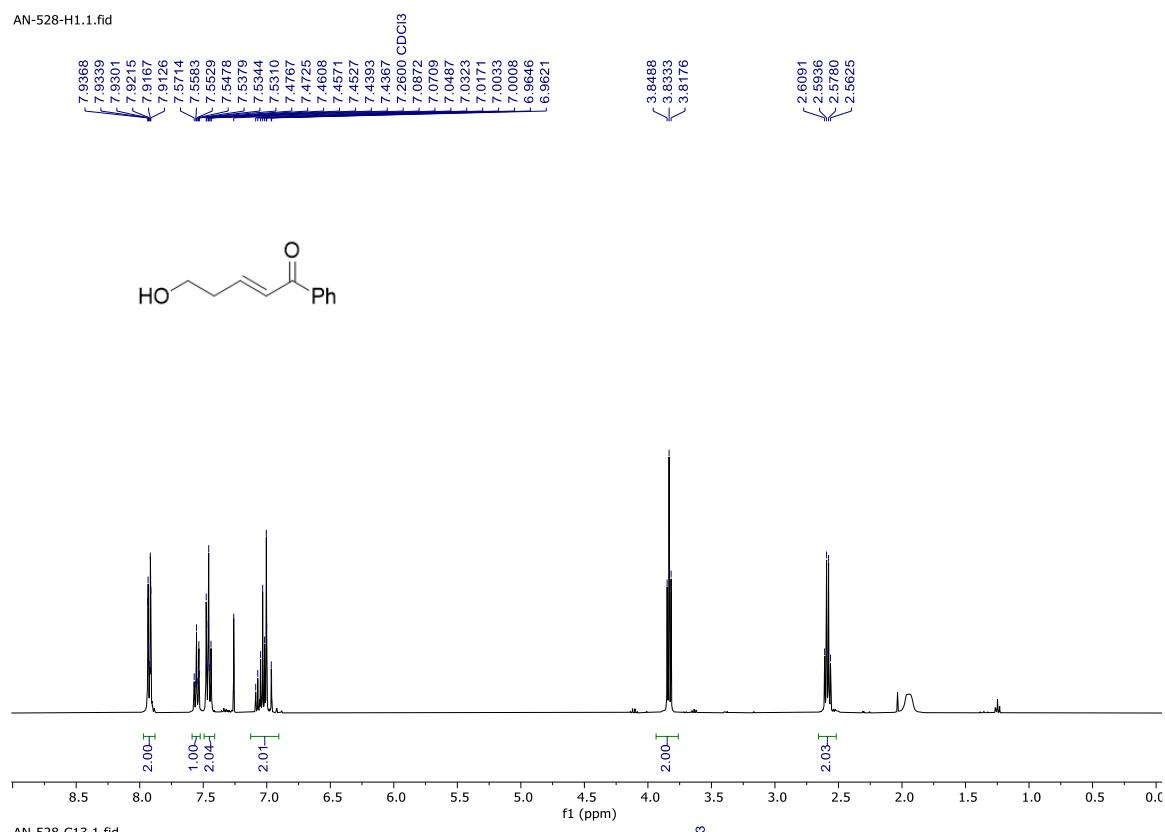
Symmetry transformations used to generate equivalent atoms:

#1 -x,-y,-z+1 #2 -x+1,-y+1,-z+2

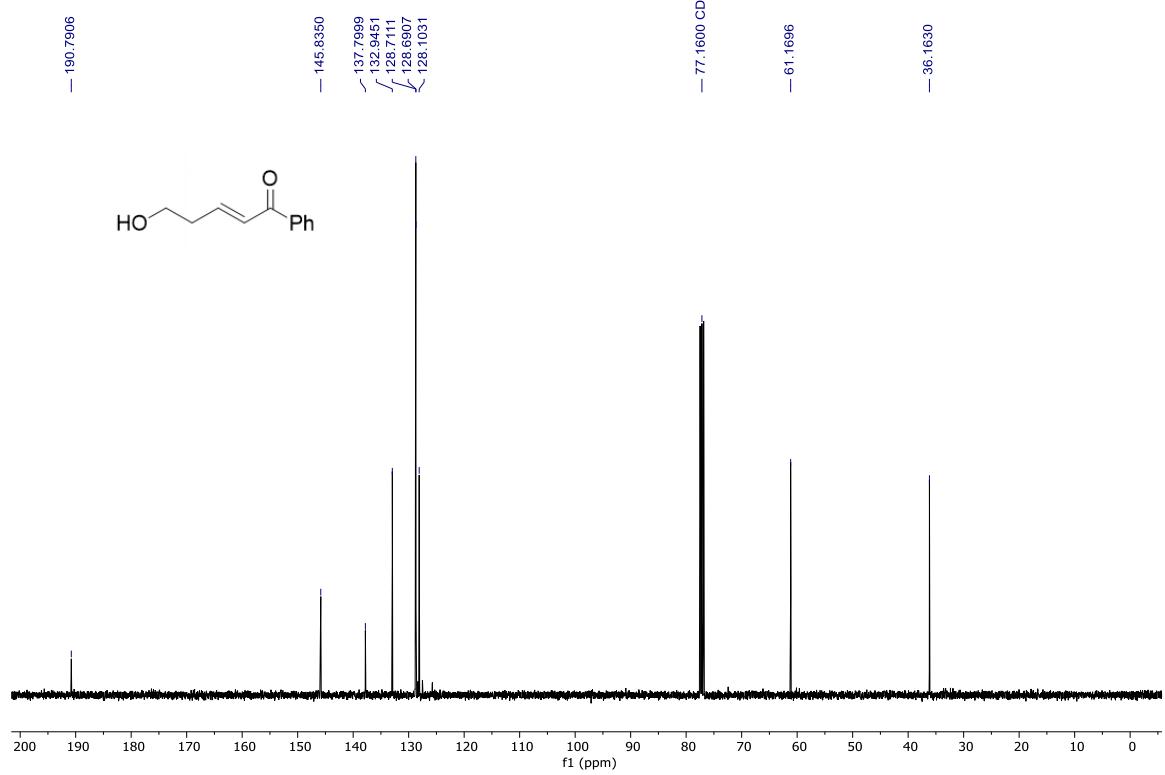
VIII. NMR Spectra

Compound 1 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-528-H1.1.fid

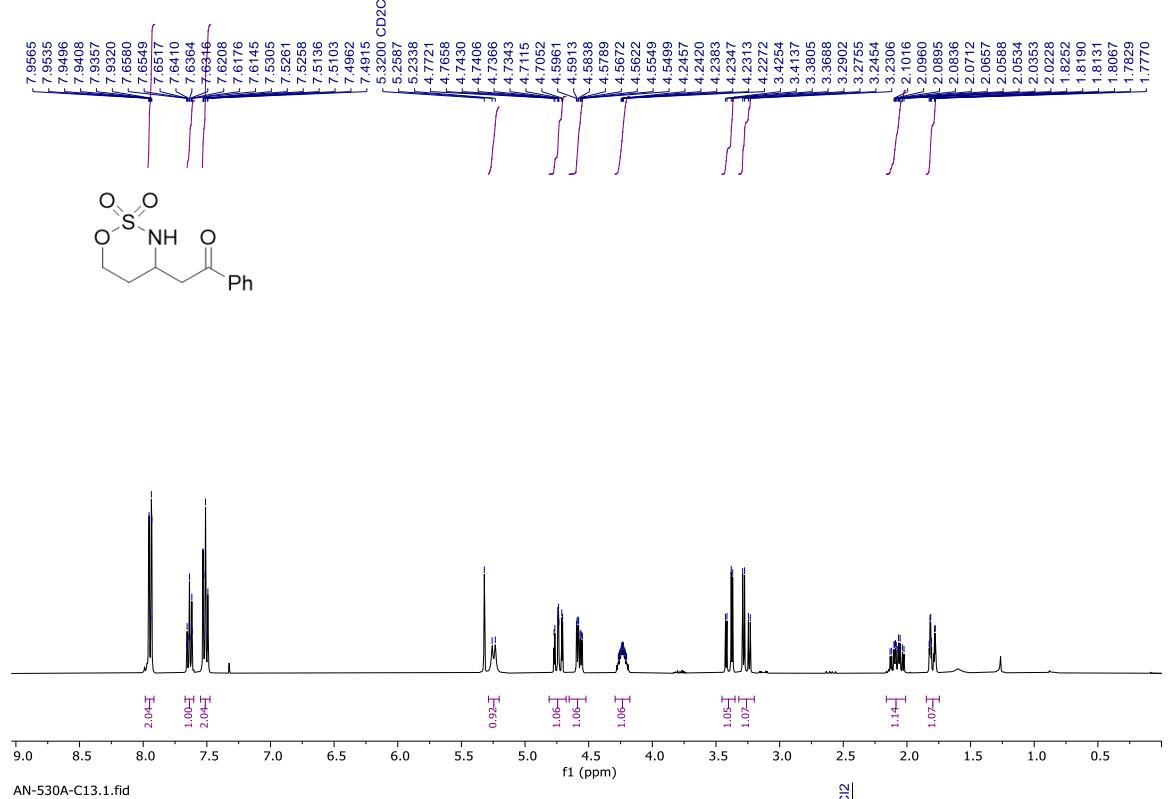


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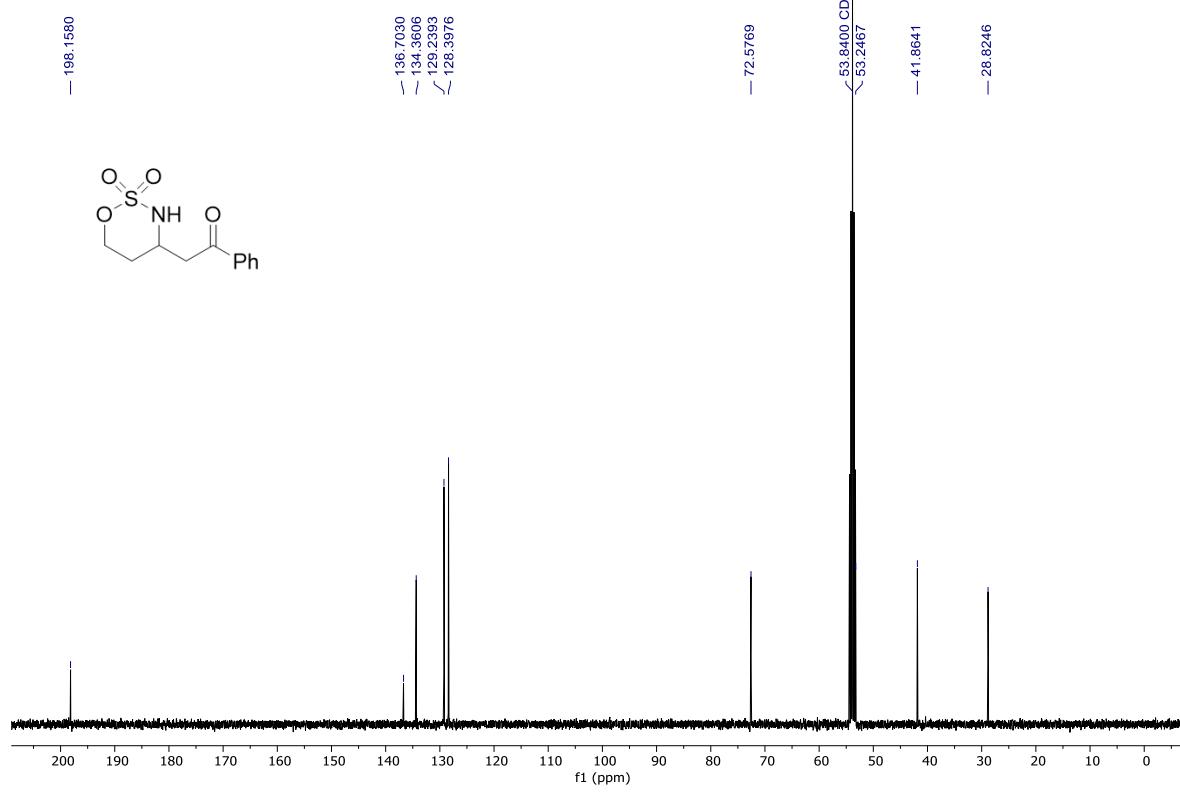


Compound 2 (CD₂Cl₂, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

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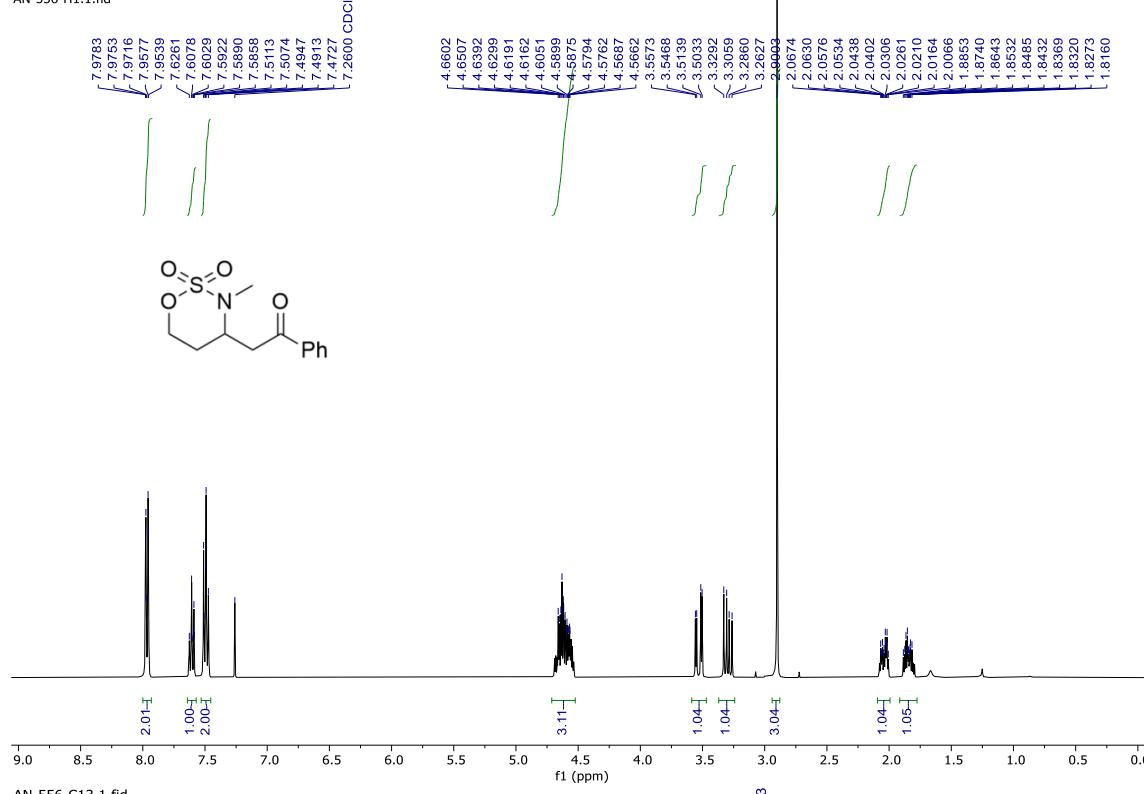


— 198.1580

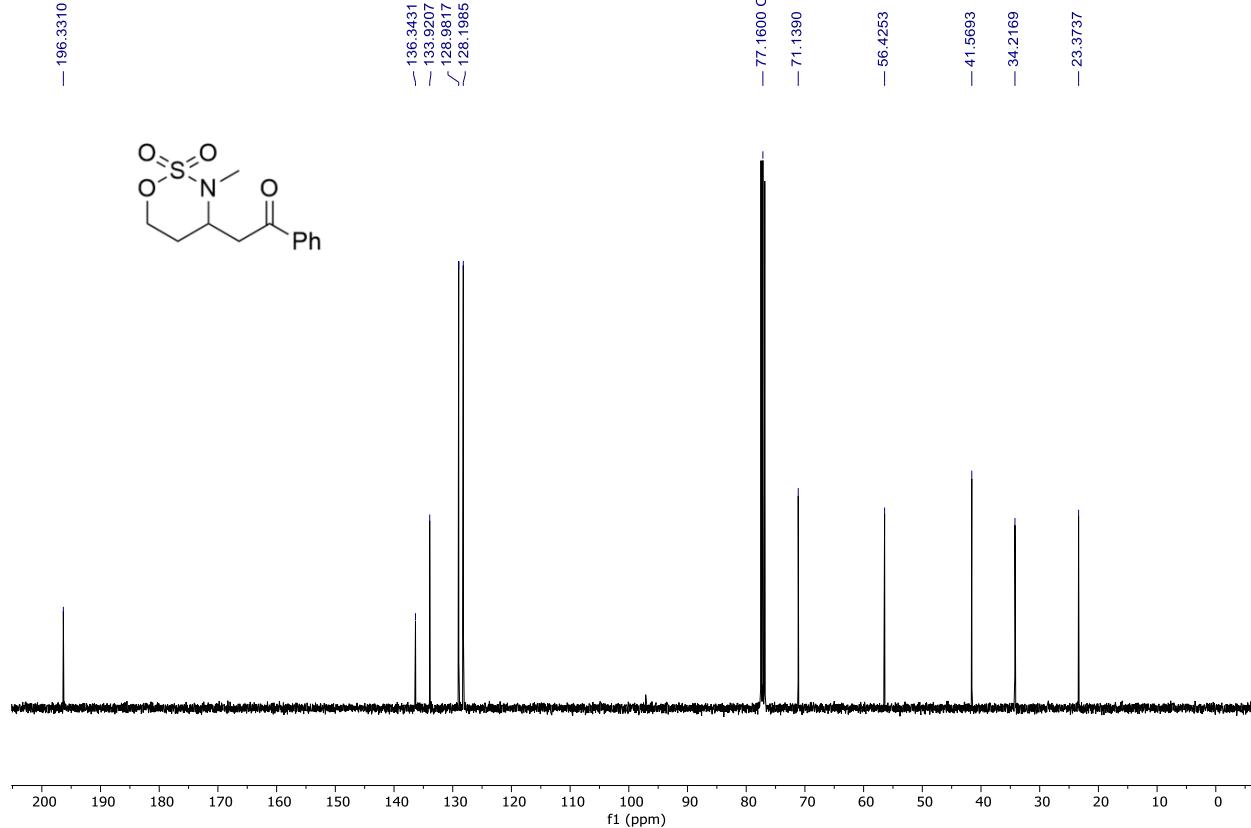


Compound 3 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-556-H1.1.fid

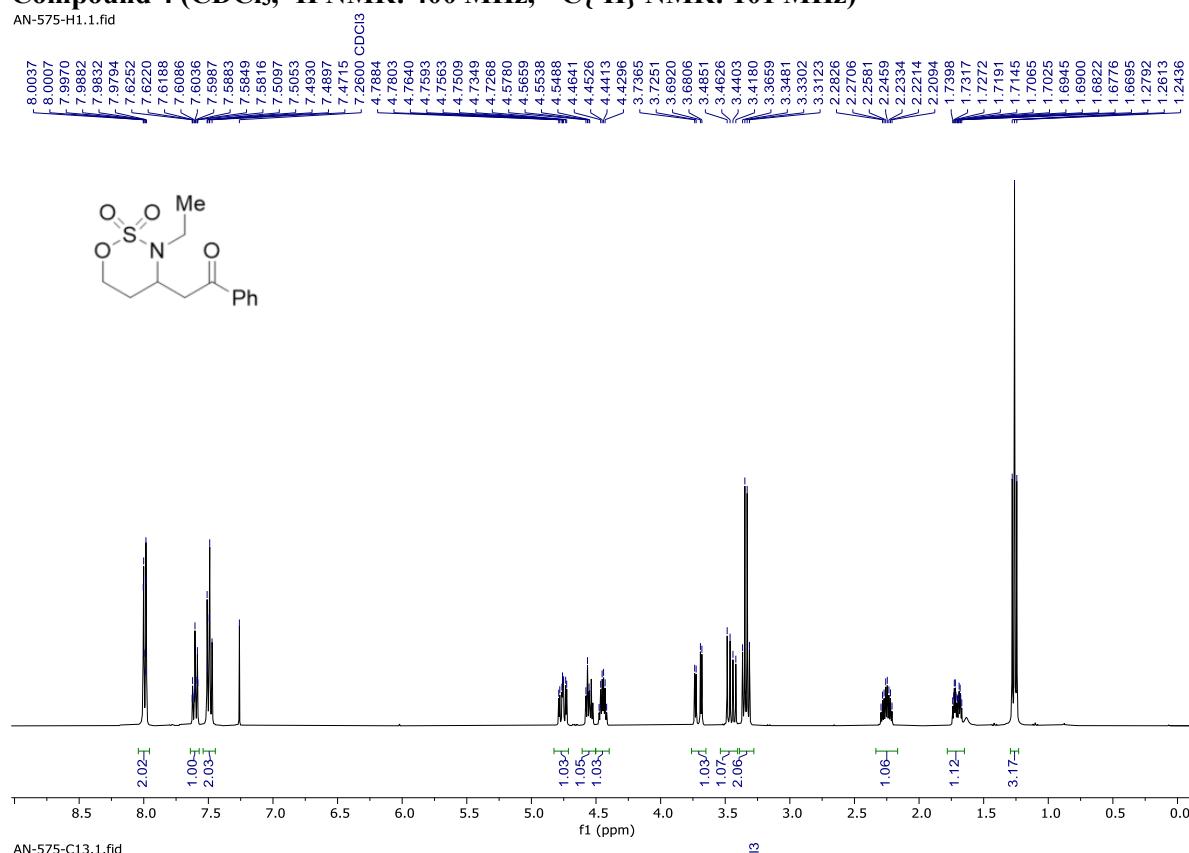


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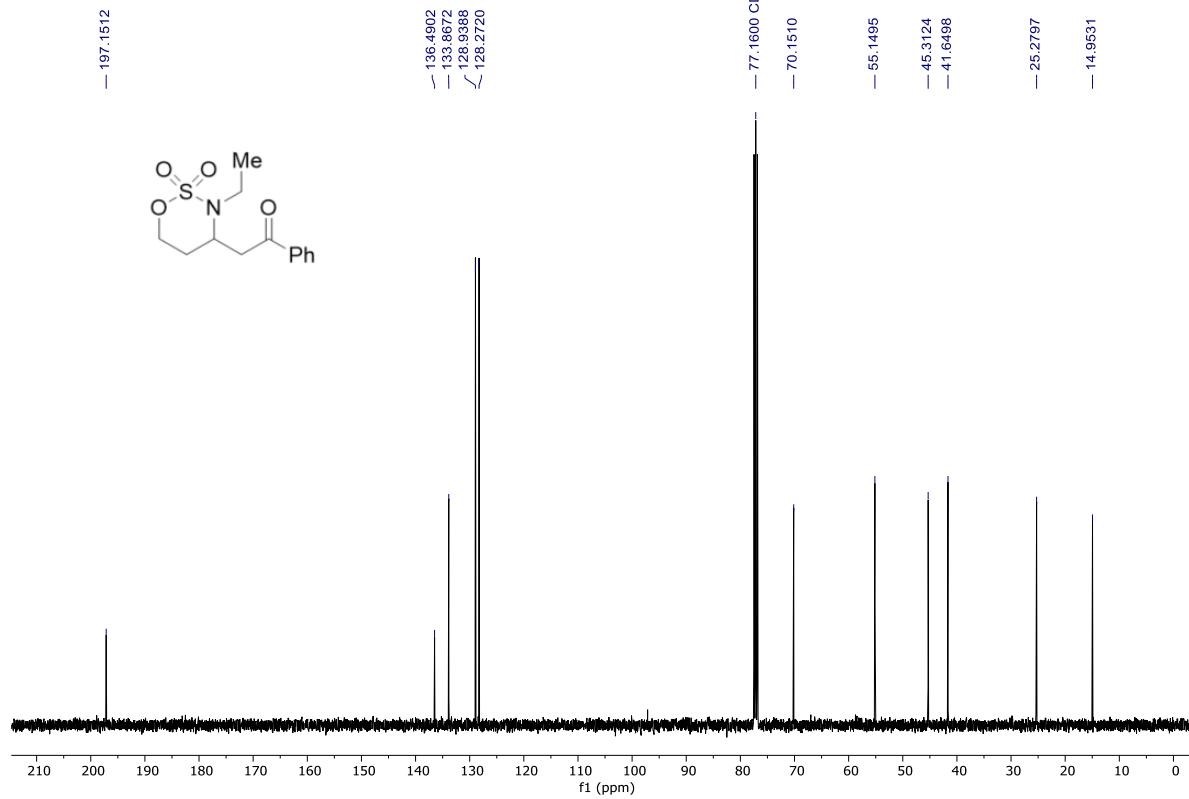


Compound 4 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

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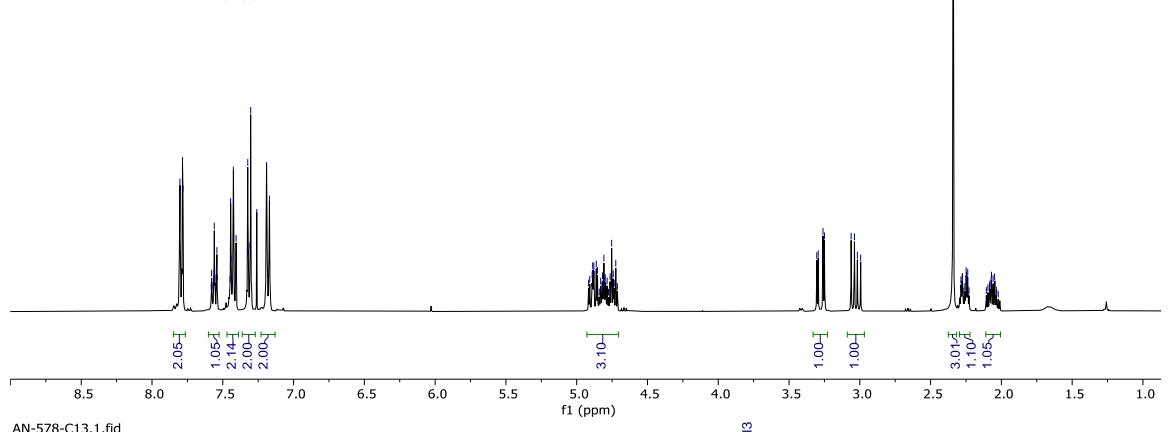
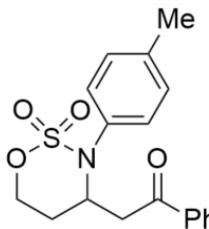


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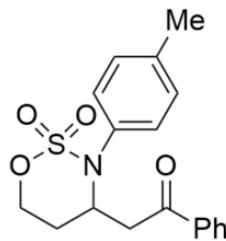
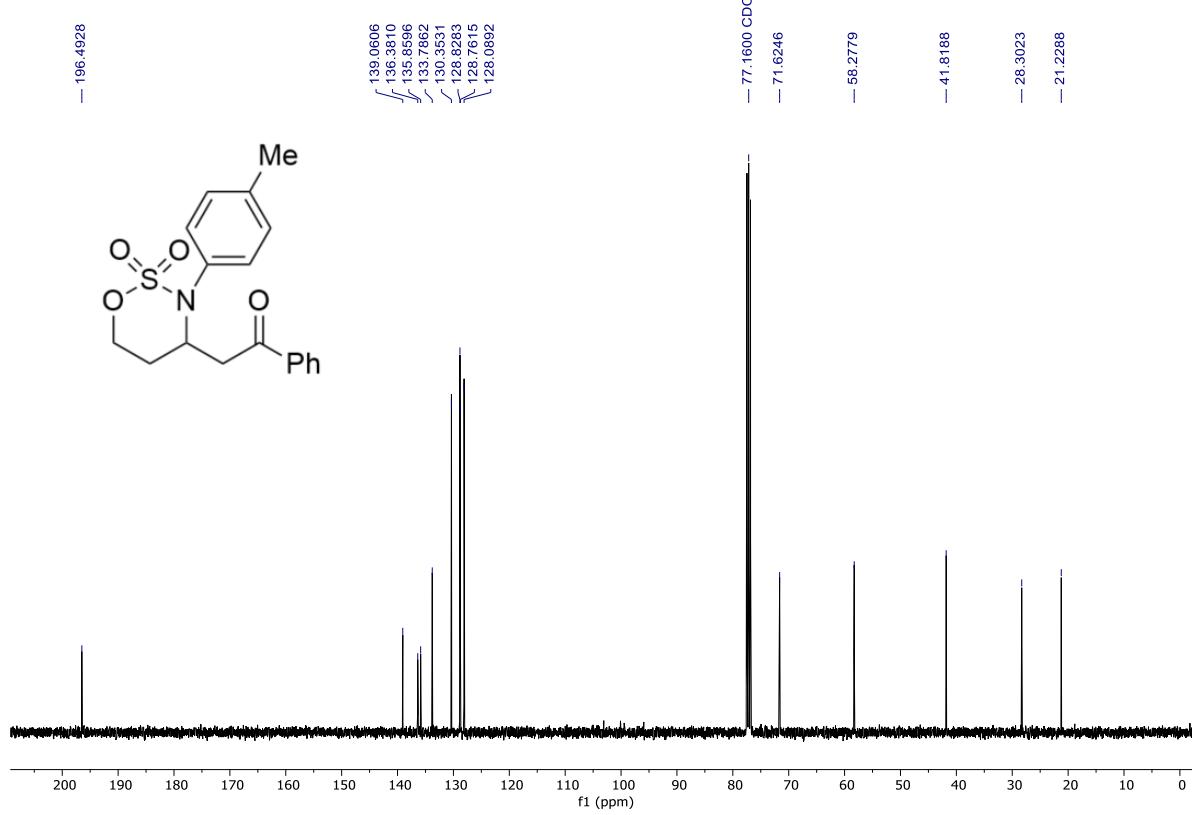


Compound 5 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-578-H1.1.fid

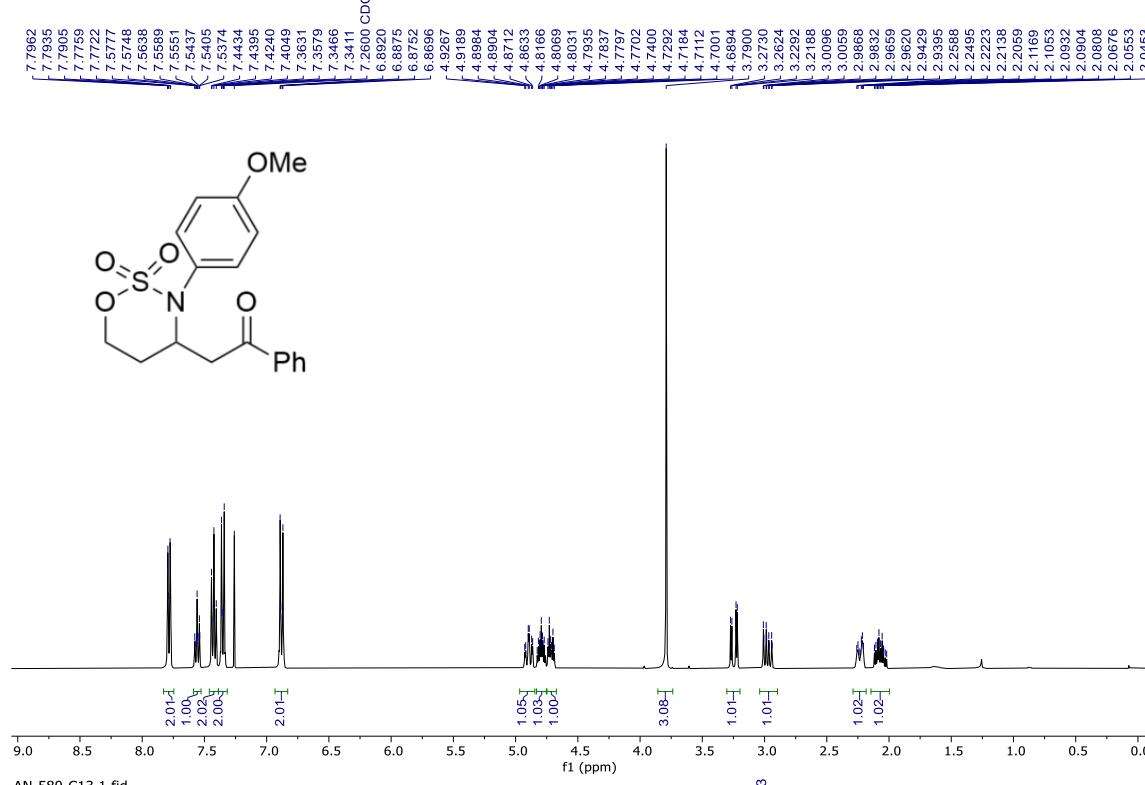


AN-578-C13.1.fid

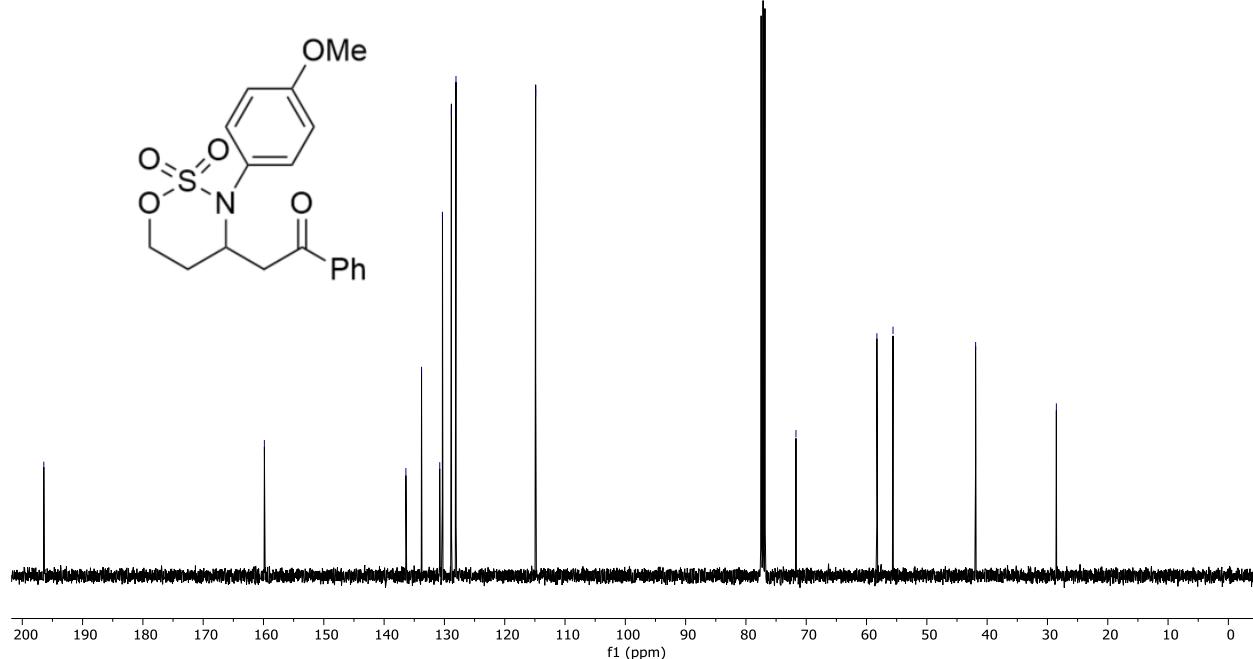


Compound 6 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

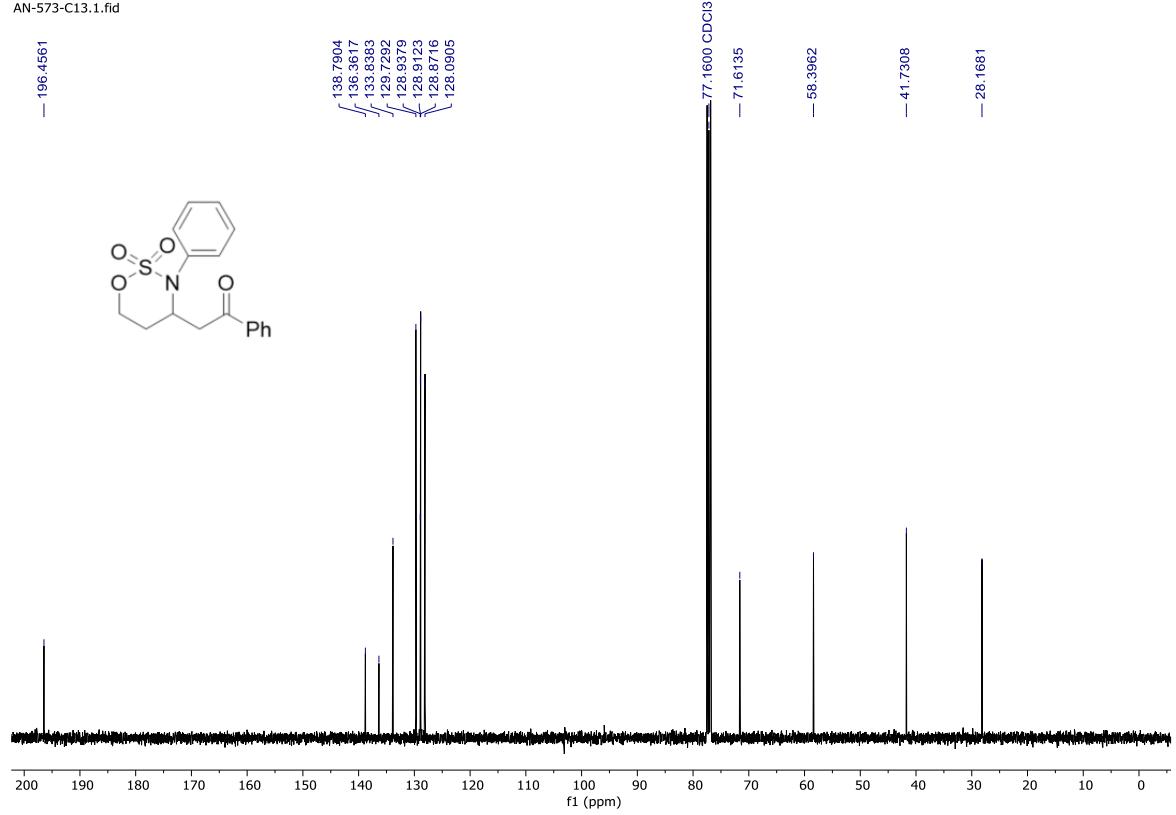
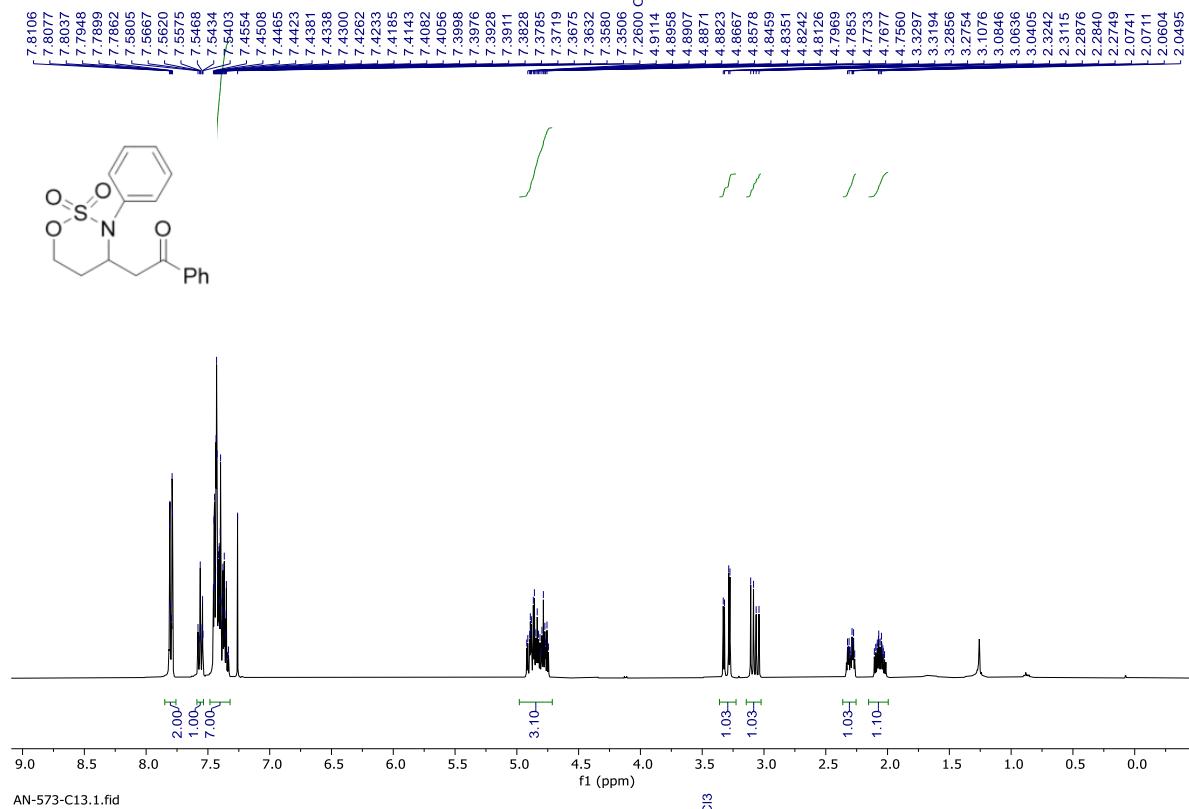
AN-580-H1.1.fid



— 196.4316

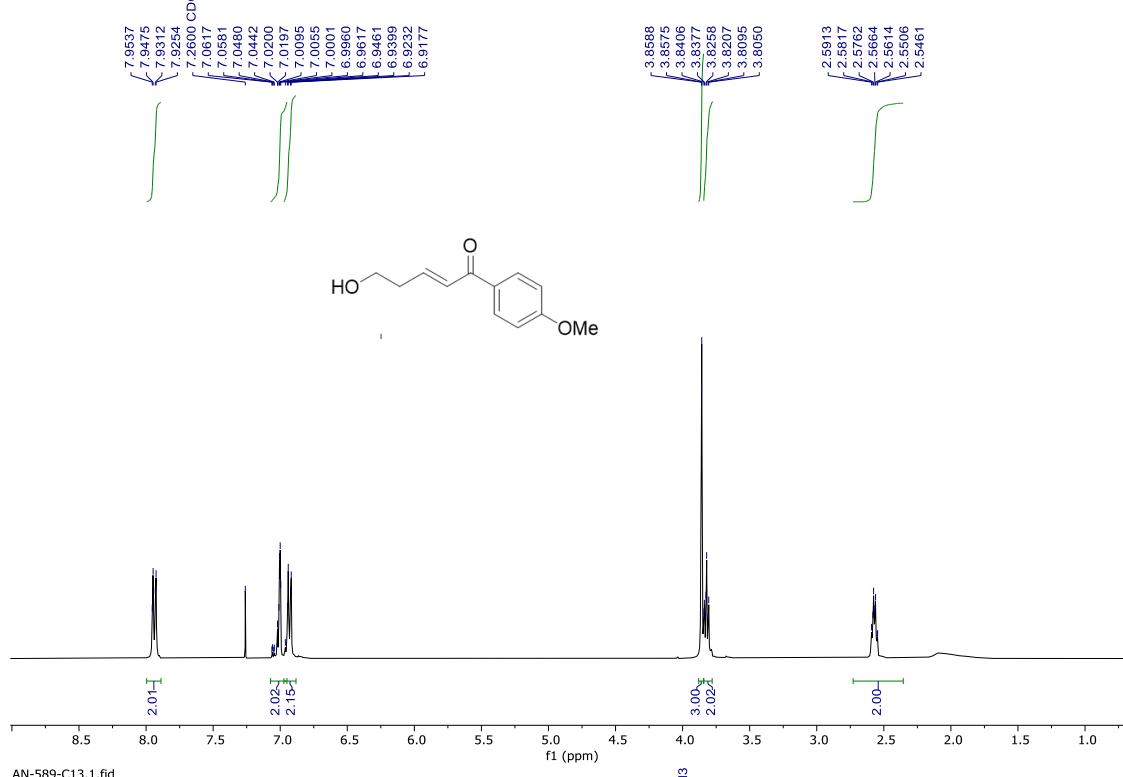


Compound 7 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)
 AN-573-H1.1.fid

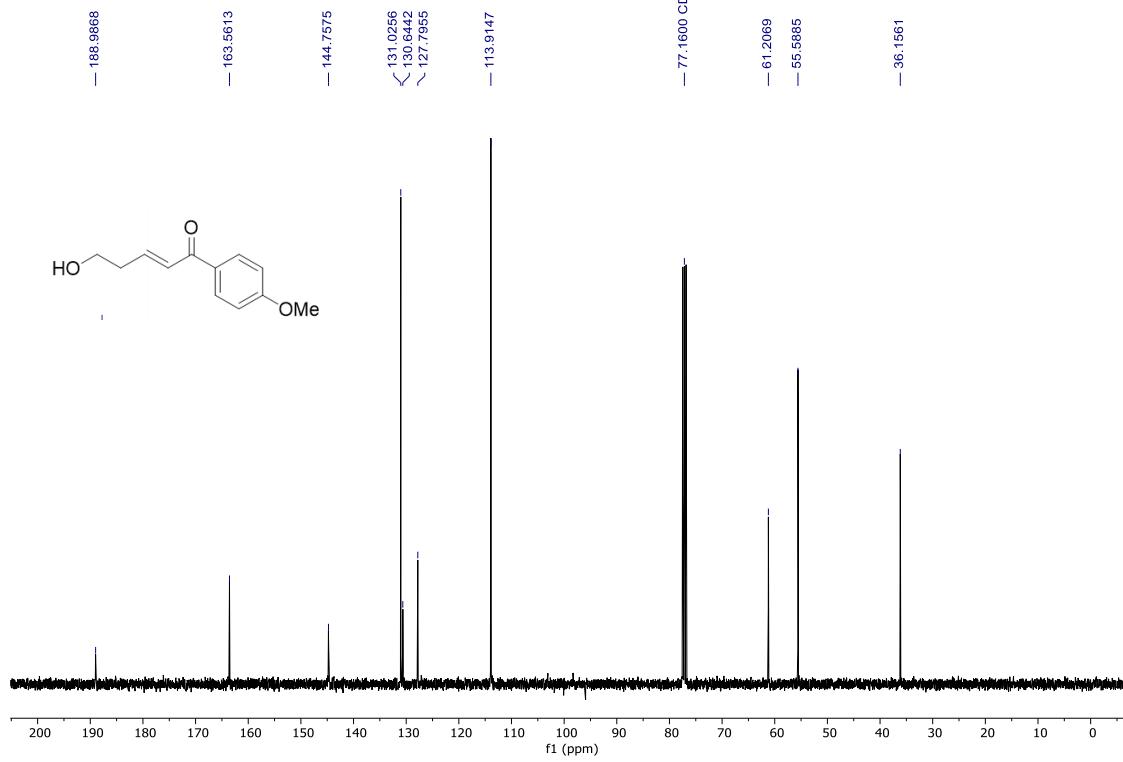


Compound 8 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-589-H1.1.fid

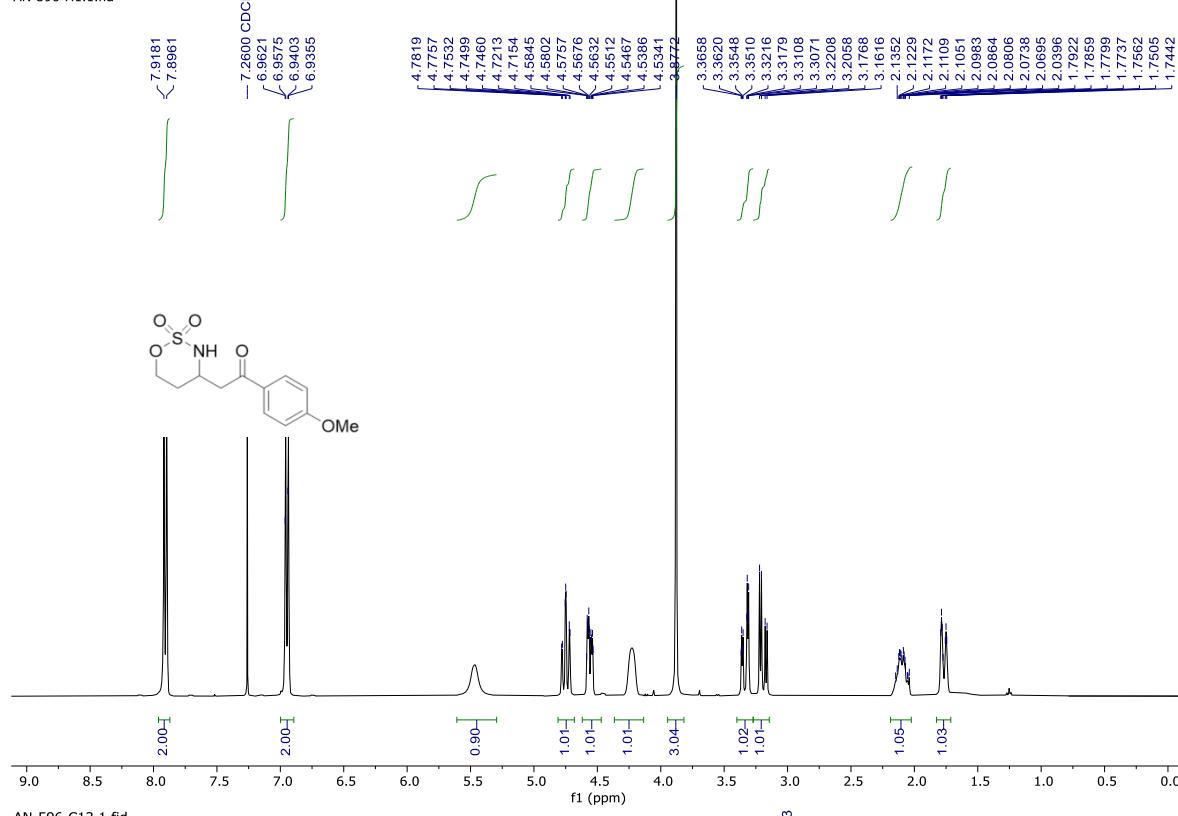


AN-589-C13.1.fid

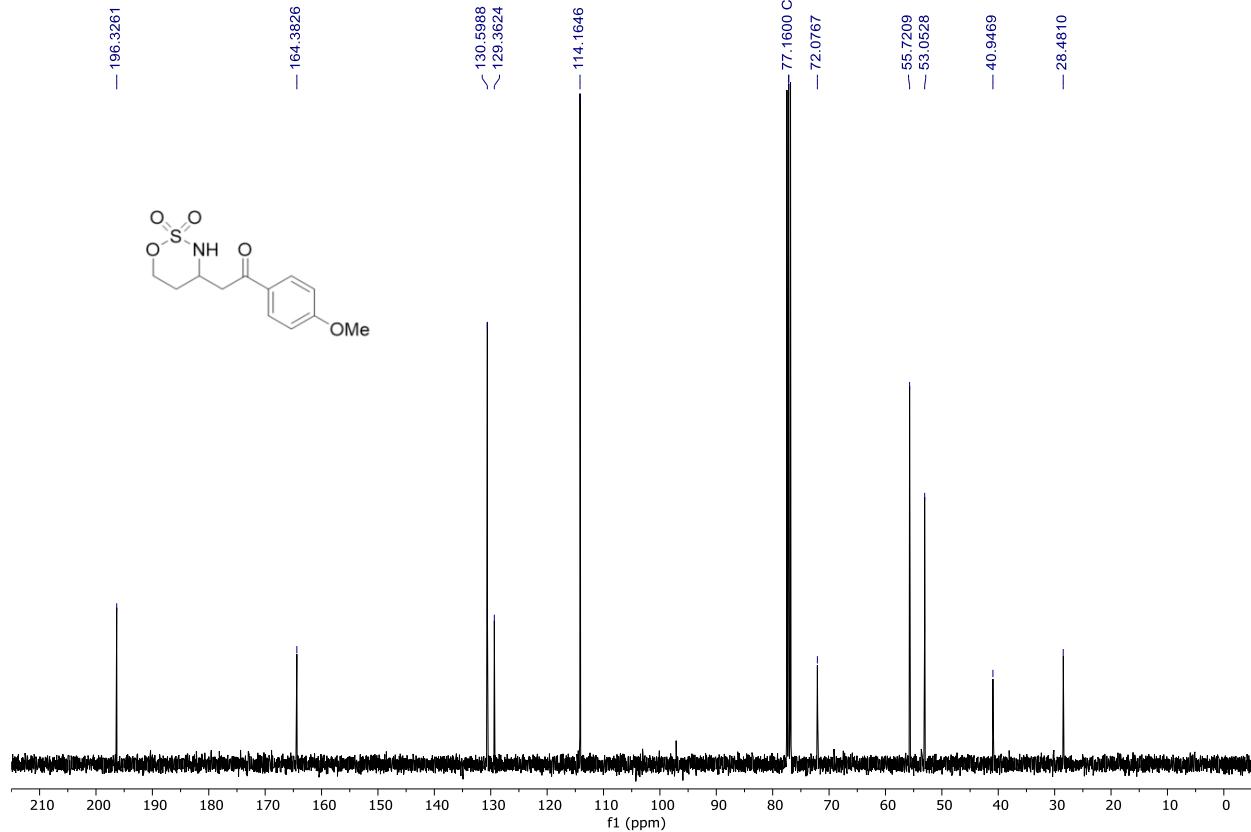


Compound 9 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-596-H1.1.fid

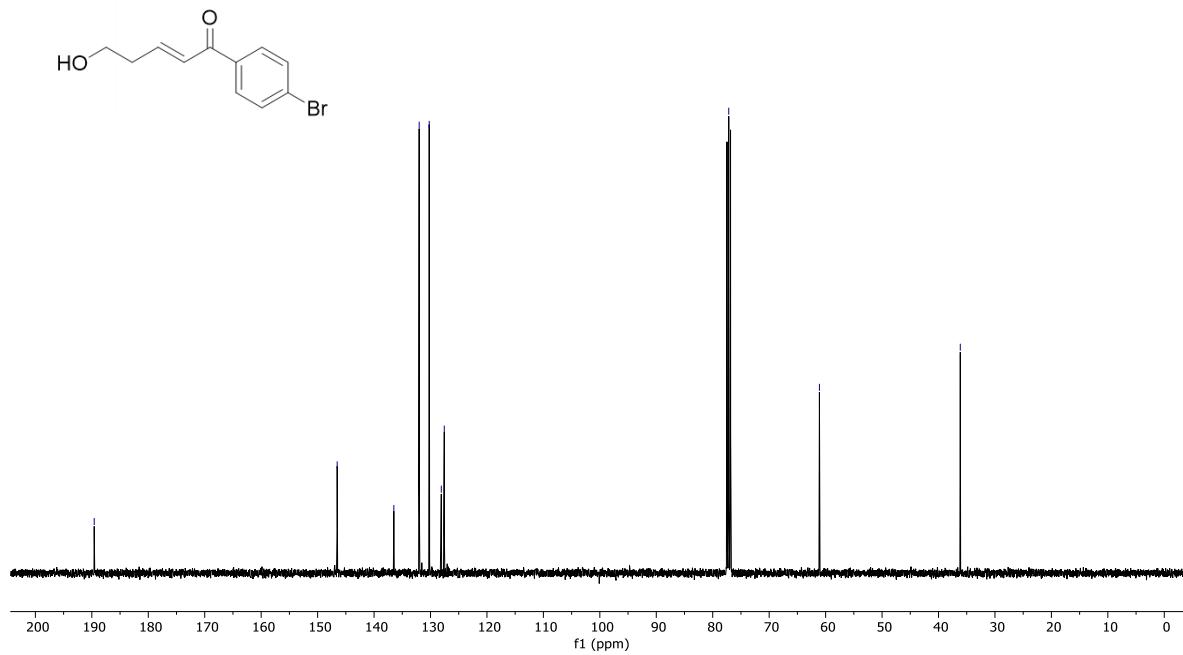
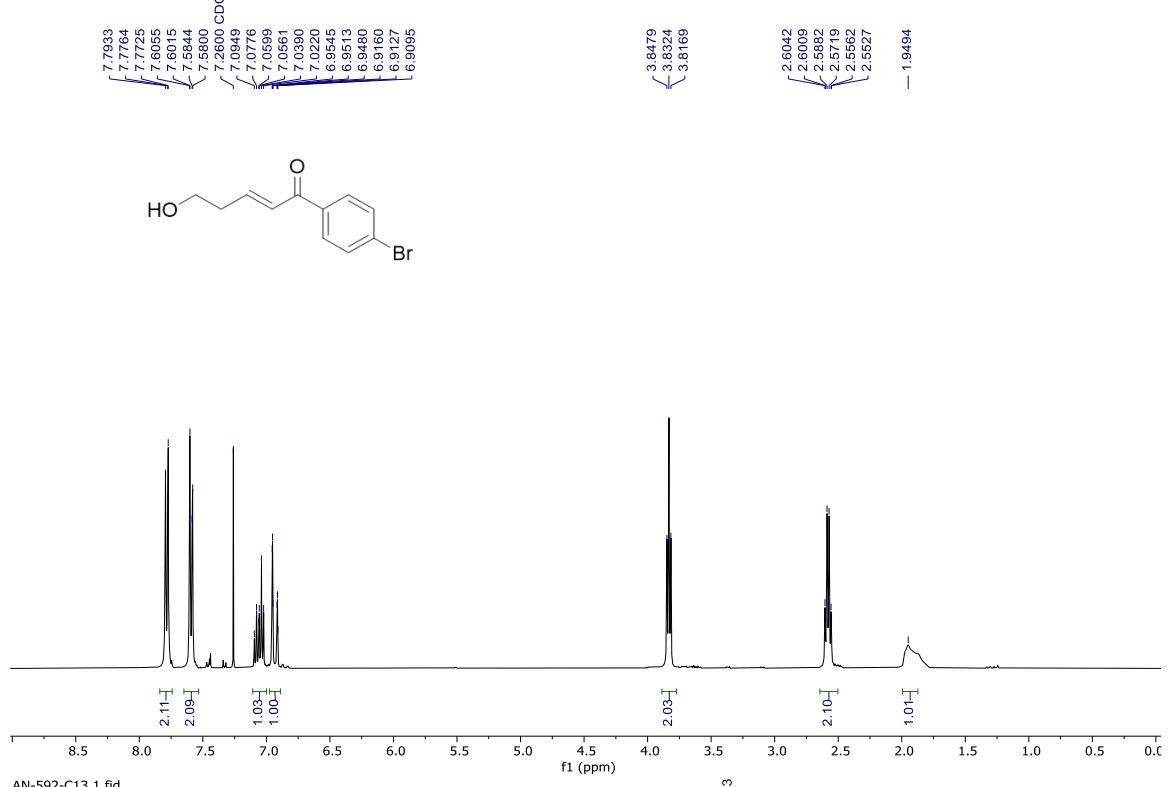


AN-596-C13.1.fid



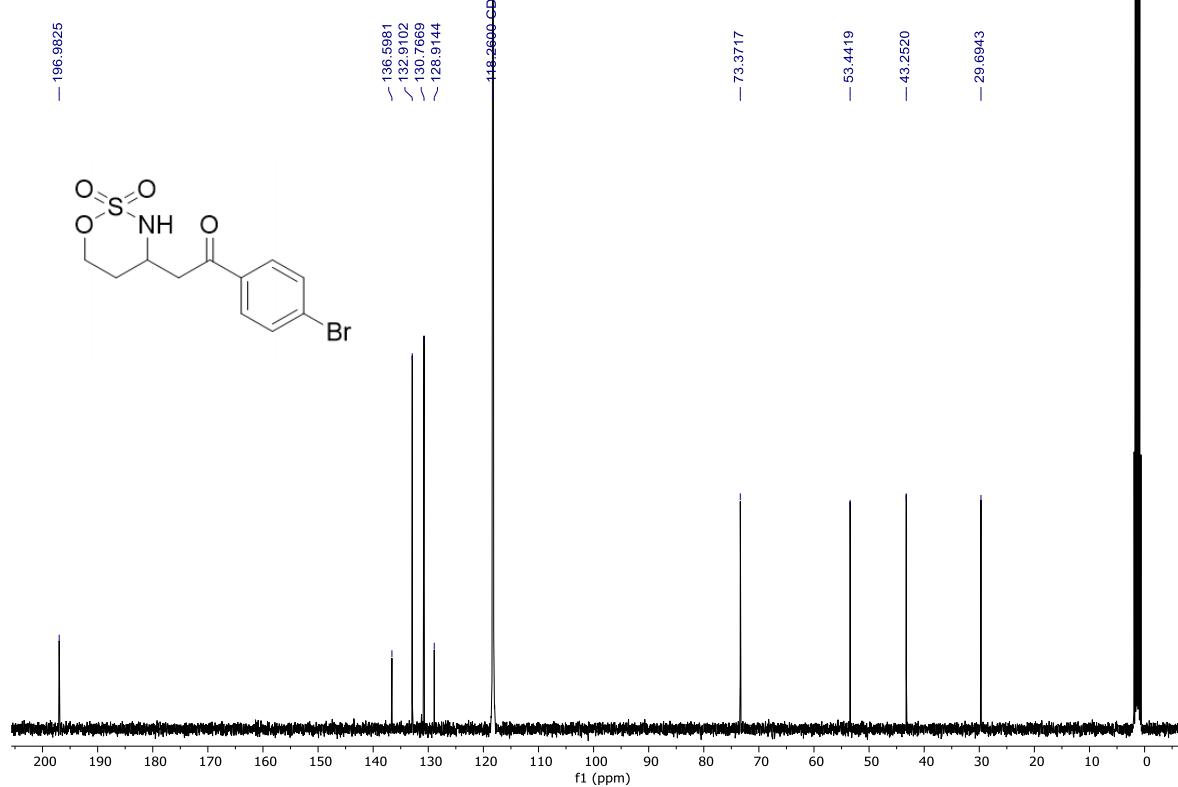
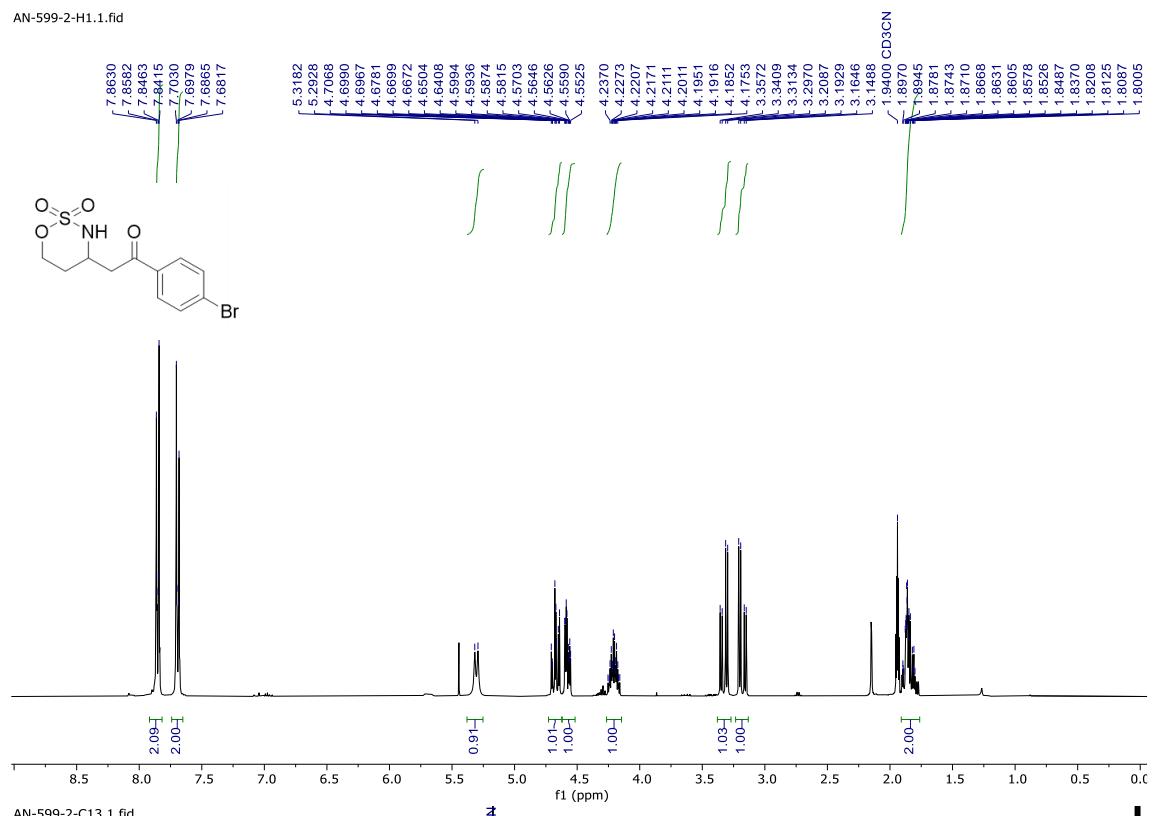
Compound 10 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

AN-592-H1.1.fid



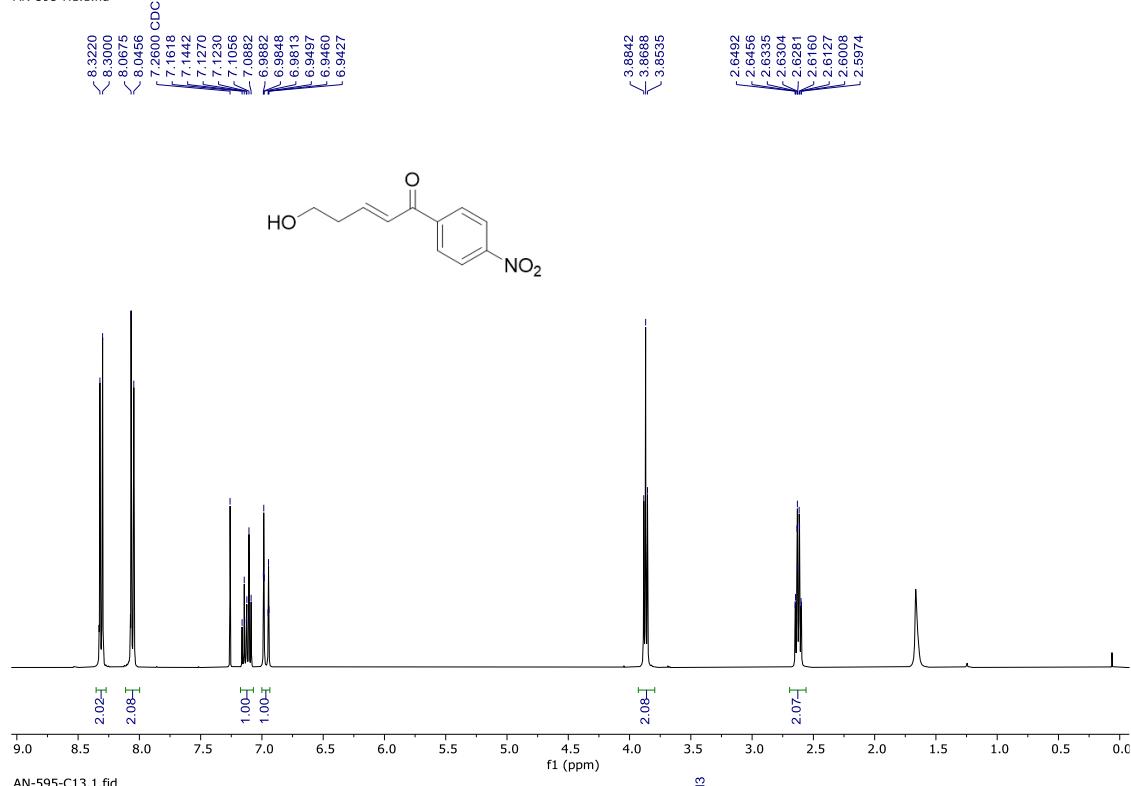
Compound 11 (CD_3CN , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-599-2-H1.1.fid

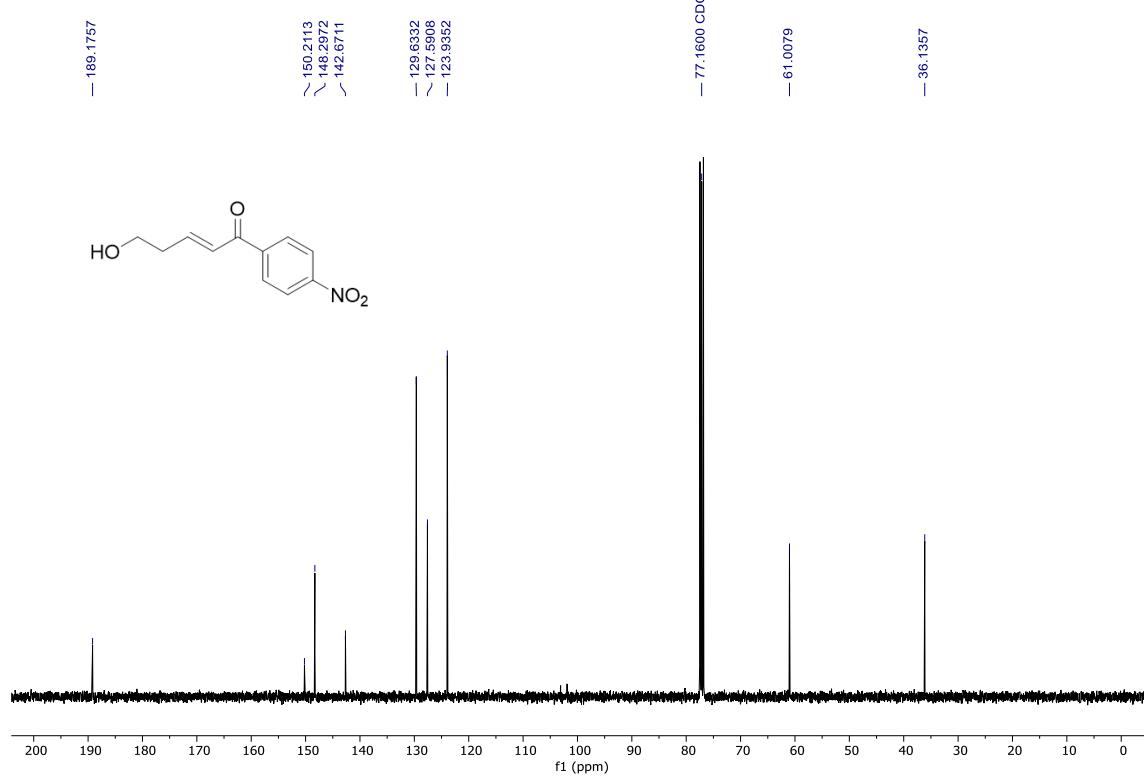


Compound 12 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-595-H1.1.fid

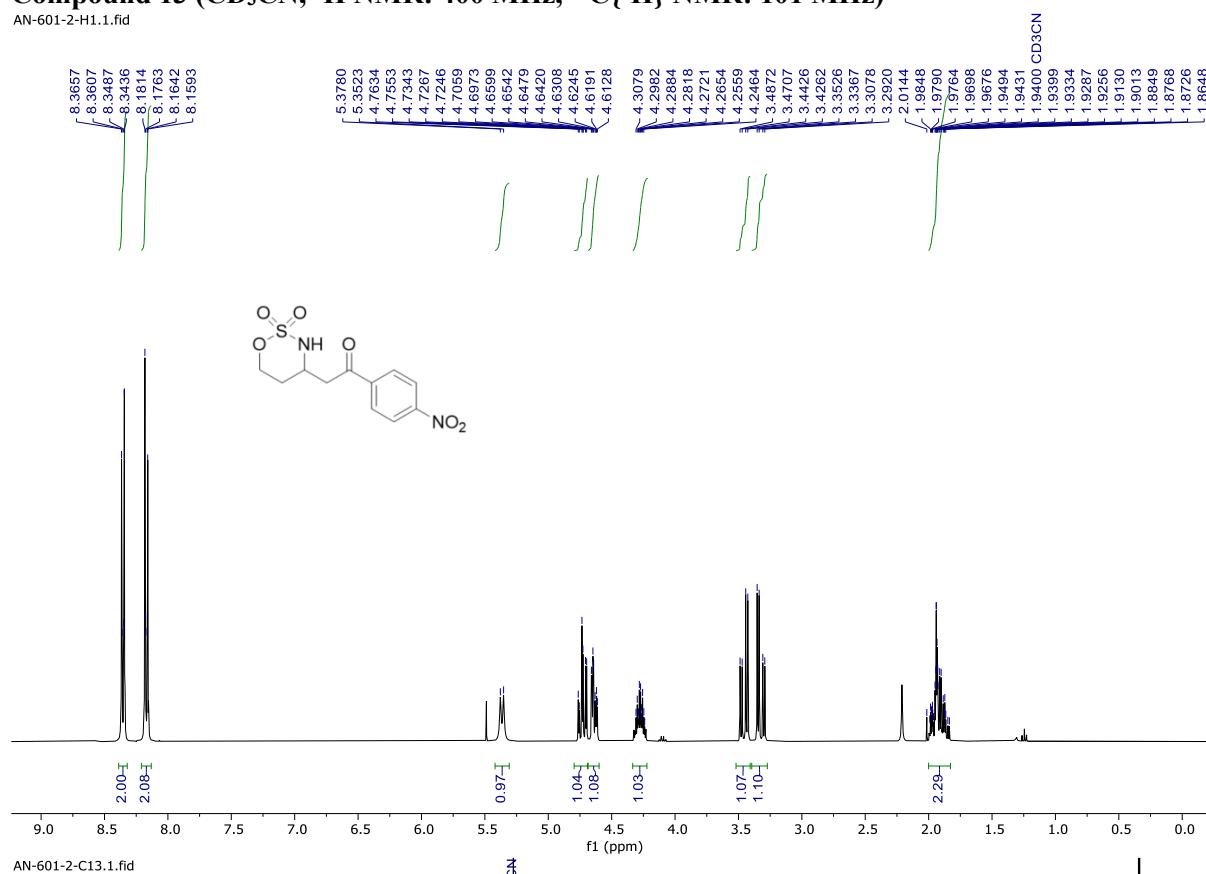


AN-595-C13.1.fid

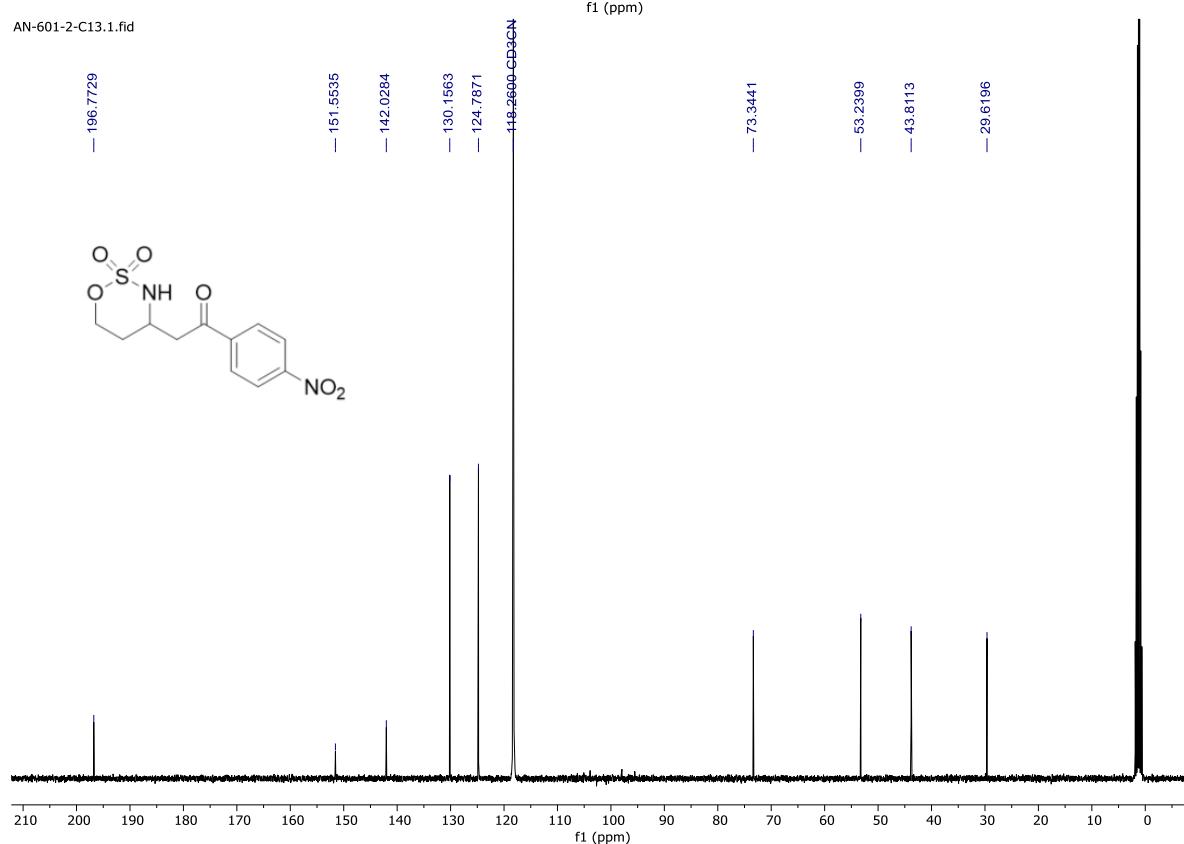


Compound 13 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-601-2-H1.1.fid

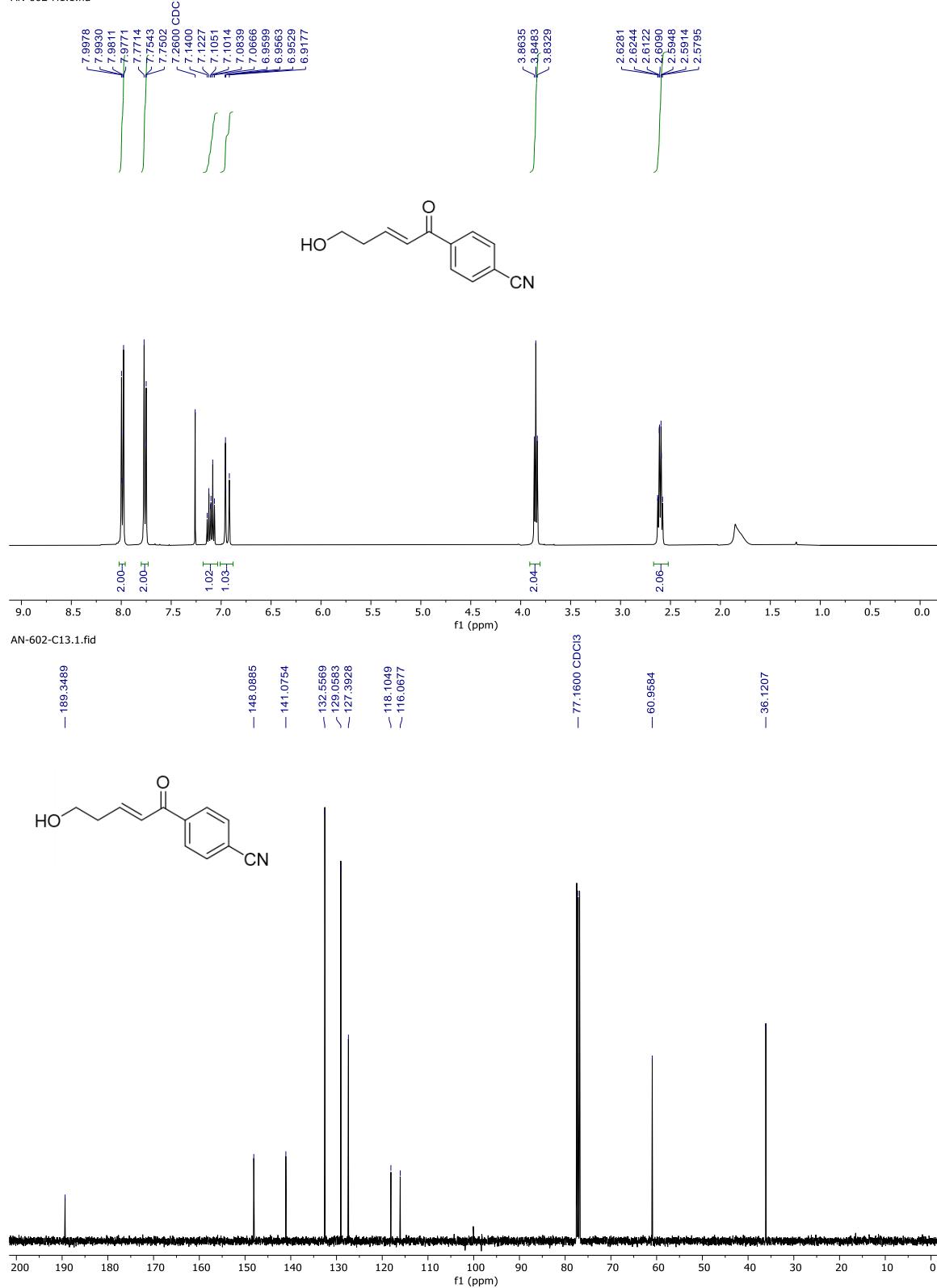


AN-601-2-C13.1.fid



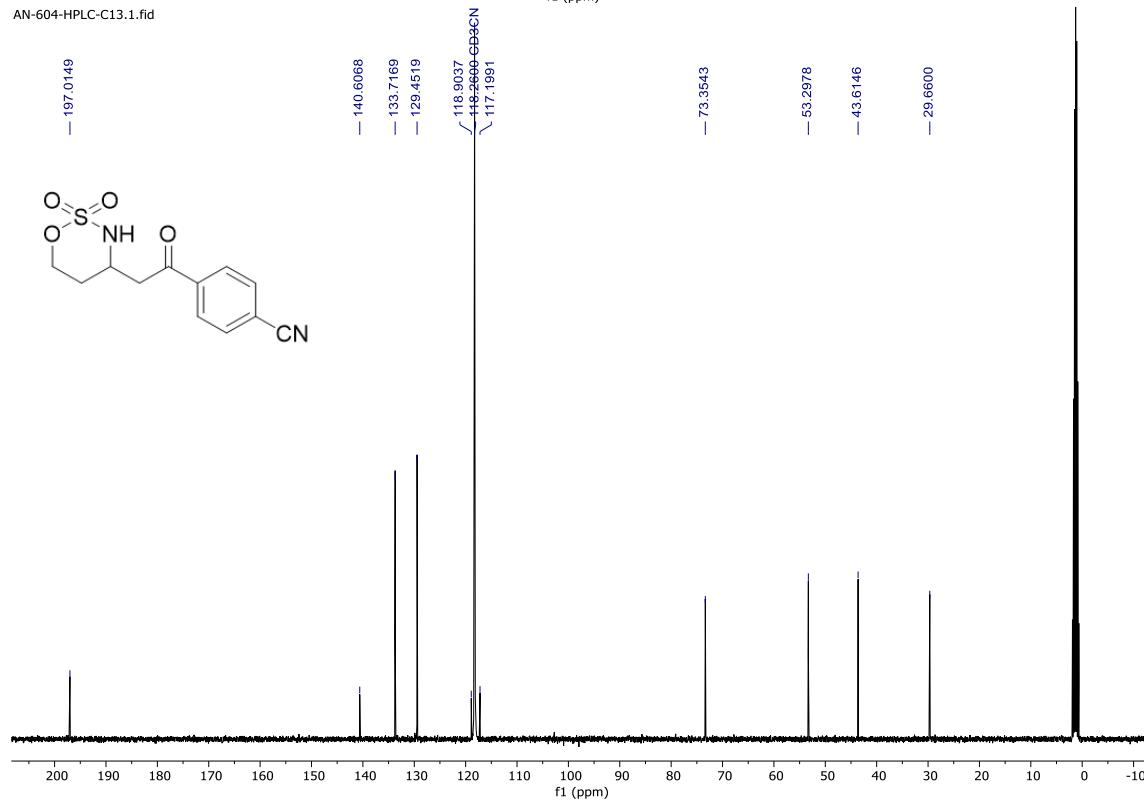
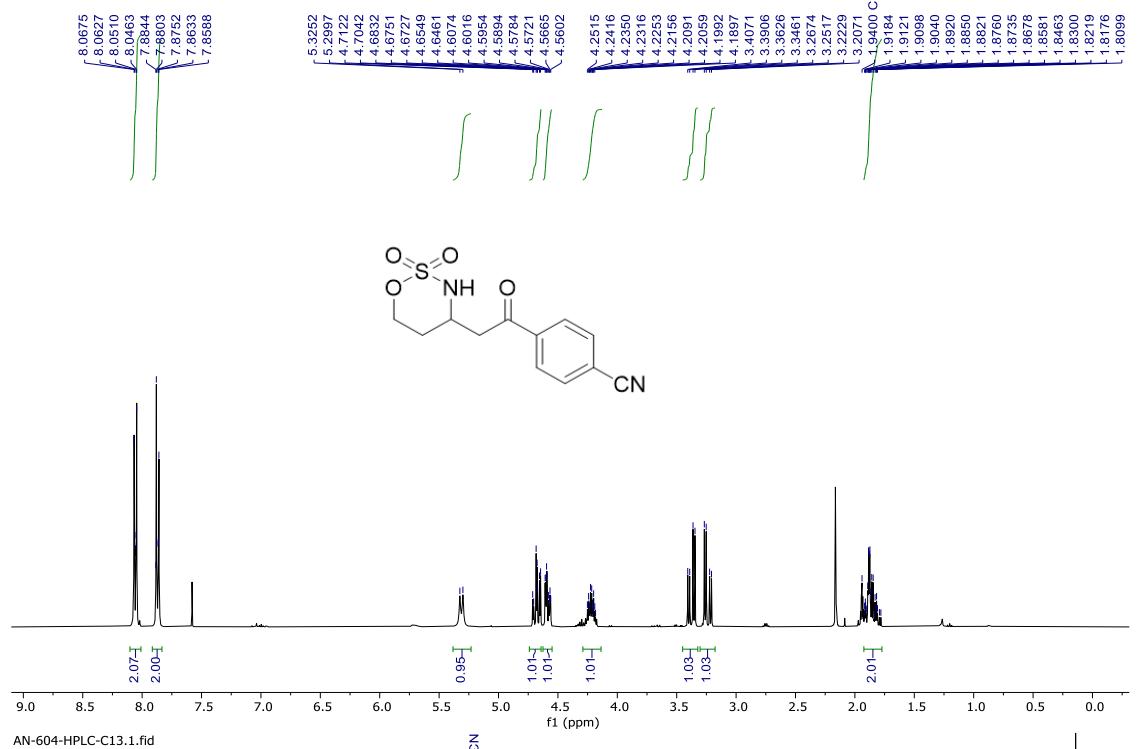
Compound 14 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-602-H1.1.fid



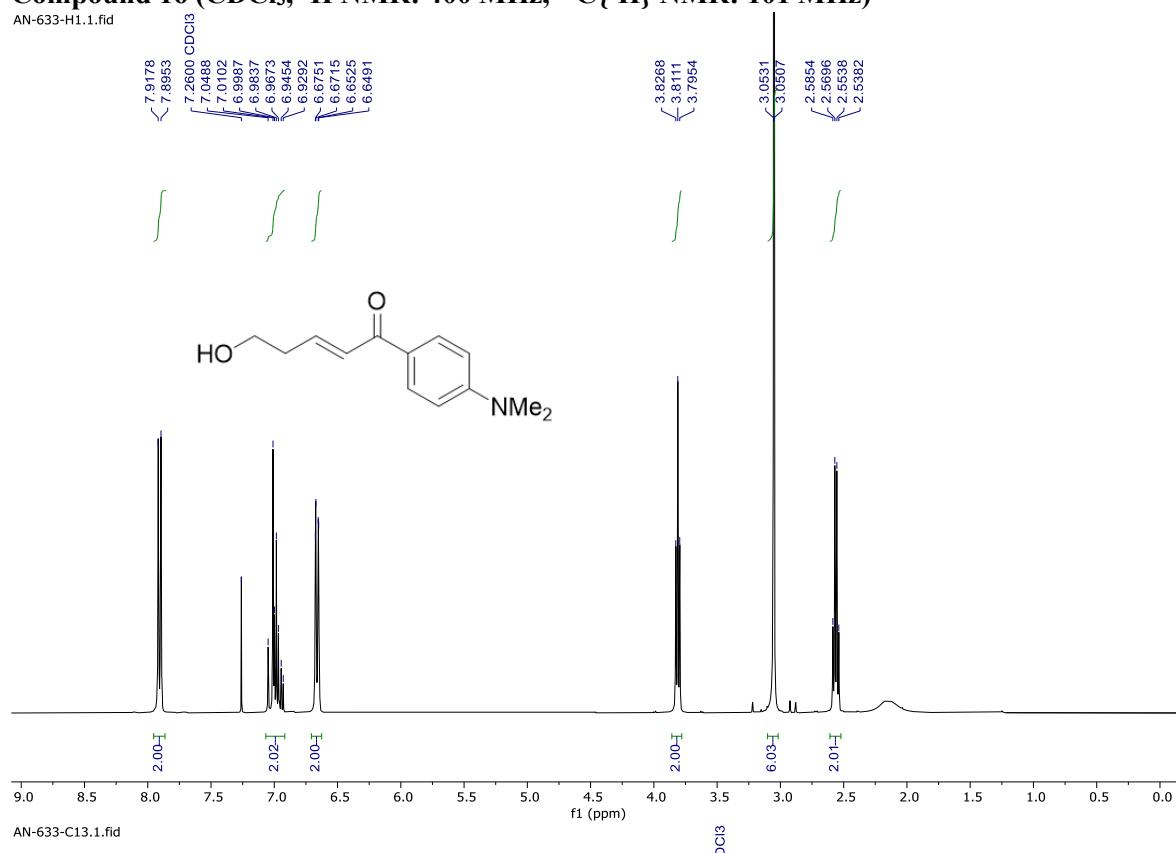
Compound 15 (CD_3CN , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-604-HPLC-H1.1.fid

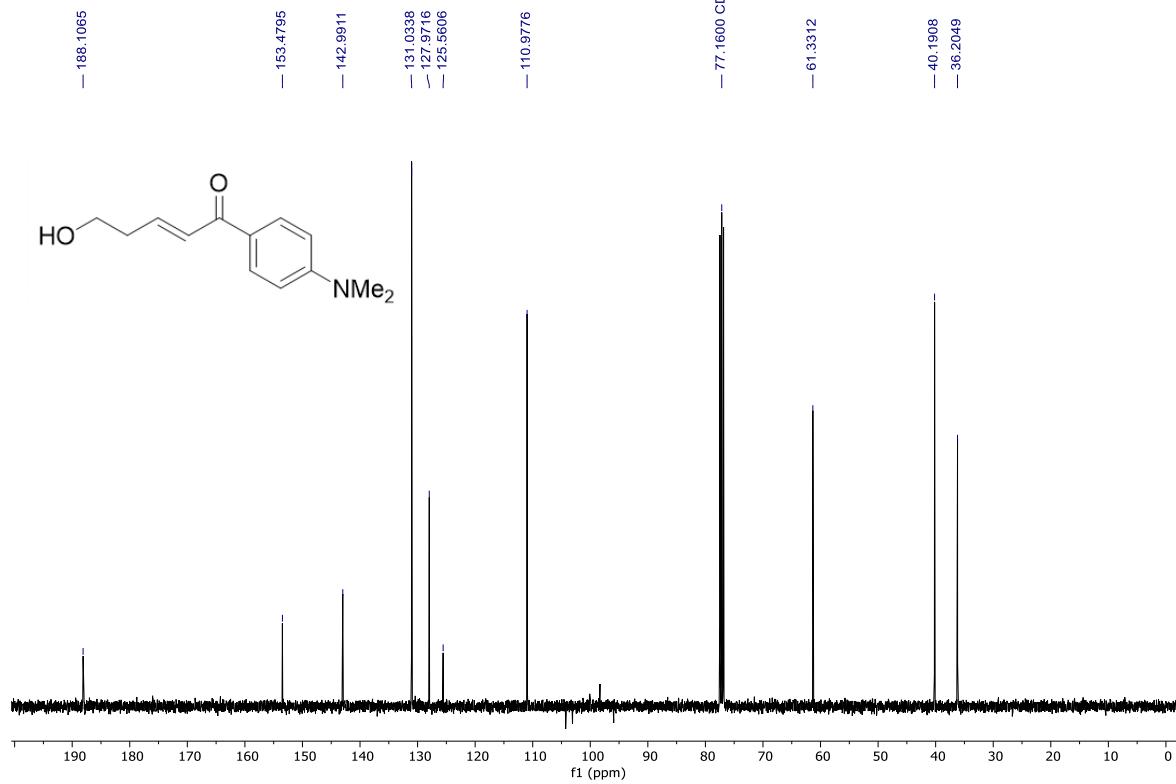


Compound 16 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

AN-633-H1.1.fid

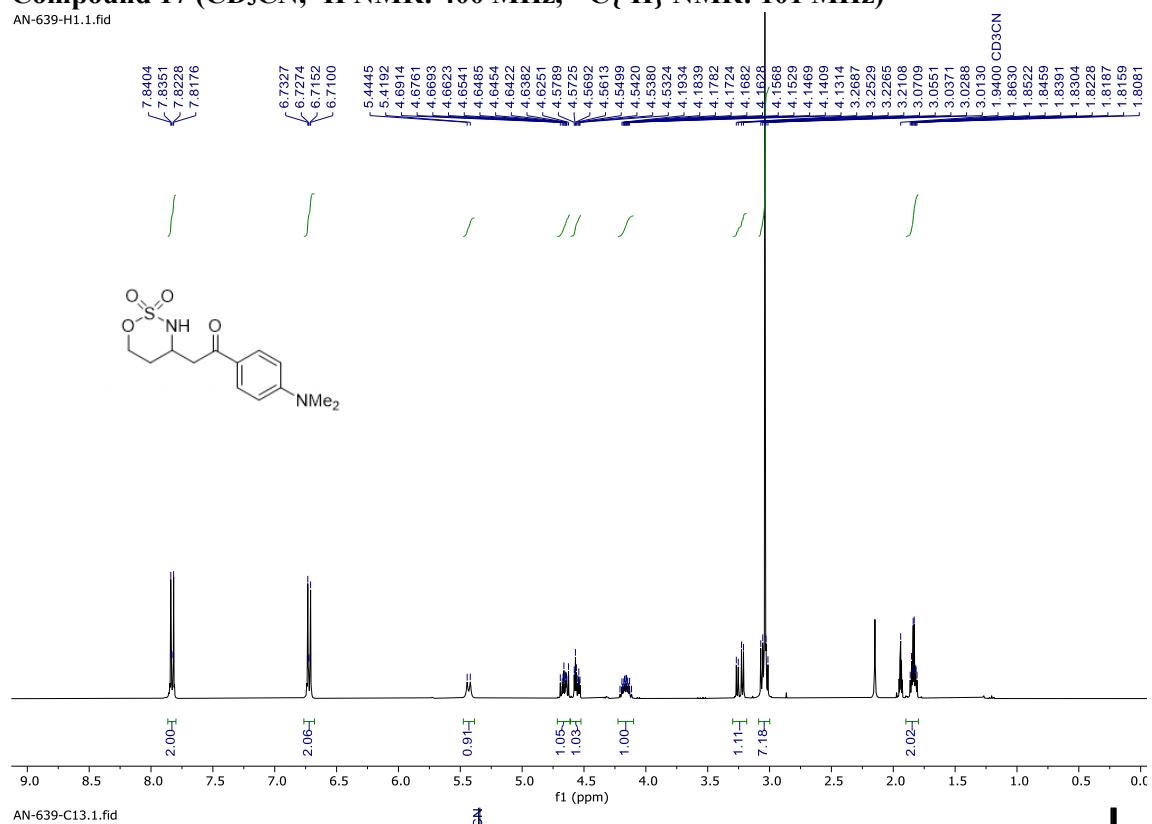


AN-633-C13.1.fid

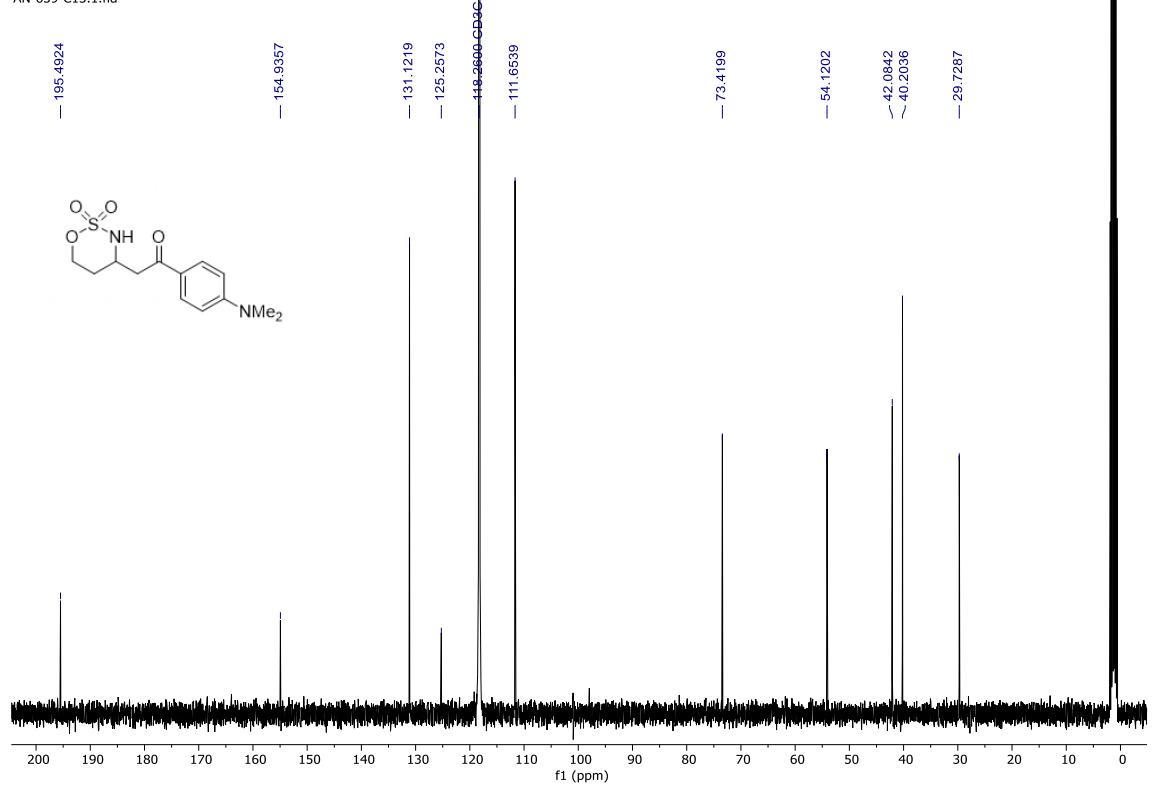


Compound 17 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-639-H1.1.fid

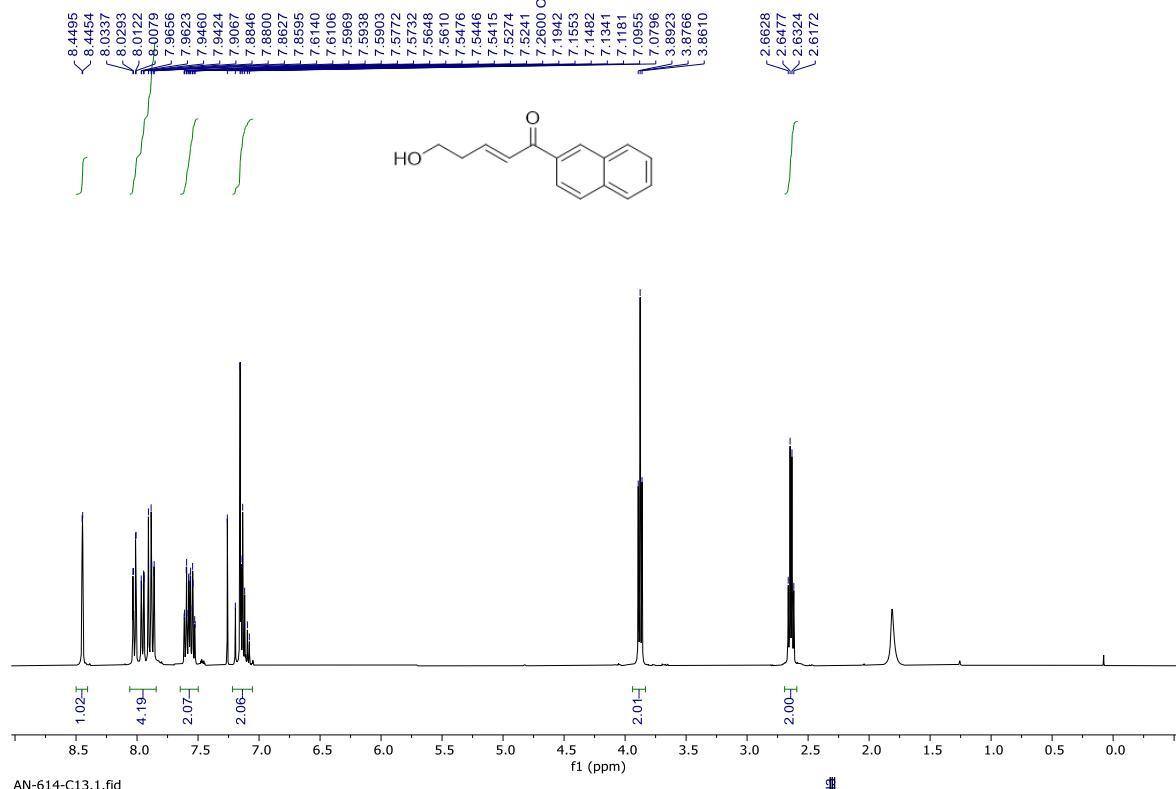


AN-639-C13.1.fid

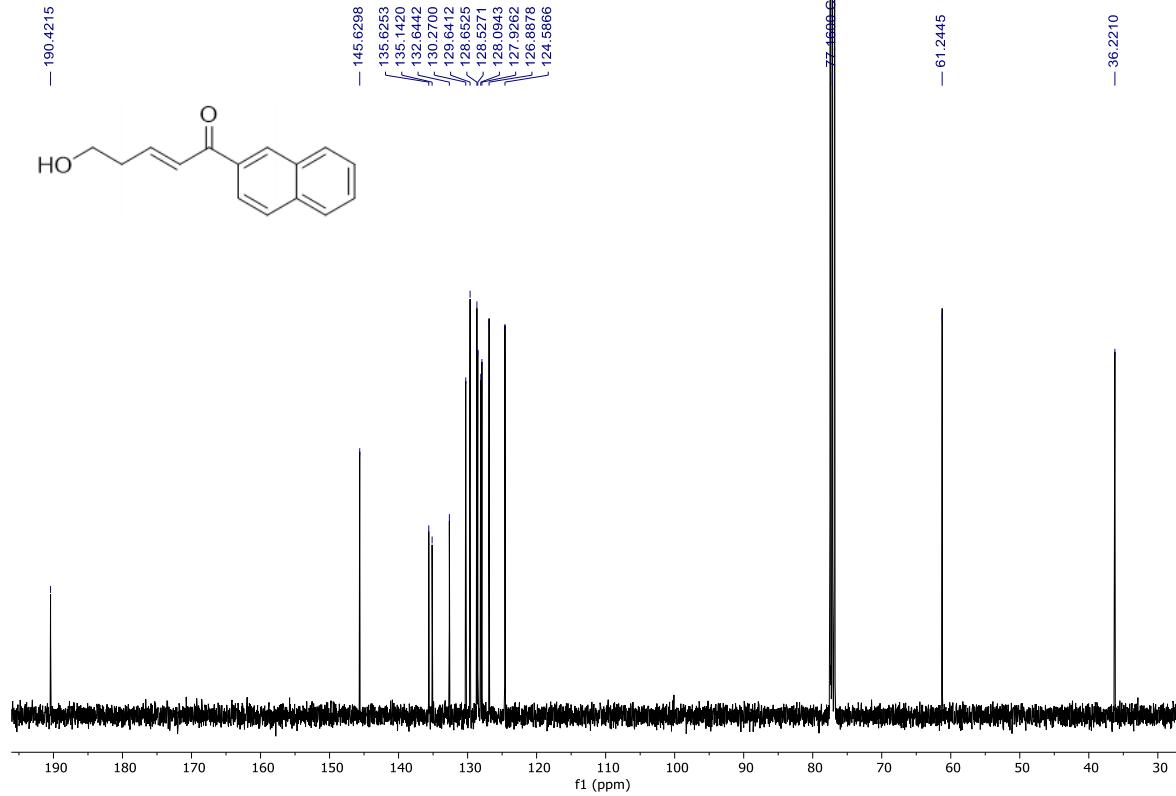


Compound 18 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-614-H1.1.fid

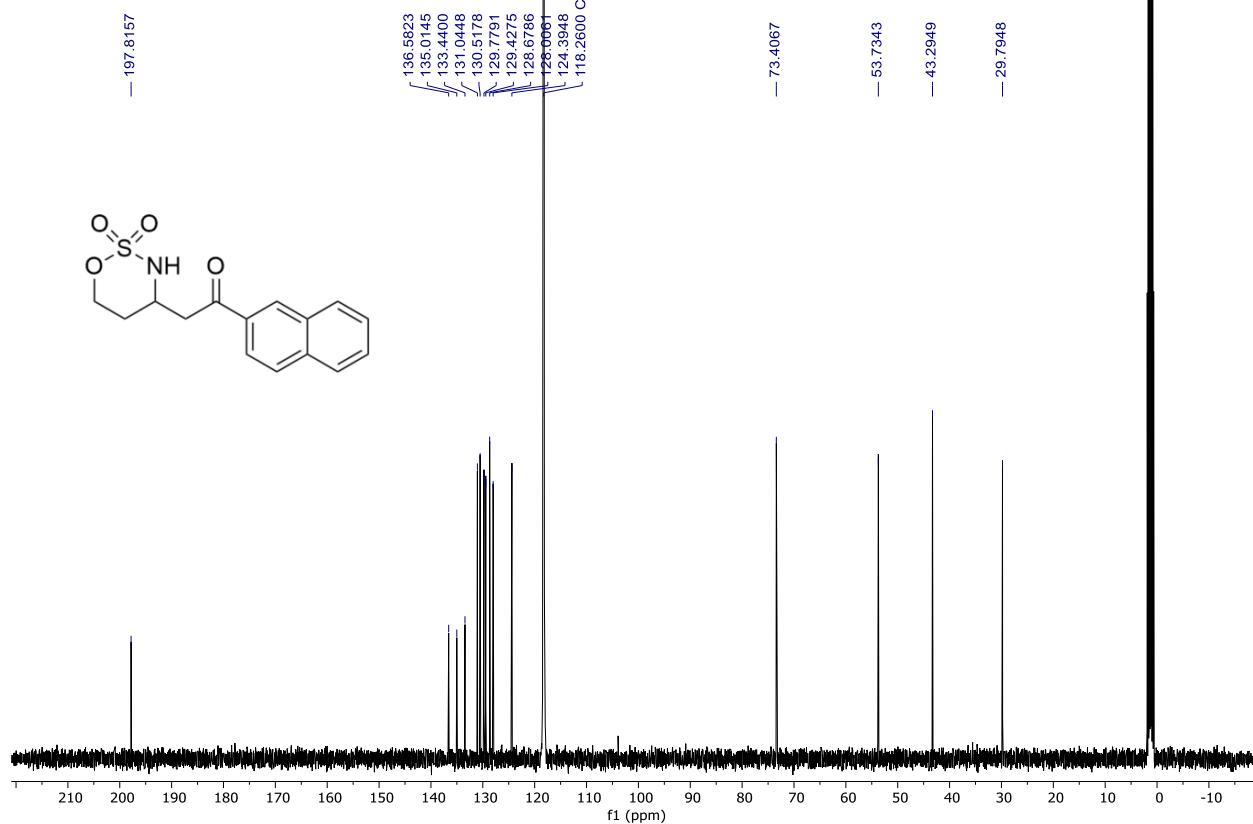
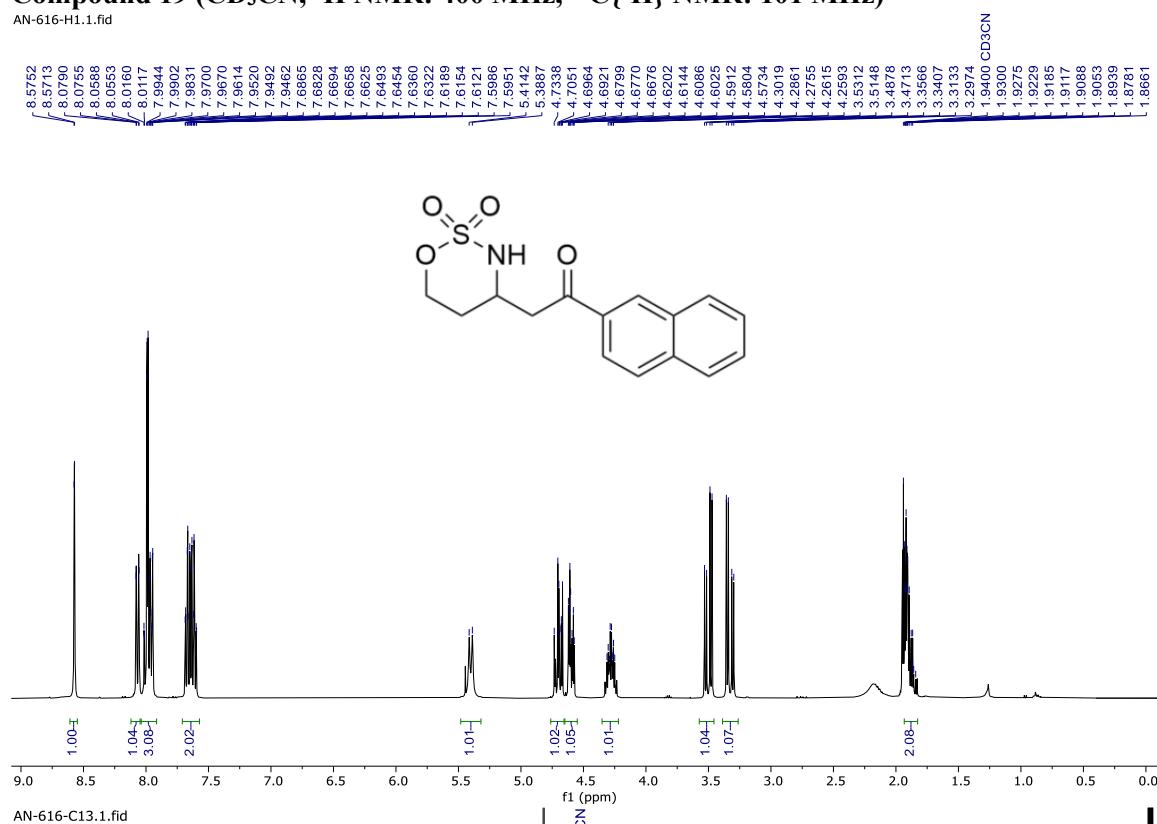


AN-614-C13.1.fid



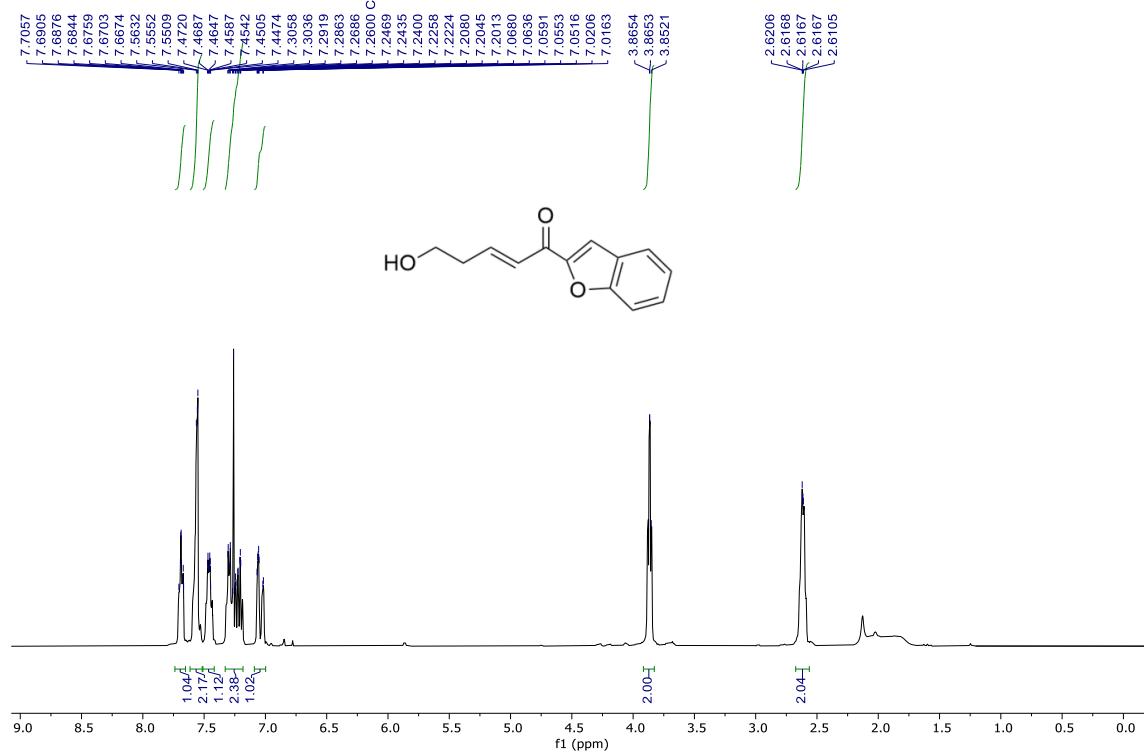
Compound 19 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-616-H1.1.fid

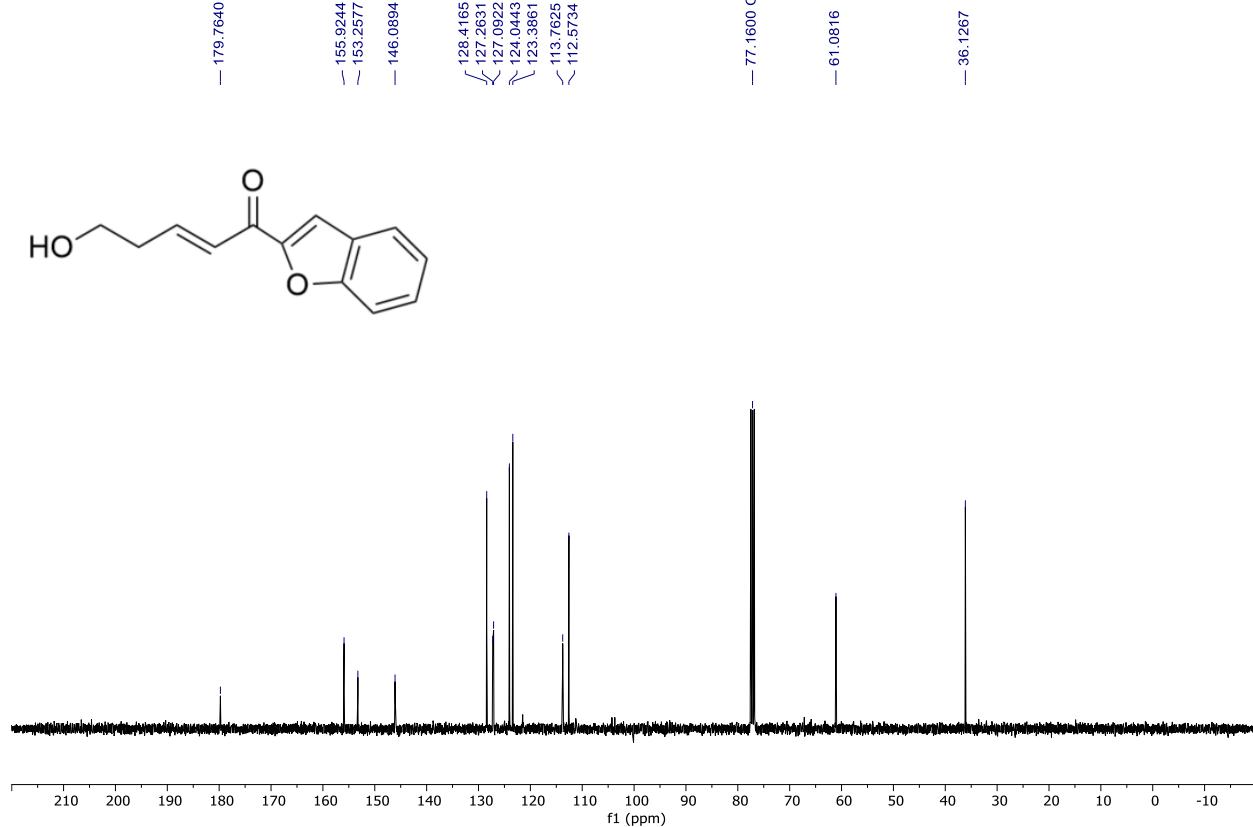


Compound 20 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-615-H1.1.fid

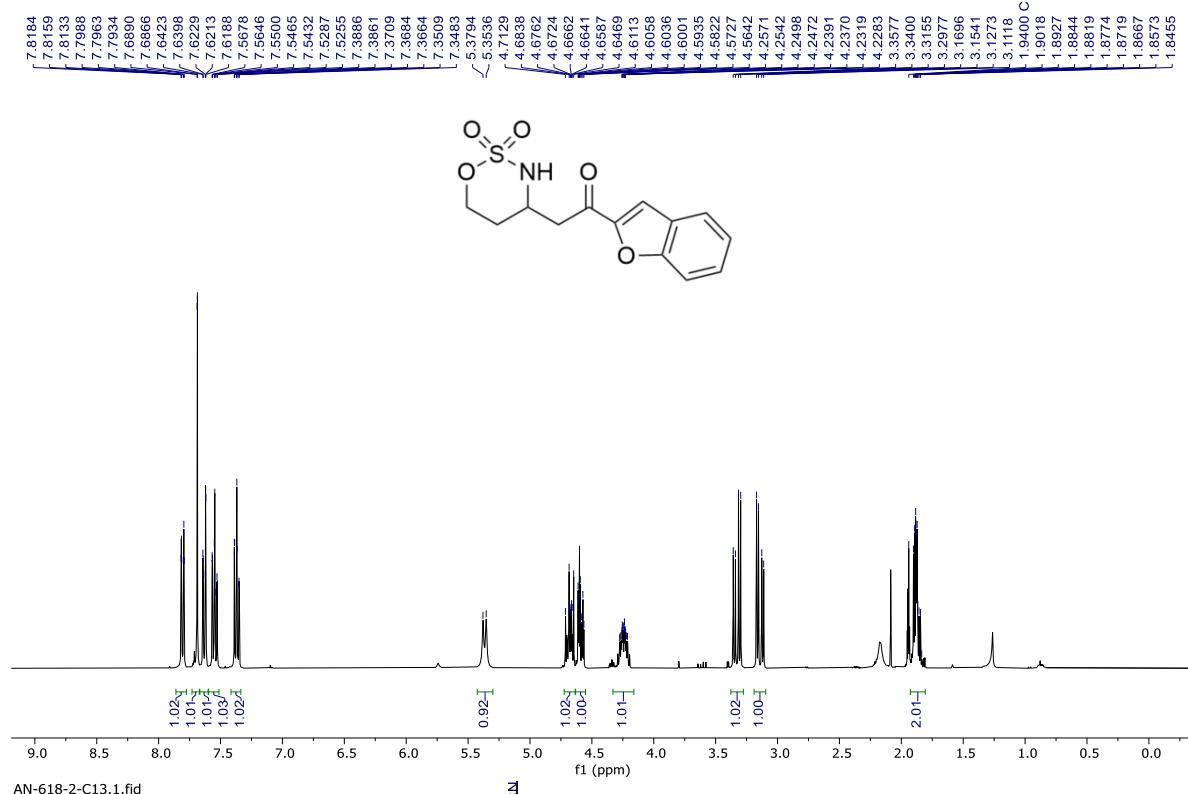


AN-615-C13.1.fid

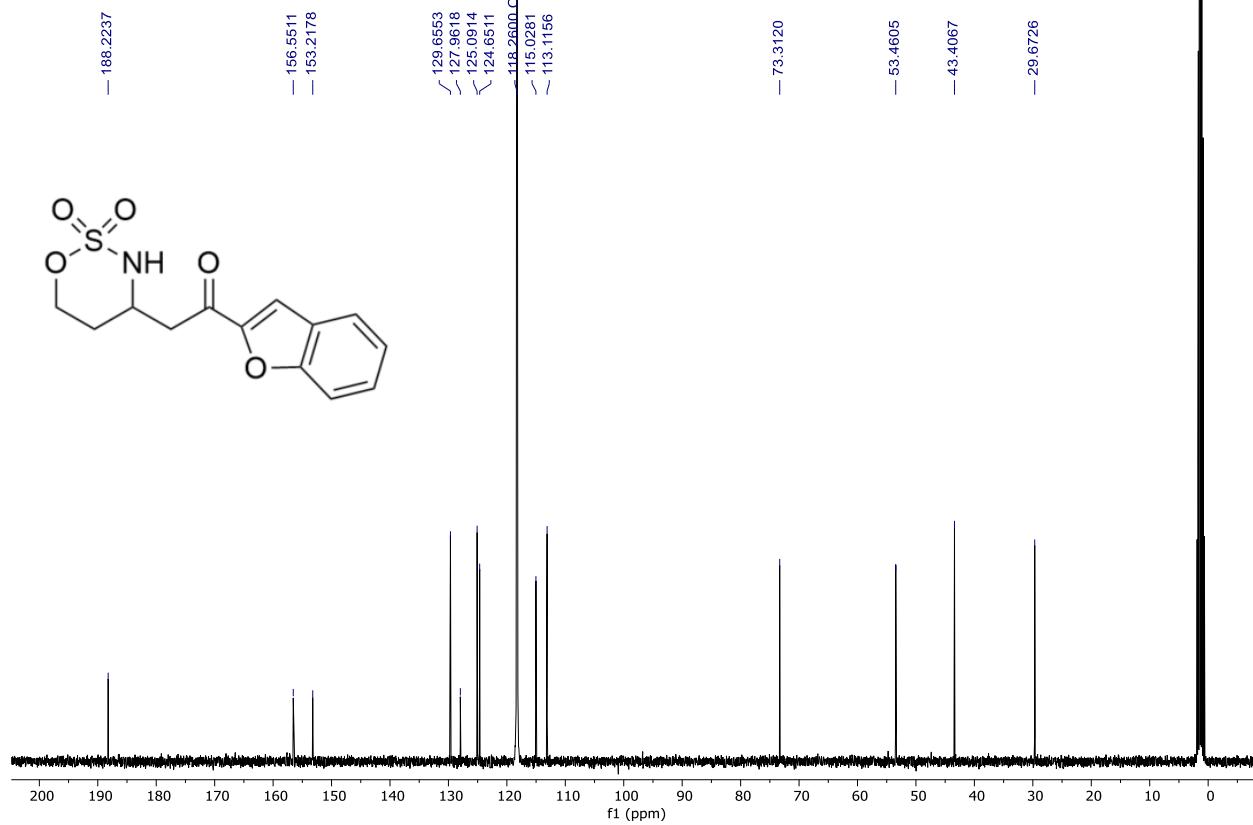


Compound 21 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-618-2-H1.1.fid

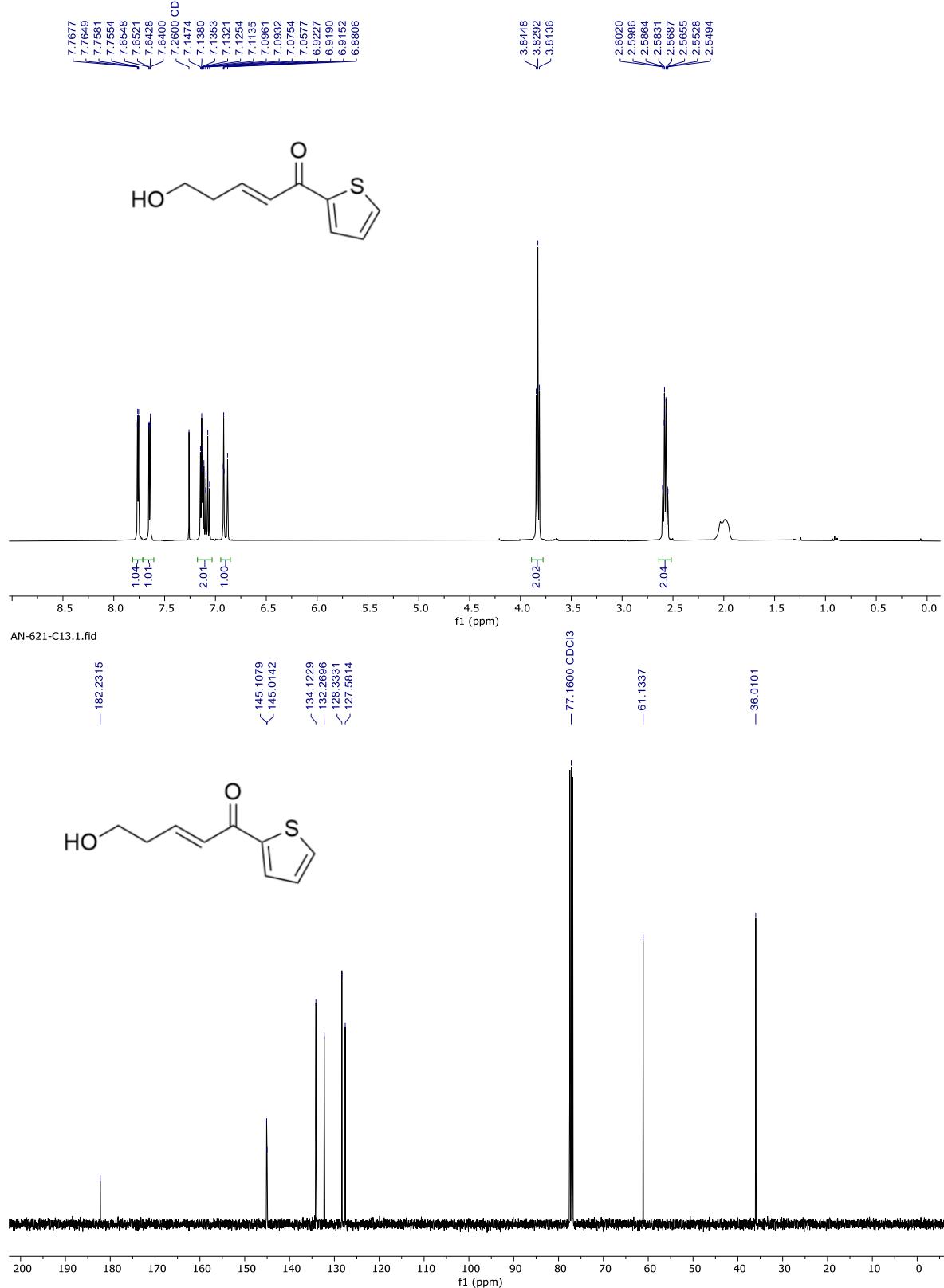


AN-618-2-C13.1.fid



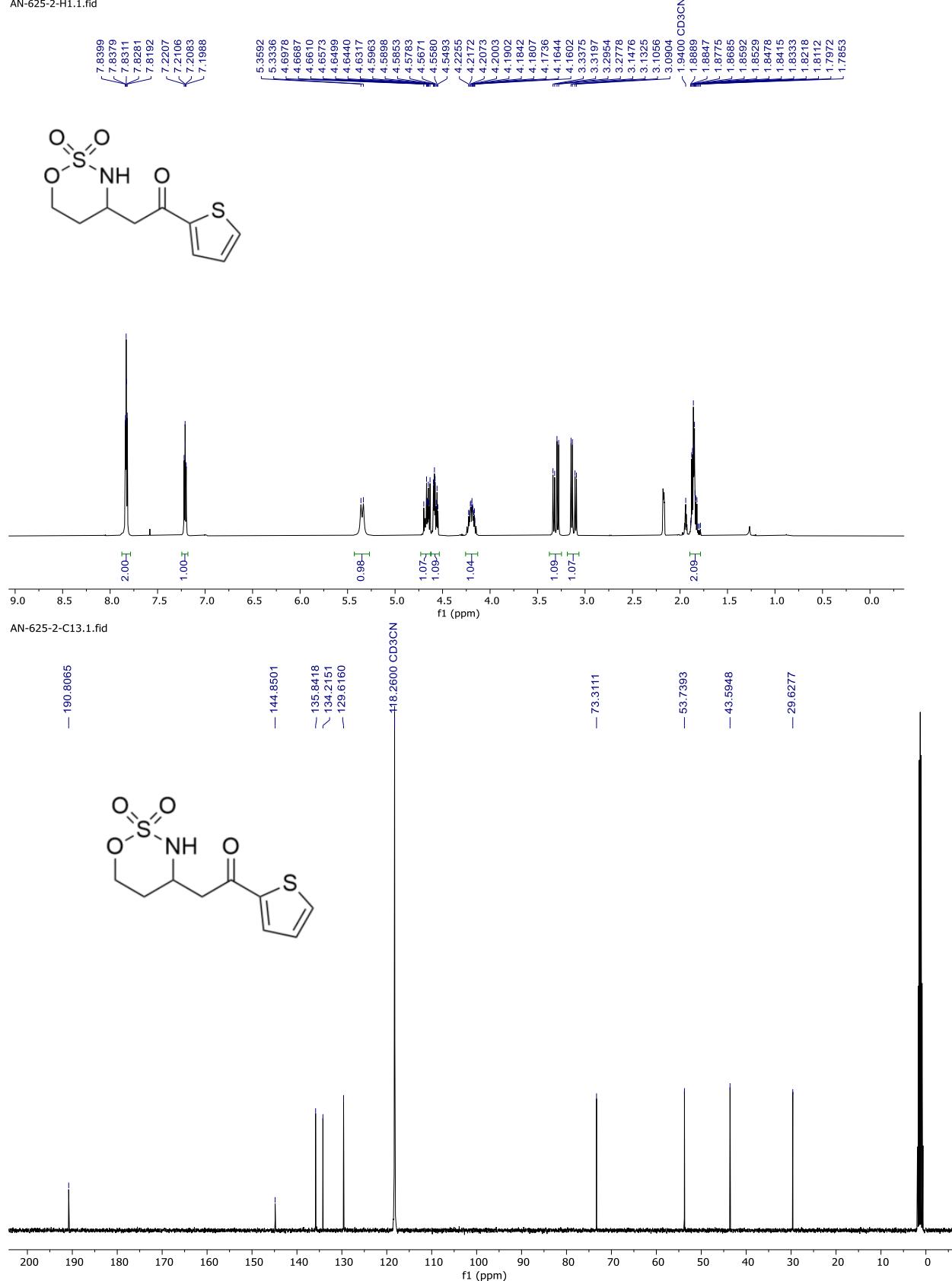
Compound 22 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

AN-621-H1.1.fid



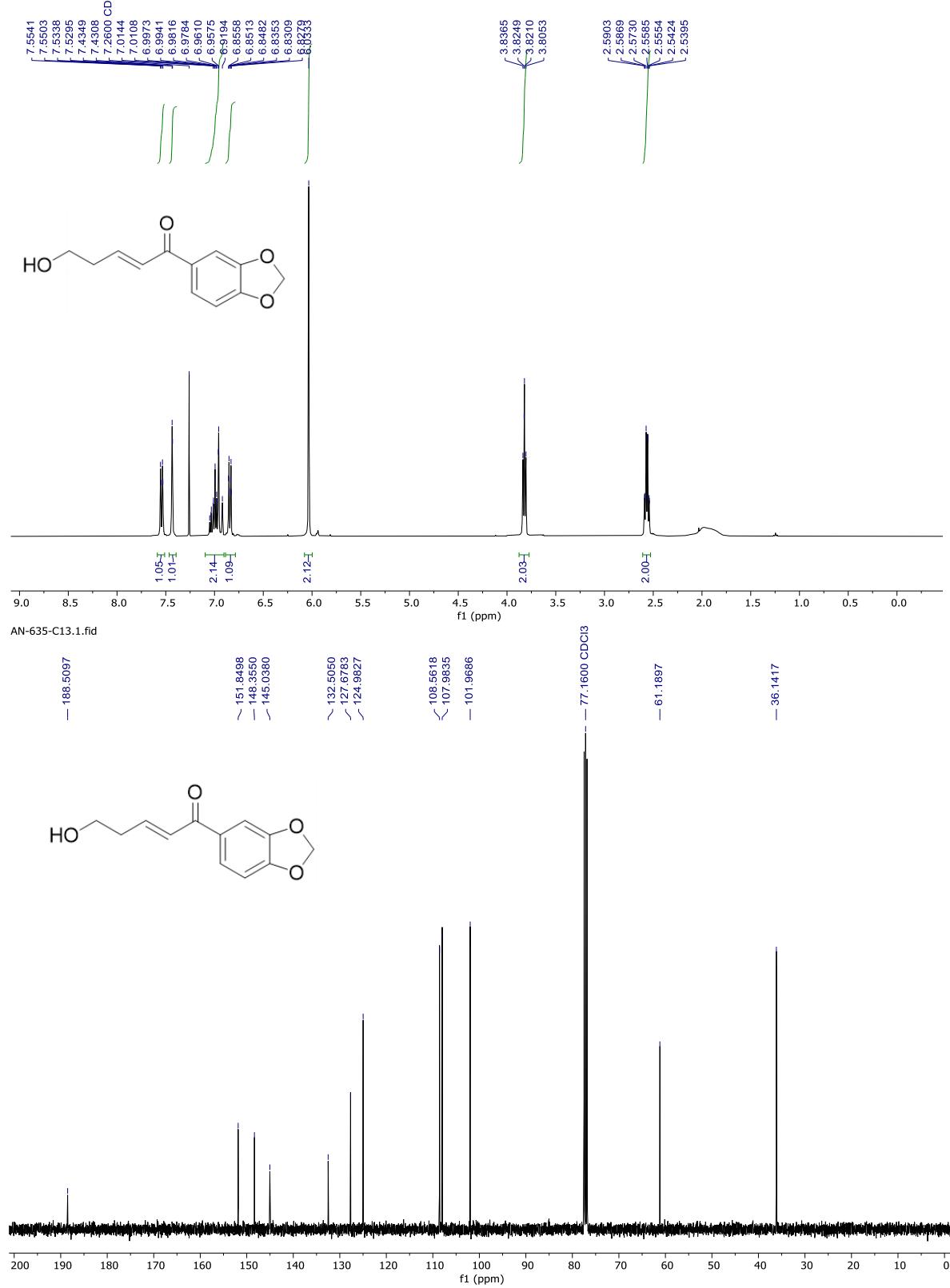
Compound 23 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-625-2-H1.1.fid



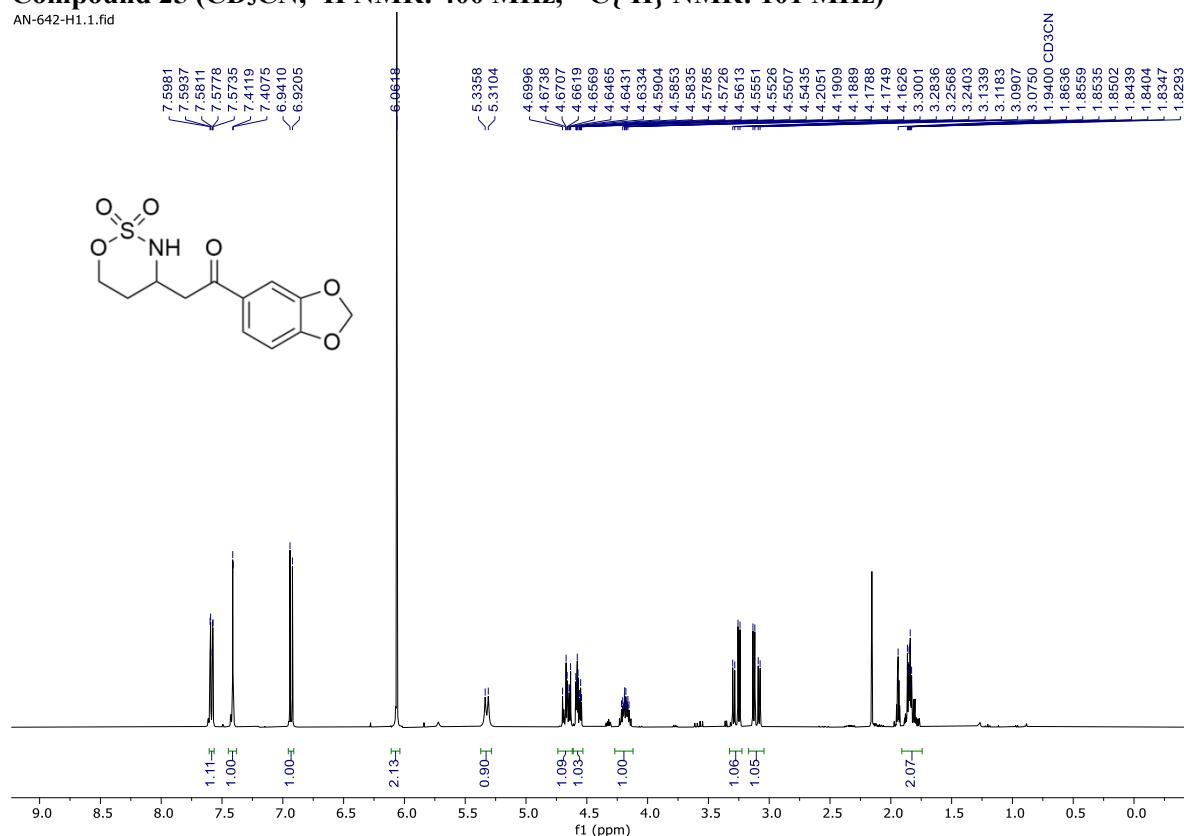
Compound 24 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-635-H1.1.fid

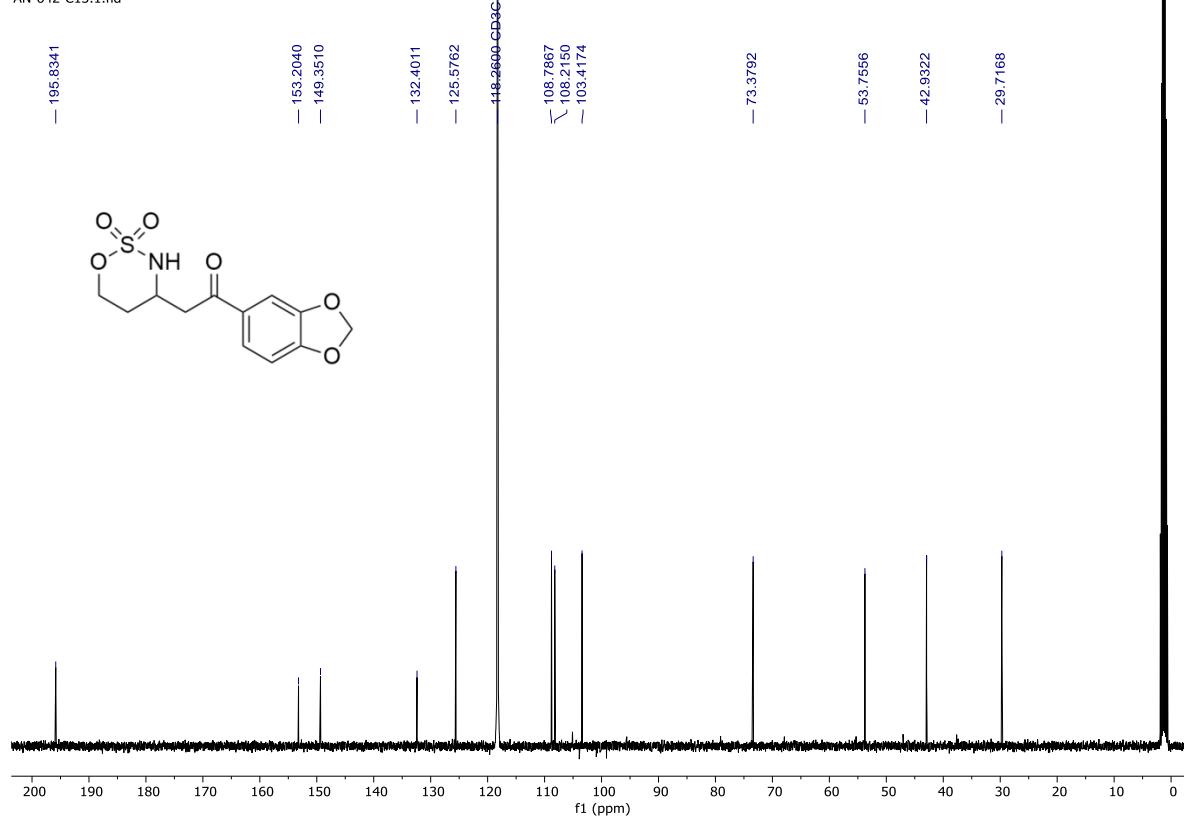


Compound 25 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-642-H1.1.fid

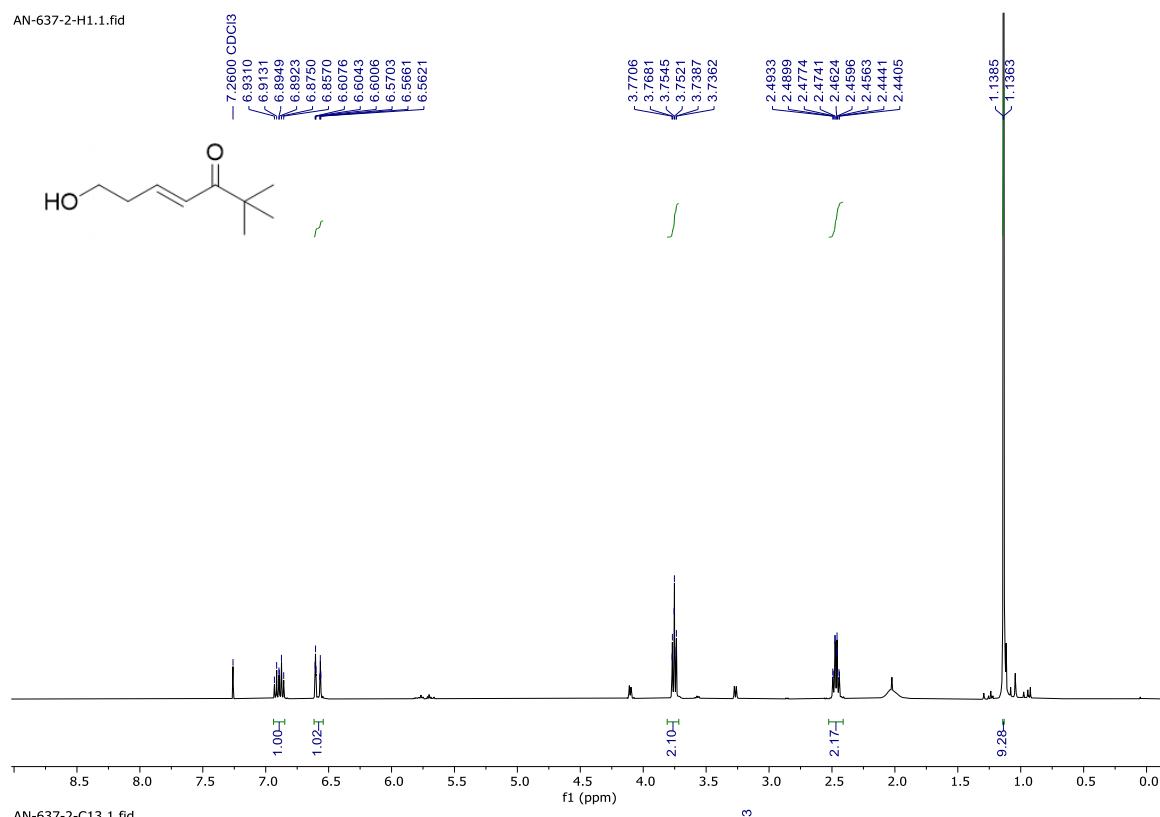


AN-642-C13.1.fid

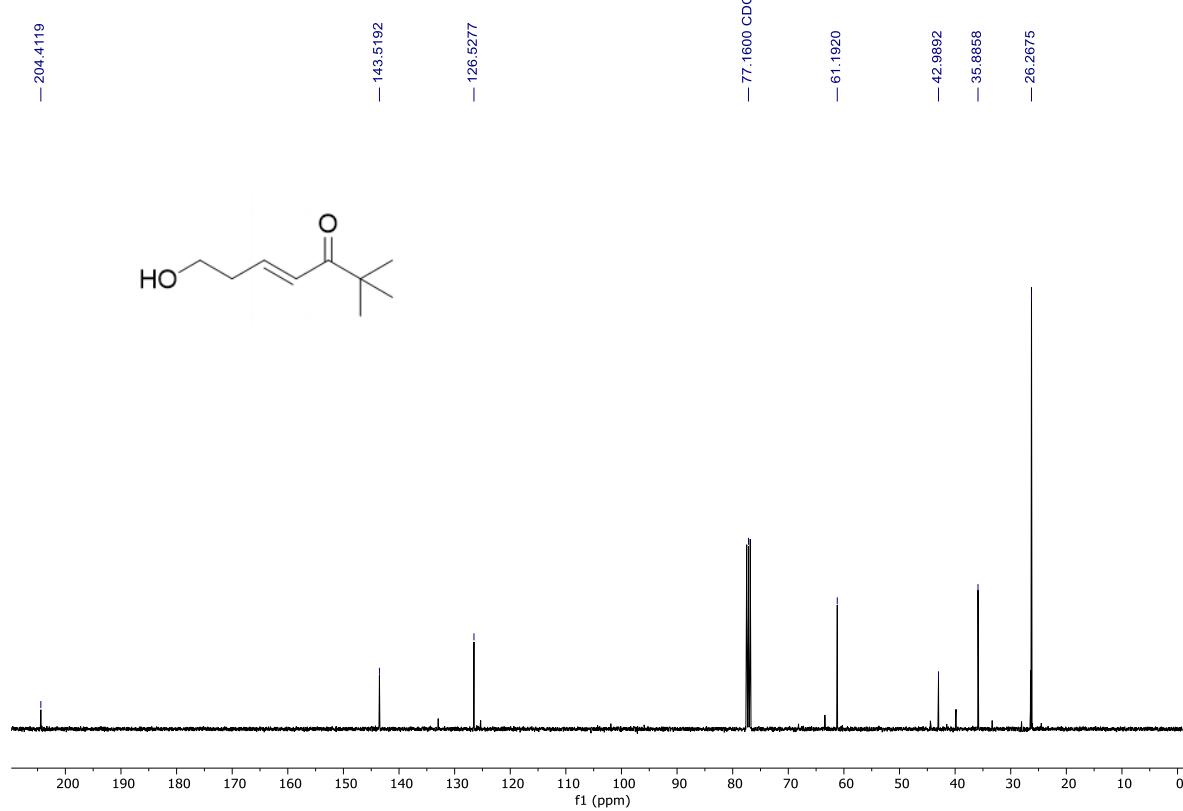


Compound 26 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

AN-637-2-H1.1.fid

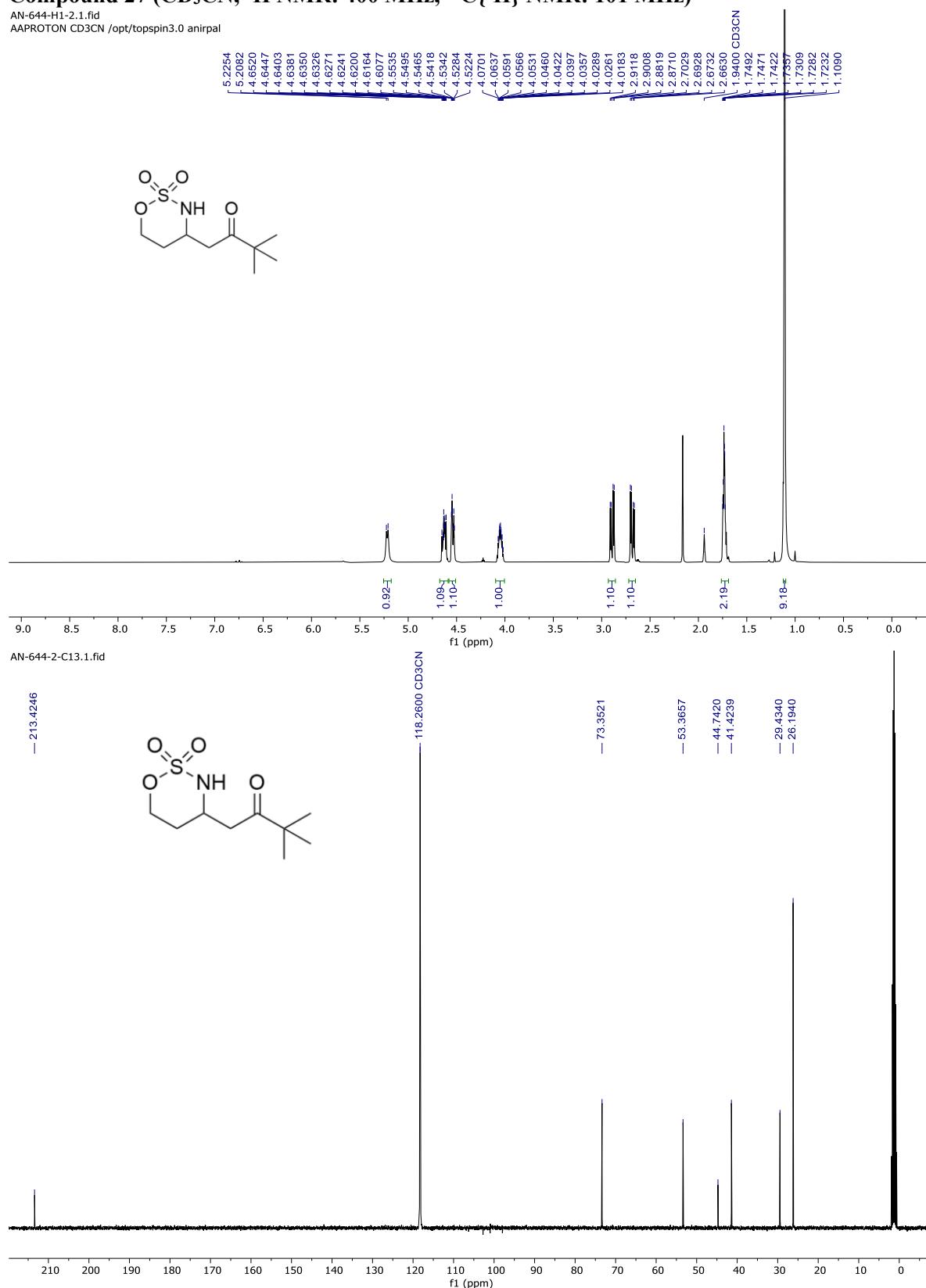


AN-637-2-C13.1.fid



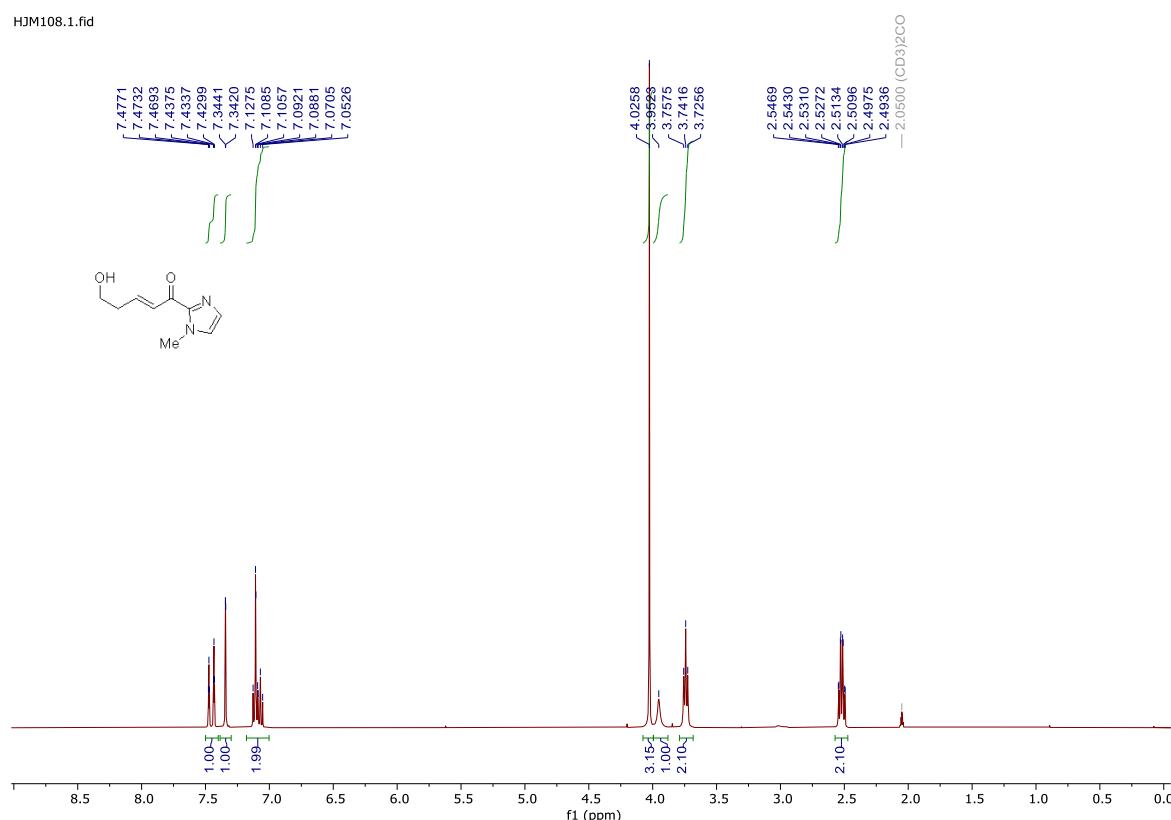
Compound 27 (CD_3CN , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-644-H1-2.1.fid
AAPROTON CD3CN /opt/topspin3.0 anirpal

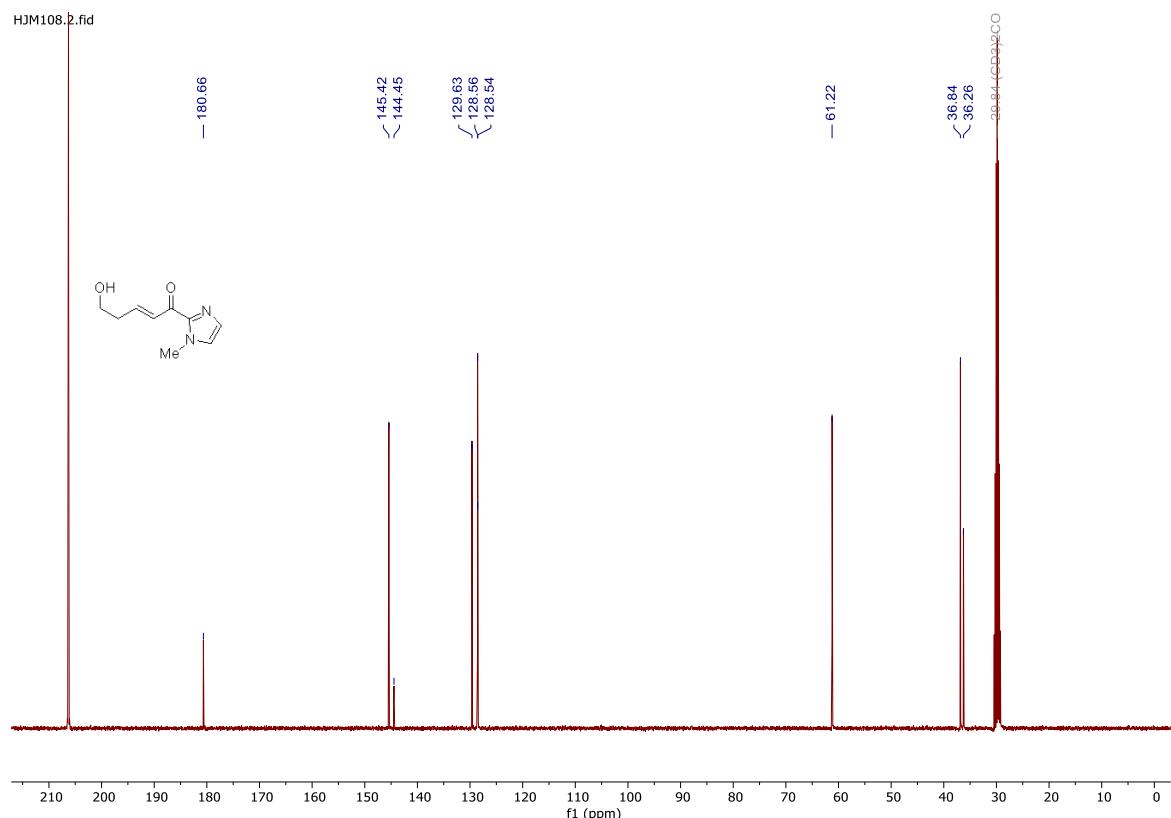


Compound 28 (Acetone-*d*₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

HJM108.1.fid

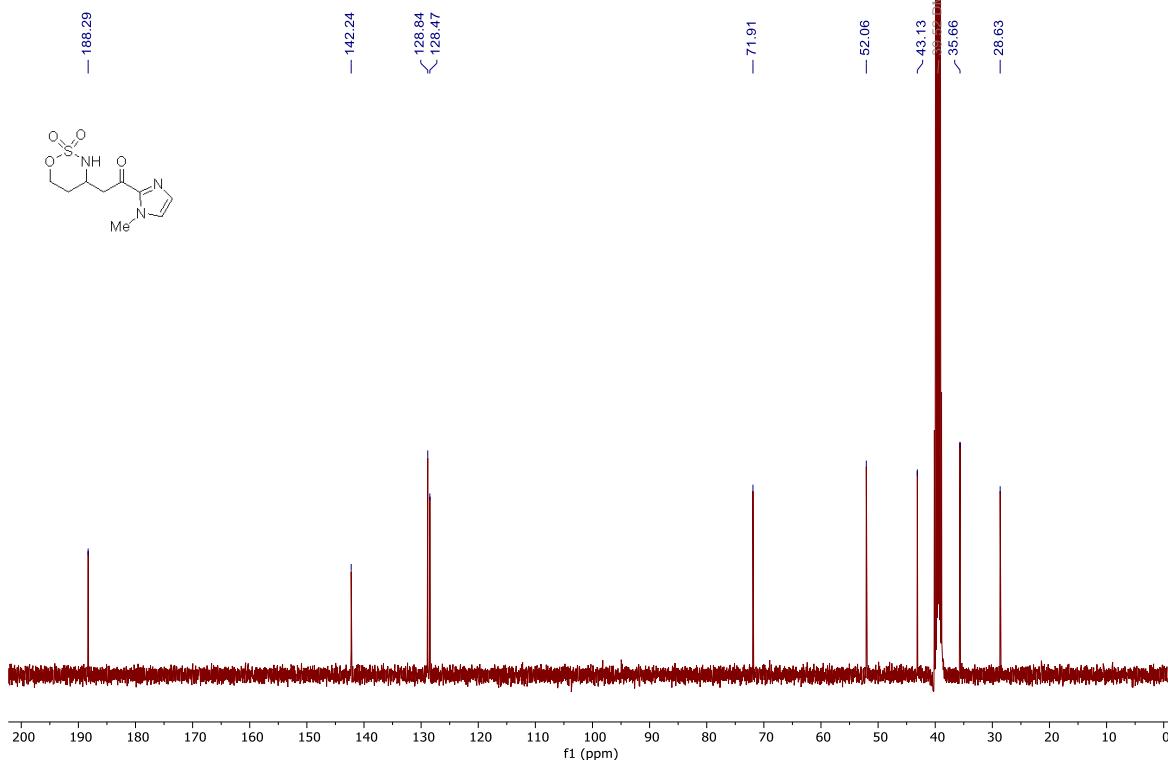
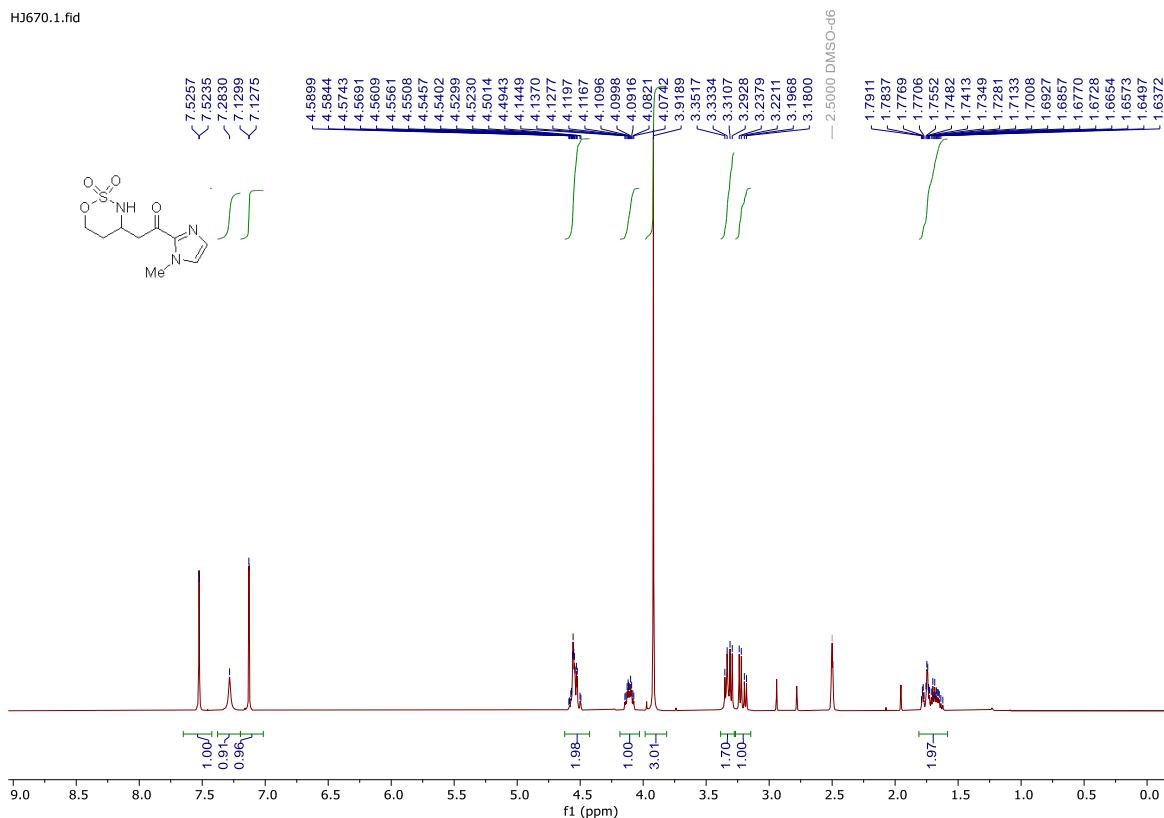


HJM108.2.fid



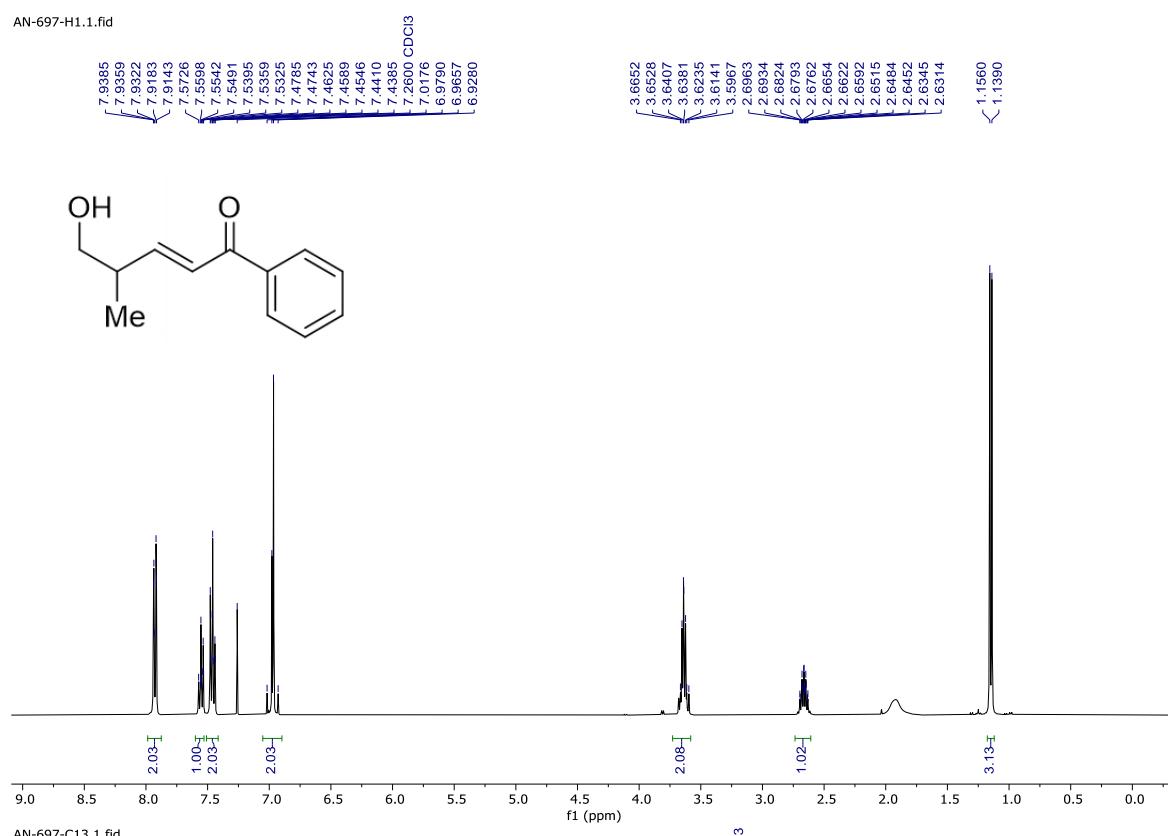
Compound 29 (DMSO-*d*₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

HJ670.1.fid

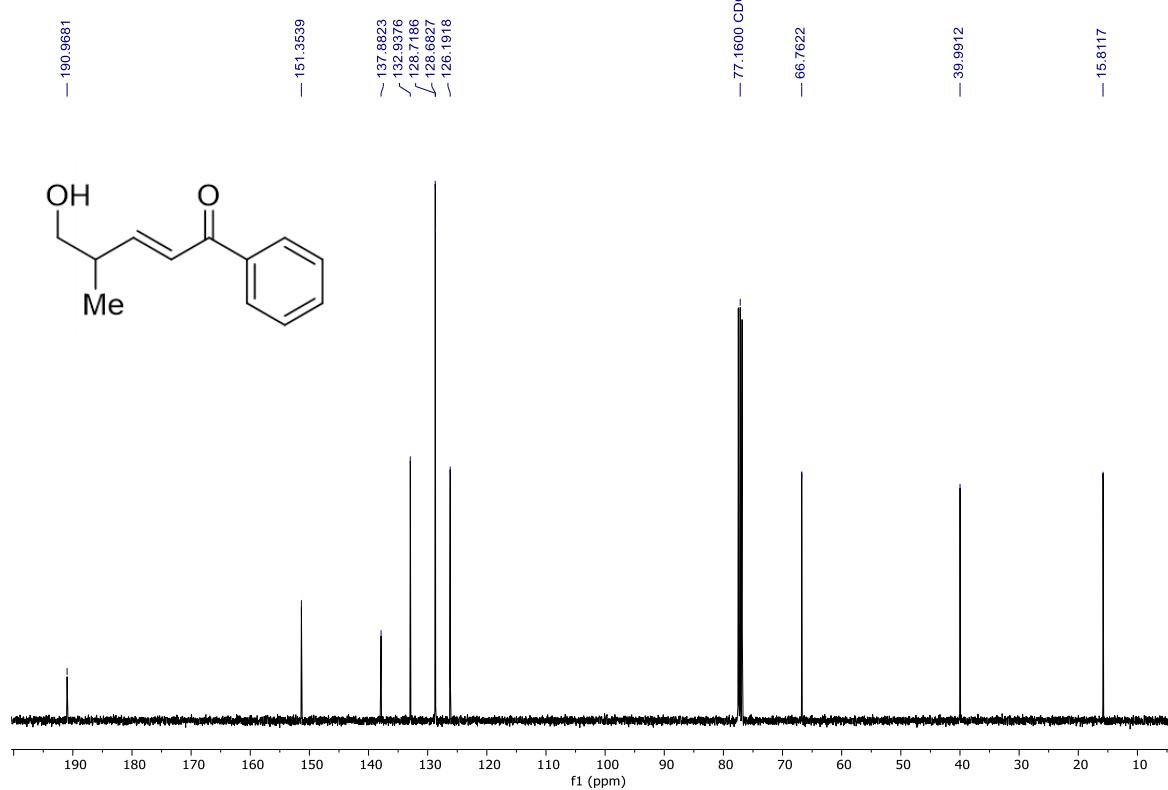


Compound 30 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-697-H1.1.fid

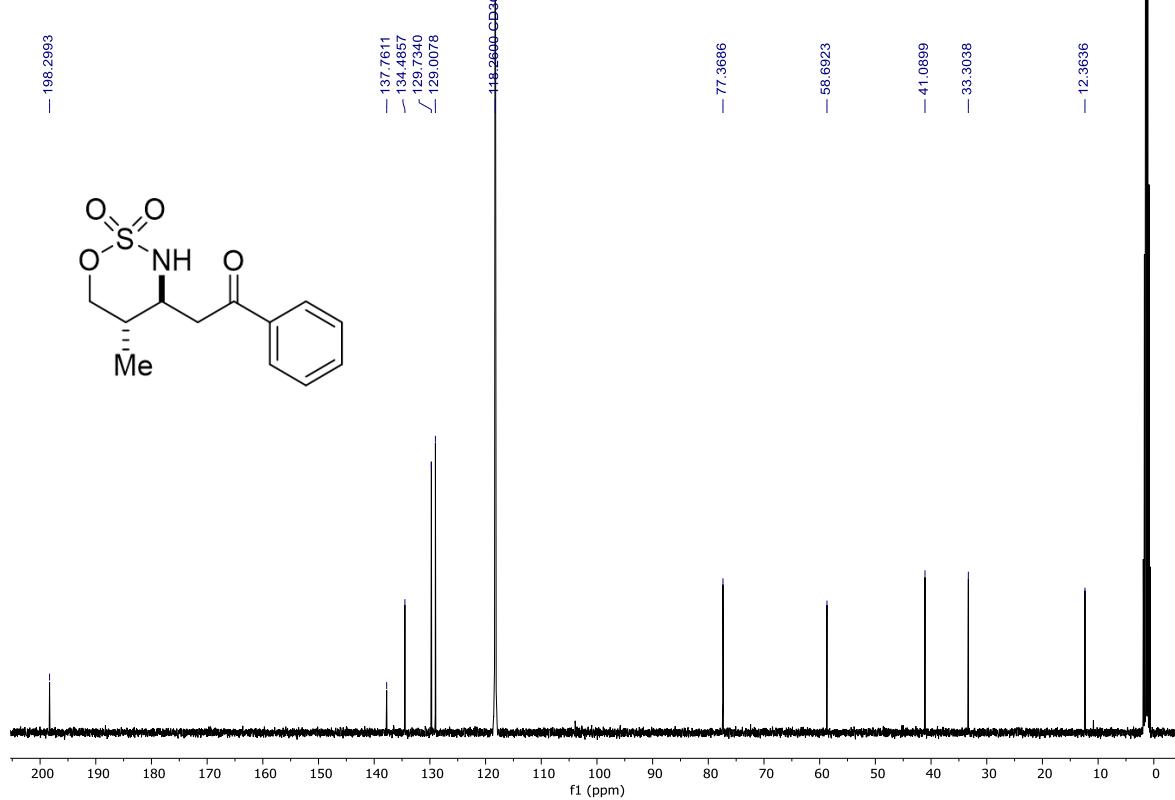
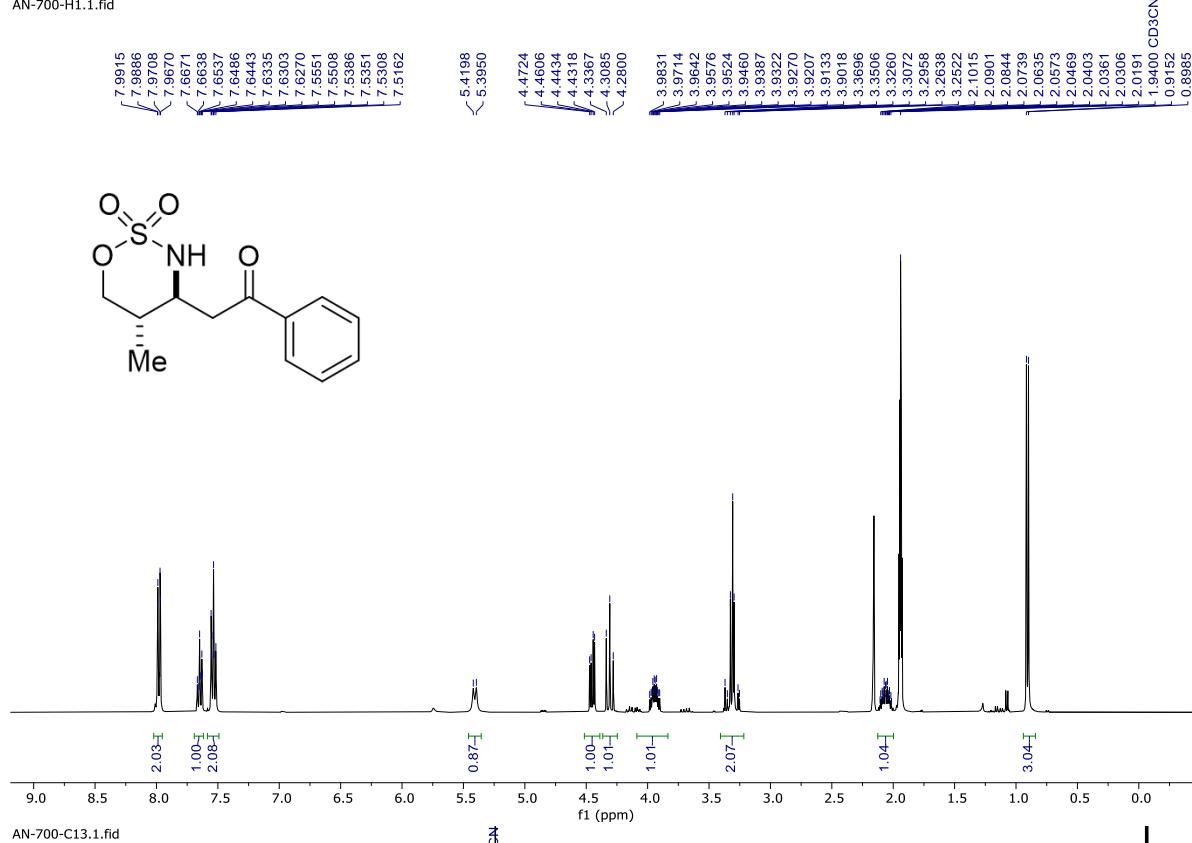


AN-697-C13.1.fid

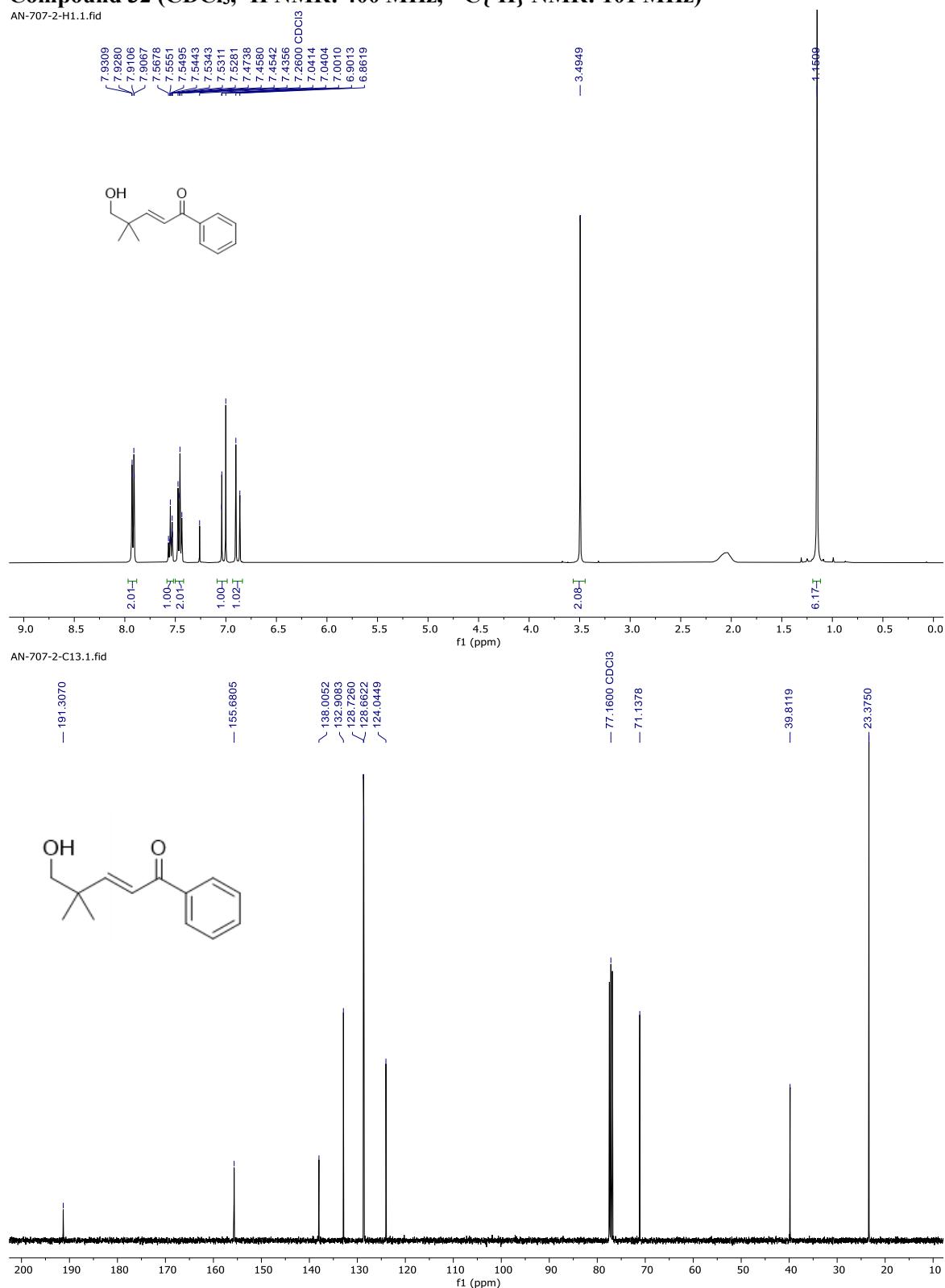


Compound 31 (CD_3CN , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-700-H1.1.fid

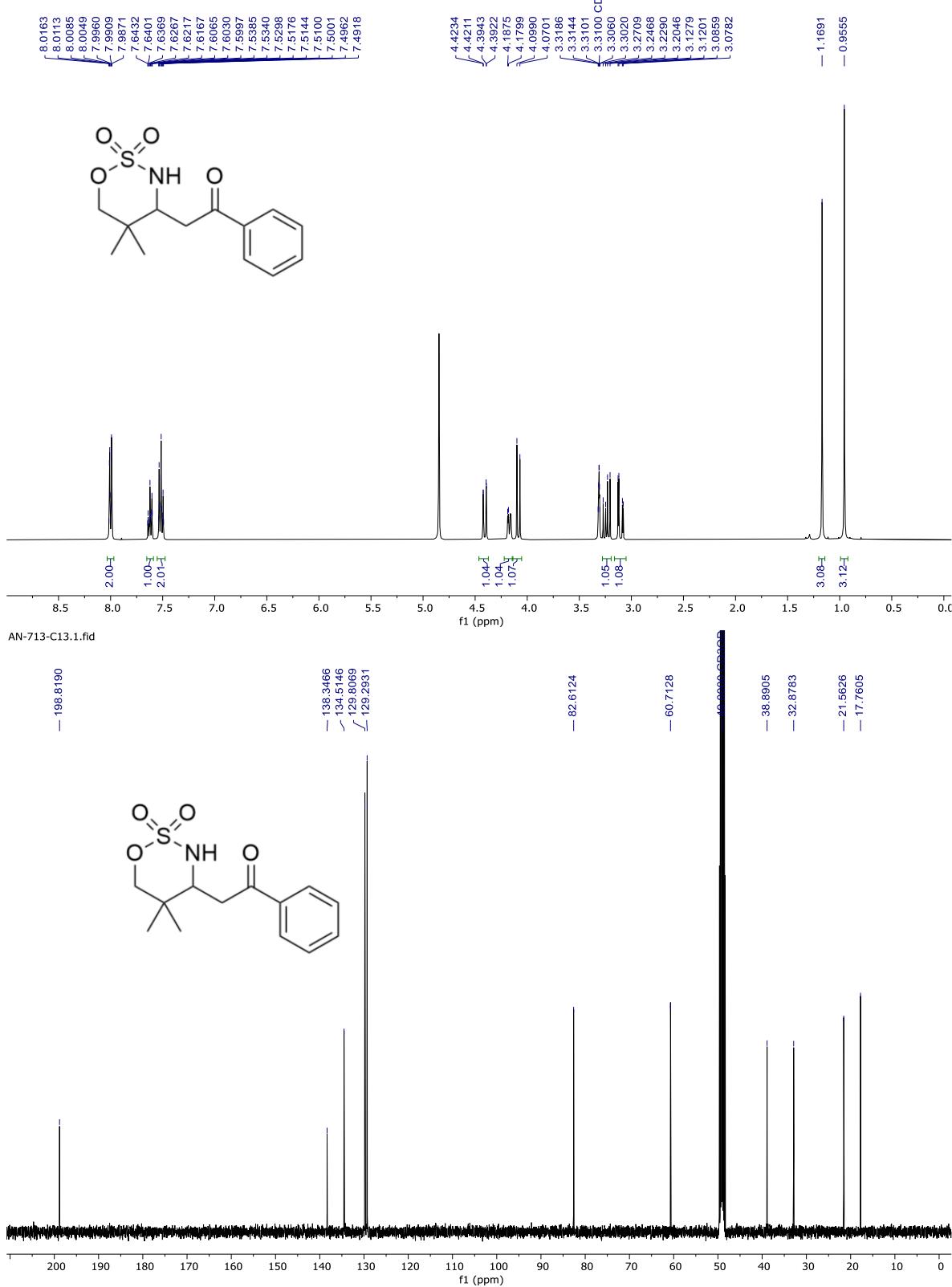


Compound 32 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)
AN-707-2-H1.1.fid



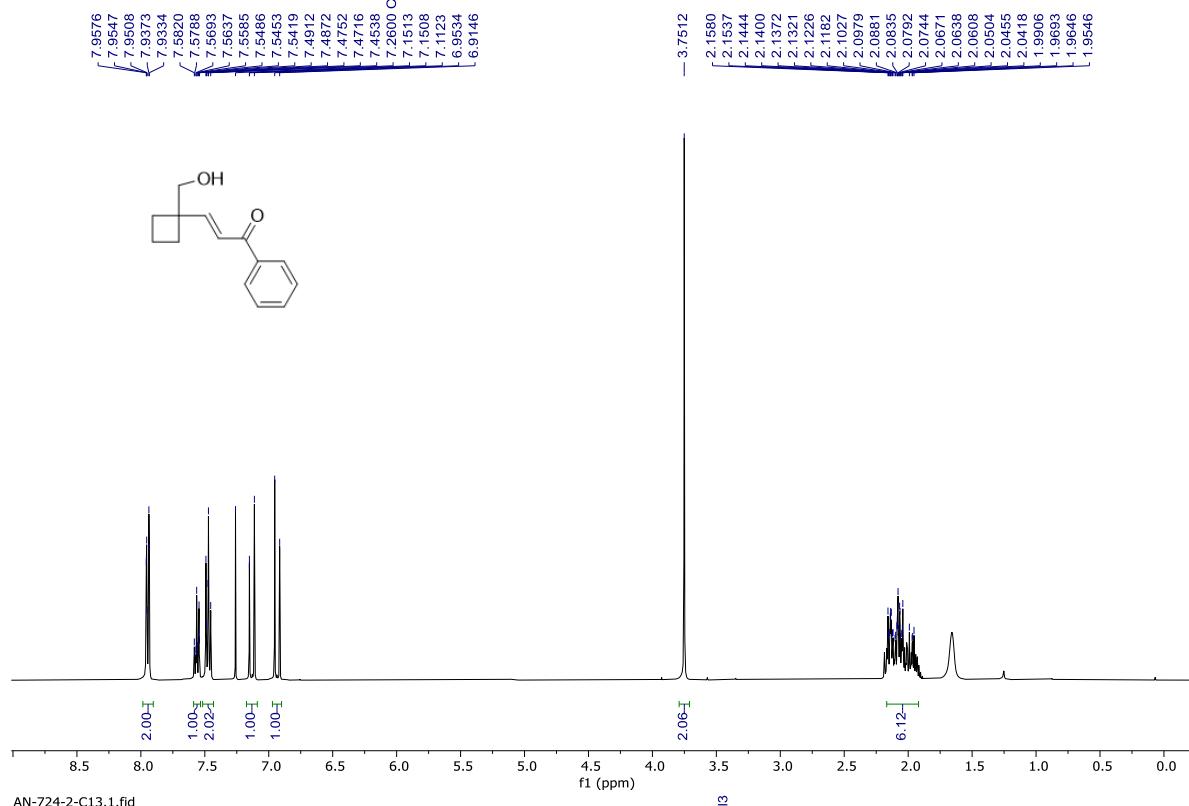
Compound 33 (CD₃OD, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-713-H1.1.fid



Compound 34 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

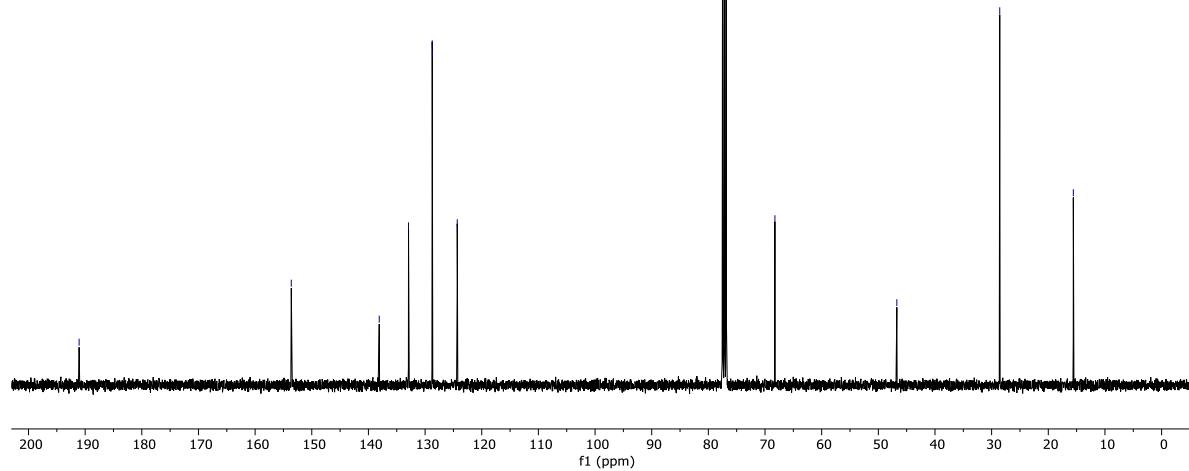
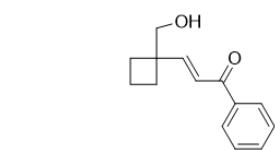
AN-724-2-H1.1.fid



— 191.0659
— 153.6148

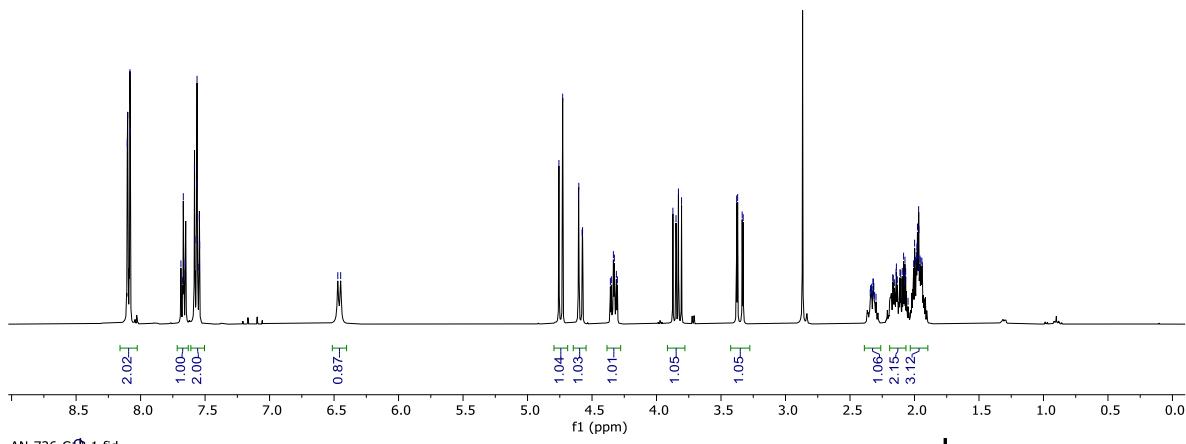
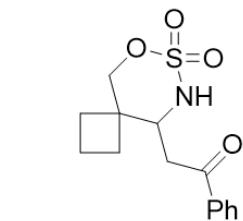
~ 138.6941
~ 132.9225
~ 128.7399
~ 128.7048
~ 124.3241

— 3.7512
— 2.1580
— 2.1537
— 2.1444
— 2.1400
— 2.1372
— 2.1321
— 2.1226
— 2.1182
— 2.1027
— 2.0979
— 2.0881
— 2.0835
— 2.0792
— 2.0744
— 2.0671
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— 2.0418
— 1.9806
— 1.9693
— 1.9546
— 1.9546

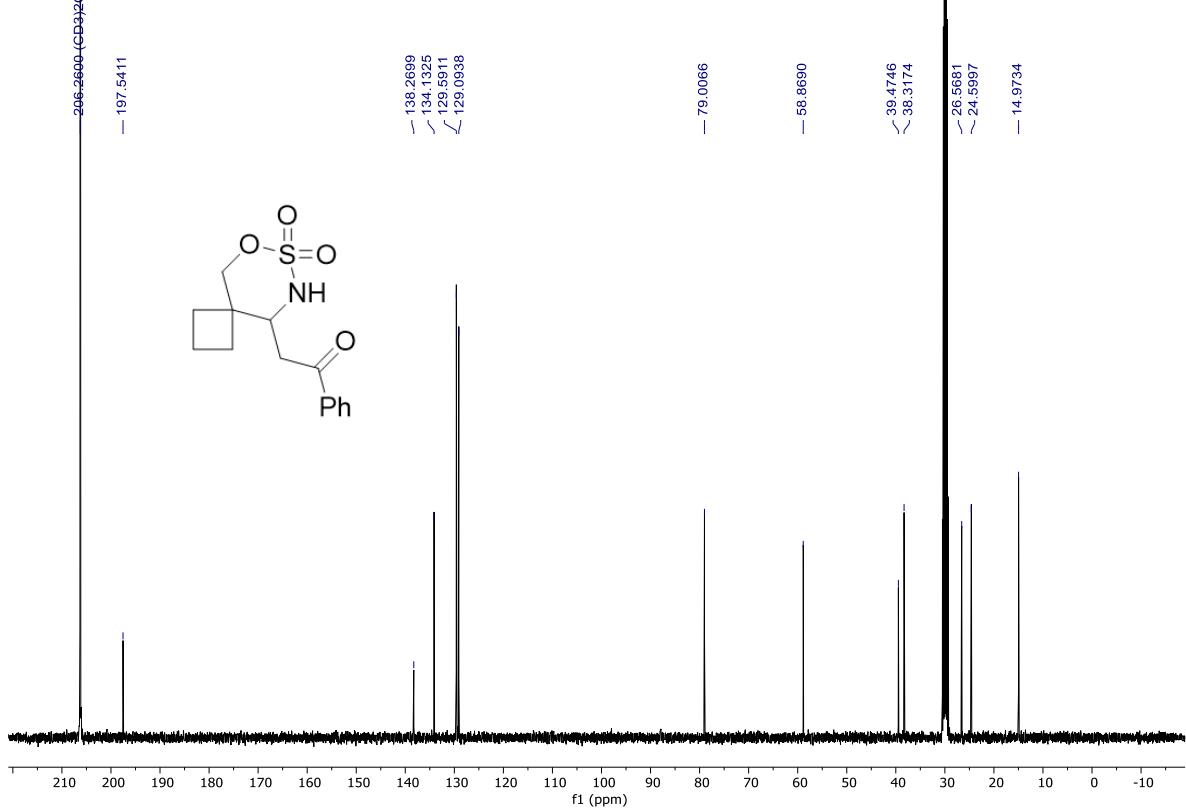


Compound 35 (Acetone-d₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-726-H1.1.fid

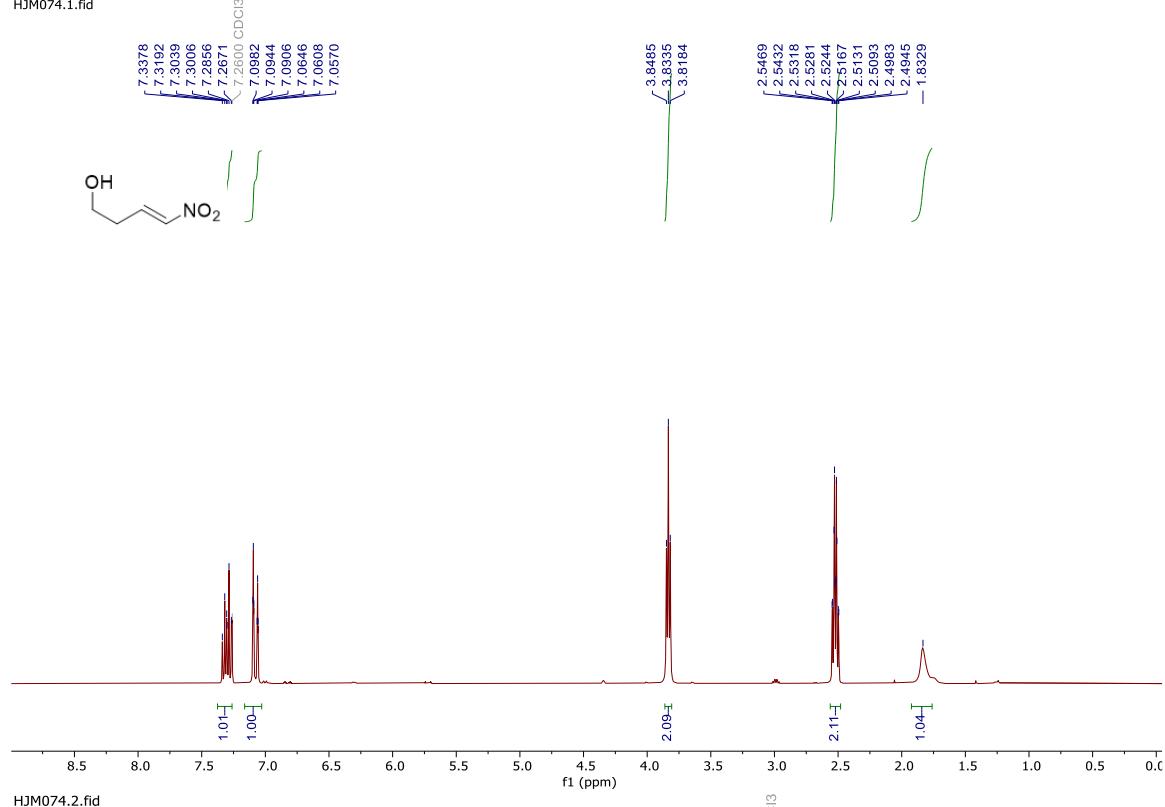


AN-726-C13.1.fid

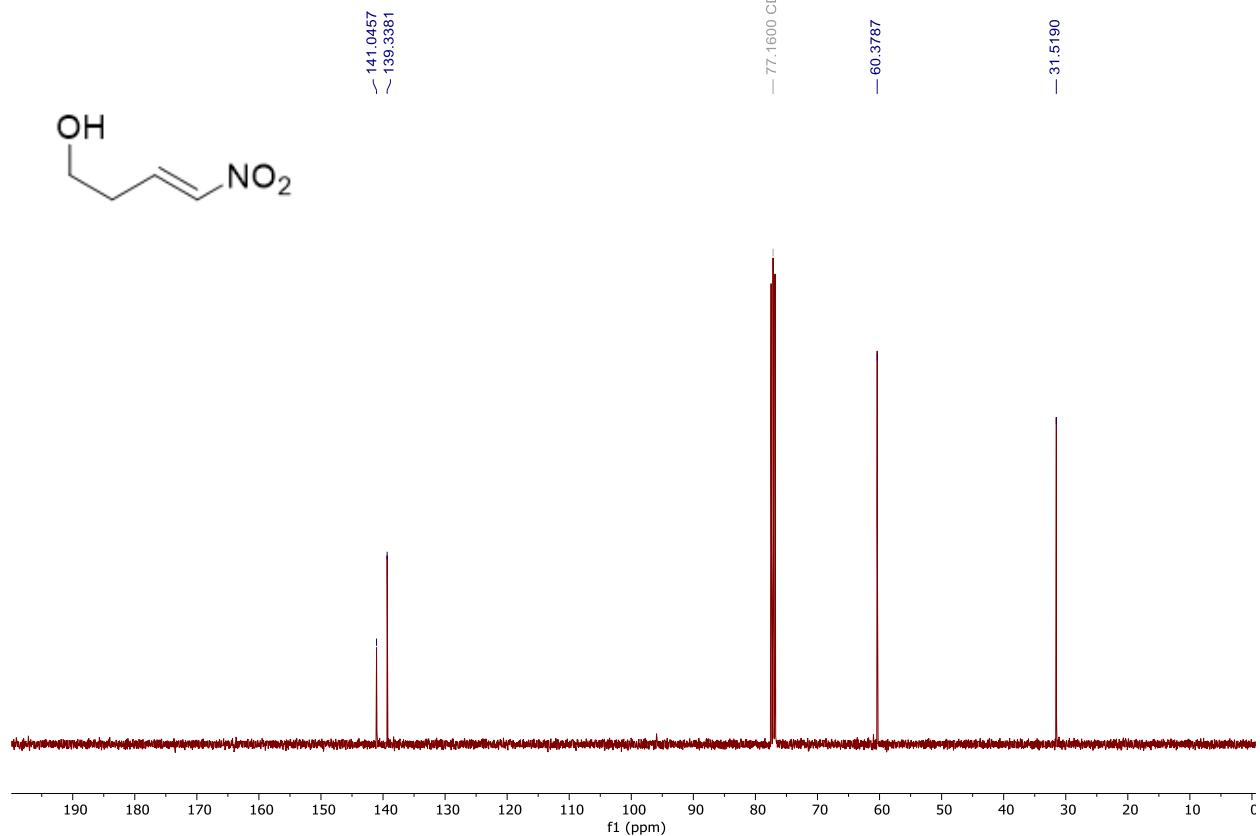


Compound 36 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

HJM074.1.fid

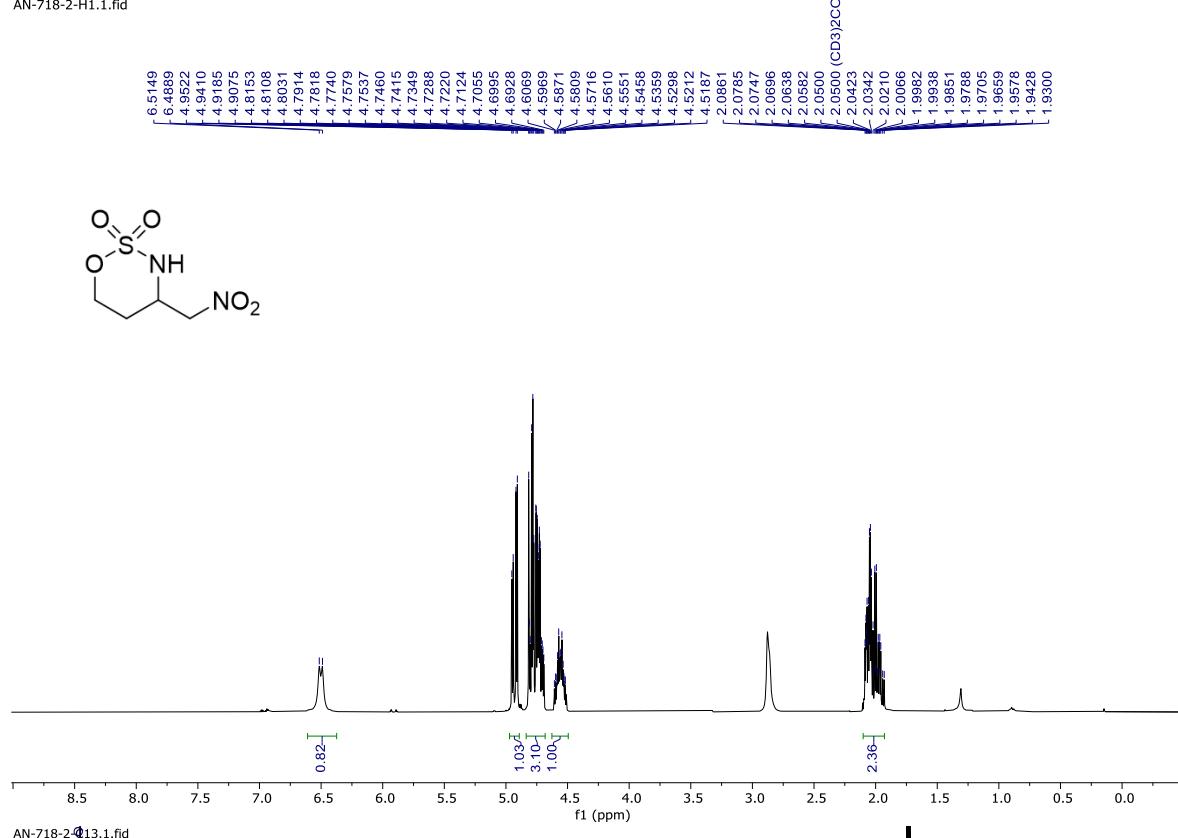


HJM074.2.fid

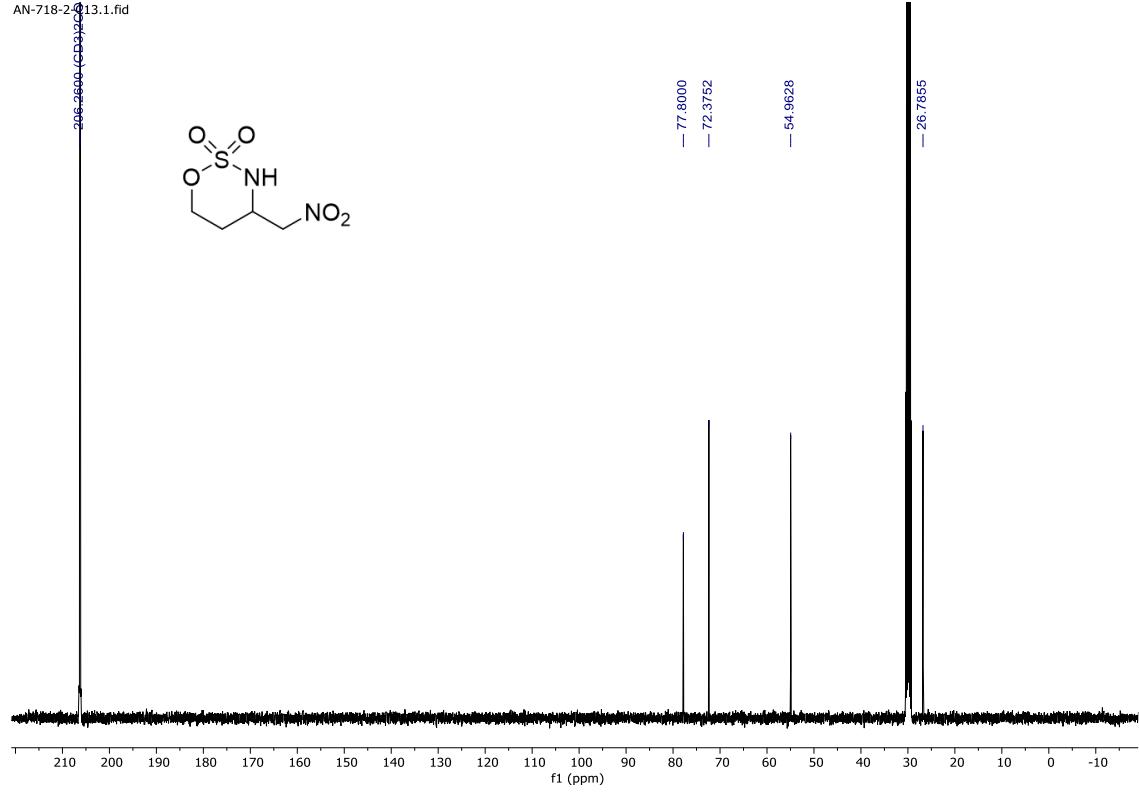


Compound 37 (Acetone-d₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-718-2-H1.1.fid

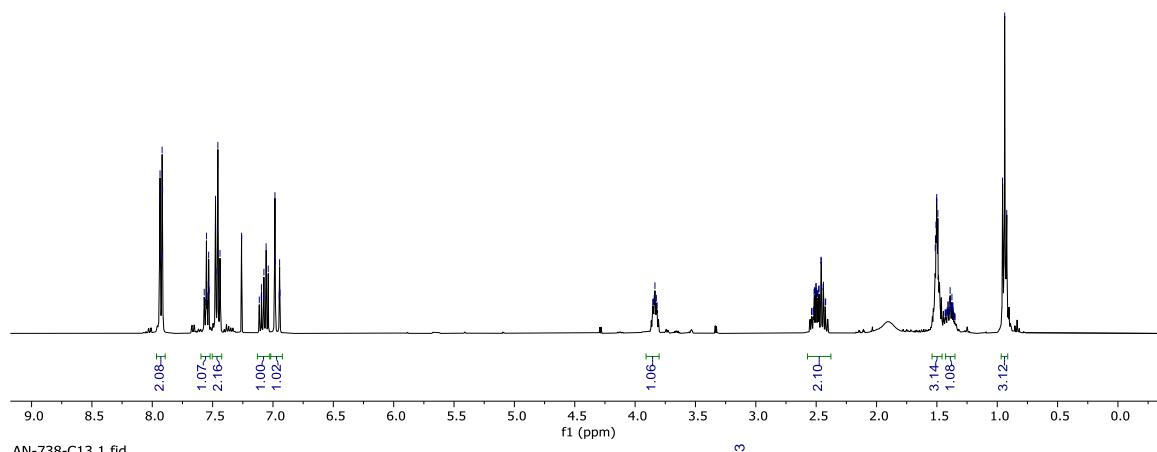
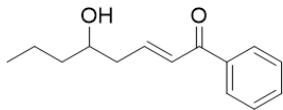


AN-718-2-C13.1.fid

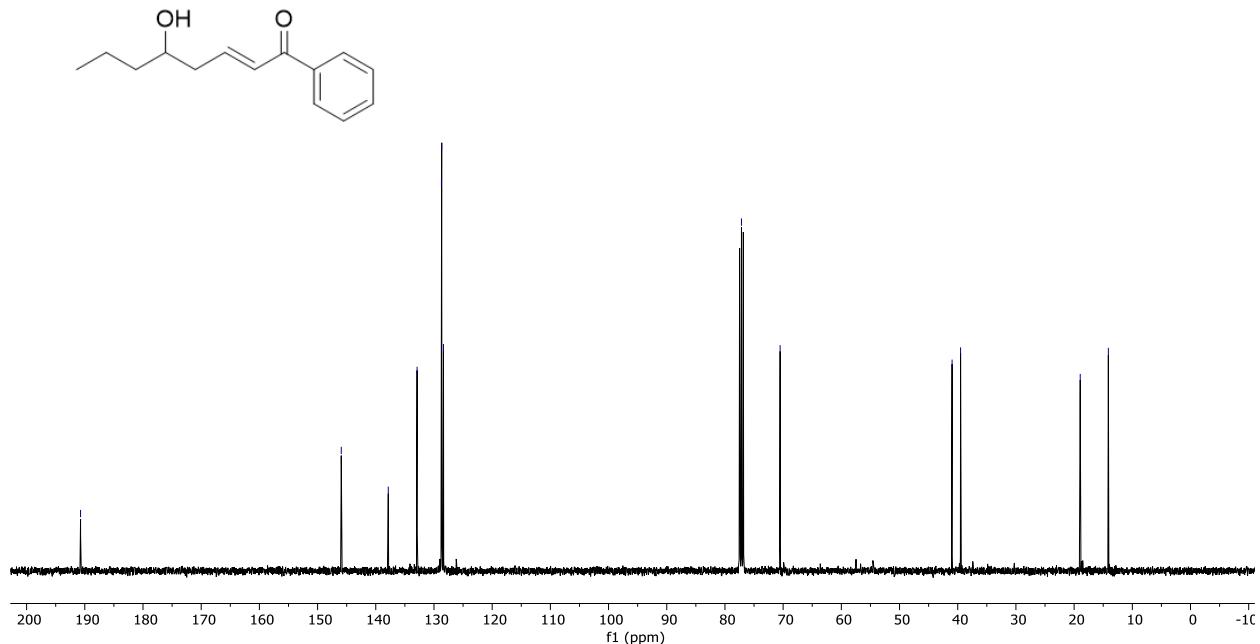


Compound 38 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-738-H1.1.fid

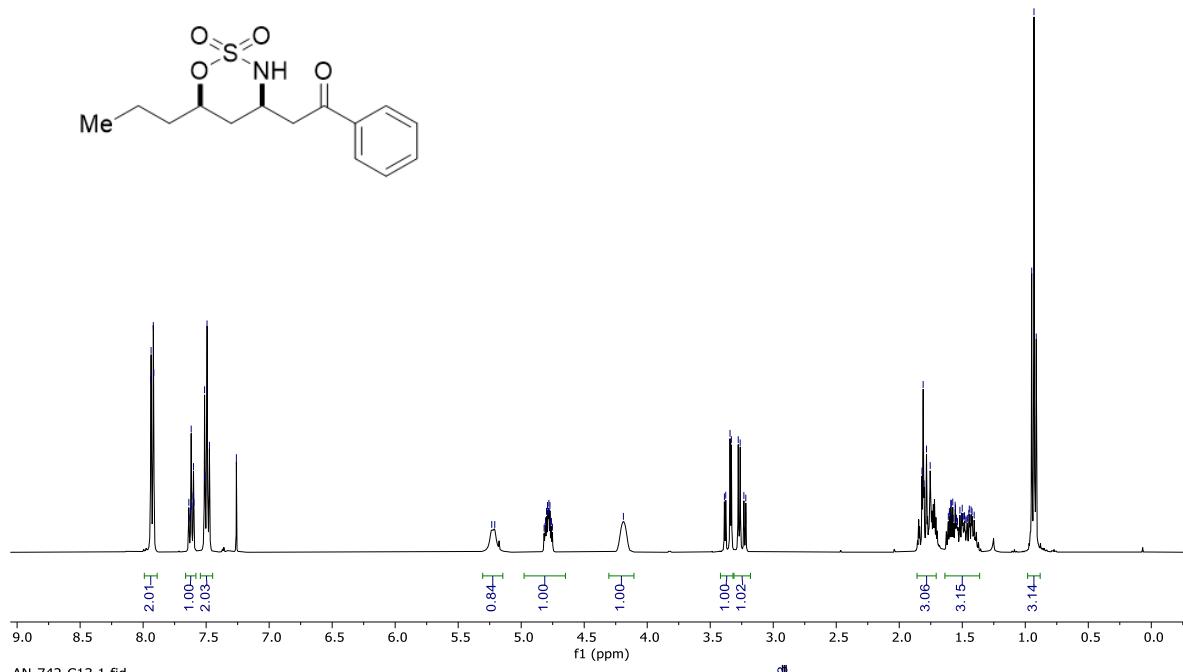


— 190.7290
— 145.9315
— 137.8527
✓ 132.9013
✓ 128.7173
✓ 128.6750
✓ 128.3670

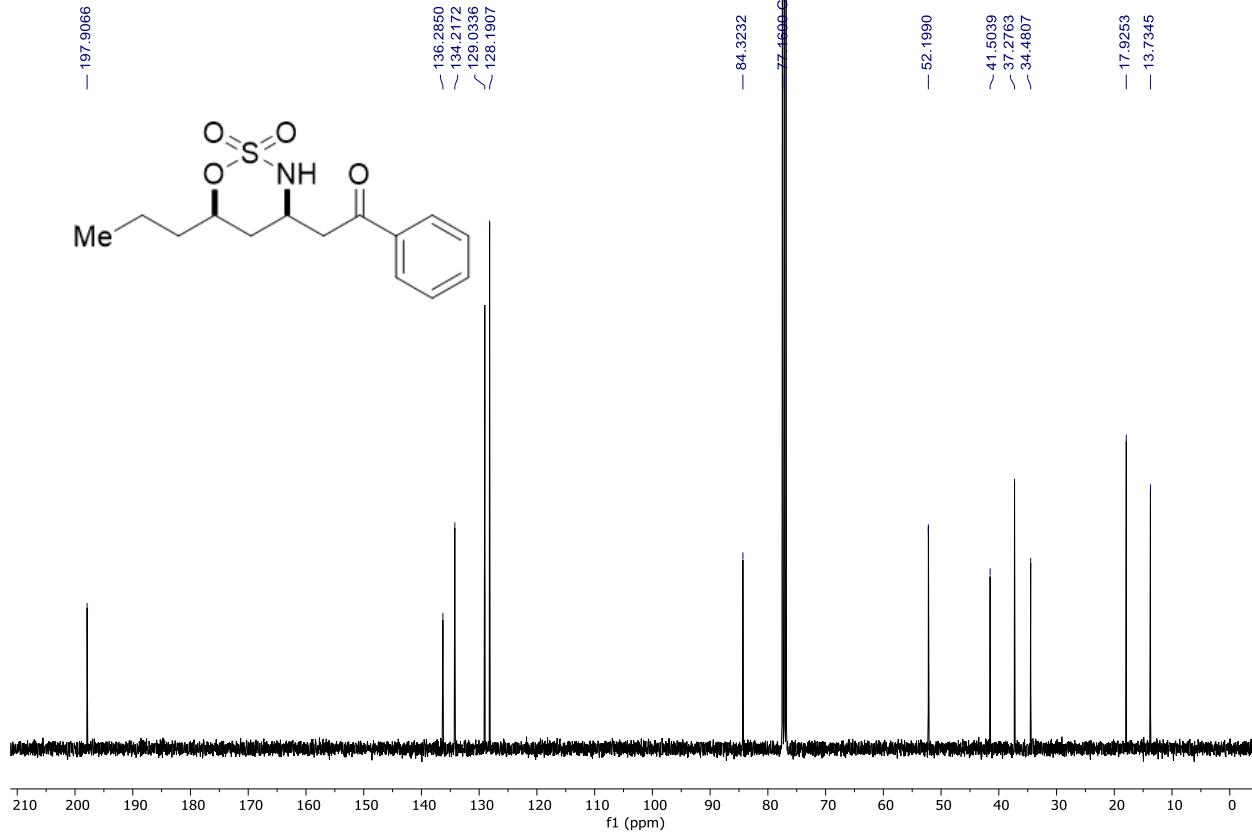


Compound 39 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-742-H1.1.fid

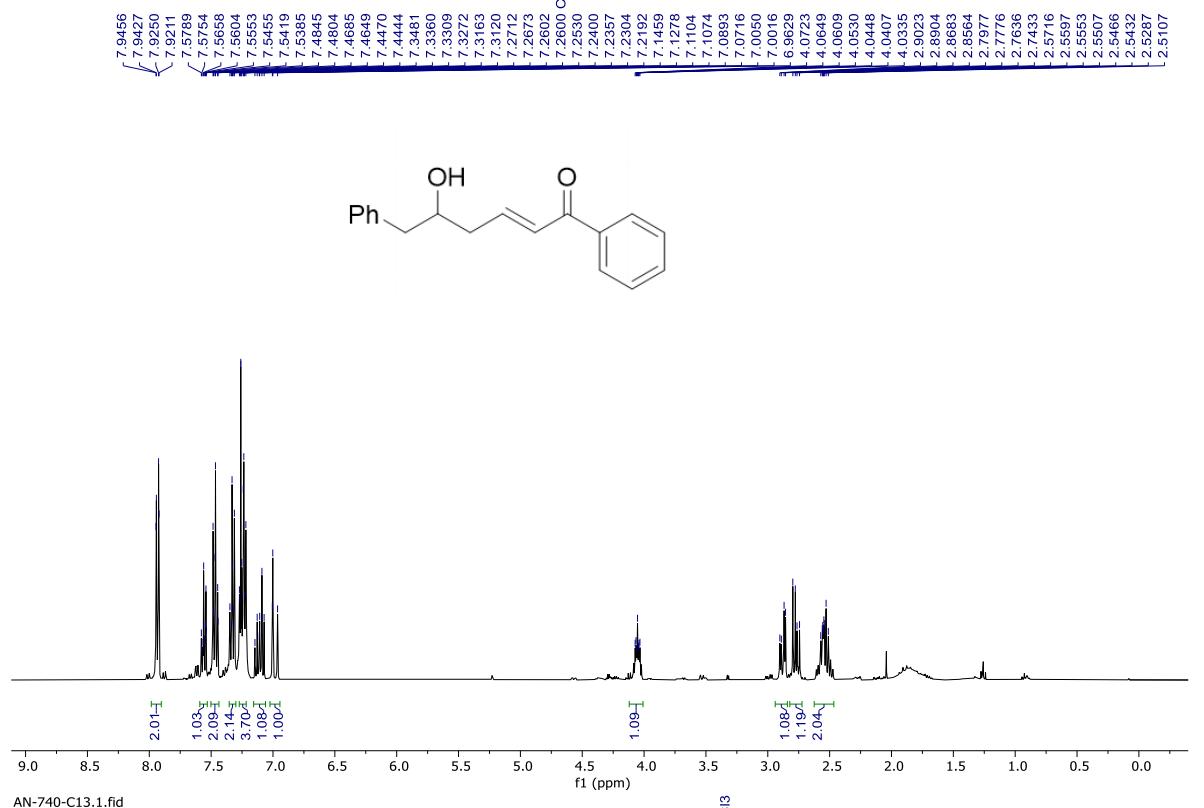


AN-742-C13.1.fid

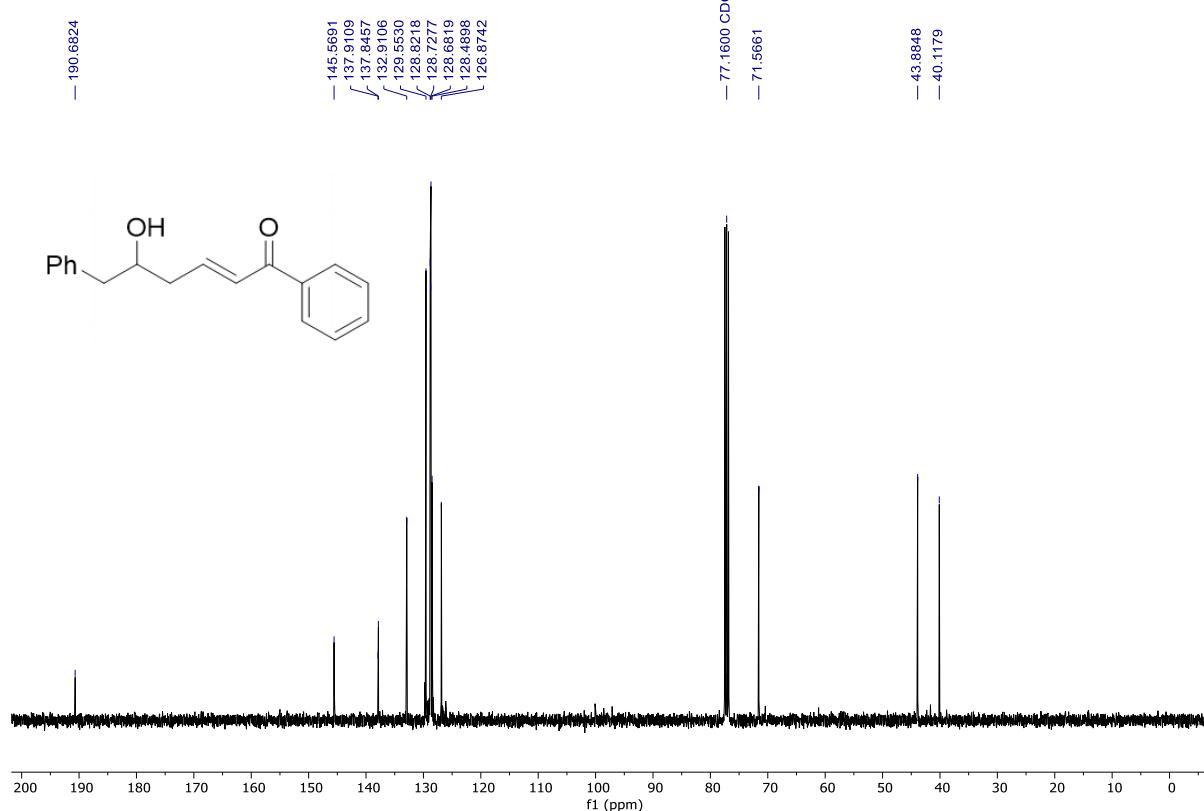


Compound 40 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-740-H1.1.fid

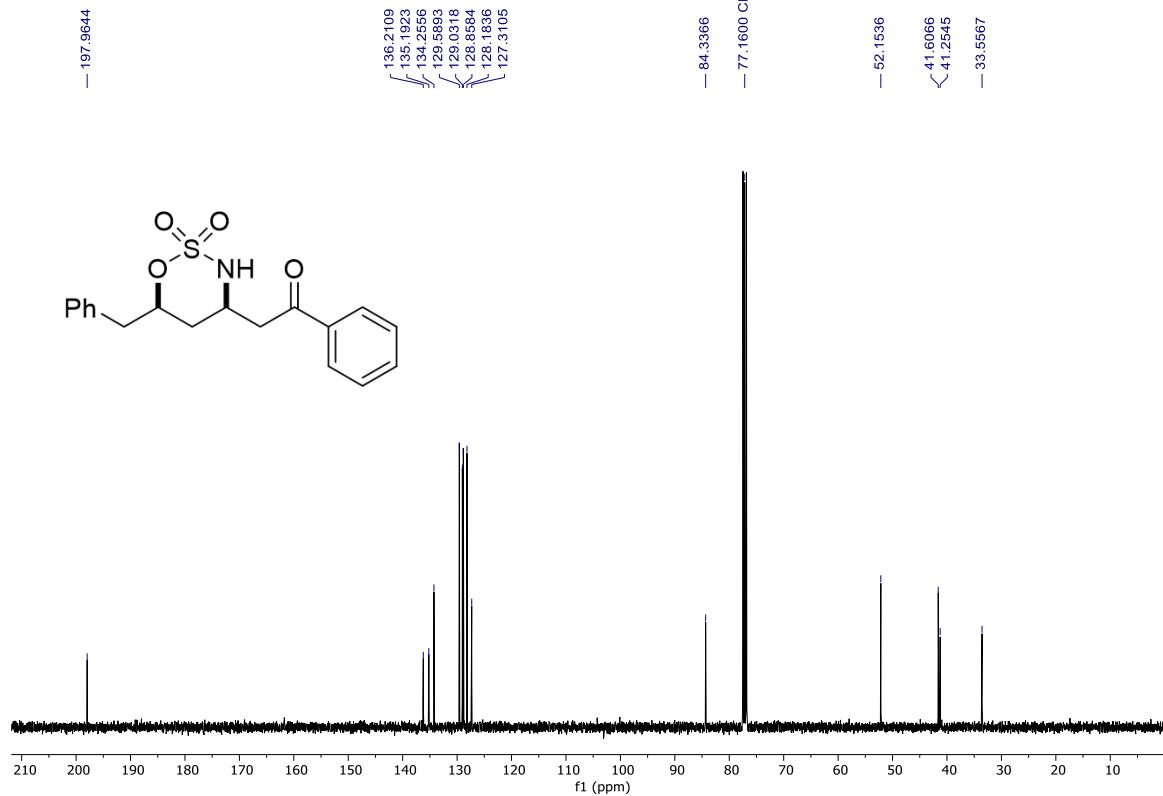
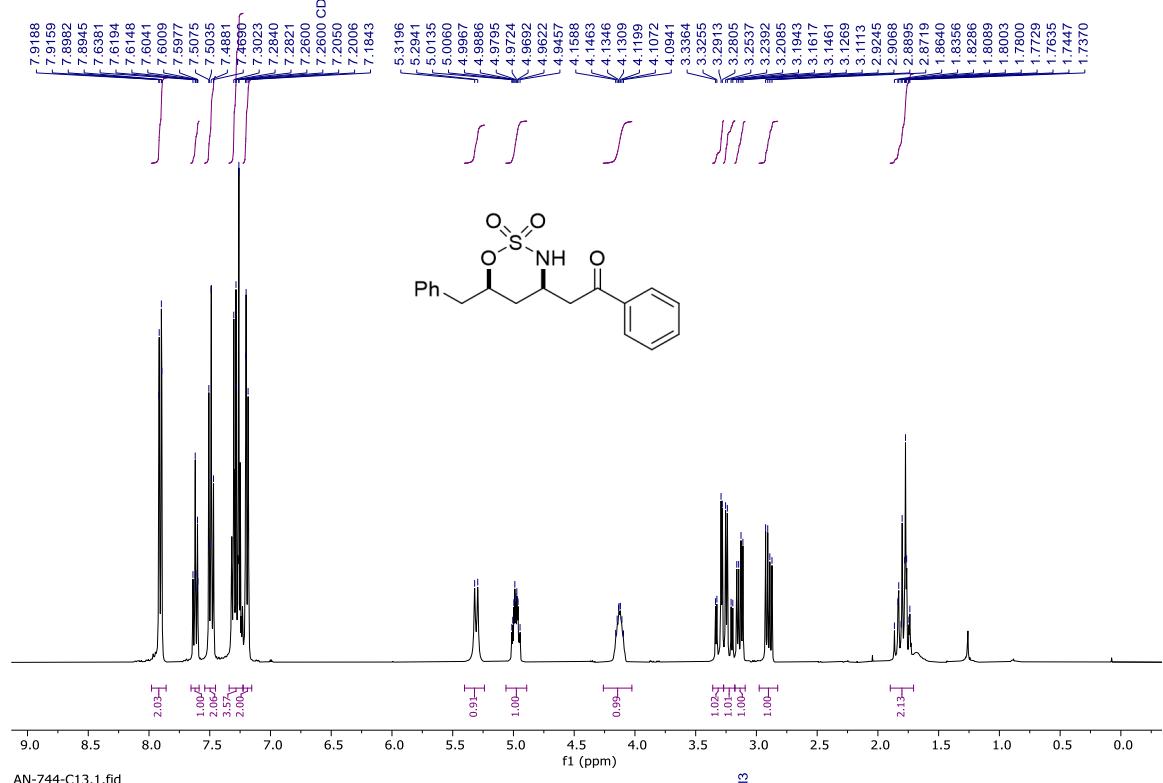


AN-740-C13.1.fid



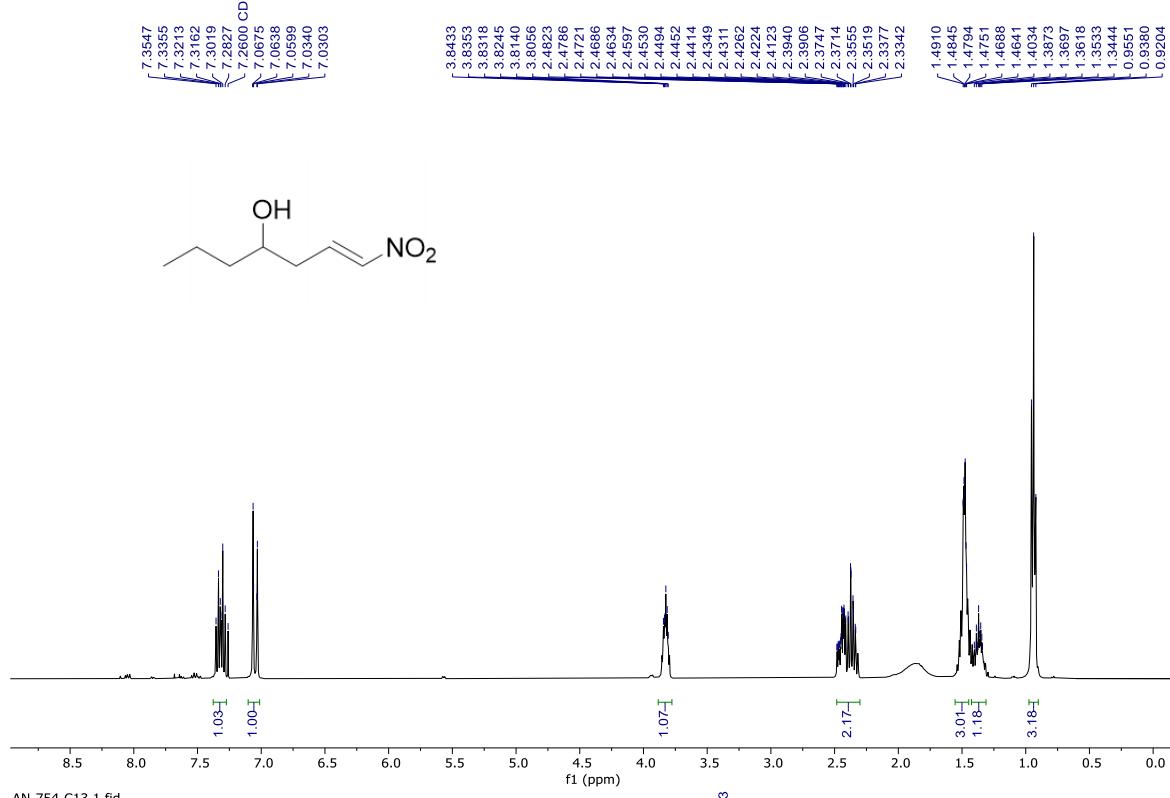
Compound 41 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-744-H1.1.fid

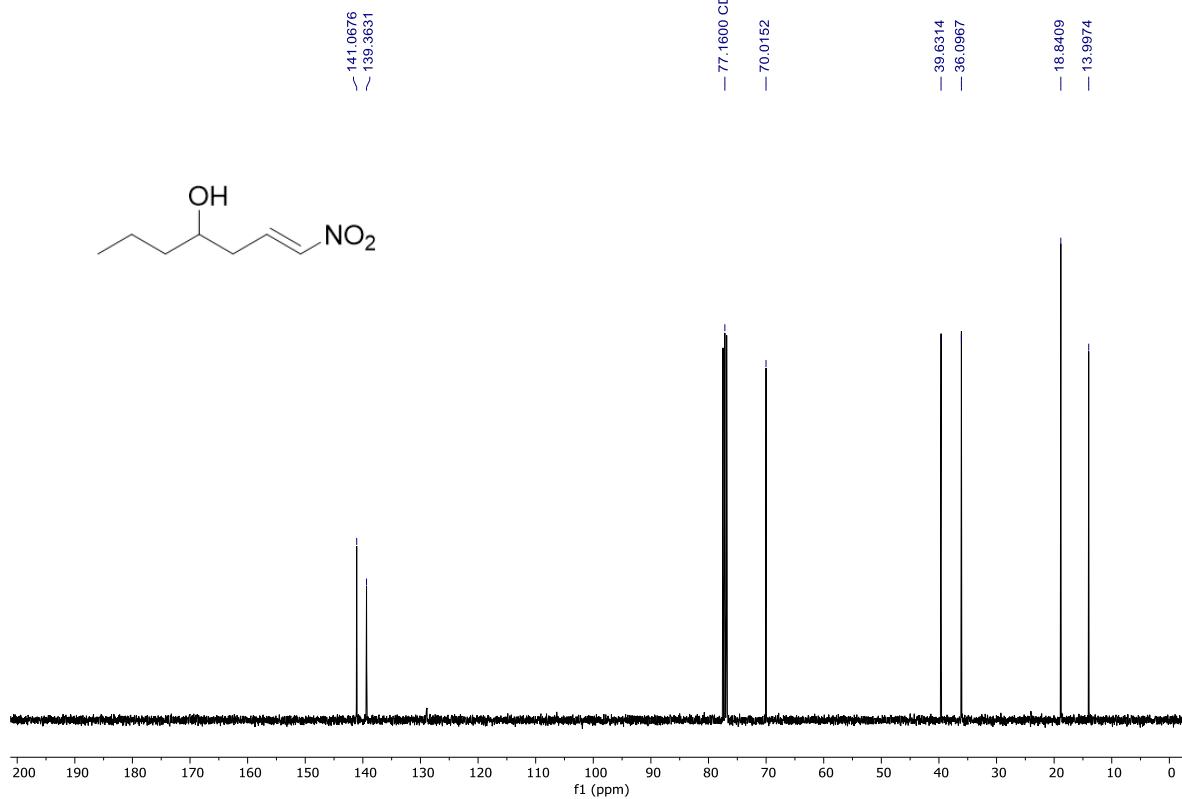


Compound 42 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

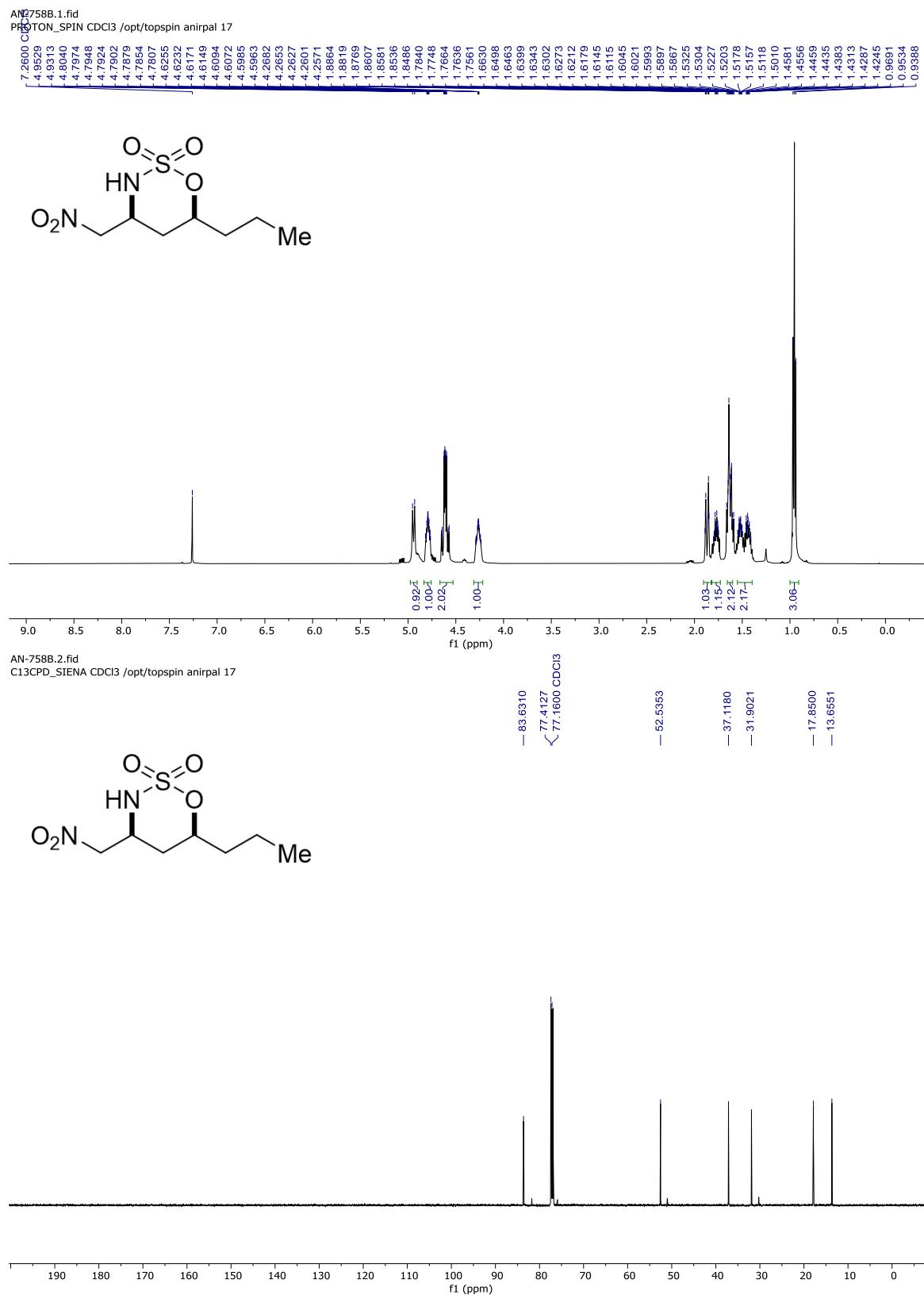
AN-754-H1.1.fid



AN-754-C13.1.fid

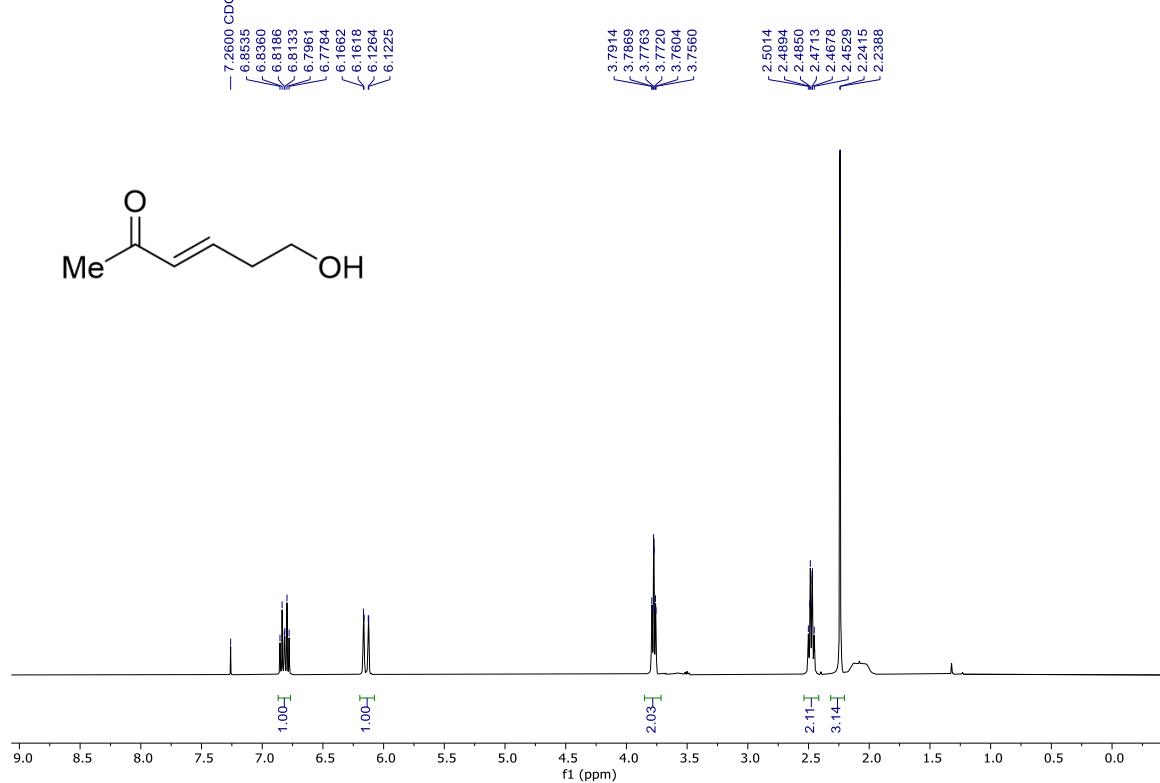


Compound 43 (CDCl₃, ¹H NMR: 500 MHz, ¹³C{¹H} NMR: 126 MHz)

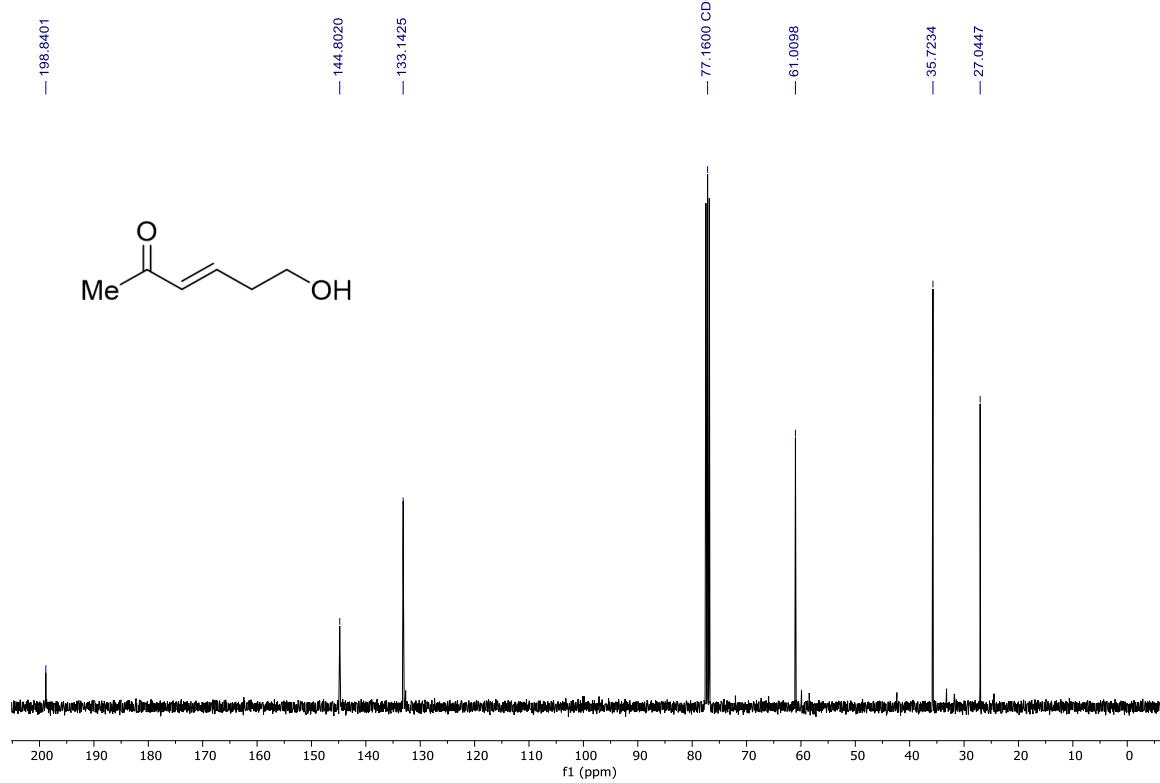


Compound 44 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-757-H1.1.fid

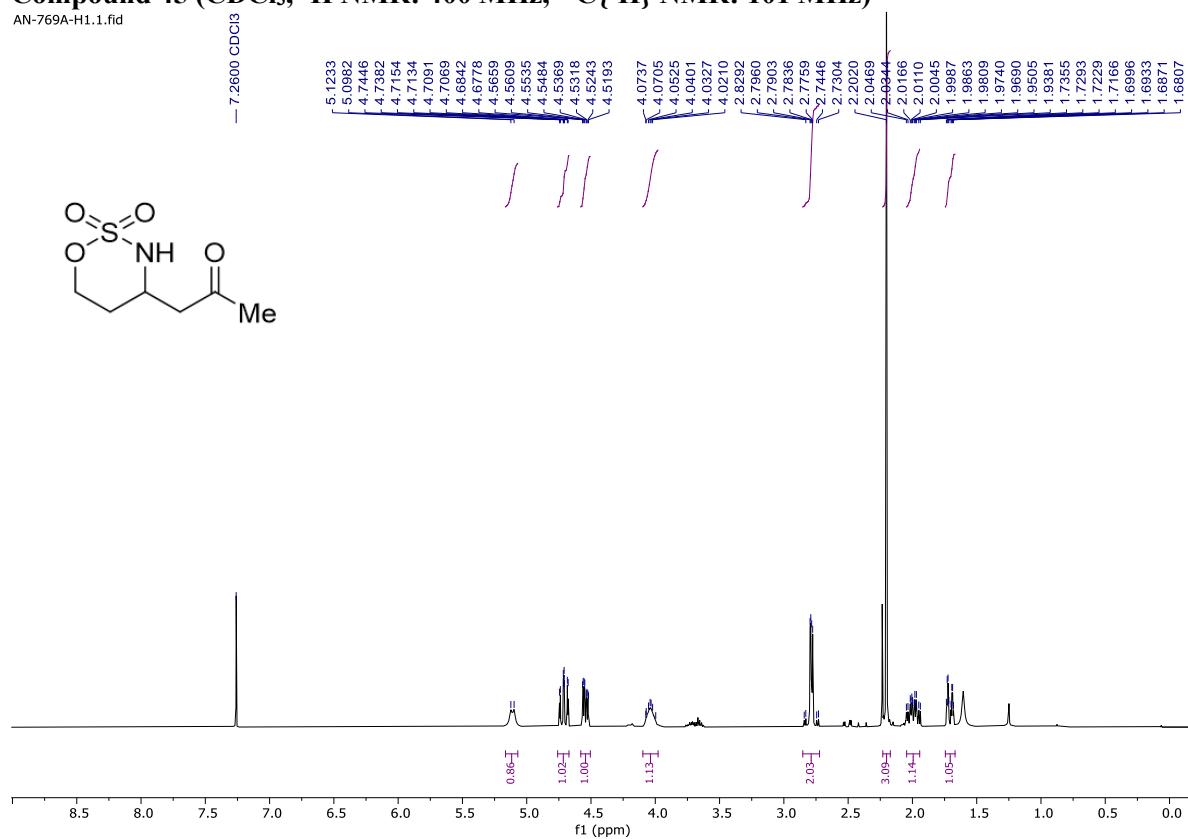


AN-757-C13.1.fid



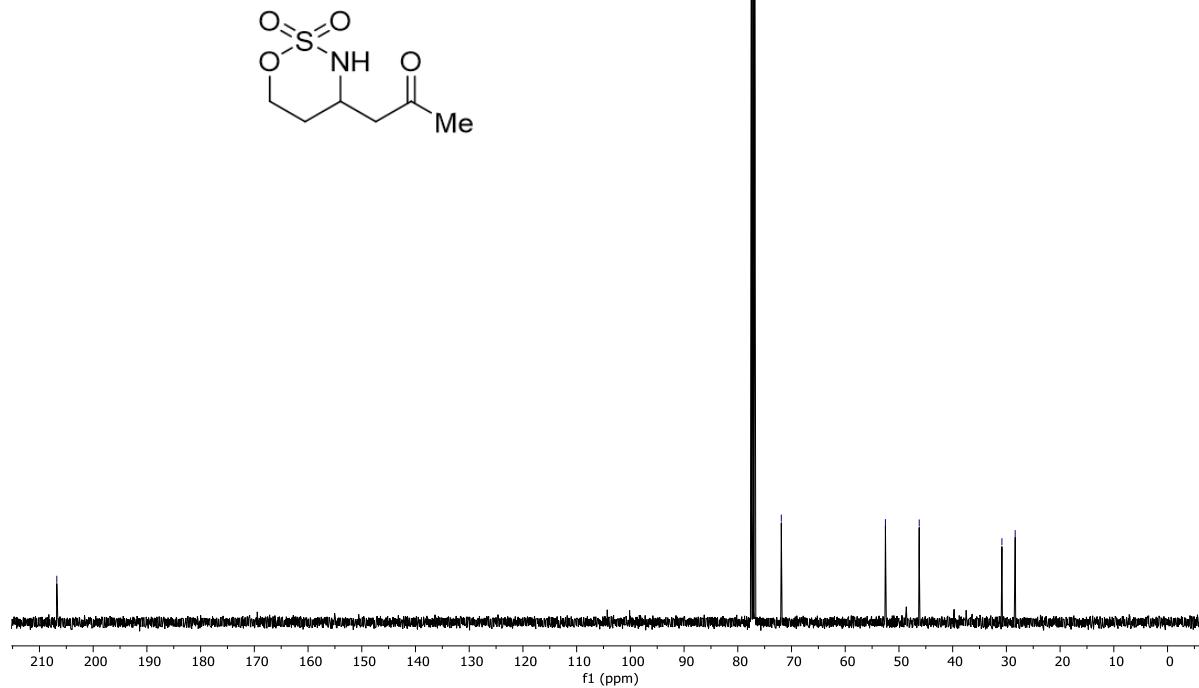
Compound 45 (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-769A-H1.1.fid



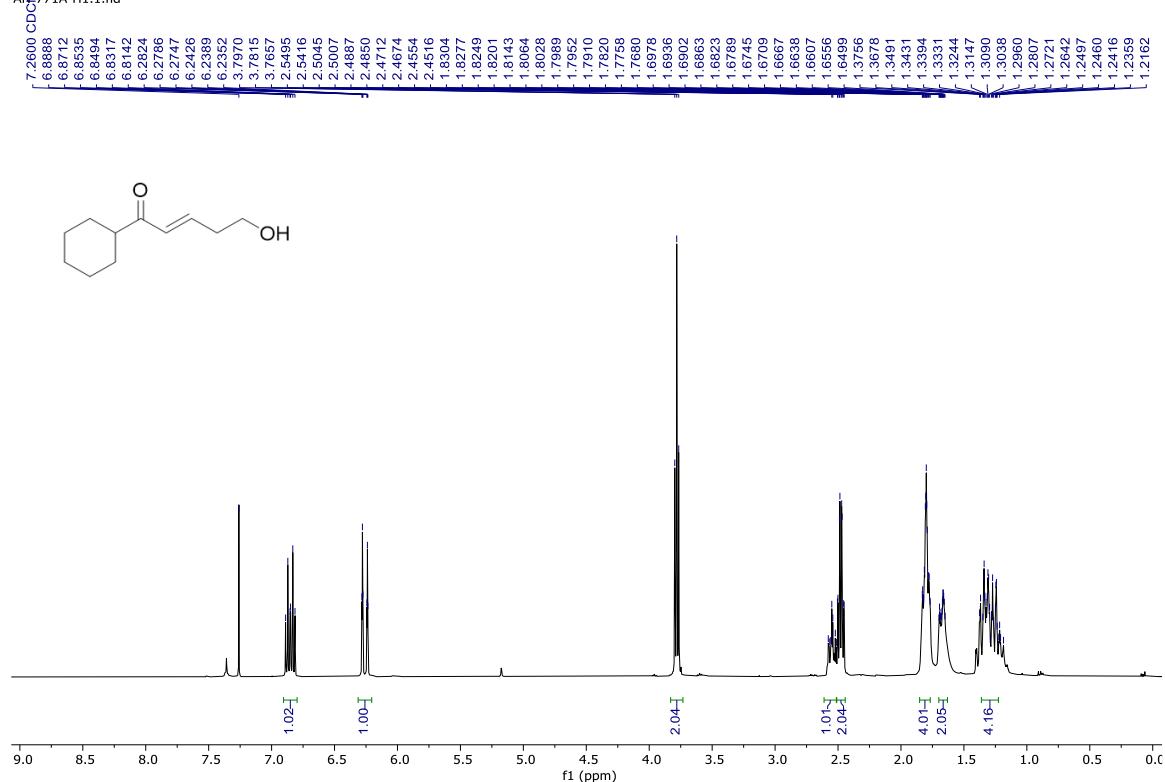
AN-769A-C13.1.fid

— 206.7706

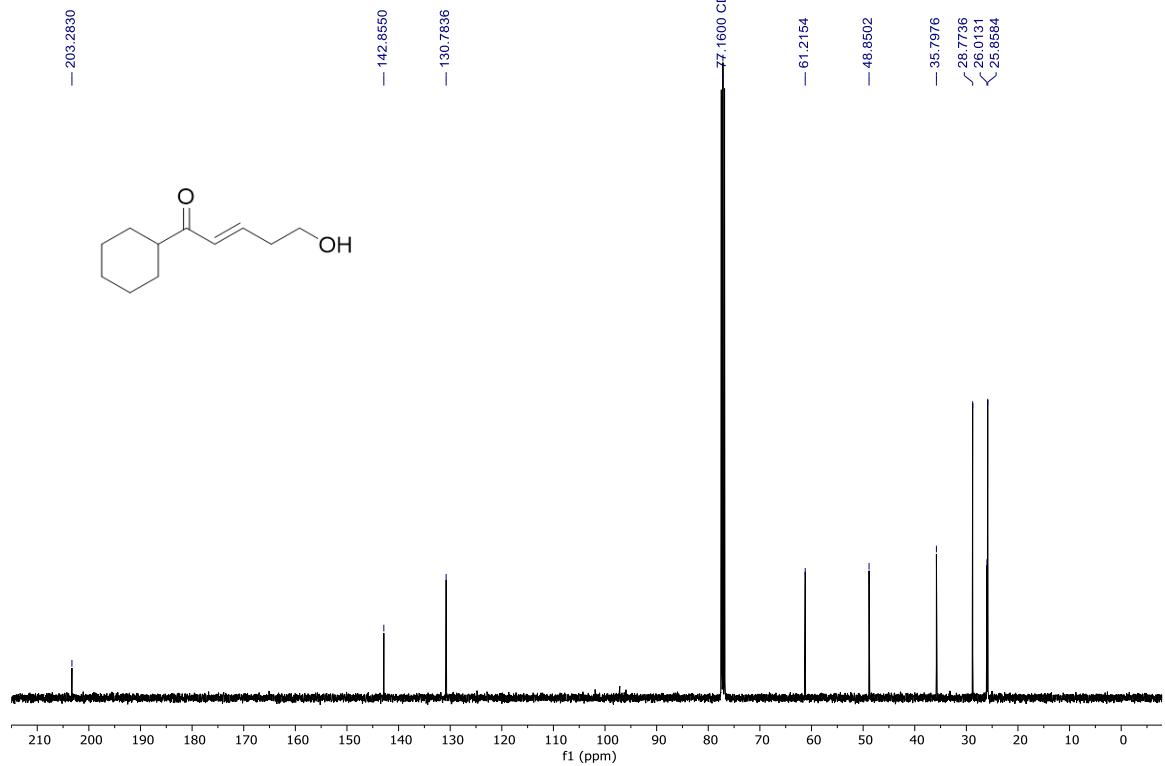


Compound 46 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-771A-H1.1.fid

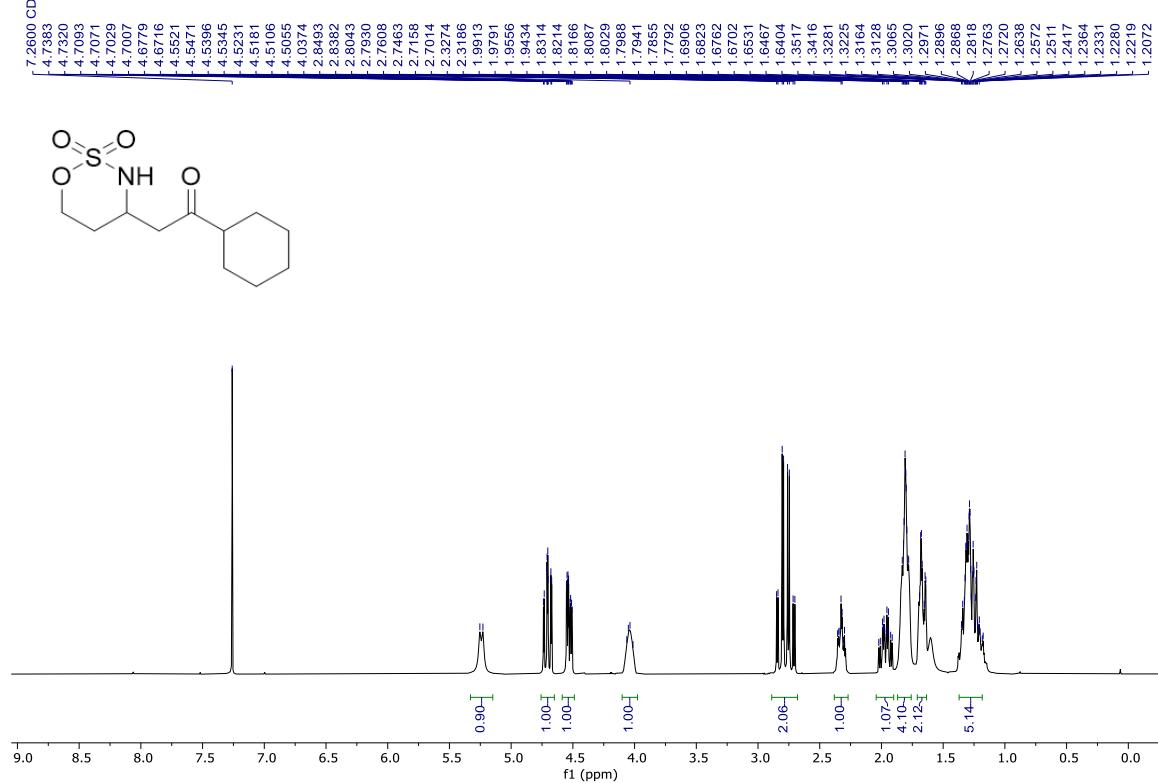


AN-771A-C13.1.fid



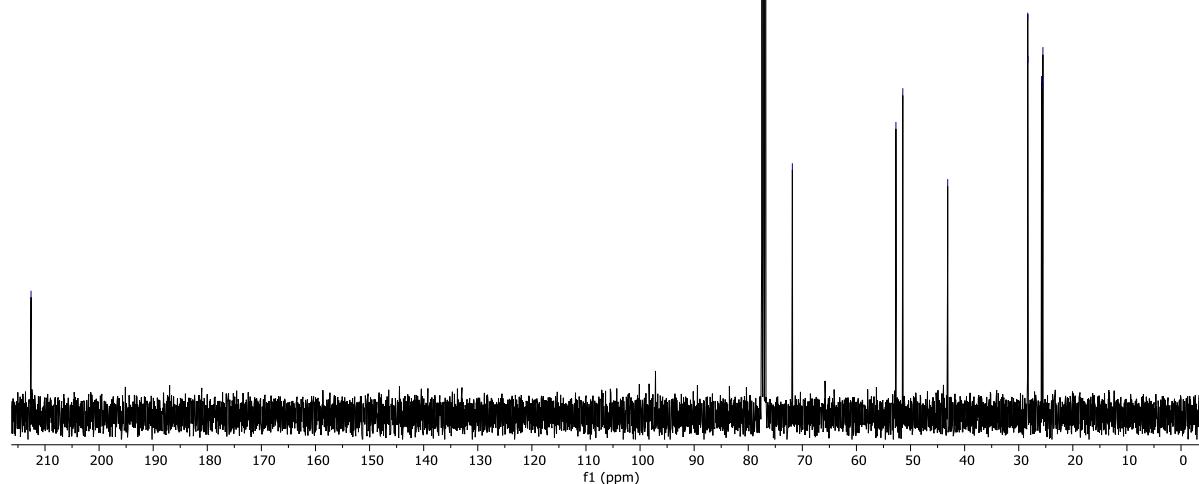
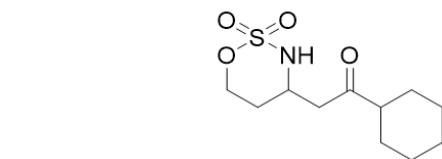
Compound 47 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-773-H1.1.fid



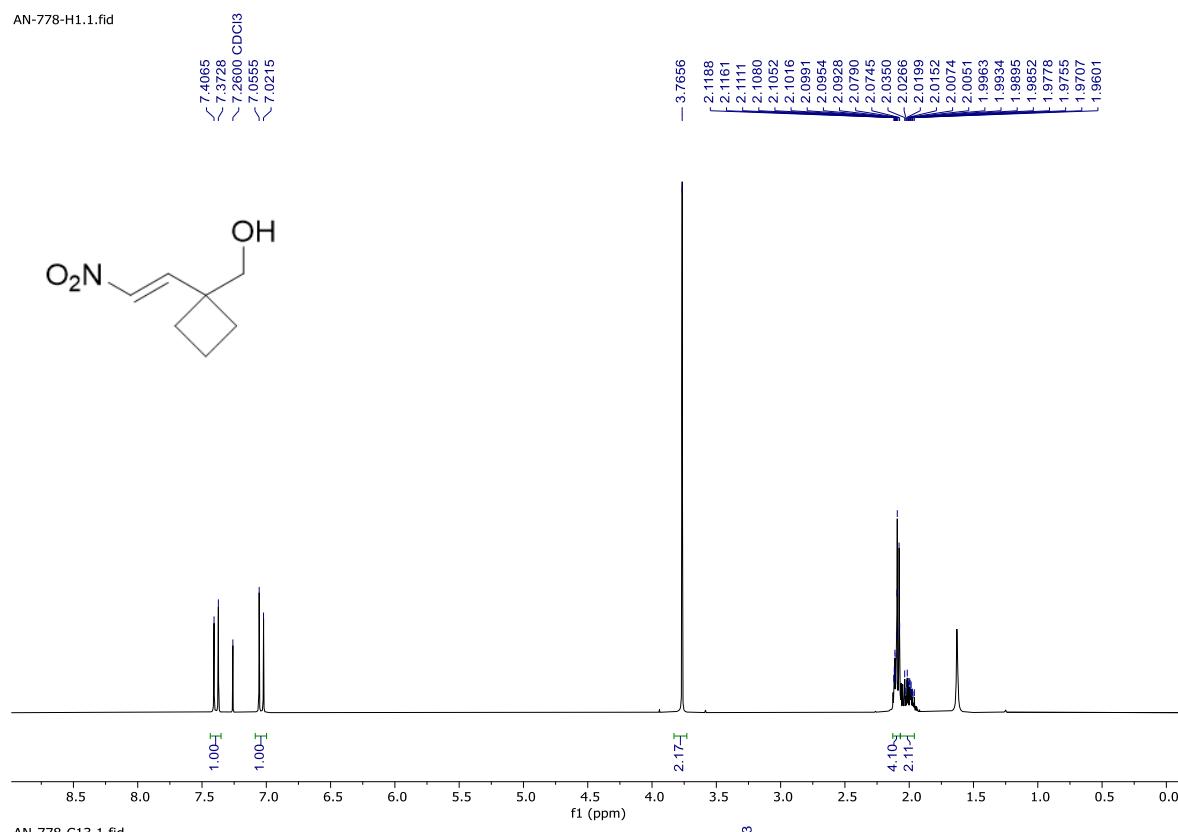
AN-773-C13.1.fid

— 212.5805

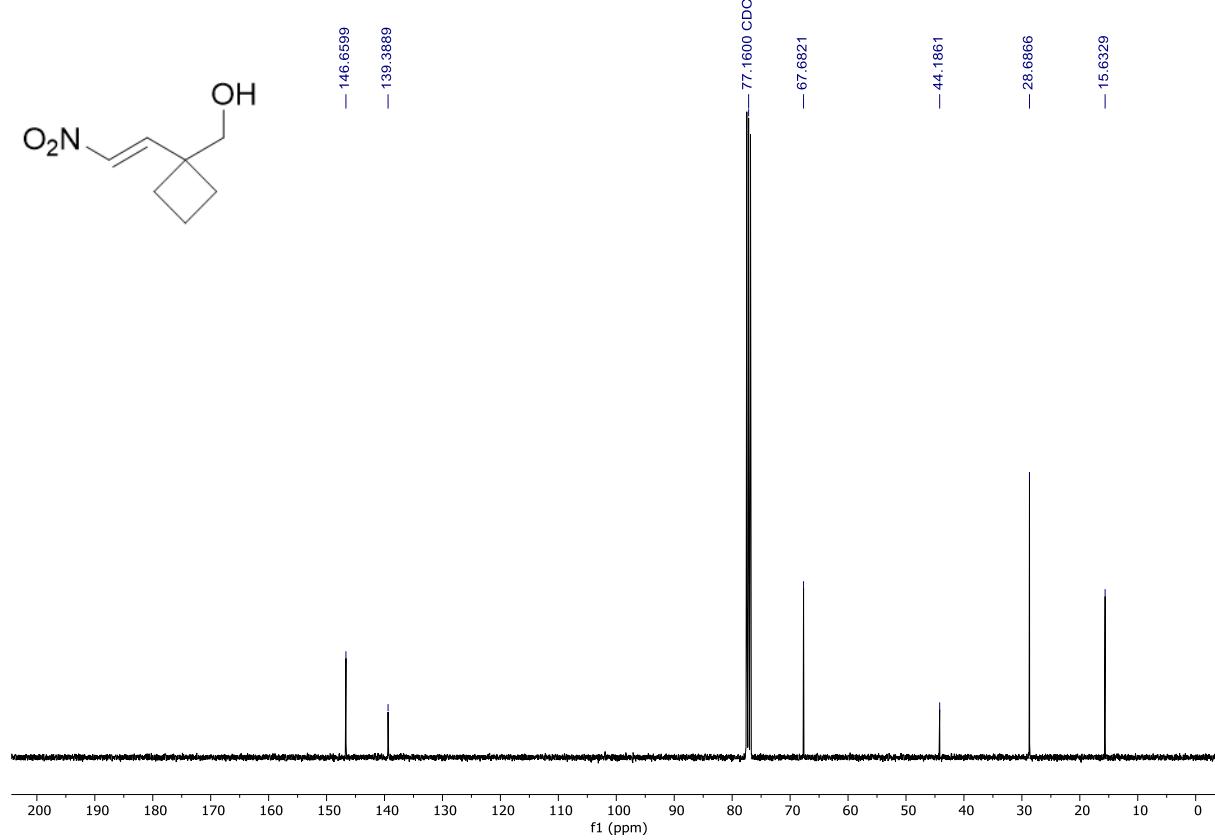


Compound 48 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-778-H1.1.fid

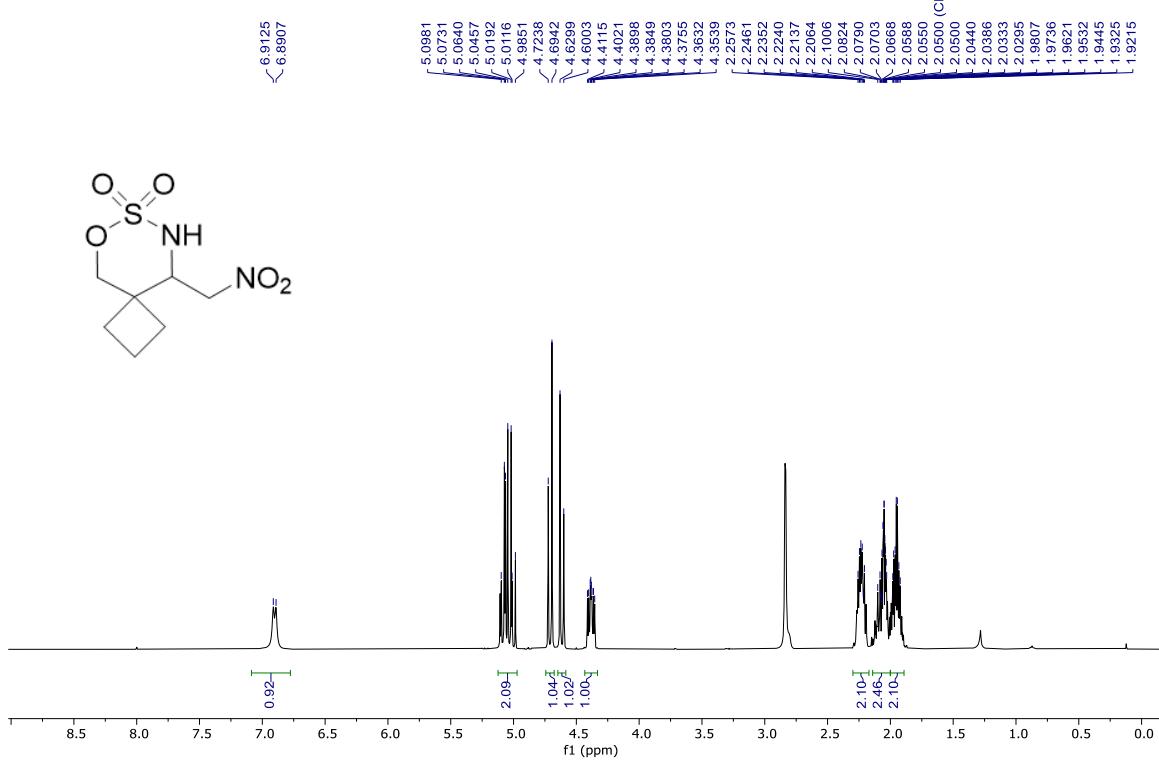


AN-778-C13.1.fid

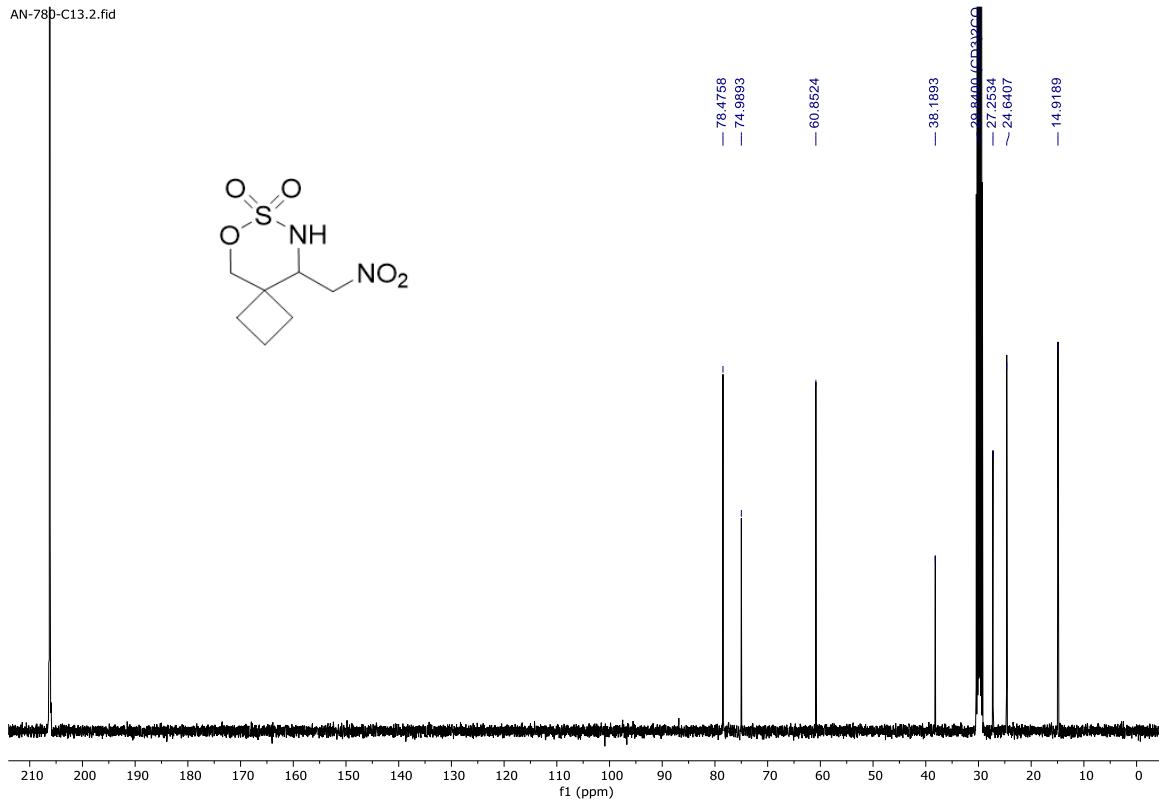


Compound 49 (Acetone-*d*₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-780-H1.2.fid

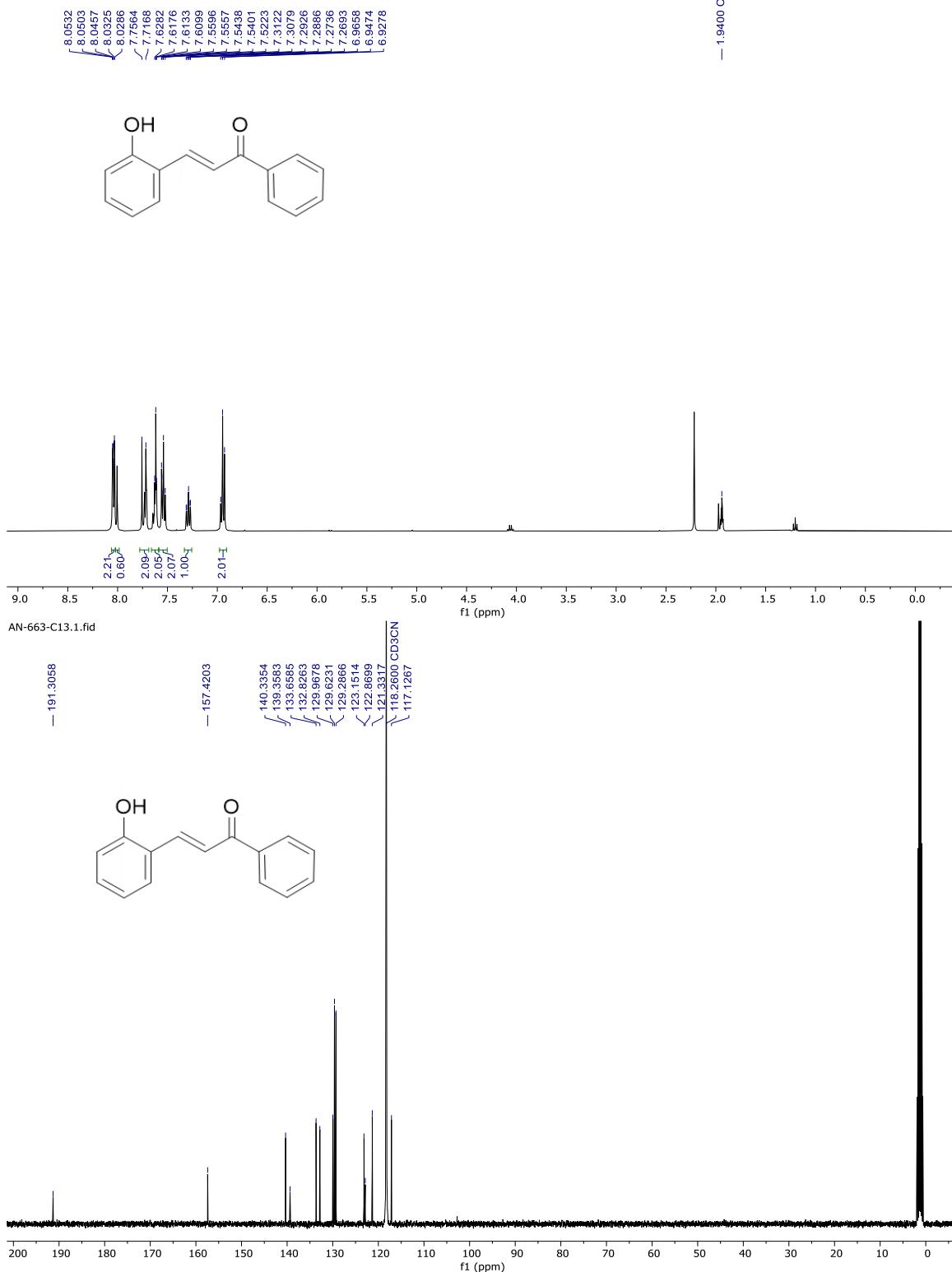


AN-780-C13.2.fid



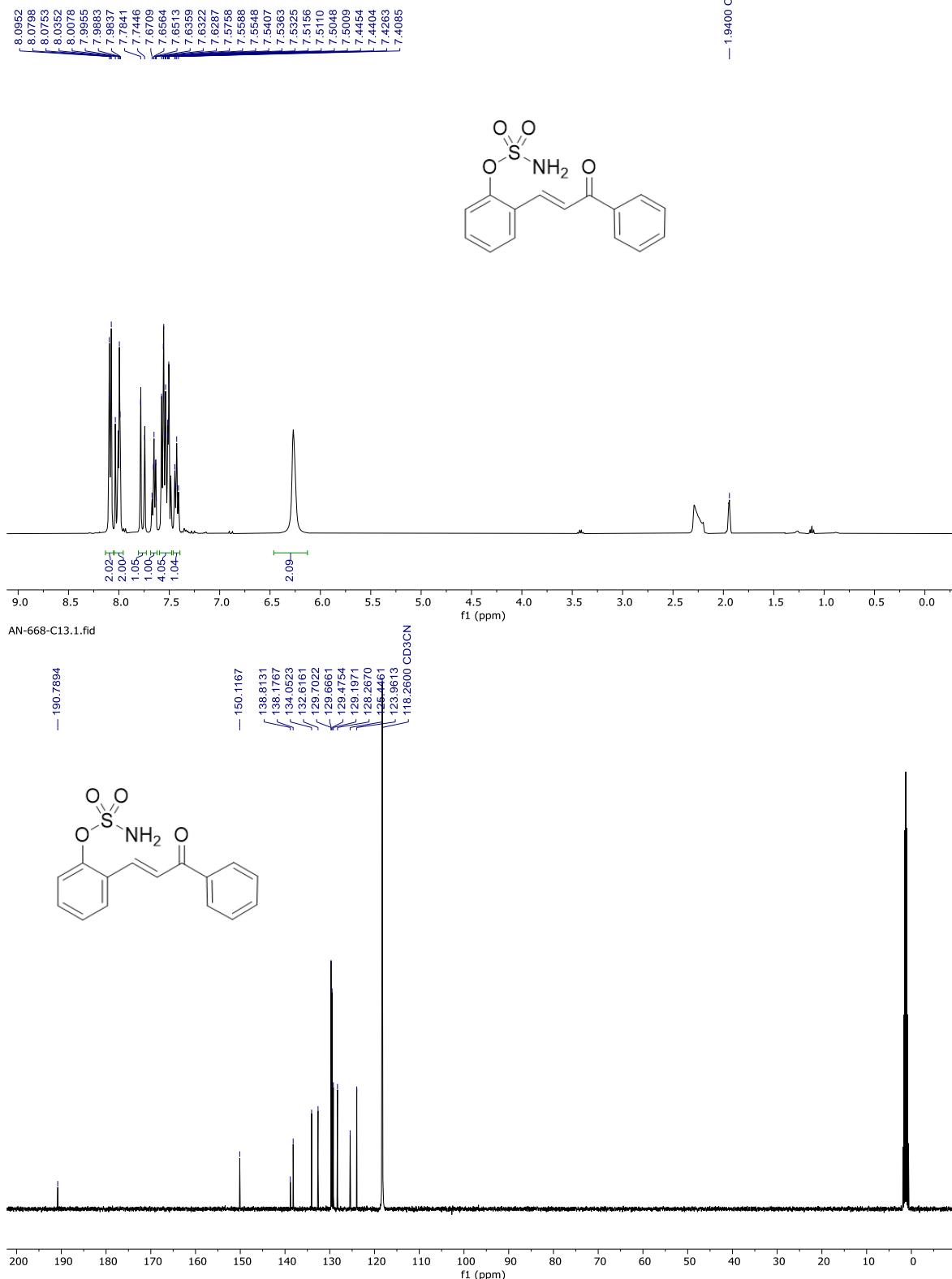
Compound S50' (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-663-H1.1.fid



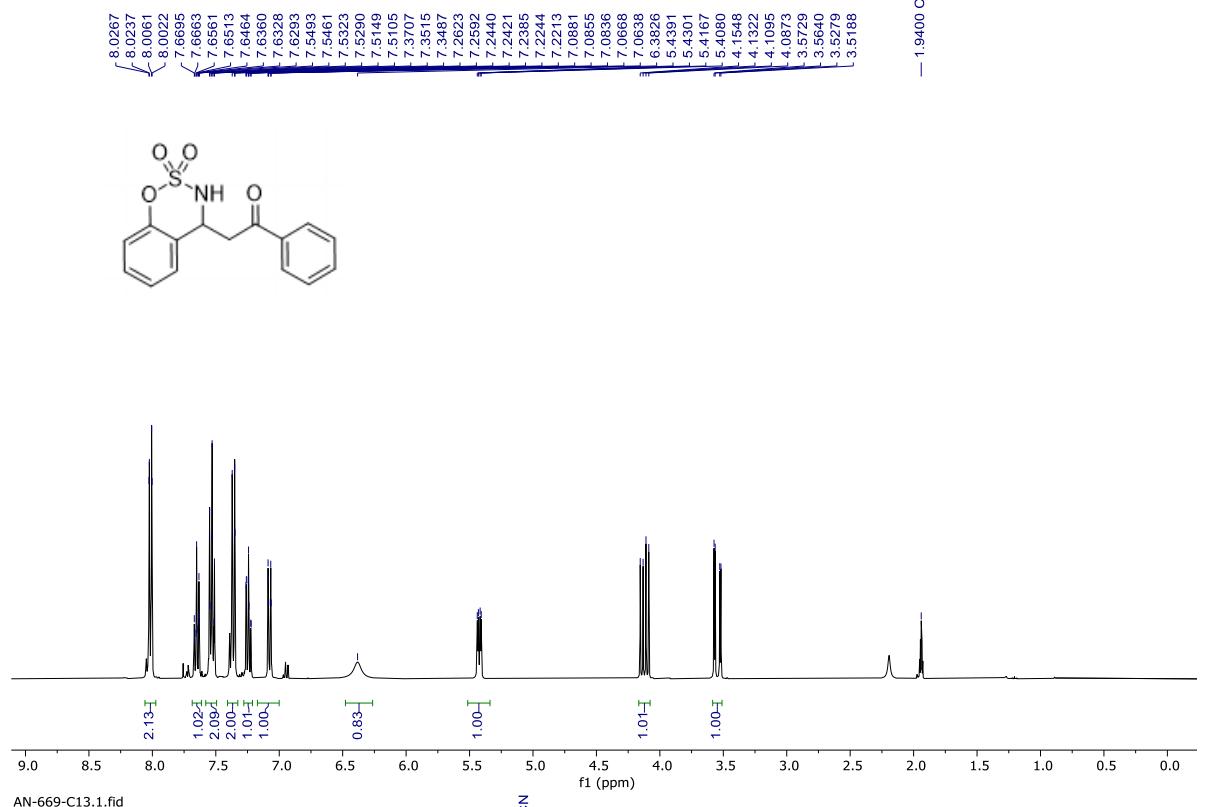
Compound S50 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-668-H1.1.fid

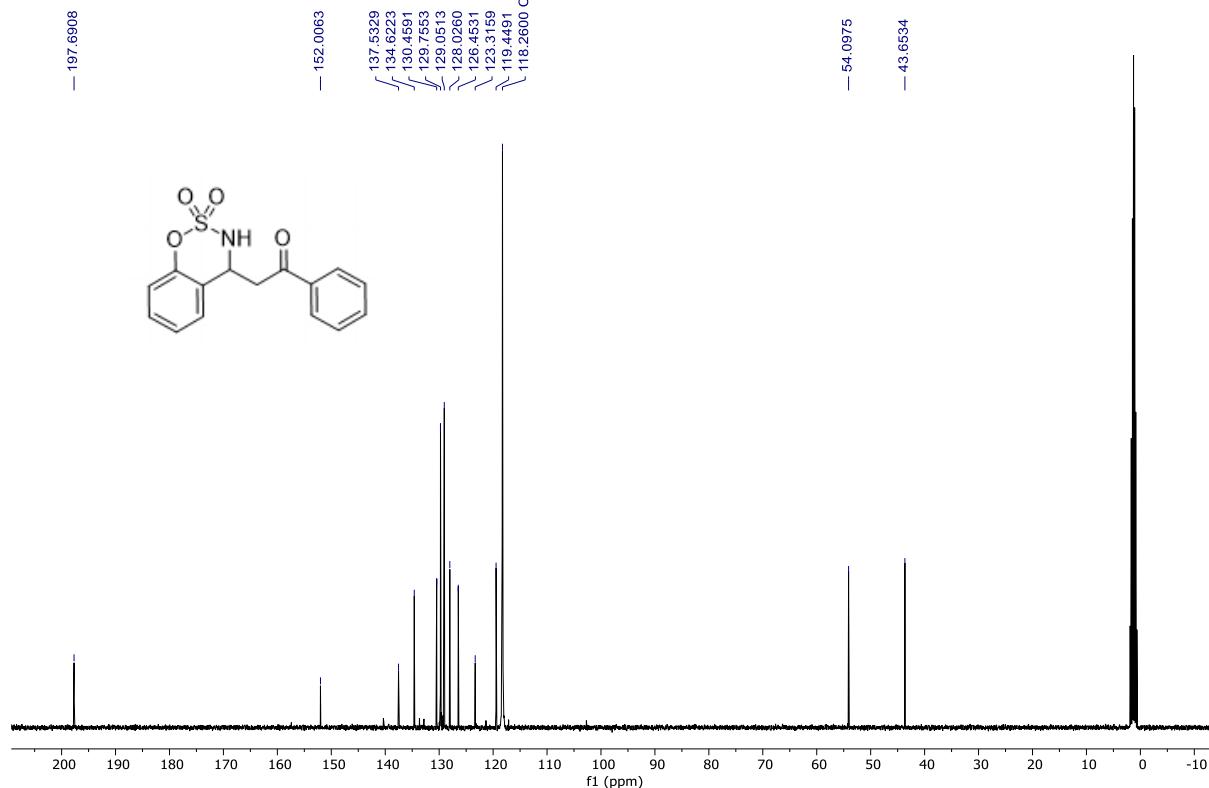


Compound 50 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-669-H1.1.fid

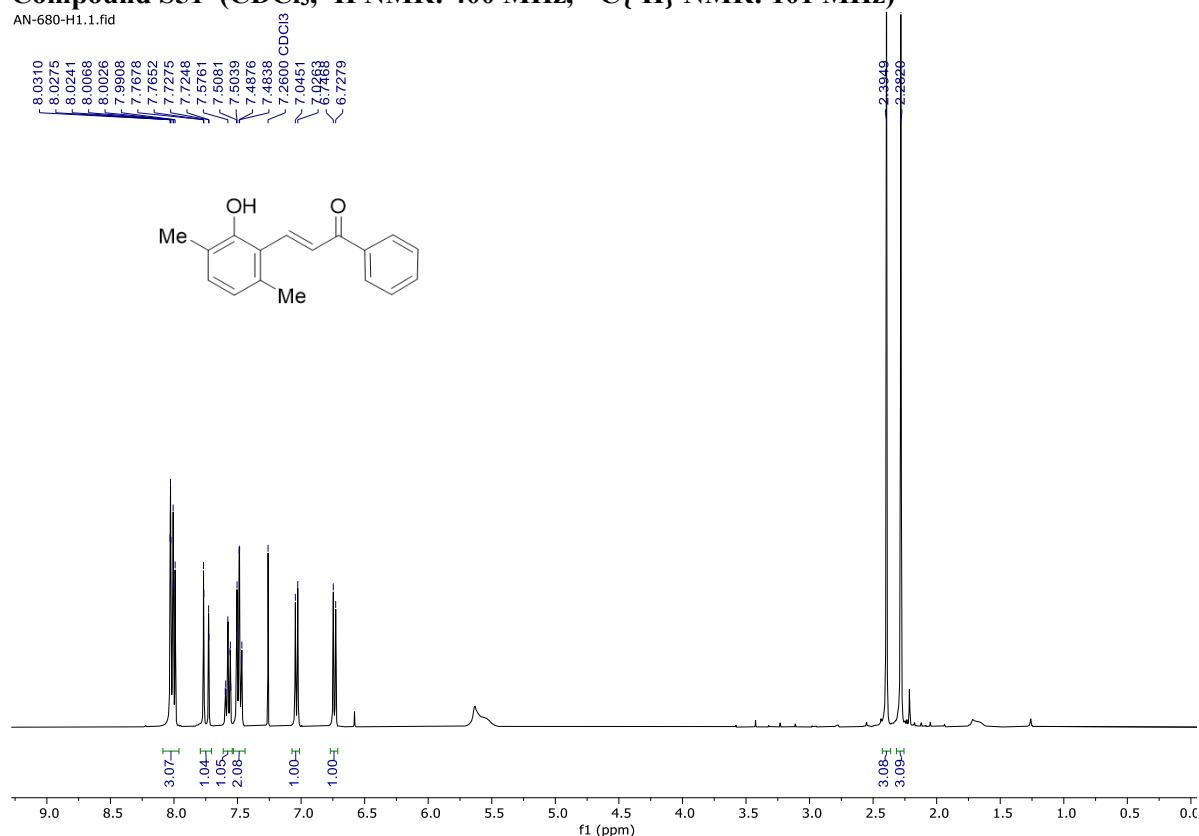


AN-669-C13.1.fid

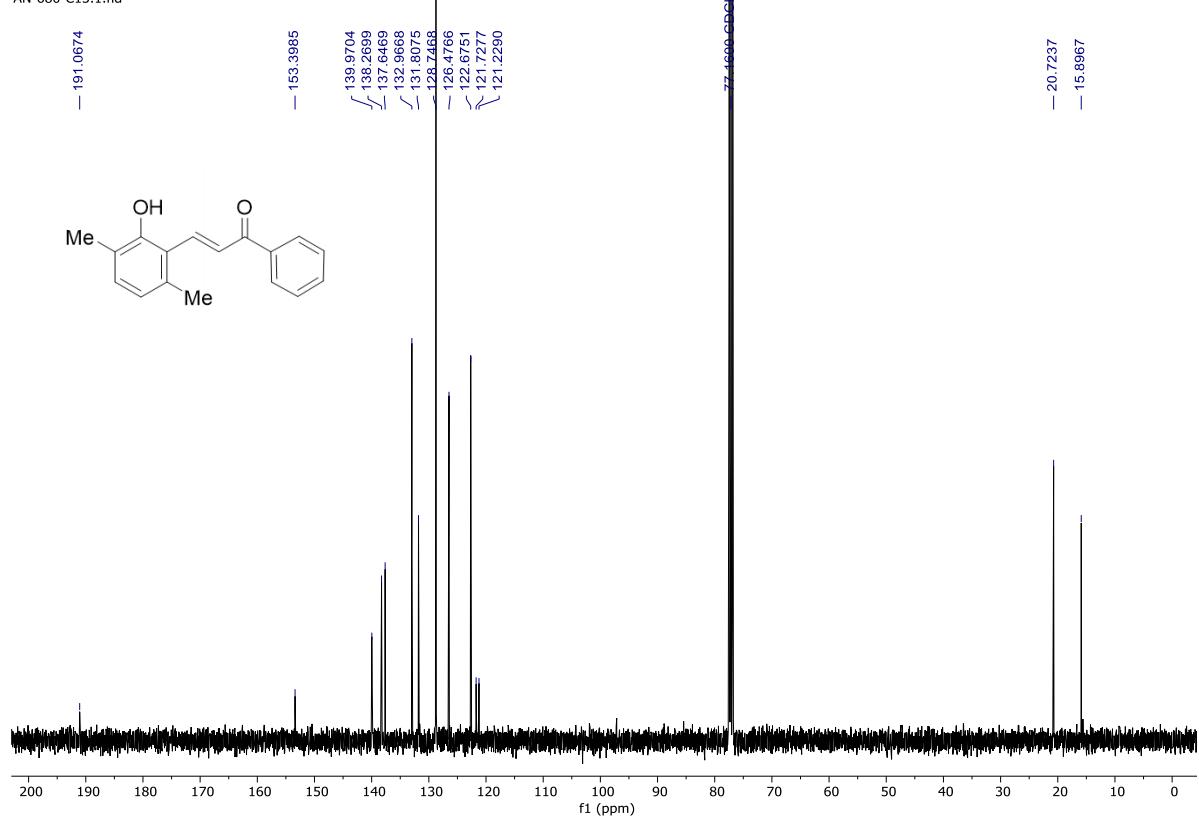


Compound S51' (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-680-H1.1.fid

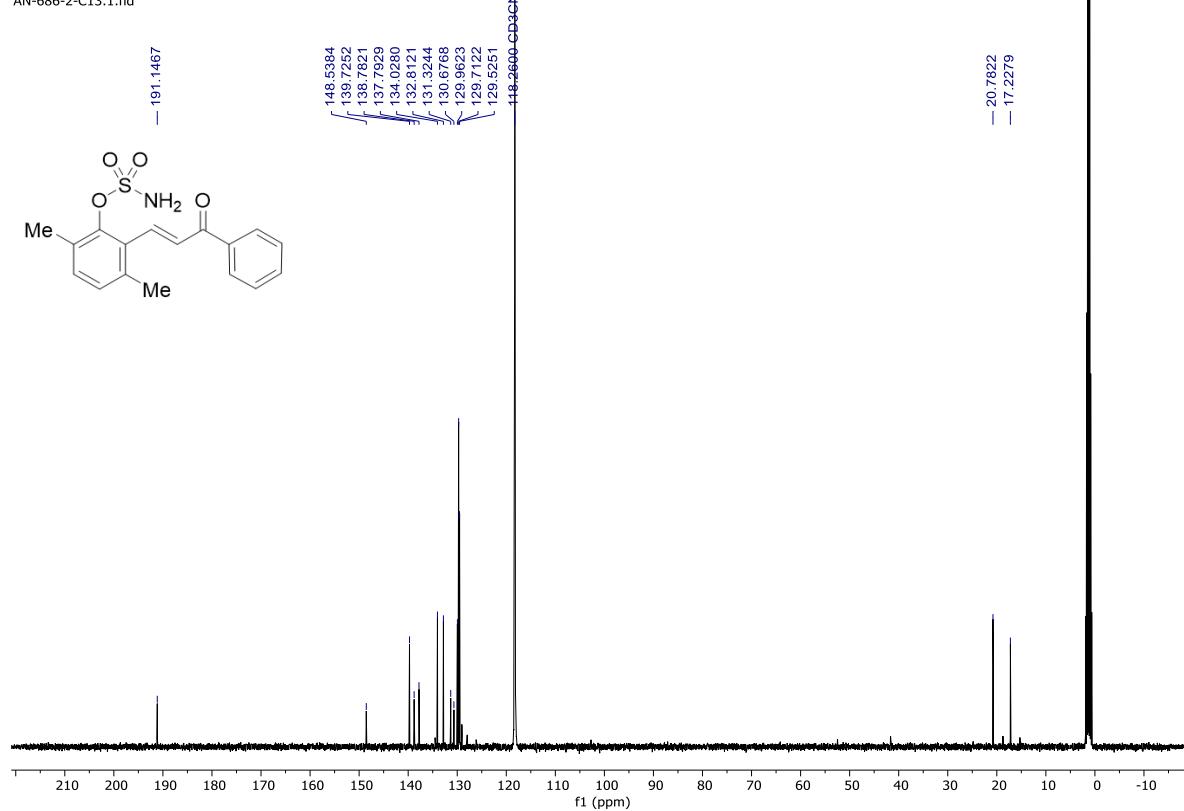
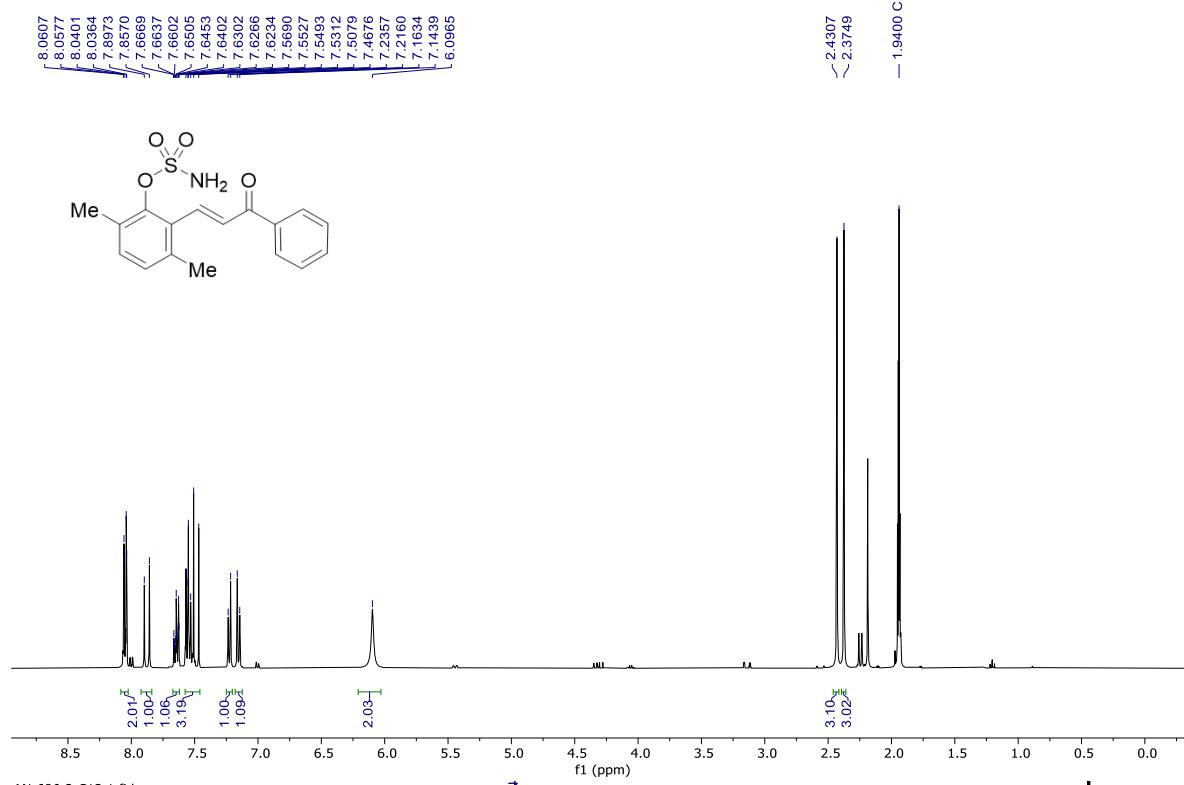


AN-680-C13.1.fid

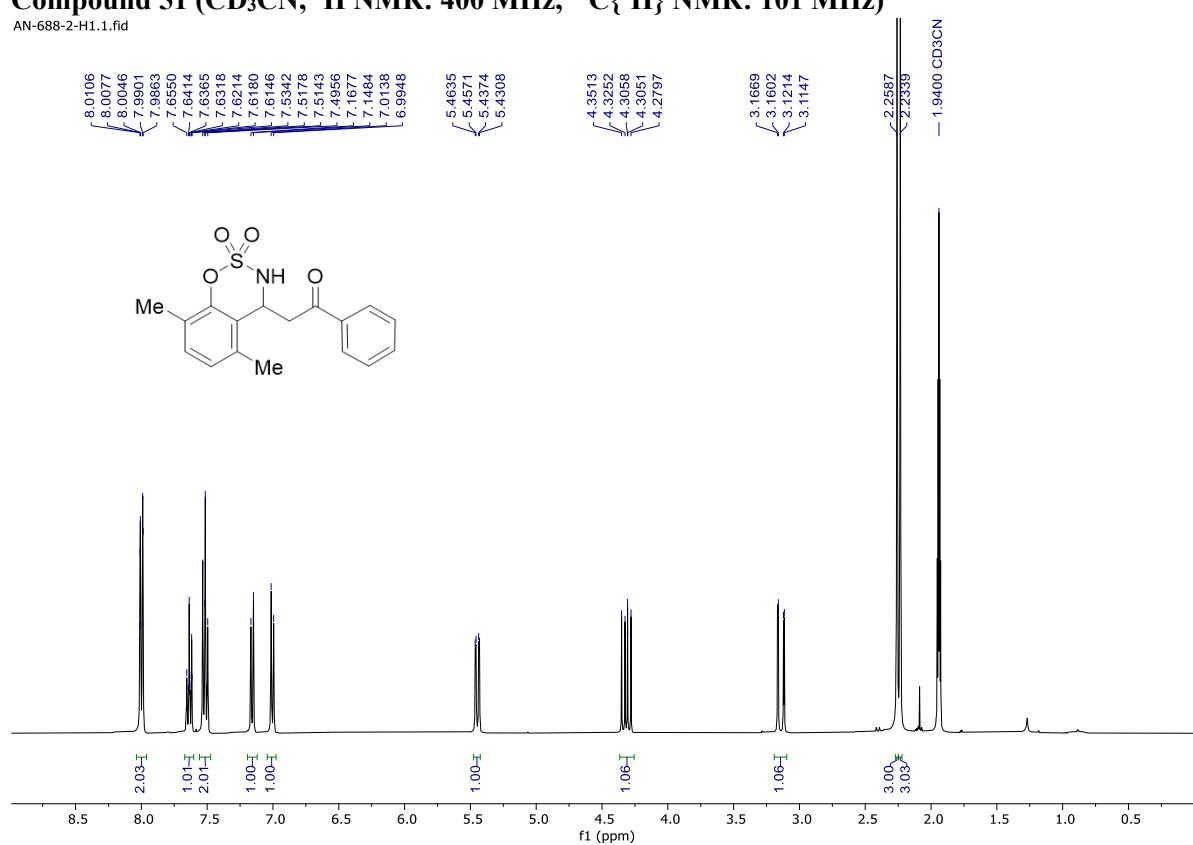


Compound S51 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

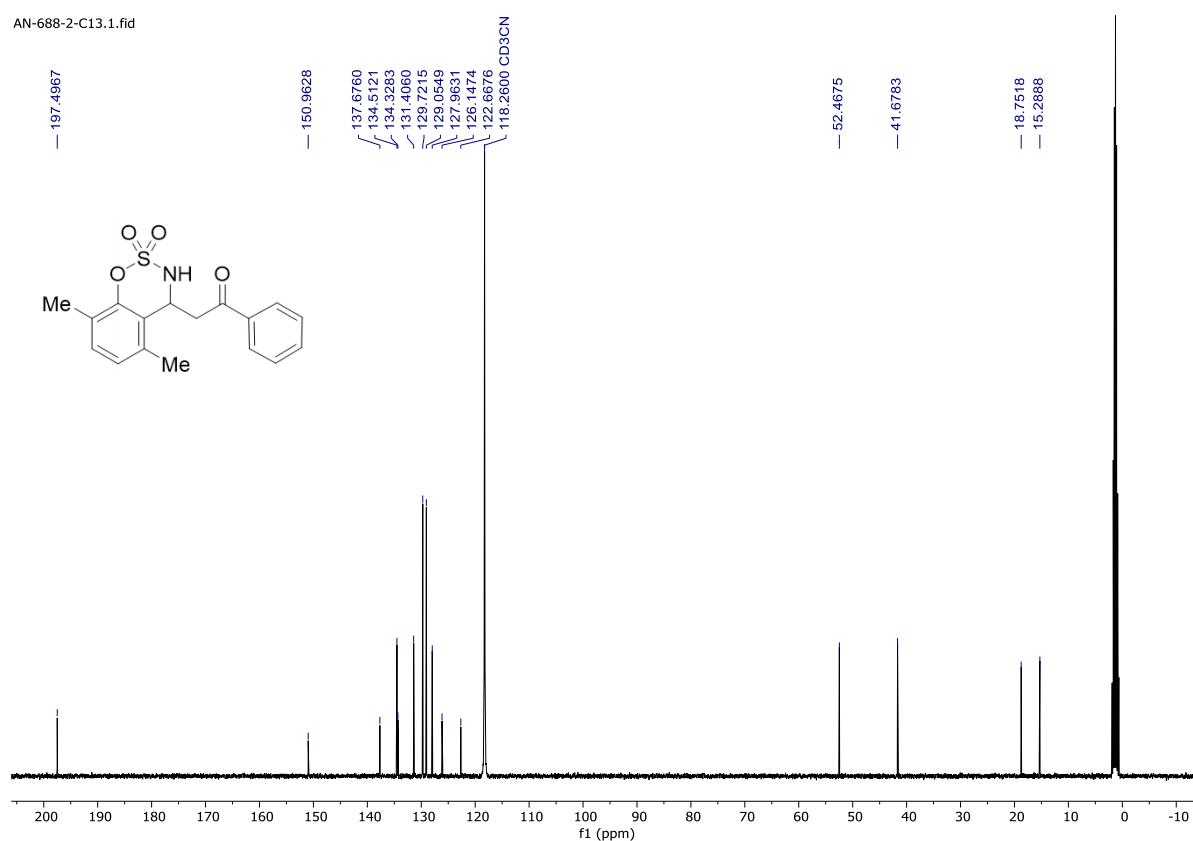
AN-686-2-H1.1.fid



Compound 51 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)
 AN-688-2-H1.1.fid

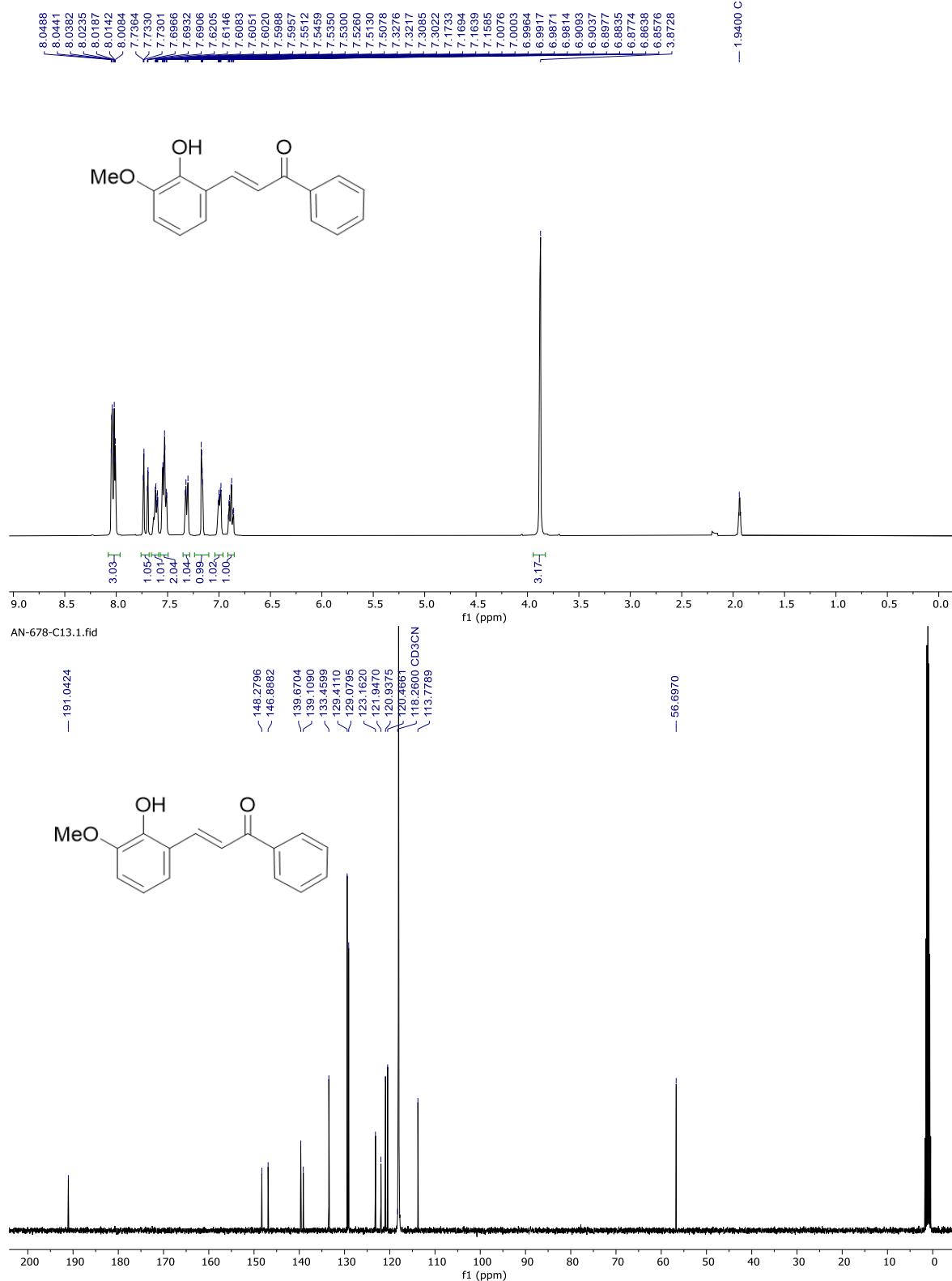


AN-688-2-C13.1.fid



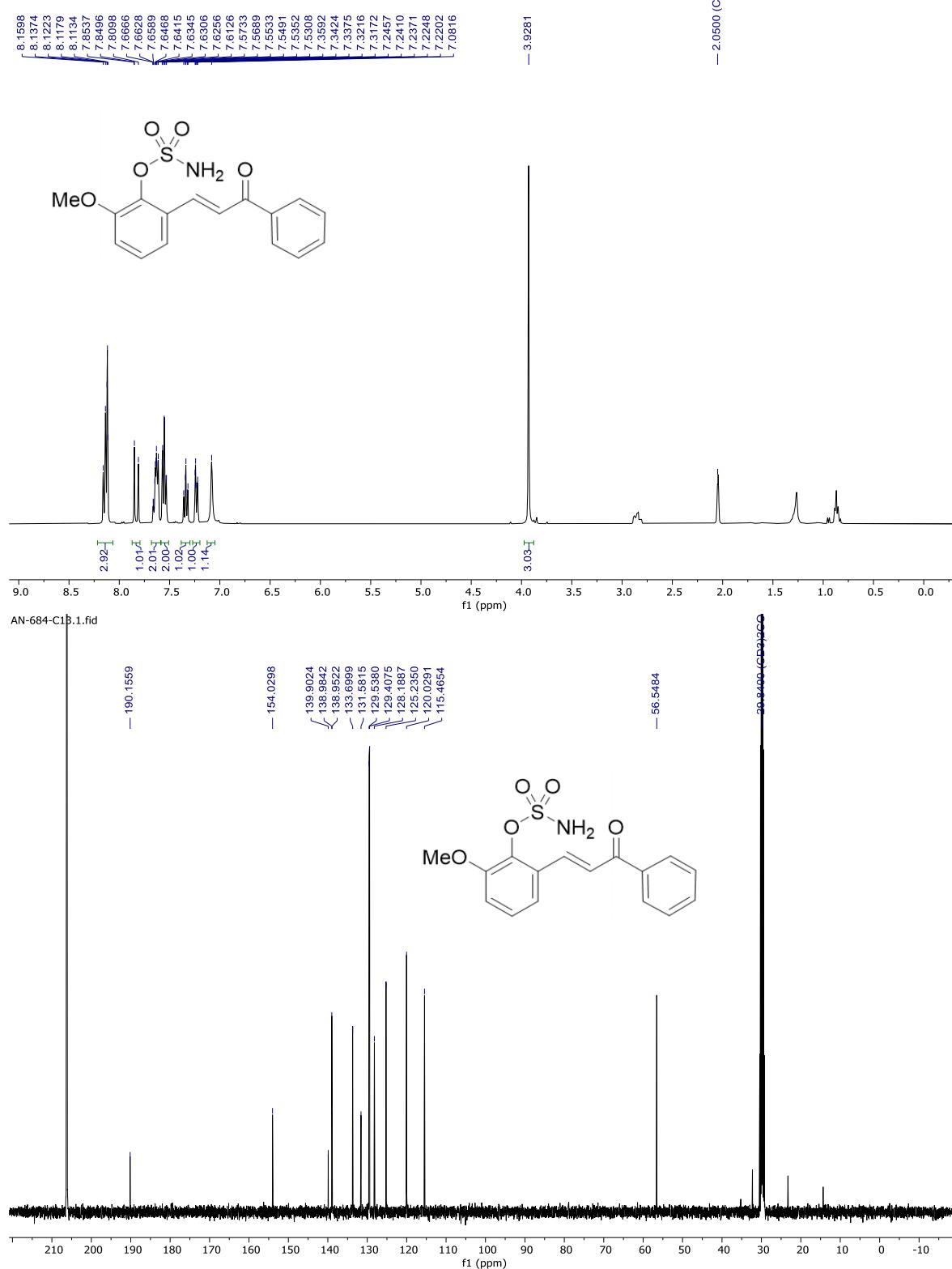
Compound S52' (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-678-H1.1.fid



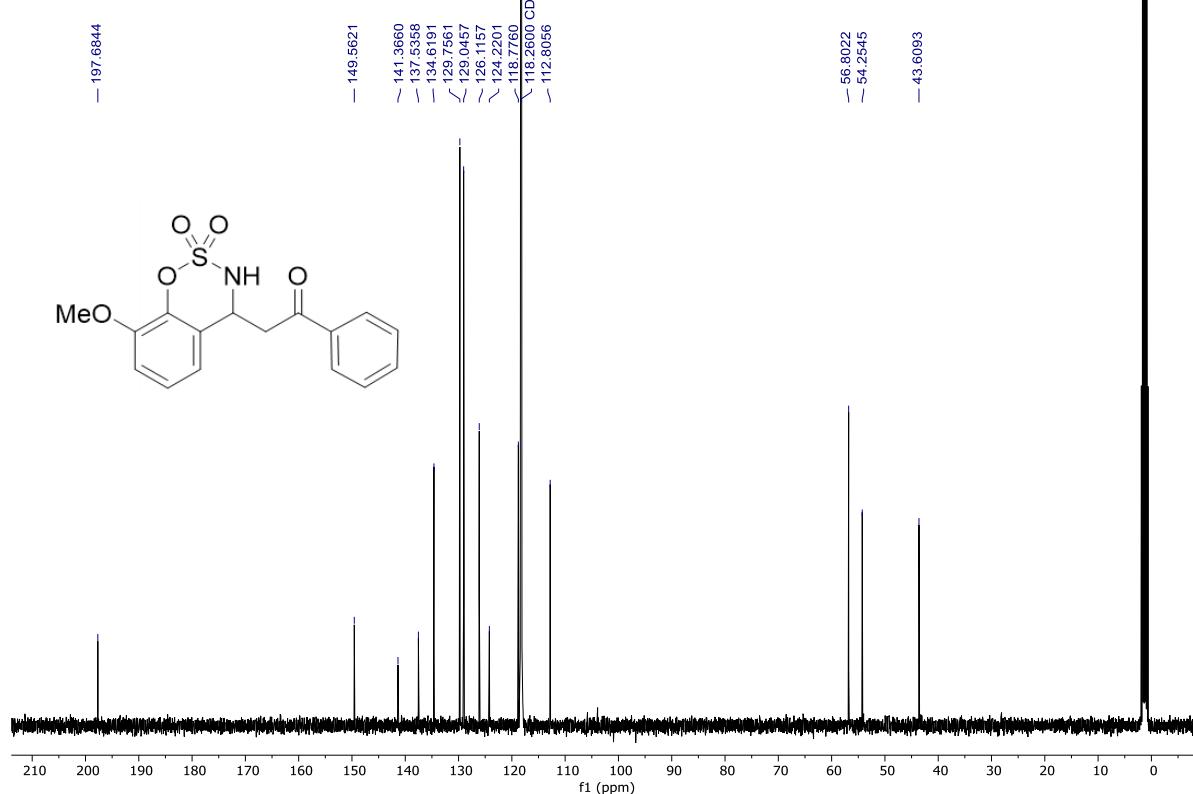
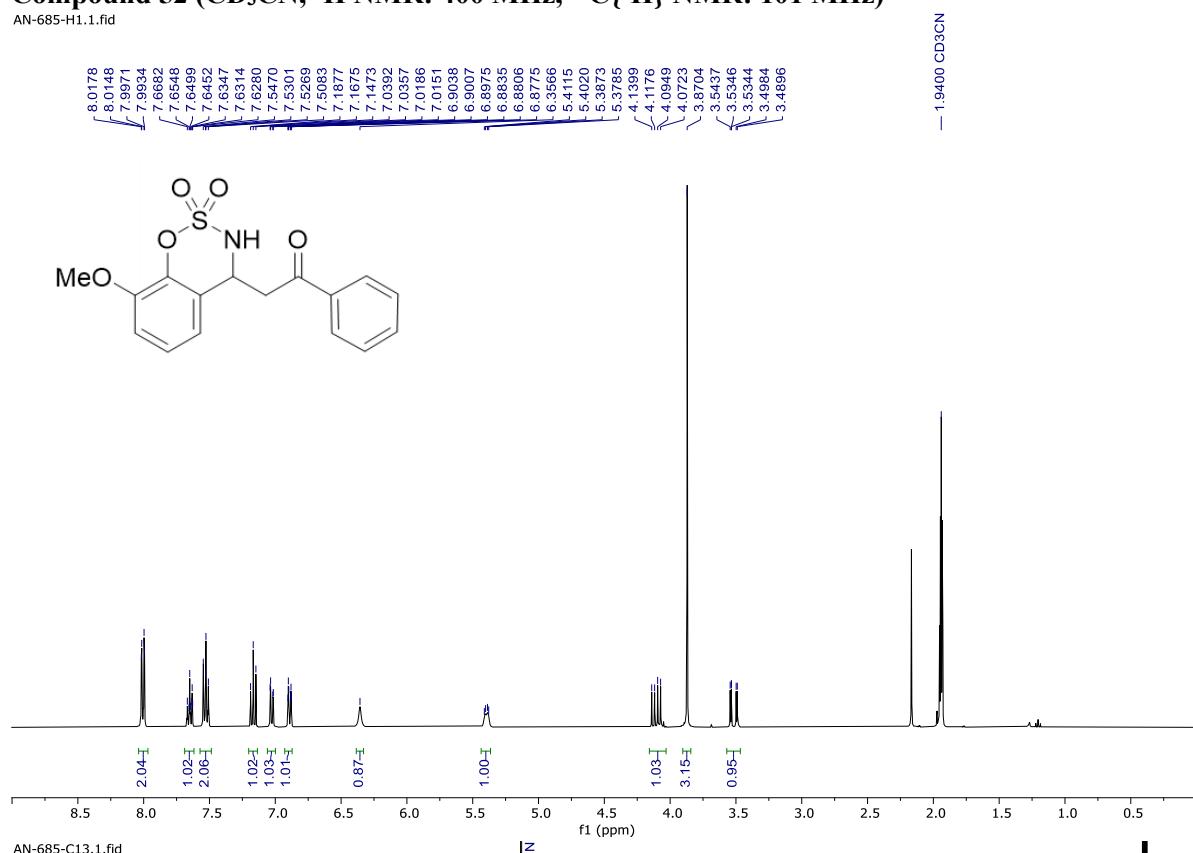
Compound S52 (Acetone-d₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-684-H1.1.fid



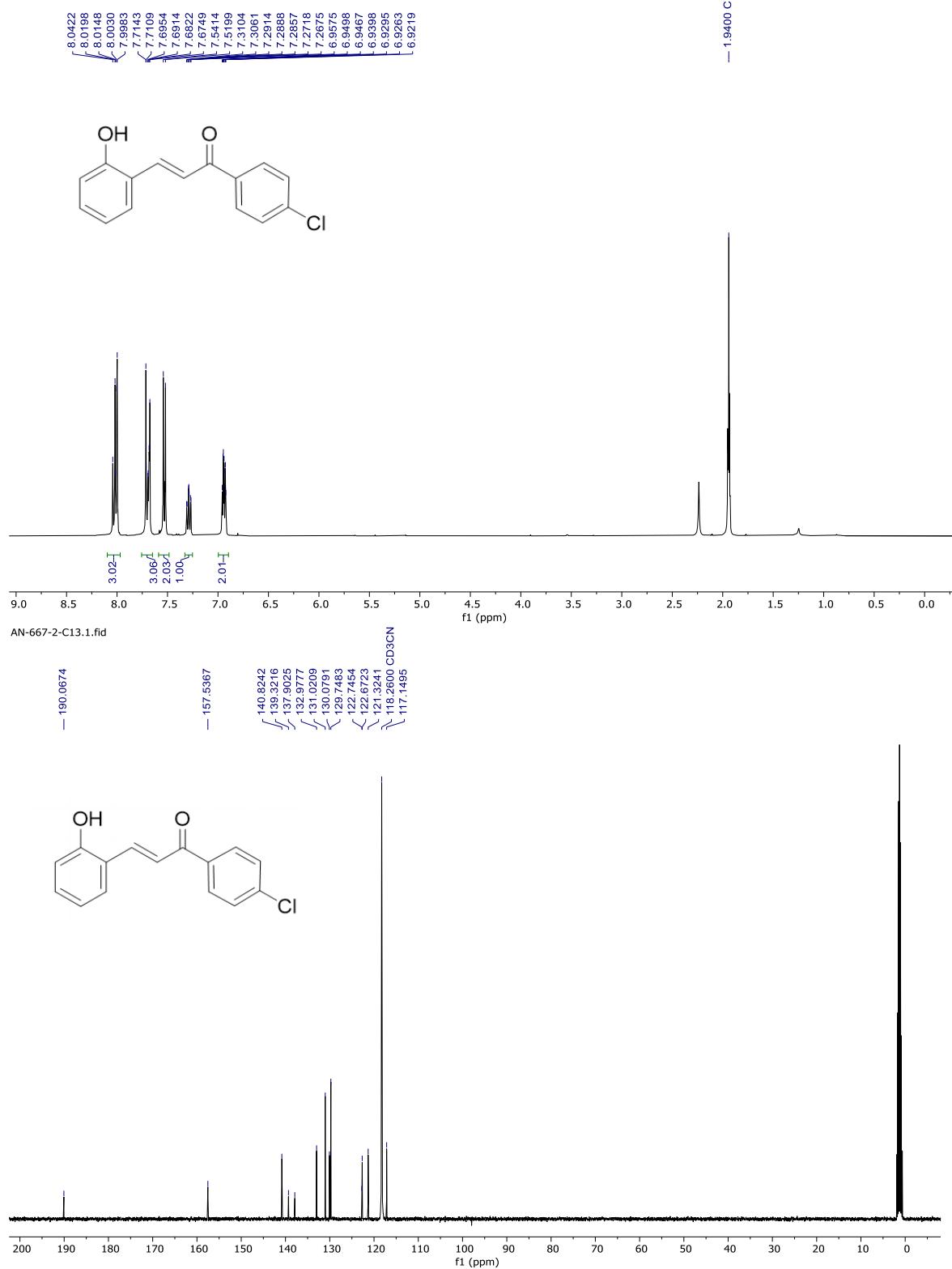
Compound 52 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-685-H1.1.fid



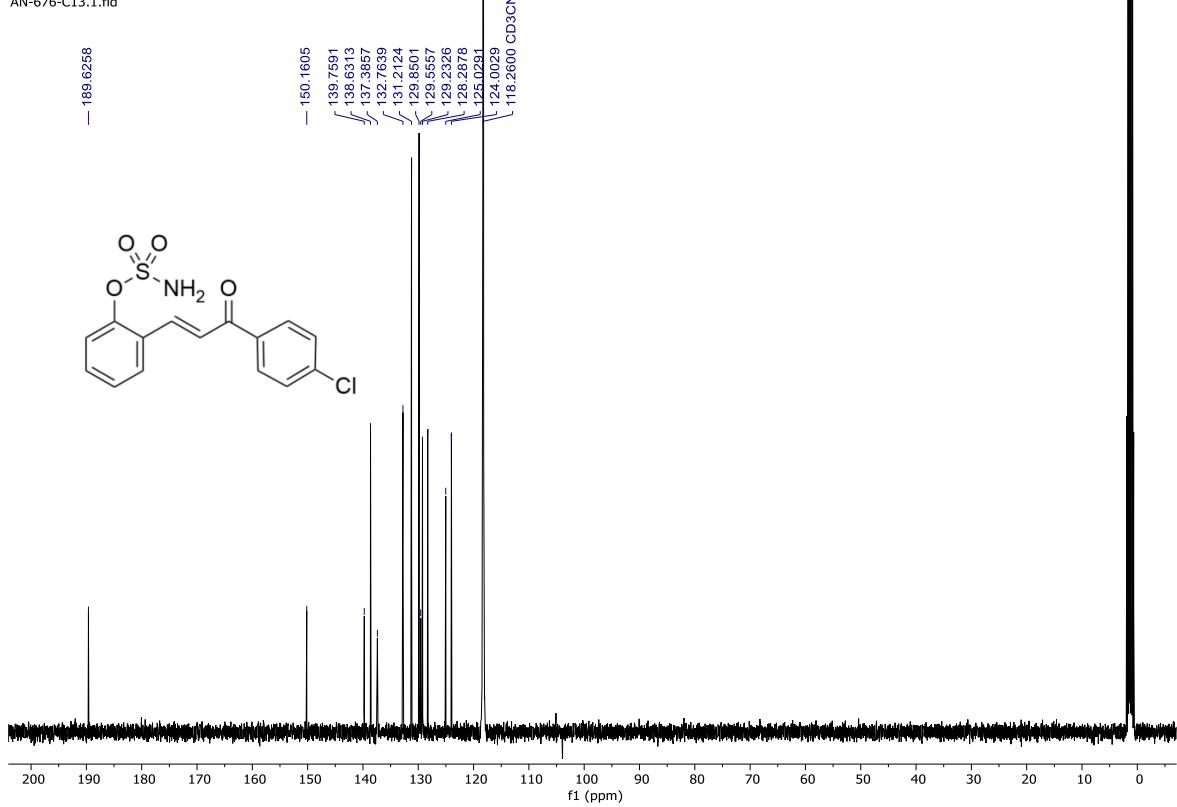
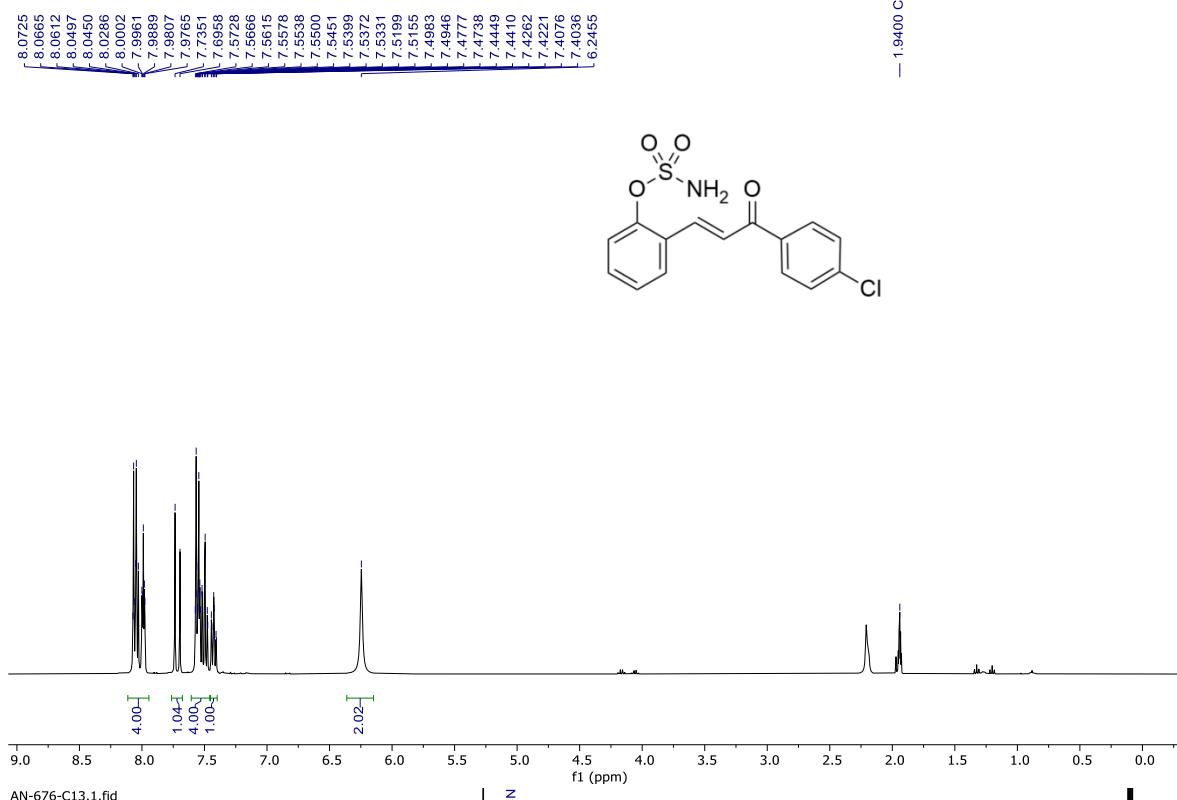
Compound S53' (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-667-2-H1.1.fid



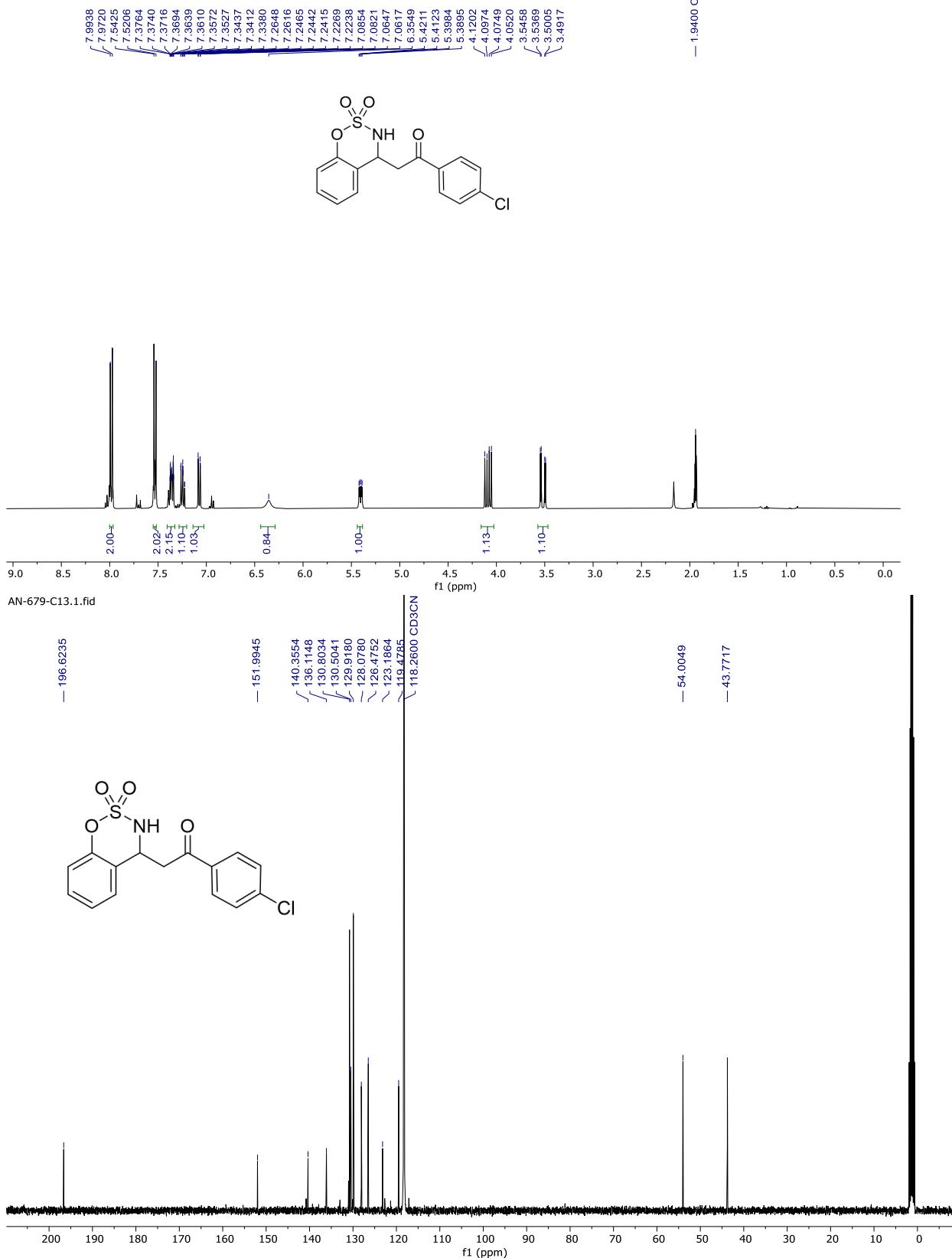
Compound S53 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-676-H1.1.fid



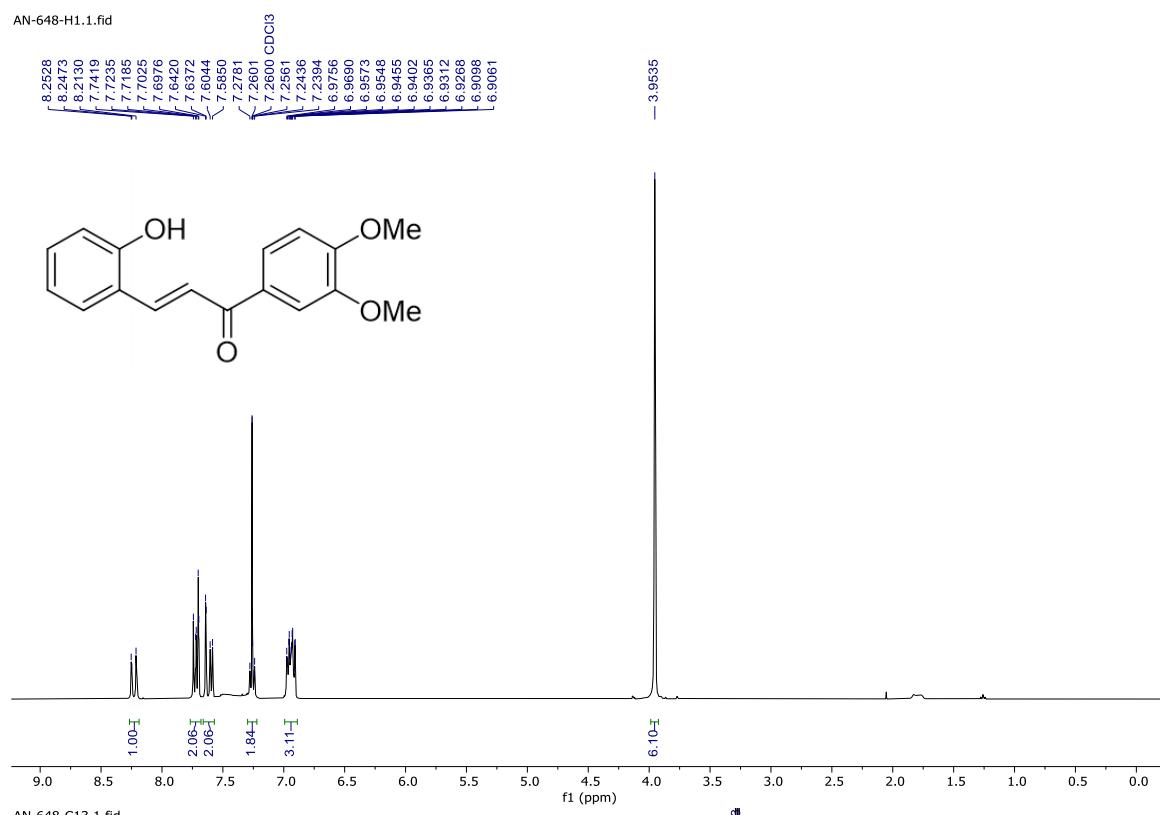
Compound 53 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-679-H1.1.fid

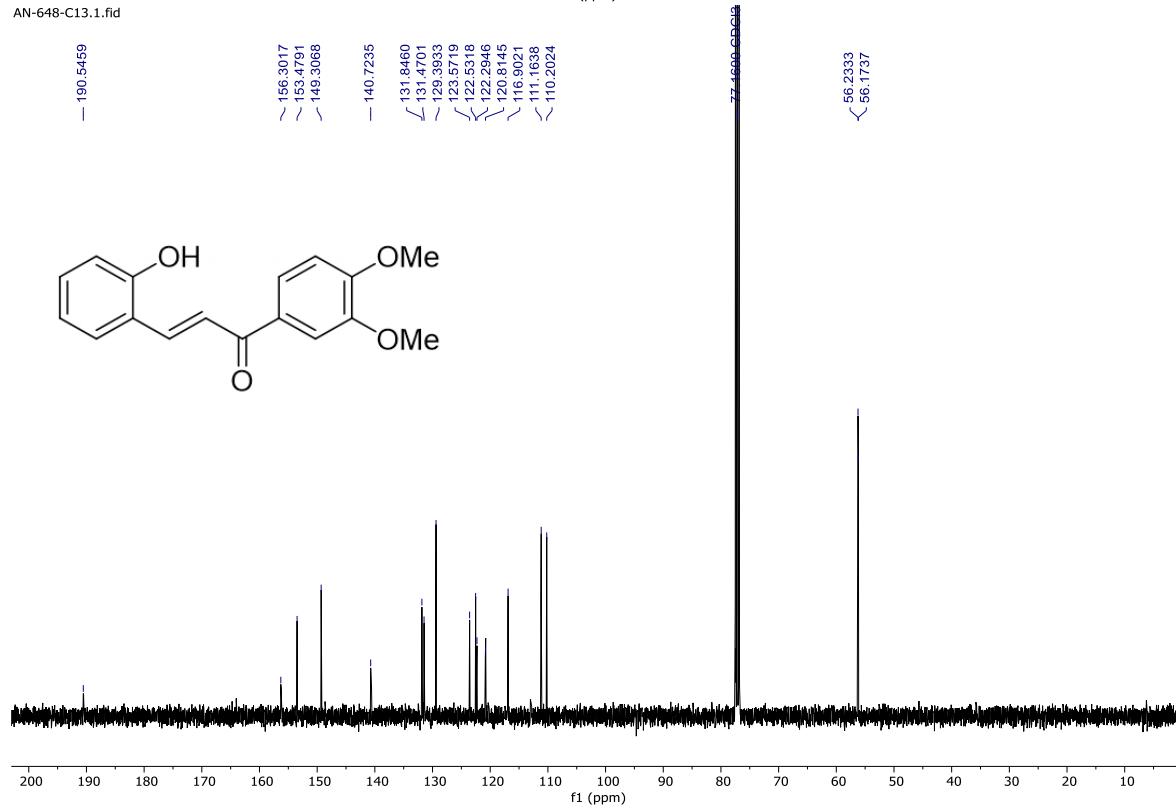


Compound S54' (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

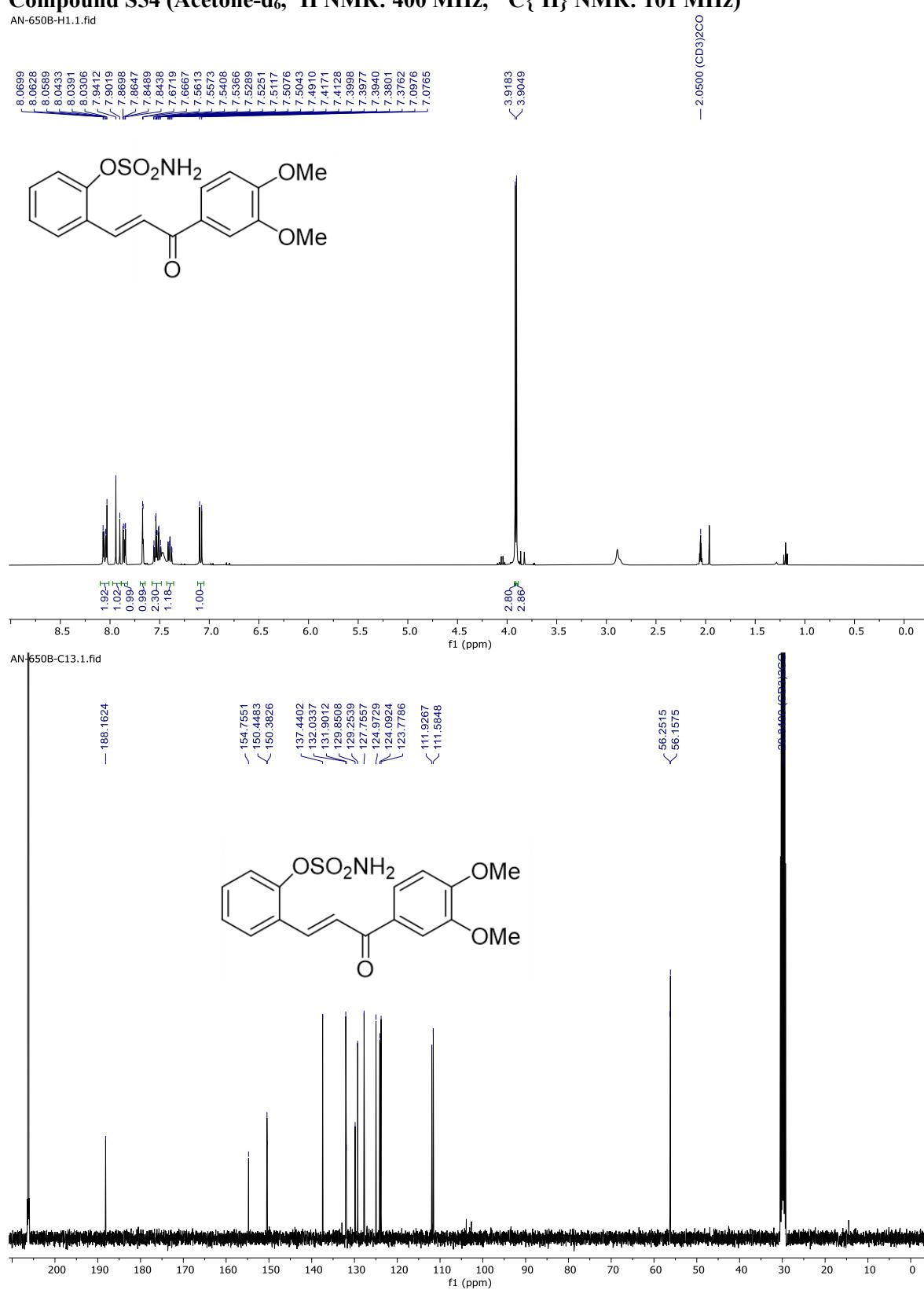
AN-648-H1.1.fid



AN-648-C13.1.fid

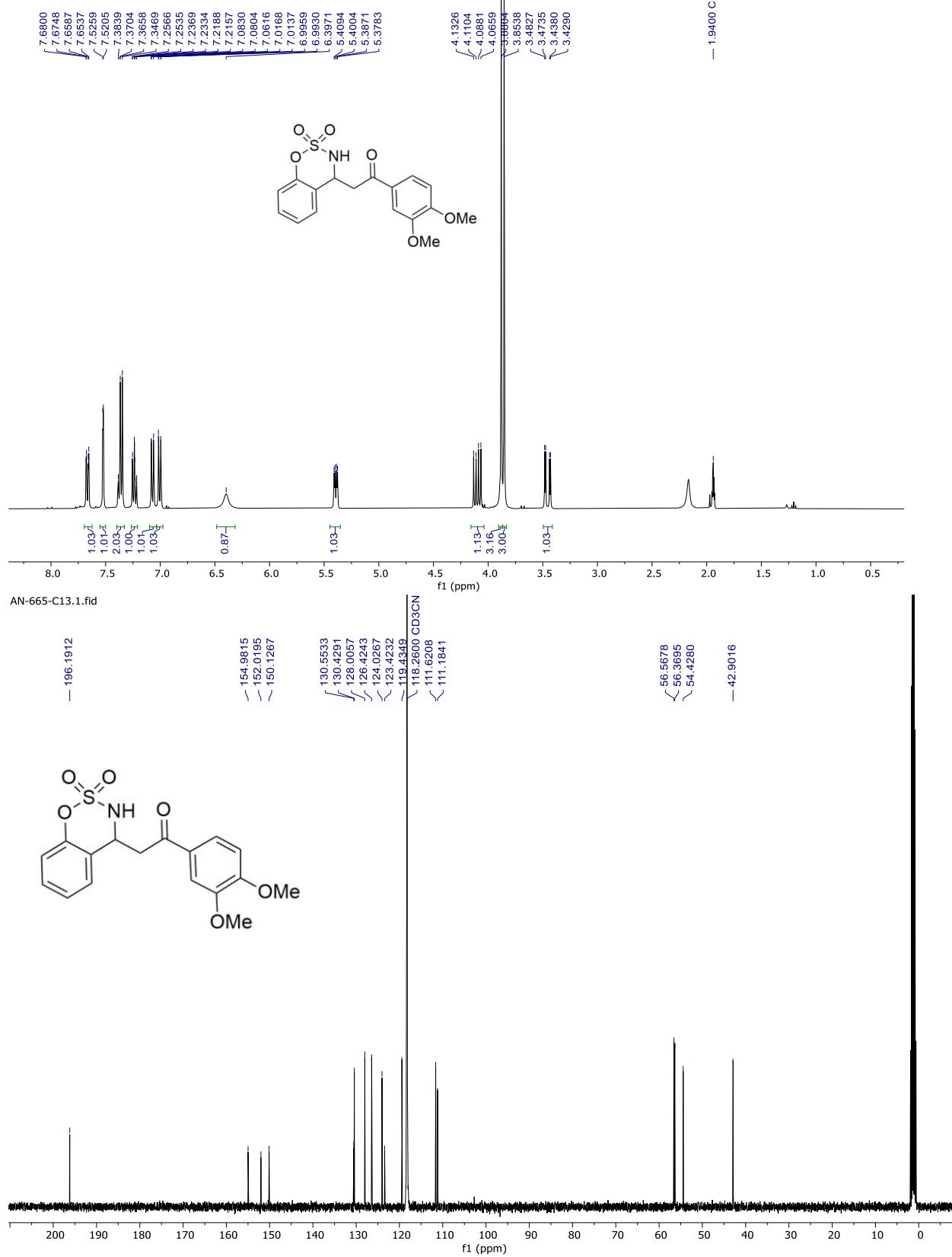


Compound S54 (Acetone-d₆, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)
AN-650B-H1.1.fid



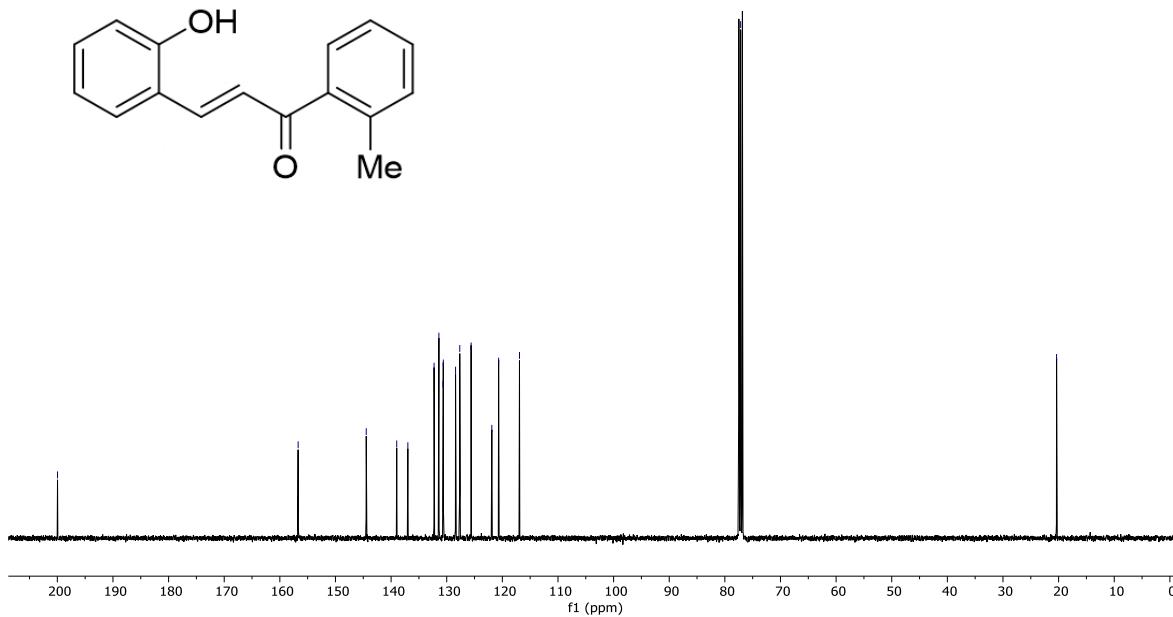
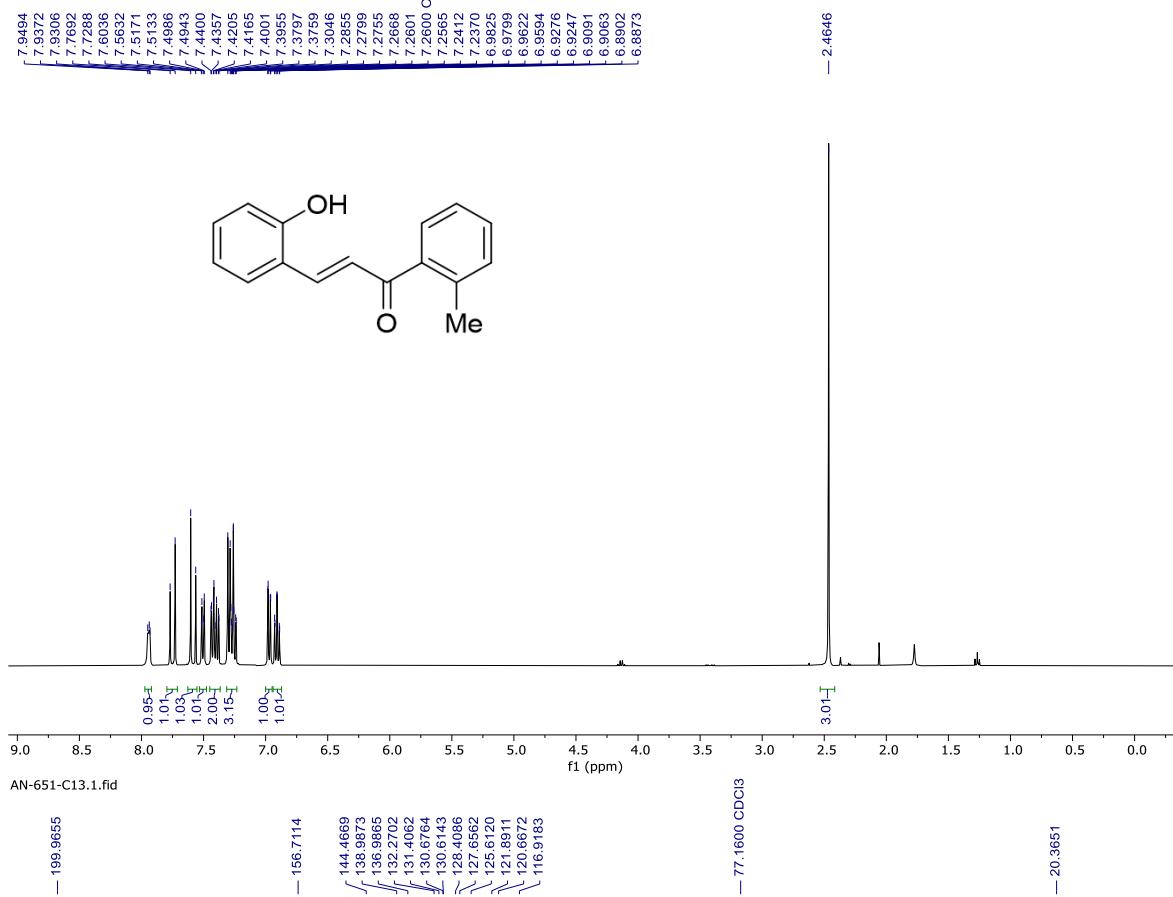
Compound 54 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-665-H1.1.fid



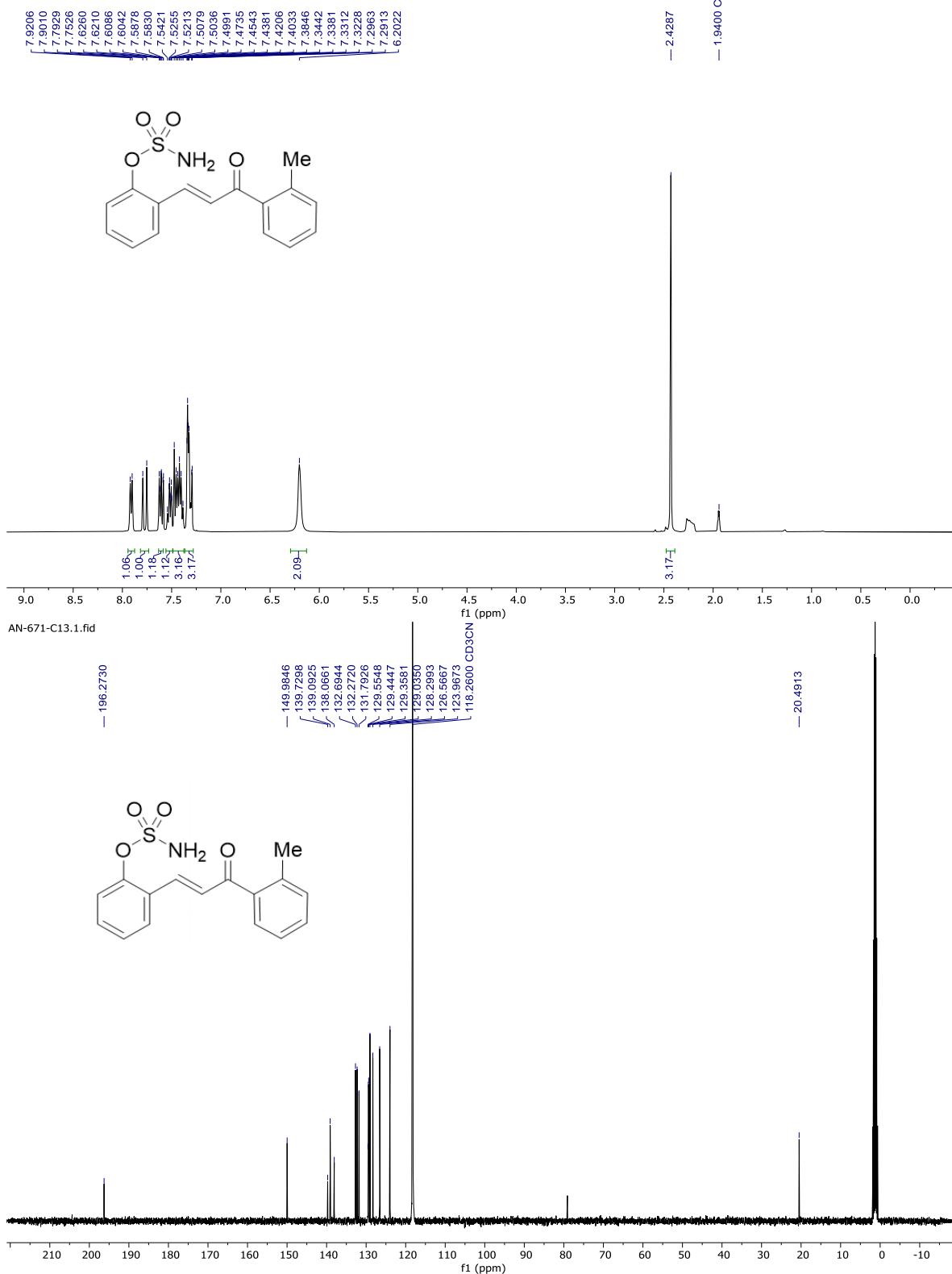
Compound S55' (CDCl₃, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

AN-651-H1.1.fid

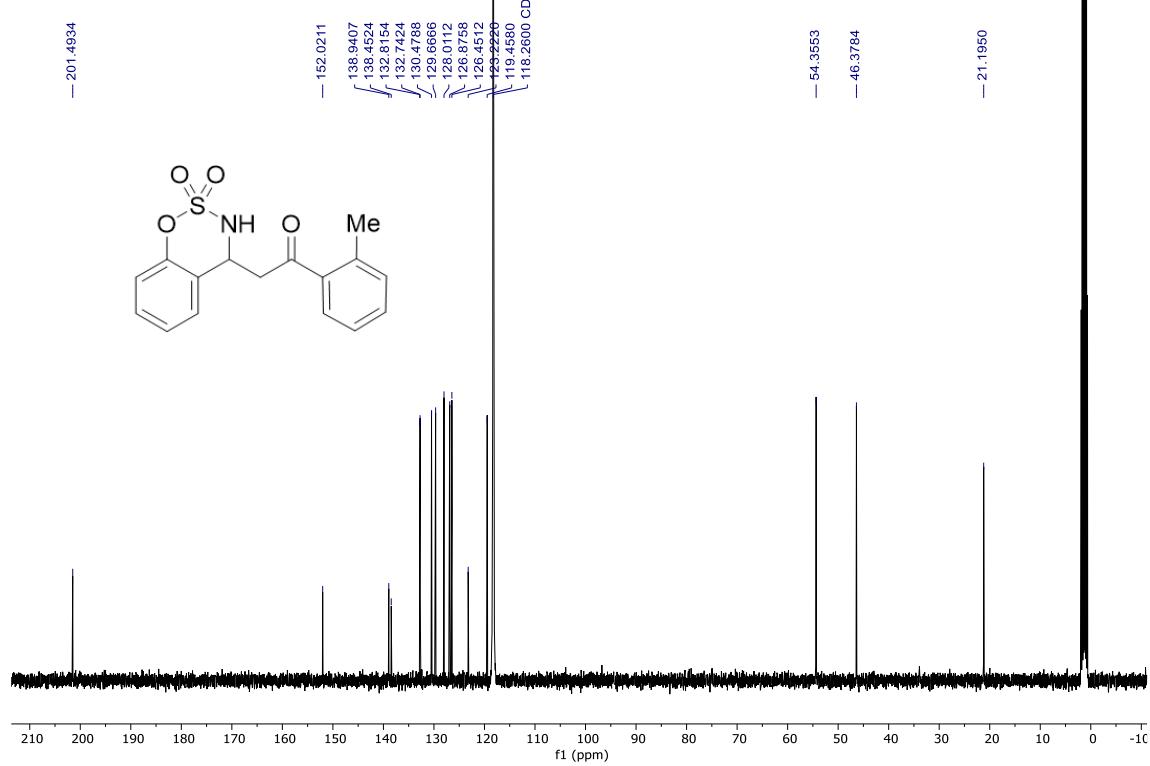
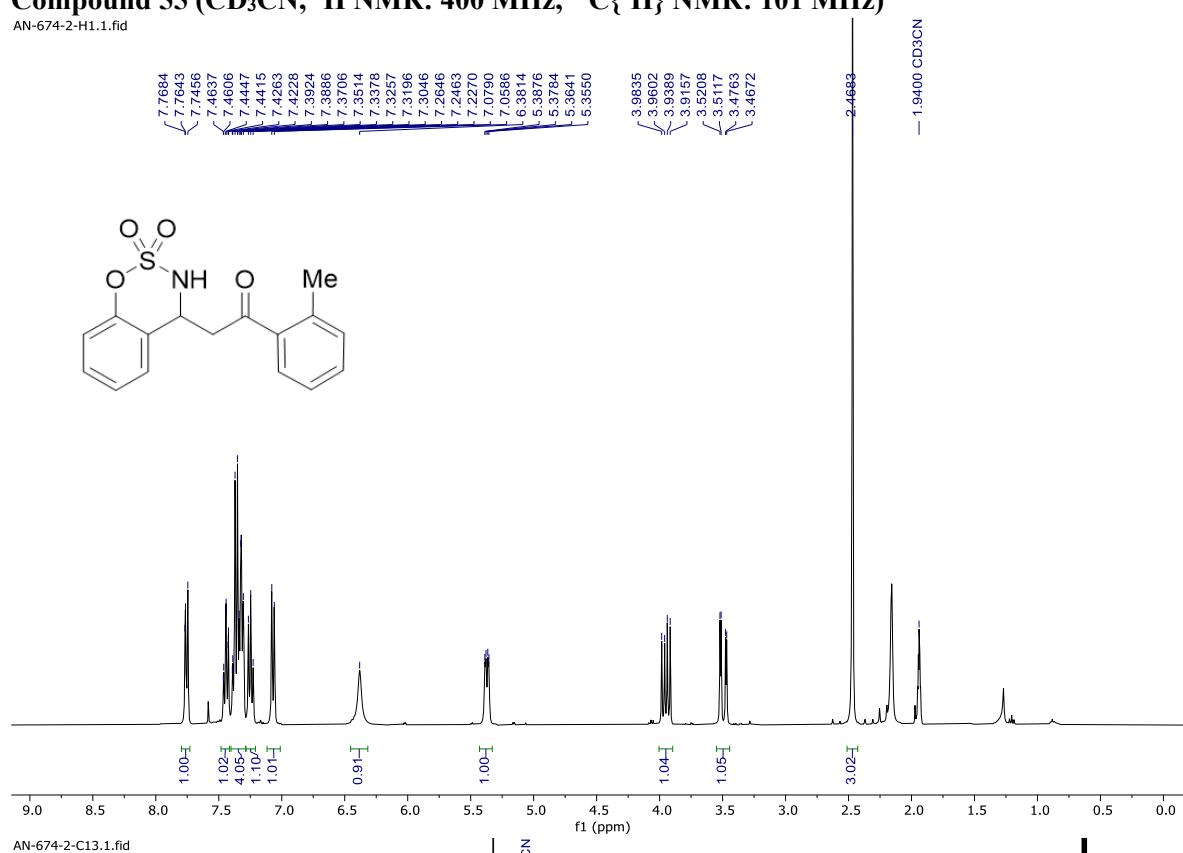


Compound S55 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)

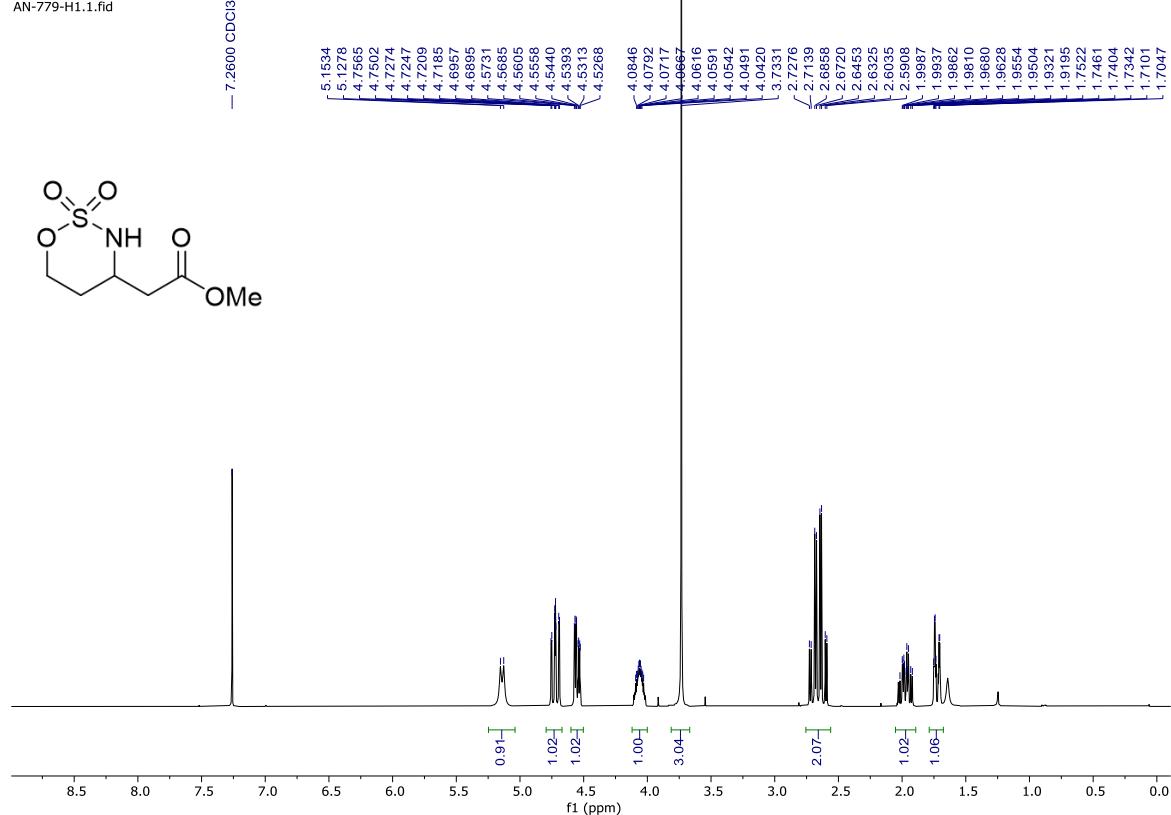
AN-671-H1.1.fid



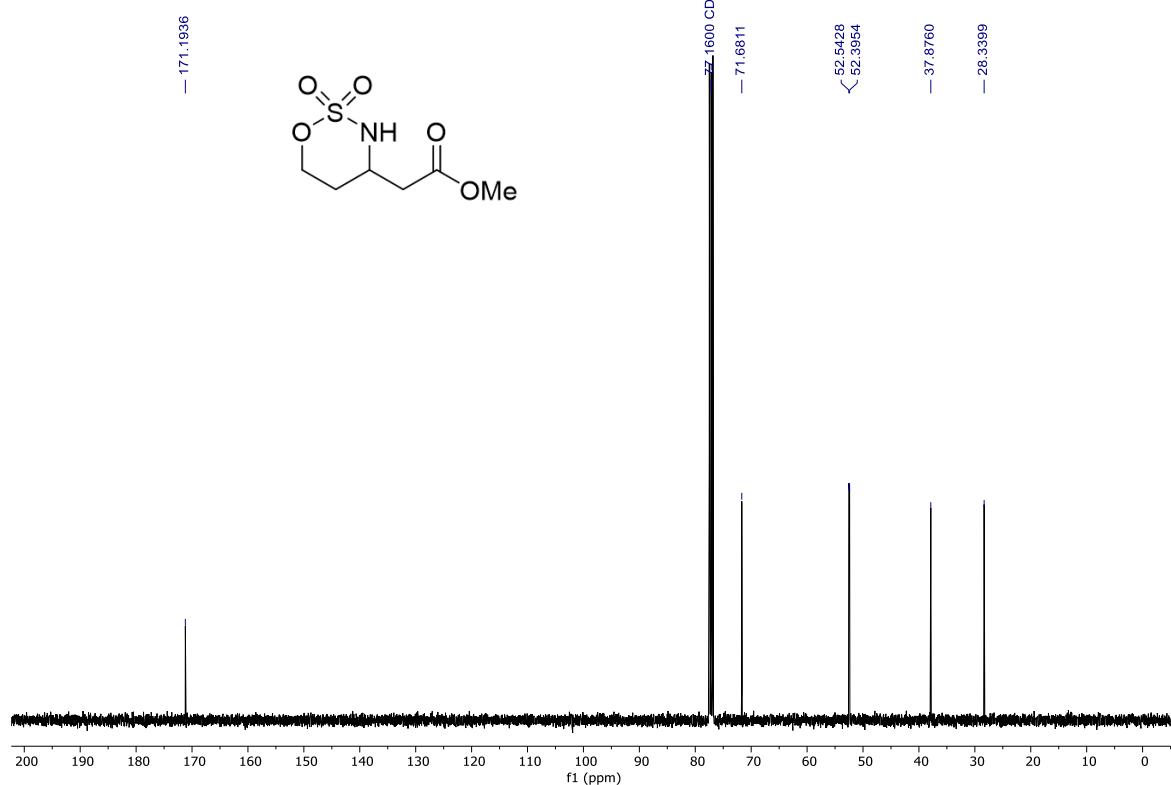
Compound 55 (CD₃CN, ¹H NMR: 400 MHz, ¹³C{¹H} NMR: 101 MHz)
AN-674-2-H1.1.fid



Compound 56 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)
 AN-779-H1.1.fid

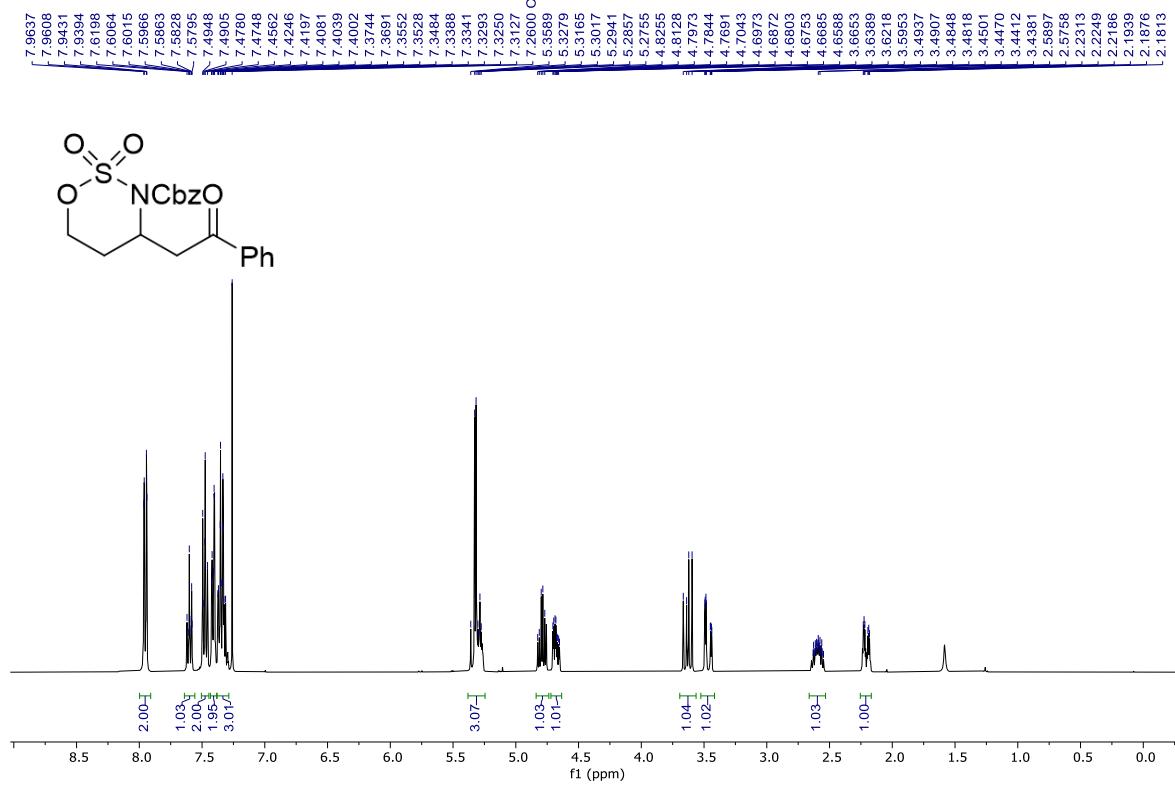


AN-779-C13.1.fid

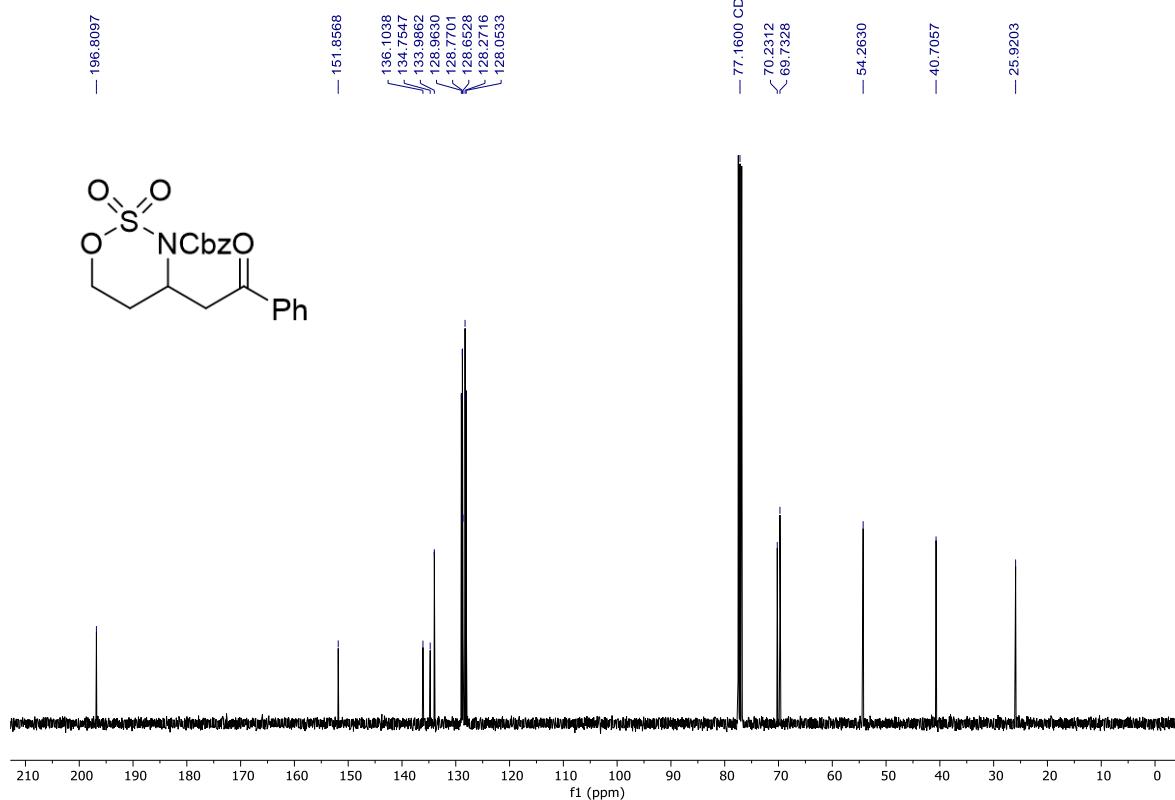


Compound 57 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{\text{H}\}$ NMR: 101 MHz)

AN-782-H1.1.fid

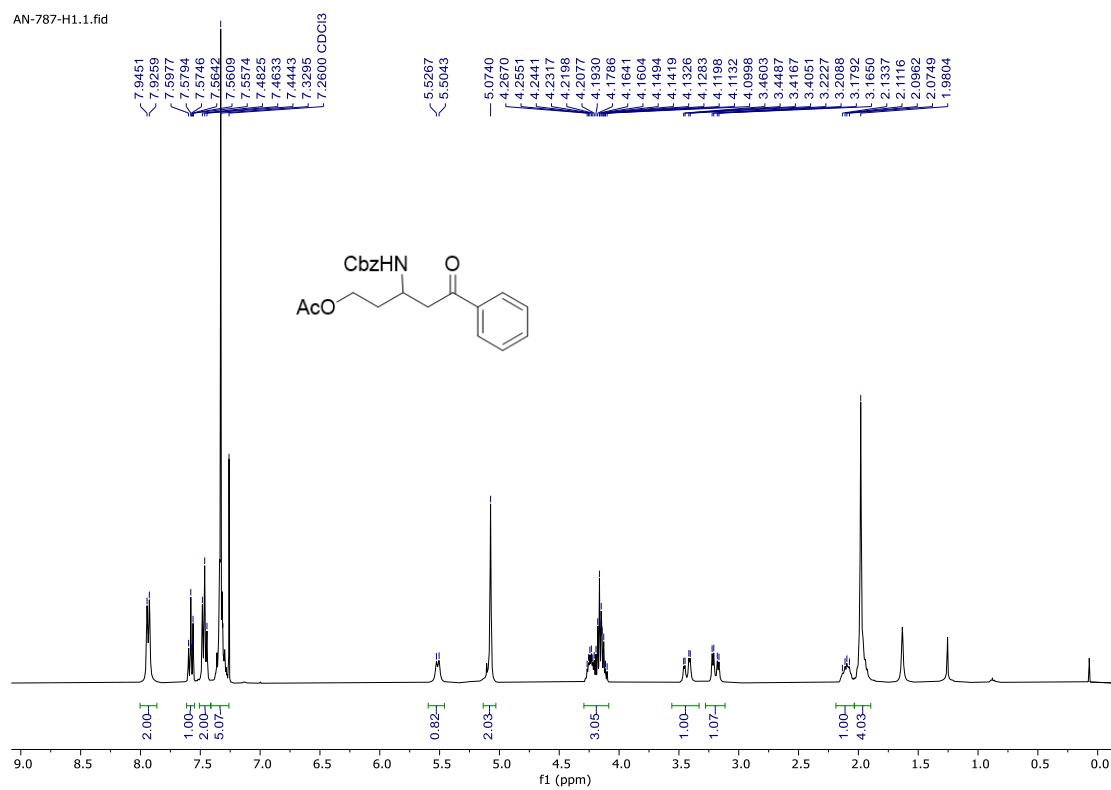


AN-782-C13.1.fid

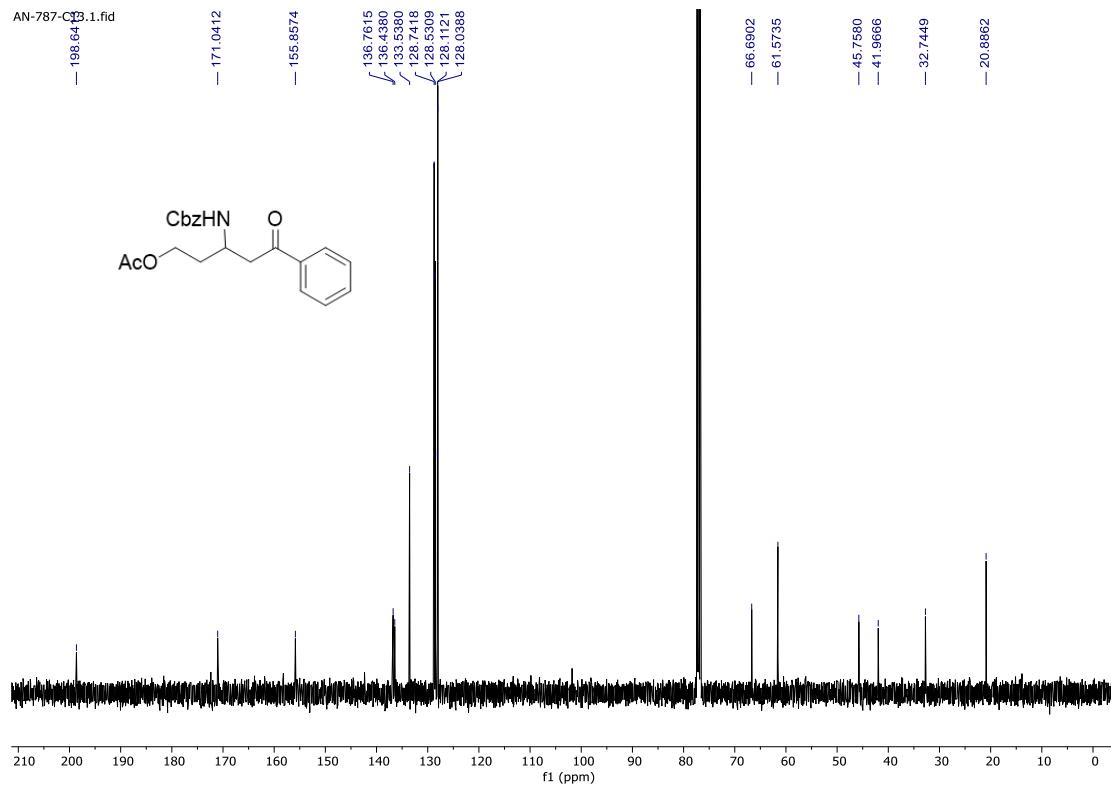


Compound 58 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-787-H1.1.fid

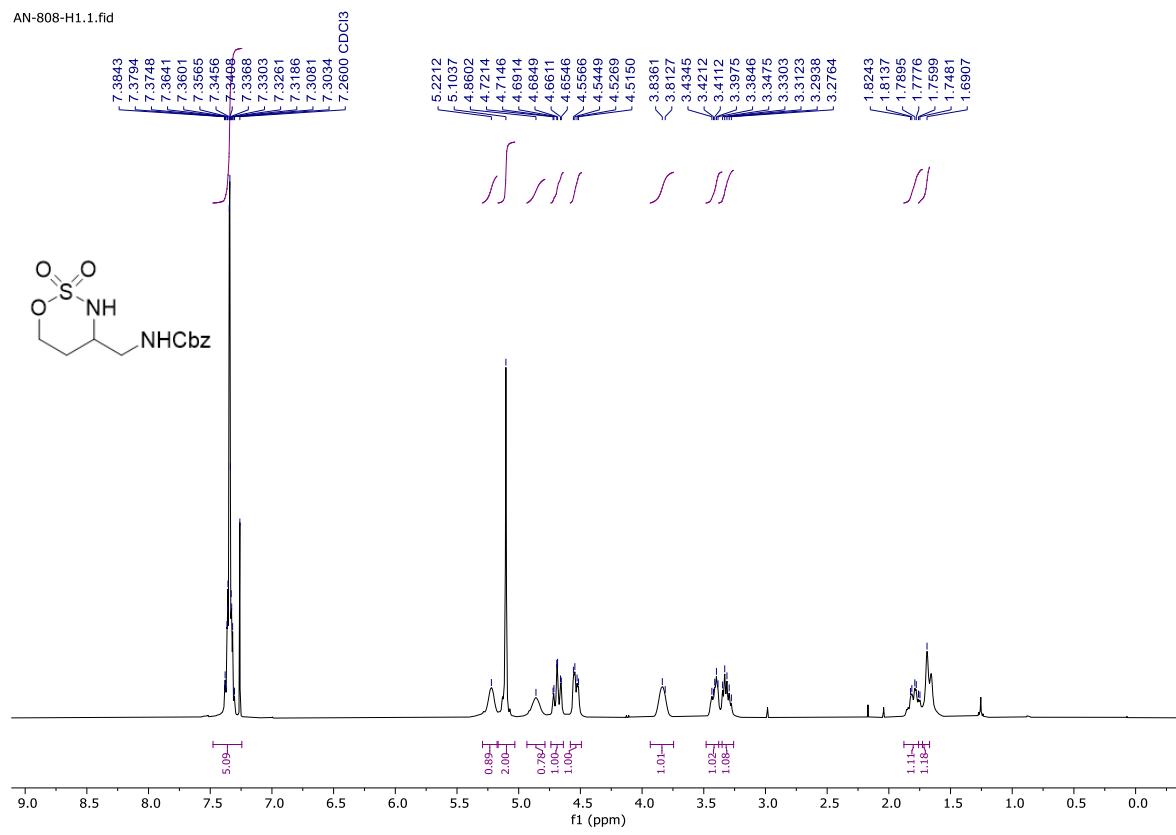


AN-787-CB1.1.fid



Compound 59 (CDCl_3 , ^1H NMR: 400 MHz, $^{13}\text{C}\{^1\text{H}\}$ NMR: 101 MHz)

AN-808-H1.1.fid



AN-808-C13.1.fid

