SUPPORTING INFORMATION *for*

A C–H Functionalization Approach to Diverse Nitrogenous Scaffolds Through Conjugate Addition of Catalytic Allyliron Nucleophiles

Sarah G. Scrivener,[a] and Yi-Ming Wang[a]*

[a]*Department of Chemistry, University of Pittsburgh, Pittsburgh, PA 15260*

Contents

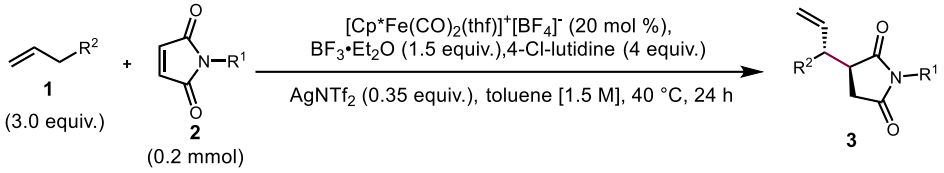
General Information

General Reagent Information: Anhydrous tetrahydrofuran (THF), 1,2-dichloroethane (DCE), toluene and trifluorotoluene were purchased from Acros (AcroSeal packaging), Sigma Aldrich (Sure/Seal packaging), and Frontier Scientific (J&KSeal packaging), respectively, and were deoxygenated by sparging with nitrogen and transferred into an argon-filled glovebox. Other dry solvents were obtained by distillation and storage over 3Å or 4Å molecular sieves. All other reagents were purchased from Oakwood, Acros, TCI, Strem, Alfa Aesar, or Sigma Aldrich and used as received. 2,2,6,6,-Tetramethylpiperidine (TMPH) was purified by short-path vacuum distillation. Cationic iron complex $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ was prepared according to literature procedure.[1] Air sensitive reagents and the iron catalyst (including $BF_3 \bullet Et_2O$, TMPH, silyl triflates, and bistriflimide salts) were stored in the glovebox. Compounds were purified by flash column chromatography using SiliCycle *SiliaFlash® F60* silica gel, unless otherwise indicated.

General Analytical Information: New compounds were characterized by ^1H NMR, ^{13}C NMR and HRMS. Copies of the ^1H NMR and ^{13}C NMR spectra can be found at the end of the Supporting Information. ^1H and ^{13}C NMR spectra were recorded on Bruker 300 MHz, 400 MHz, or 500 MHz instruments. All ^1H NMR data are reported in δ units, parts per million (ppm), and were measured relative to the residual proton signal in the deuterated solvent at 7.26 ppm ($CDCl_3$). Throughout analysis of diastereomeric mixtures, H* indicates the major diastereomer, while H' indicates the minor diastereomer. All ^{13}C NMR spectra are ^1H decoupled and reported in ppm relative to the solvent signal at 77.16 ppm ($CDCl_3$). Thin-layer chromatography (TLC) was performed on Silicycle 250 μm (analytical) or 1000 μm (preparative) silica gel plates. Compounds were visualized by irradiation with UV light, or by staining with potassium permanganate. Yields refer to isolated compounds, unless otherwise indicated. High resolution mass spectra were recorded on a Thermo Scientific Q-Exactive mass spectrometer. NMR yield was determined by using 2,4-dinotrotoluene as internal standard for ^1H spectroscopy.

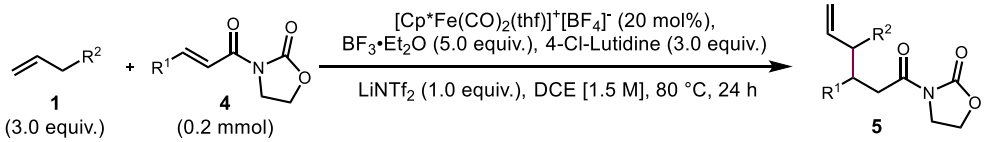
1. General procedures

General procedure A for the addition of alkenes to maleimide Michael acceptors



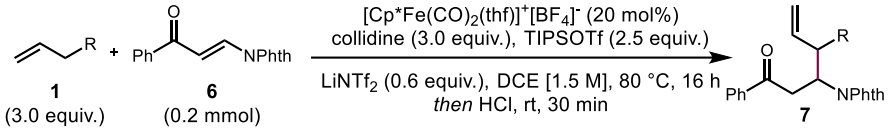
A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with an olive shaped magnetic stir bar (10 mm) was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were added Michael acceptor (**2**, 0.2 mmol, 1.0 equiv), $AgNTf_2$ (0.07 mmol, 27.7mg, 0.35 equiv), and $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 16.8 mg). Dry toluene (0.13 mL, 1.5 M), was added and the solution was briefly stirred. Then alkene (**1**, 0.9 mmol, 3.0 equiv), $BF_3•Et_2O$ (0.3 mmol, 37 μL, 1.5 equiv), and 4-chloro-2,6-lutidine (0.8 mmol, 103 μL, 4.0 equiv) were sequentially added with brief stirring after each addition. The reaction tube was then removed from the glovebox and placed in an oil bath at 40 °C for 24 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug (2 mL) using ethyl acetate (10 mL). The diastereomeric ratio (d.r.) was then determined by ^{1}H NMR analysis of a portion of the filtrate. The crude mixture was concentrated *in vacuo*. After concentration *in vacuo*, the crude mixture was purified by flash column chromatography to provide the desired product. The stereochemical outcome was assigned by analogy to the relative configuration of a crystal grown from a diastereomerically-enriched sample of **3fc**. The major diastereomer was determined to be the (*R**, *R**) configuration.

General procedure B for the addition of alkenes to *N*-acyl oxazolidinone Michael acceptors



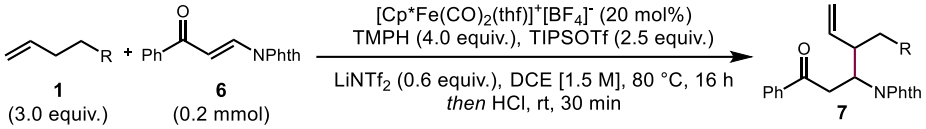
A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with an olive shaped magnetic stir bar (10 mm) was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were added Michael acceptor (**4**, 0.2 mmol, 1.0 equiv), $LiNTf_2$ (0.2 mmol, 57.4 mg, 1.0 equiv), and $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 16.8 mg). Dry DCE (0.13 mL, 1.5 M), was added and the solution was briefly stirred. Then alkene (**1**, 0.9 mmol, 3.0 equiv), $BF_3•Et_2O$ (1.0 mmol, 125 μL, 5.0 equiv), and 4-chloro-2,6-lutidine (0.6 mmol, 77 μL, 3.0 equiv) were sequentially added with brief stirring after each addition. The reaction tube was then removed from the glovebox and placed in an oil bath at 80 °C for 24 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug (2 mL) using ethyl acetate (10 mL). The diastereomeric ratio (d.r.) was then determined by ^{1}H NMR analysis of a portion of the filtrate. The crude mixture was concentrated *in vacuo*. After concentration *in vacuo*, the crude mixture was purified by flash column chromatography to provide the desired product.

General procedure C for the addition of activated alkenes to β-phthalimido Michael acceptors



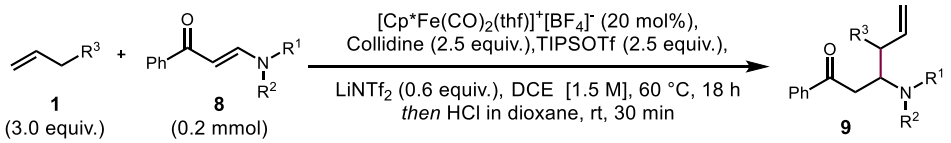
A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with an olive shaped magnetic stir bar (10 mm) was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were added Michael acceptor (**6**, 0.3 mmol, 1.0 equiv), $LiNTf_2$ (0.12 mmol, 34.4 mg, 0.6 equiv), and $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 16.8 mg). Dry DCE (0.13 mL, 1.5 M), was added and the solution was briefly stirred. Then alkene (**1**, 0.9 mmol, 3.0 equiv), TIPSOTf (0.5 mmol, 134 µL, 2.5 equiv), and 2,4,6-collidine (0.6 mmol, 75 µL, 3.0 equiv) were sequentially added with brief stirring after each addition. The reaction tube was then removed from the glovebox and placed in an oil bath at 80 °C for 16 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug (2 mL) using ethyl acetate (10 mL). 4N HCl in dioxane (0.4 mmol, 100 µL, 2.0 equiv) was added at room temperature and the mixture was stirred for 30 min at room temperature. The crude mixture was concentrated *in vacuo*. After concentration *in vacuo*, the crude mixture was purified by flash column chromatography to provide the desired product.

General procedure D for the addition of unactivated alkenes to β-phthalimido Michael acceptors



A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with an olive shaped magnetic stir bar (10 mm) was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were added Michael acceptor (**6**, 0.2 mmol, 1.0 equiv), $LiNTf_2$ (0.12 mmol, 34.4 mg, 0.6 equiv), and $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 16.8 mg). Dry DCE (0.13 mL, 1.5 M), was added and the solution was briefly stirred. Then alkene (**1**, 0.9 mmol, 3.0 equiv), TIPSOTf (0.5 mmol, 134 µL 2.5 equiv), and TMPH (0.8 mmol, 136 µL, 4.0 equiv) were sequentially added with brief stirring after each addition. The reaction tube was then removed from the glovebox and placed in an oil bath at 80 °C for 16 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug (2 mL) using ethyl acetate (10 mL). 4N HCl in dioxane (0.4 mmol, 100 µL, 2.0 equiv) was added at room temperature and the mixture was stirred for 30 min at room temperature. The crude mixture was concentrated *in vacuo*. After concentration *in vacuo*, the crude mixture was purified by flash column chromatography to provide the desired product.

General procedure E for the addition of alkenes to β-sulfonamido Michael acceptors

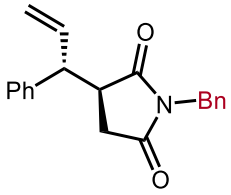


A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with an olive shaped magnetic stir bar (10 mm) was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum and transferred into an argon-filled glovebox. In the

glovebox were added Michael acceptor (**8**, 0.2 mmol, 1.0 equiv), $LiNTf_2$ (0.12 mmol, 34.4 mg, 0.6 equiv), and $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 16.8 mg). Dry DCE (0.13 mL, 1.5 M), was added and the solution was briefly stirred. Then alkene (**1**, 0.9 mmol, 3.0 equiv), TIPSOTf (0.5 mmol, 134 μL 2.5 equiv), and collidine (0.5 mmol, 66 μL, 2.5 equiv) were sequentially added with brief stirring after each addition. The reaction tube was then removed from the glovebox and placed in an oil bath at 60 °C for 18 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug (2 mL) using ethyl acetate (10 mL). 4N HCl in dioxane (0.4 mmol, 100 μL, 2.0 equiv) was added at room temperature and the mixture was stirred for 30 min at room temperature. The crude mixture was concentrated *in vacuo*. After concentration *in vacuo*, the crude mixture was purified by flash column chromatography to provide the desired product.

2. Characterization data

2.1 Synthesis of Michael adducts from Fe-catalyzed Michael addition – Maleimides

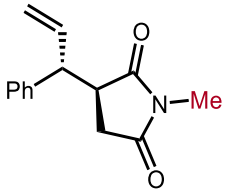


1-Benzyl-3-(1-phenylallyl)pyrrolidine-2,5-dione (3aa): (SGS-5-192) Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (54.5 mg, 89%, 7.6:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 1H*+1H'), 7.25 – 7.06 (m, 9H*+9H'), 6.11 (ddd, *J* = 17.0, 10.4, 6.4 Hz, 1H*), 5.98 – 5.89 (m, 1H'), 5.29 – 5.05 (m, 2H*+2H'), 4.62 (s, 2H'), 4.51 (s, 2H*), 4.14 – 4.06 (m, 1H*), 4.03 – 3.98 (m, 1H'), 3.37 – 3.25 (m, 1H*+1H'), 2.77 – 2.61 (m, 1H*+2H'), 2.51 (dd, *J* = 18.4, 4.4 Hz, 1H*). Throughout analysis of diastereomeric mixtures, H* indicates the major diastereomer, while H' indicates the minor diastereomer.

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.4, 175.9, 138.2, 138.0, 135.6, 134.8, 129.0_1, 128.9_9, 128.8_5, 128.8, 128.7, 128.6, 128.0, 127.9, 127.8, 127.6, 119.6, 116.6, 49.3, 48.6, 45.3, 44.0, 42.6, 42.4, 31.4, 29.8. Six peaks missing due to overlap and low intensity of minor diastereomer.

HRMS (ESI) calcd for $C_{20}H_{20}NO_2$ $[M+H]^+$: 306.14886, found: 306.14831.

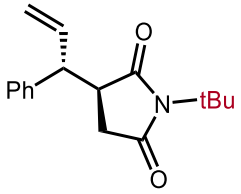


1-Methyl-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ba): (SGS-6-34) Prepared following **General procedure A** using 1-methyl-1*H*-pyrrole-2,5-dione (**2b**, 22.2 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (26.4 mg, 58%, 3.9:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H'), 7.31 – 7.19 (m, 3H*+3H'), 7.18 – 7.11 (m, 2H*), 6.15 (ddd, *J* = 17.1, 10.5, 6.4 Hz, 1H*), 5.99 (ddd, *J* = 17.0, 10.2, 8.5 Hz, 1H'), 5.29 – 5.10 (m, 2H*+2H'), 4.10 – 4.01 (m, 1H*+1H'), 3.35 – 3.25 (m, 1H*+1H'), 2.95 (s, 3H'), 2.81 (s, 3H*), 2.75 – 2.65 (m, 1H*+2H'), 2.48 (dd, *J* = 18.3, 4.3 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.8, 178.6, 176.5, 176.3, 140.4, 138.4, 137.9, 134.9, 129.0, 128.8, 128.7, 127.8, 127.7, 127.3, 119.6, 116.6, 49.2, 48.8, 45.5, 44.1, 31.5, 31.3, 24.9, 24.7.

HRMS (ESI) calcd for $C_{14}H_{16}NO_2$ $[M+H]^+$: 230.11756, found: 230.11781.

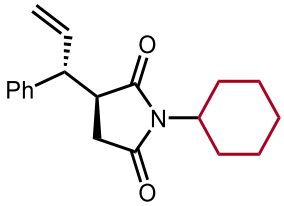


1-(Tert-butyl)-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ca): (SGS-6-35) Prepared following **General procedure A** using 1-(tert-butyl)-1*H*-pyrrole-2,5-dione (**2c**, 30.6 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (37.1 mg, 68%, 6.8:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.14 (m, 5H*+5H'), 6.11 (ddd, *J* = 17.0, 10.5, 6.2 Hz, 1H*), 5.99 (ddd, *J* = 17.0, 10.3, 8.5 Hz, 1H'), 5.27 – 5.09 (m, 2H*+2H'), 4.10 – 4.05 (m, 1H*), 4.01 (dd, *J* = 8.4, 3.9 Hz, 1H'), 3.18 – 3.08 (m, 1H*+1H'), 2.64 – 2.50 (m, 1H*+2H'), 2.41 (dd, *J* = 18.2, 4.3 Hz, 1H*), 1.54 (s, 9H'), 1.38 (s, 9H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.9, 179.7, 177.6, 177.4, 140.7, 138.3, 138.2, 134.9, 129.0, 128.9, 128.8, 127.8, 127.6, 127.2, 119.5, 116.4, 58.6, 58.4, 49.5, 48.9, 45.0, 43.8, 31.9, 31.7, 28.5, 28.3.

HRMS (ESI) calcd for $C_{17}H_{22}NO_2$ $[M+H]^+$: 272.16451, found: 272.16487.

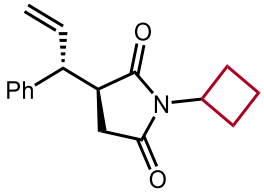


1-Cyclohexyl-3-(1-phenylallyl)pyrrolidine-2,5-dione (3da): (SGS-6-24) Prepared following **General procedure A** using 1-cyclohexyl-1*H*-pyrrole-2,5-dione (**2d**, 35.8 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (40.9 mg, 82%, 6.8:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.12 (m, 5H*+5H'), 6.12 (ddd, *J* = 17.0, 10.5, 6.2 Hz, 1H*), 5.98 (ddd, *J* = 17.0, 10.2, 8.5 Hz, 1H'), 5.28 – 5.09 (m, 2H*+2H'), 4.15 – 4.08 (m, 1H*), 4.02 (dd, *J* = 8.5, 3.9 Hz, 1H'), 3.94 (tt, *J* = 12.4, 3.9 Hz, 1H'), 3.78 (tt, *J* = 12.4, 3.8 Hz, 1H*), 3.26 – 3.15 (m, 1H*+1H'), 2.70 – 2.61 (m, 1H*+1H'), 2.58 (dd, *J* = 18.4, 4.7 Hz, 1H'), 2.45 (dd, *J* = 18.4, 4.1 Hz, 1H*), 2.17 – 1.49 (m, 6H*+6H'), 1.34 – 1.08 (m, 4H*+4H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.9, 178.7, 176.6, 176.4, 140.6, 138.2, 138.0, 134.8, 128.9, 128.8, 127.8, 127.6, 127.2, 119.5, 116.4, 51.9, 51.7, 49.3, 48.7, 44.8, 43.4, 31.2, 31.1, 29.0, 28.8, 28.7, 28.6, 26.0, 25.9, 25.2, 25.1. Three peaks missing due to overlap.

HRMS (ESI) calcd for $C_{19}H_{24}NO_2$ $[M+H]^+$: 298.18016, found: 298.18073.

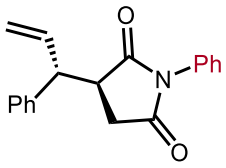


1-Cyclobutyl-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ea) (SGS-6-44) Prepared following **General procedure A** using 1-cyclobutyl-1*H*-pyrrole-2,5-dione (**2e**, 30.2 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (45.4 mg, 84%, 6.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.11 (m, 5H*+5H'), 6.13 (ddd, *J* = 16.9, 10.5, 6.2 Hz, 1H*), 5.98 (ddd, *J* = 17.1, 10.2, 8.6 Hz, 1H'), 5.32 – 5.06 (m, 2H*+2H'), 4.55 (p, *J* = 9.0 Hz, 1H'), 4.40 (p, *J* = 8.9 Hz, 1H*), 4.11 – 4.04 (m, 1H*), 4.01 (dd, *J* = 8.3, 3.9 Hz, 1H'), 3.28 – 3.14 (m, 1H*+1H'), 2.86 – 2.75 (m, 2H'), 2.73 – 2.52 (m, 3H*+2H'), 2.43 (dd, *J* = 18.3, 4.1 Hz, 1H*), 2.15 – 2.06 (m, 2H'), 2.03 – 1.93 (m, 2H*+1H'), 1.90 – 1.56 (m, 2H*+2H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.1, 178.8, 176.6, 176.4, 140.5, 138.2, 138.0, 135.0, 128.9, 128.8, 127.8, 127.6, 127.3, 119.4, 116.5, 49.4, 48.8, 45.5, 45.3, 44.8, 43.5, 31.3, 26.6, 26.5, 26.3, 15.5, 15.4. Three peaks missing due to overlap.

HRMS (ESI) calcd for $C_{17}H_{20}NO_2$ $[M+H]^+$: 270.14886, found: 270.14832.

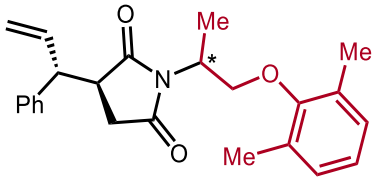


1-Phenyl-3-(1-phenylallyl)pyrrolidine-2,5-dione (3fa): (SGS-6-27) Prepared following **General procedure A** using 1-phenyl-1*H*-pyrrole-2,5-dione (**2f**, 34.6 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (44.1 mg, 75%, 3.8:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (t, *J* = 7.7 Hz, 2H'), 7.42 – 7.16 (m, 8H*+8H'), 6.94 (d, *J* = 7.6 Hz, 2H*), 6.22 – 6.09 (m, 1H*+1H'), 5.36 – 5.10 (m, 2H*+2H'), 4.27 – 4.22 (m, 1H*), 4.13 (dd, *J* = 8.6, 3.8 Hz, 1H'), 3.56 – 3.42 (m, 1H*+1H'), 2.95 – 2.77 (m, 1H*+2H'), 2.71 (dd, *J* = 18.5, 4.0 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.9, 177.6, 175.4, 175.3, 140.2, 138.0, 137.7, 134.8, 132.0, 131.8, 129.3, 129.2, 129.1, 129.0, 128.8, 128.8, 127.9, 127.8, 127.5, 126.6, 126.2, 119.9, 116.8, 49.6, 48.8, 45.5, 44.1, 31.5, 31.4. One peak missing due to overlap.

HRMS (ESI) calcd for $C_{19}H_{18}NO_2$ $[M+H]^+$: 292.13321, found: 292.13279.



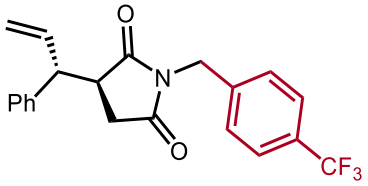
1-(1-(2,6-Dimethylphenoxy)propan-2-yl)-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ga): (SGS-6-65) Prepared following **General procedure A** using 1-(1-(2,6-dimethylphenoxy)propan-2-yl)-1*H*-pyrrole-2,5-dione (**2g**, 51.8 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (57.3

mg, 76%, 5.3:1 d.r. with respect to the newly generated stereocenters, d.r. with respect to the stereocenter designated by * could not be determined by ^{1}H NMR due to extensive signal overlap).

^{1}H NMR (601 MHz, $CDCl_3$) δ 7.37 – 7.11 (m, 5H*+5H'), 7.00 – 6.94 (m, 2H*+2H'), 6.92 – 6.87 (m, 1H*+1H'), 6.20 – 6.09 (m, 1H*), 6.07 – 5.97 (m, 1H'), 5.30 – 5.11 (m, 2H*+2H'), 4.74 – 4.67 (m, 1H'), 4.59 – 4.51 (m, 1H*), 4.30 (t, *J* = 9.3 Hz, 1H'), 4.16 – 4.04 (m, 2H*+1H'), 3.81 – 3.75 (m, 1H'), 3.75 – 3.66 (m, 1H*), 3.37 – 3.23 (m, 1H*+1H'), 2.78 – 2.66 (m, 1H*+2H'), 2.56 – 2.47 (m, 1H*), 2.23 (s, 3H'), 2.21 (s, 3H'), 2.19 (s, 3H*), 2.15 (s, 3H*), 1.40 – 1.25 (m, 3H'), 1.24 – 1.13 (m, 3H*).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.9, 178.7, 178.5, 176.6, 176.5, 176.4, 176.3, 155.5, 155.4, 155.2$_9$, 155.2$_5$, 140.6, 140.5, 138.5, 138.2, 138.0$_2$, 137.9$_9$, 135.0$_7$, 134.9$_5$, 130.8$_8$, 130.8$_4$, 130.8$_0$, 128.9$_7$, 128.9$_5$, 128.9$_0$, 128.8$_5$, 128.8$_5$, 128.7$_7$, 127.9, 127.8, 127.7 127.6, 127.3, 124.0$_9$, 124.0$_7$, 124.0$_5$, 119.6$_2$, 119.5$_6$, 116.6, 116.5, 71.0$_3$, 70.9$_6$, 70.9, 70.7, 49.2$_7$, 49.2$_5$, 49.0, 48.6, 48.1, 48.0, 47.9, 47.8, 45.0, 44.9, 43.7, 43.6, 31.5, 31.4, 31.3, 31.2, 16.5, 16.4$_3$, 16.3$_5$, 16.3, 14.4, 14.3$_4$, 14.3$_0$, 14.2. (Complexity due to diastereomers originating from the stereocenter indicated by *)

HRMS (ESI) calcd for $C_{24}H_{28}NO_3$ $[M+H]^+$: 378.20637, found: 378.20631.



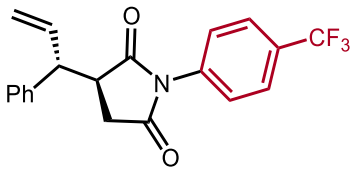
3-(1-Phenylallyl)-1-(4-(trifluoromethyl)benzyl)pyrrolidine-2,5-dione (3ha): (SGS-6-40) Prepared following **General procedure A** using 1-(4-(trifluoromethyl)benzyl)-1*H*-pyrrole-2,5-dione (**2h**, 51.0 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a brown oil (48.8 mg, 65%, 7.0:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 – 7.42 (m, 2H*+2H'), 7.22 – 7.03 (m, 7H*+7H'), 6.10 (ddd, *J* = 17.0, 10.5, 6.2 Hz, 1H*), 5.96 (ddd, *J* = 17.1, 10.2, 8.6 Hz, 1H'), 5.31 – 5.06 (m, 2H*+2H'), 4.66 (s, 2H'), 4.54 (s, 2H*), 4.13 (dd, *J* = 6.0, 4.6 Hz, 1H*), 3.99 (dd, *J* = 8.1, 4.4 Hz, 1H'), 3.37 (dt, *J* = 9.0, 4.4 Hz, 1H*), 3.31 (dt, *J* = 9.0, 4.5 Hz, 1H'), 2.82 – 2.54 (m, 2H*+2H').

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.3, 178.0, 175.9, 175.8, 140.2, 139.6, 139.4, 137.8, 137.8, 134.8, 130.12 (q, *J* = 32.3 Hz), 129.3, 129.0, 128.9, 128.8$_2$, 128.8$_1$, 127.8, 127.7, 127.4, 125.65 (q, *J* = 4.0 Hz), 124.16 (q, *J* = 272.1 Hz), 119.6, 116.6, 49.4, 48.4, 45.3, 43.9, 42.0, 41.8, 31.4, 31.2. Three peaks missing due to overlap.

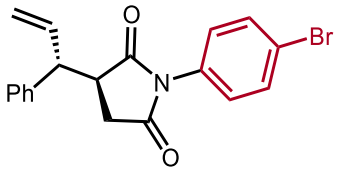
^{19}F NMR (471 MHz, $CDCl_3$) δ −62.66, −62.67.

HRMS (ESI) calcd for $C_{21}H_{19}NO_2F_3$ $[M+H]^+$: 374.13624, found: 374.13661.

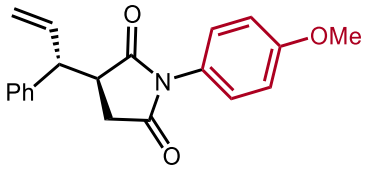


3-(1-Phenylallyl)-1-(4-(trifluoromethyl)phenyl)pyrrolidine-2,5-dione (3ia): (SGS-6-59) Prepared following **General procedure A** using 1-(4-(trifluoromethyl)phenyl)-1*H*-pyrrole-2,5-dione (**2i**, 48.2 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated

at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 5:1) to afford the title compound as a brown oil (60.0 mg, 84%, 3.5:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.2 Hz, 2H'), 7.66 (d, *J* = 8.2 Hz, 2H*), 7.41 – 7.24 (m, 3H*+8H'), 7.21 (d, *J* = 7.3 Hz, 2H*), 7.11 (d, *J* = 8.2 Hz, 2H*), 6.26 – 6.07 (m, 1H*+1H'), 5.41 – 5.14 (m, 2H*+2H'), 4.24 (s, 1H*), 4.13 (dd, *J* = 8.6, 3.7 Hz, 1H'), 3.59 – 3.45 (m, 1H*+1H'), 2.98 – 2.88 (m, 1H*+1H'), 2.84 (dd, *J* = 18.6, 4.5 Hz, 1H'), 2.76 (dd, *J* = 18.6, 3.8 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 177.5, 177.1, 174.9, 174.7, 139.9, 137.8, 137.5, 135.0, 134.9, 134.7, 130.7_1 (q, *J* = 33.2 Hz), 130.6_8 (q, *J* = 33.0 Hz), 129.1, 129.0, 128.9, 128.0, 127.8, 127.6, 126.8_4, 126.8_0, 126.4 – 126.2 (m), 123.8_0 (q, *J* = 271.9 Hz), 123.7_7 (q, *J* = 272.4 Hz), 120.0, 117.0, 49.7, 48.8, 45.5, 44.2, 31.6, 31.4.
^{19}F NMR (471 MHz, $CDCl_3$) δ −62.75, −62.78.
HRMS (ESI) calcd for $C_{20}H_{17}NO_2F_3$ $[M+H]^+$: 360.12059, found: 360.11995.

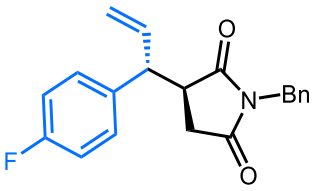


1-(4-Bromophenyl)-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ja): (SGS-6-22) Prepared following **General procedure A** using 1-(4-bromophenyl)-1*H*-pyrrole-2,5-dione (**2j**, 50.4 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a brown solid (52.0 mg, 70%, 3.4:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 – 7.57 (m, 2H'), 7.55 – 7.49 (m, 2H*), 7.39 – 7.27 (m, 3H*+6H'), 7.23 – 7.19 (m, 2H*), 7.13 – 7.09 (m, 2H'), 6.87 – 6.82 (m, 2H*), 6.23 – 6.08 (m, 1H*+1H'), 5.37 – 5.16 (m, 2H*+2H'), 4.23 (dd, *J* = 5.7, 4.5 Hz, 1H*), 4.12 (dd, *J* = 8.6, 3.8 Hz, 1H'), 3.53 – 3.48 (m, 1H*), 3.48 – 3.43 (m, 1H'), 2.95 – 2.86 (m, 1H*+1H'), 2.82 (dd, *J* = 18.6, 4.6 Hz, 1H'), 2.73 (dd, *J* = 18.6, 3.9 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 177.6, 177.2, 175.0, 174.9, 137.9, 137.6, 134.7, 132.5, 132.4, 130.8, 129.1, 129.0, 128.9, 128.1_0, 128.0_8, 128.0, 127.8, 127.6, 122.7, 120.0, 116.9, 49.7, 48.8, 45.5, 44.2, 31.6, 31.4. Three peaks missing due to overlap and low intensity of minor diastereomer.
HRMS (ESI) calcd for $C_{19}H_{15}NO_2Br$ $[M+H]^+$: 368.02807, found: 368.02870.
m.p. = 98 – 100 °C

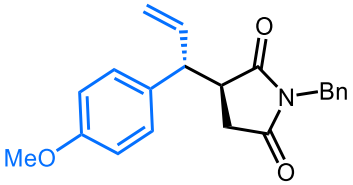


1-(4-Methoxyphenyl)-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ka): (SGS-6-38) Prepared following **General procedure A** using 1-(4-methoxyphenyl)-1*H*-pyrrole-2,5-dione (**2k**, 40.1 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (43.7 mg, 68%, 3.4:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.20 (m, 5H*+5H'), 7.13 – 7.08 (m, 2H'), 6.98 – 6.04 (m, 2H'), 6.92 – 6.87 (m, 2H*), 6.87 – 6.82 (m, 2H*), 6.22 – 6.08 (m, 1H+1H'), 5.34 – 5.15 (m, 2H*+2H'), 4.26 – 4.20 (m, 1H*), 4.14 – 4.09 (m, 1H'), 3.81 (s, 3H'), 3.78 (s, 3H*), 3.51 – 3.40 (m, 1H*+1H'), 2.93 – 2.83 (m, 1H*+1H'), 2.79 (dd, *J* = 18.5, 4.6 Hz, 1H'), 2.69 (dd, *J* = 18.5, 4.0 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 178.2, 177.8, 175.7, 175.5, 159.6_8, 159.6_5, 140.2, 138.0, 137.7, 134.8, 129.0, 129.0, 128.9, 127.9, 127.8, 127.4, 124.6, 124.4, 119.8, 116.7, 114.6_2, 114.5_6, 55.6_0, 55.5_6, 49.6, 48.7, 45.4, 44.1, 31.5, 31.3. One peak missing due to overlap.
HRMS (ESI) calcd for $C_{20}H_{20}NO_3$ $[M+H]^+$: 322.14377, found: 322.14390.



1-Benzyl-3-(1-(4-fluorophenyl)allyl)pyrrolidine-2,5-dione (3ab): (SGS-6-56)
Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and 4-fluoroallylbenzene (**1b**, 81 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a brown oil (46.1 mg, 71%, 6.5:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.21 (m, 3H*+5H'), 7.16 – 7.10 (m, 2H*+2H'), 7.05 – 6.99 (m, 2H*), 6.96 (t, *J* = 8.6 Hz, 2H'), 6.81 (t, *J* = 8.6 Hz, 2H*), 6.05 (ddd, *J* = 17.0, 10.5, 6.2 Hz, 1H*), 5.92 (ddd, *J* = 17.0, 10.3, 8.4 Hz, 1H'), 5.29 – 5.04 (m, 2H*+2H'), 4.64 – 4.45 (m, 2H*+2H'), 4.11 (dd, *J* = 5.7, 4.5 Hz, 1H*), 3.95 (dd, *J* = 8.2, 4.3 Hz, 1H'), 3.31 (dt, *J* = 8.9, 4.3 Hz, 1H*), 3.24 (dt, *J* = 9.0, 4.5 Hz, 1H'), 2.79 – 2.69 2.60 (m, 1H*+1H'), (dd, *J* = 18.4, 4.6 Hz, 1H'), 2.48 (dd, *J* = 18.5, 4.4 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 178.3, 175.8, 162.1 (d, J = 246.3 Hz), 137.6, 135.6, 135.0, 133.7 (d, J = 3.2 Hz), 130.3 (d, J = 8.1 Hz), 129.3 (d, J = 8.2 Hz), 129.0, 128.7 (d, J = 3.0 Hz), 128.1, 128.0, 119.5, 116.8, 115.9, 115.8, 115.6, 48.8, 47.6, 46.1, 45.2, 43.8, 42.4, 31.5, 31.1. Four peaks missing due to overlap.
^{19}F NMR (471 MHz, $CDCl_3$) δ −114.83, −115.33.
HRMS (ESI) calcd for $C_{20}H_{19}NO_2F$ $[M+H]^+$: 324.13943, found: 324.13974.

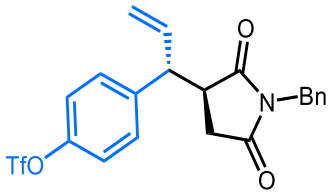


1-Benzyl-3-(1-(4-methoxyphenyl)allyl)pyrrolidine-2,5-dione (3ac) (SGS-6-46) Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and 4-methoxyallylbenzene (**1i**, 92 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (60.8 mg, 90%, 6.2:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.10 (m, 5H*+5H'), 7.07 (d, *J* = 8.5 Hz, 2H'), 6.99 (d, *J* = 8.4 Hz, 2H*), 6.81 (d, *J* = 8.5 Hz, 2H'), 6.68 (d, *J* = 8.5 Hz, 2H*), 6.08 (ddd, *J* = 16.9, 10.4, 6.1 Hz, 1H*), 5.98 – 5.89 (m, 1H'), 5.29 – 5.02 (m, 2H*+2H'), 4.62 (s, 2H'), 4.51 (s, 2H*), 4.12 – 4.07 (m, 1H*), 3.93 (dd, *J*

= 7.6, 3.9 Hz, 1H'), 3.78 (s, 3H'), 3.75 (s, 3H*), 3.35 – 3.22 (m, 1H*+1H'), 2.77 – 2.59 (m, 1H*+2H'), 2.51 (dd, *J* = 18.4, 4.3 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.6, 178.2, 176.0, 158.9, 158.7, 138.3, 135.8, 135.6, 135.4, 132.2, 129.9, 129.8, 129.0, 128.8, 128.7, 128.6, 128.0, 127.8, 119.1, 116.2, 114.3, 114.2, 55.4, 55.3, 48.7, 47.6, 45.4, 44.0, 42.5, 42.3, 31.5, 31.2. One peak missing due to overlap.

HRMS (ESI) calcd for $C_{21}H_{22}NO_3$ $[M+H]^+$: 336.15942, found: 336.15950.



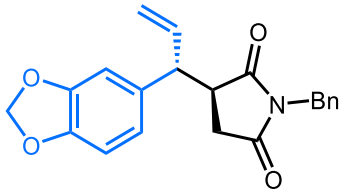
4-(1-(1-Benzyl-2,5-dioxopyrrolidin-3-yl)allyl)phenyl trifluoromethanesulfonate (3ad): (SGS-6-54) Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and 4-allylphenyl trifluoromethanesulfonate (**1d**, 159.7 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a brown oil (65.5 mg, 72%, 5.0:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.13 (m, 7H*+9H'), 7.02 (d, *J* = 8.6 Hz, 2H*), 6.07 – 5.98 (m, 1H*), 5.95 – 5.82 (m, 1H'), 5.36 – 5.06 (m, 2H*+2H'), 4.62 (s, 2H'), 4.52 (q, *J* = 14.0 Hz, 2H*), 4.14 – 4.09k (m, 1H*), 4.01 (dd, *J* = 8.1, 4.3 Hz, 1H'), 3.34 (dt, *J* = 8.9, 4.3 Hz, 1H*), 3.26 (dt, *J* = 9.1, 4.5 Hz, 1H'), 2.83 – 2.70 (m, 1H*+1H'), 2.60 (dd, *J* = 18.4, 4.5 Hz, 1H'), 2.43 (dd, *J* = 18.4, 4.4 Hz, 1H*).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.0, 177.6, 175.5, 175.4, 148.7, 148.6, 141.0, 138.8, 136.7, 135.7, 135.6, 134.1, 130.6, 129.6, 129.1, 128.9, 128.8, 128.7, 128.2, 128.1, 121.9, 121.6, 120.3, 119.9, 48.8, 47.9, 44.9, 43.6, 42.7, 42.5, 31.4, 31.3.

^{19}F NMR (471 MHz, $CDCl_3$) δ −72.85, −72.93.

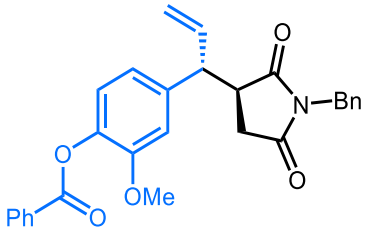
HRMS (ESI) calcd for $C_{21}H_{19}NO_5SF_3$ $[M+H]^+$: 454.09305, found: 454.09191.



3-(1-(Benzo[*d*][1,3]dioxol-5-yl)allyl)-1-benzylpyrrolidine-2,5-dione (3ae): (SGS-6-45) Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and safrole (**1e**, 89 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (50.9 mg, 72%, 7.4:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.13 (m, 5H*+5H'), 6.74 – 6.49 (m, 3H*+3H'), 6.05 (ddd, *J* = 16.9, 10.4, 6.2 Hz, 1H*), 5.92 (d, *J* = 14.2 Hz, 2H*+3H'), 5.27 – 5.03 (m, 1H*+1H'), 4.66 – 4.58 (m, 2H'), 4.57-4.09 (m, 2H*), 4.08 – 4.01 (s, 1H*), 3.89 (dd, *J* = 7.8, 3.8 Hz, 1H'), 3.29 (dt, *J* = 8.6, 4.1 Hz, 1H*), 3.22 (dt, *J* = 8.6, 4.2 Hz, 1H'), 2.72 (dt, *J* = 14.4, 7.2 Hz, 1H*+1H'), 2.62 (dd, *J* = 18.6, 4.3 Hz, 1H'), 2.52 (dd, *J* = 18.5, 4.3 Hz, 1H*).

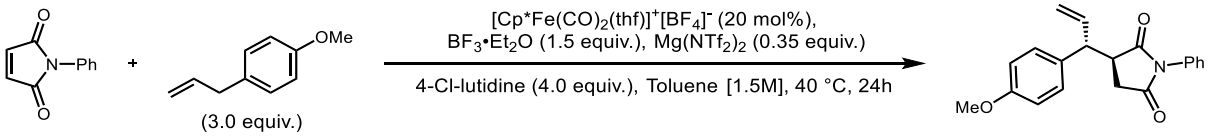
^{13}C NMR (126 MHz, $CDCl_3$) δ 178.4, 178.0, 176.0, 148.1, 147.9, 146.9, 146.7, 138.0, 135.8, 135.6, 135.1, 134.1, 131.8, 128.9, 128.6$_4$, 128.6$_0$, 128.6, 128.0, 127.8, 122.1, 120.8, 119.3, 116.3, 109.1, 108.6, 108.5, 108.2, 101.2, 49.1, 48.0, 45.4, 44.0, 42.5, 42.4, 31.4, 31.2.
HRMS (ESI) calcd for $C_{21}H_{20}NO_4$ $[M+H]^+$: 350.13868, found: 350.13750.



4-(1-(1-Benzyl-2,5-dioxopyrrolidin-3-yl)allyl)-2-methoxyphenyl benzoate (3af): (SGS-6-55) Prepared following **General procedure A** using 1-benzyl-1*H*-pyrrole-2,5-dione (**2a**, 37.4 mg, 0.2 mmol, 1.0 equiv) and 2-methoxy-4-(6-oxo-4,6-diphenylhex-1-en-3-yl)phenyl benzoate (**1f**, 160.9 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 40 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a yellow oil (75.2 mg, 83%, 8.5:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 – 8.18 (m, 2H*+2H'), 7.65 – 7.60 (m, 1H*+1H'), 7.54 – 7.47 (m, 2H*+2H'), 7.36 – 7.20 (m, 3H*+4H'), 7.15 – 7.10 (m, 2H*+1H'), 7.07 (d, *J* = 8.1 Hz, 1H'), 6.98 (d, *J* = 8.1 Hz, 1H*), 6.83 – 6.69 (m, 2H*+2H'), 6.10 (ddd, *J* = 17.0, 10.5, 6.3 Hz, 1H*), 5.91 (ddd, *J* = 17.1, 10.2, 8.6 Hz, 1H'), 5.30 – 5.07 (m, 2H*+2H'), 4.65 – 4.48 (m, 2H*+2H'), 4.11 (s, 1H*), 4.05 (dd, *J* = 8.4, 3.6 Hz, 1H'), 3.75 (s, 3H'), 3.62 (s, 3H*), 3.38 – 3.31 (m, 1H*), 3.31 – 3.25 (m, 1H'), 2.82 – 2.65 (m, 1H*+2H'), 2.57 (dd, *J* = 18.4, 4.7 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 178.3, 178.1, 176.0, 175.8, 164.8, 164.6, 151.6, 151.4, 139.4, 139.3, 139.1, 137.6, 137.1, 135.7, 135.5, 134.2, 133.6, 130.4, 129.5, 128.9, 128.6$_9$, 128.6$_5$, 128.3, 128.0, 127.9, 123.2, 123.1, 120.7, 120.0, 119.6, 116.7, 113.3, 112.3, 56.0, 55.9, 48.9, 48.3, 45.5, 43.9, 42.6, 42.4, 31.5, 31.2. Three peaks missing due to overlap.
HRMS (ESI) calcd for $C_{28}H_{26}NO_5$ $[M+H]^+$: 456.18055, found: 456.17893.

2.1.1 Gram Scale Synthesis

1 mmol scale synthesis of 3-(1-(4-methoxyphenyl)allyl)-1-phenylpyrrolidine-2,5-dione (3fc) (SGS-6-47)



1 mmol scale reaction: A 25 mL round both flask equipped with a magnetic stir bar and septa was flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were sequentially added $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 82.2 mg), $Mg(NTf_2)_2$ (204.6 mg, 0.35 equiv), 1-phenyl-1*H*-pyrrole-2,5-dione (**2f**, 173.2 mg, 1.0 mmol, 1.0 equiv), and dry toluene (0.65 mL, 1.5 M). The solution was briefly stirred. Then 4-allylanisole (**1c**, 0.46 mL, 3.0 equiv), BF_3•Et_2O (186 µL, 1.5 equiv), and 4-Cl-lutidine (0.52 mL, 4.0 equiv) were sequentially added with brief stirring after each addition. The flask was

then sealed and removed from the glovebox and placed in an oil bath at 40 °C for 24 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug using ethyl acetate. The crude mixture was concentrated *in vacuo*. The crude mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a brown oil (311.0 mg, 96%, 4.9:1 isolated d.r.). Succinimide **3fc** was crystallized from a diastereomerically enriched sample and relative stereochemistry was assigned through X-ray diffraction.

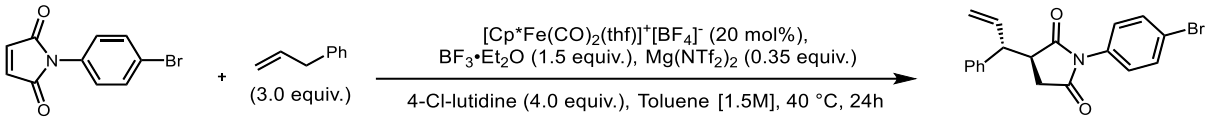
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.26 (m, 3H*+3H'), 7.21 – 7.07 (m, 2H*+4H'), 6.96 (d, *J* = 7.6 Hz, 2H*), 6.89 – 6.79 (m, 2H*+2H'), 6.18 – 6.03 (m, 1H*+1H'), 5.32 – 5.07 (m, 2H*+2H'), 4.22 – 4.14 (m, 1H*), 4.03 (dd, *J* = 8.4, 3.7 Hz, 1H'), 3.76 (s, 3H'), 3.74 (s, 3H*), 3.44 – 3.39 (m, 1H*), 3.38 – 3.34 (m, 1H'), 2.89 – 2.78 (m, 1H*+1H'), 2.74 (dd, *J* = 18.5, 4.4 Hz, 1H'), 2.65 (dd, *J* = 18.5, 3.9 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.0, 177.5, 175.4, 175.2, 159.1, 158.7, 137.9, 135.2, 132.0, 131.9, 131.8, 129.9, 129.7, 129.1_2, 129.0_6, 128.7, 128.6_1, 128.5_8, 126.5, 119.3, 116.3, 114.3, 114.2, 55.3, 48.8, 47.7, 45.4, 44.0, 31.5, 31.2. One peak missing due to overlap.

HRMS (ESI) calcd for $C_{20}H_{20}NO_3$ $[M+H]^+$: 322.14377, found: 322.14220.

m.p. = 93 – 97 °C

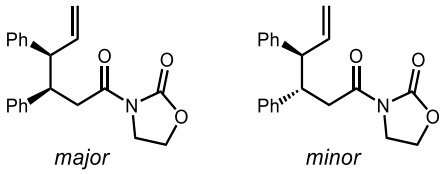
3 mmol scale synthesis of 1-(4-Bromophenyl)-3-(1-phenylallyl)pyrrolidine-2,5-dione (3ka) (SGS-6-82)



3 mmol scale reaction: A 25 mL round both flask equipped with a magnetic stir bar and septa was flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were sequentially added $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 246.6 mg), $Mg(NTf_2)_2$ (613.8 mg, 0.35 equiv), 1-(4-bromophenyl)-1*H*-pyrrole-2,5-dione (**2k**, 752.9 mg, 3.0 mmol, 1.0 equiv), and dry toluene (2.0 mL, 1.5 M). The solution was briefly stirred. Then allylbenzene (**1a**, 1.19 mL, 3.0 equiv), BF_3•Et_2O (558 µL, 1.5 equiv), and 4-Cl-lutidine (1.56 mL, 4.0 equiv) were sequentially added with brief stirring after each addition. The flask was then sealed and removed from the glovebox and placed in an oil bath at 40 °C for 24 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug using ethyl acetate. The crude mixture was concentrated *in vacuo*. The crude mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a brown oil (1.0224 g, 92%, 4.0:1 d.r.).

Characterization data match that of 0.2 mmol scale synthesis entry for **3ka**.

2.2 Synthesis of Michael adducts from Fe-catalyzed Michael addition – Oxazolidinones



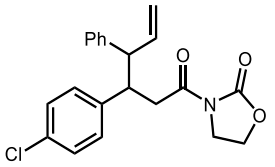
3-(3,4-Diphenylhex-5-enoyl)oxazolidin-2-one (5aa): (SGS-6-21) Prepared following **General procedure B** using 3-cinnamoyloxazolidin-2-one (**4a**, 43.4 mg, 0.2 mmol, 1.0 equiv) and allylbenzene

(**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 3:1) to afford the title compound as a brown oil (58.8 mg, 88%, 2.2:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 6.97 (m, 10H* + 10H'), 6.09 (dt, *J* = 17.0, 9.8 Hz, 1H'), 5.79 (ddd, *J* = 17.1, 10.2, 8.1 Hz, 1H*), 5.13 (dd, *J* = 17.0, 1.1 Hz, 1H'), 5.03 (dd, *J* = 10.0, 1.6 Hz, 1H'), 4.75 (ddd, *J* = 10.3, 1.5, 0.7 Hz, 1H*), 4.70 – 4.61 (m, 1H*), 4.30 (td, *J* = 9.0, 6.7 Hz, 1H'), 4.26 – 4.09 (m, 2H* +1H'), 3.89 – 3.76 (m, 2H'), 3.73 – 3.62 (m, 3H*+ 1H'), 3.60 – 3.48 (m, 1H* + 2H'), 3.46 – 3.34 (m, 1H*+1H'), 3.01 (dd, *J* = 16.8, 4.8 Hz, 1H*). Throughout analysis of diastereomeric mixtures, H* indicates the major diastereomer, while H' indicates the minor diastereomer.

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.4, 172.1, 153.9, 153.4, 142.51, 142.46, 142.4, 142.1, 140.3, 139.8, 128.8, 128.6, 128.5, 128.4, 128.3, 128.0, 126.80, 126.75, 126.4, 126.2, 116.6, 116.2, 62.0, 61.9, 57.3, 56.1, 46.9, 46.5, 42.6, 42.5, 39.71, 39.68.

HRMS (ESI) calcd for $C_{21}H_{22}O_3N$ $[M+H]^+$: 336.15942, found: 336.15835.

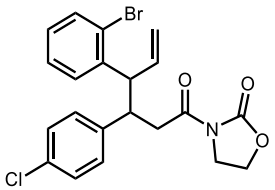


3-(3-(4-Chlorophenyl)-4-phenylhex-5-enoyl)oxazolidin-2-one (5ba): (SGS-6-71). Prepared following **General procedure B** using 3-(3-(4-chlorophenyl)acryloyl)oxazolidin-2-one (**4b**, 43.4 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 3:1) to afford the title compound as a yellow foam (55.2 mg, 75%, 2.3:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.30 (m, 2H*), 7.28 – 7.21 (m, 3H*+4H'), 7.19 – 7.11 (m, 2H*+2H'), 7.10 – 7.03 (m, 1H*+1H'), 7.02 – 6.97 (m, 1H*+2H'), 6.07 (dt, *J* = 17.0, 9.8 Hz, 1H'), 5.78 (ddd, *J* = 17.1, 10.2, 8.4 Hz, 1H*), 5.21 (d, *J* = 16.9 Hz, 1H'), 5.12 (dd, *J* = 10.1, 1.3 Hz, 1H'), 4.86 (d, *J* = 10.3 Hz, 1H*), 4.76 (d, *J* = 17.0 Hz, 1H*), 5.25 – 5.10 (m, 2H'), 4.81 (dd, *J* = 47.8, 13.6 Hz, 2H*), 4.35 – 4.18 (m, 2H*+2H'), 3.91 – 3.78 (m, 1H*), 3.72 (td, *J* = 7.9, 2.0 Hz, 1H*+2H'), 3.69 – 3.62 (m, 1H*+1H'), 3.57 – 3.35 (m, 2H*+3H'), 2.99 (dd, *J* = 17.1, 4.7 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.0, 171.7, 153.5, 153.3, 142.0_4, 142.0_2, 140.8, 140.6, 139.9, 139.3, 132.3, 131.9, 130.0, 129.7, 128.7, 128.3_9, 128.3_5, 128.1, 127.9, 126.8, 126.3, 116.8, 116.5, 62.0, 61.8, 57.1, 55.9, 46.1, 45.7, 42.5, 42.4, 39.4_9, 39.4_5. One peak missing due to overlap.

HRMS (ESI) calcd for $C_{21}H_{21}NO_3Cl$ $[M+H]^+$: 370.12045, found: 370.12052.



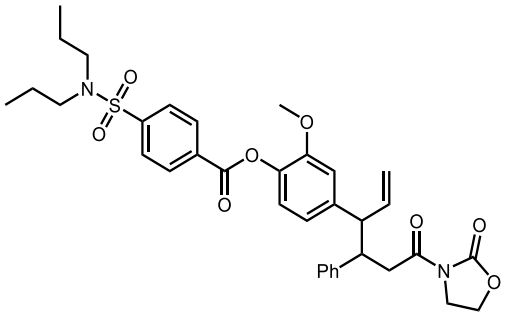
3-(4-(2-Bromophenyl)-3-(4-chlorophenyl)hex-5-enoyl)oxazolidin-2-one (5bg): (SGS-6-77) Prepared following **General procedure B** using 3-(3-(4-chlorophenyl)acryloyl)oxazolidin-2-one (**4b**, 43.4 mg, 0.2 mmol, 1.0 equiv) and 2-bromoallylbenzene (**1g**, 89 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 24 h. The crude reaction mixture was purified by flash column

chromatography (hexanes/ethyl acetate = 3:1) to afford the title compound as a yellow foam (76.5 mg, 85%, 2.3:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (dd, *J* = 8.0, 1.1 Hz, 1H*), 7.36 (dd, *J* = 8.0, 1.0 Hz, 1H'), 7.30 – 7.21 (m, 3H*+2H'), 7.20 – 7.13 (m, 2H*+3H'), 7.12 – 7.06 (m, 2H*+1H'), 6.90 (td, *J* = 8.0, 1.6 Hz, 1H'), 5.92 (dt, *J* = 17.0, 9.7 Hz, 1H'), 5.72 (ddd, *J* = 17.1, 10.2, 8.3 Hz, 1H*), 5.23 (d, *J* = 16.6 Hz, 1H'), 5.07 (dd, *J* = 10.0, 1.2 Hz, 1H'), 4.84 (d, *J* = 10.2 Hz, 1H*), 4.78 (d, *J* = 17.0 Hz, 1H*), 4.36 – 4.18 (m, 3H*+3H'), 3.92 – 3.79 (m, 2H'), 3.79 – 3.72 (m, 2H*+1H'), 3.67 (td, *J* = 10.1, 4.0 Hz, 1H*), 3.50 (dd, *J* = 16.7, 10.4 Hz, 1H*), 3.43 (dd, *J* = 17.2, 9.5 Hz, 1H'), 3.35 (dd, *J* = 17.2, 4.5 Hz, 1H'), 2.85 (dd, *J* = 16.7, 4.0 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 171.6, 153.6, 153.4, 141.1$_1$, 141.0$_5$, 140.3, 138.7, 137.8, 133.4, 133.1, 132.6, 132.2, 130.2, 129.9, 129.2, 128.9, 128.6, 128.3, 128.2, 127.8$_3$, 127.8$_1$, 127.5, 125.5, 124.8, 117.9, 117.6, 62.1, 62.0, 53.9, 53.3, 45.7, 44.7, 42.6, 42.5, 39.8, 39.2. One peak missing due to overlap.

HRMS (ESI) calcd for $C_{21}H_{20}NO_3ClBr$ $[M+H]^+$: 448.03096, found: 448.03139.



2-Methoxy-4-(6-oxo-6-(2-oxooxazolidin-3-yl)-4-phenylhex-1-en-3-yl)phenyl 4-(*N*,*N*-dipropylsulfamoyl)benzoate (5ah): (SGS-6-76) Prepared following **General procedure B** using 3-cinnamoyloxazolidin-2-one (**4a**, 43.4 mg, 0.2 mmol, 1.0 equiv) and 4-allyl-2-methoxyphenyl 4-(*N*,*N*-dipropylsulfamoyl)benzoate (**1h**, 258.9 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 3:1) to afford the title compound as a yellow foam with diastereomers eluting separately as yellow foams with a 1.5:1 d.r. and 89% total yield. (major diastereomers 69.5 mg 53%) (minor diastereomers 46.4 mg, 36%).

Major Isomer

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.3 Hz, 2H), 7.93 (d, *J* = 8.3 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 6.8 Hz, 1H), 7.21 (t, *J* = 7.1 Hz, 1H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.94 – 6.86 (m, 2H), 5.85 – 5.71 (m, 1H), 4.85 (d, *J* = 10.2 Hz, 1H), 4.76 (d, *J* = 17.0 Hz, 1H), 4.29 (q, *J* = 8.5 Hz, 1H), 4.19 (q, *J* = 8.6 Hz, 1H), 3.82 (s, 3H), 3.78 – 3.66 (m, 3H), 3.63 – 3.56 (m, 1H), 3.33 (dd, *J* = 16.6, 6.0 Hz, 1H), 3.24 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.15 – 3.09 (m, 4H), 1.61 – 1.53 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H).

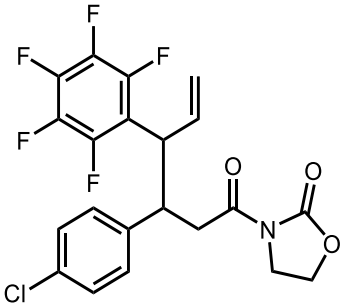
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 163.6, 153.5, 151.0, 144.8, 142.4, 141.6, 139.5, 138.4, 132.8, 131.0, 128.6, 128.4, 127.2, 126.8, 122.6, 120.9, 116.4, 112.9, 62.0, 56.1, 56.0, 50.1, 46.8, 42.5, 39.7, 22.1, 11.3.

HRMS (ESI) calcd for $C_{35}H_{41}N_2O_8S$ $[M+H]^+$: 649.25781, found: 649.25814

Minor Isomer

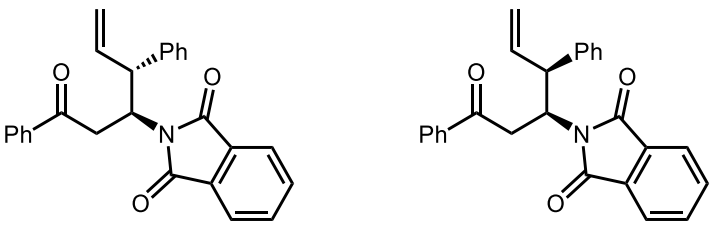
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.5 Hz, 2H), 7.15 (t, *J* = 7.4 Hz, 2H), 7.12 – 7.02 (m, 3H), 6.92 (d, *J* = 8.1 Hz, 1H), 6.69 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.55 (d, *J* = 1.7 Hz, 1H), 6.11 (dt, *J* = 17.0, 9.8 Hz, 1H), 5.23 (d, *J* = 16.9 Hz, 1H), 5.16 (dd, *J* = 10.1, 1.3 Hz, 1H), 4.32 (td, *J* = 9.0, 6.7

Hz, 1H), 4.24 (td, *J* = 9.0, 7.3 Hz, 1H), 3.90 – 3.77 (m, 2H), 3.68 – 3.60 (m, 4H), 3.59 – 3.40 (m, 3H), 3.15 – 3.05 (m, 4H), 1.56 (dq, *J* = 14.9, 7.4 Hz, 4H), 0.88 (t, *J* = 7.4 Hz, 6H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 163.5, 153.6, 150.6, 144.8, 141.9, 141.8, 139.6, 137.9, 132.9, 130.95, 128.5, 128.2, 127.2, 126.6, 122.3, 120.2, 117.1, 112.7, 62.1, 57.1, 56.0, 50.1, 46.7, 42.6, 39.4, 22.1, 11.3.
HRMS (ESI) calcd for $C_{35}H_{41}N_2O_8S$ $[M+H]^+$: 649.25781, found: 649.25785.



3-(3-(4-Chlorophenyl)-4-(perfluorophenyl)hex-5-enoyl)oxazolidin-2-one (5bj): (SGS-7-8) Prepared following **General procedure B** using 3-(3-(4-chlorophenyl)acryloyl)oxazolidin-2-one (**4b**, 43.4 mg, 0.2 mmol, 1.0 equiv) and allylpentafluorobenzene (**1j**, 91 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 24 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 3:1) to afford the title compound as a brown foam (58.2 mg, 63%, 1.4:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H*), 7.21 – 7.09 (m, 2H*+4H'), 6.10 (dt, *J* = 17.1, 9.7 Hz, 1H'), 5.90 – 5.78 (m, 1H*), 5.30 (d, *J* = 16.8 Hz, 1H'), 5.19 (d, *J* = 9.9 Hz, 1H'), 4.85 (d, *J* = 10.1 Hz, 2H*), 4.77 (d, *J* = 16.9 Hz, 2H*), 4.38 – 4.21 (m, 2H*+2H'), 4.03 – 3.94 (m, 1H*+1H'), 3.93 – 3.72 (m, 3H*+3H'), 3.54 – 3.48 (m, 1H*+1H'), 3.39 (dd, *J* = 17.3, 3.9 Hz, 1H'), 2.83 (dd, *J* = 16.7, 3.8 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 170.8, 153.6, 153.4, 139.9, 139.6, 135.8, 135.1, 133.1, 133.0, 130.0, 129.7, 129.1, 129.0, 128.9, 128.8, 120.2, 118.9, 62.2, 62.1, 46.7, 45.9, 43.8, 42.9, 42.6, 42.5, 40.3, 39.6. Peaks missing from complex C-F coupling and overlap.
^{19}F NMR (376 MHz, $CDCl_3$) δ –140.68, –141.75, –155.56 (t, *J* = 21.0 Hz), –156.01 (t, *J* = 21.1 Hz), –161.24 (td, *J* = 22.1, 8.0 Hz), –161.92 (td, *J* = 22.1, 7.7 Hz).
HRMS (ESI) calcd for $C_{21}H_{16}NO_3ClF_5$ $[M+H]^+$: 460.07334, found: 460.07292.

2.3 Synthesis of Michael adducts from Fe-catalyzed Michael addition – Phthalimides

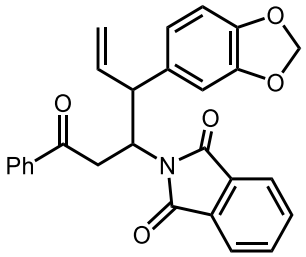


major *minor*

2-(1-Oxo-1,4-diphenylhex-5-en-3-yl)isoindoline-1,3-dione (**7aa**): (SGS-5-144) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2

mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a tan foam (73.2 mg, 93%, 1.2:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 – 7.90 (m, 2H'), 7.86 – 7.10 (m, 14H* + 11H'), 7.06 – 7.00 (m, 1H'), 6.07 (dt, *J* = 16.9, 9.8 Hz, 1H'), 5.95 (dt, *J* = 16.9, 9.9 Hz, 1H*), 5.35 (d, *J* = 16.8 Hz, 1H'), 5.26 – 5.15 (m, 1H* + 2H'), 5.02 (dd, *J* = 16.8, 0.9 Hz, 1H*), 4.86 (dd, *J* = 10.0, 1.4 Hz, 1H*), 4.27 – 4.04 (m, 2H* + 2H'), 3.59 (dd, *J* = 17.8, 3.3 Hz, 1H'), 2.93 (dd, *J* = 17.7, 2.8 Hz, 1H*). Throughout analysis of diastereomeric mixtures, H* indicates the major diastereomer, while H' indicates the minor diastereomer.
^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 197.5, 168.6, 168.2, 140.9, 140.4, 139.0, 138.6, 136.7, 136.5, 134.0, 133.7, 133.4, 133.3, 131.8, 131.5, 129.4, 128.7, 128.6, 128.2, 128.1_2, 128.0_5, 127.8, 127.5, 127.1, 123.3, 123.1, 118.1, 117.2, 53.5_0, 53.4_5, 51.2, 50.2, 39.0, 38.1 (one carbon missing due to overlap). Note: ^{13}C signals for the phthalimide carbonyl peaks are extremely broad for this and similar examples and many scans are required to adequate S/N for these peaks.
HRMS (ESI) calcd for $C_{26}H_{22}O_3N$ $[M+H]^+$: 396.16116, found: 396.15942.

1 mmol scale reaction: A 25 mL round both flask equipped with a magnetic stir bar and septa was flame dried under vacuum and transferred into an argon-filled glovebox. In the glovebox were sequentially added $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol %, 82.2 mg), $LiNTf_2$ (170.0 mg, 0.6 equiv), 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 277.5 mg, 1.0 mmol, 1.0 equiv), and dry DCE (0.65 mL, 1.5 M). The solution was briefly stirred. Then allylbenzene (**1a**, 395 μL, 3.0 equiv), TIPSOTf (670 μL, 1.5 equiv), and collidine (375 μL, 3.0 equiv) were sequentially added with brief stirring after each addition. The flask was then sealed and removed from the glovebox and placed in an oil bath at 80 °C for 16 h at 400 rpm. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered through a silica plug using ethyl acetate. The crude mixture was concentrated *in vacuo*. The crude mixture was purified by flash column chromatography (hexanes/ethyl acetate = 20:1) to afford the title compound as a brown oil (350.4 mg, 89% yield, 1.2:1 d.r.).

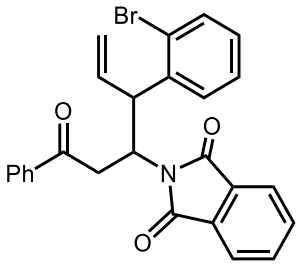


2-(4-(Benzo[*d*][1,3]dioxol-5-yl)-1-oxo-1-phenylhex-5-en-3-yl)isoindoline-1,3-dione (**7ae**): (SGS-6-1) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and safrole (**1e**, 89 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a white foam (62.3 mg, 71%, 1.2:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H'), 7.85 – 7.74 (m, 4H*), 7.70 – 7.63 (m, 2H*+2H'), 7.60 – 7.55 (m, 2H'), 7.55 – 7.50 (m, 1H), 7.50 – 7.45 (m, 1H), 7.44 – 7.39 (m, 2H), 7.39 – 7.34 (m, 2H), 6.89 (d, *J* = 1.6 Hz, 1H*), 6.86 (dd, *J* = 8.0, 1.7 Hz, 1H*), 6.80 (d, *J* = 7.9 Hz, 1H*), 6.74 (d, *J* = 1.7 Hz, 1H'), 6.65 (dd, *J* = 8.0, 1.7 Hz, 1H'), 6.57 (d, *J* = 8.0 Hz, 1H'), 6.00 (dt, *J* = 16.9, 9.8 Hz, 1H*), 5.96 – 5.85

(m, 2H*+ 1H'), 5.80 (dd, *J* = 12.4, 1.5 Hz, 2H'), 5.33 (d, *J* = 16.7 Hz, 1H'), 5.20 – 5.09 (m, 1H*+ 2H'), 5.00 (dd, *J* = 16.8, 0.8 Hz, 1H*), 4.85 (dd, *J* = 10.0, 1.4 Hz, 1H*), 4.17 – 4.01 (m, 2H*+2H'), 3.56 (dd, *J* = 17.8, 3.3 Hz, 1H'), 3.00 (dd, *J* = 17.8, 2.9 Hz, 1H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.6, 197.5, 148.4, 147.7, 146.9, 146.5, 139.1, 138.6, 136.8, 136.6, 134.7, 134.3, 134.0, 133.8, 133.4, 133.3, 131.8, 131.6, 128.7$_4$, 128.6$_5$, 128.2$_1$, 128.1$_5$, 123.2, 121.3, 120.9, 117.9, 117.1, 109.0, 108.4, 108.2, 108.1, 101.3, 101.0, 53.0$_7$, 53.0$_5$, 51.3, 50.4, 39.0, 38.1. One peak missing due to overlap. Phthalimide carbonyls not observed.

HRMS (ESI) calcd for $C_{27}H_{22}NO_5$ $[M+H]^+$: 440.14925, found: 440.14857.

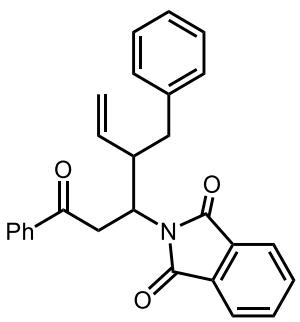


2-(4-(2-Bromophenyl)-1-oxo-1-phenylhex-5-en-3-yl)isoindoline-1,3-dione (**7ag**): (SGS-6-2) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and 2-bromoallylbenzene (**1g**, 89 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a white foam (75.5 mg, 81%, 1.6:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.5 Hz, 2H'), 7.88 – 7.71 (m, 3H*+1H'), 7.71 – 7.51 (m, 4H*+4H'), 7.51 – 7.32 (m, 5H*+4H'), 7.23 (t, *J* = 7.5 Hz, 1H'), 7.10 (t, *J* = 7.3 Hz, 1H*), 6.93 (t, *J* = 7.3 Hz, 1H'), 5.96 – 5.82 (m, 2H*+2H'), 5.46 (d, *J* = 16.9 Hz, 1H'), 5.37 (td, *J* = 11.3, 3.5 Hz, 1H'), 5.31 – 5.20 (m, 1H*), 5.18 (d, *J* = 10.0 Hz, 1H'), 5.13 (d, *J* = 16.8 Hz, 1H*), 4.96 – 4.83 (m, 2H*+1H'), 4.30 (dd, *J* = 17.9, 10.9 Hz, 1H*), 4.13 (dd, *J* = 17.9, 9.7 Hz, 1H'), 3.65 (dd, *J* = 17.9, 3.6 Hz, 1H'), 2.90 (dd, *J* = 17.9, 2.7 Hz, 1H*).

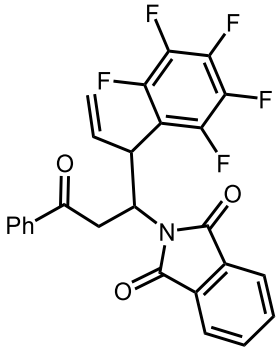
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.5, 197.3, 168.8, 168.0, 140.0, 139.2, 138.0, 137.2, 136.7, 136.5, 134.0, 133.8, 133.6, 133.4, 133.4, 133.2, 131.6, 129.0, 128.7, 128.6, 128.4, 128.3, 128.2, 128.1, 127.8, 125.3, 124.8, 123.7, 123.2, 123.1, 118.8, 118.5, 51.8, 51.3, 50.7, 49.3, 39.2, 37.5. Missing two due to overlap.

HRMS (ESI) calcd for $C_{26}H_{21}NO_3Br$ $[M+H]^+$: 474.06993, found: 474.07021.



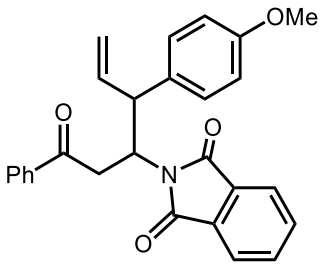
2-(4-Benzyl-1-oxo-1-phenylhex-5-en-3-yl)isoindoline-1,3-dione (7ak): (SGS-6-3) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5

mg, 0.2 mmol, 1.0 equiv) and 4-phenyl-1-butene (**1k**, 90 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a white foam (53.8 mg, 66%, 1.0:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 – 7.85 (m, 2H*+2H'), 7.79 – 7.72 (m, 2H*+2H'), 7.68 – 7.63 (m, 2H*+2H'), 7.56 – 7.49 (m, 1H*+1H'), 7.44 – 7.39 (m, 2H*+2H'), 7.29 – 7.23 (m, 1H*+1H'), 7.17 (t, *J* = 7.4 Hz, 1H*+2H'), 7.10 (t, *J* = 7.5 Hz, 1H*+1H'), 7.03 (d, *J* = 7.2 Hz, 1H*+1H'), 6.98 (t, *J* = 7.3 Hz, 1H*), 5.68 – 5.51 (m, 1H*+1H'), 5.10 – 5.00 (m, 1H*+1H'), 4.97 (td, *J* = 9.7, 3.8 Hz, 1H'), 4.88 (td, *J* = 10.5, 3.4 Hz, 1H*), 4.75 (ddd, *J* = 18.3, 13.6, 1.5 Hz, 1H*+1H'), 4.19 (dd, *J* = 17.8, 9.8 Hz, 1H*), 4.03 (dd, *J* = 17.9, 10.3 Hz, 1H'), 3.53 – 3.49 (m, 1H*), 3.49 – 3.44 (m, 1H'), 3.30 (qd, *J* = 9.6, 5.3 Hz, 1H'), 3.16 (qd, *J* = 9.9, 4.1 Hz, 1H*), 3.01 (dd, *J* = 13.7, 4.1 Hz, 1H'), 2.76 (dd, *J* = 13.9, 5.2 Hz, 1H*), 2.65 – 2.53 (m, 1H*+1H').
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.7, 197.4, 168.5$_8$, 168.5$_7$, 139.4, 139.3, 138.7, 137.9, 136.6, 136.7, 133.9, 133.4, 133.3, 131.8, 131.8, 129.3, 129.1, 128.7, 128.7, 128.3, 128.2, 128.1$_4$, 128.1$_0$, 126.6, 125.8, 123.3, 119.0, 118.3, 50.6, 50.4, 49.4, 48.3, 39.1, 38.8, 38.7, 38.6.
HRMS (ESI) calcd for $C_{27}H_{24}NO_3$ $[M+H]^+$: 410.17507, found: 410.17352.

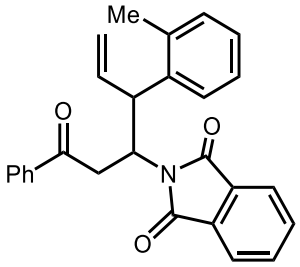


2-(1-Oxo-4-(perfluorophenyl)-1-phenylhex-5-en-3-yl)isoindoline-1,3-dione (7aj): (SGS-6-4) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and allylpentafluorobenzene (**1j**, 91 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a white foam (72.1 mg, 74%, 1.2:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.89 (m, 2H'), 7.87 – 7.75 (m, 3H*+1H'), 7.75 – 7.68 (m, 3H*+1H'), 7.67 – 7.62 (m, 2H'), 7.57 – 7.49 (m, 1H*+1H'), 7.45 – 7.36 (m, 2H*+2H'), 6.25 – 6.15 (m, 1H'), 6.15 – 6.04 (m, 1H*), 5.51 – 5.40 (m, 1H*+2H'), 5.32 (d, *J* = 9.9 Hz, 1H'), 5.16 (d, *J* = 16.8 Hz, 1H*), 5.02 (d, *J* = 10.0 Hz, 1H*), 4.64 (t, *J* = 10.5 Hz, 1H*+1H'), 4.14 (dd, *J* = 17.6, 9.8 Hz, 1H*+1H'), 3.57 (dd, *J* = 18.0, 3.8 Hz, 1H'), 3.04 (dd, *J* = 17.6, 4.0 Hz, 1H*).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 196.3, 168.8, 167.9, 146.7 – 146.2 (m), 144.2 – 143.8 (m), 142.1 – 141.3 (m), 139.7 – 138.4 (m), 136.9 – 136.6 (m), 136.5, 136.3, 136.2 – 135.8 (m), 134.2$_3$, 134.2$_1$, 133.9, 133.6, 133.6, 131.6, 131.3, 128.7$_9$, 128.7$_7$, 128.2, 128.1, 123.7, 121.4, 120.5, 114.9 – 113.2 (m), 48.6, 48.5, 43.8, 43.2, 38.8, 38.4. Carbon spectra was run an extended time to attempt to resolve low intensity peaks, however, some carbons are still missing due to complex C-F coupling and overlap.
^{19}F NMR (471 MHz, $CDCl_3$) δ −140.61, −154.70 (t, *J* = 20.9 Hz), −155.09 (t, *J* = 21.0 Hz), −160.77 (td, *J* = 21.9, 7.9 Hz), −161.33 (td, *J* = 22.0, 7.8 Hz). One peak missing due to overlap.

HRMS (ESI) calcd for $C_{26}H_{17}NO_3F_5$ $[M+H]^+$: 486.11231, found: 486.11205.

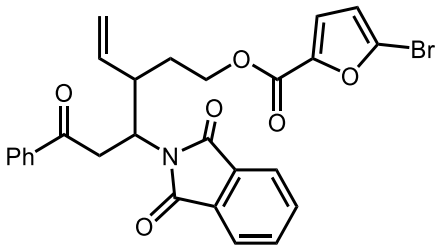


2-(4-(4-Methoxyphenyl)-1-oxo-1-phenylhex-5-en-3-yl)isoindoline-1,3-dione (7ac): (SGS-6-5) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and allylanisole (**1c**, 92 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a clear oil (75.2 mg, 88%, 1.5:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 – 7.90 (m, 2H'), 7.80 (s (br), 1H*+1H'), 7.77 – 7.73 (m, 2H*), 7.70 – 7.60 (m, 2H*+2H'), 7.58 – 7.49 (m, 2H*+1H'), 7.47 (t, *J* = 7.4 Hz, 1H*), 7.42 (t, *J* = 7.7 Hz, 2H'), 7.38 – 7.29 (m, 2H*+2H'), 7.16 – 7.11 (m, 2H'), 6.94 – 6.88 (m, 2H*), 6.71 – 6.66 (m, 2H'), 6.04 (dt, *J* = 17.0, 9.8 Hz, 1H'), 5.93 (dt, *J* = 16.9, 9.8 Hz, 1H*), 5.32 (d, *J* = 16.9 Hz, 1H'), 5.22 – 5.14 (m, 1H*+2H'), 5.00 (dd, *J* = 16.8, 0.9 Hz, 1H*), 4.84 (dd, *J* = 10.0, 1.4 Hz, 1H*), 4.21 – 4.05 (m, 2H*+2H'), 3.79 (s, 3H*), 3.65 (s, 3H'), 3.59 (dd, *J* = 17.7, 3.2 Hz, 1H'), 2.98 (dd, *J* = 17.7, 2.8 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.7, 197.5, 158.9, 158.5, 139.4, 138.9, 136.8, 136.6, 133.9, 133.7, 133.4, 133.3, 132.9, 132.5, 131.9, 131.5, 129.0, 128.7, 128.6, 128.2, 128.1, 123.1, 117.6, 116.8, 114.8, 114.1, 55.4, 55.2, 52.6, 52.6, 51.4, 50.4, 39.1, 38.1. One peak missing due to overlap. Phthalimide carbonyls not observed.
HRMS (ESI) calcd for $C_{27}H_{24}NO_4$ $[M+H]^+$: 426.16961, found: 426.16998.

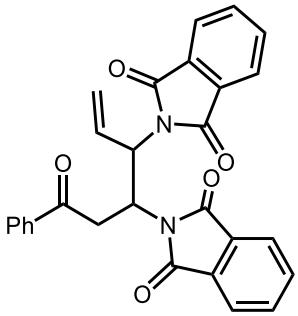


2-(1-Oxo-1-phenyl-4-(o-tolyl)hex-5-en-3-yl)isoindoline-1,3-dione (7ai): (SGS-6-6) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and 2-methylallylbenzene (**1i**, 88 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a clear oil (68.2 mg, 83%, 1.8:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.5 Hz, 2H'), 7.90 – 7.71 (m, 3H*+2H'), 7.70 – 7.65 (m, 2H*), 7.65 – 7.61 (m, 2H'), 7.57 – 7.50 (m, 1H*+1H'), 7.50 – 7.39 (m, 2H*+2H'), 7.38 – 7.31 (m, 2H*+1H'), 7.26 – 7.18 (m, 2H*), 7.15 (t, *J* = 7.3 Hz, 1H*), 7.09 (t, *J* = 7.2 Hz, 1H'), 6.99 – 6.91 (m, 2H'), 5.95 – 5.83 (m, 1H*+1H'), 5.38 – 5.24 (m, 1H*+2H'), 5.13 (d, *J* = 10.0 Hz, 1H'), 5.00 (d, *J* = 16.9 Hz, 1H*), 4.85 (d,

J = 10.0 Hz, 1H*), 4.58 (t, *J* = 10.4 Hz, 1H'), 4.53 – 4.45 (t, *J* = 10.4 Hz, 1H*), 4.17 – 4.02 (m, 1H*+1H'), 3.65 (dd, *J* = 17.6, 3.2 Hz, 1H'), 2.92 (dd, *J* = 17.5, 2.5 Hz, 1H*), 2.51 (s, 3H*), 2.33 (s, 3H').
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.6, 197.4, 169.0, 168.0, 139.3, 138.7, 138.4, 138.0, 136.7, 136.4, 136.4, 136.2, 133.9, 133.7, 133.3, 133.2, 131.0, 130.6, 128.6, 128.5, 128.1, 128.0, 127.0, 126.9, 126.8, 126.5, 126.4, 126.2, 123.6, 123.0, 117.4, 117.0, 51.3, 49.3, 48.8, 47.6, 39.2, 37.4, 20.1, 19.4.
HRMS (ESI) calcd for $C_{27}H_{24}NO_3$ $[M+H]^+$: 410.17507, found: 410.17531.



4-(1,3-Dioxoisoindolin-2-yl)-6-oxo-6-phenyl-3-vinylhexyl 5-bromofuran-2-carboxylate (7an): (SGS-6-7) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and pent-4-en-1-yl 5-bromofuran-2-carboxylate (**1n**, 155.0 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a clear oil (44.0 mg, 41%, 1.1:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 – 7.90 (m, 2H'), 7.89 – 7.85 (m, 2H*), 7.83 – 7.79 (m, 2H*), 7.79 – 7.75 (m, 2H'), 7.72 – 7.68 (m, 2H*), 7.68 – 7.63 (m, 2H'), 7.52 (dd, *J* = 15.7, 7.5 Hz, 1H*+1H'), 7.41 (dd, *J* = 16.4, 8.2 Hz, 2H*+2H'), 7.13 (d, *J* = 3.5 Hz, 1H'), 7.08 (d, *J* = 3.5 Hz, 1H*), 6.46 (d, *J* = 3.5 Hz, 1H'), 6.43 (d, *J* = 3.5 Hz, 1H*), 5.68 – 5.55 (m, 1H*+1H'), 5.35 – 5.24 (m, 2H*), 5.07 – 4.98 (m, 2H'), 4.90 (td, *J* = 9.6, 3.9 Hz, 1H'), 4.80 (td, *J* = 10.5, 3.5 Hz, 1H*), 4.39 – 4.33 (m, 1H'), 4.32 – 4.23 (m, 1H*+1H'), 4.20 – 4.13 (m, 1H*+1H'), 4.02 (dd, *J* = 18.0, 10.2 Hz, 1H*), 3.50 – 3.38 (m, 1H*+1H'), 3.07 (qd, *J* = 10.9, 2.9 Hz, 1H*), 2.98 (qd, *J* = 10.2, 3.2 Hz, 1H'), 2.14 – 2.05 (m, 1H'), 1.89 – 1.79 (m, 1H*), 1.79 – 1.70 (m, 1H'), 1.69 – 1.60 (m, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.6, 197.3, 168.6, 168.5, 157.6, 157.5, 146.4, 138.0, 137.3, 136.7, 136.6, 134.2, 134.0, 133.5, 133.4, 131.8, 131.6, 128.8, 128.7, 128.2$_1$, 128.1$_7$, 127.8, 127.7, 123.5, 123.4, 120.3, 120.2, 120.1, 119.1, 114.1, 114.0, 63.3, 62.9, 50.3, 50.0, 44.8, 44.1, 38.9, 38.3, 30.7, 30.0. One peak missing due to overlap.
HRMS (ESI) calcd for $C_{27}H_{23}NO_6Br$ $[M+H]^+$: 536.07033, found: 536.07027.



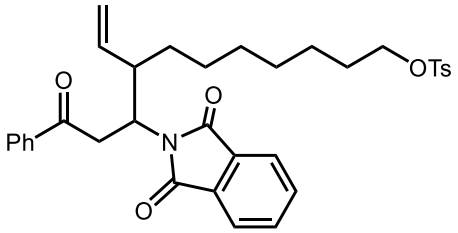
2,2'-(6-Oxo-6-phenylhex-1-ene-3,4-diyl)bis(isoindoline-1,3-dione) (7ap): (SGS-6-8) Prepared following **General procedure C** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5

mg, 0.2 mmol, 1.0 equiv), 2-allylisoindoline-1,3-dione (**1p**, 124.7 mg, 0.6 mmol, 3.0 equiv), and $LiNTf_2$ (46.0 mg, 0.16 mmol, 0.8 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (69.4 mg, 75%, 1.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.87 (m, 2H'), 7.86 – 7.77 (m, 2H*+1H'), 7.75 – 7.65 (m, 4H*+4H'), 7.66 – 7.61 (m, 2H), 7.61 – 7.57 (m, 2H), 7.53 (t, *J* = 7.4 Hz, 1H'), 7.47 (t, *J* = 7.4 Hz, 1H*), 7.42 (t, *J* = 7.7 Hz, 2H'), 7.35 (t, *J* = 7.8 Hz, 2H*), 6.58 (dt, *J* = 17.1, 9.9 Hz, 1H'), 6.42 (dt, *J* = 17.1, 9.8 Hz, 1H*), 5.73 (td, *J* = 9.8, 4.1 Hz, 1H'), 5.66 – 5.55 (m, 1H*+1H'), 5.49 – 5.37 (m, 1H*+2H'), 5.30 (d, *J* = 17.1 Hz, 1H*), 5.14 (d, *J* = 10.2 Hz, 1H*), 4.25 – 4.13 (m, 1H*+1H'), 3.49 (dd, *J* = 18.1, 3.9 Hz, 1H*), 3.34 (dd, *J* = 17.8, 4.1 Hz, 1H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 196.7, 196.5, 168.3, 168.0, 167.8, 136.7, 136.5, 134.4, 134.2, 134.1, 133.6, 133.5, 132.5, 132.1, 131.9_3, 131.9_0, 131.8, 131.7, 128.9, 128.8, 128.3_0, 128.2_9, 123.7, 123.6, 123.6, 122.6, 121.6, 56.5, 55.9, 48.1_0, 48.0_5, 37.9, 37.5. Three peaks missing due to overlap.

HRMS (ESI) calcd for $C_{28}H_{21}N_2O_5$ $[M+H]^+$: 465.14450, found: 465.14344.

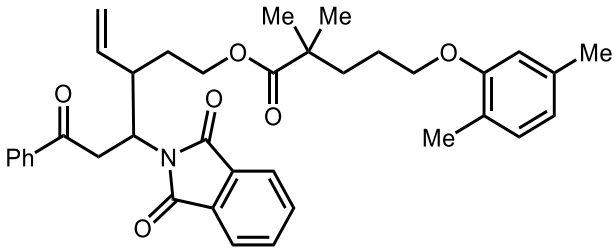


9-(1,3-Dioxoisoindolin-2-yl)-11-oxo-11-phenyl-8-vinylundecyl 4-methylbenzenesulfonate (7am): (SGS-6-18) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and dec-9-en-1-yl 4-methylbenzenesulfonate (**1m**, 279.4 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a brown oil (91.8 mg, 78%, 1.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.80 (m, 2H*), 7.80 – 7.76 (m, 2H'), 7.74 – 7.63 (m, 4H*+4H'), 7.61 – 7.57 (m, 2H'), 7.57 – 7.53 (m, 2H*), 7.46 – 7.38 (m, 1H*+1H'), 7.35 – 7.28 (m, 2H*+2H'), 7.26 – 7.21 (m, 2H*+2H'), 5.43 (ddt, *J* = 27.1, 17.0, 10.0 Hz, 1H*+1H'), 5.17 – 5.06 (m, 2H'), 4.87 – 4.76 (m, 2H*), 4.70 (td, *J* = 9.9, 3.8 Hz, 1H*), 4.62 (td, *J* = 10.5, 3.4 Hz, 1H'), 4.01 (dd, *J* = 17.8, 9.9 Hz, 1H*), 3.95 – 3.80 (m, 2H*+3H'), 3.35 (dd, *J* = 17.9, 3.4 Hz, 1H'), 3.29 (dd, *J* = 17.8, 3.8 Hz, 1H*), 2.80 – 2.63 (m, 1H*+1H'), 2.34 (s, 3H*), 2.34 (s, 3H'), 1.56 – 1.37 (m, 4H*+4H'), 1.28 – 0.93 (m, 10H*+10H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 198.0, 197.7, 168.6, 144.8, 144.7, 139.5, 138.9, 136.8, 134.1, 133.9, 133.4, 133.3, 131.9, 131.8, 129.9_4, 129.9_2, 128.8, 128.7, 128.2_1, 128.1_8, 128.0_2, 128.0_1, 123.4, 123.3, 118.8, 117.8, 70.8, 50.6, 50.3, 47.7, 47.0, 38.9, 38.5, 31.8, 30.9, 29.5, 29.2, 29.0, 28.9_2, 28.9_0, 28.9, 27.2, 26.7, 25.4, 25.3, 21.8. Four peaks missing due to overlap.

HRMS (ESI) calcd for $C_{34}H_{38}NO_6S$ $[M+H]^+$: 588.24144, found: 588.24084.

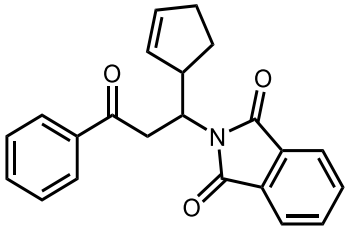


4-(1,3-Dioxoisoindolin-2-yl)-6-oxo-6-phenyl-3-vinylhexyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (7aq): (SGS-6-17) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and pent-4-en-1-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (**1q**, 273.9 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (71.4 mg, 60%, 1.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.81 (m, 2H'), 7.81 – 7.77 (m, 2H*), 7.71 – 7.65 (m, 2H*+2H'), 7.60 – 7.54 (m, 2H*+2H'), 7.47 – 7.40 (m, 1H*+1H'), 7.34 – 7.30 (m, 2H*+2H'), 6.90 (dd, *J* = 7.4, 3.2 Hz, 1H*+1H'), 6.60 – 6.55 (m, 1H*+1H'), 6.53 (d, *J* = 11.4 Hz, 1H*+1H'), 5.57 – 5.43 (m, 1H*+1H'), 5.26 – 5.15 (m, 2H*), 4.95 – 4.87 (m, 2H'), 4.79 (td, *J* = 9.7, 3.9 Hz, 1H'), 4.70 (td, *J* = 10.5, 3.5 Hz, 1H*), 4.11 – 3.75 (m, 5H*+5H'), 3.38 (dd, *J* = 18.0, 3.5 Hz, 1H*), 3.30 (dd, *J* = 17.8, 3.9 Hz, 1H'), 2.97 (qd, *J* = 11.1, 2.9 Hz, 1H*), 2.87 (qd, *J* = 10.4, 3.0 Hz, 1H'), 2.22 (s, 3H*), 2.21 (s, 3H'), 2.08 (s, 3H'), 2.05 (s, 3H*), 1.95 – 1.86 (m, 1H'), 1.71 – 1.39 (m, 6H*+5H'), 1.16 (s, 6H'), 1.09 (s, 6H*).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.7, 197.3, 177.8, 177.7, 168.6, 168.5, 157.1, 138.1, 137.5, 136.7_1, 136.6_7, 136.5_5, 136.5_3, 134.1_0, 133.9_6, 133.5, 133.4, 131.8, 131.7, 130.4_0, 130.3_9, 128.8, 128.7, 128.1_7, 128.1_5, 123.7, 123.4, 123.3, 120.8_1, 120.7_7, 120.0, 118.9, 112.1_2, 112.0_9, 68.0, 62.4, 61.9, 50.3, 50.0, 44.8, 44.1, 42.2, 42.1, 38.9, 38.3, 37.2, 37.1, 30.6, 30.0, 25.4, 25.3_3, 25.2_9, 25.3, 25.2, 21.5, 15.8_9, 15.8_6. Five peaks missing due to overlap.

HRMS (ESI) calcd for $C_{37}H_{42}NO_6$ $[M+H]^+$: 596.30066, found: 596.30116.



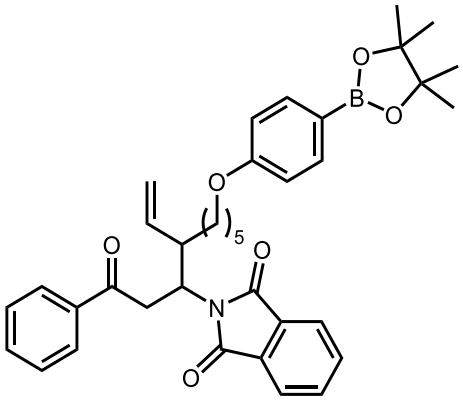
2-(1-(Cyclopent-2-en-1-yl)-3-oxo-3-phenylpropyl)isoindoline-1,3-dione (7al): (SGS-6-23) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and cyclopentene (**1l**, 53 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow oil (45.3 mg, 65%, 1.2:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.6 Hz, 2H*+2H'), 7.82 – 7.76 (m, 2H*+2H'), 7.71 – 7.64 (m, 2H*+2H'), 7.52 (t, *J* = 7.2 Hz, 1H*+1H'), 7.42 (t, *J* = 7.5 Hz, 2H*+2H'), 5.96 – 5.49 (m, 2H*+2H'), 4.79 – 4.66 (m, 1H*+1H'), 4.30 – 4.16 (m, 1H*+1H'), 3.56 – 3.43 (m, 1H*+1H'), 3.37 – 3.27 (m,

1H*+1H'), 2.51 – 2.22 (m, 2H*+2H'), 2.21 – 2.11 (m, 1H'), 1.99 – 1.89 (m, 1H*), 1.75 – 1.62 (m, 1H*+1H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.8, 168.9, 168.7, 136.9, 134.2, 134.0, 133.3, 132.0, 130.8_9, 130.8_7, 128., 128.2, 123.4, 123.3, 51.8, 51.5, 48.9_3, 48.8_6, 38.7, 38.6, 32.4, 31.7, 27.9, 27.2. Eight peaks missing due to overlap.

HRMS (ESI) calcd for $C_{22}H_{20}NO_3$ $[M+H]^+$: 346.14377, found: 346.14273.



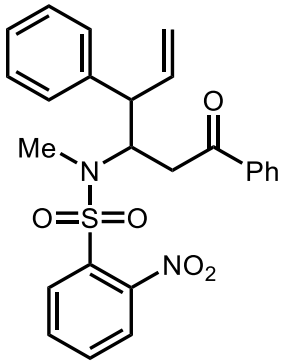
2-(1-Oxo-1-phenyl-9-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenoxy)-4-vinylnonan-3-yl)isoindoline-1,3-dione (7ao): (SGS-6-53) Prepared following **General procedure D** using 2-(3-oxo-3-phenylprop-1-en-1-yl)isoindoline-1,3-dione (**6a**, 55.5 mg, 0.2 mmol, 1.0 equiv) and 4,4,5,5-tetramethyl-2-(4-(oct-7-en-1-yloxy)phenyl)-1,3,2-dioxaborolane (**1o**, 198.2 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 80 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 5:1) to afford the title compound as a tan foam (75.9 mg, 62%, 1.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.5 Hz, 2H'), 7.88 (d, *J* = 7.5 Hz, 2H*), 7.83 – 7.61 (m, 6H*+6H'), 7.56 – 7.47 (m, 1H*+1H'), 7.46 – 7.36 (m, 2H*+2H'), 6.87 (d, *J* = 8.3 Hz, 2H*), 6.82 (d, *J* = 8.4 Hz, 2H'), 5.64 – 5.47 (m, 1H*+1H'), 5.22 (dd, *J* = 20.2, 13.6 Hz, 2H'), 4.99 – 4.88 (m, 2H*), 4.73 (td, *J* = 10.4, 3.1 Hz, 1H'), 4.81 (td, *J* = 9.9, 3.5 Hz, 1H*), 4.73 (td, *J* = 10.4, 3.1 Hz, 1H'), 4.13 (dd, *J* = 17.7, 10.0 Hz, 1H*), 3.99 (m, 2H*+1H'), 3.88 (t, *J* = 6.4 Hz, 2H'), 3.49 – 3.37 (m, 1H*+1H'), 2.92 – 2.76 (m, 1H*+1H'), 1.83 – 1.16 (m, 20H*+20H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.9, 197.6, 168.6, 161.7_9, 161.7_7, 139.4, 138.8, 136.7_9, 136.7_6, 136.6, 136.5, 134.0, 133.9, 133.4, 133.3, 131.8_4, 131.7_7, 128.7_2, 128.6_6, 128.2, 128.1, 123.4, 123.2, 118.9, 117.8, 114.0, 113.9, 83.6, 67.6_9, 67.6_6, 50.6, 50.3, 47.6, 47.0, 38.9, 38.4, 31.7, 30.9, 29.2_3, 29.1_5, 27.1, 26.7, 26.1, 25.9, 25.0. Three peaks missing due to overlap.

HRMS (ESI) calcd for $C_{37}H_{43}BNO_6$ $[M+H]^+$: 608.31779, found: 608.31985.

2.4 Synthesis of Michael adducts from Fe-catalyzed Michael addition – Sulfonamides

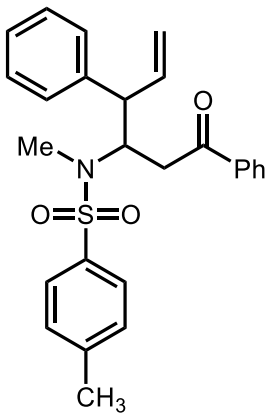


***N*-Methyl-2-nitro-*N*-(1-oxo-1,4-diphenylhex-5-en-3-yl)benzenesulfonamide (9aa):** (SGS-5-184) Prepared following **General procedure E** using *N*-methyl-2-nitro-*N*-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (**8a**, 69.3 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 5:1) to afford the title compound as a tan foam (66.9 mg, 72%, 1.2:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 – 7.94 (m, 1H'), 7.84 – 7.80 (m, 1H*+1H'), 7.74 (d, *J* = 7.9 Hz, 1H*), 7.56 – 7.38 (m, 6H*+6H'), 7.29 – 7.23 (m, 3H*+3H'), 7.18 – 7.12 (m, 1H*+2H'), 7.10 – 7.05 (m, 2H*+1H'), 6.11 (dt, *J* = 16.9, 9.7 Hz, 1H'), 5.87 (dt, *J* = 16.9, 9.9 Hz, 1H*), 5.10 (d, *J* = 4.9 Hz, 1H*), 5.06 (d, *J* = 5.4 Hz, 1H'), 5.02 – 4.90 (m, 1H*+2H'), 4.87 (dd, *J* = 10.0, 1.2 Hz, 1H*), 3.65 – 3.57 (m, 2H'), 3.46 (dd, *J* = 17.4, 6.7 Hz, 1H*), 3.25 (dd, *J* = 17.4, 4.2 Hz, 1H*), 3.05 (s, 3H'), 3.03 – 2.90 (m, 4H*+1H'). Throughout analysis of diastereomeric mixtures, H* indicates the major diastereomer, while H' indicates the minor diastereomer.

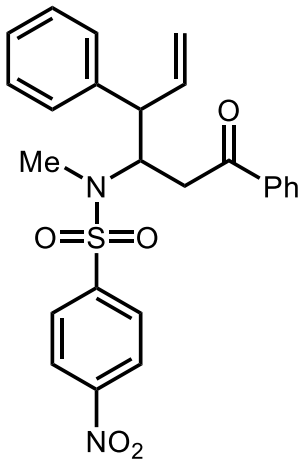
^{13}C NMR (126 MHz, $CDCl_3$) δ 196.8$_3$, 196.7$_6$, 147.9, 147.6, 140.8, 140.5, 138.6, 138.3, 136.6, 136.2, 133.9, 133.4, 133.3, 133.2$_4$, 133.1$_6$, 131.7, 131.5, 131.4, 131.3, 129.2, 128.7$_3$, 128.6$_5$, 128.5, 128.4, 128.1, 128.0, 127.7, 127.5, 126.9, 124.4, 123.7, 117.7, 117.0, 59.1, 57.7, 55.6, 54.9, 41.0, 39.4. Two peaks missing due to overlap.

HRMS (ESI) calcd for $C_{25}H_{25}N_2O_5S$ $[M+H]^+$: 465.14787, found: 465.14707.

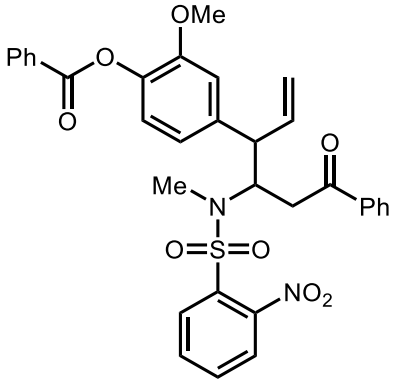


***N*,4-Dimethyl-*N*-(1-oxo-1,4-diphenylhex-5-en-3-yl)benzenesulfonamide (9ca):** (SGS-6-20) Prepared following **General procedure E** using *N*,4-dimethyl-*N*-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (**8c**, 63.1 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by

flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a tan foam (48.3 mg, 56%, 1.2:1 d.r.).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H'), 7.59 – 7.04 (m, 14H*+10H'), 6.98 (d, *J* = 7.9 Hz, 2H'), 6.24 – 6.07 (m, 1H*), 5.93 (dt, *J* = 17.2, 9.8 Hz, 1H'), 5.16 – 4.83 (m, 3H*+3H'), 3.69 – 3.52 (m, 1H*+1H'), 3.30 (dd, *J* = 17.3, 6.3 Hz, 1H'), 3.03 (dd, *J* = 17.3, 5.1 Hz, 1H'), 2.96 – 2.75 (m, 5H*), 2.68 (s, 3H'), 2.29 (s, 3H*), 2.24 (s, 3H').
^{13}C NMR (101 MHz, $CDCl_3$) δ 197.1, 196.9, 142.9, 142.9, 141.4, 140.9, 139.1, 138.8, 137.6, 136.8, 136.7, 136.3, 133.2, 133.1, 129.5, 129.4, 129.1, 128.8, 128.6, 128.5, 128.4, 128.1, 128.0, 127.4, 127.3, 127.0, 117.4, 116.8, 59.3, 57.6, 56.1, 55.1, 40.0, 39.6, 31.5, 30.5, 21.5_1, 21.4_6. Two peaks missing due to overlap.
HRMS (ESI) calcd for $C_{26}H_{28}NO_3S$ $[M+H]^+$: 434.17844, found: 434.17736.



***N*-Methyl-4-nitro-*N*-(1-oxo-1,4-diphenylhex-5-en-3-yl)benzenesulfonamide (9ba):** (SGS-5-168-8) Prepared following **General procedure E** using *N*-methyl-4-nitro-*N*-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (**8b**, 69.3 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a tan foam (62.2 mg, 67%, 1.1:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 – 7.98 (m, 2H*), 7.93 – 7.88 (m, 2H'), 7.75 – 7.71 (m, 2H*), 7.69 – 7.65 (m, 2H'), 7.51 – 7.14 (m, 10H*+10H'), 6.14 (dt, *J* = 16.9, 9.8 Hz, 1H*), 5.90 (dt, *J* = 16.9, 9.9 Hz, 1H'), 5.15 – 4.92 (m, 2H*+2H'), 4.92 – 4.82 (m, 1H*+1H'), 3.61 (t, *J* = 10.0 Hz, 1H'), 3.50 (t, *J* = 9.9 Hz, 1H*), 3.31 (dd, *J* = 17.2, 5.3 Hz, 1H'), 3.11 (dd, *J* = 17.2, 6.8 Hz, 1H'), 2.90 (s, 3H*), 2.82 – 2.77 (m, 2H*), 2.74 (s, 3H').
^{13}C NMR (126 MHz, $CDCl_3$) δ 196.8, 196.7, 149.7, 149.6, 146.3, 145.7, 141.1, 140.7, 138.6, 138.1, 136.2, 135.7, 133.7, 129.4, 129.0, 128.8, 128.7, 128.4, 128.3, 128.3, 128.1, 128.0_1, 127.9_9, 127.7, 127.3, 124.0, 118.2, 117.4, 60.1, 58.9, 55.8, 55.3, 40.0, 39.1. Two peaks missing due to overlap.
HRMS (ESI) calcd for $C_{25}H_{25}N_2O_5S$ $[M+H]^+$: 465.14787, found: 465.14648.

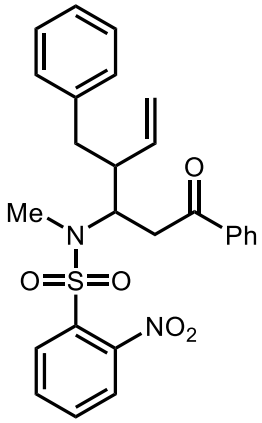


2-Methoxy-4-(4-((*N*-methyl-2-nitrophenyl)sulfonamido)-6-oxo-6-phenylhex-1-en-3-yl)phenyl benzoate (9af): (SGS-6-30) Prepared following **General procedure E** using *N*-methyl-2-nitro-*N*-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (**8a**, 69.3 mg, 0.2 mmol, 1.0 equiv) and 2-methoxy-4-(6-oxo-4,6-diphenylhex-1-en-3-yl)phenyl benzoate (**1f**, 160.9 mg, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a brown foam (96.4 mg, 78%, 1.6:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (t, *J* = 6.5 Hz, 2H*+2H'), 7.97 – 7.91 (m, 1H'), 7.83 (d, *J* = 7.6 Hz, 1H*+1H'), 7.77 – 7.71 (m, 1H*), 7.67 – 7.31 (m, 10H*+10H'), 7.06 (d, *J* = 8.0 Hz, 1H'), 6.97 – 6.83 (m, 2H*+2H'), 6.76 (d, *J* = 7.9 Hz, 1H*), 6.15 – 6.04 (m, 1H'), 5.95 – 5.84 (m, 1H*), 5.18 – 5.06 (m, 1H*+1H'), 5.04 – 4.89 (m, 2H*+2H'), 3.79 (s, 3H'), 3.78 (s, 3H*), 3.73 – 3.59 (m, 2H'), 3.49 (dd, *J* = 17.2, 6.2 Hz, 1H*), 3.33 – 3.23 (m, 1H*), 3.15 – 2.92 (m, 4H*+4H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.0, 196.9, 164.7, 151.8, 151.4, 148.0, 147.8, 139.8, 139.5, 139.3, 138.9, 138.3, 137.9, 136.5, 136.2, 133.8, 133.7, 133.6, 133.5, 133.4, 133.3_3, 133.2_6, 133.0, 131.7, 131.5, 131.1 131.0, 130.4, 129.6, 129.5, 128.8, 128.7, 128.6_3, 128.6_0, 128.1_1, 128.1_0, 124.5, 123.7, 123.4, 122.9, 120.6, 119.6, 118.0, 117.2, 112.5, 59.2, 58.0, 56.2, 56.1, 55.4, 54.7, 40.9, 39.6.

HRMS (ESI) calcd for $C_{33}H_{31}N_2O_8S$ $[M+H]^+$: 615.17956, found: 615.17866.



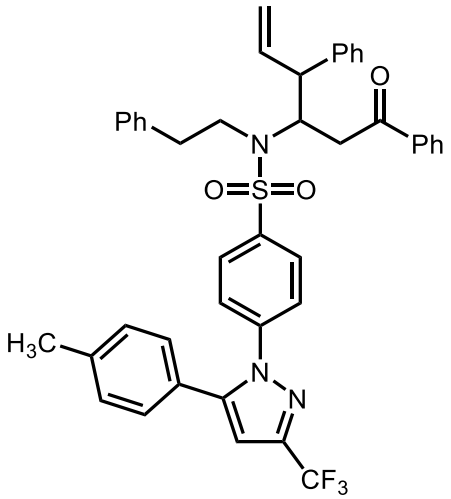
***N*-(4-Benzyl-1-oxo-1-phenylhex-5-en-3-yl)-*N*-methyl-2-nitrobenzenesulfonamide (9ak):** (SGS-6-19) Prepared following **General procedure E** using *N*-methyl-2-nitro-*N*-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (**8a**, 69.3 mg, 0.2 mmol, 1.0 equiv) and 4-phenyl-1-butene (**1k**, 90 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by

flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a yellow foam (37.7 mg, 40%, 1.1:1 d.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (dd, *J* = 7.6, 0.6 Hz, 1H'), 8.09 – 8.04 (m, 1H*), 7.82 (d, *J* = 7.6 Hz, 2H*), 7.74 (d, *J* = 7.5 Hz, 2H'), 7.64 – 7.37 (m, 6H*+6H'), 7.25 – 7.11 (m, 3H*+3H'), 7.07 (d, *J* = 7.4 Hz, 2H*+2H'), 5.58 (dt, *J* = 17.1, 9.9 Hz, 1H*), 5.46 (dt, *J* = 17.1, 9.8 Hz, 1H'), 4.91 – 4.59 (m, 3H*+3H'), 3.40 (dd, *J* = 17.3, 7.2 Hz, 1H*), 3.31 (dd, *J* = 17.1, 6.7 Hz, 1H'), 3.16 – 3.09 (m, 3H'+1H*), 3.05 – 2.98 (m, 3H*+1H'), 2.95 (dd, *J* = 17.1, 4.4 Hz, 1H*), 2.80 – 2.48 (m, 2H*+2H'), 2.31 (dd, *J* = 13.6, 10.9 Hz, 1H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 196.8, 196.6, 147.9, 147.8, 139.8, 139.5, 137.8, 137.6, 136.6, 136.5, 133.7, 133.5, 133.4, 133.4, 133.3, 132.0, 131.8_1, 131.7_8, 131.7, 129.4, 129.3, 128.8, 128.7, 128.3, 128.1, 128.0, 126.2, 126.1, 124.0, 123.9, 119.2, 118.6, 57.1, 56.5, 51.4, 50.4, 40.4, 40.3, 38.5, 38.1, 31.5, 30.6. Two peaks missing due to overlap.

HRMS (ESI) calcd for $C_{26}H_{27}N_2O_5S$ $[M+H]^+$: 479.16352, found: 479.16176.



***N*-(1-Oxo-1,4-diphenylhex-5-en-3-yl)-N-phenethyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (9da):** (SGS-6-63) Prepared following **General procedure E** using *N*-(3-oxo-3-phenylprop-1-en-1-yl)-*N*-phenethyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (**8d**, 123.1 mg, 0.2 mmol, 1.0 equiv) and allylbenzene (**1a**, 79 μL, 0.6 mmol, 3.0 equiv). The reaction mixture was heated at 60 °C for 16 h. The crude reaction mixture was purified by flash column chromatography (hexanes/ethyl acetate = 10:1) to afford the title compound as a very sticky brown solid, which trapped ethyl acetate despite significant removal attempts. (104.5 mg, 70%, 1.1:1 d.r.).

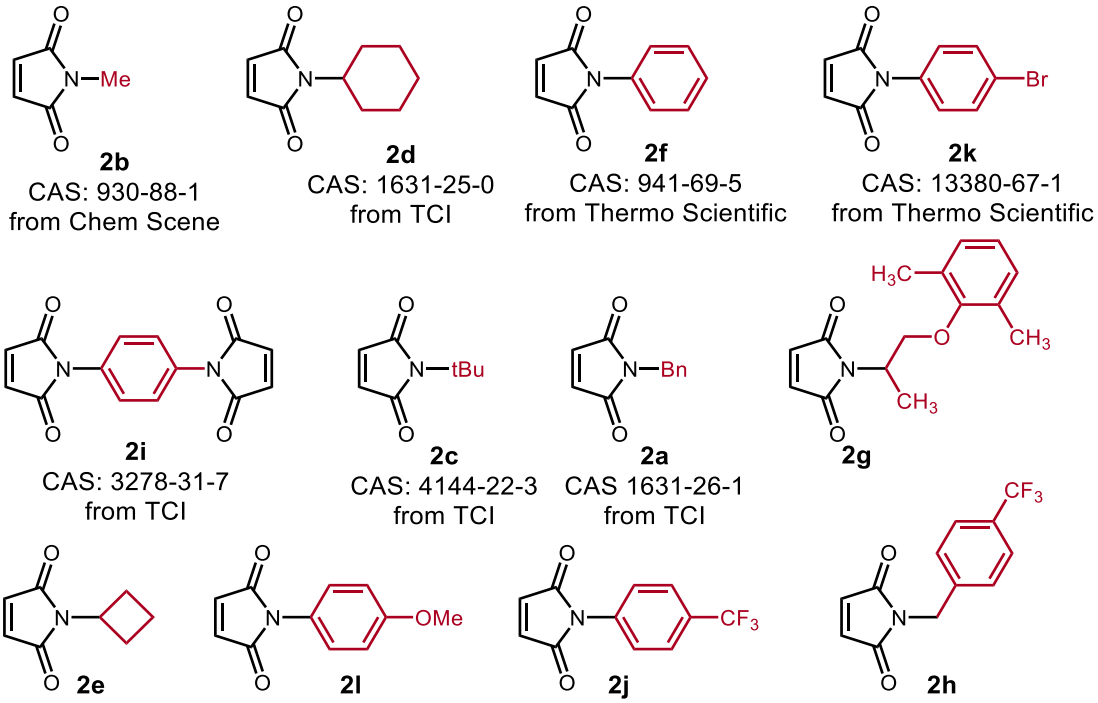
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 – 7.78 (t, *J* = 7.4 Hz, 2H*+2H'), 7.56 – 7.48 (m, 1H*+1H'), 7.45 – 7.37 (dd, *J* = 16.7, 8.4 Hz, 2H*+2H'), 7.35 – 7.14 (m, 12H*+12H'), 7.13 – 6.98 (m, 6H*+6H'), 6.69 (s, 1H*), 6.68 (s, 1H'), 6.19 – 6.07 (m, 1H'), 5.77 (dt, *J* = 16.9, 9.9 Hz, 1H*), 5.14 – 4.79 (m, 3H*+3H'), 3.81 – 3.60 (m, 1H*+1H'), 3.59 – 3.27 (m, 2H*+2H'), 3.21 – 2.56 (m, 4H*+4H'), 2.32 (s, 3H*), 2.28 (s, 3H').

^{13}C NMR (151 MHz, $CDCl_3$) δ 197.0, 196.6, 145.2_4, 145.2_1, 144.0_6 (q, *J* = 38.5 Hz), 144.0_4 (q, *J* = 38.5 Hz), 142.3_4, 142.3_2, 141.3, 140.6, 140.5, 139.8_3, 139.8_1, 139.6, 139.2, 139.0, 138.8, 138.6_3, 136.5_5, 136.2, 133.4, 133.3, 129.8, 129.0, 128.9, 128.9, 128.8_4, 128.8_1, 128.7_7, 128.7_6, 128.7, 128.5_7, 128.5_5, 128.5, 128.2, 128.1, 127.9, 127.5, 127.2, 126.8_2, 126.7_7, 125.9, 125.3_0, 125.2_7, 121.2 (q, *J* = 269.1 Hz), 117.6, 117.4, 106.3, 56.9, 55.6, 42.4, 41.3, 36.9, 36.4, 29.8, 21.4_1, 21.3_7. Six peaks missing due to overlap.

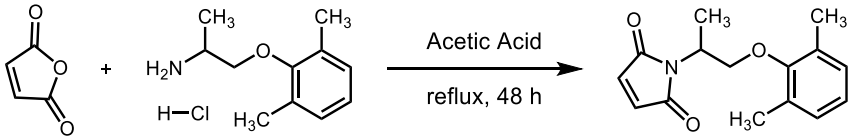
^{19}F NMR (471 MHz, $CDCl_3$) δ −62.40. One peak missing due to overlap.
HRMS (ESI) calcd for $C_{43}H_{39}N_3O_3F_3S$ $[M+H]^+$:734.26587, found: 734.26482.

2.5 Synthesis of Michael acceptor substrates

2.5.1 Maleimide Michael Acceptors



Maleimides **2a, 2b, 2c, 2d, 2f, 2i,** and **2k** are commercial and were used directly without further purification.
Maleimides **2e,**[2] **2h,**[3] **2j,**[4] and **2l**[5] were synthesized according to a known literature procedure and have been previously characterized.



1-(1-(2,6-Dimethylphenoxy)propan-2-yl)-1*H*-pyrrole-2,5-dione (2g): (SGS-5-191)
Mexiletine hydrochloride (647 mg, 3.0 mmol, 1.0 equiv) (note that the hydrochloride salt was simply chosen for the commercial availability of the compound and had no additional bearing on the reaction) and maleic anhydride (588.4 mg, 6.0 mmol, 2.0 equiv) were suspended in glacial acetic acid (4.5 mL). The reaction was refluxed with stirring at 125 °C for 48 hours. The reaction mixture was then cooled to room temperature and transferred to a 500 mL beaker. Saturated aqueous sodium bicarbonate was added until no additional gas evolution was observed. Ethyl acetate was added and the mixture was extracted with ethyl acetate. The combined organic fractions were washed with a 1N HCl solution, followed by a brine solution, and were then dried with magnesium sulfate, filtered, and concentrated. The crude product was purified by flash column chromatography on silica (ethyl acetate/hexanes = 3:1), affording **2g** (165.7 mg, 21% yield) as off white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.97 (d, *J* = 7.5 Hz, 2H), 6.89 (dd, *J* = 8.0, 6.9 Hz, 1H), 6.70 (s, 2H), 4.71 – 4.62 (m, 1H), 4.23 (t, *J* = 9.3 Hz, 1H), 3.80 (dd, *J* = 9.4, 5.5 Hz, 1H), 2.20 (s, 6H), 1.46 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 155.3, 134.2, 130.8, 129.0, 124.1, 71.6, 47.2, 16.3, 15.2.
HRMS (ESI) calcd for $C_{15}H_{18}O_3N$ $[M+H]^+$: 260.12812, found: 260.12816.
m.p. = 64 – 65 °C

2.5.2 *N*-Acyl oxazolidinone Michael Acceptors

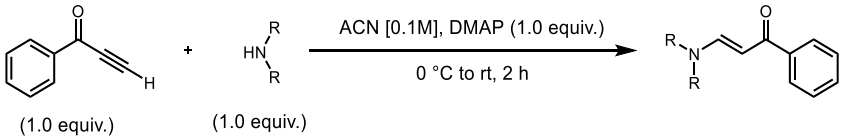
All Michael acceptor starting materials were synthesized with an E/Z of >20% by NMR.
N-Acyl oxazolidinone Michael Acceptors **4a** and **4b** were synthesized according to a known literature procedure and have been previously characterized.[6]

2.5.3 Phthalimide protected Michael Acceptors

All Michael acceptor starting materials were synthesized with an E/Z of >20% by NMR.
The Michael acceptor **6a** was synthesized according a literature procedure and has been previously characterized.[7]

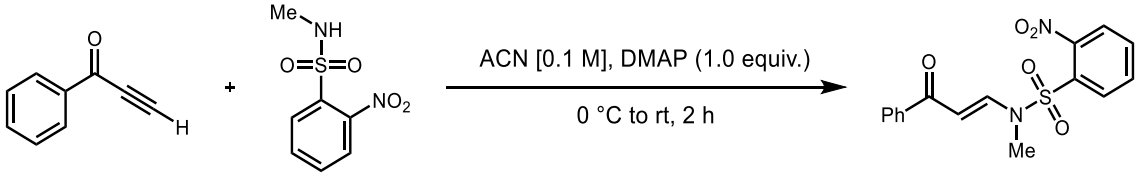
2.5.4 Sulfonamide protected Michael Acceptors:

All Michael acceptor starting materials were synthesized with an E/Z of >20% by NMR.
General Procedure F: Synthesis of sulfonamide protected Michael acceptors from alkynones



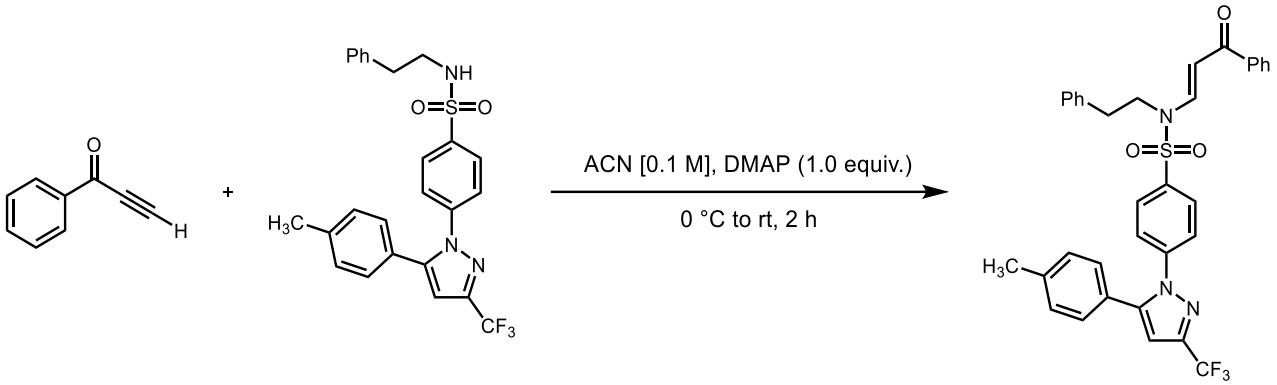
To a flame dried round bottom flask containing the sulfonamide (2.0 mmol, 1.0 equiv) and DMAP (2.0 mmol 1.0 equiv) was added acetonitrile [0.1 M]. Alkyne was dissolved in minimal acetonitrile and then added dropwise at 0 °C with rapid stirring. Minimal color change upon alkyne addition is preferable, with poor yields observed with significant darkening of the reaction mixture. The reaction was stirred for two hours, gradually warming to room temperature. The mixture was filtered through a celite plug, then concentrated to dryness and purified by column chromatography.

The sulfonamide protected Michael acceptors **8b** and **8c** were synthesized according to General Procedure F and have been previously characterized.[8]



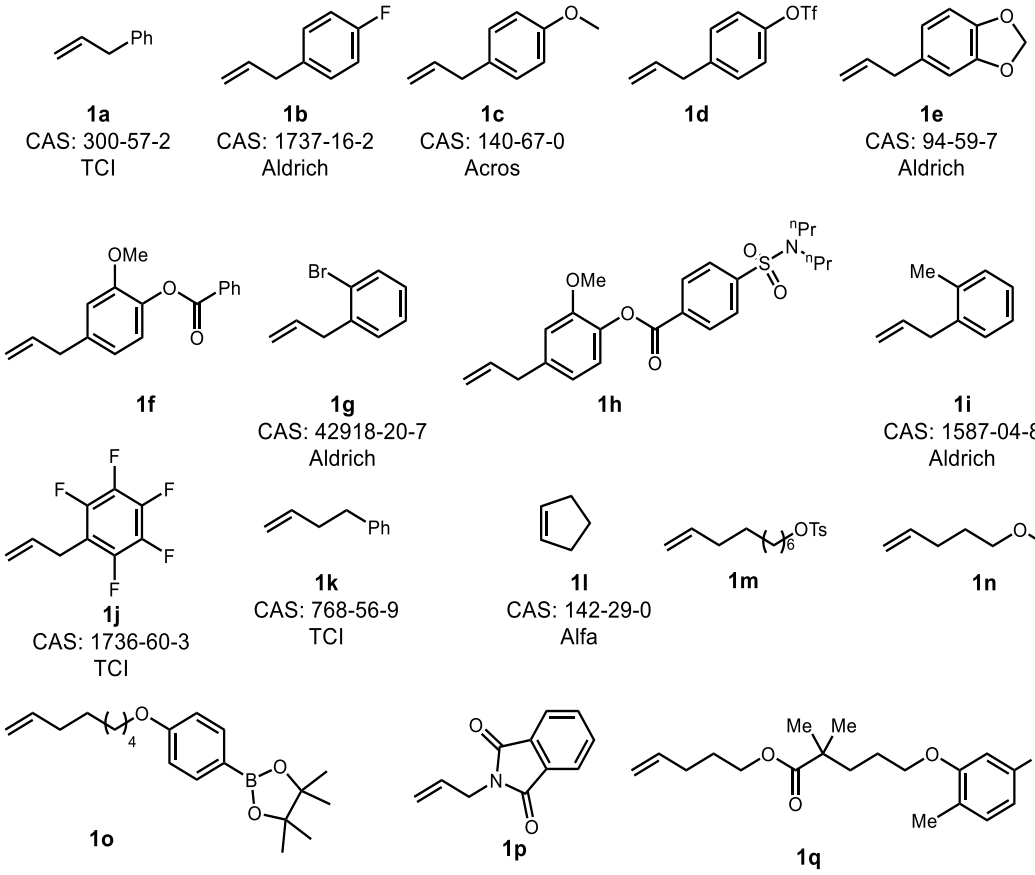
***N*-methyl-2-nitro-N-(3-oxo-3-phenylprop-1-en-1-yl)benzenesulfonamide (8a):** (SGS-5-181)
Prepared following **General Procedure F**: The crude product was purified by flash column chromatography on silica (ethyl acetate/hexanes = 2:1), affording **8a** (0.3027 g, 70% yield) as off white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.20 (d, *J* = 13.5 Hz, 1H), 8.12 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.93 – 7.89 (m, 2H), 7.82 – 7.73 (m, 3H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 6.28 (d, *J* = 13.5 Hz, 1H), 3.30 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 189.5, 143.1, 138.4, 135.1, 132.8, 132.6, 131.5, 131.3, 128.7, 128.3, 125.1, 104.6, 33.5.
HRMS (ESI) calcd for $C_{16}H_{15}O_5N_2S$ $[M+H]^+$: 347.06962, found: 347.07065.
m.p. = 158 – 159 °C



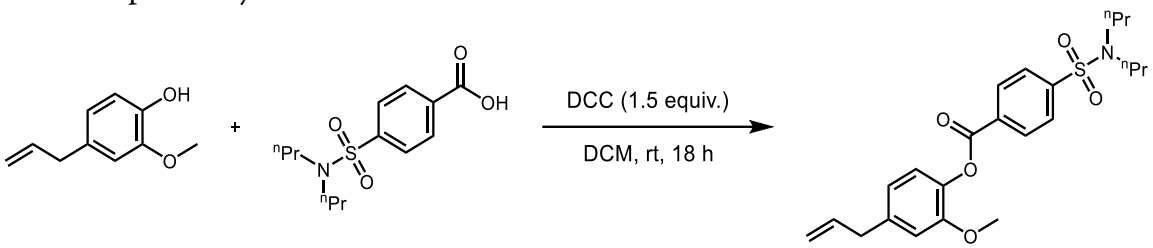
***N*-(3-oxo-3-phenylprop-1-en-1-yl)-*N*-phenethyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)benzenesulfonamide** (**8d**): (SGS-5-183)
Step 1. N-phenethyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)benzenesulfonamide was synthesized according to a known literature procedure and has been previously characterized.[9]
Step 2. Prepared following **General Procedure F**: The crude product was purified by flash column chromatography on silica (ethyl acetate/hexanes = 5:1), affording **8d** (0.2745 g, 73% yield) as yellow foam.
^{1}H NMR (500 MHz, $CDCl_3$) 8.23 (d, *J* = 13.7 Hz, 1H), 7.85 – 7.79 (m, 4H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.43 (m, 4H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.28 – 7.22 (m, 1H), 7.20 (d, *J* = 7.1 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 6.72 (s, 1H), 6.21 (d, *J* = 13.7 Hz, 1H), 3.75 – 3.68 (m, 2H), 3.01 – 2.91 (m, 2H), 2.35 (s, 3H).
^{13}C NMR (126 MHz, C DCl_3) 189.3, 145.5, 144.5 (q, *J* = 38.9 Hz), 143.6, 142.3, 140.2, 138.5, 137.5, 137.4, 132.7, 123.0, 129.1, 128.8, 128.7, 128.4, 128.2, 127.3, 125.9, 125.7, 121.1 (q, *J* = 269.3 Hz). 106.8, 103.7, 48.2, 33.9, 21.5.
^{19}F NMR (471 MHz, $CDCl_3$) δ −62.55.
HRMS (ESI) calcd for $C_{34}H_{29}O_3N_3F_3S$ $[M+H]^+$: 616.18762, found: 616.18750.
m.p. = 86 – 91 °C

2.6 Synthesis of alkene substrates



The alkenes **1a**-**1c**, **1e**, **1g**, and **1j**-**1l** are commercial, and were used directly without purification.

The alkenes **1f** and **1m**-**1q**,[10] and **1d**[11] were synthesized according to a known literature procedure and have been previously characterized.



4-Allyl-2-methoxyphenyl 4-(N,N-dipropylsulfamoyl)benzoate (**1h**) (SGS-6-62)

DCC (928 mg, 4.5 mmol, 1.5 equiv) was added to the solution of probenecid (856 mg, 3.0 mmol, 1.0 equiv) and eugenol (0.558 ml, 3.6 mmol, 1.2 equiv) in 15 mL of CH_2Cl_2. The reaction mixture was stirred at room temperature overnight. The crude mixture filtered through celite and purified by flash chromatography (hexanes/ethyl acetate = 5:1) to obtain the pure compound as a white solid (342.8 mg, 26% yield).

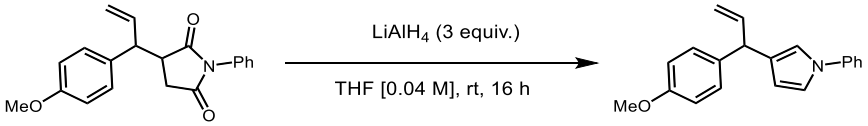
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.5 Hz, 2H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 1H), 6.88 – 6.79 (m, 2H), 5.99 (ddt, *J* = 16.8, 10.1, 6.7 Hz, 1H), 5.20 – 5.07 (m, 2H), 3.81 (s, 3H), 3.42 (d, *J* = 6.7 Hz, 2H), 3.16 – 3.07 (m, 4H), 1.67 – 1.48 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.7, 151.0, 144.8, 139.6, 138.0, 137.1, 133.0, 131.0, 127.3, 122.6, 120.9, 116.4, 113.0, 56.0, 50.2, 40.3, 22.1, 11.3.

HRMS (ESI) calcd for $C_{23}H_{30}O_5NS$ $[M+H]^+$: 432.18392, found: 432.18363.
m.p. = 105 – 106 °C

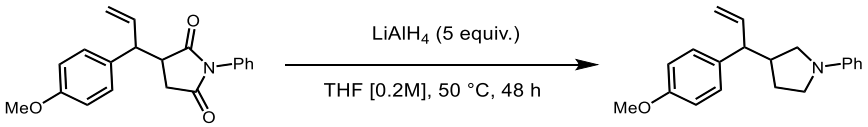
3. Synthetic applications of products

Synthesis of 3-(1-(4-Methoxyphenyl)allyl)-1-phenyl-1*H*-pyrrole (10) (SGS-6-94)



The following reaction was based on a literature procedure.[12] A reaction tube equipped with a magnetic stir bar was capped with a septum and flame dried under vacuum. **3fc** (32.3 mg, 0.1 mmol, 1.0 equiv), is dissolved in anhydrous THF (2.5 mL). $LiAlH_4$ (10.7 mg, 0.3 mmol, 3 equiv) was added in one portion under nitrogen stream. The flask sealed and stirred at room temperature for 16 h. The reaction is quenched with water at 0 °C. The crude reaction mixture is extracted with ethyl acetate, dried over magnesium sulfate, filtered, and concentrated. The crude reaction mixture was immediately purified by flash chromatography (hexanes/ethyl acetate = 5:1) to give the pure product **10** as a yellow oil (24.6 mg, 85% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 4H), 7.25 – 7.18 (m, 3H), 7.05 (t, *J* = 2.6 Hz, 1H), 6.90 – 6.85 (m, 2H), 6.81 (s, 1H), 6.26 (ddd, *J* = 17.3, 10.0, 7.5 Hz, 1H), 6.15 (dd, *J* = 2.6, 1.9 Hz, 1H), 5.16 – 5.06 (m, 2H), 4.61 (d, *J* = 7.4 Hz, 1H), 3.81 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 158.2, 141.7, 140.8, 136.3, 129.6, 129.4, 128.9, 125.4, 120.2, 119.4, 117.3, 114.8, 113.9, 110.9, 55.4, 47.7.
HRMS (ESI) calcd for $C_{20}H_{20}ON$ $[M+H]^+$: 290.15394, found: 290.15333.

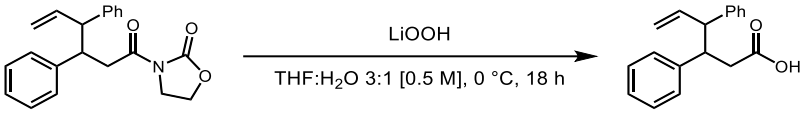
Synthesis of 3-(1-(4-Methoxyphenyl)allyl)-1-phenylpyrrolidine (11) (SGS-6-67)



The following reaction was based on a literature procedure.[13] A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap and flame dried under vacuum. **3fc** (64.3 mg, 0.2 mmol, 1.0 equiv), is dissolved in anhydrous THF (1.0 mL). $LiAlH_4$ (2.7 mg, 0.012 mmol, 5 equiv) was added into the reaction tube in one portion. The reaction tube sealed and stirred at 50 °C, 48 h. The reaction is quenched with water at 0 °C. The crude reaction mixture is extracted with ethyl acetate, dried over magnesium sulfate, filtered, and concentrated. The crude reaction mixture was purified by flash chromatography (hexanes/ethyl acetate = 5:1) to give the pure product **11** as a clear oil (21.5 mg, 37% yield, 5.0:1 d.r.).
^{1}H NMR (601 MHz, $CDCl_3$) δ 7.24 – 7.19 (m, 2H*), 7.18 – 7.14 (m, 2H'), 7.14 – 7.10 (m, 2H*+2H'), 6.89 – 6.83 (m, 2H*+2H'), 6.65 (t, *J* = 7.3 Hz, 1H*), 6.61 (t, *J* = 7.3 Hz, 1H'), 6.55 (d, *J* = 8.0 Hz, 2H*), 6.44 (d, *J* = 8.0 Hz, 2H'), 6.04 – 5.94 (m, 1H*+1H'), 5.11 – 4.99 (m, 2H*+2H'), 3.79 (s, 3H'), 3.79 (s, 3H*), 3.46 (dd, *J* = 9.1, 7.6 Hz, 1H*), 3.39 (td, *J* = 8.8, 2.8 Hz, 1H'), 3.35 – 3.27 (m, 1H*+1H'), 3.23 - 3.17 (m, 1H*), 3.14 – 3.04 (m, 2H*+2H'), 2.83 (t, *J* = 8.8 Hz, 1H'), 2.68 – 2.56 (m, 1H*+1H'), 2.21 (dtd, *J* = 9.4, 6.7, 2.7 Hz, 1H'), 1.86 – 1.73 (m, 1H*+1H'), 1.57 (dq, *J* = 12.4, 8.8 Hz, 1H*+1H').

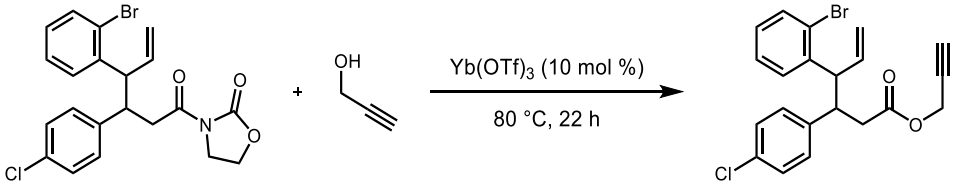
^{13}C NMR (151 MHz, $CDCl_3$) δ 158.3, 158.3, 148.0, 147.9, 141.2$_2$, 141.1$_7$, 135.5, 135.4, 129.3, 129.2, 128.7, 128.6, 115.6$_1$, 115.5$_7$, 114.9, 114.8, 114.1$_9$, 114.1$_6$, 111.6$_1$, 111.5$_7$, 55.4, 54.1$_7$, 54.1$_5$, 52.5, 52.4, 47.7, 47.5, 43.6, 43.5, 30.8, 30.5. One peak missing due to overlap.
HRMS (ESI) calcd for $C_{20}H_{24}ON$ $[M+H]^+$: 294.18524, found: 294.18549.

Synthesis of 3,4-Diphenylhex-5-enoic acid (12) (SGS-6-83)



The following reaction was based on a literature procedure.[14] *N*-Acyl oxazolidinone **5aa** (67.1 mg, 0.2 mmol, 1.0 equiv), is dissolved in THF:H_2O 3:1 (0.4 mL) in a 5mL round bottomed flask and cooled to 0 °C. 30% H_2O_2 (0.16 mL, 8.0 equiv) and LiOH (16.8 mg, 2.0 equiv) are sequentially added and the reaction vessel is allowed to warm to room temperature and stirred for 18 h. The peroxide is quenched by the addition of saturated aqueous Na_2SO_3, and the mixture is acidified to pH 1-2 with 6 M HCl. The crude reaction mixture is extracted with ethyl acetate, dried over magnesium sulfate, filtered, and concentrated. The crude reaction mixture was purified by flash chromatography (hexanes/ethyl acetate = 3:1) to give the pure product **12** as a clear oil (51.3 mg, 96% yield, 1:2.2 d.r.)
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.24 (m, 3H*+3H'), 7.24 – 7.07 (m, 6H*+5H'), 7.05 (t, *J* = 6.8 Hz, 2H'), 6.99 – 6.93 (m, 1H*), 6.09 – 6.00 (m, 1H'), 5.84 – 5.74 (m, 1H*), 5.21 – 5.06 (m, 2H'), 4.91 – 4.70 (m, 2H*), 3.53 – 3.38 (m, 2H*+2H'), 2.98 – 2.92 (m, 1H'), 2.67 – 2.59 (m, 1H'), 2.58 – 2.46 (m, 2H*). Carboxylic acid proton was not observed.
^{13}C NMR (126 MHz, $CDCl_3$) δ 177.5, 177.2, 142.3, 142.1, 141.5, 140.0, 139.3, 128.8, 128.6, 128.4, 128.3, 128.1, 128.1, 126.9, 126.6, 126.3, 116.8, 116.5, 57.2, 55.8, 47.2, 46.7, 39.1$_4$, 39.1$_0$.
HRMS (ESI) calcd for $C_{18}H_{17}O_2$ $[M–H]^-$: 265.12231, found: 265.12271.

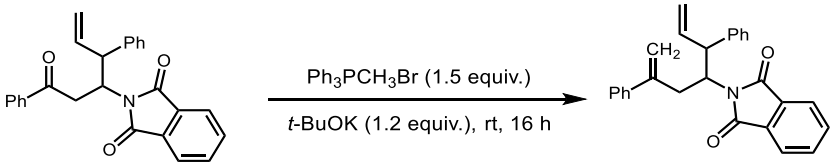
Synthesis of Prop-2-yn-1-yl 4-(2-bromophenyl)-3-(4-chlorophenyl)hex-5-enoate (13) (SGS-7-4)



The following reaction was based on a literature procedure.[15] A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap was flame dried under vacuum. $Yb(OTf)_3$ (6.2 mg, 10 mol %) was added and the reaction tube was purged with nitrogen. Propargyl alcohol (680 μL) was added to the reaction tube and the catalyst solution was heated to 60 °C for 30 minutes. The reaction vial was removed from the heat for 5 minutes and **5bg** (44.8 mg, 0.100 mmol) dissolved in propargyl alcohol (680 μL) was added. The reaction vial was stirred at 80 °C for 22 hours. The reaction mixture was then cooled, filtered through a silica plug, and the crude reaction mixture was purified by flash chromatography (hexanes/ethyl acetate = 3:1) to give the pure product **13** as a clear oil (37.6 mg, 90% yield, 1:2.5 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 8.0, 1.0 Hz, 1H*), 7.37 (dd, *J* = 8.0, 0.9 Hz, 1H'), 7.31 – 7.02 (m, 7H*+6H'), 6.94 – 6.90 (m, 1H'), 5.90 (dt, *J* = 17.0, 9.7 Hz, 1H'), 5.72 (ddd, *J* = 17.1, 10.2, 8.3 Hz, 1H*), 5.33 – 5.11 (m, 2H'), 4.89 (dd, *J* = 37.5, 13.6 Hz, 2H*), 4.59 – 4.34 (m, 2H*+2H'), 4.17 (t, *J* = 9.3 Hz, 1H*+1H'), 3.61 (dt, *J* = 10.3, 5.1 Hz, 1H'), 3.50 (td, *J* = 10.4, 4.6 Hz, 1H*), 3.01 (dd, *J* = 15.9, 4.7

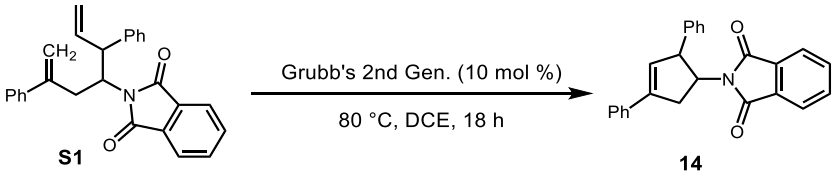
Hz, 1H'), 2.68 – 2.60 (m, 1H*+1H'), 2.50 (dd, *J* = 15.8, 4.7 Hz, 1H*), 2.40 (t, *J* = 2.4 Hz, 1H'), 2.37 (t, *J* = 2.4 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 171.1, 141.2, 140.9, 139.8, 139.7, 138.5, 137.7, 133.4, 133.2, 132.8, 132.4, 130.0, 129.6, 129.1, 128.9, 128.7, 128.4, 128.4, 128.0, 127.9, 127.6, 125.4, 118.0, 117.7, 77.5_4, 77.4_9, 74.9_6, 74.9_2, 54.0, 53.2, 52.1, 52.0, 46.6, 45.4, 39.6, 38.9.
HRMS (ESI) calcd for $C_{21}H_{19}O_2BrCl$ $[M+H]^+$: 417.02515, found: 417.02509.

Synthesis of 2-(2,5-Diphenylhepta-1,6-dien-4-yl)isoindoline-1,3-dione (S1) (SGS-6-79)



The following reaction was based on a literature procedure.[16] A flame dried round bottom flask with stir bar was transferred into the glove box. Methyltriphenylphosphonium bromide (160.8 mg, 0.45 mmol, 1.5 equiv) and potassium tert-butoxide (50.5 mg, 0.45 mmol, 1.5 equiv) are transferred into the flask and subsequently dissolved in anhydrous THF (1.0 mL) at room temperature. The resulting yellow solution was stirred at rt for 1 hr. **7aa** (118.6 mg, 0.3 mmol, 1.0 equiv) is added to the reaction mixture. The flask was sealed, removed from the glove box, and stirred at rt for 16 hours. The reaction is quenched by the addition of water and ethyl acetate. The crude reaction mixture is extracted with ethyl acetate. The combined organic layers were washed with water and brine, dried over $MgSO_4$, and evaporated under reduced pressure. The crude product was purified by flash chromatography (hexanes/ethyl ether = 100:1) to give the pure product **S1** as a clear oil (55.0 mg, 47% yield, 2.2:1 d.r.).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 – 7.46 (m, 4H*+4H'), 7.40 (t, *J* = 7.4 Hz, 2H*), 7.35 (d, *J* = 7.0 Hz, 2H*), 7.31 (t, *J* = 7.2 Hz, 1H*), 7.25 – 6.94 (m, 5H*+10H'), 6.04 (dt, *J* = 17.0, 9.7 Hz, 1H'), 5.82 (dt, *J* = 16.9, 9.8 Hz, 1H*), 5.39 – 4.76 (m, 4H*+4H'), 4.57 (td, *J* = 11.6, 3.0 Hz, 1H*+1H'), 4.26 – 4.20 (m, 1H'), 4.19 – 4.12 (m, 1H*), 3.40 – 3.19 (m, 1H*+2H'), 2.63 (dd, *J* = 14.6, 2.0 Hz, 1H*).
^{13}C NMR (126 MHz, $CDCl_3$) δ 146.3, 146.0, 141.5, 141.1, 140.7, 140.2, 139.6, 139.0, 133.8, 133.5, 129.2, 128.5, 128.4, 128.3, 128.1, 127.6, 127.5_3, 127.5_1, 127.3, 126.8, 126.4, 126.3, 123.4, 122.9, 117.6, 116.7, 115.6, 115.2, 55.1, 54.4, 53.6, 53.5, 36.2, 35.3. Two peaks missing due to overlap. The broad phthalimide carbonyl peaks were not resolved from baseline.
HRMS (ESI) calcd for $C_{27}H_{24}O_2N$ $[M+H]^+$: 394.18016, found: 394.18021.

Synthesis of 2-(2,4-Diphenylcyclopent-3-en-1-yl)isoindoline-1,3-dione (14) (SGS-6-80)



The following reaction was based on a literature procedure.[17] A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap and flame dried under vacuum. **S1** (39.3 mg, 0.1 mmol, 1.0 equiv), Grubb's second generation catalyst (8.5 mg, 0.01 mmol, 10% mol), and anhydrous DCE (5 mL) were added into the reaction tube in the glovebox. The reaction tube was sealed, removed from the glove box, and was stirred for 18 h at 80 °C. The reaction mixture was cooled to room

temperature and filtered through a plug of silica gel and washed with CH_2Cl_2 (ca. 30 mL). The crude product was purified by flash chromatography (hexanes/ethyl ether = 20:1) to give the pure product **14** as a yellow oil (12.0 mg, 33% yield, 1:1.6 dr).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.80 (m, 2H*), 7.74 – 7.69 (m, 2H*), 7.62 – 7.55 (m, 1H*), 7.48 (d, *J* = 7.7 Hz, 2H*), 7.42 – 7.19 (m, 7H*+9H'), 7.12 (d, *J* = 7.7 Hz, 2H'), 7.05 (t, *J* = 7.5 Hz, 2H'), 6.98 (t, *J* = 7.3 Hz, 1H'),

6.36 (d, *J* = 2.0 Hz, 1H'), 6.27 (s, 1H*), 5.40 (td, *J* = 9.1, 6.2 Hz, 1H'), 4.96 (q, *J* = 8.8 Hz, 1H*), 4.82 – 4.77 (m, 1H*), 4.50 (d, *J* = 8.8 Hz, 1H'), 3.97 (ddt, *J* = 16.1, 6.1, 1.9 Hz, 1H'), 3.46 – 3.38 (m, 1H*), 3.17 – 3.07 (m, 1H*+1H').

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.5, 168.4, 142.8, 141.3, 135.8, 134.1, 133.7, 132.1, 128.9, 128.9, 128.7, 128.6, 128.0, 127.8$_7$, 127.8$_0$, 127.6, 127.2, 127.0, 126.0, 125.8, 123.4, 122.8, 58.4, 55.1, 54.2, 54.1, 36.8, 34.7.

HRMS (ESI) calcd for $C_{25}H_{20}O_2N$ $[M+H]^+$: 366.14886, found: 366.14940.

4. Assignment of diastereomers

4.1 Assignment for succinimide products 3 by X-ray crystallography

A sample **3fc** was crystallized was from previously isolated material using slow evaporation out of dichloromethane and hexanes to achieve crystals with further diastereomeric enrichment.

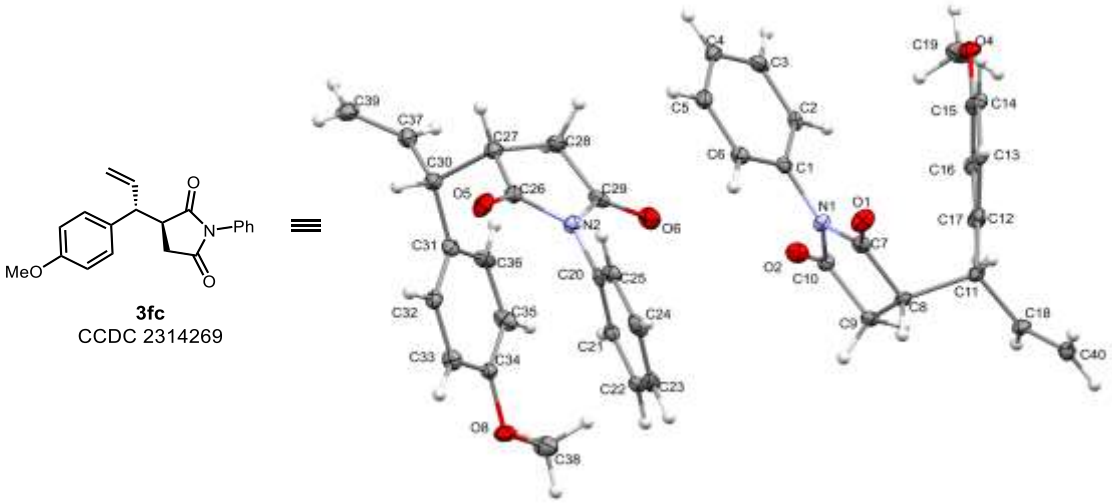


Figure S1: Structure of diastereomerically enriched **3fc**. Ellipsoid contour probability is set at 50%

The observed relative configuration of the major product is consistent with a Mukaiyama-type open transition state, in which additive coordinated to the γ carbon of the Michael acceptor is close enough to the incoming allyliron to have an effect on the diastereoselectivity of the allylation product.

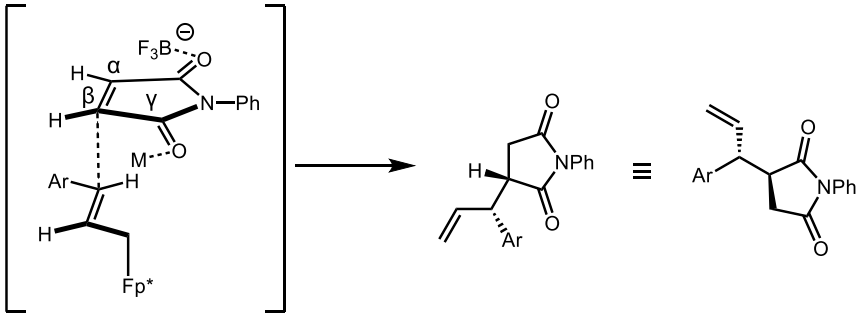


Figure S2: Postulated open transition state and anticipated stereochemical outcome

Datablock: Sarah120723

Bond precision: C-C = 0.0061 A Wavelength=1.54184

Cell: a=13.4572(6) b=9.9639(5) c=12.5603(6)
alpha=90 beta=102.524(2) gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	1644.09(14)	1644.09(14)
Space group	P c	P 1 c 1
Hall group	P -2yc	P -2yc
Moiety formula	C20 H19 N O3	?
Sum formula	C20 H19 N O3	C20 H19 N O3
Mr	321.36	321.36
Dx,g cm-3	1.298	1.298
Z	4	4
Mu (mm-1)	0.704	0.704
F000	680.0	680.0
F000'	682.07	
h,k,lmax	16,12,15	16,12,15
Nref	6518[3265]	6286
Tmin,Tmax	0.959,0.979	0.320,0.760
Tmin'	0.939	

Correction method= # Reported T Limits: Tmin=0.320 Tmax=0.760
AbsCorr = MULTI-SCAN

Data completeness= 1.93/0.96 Theta(max)= 72.370

R(reflections)= 0.0695(6152) wR2(reflections)= 0.1759(6286)

S = 1.045 Npar= 436

4.2 Assignment for β-aminoketones products 7 and 9 by nOe experiments and multiplet analysis

Comparison of coupling constants and nuclear Overhauser effect experiments were conducted on the cyclic derivative **14** and the *anti*-diastereomer was determined to be the major product.

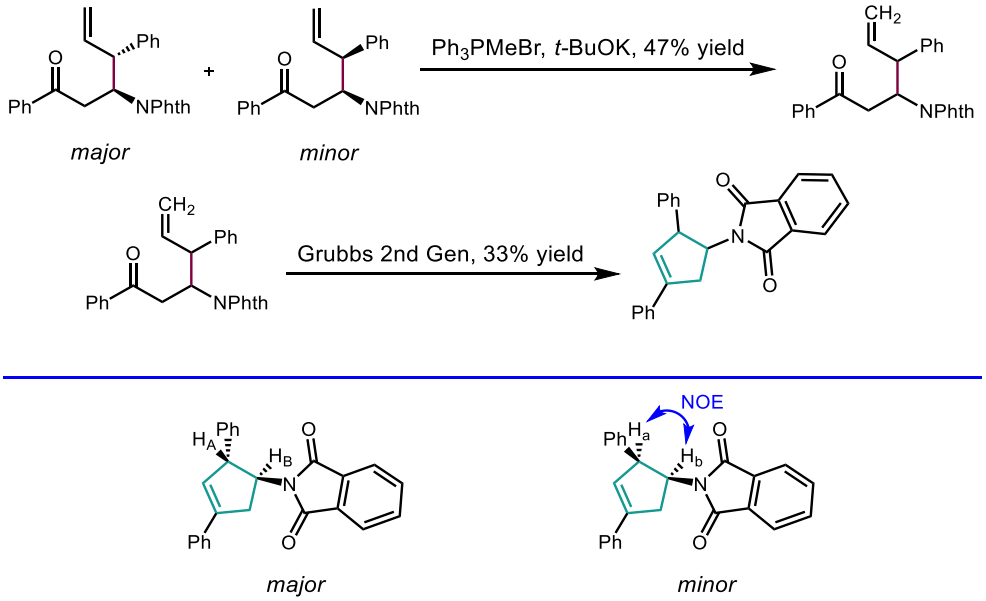


Figure S3: Derivatization reactions and observed stereochemical outcomes

The major product of the Wittig reaction was identified as the major product of the iron-catalyzed reaction based on comparison of coupling constants for the alkene protons at 6.07 (minor) and 5.92 (major), which remain at (dt, J = 16.9, 9.8 Hz) and (dt, J = 16.9, 9.9 Hz), respectively. The major product of olefin metathesis was identified as the major product of the Wittig reaction by integration comparison.

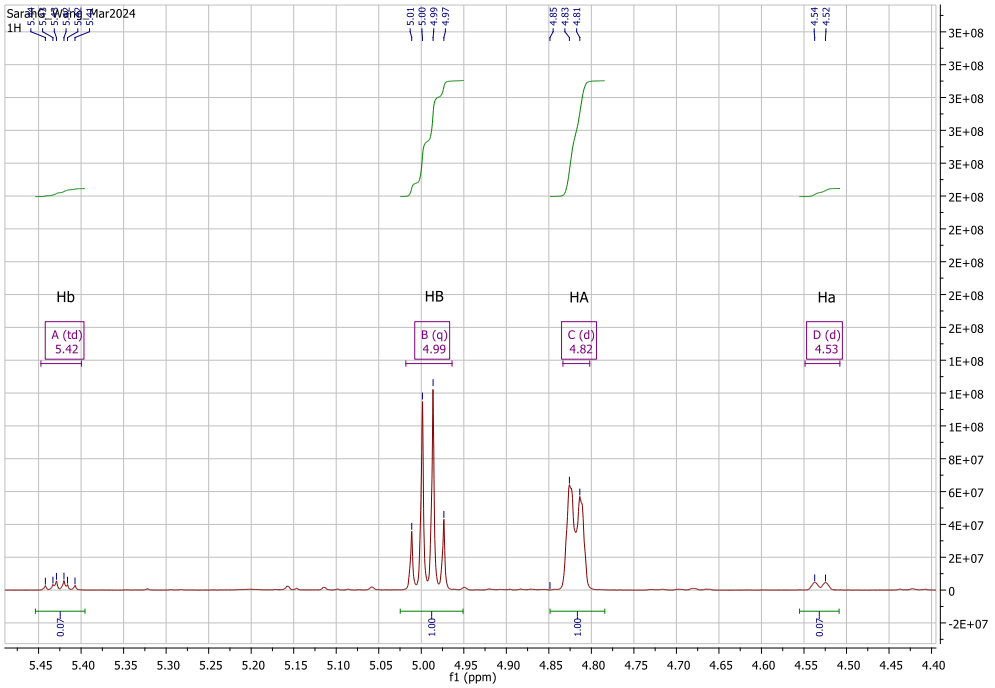


Figure S4: ^{1}H NMR of **14** showing protons H_a, H_b, H_A, and H_B

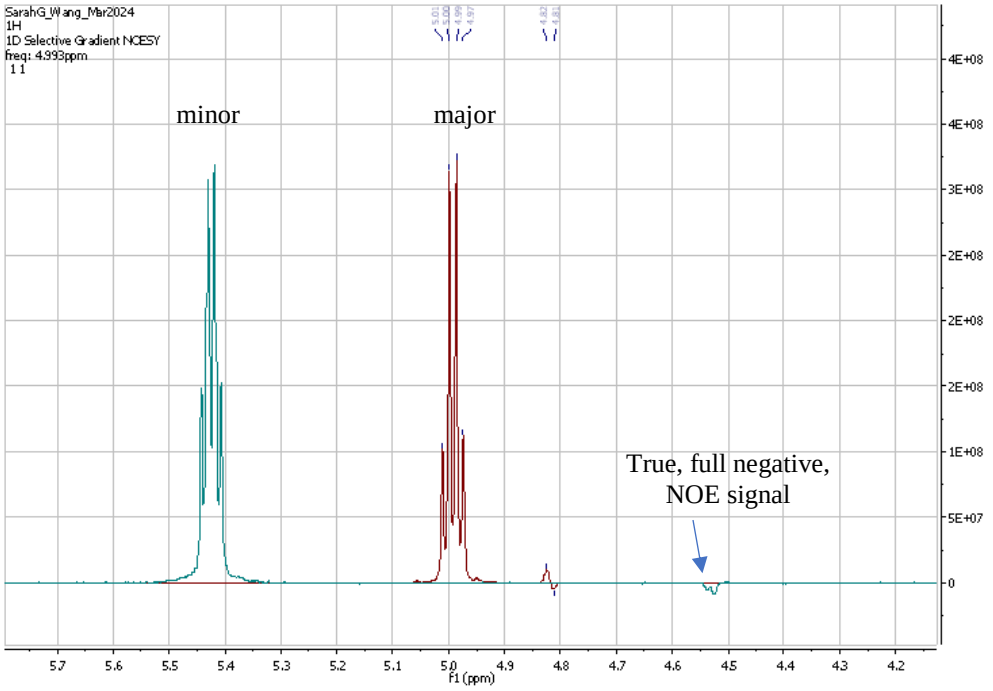


Figure S5: NOE irradiation at protons H_b, and H_B overlapped spectra

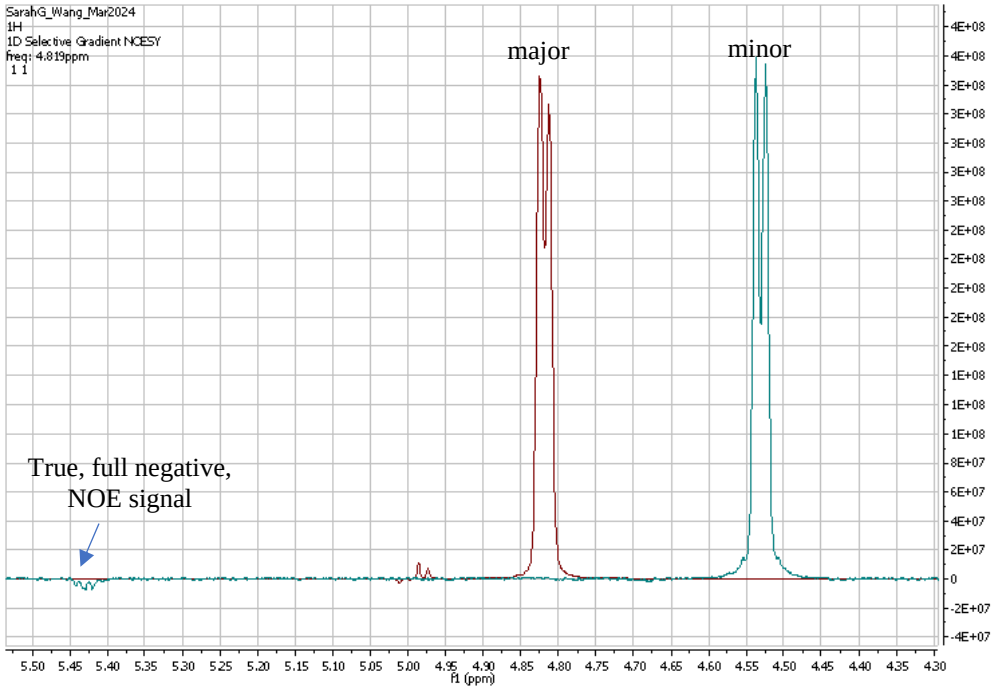


Figure S6: NOE irradiation at protons H_a and H_A overlapped spectra

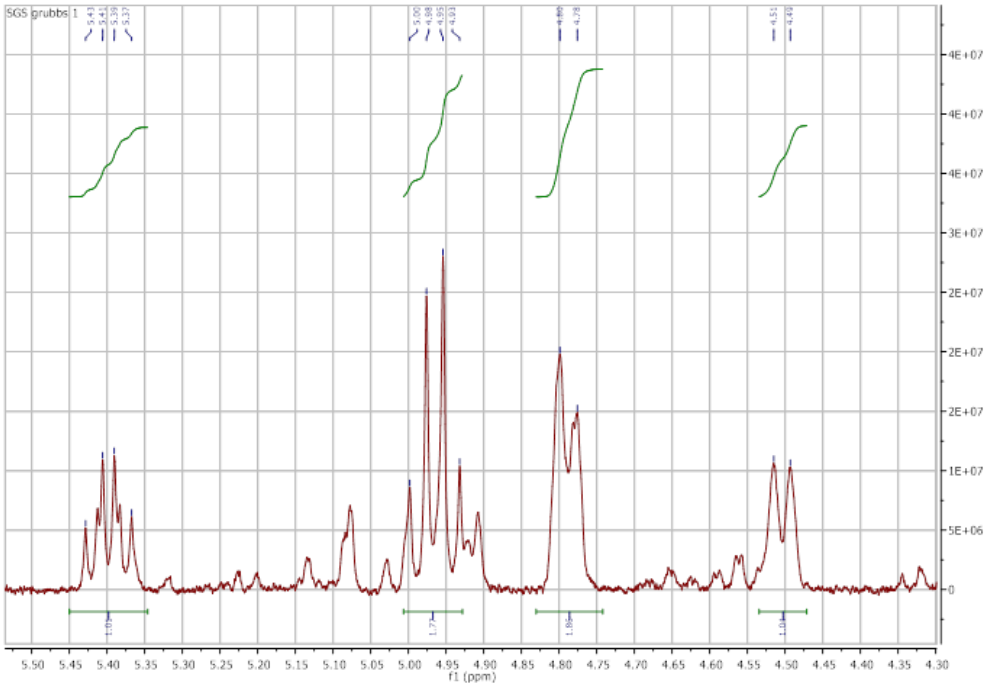
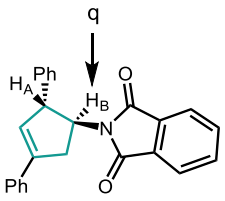
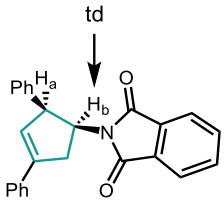


Figure S7: Crude NMR spectrum of transformation showing original diastereomeric ratio before enrichment on silica. The diastereomeric ratio is consistent with the crude spectra of the Michael addition.

Comparison of *syn*- and *anti*-configurations by DFT optimized geometry shows dihedral angles of which are consistent with a quartet splitting pattern for the *anti*-isomer, and a triplet of doublets for the *syn*-isomer.



major
all similar coupling constants



minor
two distinct coupling constants

Entry	Chemical Shift (experimental)	Coupling Constants (experimental)	Dihedral Angles (DFT)	Coupling Constants (Karplus curve prediction based on cyclopentane)
syn	5.45	9.1, 9.1, 6.2	13.5, 6.4, 112.4	11, 11, 4
anti	4.99	8.9, 8.9, 8.9	152.6, 155.2, 32.4	10.5, 10.5, 9

Karplus curve predictions were based on cyclopentane Karplus curve data from professor Hans J. Reich's NMR spectroscopy collection: https://organicchemistrydata.org/hansreich/resources/nmr/?page=05-hmr-05-3j%2F#05-hmr-05-3j-cyclopentane Accessed 4/12/2024

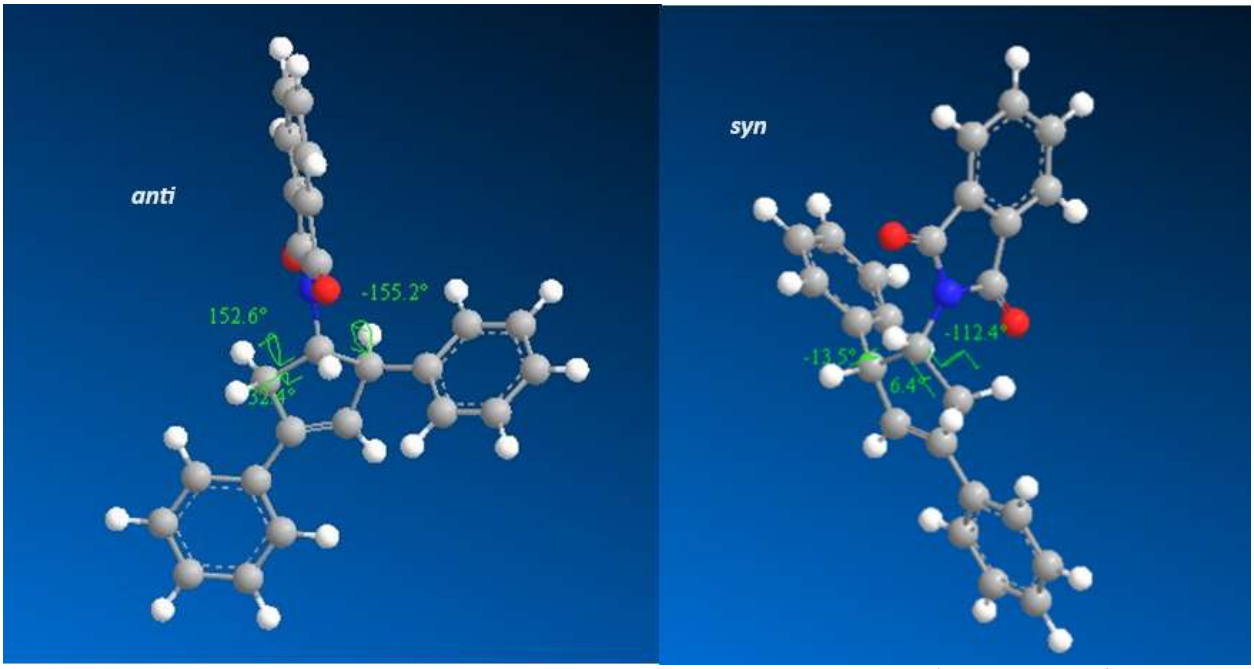
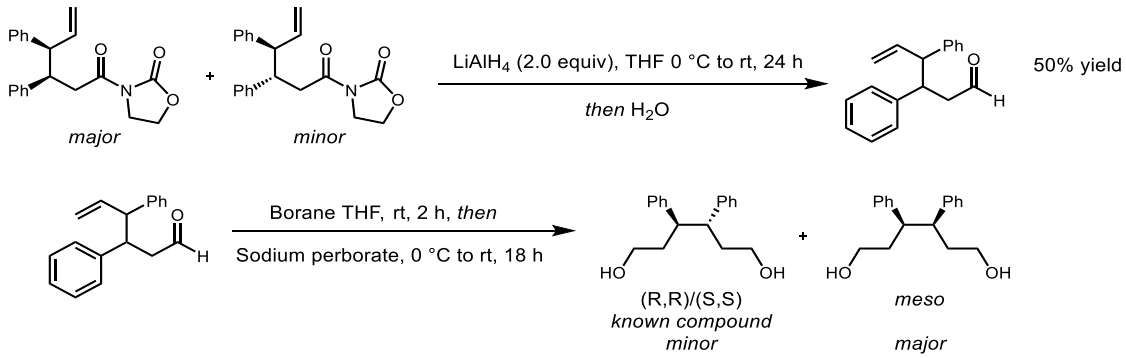
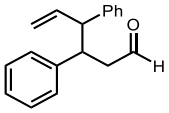


Figure S8: Comparison of dihedral angles for *syn*- and *anti*-configurations by DFT (B3LYP/6-31G) optimized geometry

4.3 Assignment for oxazolidinone products 5 by chemical correlation with known diol



A sample of **5aa** was reduced to the aldehyde with LAH and subsequently transformed into diol with Borane THF to give a diastereomeric mixture of 3,4-diphenyl-hexane-1,6-diol.
This was compared to published values in N. Kise, K. Iwasaki, N. Tokieda, and N. Ueda, *Org. Lett.*, 2001, **3**, 3241-3244.



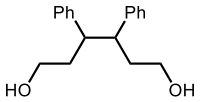
3,4-diphenylhex-5-enal (S2): (SGS-7-22)
5aa (0.19mmol, 1.0 equiv, 2.1:1 dr) is placed in a flame dried round bottom flask under nitrogen and dissolved in dry THF (0.2 M). Lithium aluminum hydride (14.4 mg, 2.0 equiv) is added at 0 °C and the reaction mixture is allowed to warm to room temperature and is stirred for 24 h or until consumption of

the starting material. The reaction mixture is then cooled to 0 °C and treated with water, extracted with ethyl acetate, dried over magnesium sulfate, filtered, and concentrated *in vacuo*. ^{1}H NMR analysis of the crude mixture showed 1:2.2 dr. Purification by preparatory TLC in 2:1 hexanes:ethyl acetate gave pure 3,4-diphenylhex-5-enal as a white solid (25.0 mg, 50% yield, 2.2:1 dr).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 – 9.61 (m, 1H'), 9.45 – 9.42 (m, 1H*), 7.36 – 7.10 (m, 8H*+8H'), 7.09 – 7.03 (m, 2H'), 7.02 – 6.95 (m, 2H*), 6.04 (dt, *J* = 16.9, 9.8 Hz, 1H'), 5.90 – 5.74 (m, 1H*), 5.17 (dd, *J* = 16.9, 0.8 Hz, 1H'), 5.12 (dd, *J* = 10.1, 1.5 Hz, 1H', 4.89 (d, *J* = 1.0 Hz, 1H*), 4.87 (d, *J* = 0.7 Hz, 1H*), 4.76 (dd, *J* = 9.6, 8.5 Hz, 1H), 3.64 – 3.42 (m, 2H*+2H'), 3.00 (ddd, *J* = 17.1, 5.0, 1.4 Hz, 1H'), 2.79 (ddd, *J* = 17.1, 9.2, 2.2 Hz, 1H'), 2.68 (ddd, *J* = 17.0, 9.1, 2.3 Hz, 1H*), 2.56 (ddd, *J* = 17.1, 4.6, 1.4 Hz, 1H*).

^{13}C NMR (101 MHz, $CDCl_3$) δ 201.8, 201.5, 142.2, 142.0, 141.8, 141.7, 140.2, 139.2, 128.9, 128.6, 128.4, 128.3, 128.1, 127.0_4, 126.9_9, 126.6, 126.4, 116.8, 116.6, 57.5, 56.0, 48.6, 48.5, 45.7, 45.2. Three peaks missing due to overlap.

HRMS (ESI) calcd for $C_{18}H_{19}O$ $[M+H]^+$: 251.14304, found: 251.14280.



3,4-diphenylhexane-1,6-diol: (SGS-7-34)

3,4-diphenylhex-5-enal (0.07mmol, 1.0 equiv, 2.2:1 dr) is placed in a flame dried round bottom flask under nitrogen. Borane THF (0.2 mL, 3.0 equiv, 1 M in THF) is added to the neat material at room temperature. The reaction mixture is stirred for 2 h, then cooled to 0 °C and treated with water (0.14 mL, 2 mL/mmol) followed by sodium perborate (34.9 mg, 5 equiv). The reaction mixture is allowed to gradually return to room temperature over 18 h. The crude reaction mixture is then extracted with ethyl acetate, dried over magnesium sulfate, filtered, and concentrated *in vacuo*. ^{1}H NMR analysis of the crude mixture showed 1:2.7 dr. Purification by preparatory TLC in 3:1 hexanes:ethyl acetate gave pure 3,4-diphenylhexane-1,6-diol as a white solid with diastereomers separated.

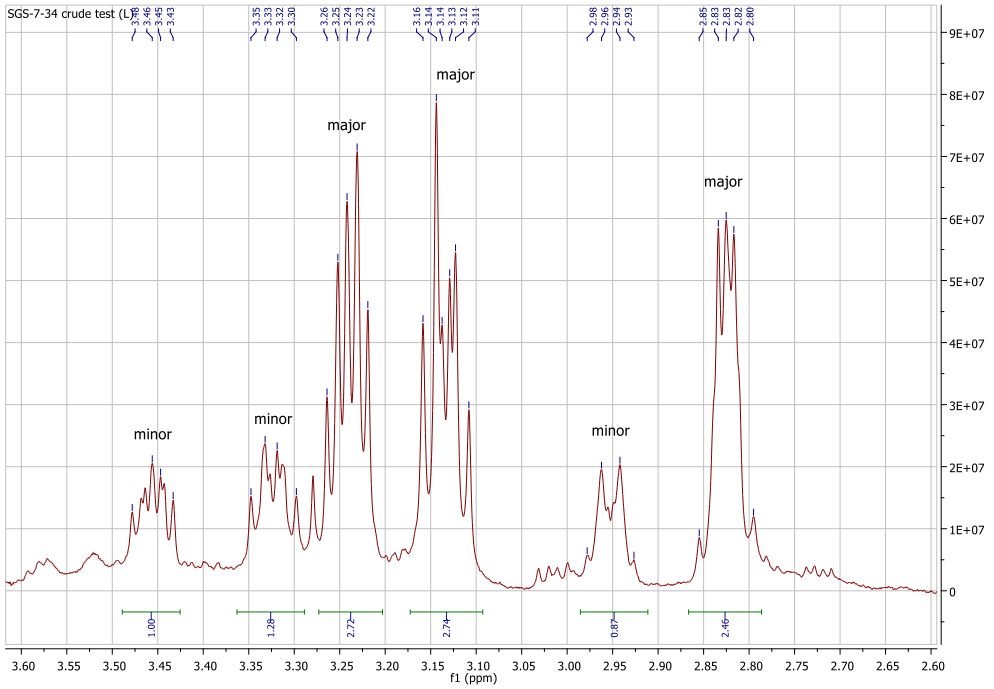
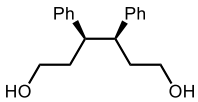


Figure S9: Crude NMR spectrum of transformation showing diastereomeric ratio before separation of diastereomers. The diastereomeric ratio is consistent with the crude spectra of the Michael addition.

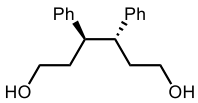


***meso*-3,4-diphenylhexane-1,6-diol (S3)**: (SGS-7-34-P3)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.6 Hz, 4H), 7.28 – 7.26 (m, 6H), 3.36 – 3.30 (m, 2H), 3.23 (dt, *J* = 10.5, 7.4 Hz, 2H), 2.95 – 2.88 (m, 2H), 1.68 – 1.62 (m, 4H), 1.59 (s broad, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.6, 128.9, 128.4, 126.8, 61.3, 49.0, 37.6.

HRMS (ESI) calcd for $C_{18}H_{23}O_2$ $[M+H]^+$: 271.16926, found: 271.16881.



(3R,4R)-3,4-diphenylhexane-1,6-diol and (3S,4S)-3,4-diphenylhexane-1,6-diol (S4): (SGS-7-34-P4)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 – 7.08 (m, 6H), 6.90 (d, *J* = 6.9 Hz, 4H), 3.53 (ddd, *J* = 10.8, 7.1, 4.8 Hz, 1H), 3.40 (ddd, *J* = 10.6, 7.9, 6.5 Hz, 1H), 3.07 – 2.98 (m, 2H), δ 2.18 (dtd, *J* = 10.8, 7.8, 3.6 Hz, 1H), 1.92 – 1.81 (m, 2H), 1.55 (s broad, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.0, 129.0, 128.0, 126.4, 61.4, 48.1, 36.1.

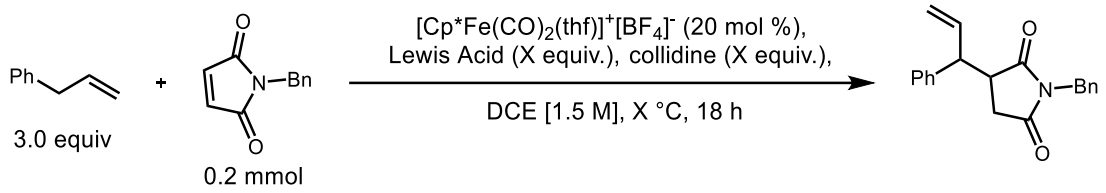
HRMS (ESI) calcd for $C_{18}H_{23}O_2$ $[M+H]^+$: 271.16926, found: 271.16897.

This spectra data matches literature the values (N. Kise, K. Iwasaki, N. Tokieda, and N. Ueda, *Org. Lett.*, 2001, **3**, 3241-3244). ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.05-7.17 (m, 6H), 6.87-6.92 (m, 4 H), 3.48-3.59 (m, 2 H), 3.34-3.46 (m, 2 H), 2.98-3.07 (m, 2 H), 2.12-2.24 (m, 2H), 1.80-1.93 (m, 2 H), 1.43 (brs, 2 H). ^{13}C NMR ($CDCl_3$) δ 147.76, 128.75, 127.58, 125.94, 60.86, 47.63, 35.94.

5. Reaction optimization

5.1 Optimization of Maleimide Michael Acceptors

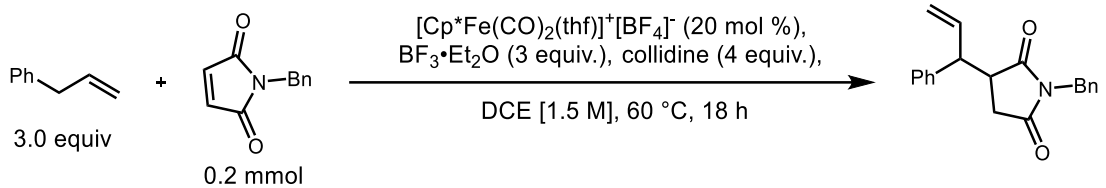
Table S1. Rection discovery and Lewis acid investigation



Entry	Lewis Acid	Lewis Acid Equiv	Base Equiv	Time (hr)	Temp. (°C)	NMR Maleimide (%)	NMR Yield (%)[a]
SGS-5-94-1	TIPSOTf	1.5	2.5	18	80	0	<10
SGS-5-94-2	TIPSOTf	2.2	2.5	18	60	0	<10
SGS-5-136	TMSTOf	2.5	3	18	60	0	0
SGS-5-101-1	BF_3•Et_2O	2	3	4	80	24	28
SGS-5-101-2	BF_3•Et_2O	2	3	4	50	57	27
SGS-5-137-1	BF_3•Et_2O	3	4	18	60	0	29
SGS-5-137-2	BF_3•Et_2O	6	4	18	60	0	<10
SGS-5-141	BF_3•Et_2O	3	4	3	60	15	30

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine.

Table S2: Exploration to find significant parameters

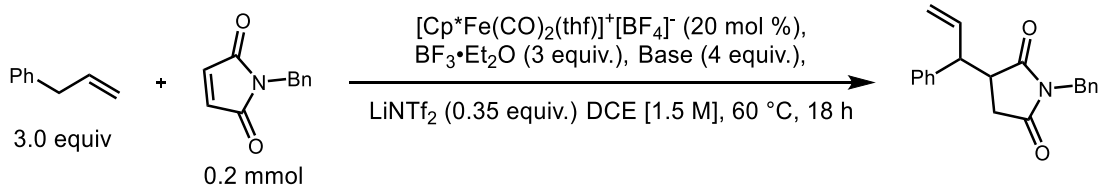


Entry	Variation from Standard Conditions	NMR Maleimide SM (%)	NMR Yield (%)[a]
SGS-5-142-1	100 °C	0	19
SGS-5-142-2	0.5 M DCE	12	15
SGS-5-142-3	0.35 equiv $LiNTf_2$	0	37
SGS-5-142-4	0.6 equiv $LiNTf_2$	0	32

SGS-5-142-5	1.0 equiv $LiNTf_2$	0	38
SGS-5-142-6	0.1 equiv $LiNTf_2$	trace	23

[a]Yields determined by ^1H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine.

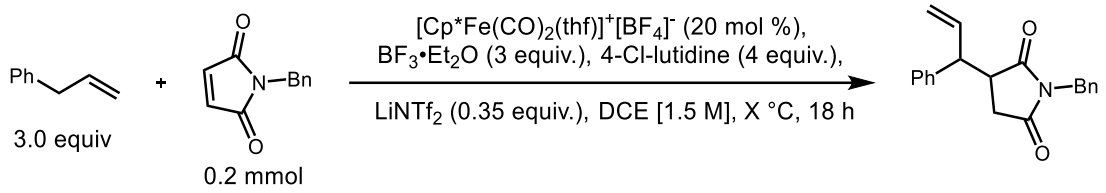
Table S3: Base optimization



Entry	Base	NMR Maleimide SM (%)	NMR Yield (%)[a]
SGS-5-143-1	TMPH	0	0
SGS-5-143-2	4-Cl-Lutidine	0	47
SGS-5-143-3	Lutidine	0	40
SGS-5-143-4	4-Br-Lutidine	0	44
SGS-7-15	2,3,5,6-Tetramethylpyrazine	0	16 (5.2:1 dr)

[a]Yields determined by ^1H NMR using 2,4-dinitrotoluene as the internal standard. TMPH = 2,2,6,6-tetramethylpiperidine.

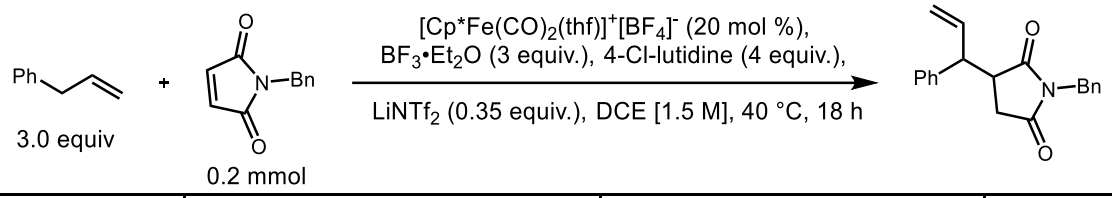
Table S4: Temperature optimization



Entry	Temperature (°C)	NMR Maleimide SM (%)	NMR Yield (%)[a]
SGS-5-143-2	60	0	47
SGS-5-159	50	0	54
SGS-5-147-1	40	0	64
SGS-5-148-1	30	30	37
SGS-5-148-2	RT	24	23

[a]Yields determined by ^1H NMR using 2,4-dinitrotoluene as the internal standard.

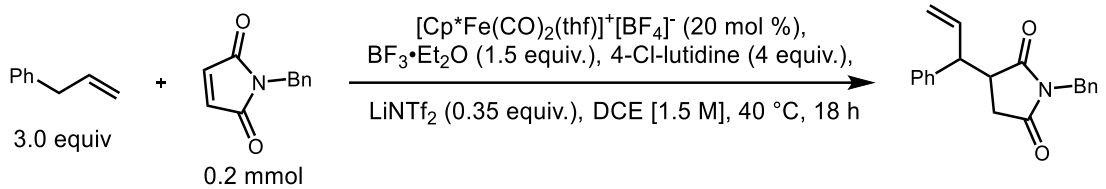
Table S5: Stoichiometry optimization



Entry	Ratio (Lewis Acid/Base)	NMR Maleimide SM (%)	NMR Yield (%)[a]
SGS-5-147-1	3/4	0	64
SGS-5-158-1	3/3	0	45
SGS-5-158-2	2.2/4	0	69
SGS-5-158-3	2.2/3	0	57
SGS-5-158-5	2.2/5	0	70
SGS-5-158-6B	1.0/4	0	71
SGS-5-158-4	1.5/4	0	80
SGS-5-158-7	1.5/4 + 8hr run time	9	53

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard.

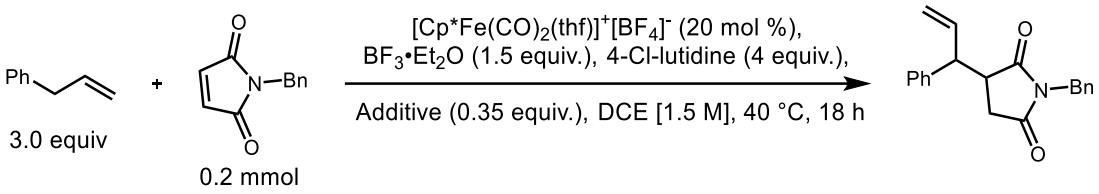
Table S6: Diastereomeric ratio optimization



Entry	Variation from Standard Conditions	NMR Maleimide SM (%)	NMR Yield (%)[a]	d.r.
SGS-6-11-1	none	0	83	3.7:1
SGS-6-11-2	35 °C	0	82	3.8:1
SGS-6-11-3	30 °C	0	55	4.2:1
SGS-6-11-5	0.6 equiv $LiNTf_2$	5	66	4.0:1
SGS-6-11-6	0.2 equiv $LiNTf_2$	4	74	3.9:1

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard.

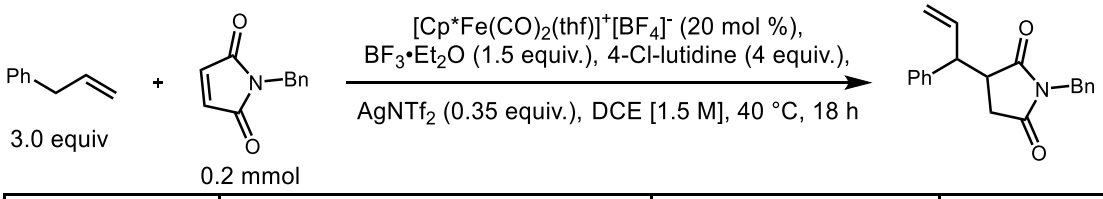
Table S7: Additive optimization for diastereomeric ratio



Entry	Additive	NMR Maleimide SM (%)	NMR Yield (%)[a]	d.r.
SGS-6-11-4	$CuNTf_2$	61	17	3.8:1
SGS-5-185-1	$Ca(NTf_2)_2$	86	*trace*	-
SGS-5-185-2	$Mg(NTf_2)_2$	21	75	6.4:1
SGS-5-185-3	$Zn(NTf_2)_2$	0	19	1.7:1
SGS-5-185-4	$Zn(OTf)_2$	60	20	5.7:1
SGS-5-185-5	LiOTf	58	29	6.4:1
SGS-5-185-6	$AgNTf_2$	0	77	7.3:1
SGS-5-187-1	$Yb(OTf)_3$	80	12	-
SGS-5-187-2	$Bi(OTf)_3$	80	<10	-
SGS-5-187-4	AgOTf	89	0	-
SGS-5-187-5	$Eu(OTf)_3$	96	0	-
SGS-5-187-6	$Er(OTf)_3$	87	<10	-
SGS-7-14-1	Tritylium tetrafluoroborate	43	12	5.8:1
SGS-7-14-2	Triphenyl borane	40	10	6.0:1

[a]Yields determined by ^1H NMR using 2,4-dinitrotoluene as the internal standard.

Table S8: Exploration with silver bistriflimide additive



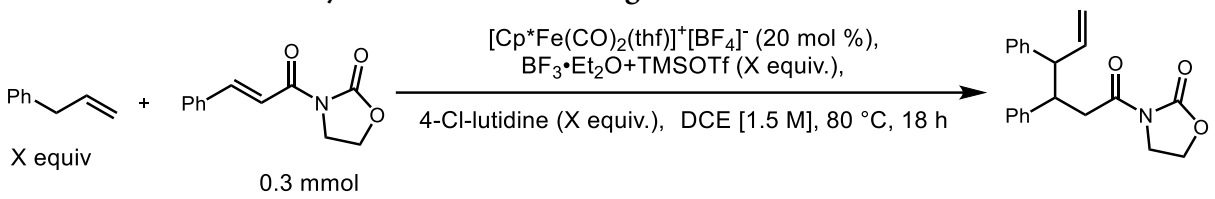
Entry	Variation from Standard Conditions	NMR Maleimide SM (%)	NMR Yield (%)[a]	d.r.
SGS-5-185-6	none	0	77	7.3:1

SGS-5-188-8	0.5 equiv additive	8	63	6.8:1
SGS-5-188-7	0.3 equiv additive	11	67	6.3:1
SGS-5-188-5	No additive	41	25	6.4:1
SGS-5-189-1	No BF_3•Et_2O	88	0	-
SGS-5-188-9	No Catalyst	100	0	-
SGS-5-188-6	Toluene as solvent	8	75	7.2:1
SGS-5-192	Toluene as solvent + 24 hr reaction	0	(89)[b]	7.6:1
SGS-7-1	No Additive + Toluene as solvent + 24 hr reaction	67	9	6.3:1
SGS-7-7	Toluene as solvent + 24 hr reaction + lutidine as base	61	27	6.7:1
SGS-7-18	Pentaethyl substituted cyclopentadiene ligand	21	24	14.3:1

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. [b]Isolated Yield.

5.2 Optimization of *N*-Acyl-Oxazolidinone Michael Acceptors

Table S9. Rection discovery and Lewis acid investigation

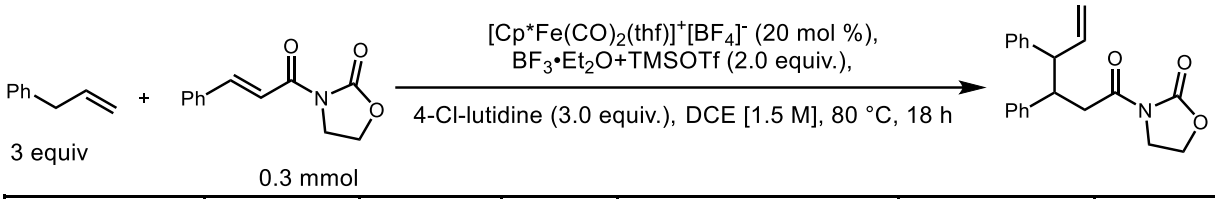


Entry	Additive (20%)	Ratio (alkene/Co-Lewis Acids/Base)	Temp. (°C)	NMR SM (%)	NMR Yield (%)[a]	d.r.
SGS-4-96-1	None	2/2/3	80	19	17	2.4:1
SGS-4-96-2	$Yb(OTf)_3$	2/2/3	80	30	19	2.4:1
SGS-4-96-3	CuOTf	2/2/3	80	23	14	2.0:1
SGS-5-1-1	$Yb(OTf)_3$	3/2/3	80	23	27	3.2:1
SGS-5-1-2	$Yb(OTf)_3$	2/2/4	80	23	16	2.0:1
SGS-5-1-3	$Yb(OTf)_3$	2/1.2/4	80	43	7	1.4:1

SGS-5-1-4	$Yb(OTf)_3$	2/1.2/3	80	47	14	2.2:1
SGS-5-1-5A	$Yb(OTf)_3$	2/1.2/2	80	30	16	2.8:1
SGS-5-1-5B	$Yb(OTf)_3$	3/1.2/2	80	51	19	3.2:1
SGS-5-1-5C	$Yb(OTf)_3$	2/1.2/2	100	45	11	2.3:1

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine.

Table S10. Control reactions and comparison of Lewis acids needed

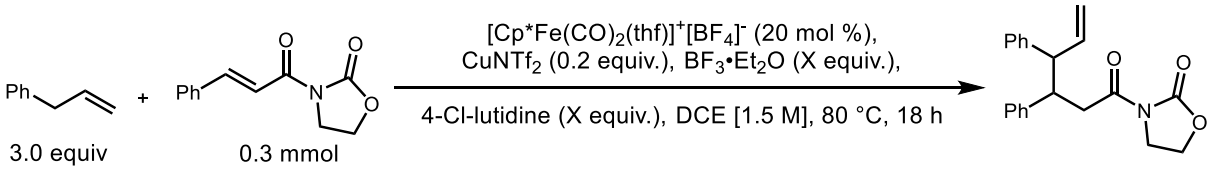


Entry	BF_3•Et_2O	TMSOTf	Additive	NMR SM (%)	NMR Yield (%)[a]	d.r.
SGS-5-1-1	Y	Y	Y	23	27	2.3:1
SGS-5-16-1	Y	-	-	24	<10	-
SGS-5-16-2	Y	-	Y	58	0	-
SGS-4-96-1	Y	Y	-	19	17	2.4:1
SGS-5-16-3	-	Y	-	79	0	-
SGS-5-16-4	-	Y	Y	73	0	-

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine.

The results of a control reaction indicated that BF_3•Et_2O alone is sufficient for the reaction, but the addition of TMSOTf and Additive increase the yield. Reactions tried with the stronger base 2,4,6-trimethyl pyridine gave no product, presumably because it ties up more of the available Lewis acid. Together, these indicate that a significant increase in the amount of Lewis acid is necessary.

Table S11. Increasing free Lewis acid



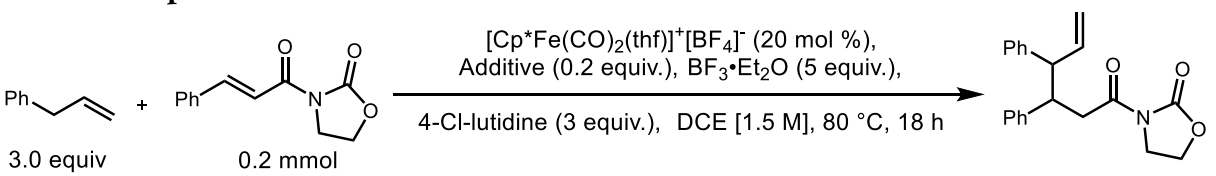
Entry	Ratio (BF_3•Et_2O/Base)	Base	NMR SM (%)	NMR Yield (%)[a]	d.r.

SGS-5-19-1	3/3	4-Cl-Lutidine	19	41	2.6:1
SGS-5-19-2	2/2	Collidine	48	21	3.9:1
SGS-5-19-3	3/3	Collidine	31	19	6.1:1
SGS-5-19-4	4/3	Collidine	47	29	6.6:1
SGS-5-21-1	4/3	4-Cl-Lutidine	4	39	2.1:1
SGS-5-21-2	3/2	4-Cl-Lutidine	44	trace	-
SGS-5-21-3	4/2	Collidine	39	<10%	-
SGS-5-24-1	3/2	Collidine	20	21	-
SGS-5-24-2	3/1.2	Collidine	35	0	-
SGS-5-24-3	3/1.2	Collidine	63	0	-
SGS-5-24-4	5/3	Collidine	9	35	2.4:1
SGS-5-36-1	5/3	4-Cl-Lutidine	48	46	3.8:1

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine.

These results validate the hypothesis that significant free BF_3•Et_2O is needed in the reaction. Good yields are observed with equivalents of Lewis acid greater than the equivalents of base, which was unprecedented for our group's explorations of cyclopentadienyldicarbonyl iron complex catalysis.

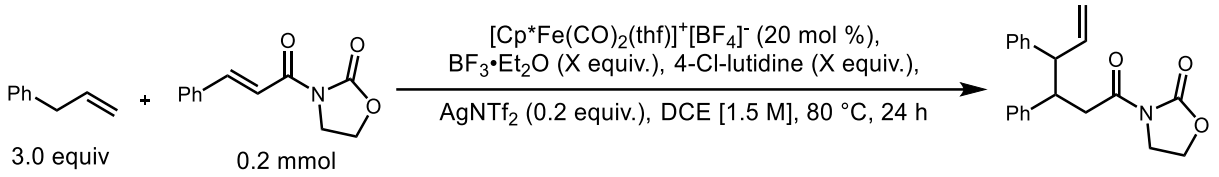
Table S12. Optimization of Additive



Entry	Additive	Additive Equivalents	NMR SM (%)	NMR Yield (%)[a]	d.r.
SGS-5-39-4	$LiNTf_2$	0.2	18	67	2.5:1
SGS-5-39-3	$Cu(NTf_2)_2$	0.2	34	42	2.8:1
SGS-5-39-5	$Yb(OTf)_3$	0.2	40	54	2.7:1
SGS-5-39-6	$Zn(OTf)_2$	0.2	30	57	2.8:1
SGS-6-21	$LiNTf_2$	1.0	0	85 (88%)[b]	2.2:1

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. [b]Isolated Yield.

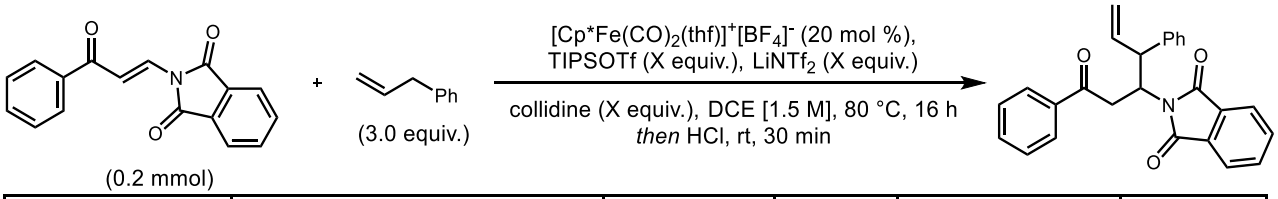
Table S13. Control Experiments



Entry	$BF_3•Et_2O$ Equivalents	Base Equivalents	NMR SM (%)	NMR Yield (%)[a]	d.r.
SGS-7-6-1	1.5	4	54	*trace*	-
SGS-7-6-2	5.0	3	0	19	1.4:1

5.3 Optimization of Phthalimide Michael Acceptors

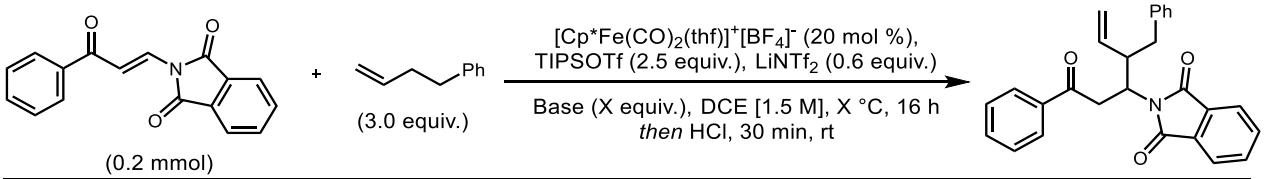
Table S14. Optimization for substituted allylbenzene derivatives



Entry	Ratio (SiR_3/Base/additive)	Silyl Triflate	Temp.	NMR SM (%)	NMR Yield (%)[a]
SGS-5-114-1	1.5/2.0/0.6	TIPSOTf	80	41	29
SGS-5-114-2	1.5/2.0/0.6	TMSOTf	80	23	18
SGS-5-144-2	2.5/3.0/0.6	TIPSOTf	80	0	90 (93%)[b]
SGS-5-144-1	2.5/3.0/0.6	TMSOTf	80	0	37
SGS-5-144-3	2.5/3.0/0.3	TIPSOTf	80	0	73
SGS-5-164-1	2.5/3.0/0.6	TIPSOTf	60	0	85

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. [b]Isolated Yield.

Table S15. Optimization for unactivated alkene substrates



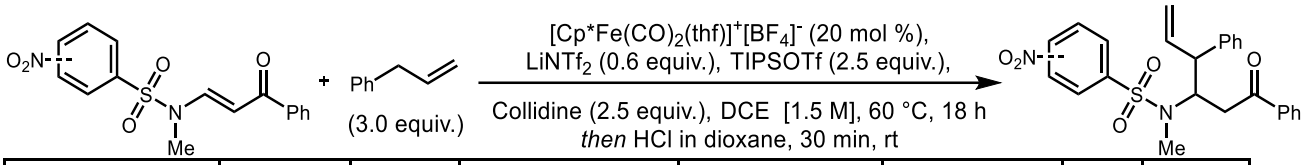
Entry	Temp.	Base	Base (equiv)	NMR Yield (%)[a]
SGS-6-3-1	80	Collidine	3	<20

SGS-6-3-2	90	Collidine	3	30
SGS-6-3-3	80	Collidine	4	44
SGS-6-3-4	80	TMPH	3	45
SGS-6-3-5	80	TMPH	4	79 (66)[b]

[a]Yields determined by ^{1}H NMR using 2,4-dinitrotoluene as the internal standard. [b]Isolated Yield. TMPH = 2,2,6,6-tetramethylpiperidine.

5.4 Optimization of Sulfonamide Michael Acceptors

Table S16. Optimization of reaction



Entry	Solvent	Temp	TIPSOTf equiv	Base equiv	NMR Yield (%)	NO_2	SM (%)
SGS-5-146-1	DCE	60	1.5	2.5 (Collidine)	43	*-p*	0
SGS-5-168-4	DCE	60	1.5	2.5 (4-Cl-Lutidine)	18	*-p*	10
SGS-5-146-2	DCE	60	2.5	3.0 (Collidine)	43	*-p*	0
SGS-5-168-1	DCE	80	2.5	3.0 (Collidine)	22	*-p*	0
SGS-5-146-3	PhCl	60	2.5	3.0 (Collidine)	31	*-p*	7
SGS-5-146-5	DCE	60 (8 h)	2.5	3.0 (Collidine)	22	*-p*	20
SGS-5-168-3	DCE	60	2.5	2.5 (Collidine)	46	*-p*	*trace*
SGS-5-168-6	DCE	50	2.5	2.5 (Collidine)	18	*-p*	14
SGS-5-168-5	DCE	60	2.5	2.5 (4-Cl-Lutidine)	35	*-p*	0
SGS-5-168-7	DCE	60	2.5	2.5 (TMPH)	42	*-p*	*trace*

SGS-5-168-8	DCE	60	2.5+0.6 equiv $LiNTf_2$	2.5 (Collidine)	67	*-p*	0
SGS-5-172-1	DCE	60	2.0+0.6 equiv $LiNTf_2$	2.0 (Collidine)	60	*-p*	0
SGS-5-172-2	DCE	60	2.5+0.8 equiv $LiNTf_2$	2.5 (Collidine)	67	*-p*	0
SGS-5-184-1	DCE	60	2.5+0.6 equiv $LiNTf_2$	2.5 (Collidine)	84 (72)[b]	*-o*	0
SGS-5-184-2	DCE	60	2.5+0.6 equiv $LiNTf_2$	3.5 (Collidine)	76	*-o*	0

[a]Yields determined by ^1H NMR using 2,4-dinitrotoluene as the internal standard. Collidine = 2,4,6-trimethyl pyridine. TMPH = 2,2,6,6-tetramethylpiperidine.

6. Unsuccessful substrates

Unsucessful Michael Acceptors:

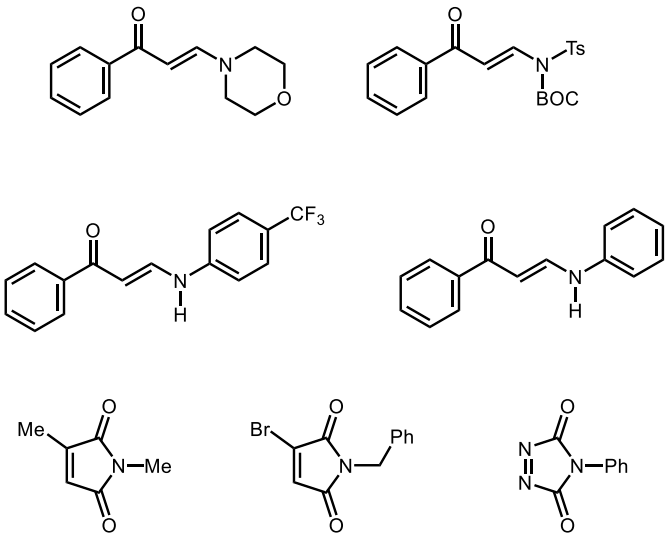
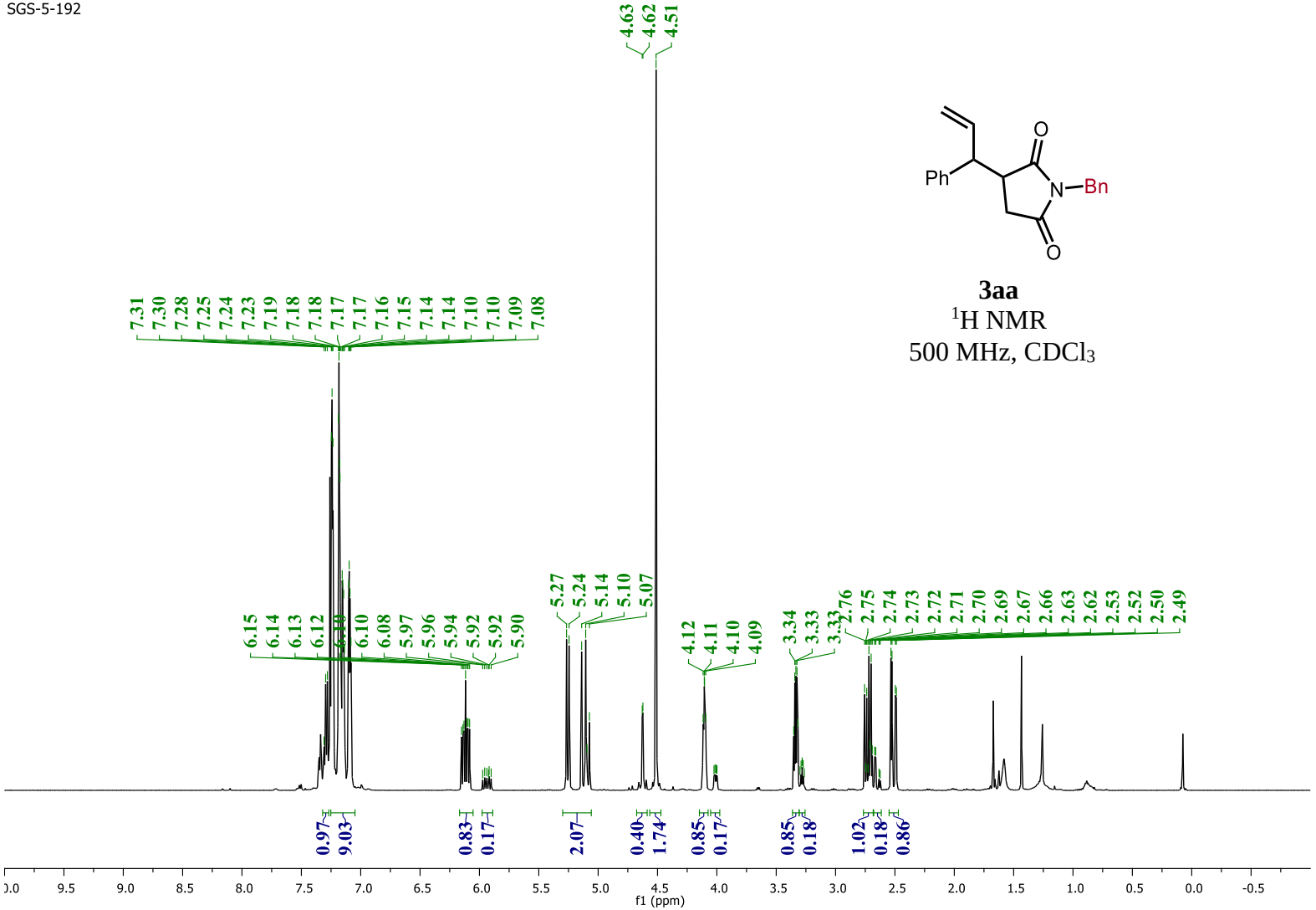


Figure S10: Unsuccessful nitrogen-containing Michael acceptors

7. Copies of NMR spectra of products and substrates

7.1 NMR spectra: Maleimide Scope

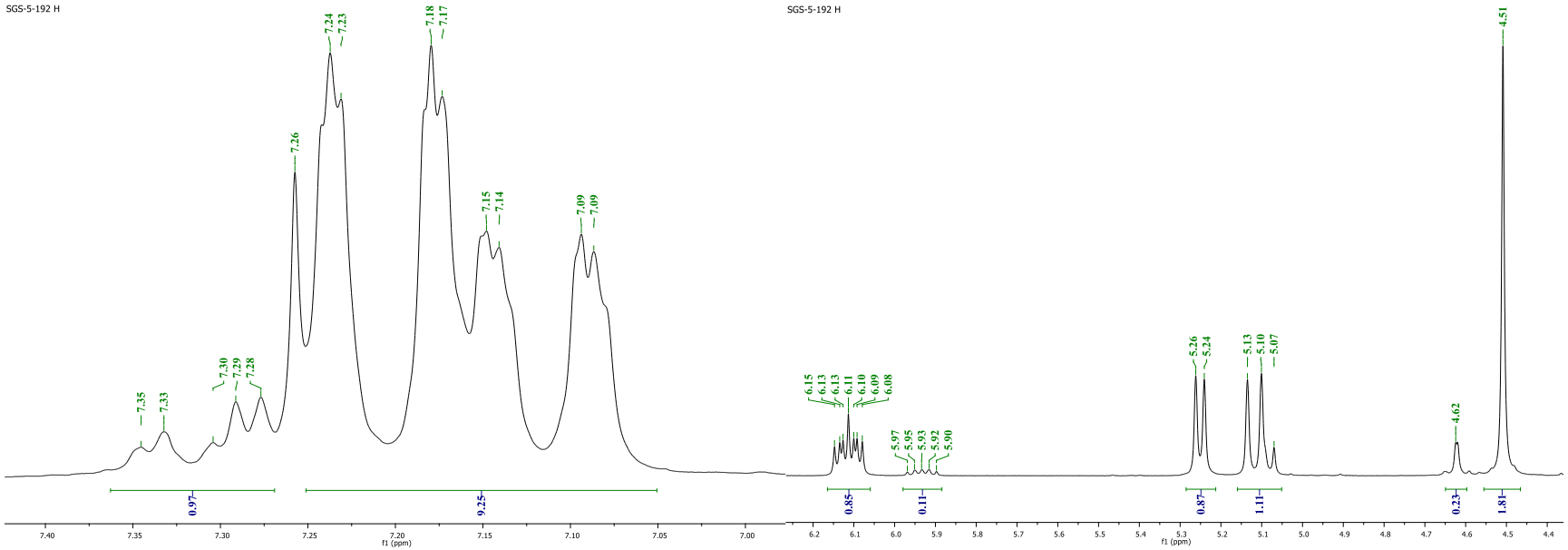
SGS-5-192

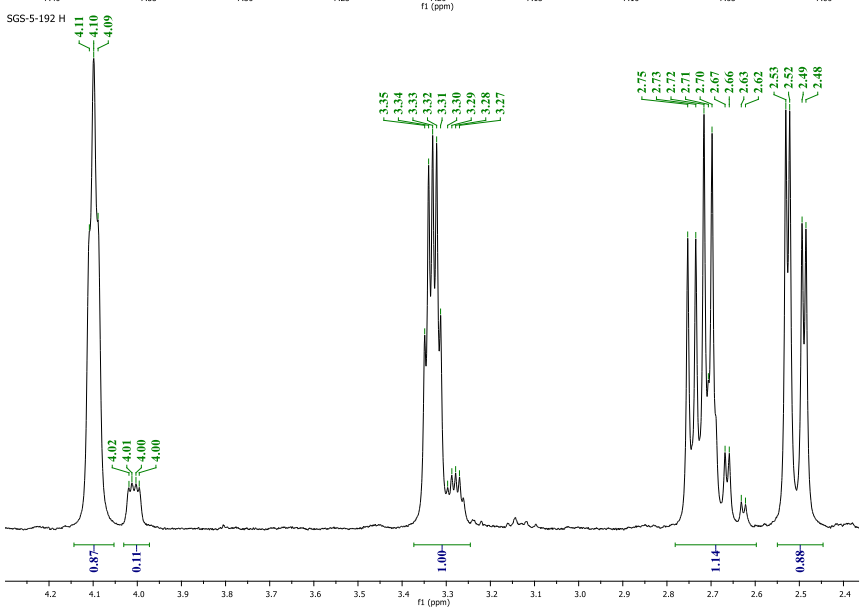


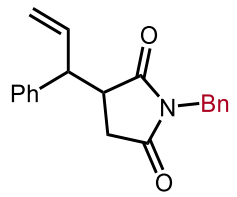
3aa

^{1}H NMR

500 MHz, $CDCl_3$

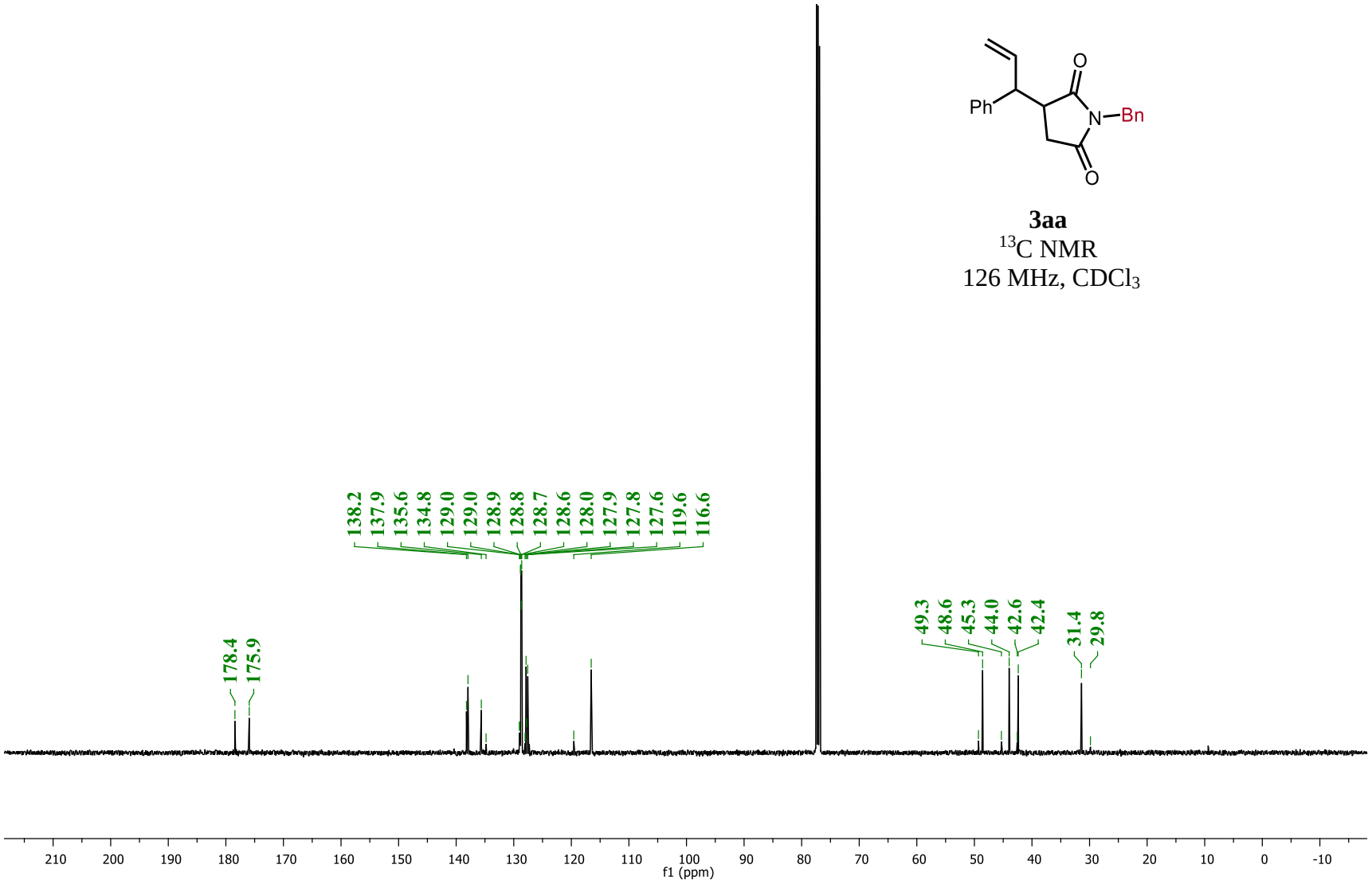






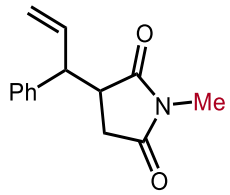
3aa
^{1}H NMR
500 MHz, $CDCl_3$

SGS-5-192 C



3aa
^{13}C NMR
126 MHz, $CDCl_3$

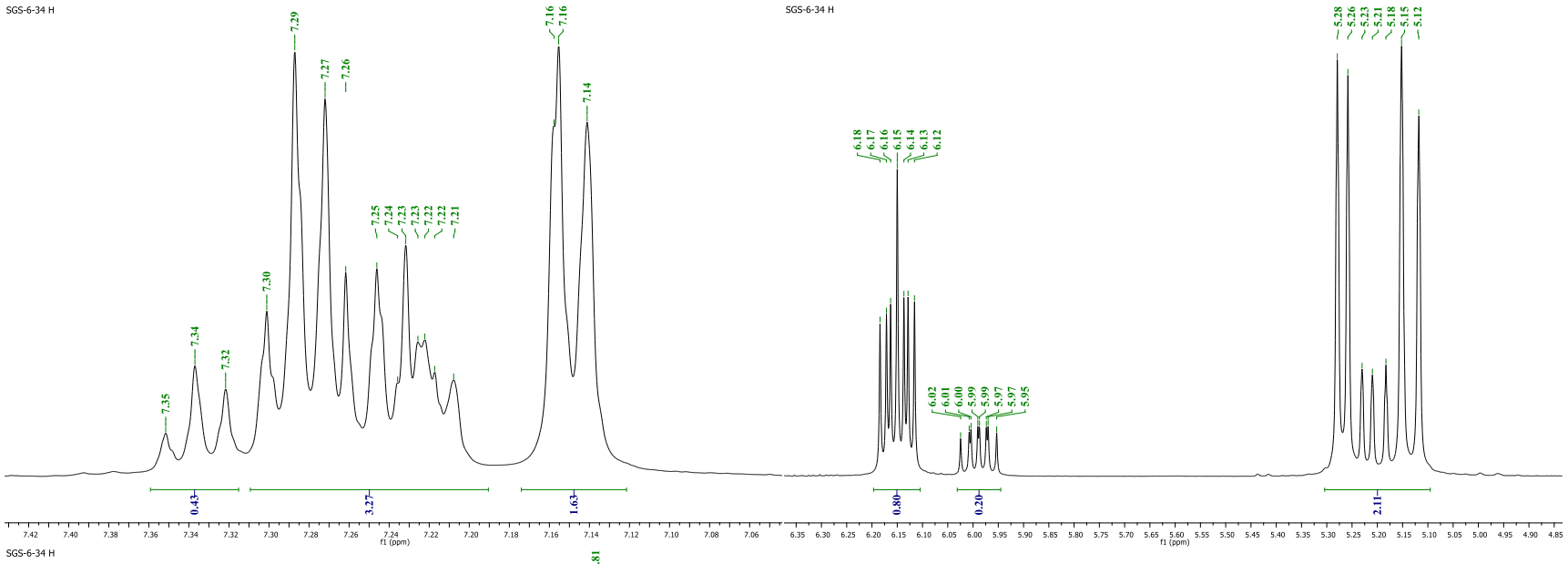
SGS-6-34 H

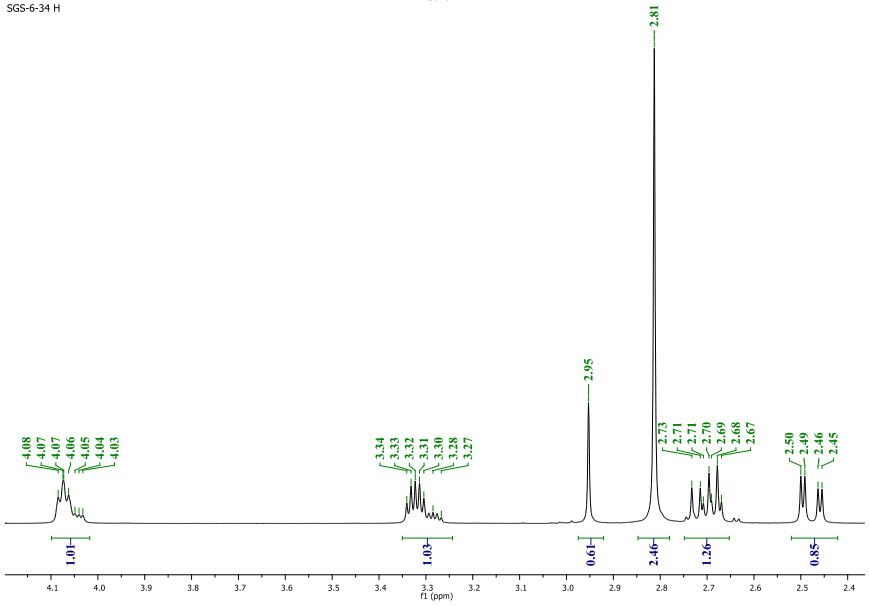


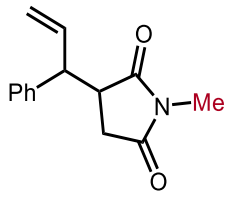
3ba

^{1}H NMR

500 MHz, $CDCl_3$





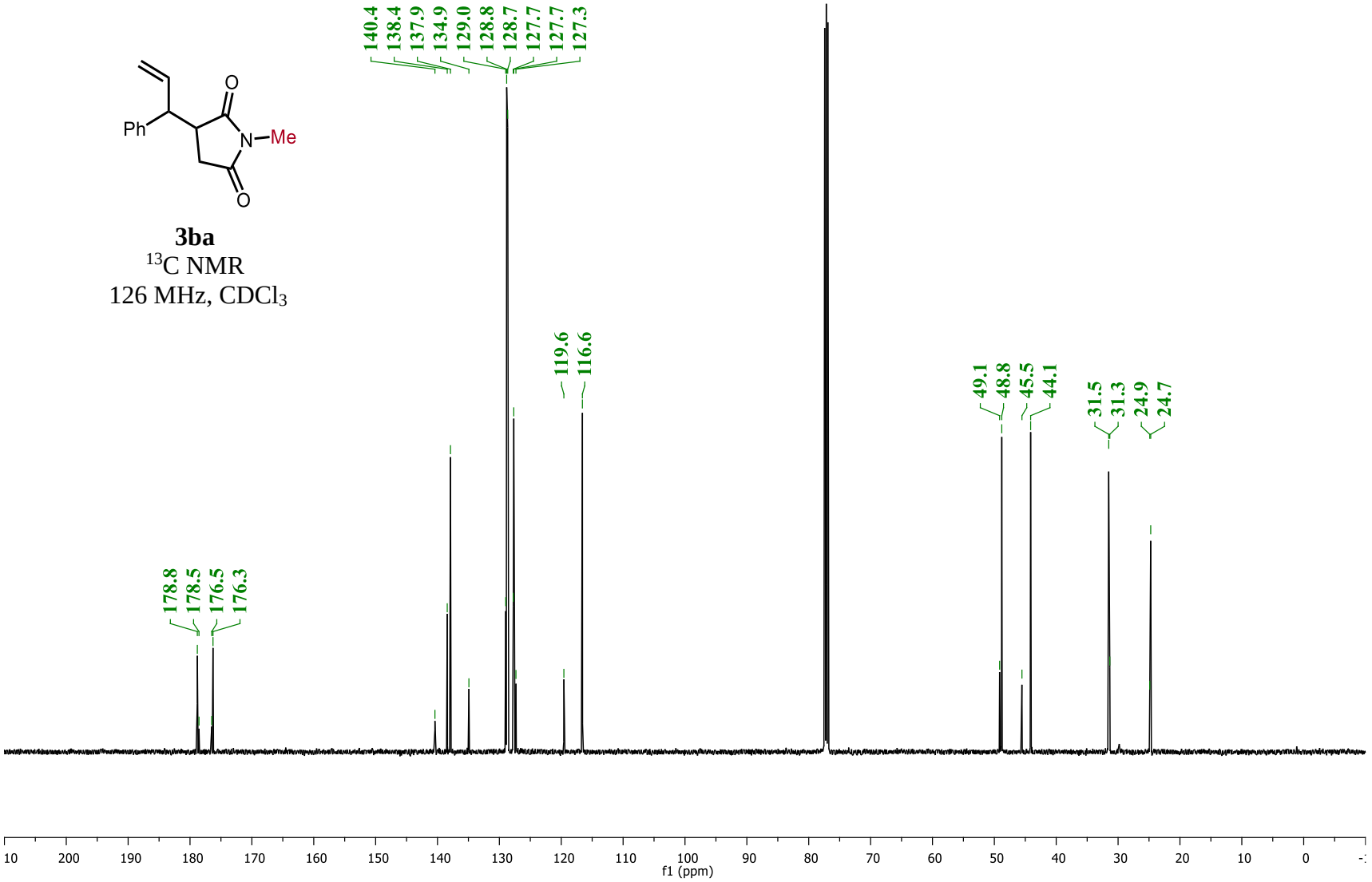


3ba

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-34 C

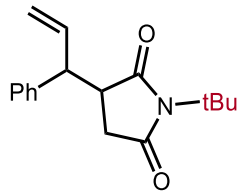


3ba

^{13}C NMR

126 MHz, $CDCl_3$

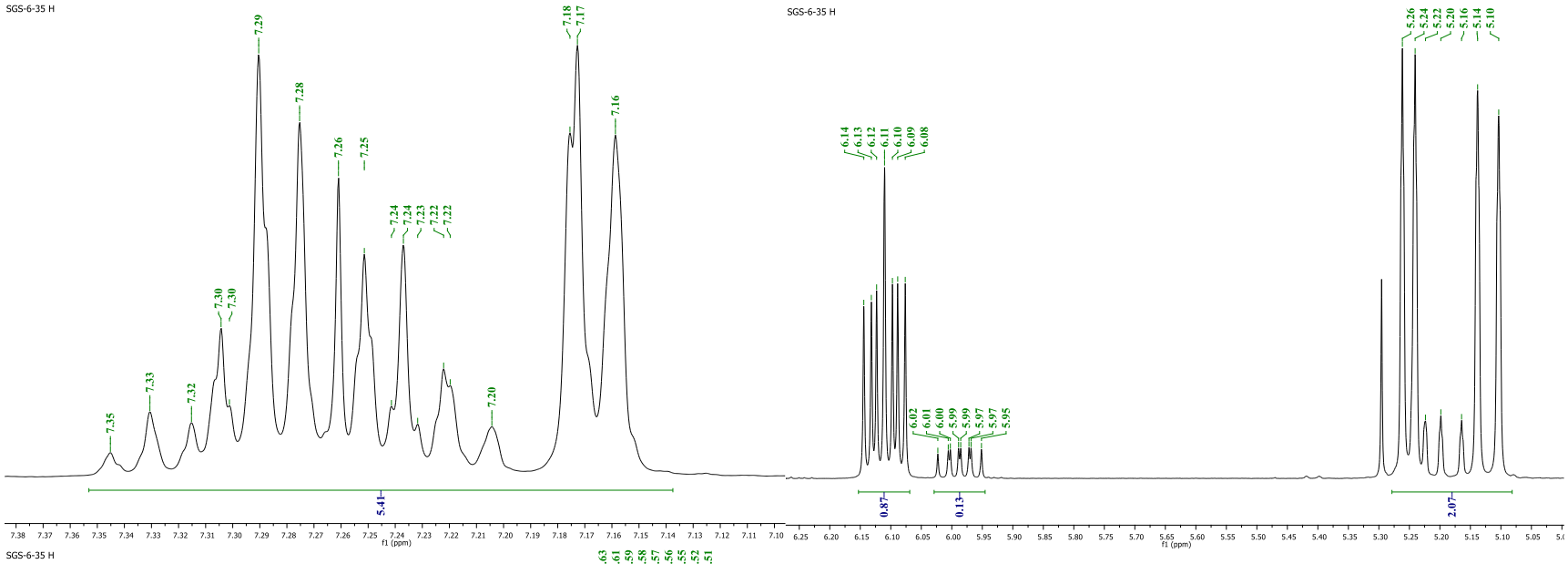
SGS-6-35 H

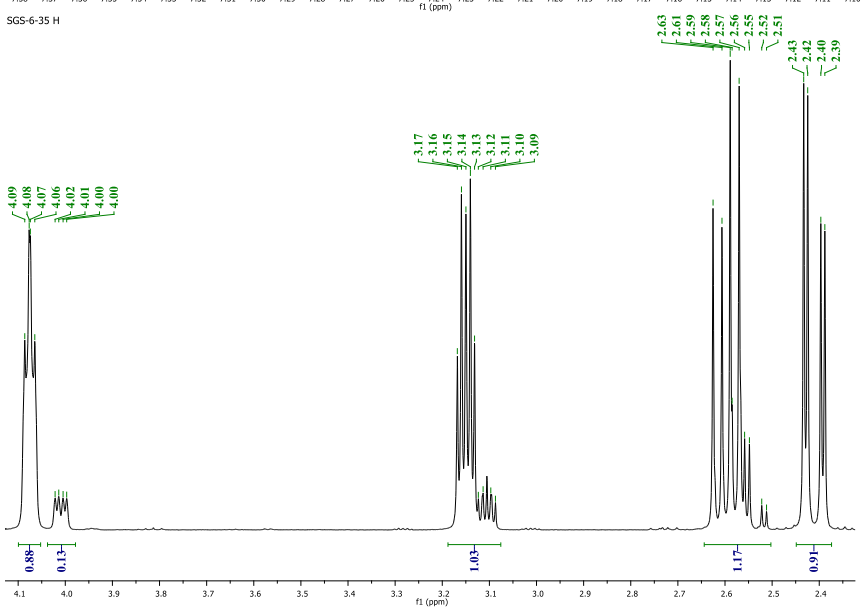


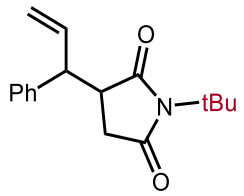
3ca

^{1}H NMR

500 MHz, $CDCl_3$

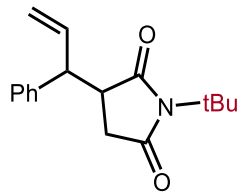






3ca
^{1}H NMR
500 MHz, $CDCl_3$

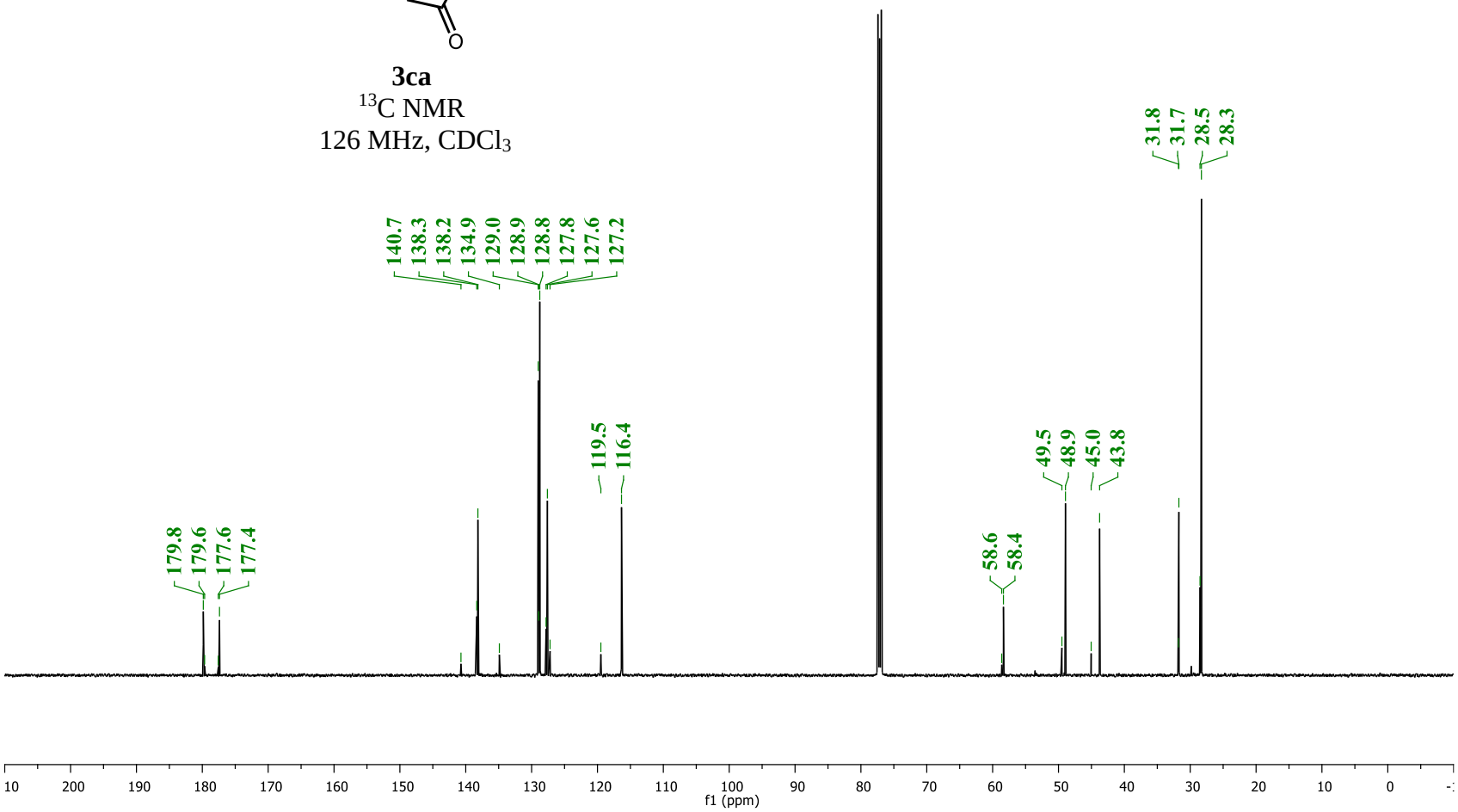
SGS-6-35 C

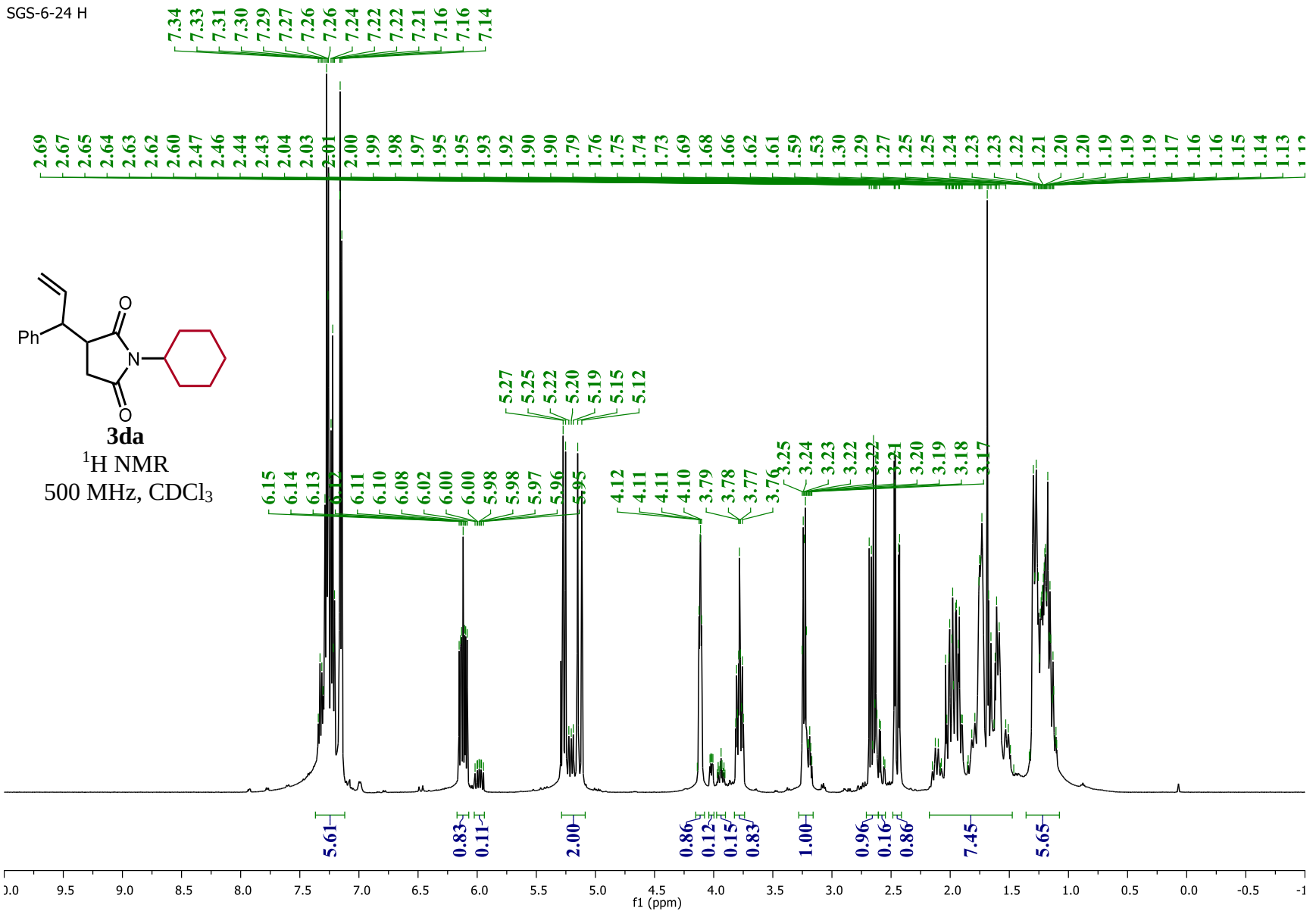


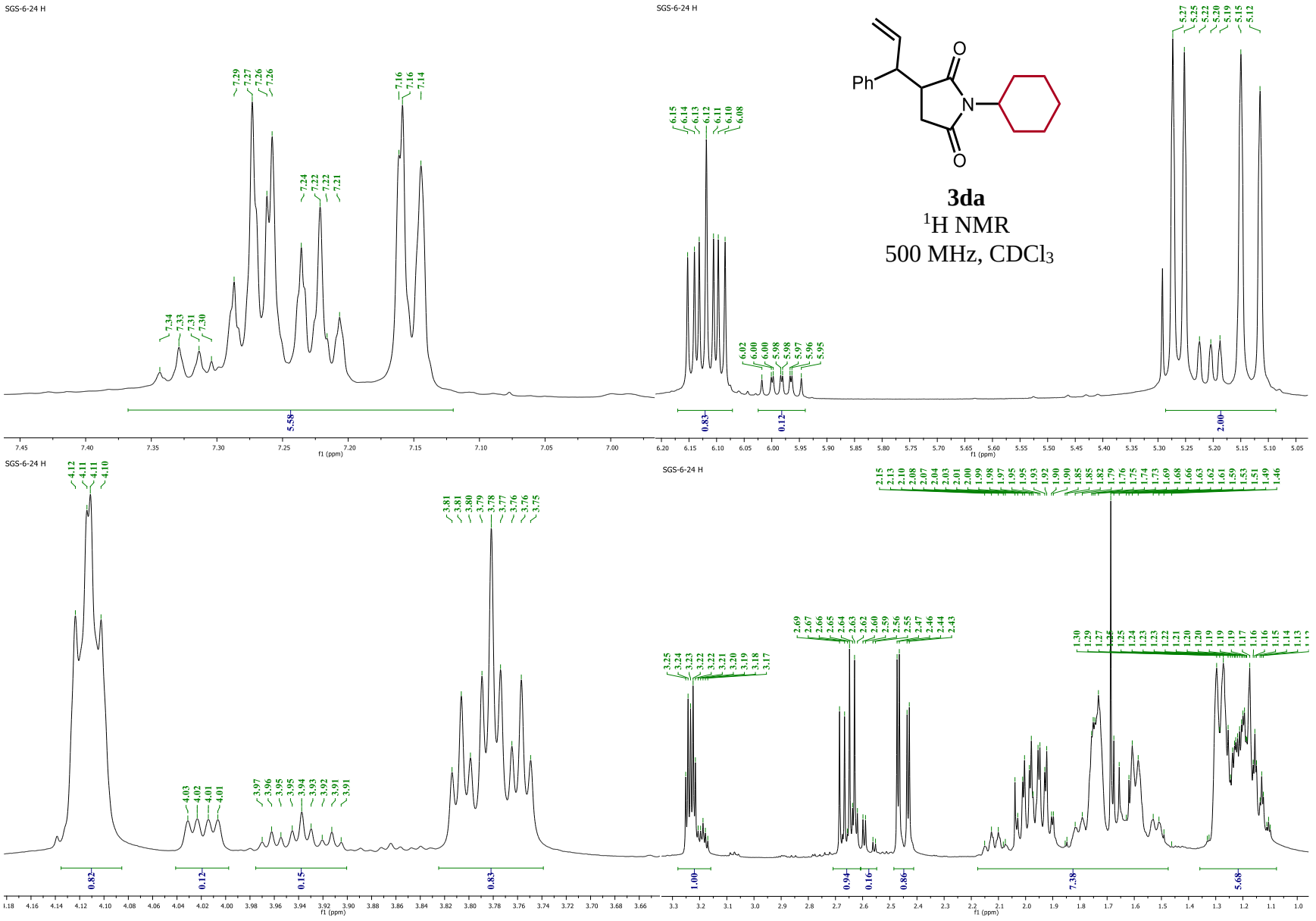
3ca

^{13}C NMR

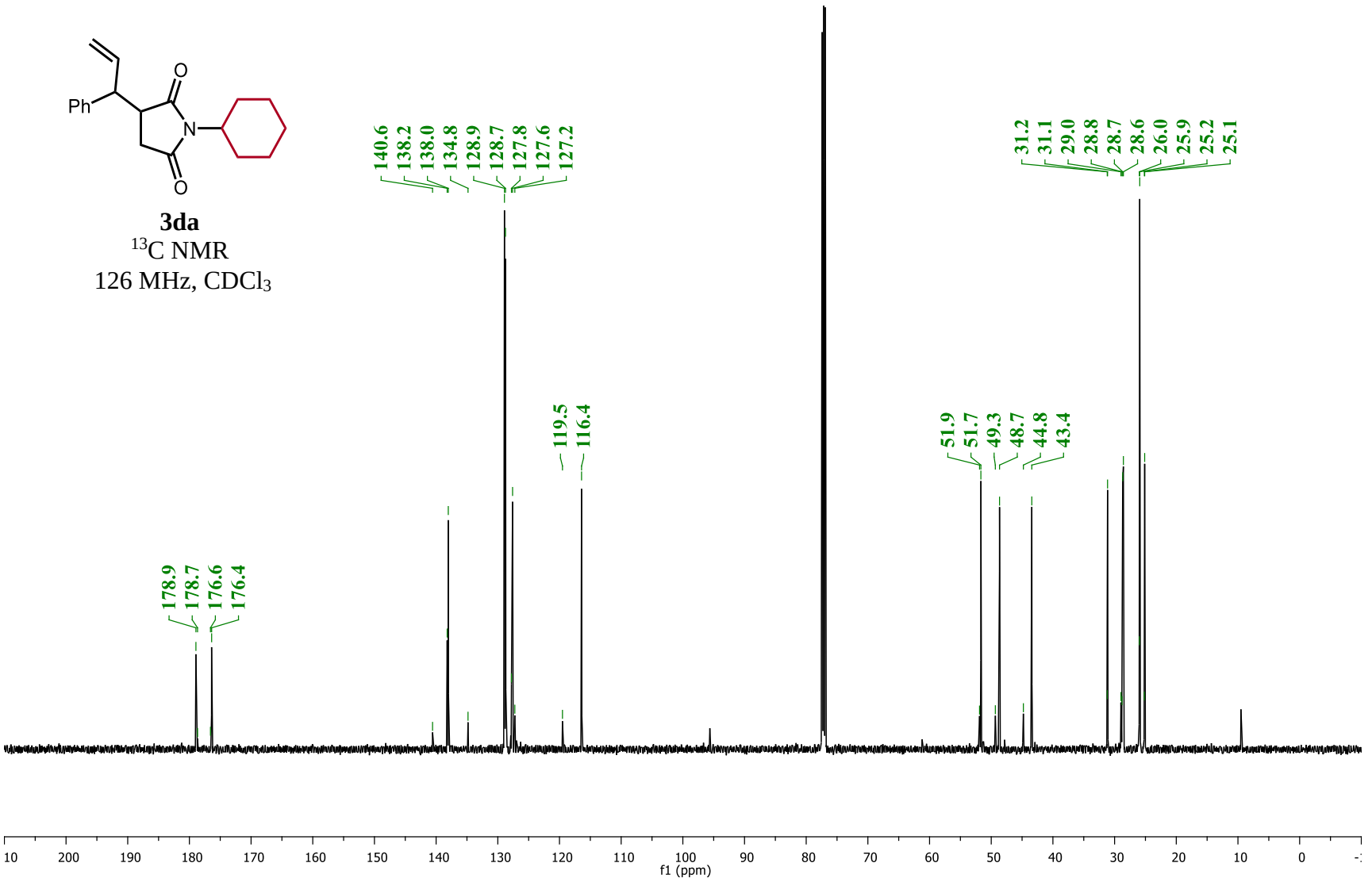
126 MHz, $CDCl_3$

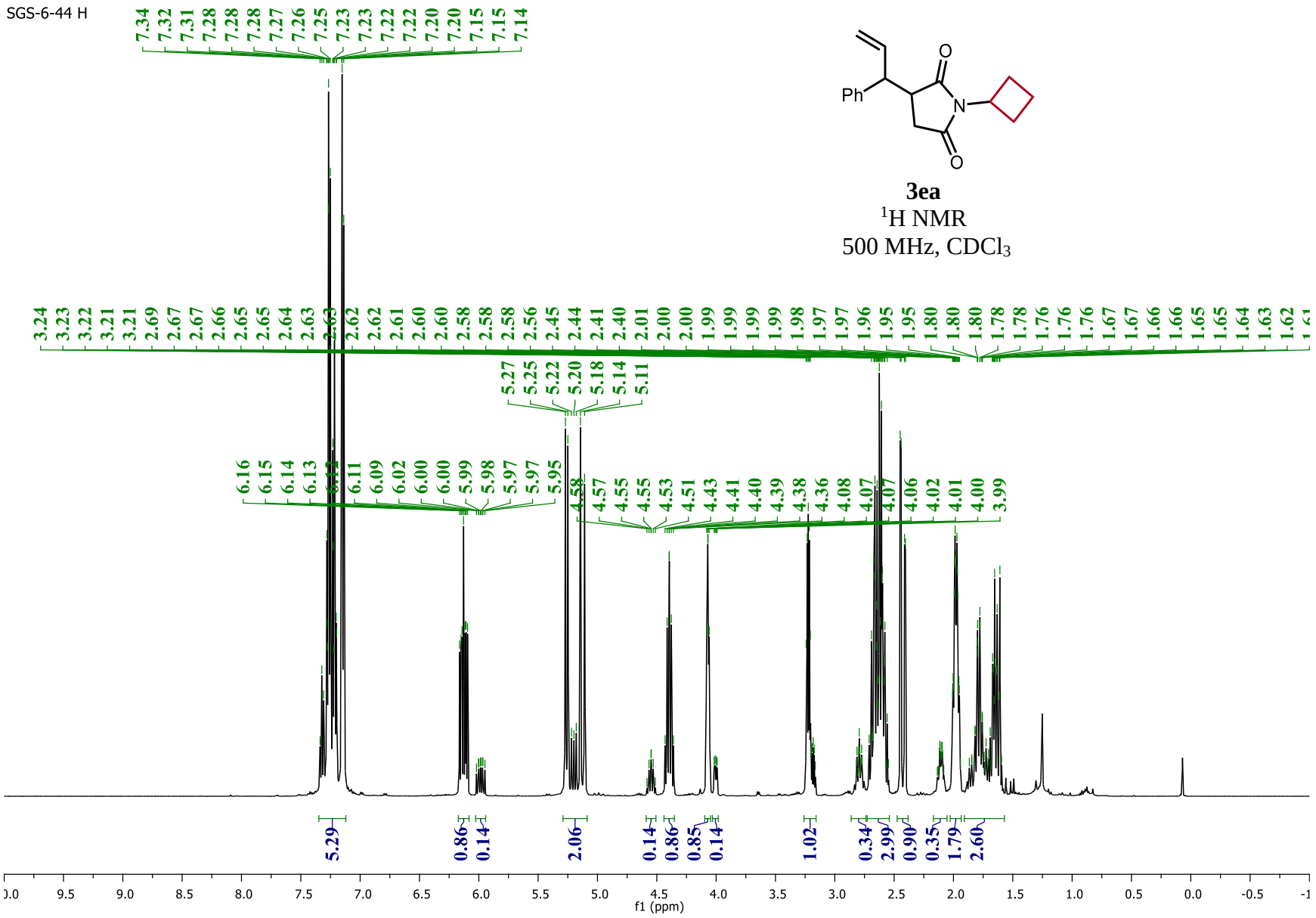



SGS-6-24 H
Ph
O
N
O
3da
1H NMR
500 MHz, CDCl3
f1 (ppm)


SGS-6-24 H
SGS-6-24 H
Ph
O
N
O
3da
1H NMR
500 MHz, CDCl3
SGS-6-24 H
SGS-6-24 H
f1 (ppm)

SGS-6-24 C

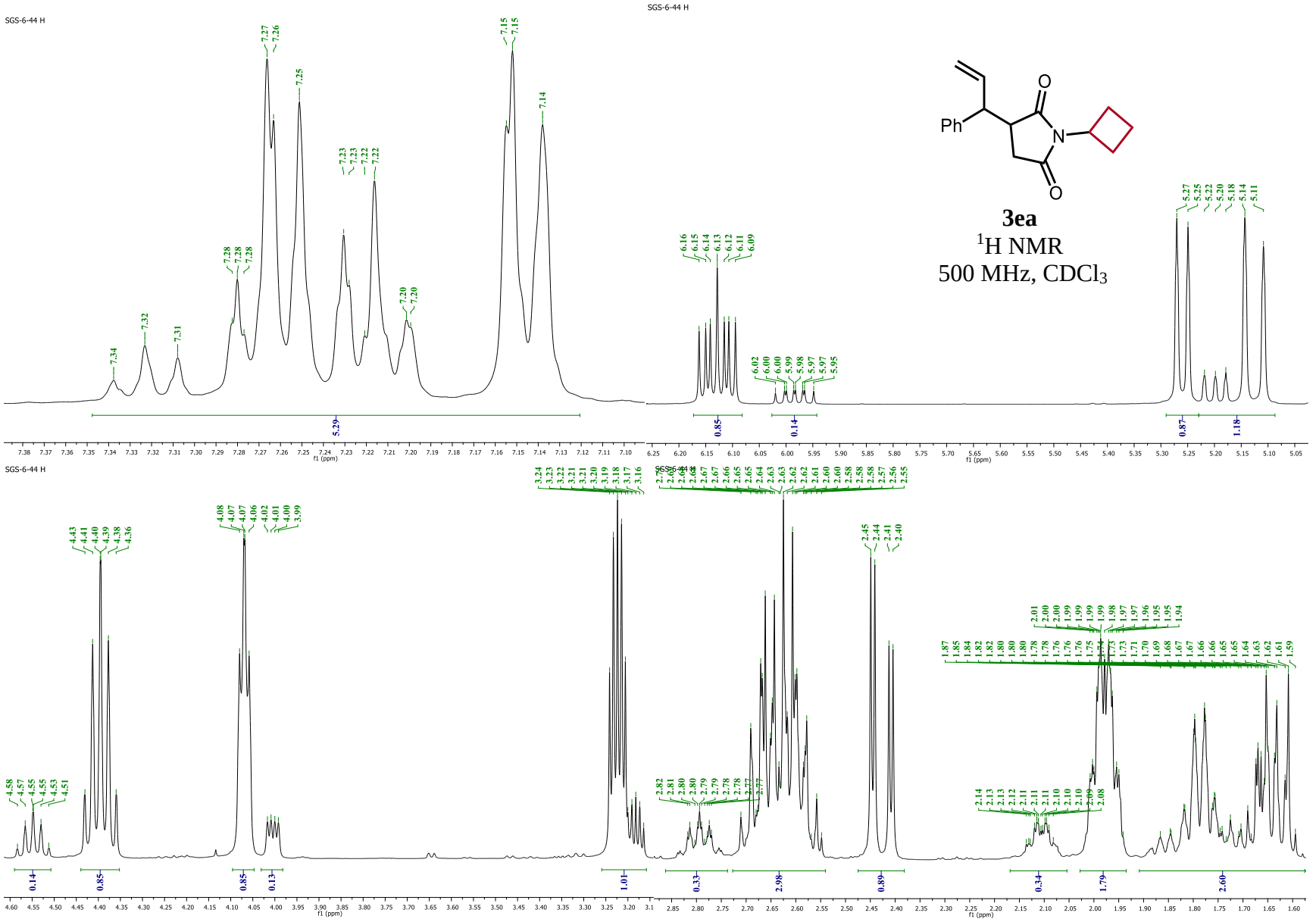

Ph
O
N
O
3da
13C NMR
126 MHz, CDCl3
140.6
138.2
138.0
134.8
128.9
128.7
127.8
127.6
127.2
119.5
116.4
178.9
178.7
176.6
176.4
51.9
51.7
49.3
48.7
44.8
43.4
31.2
31.1
29.0
28.8
28.7
28.6
26.0
25.9
25.2
25.1
f1 (ppm)



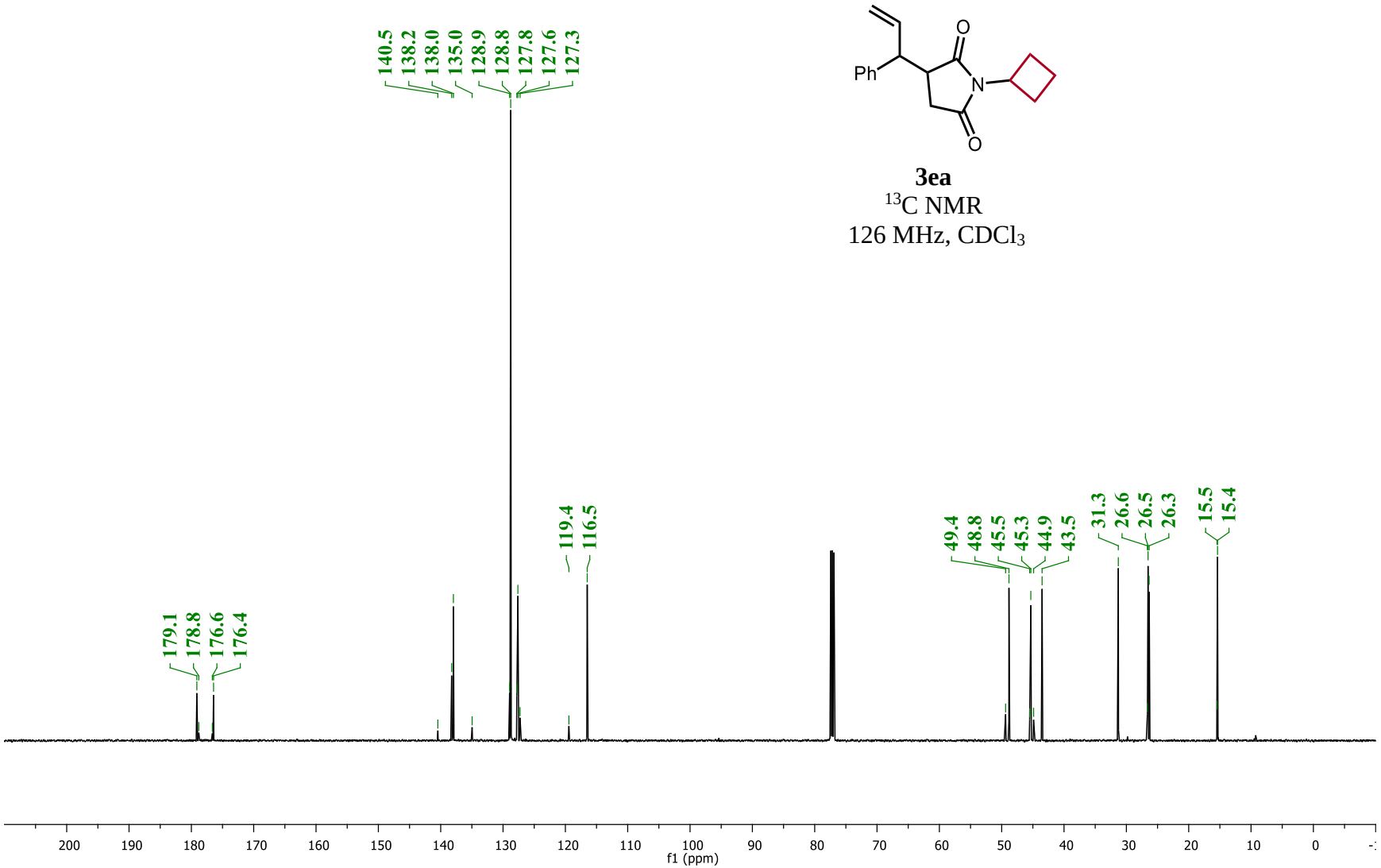
3ea

^{1}H NMR

500 MHz, $CDCl_3$


SGS-6-44 H
Ph
O
N
O
O
3ea
1H NMR
500 MHz, CDCl3
f1 (ppm)

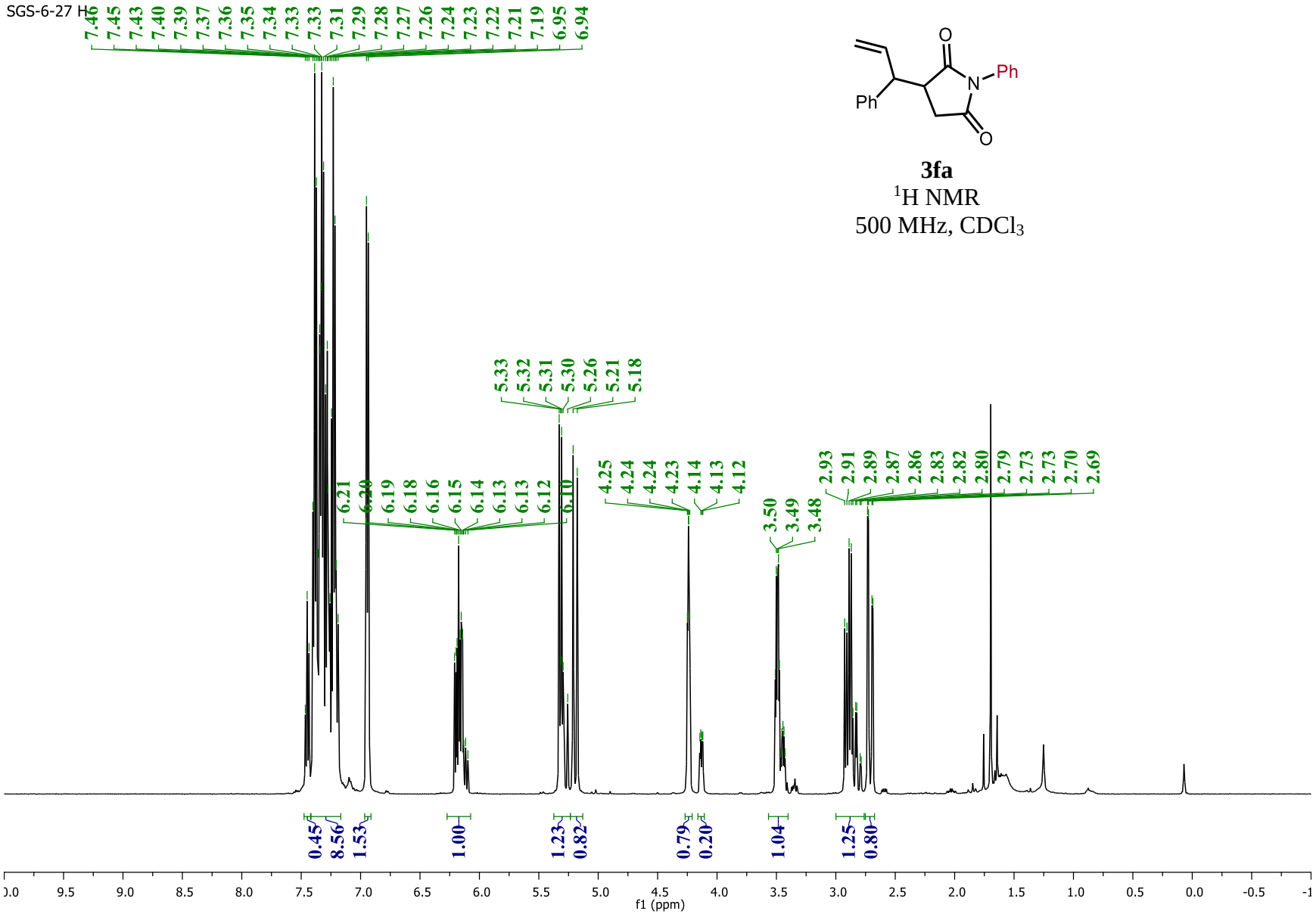
SGS-6-44 C



3ea

^{13}C NMR

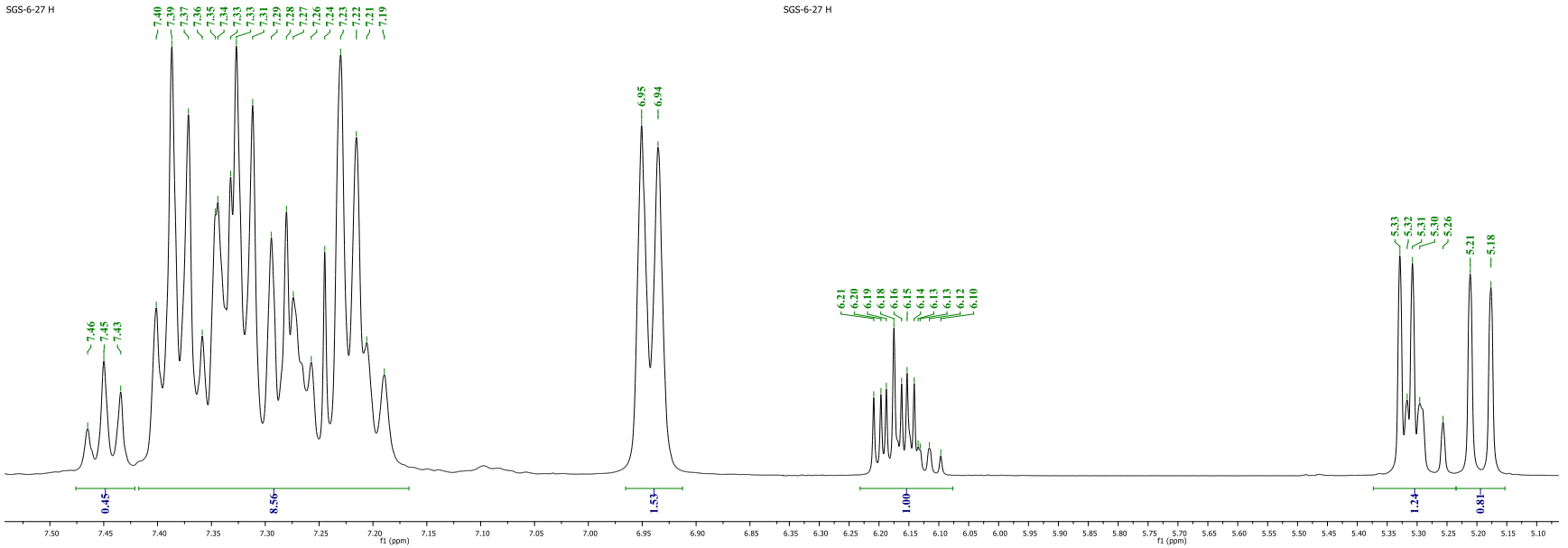
126 MHz, $CDCl_3$

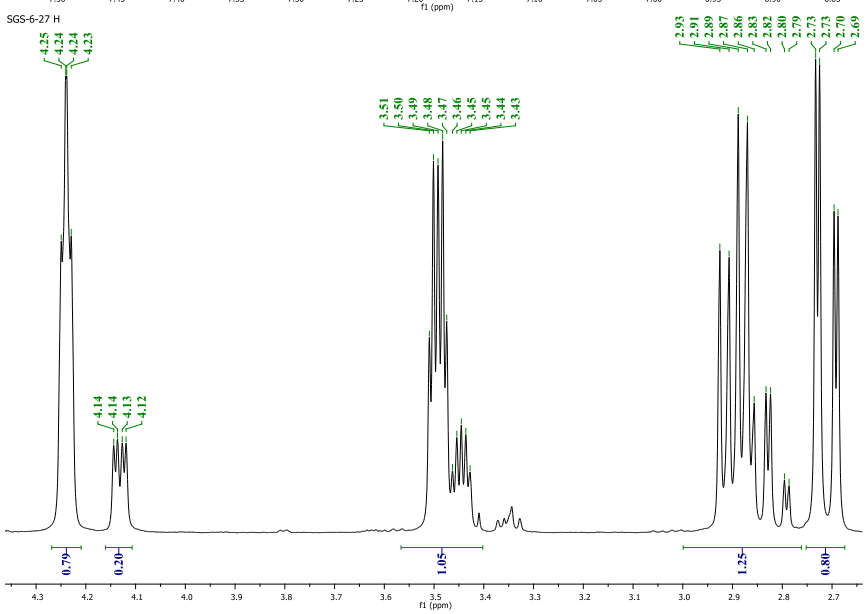


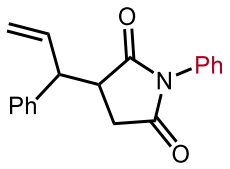
3fa

^{1}H NMR

500 MHz, $CDCl_3$





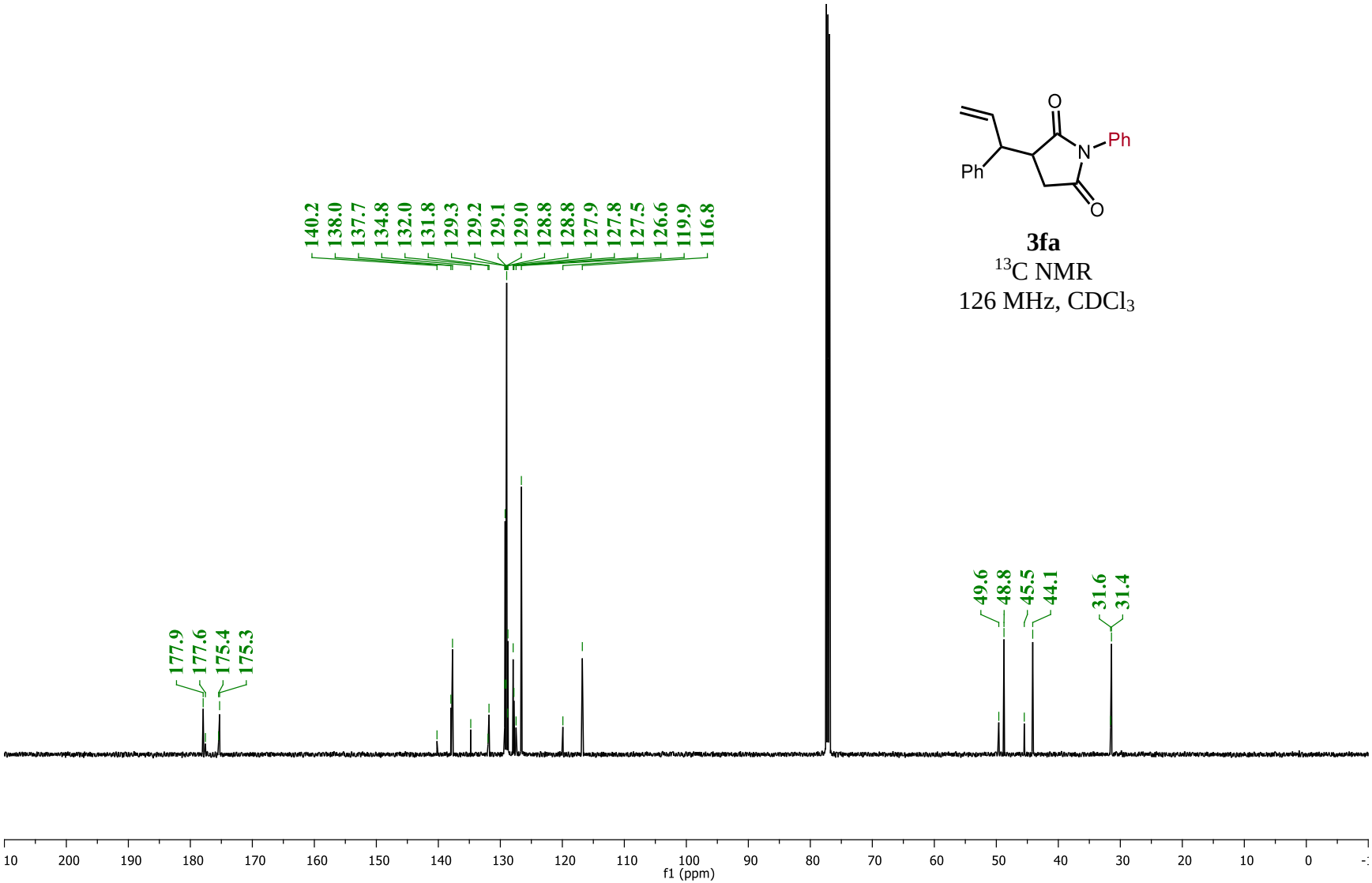


3fa

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-27 C

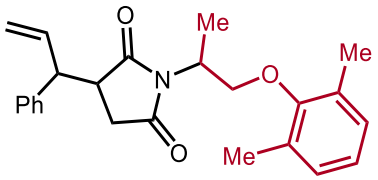


3fa

^{13}C NMR

126 MHz, $CDCl_3$

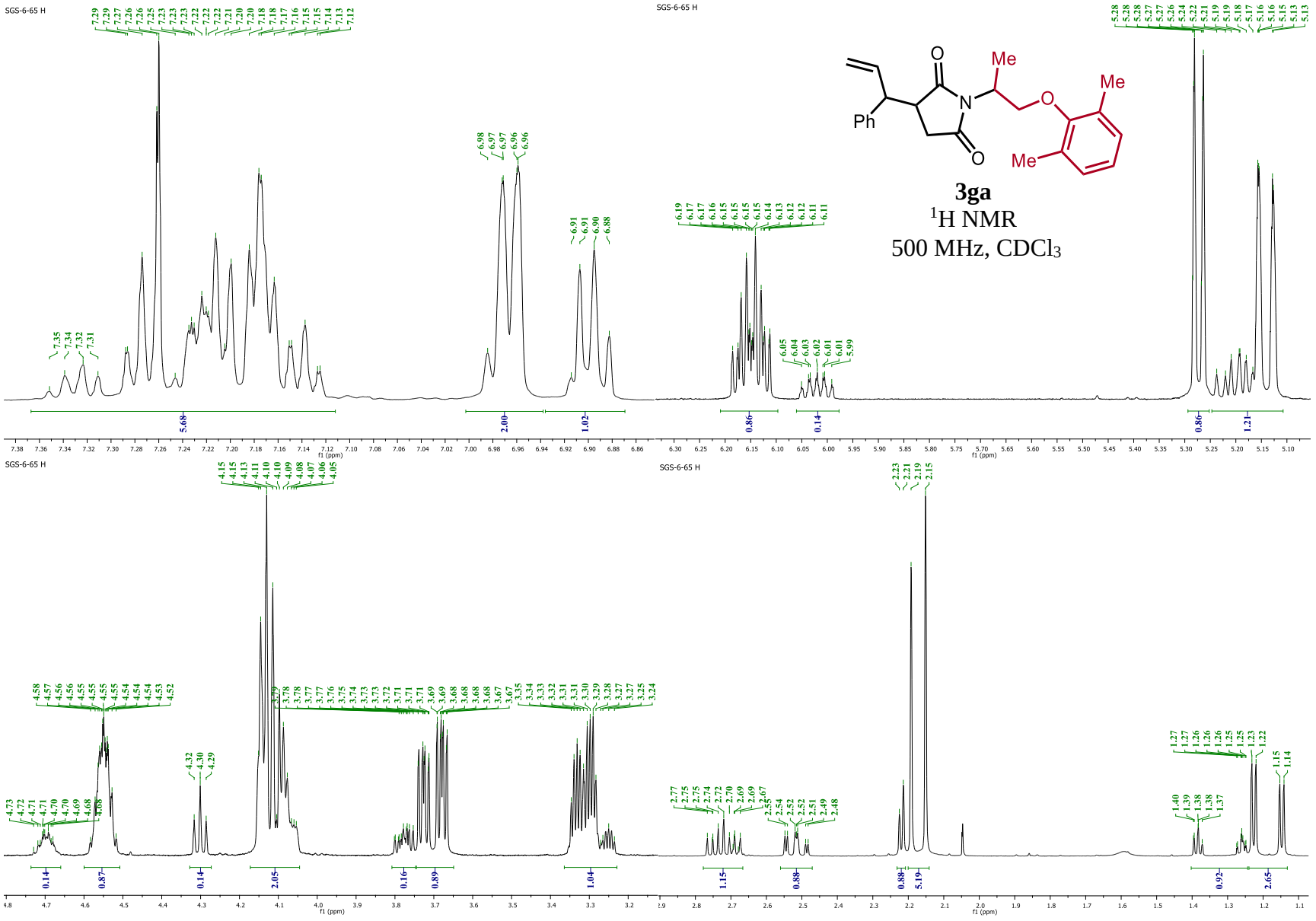
SGS-6-65 H



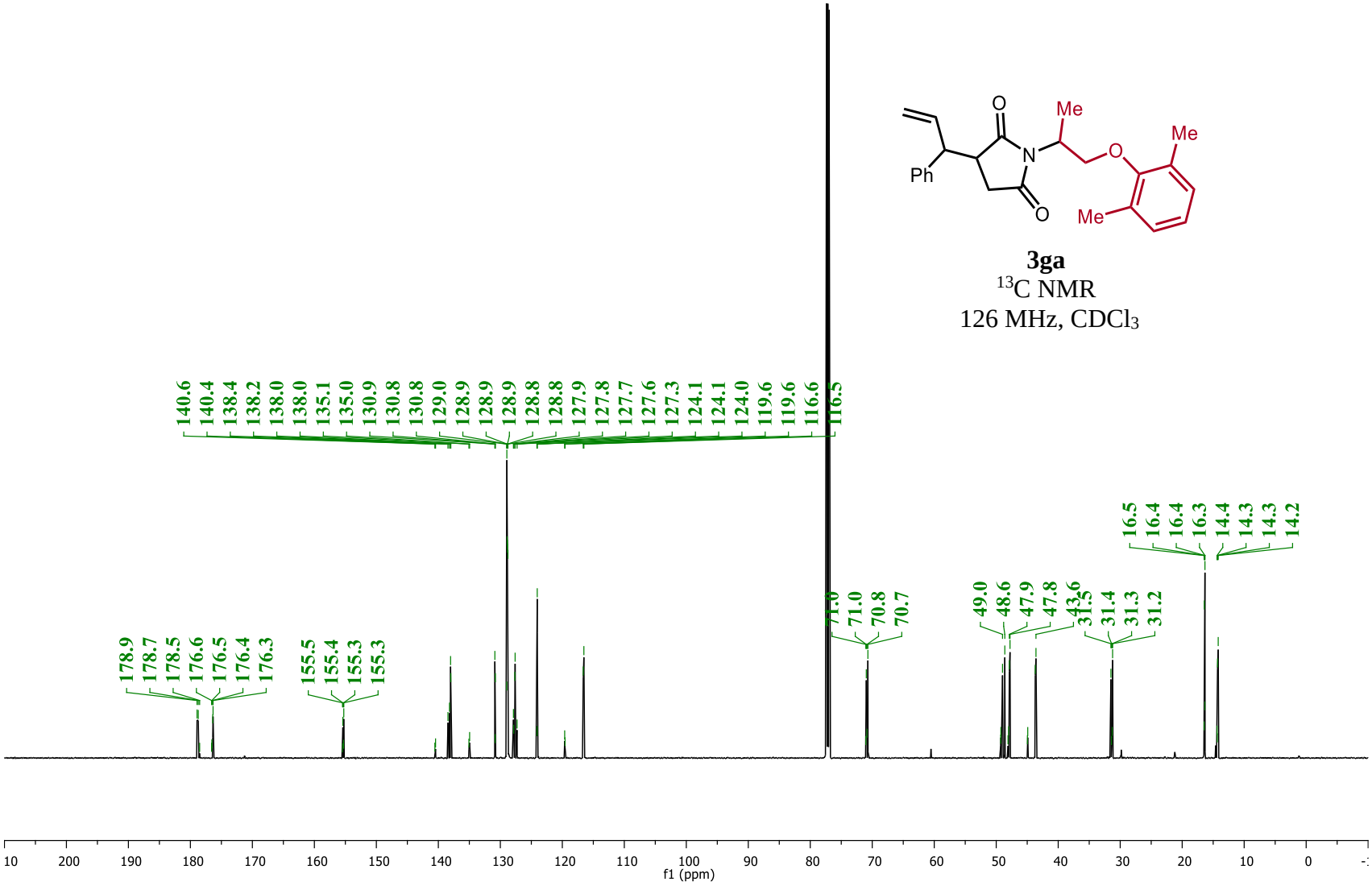
3ga

^{1}H NMR

500 MHz, $CDCl_3$


SGS-6-65 H
SGS-6-65 H
SGS-6-65 H
SGS-6-65 H
Ph
N
O
O
O
Me
Me
Me
3ga
1H NMR
500 MHz, CDCl3
f1 (ppm)
f1 (ppm)

SGS-6-65 C

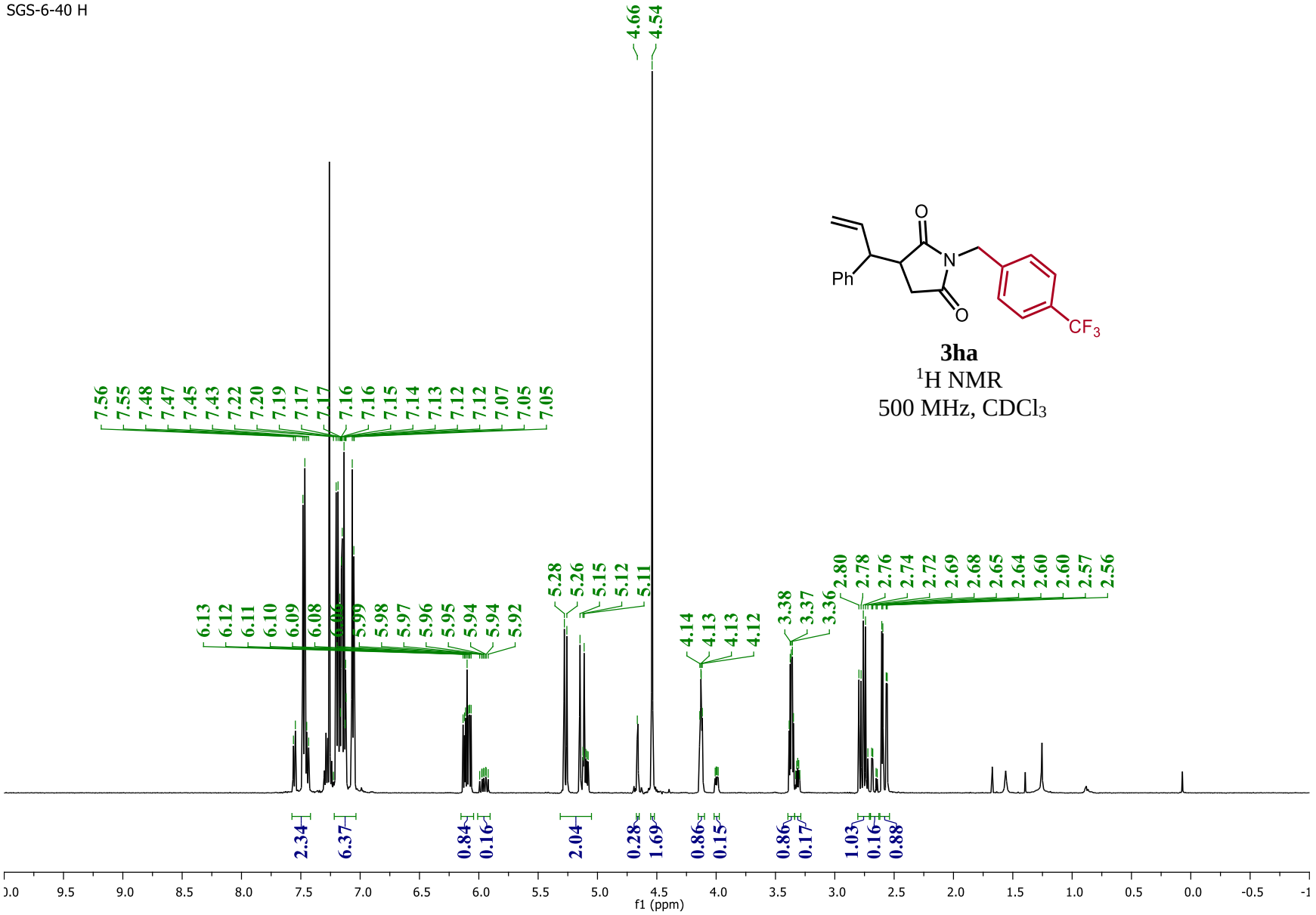


3ga

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-40 H

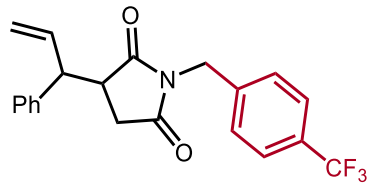


3ha

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-40 C

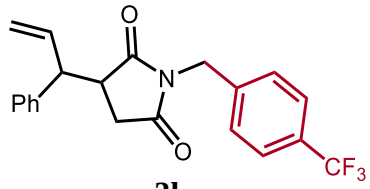


3ha

^{13}C NMR

151 MHz, $CDCl_3$

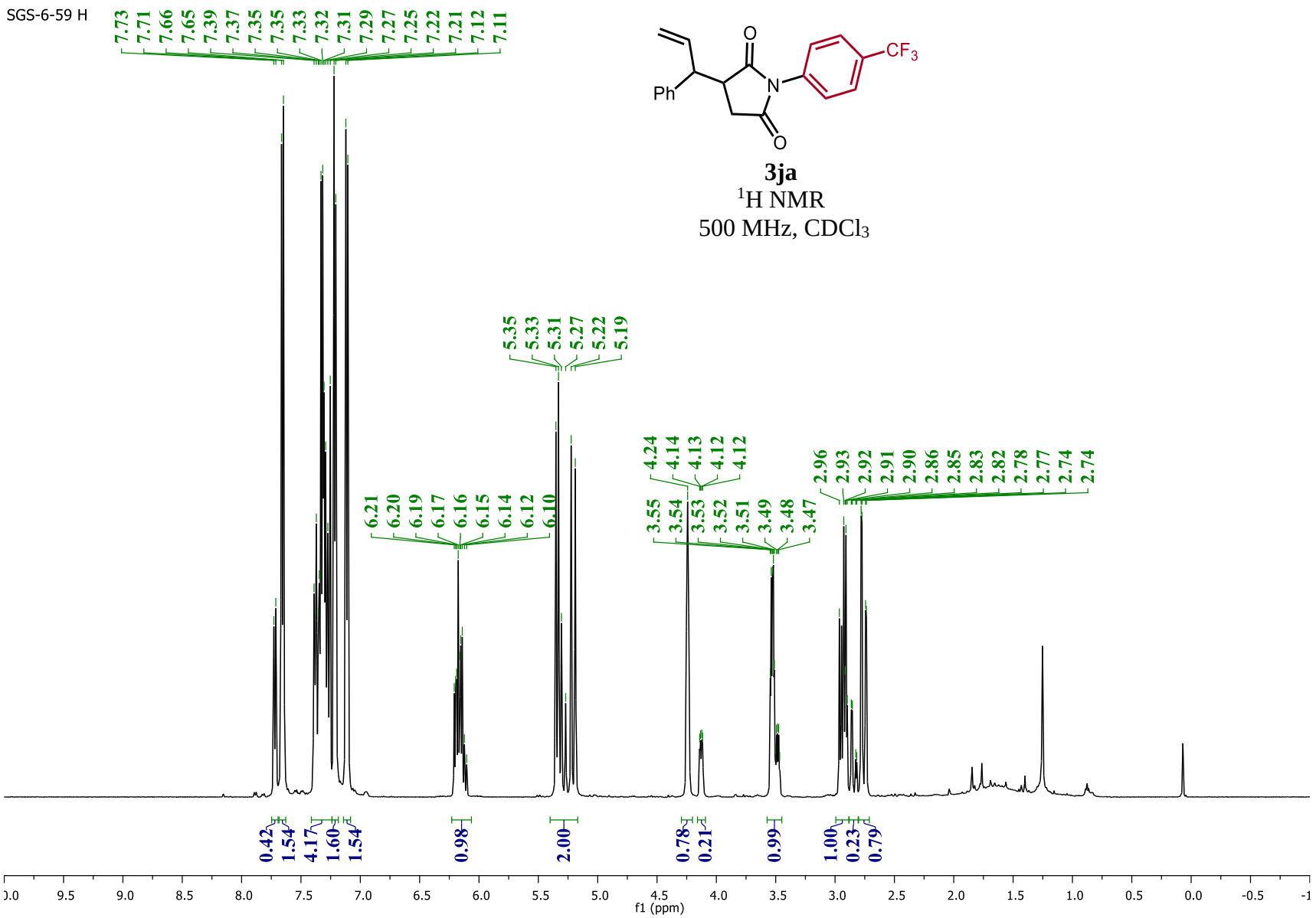
SGS-6-40 F



3ha

^{19}F NMR

471 MHz, $CDCl_3$

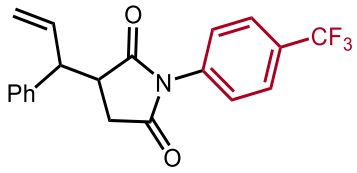


3ja

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-59 C

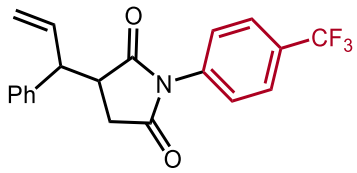


3ja

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-59 F

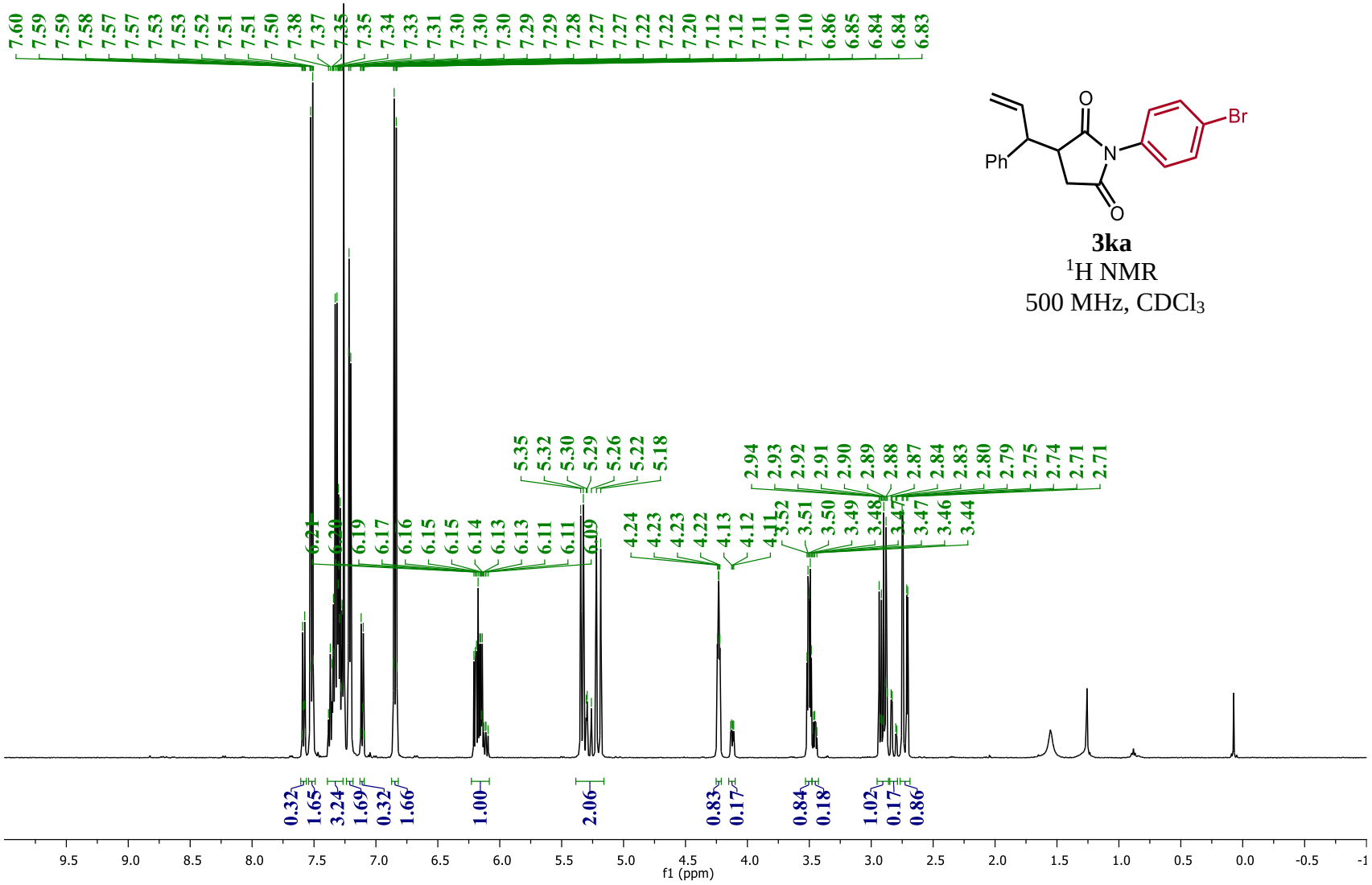


3ja

^{19}F NMR

471 MHz, $CDCl_3$

SGS-6-22 H

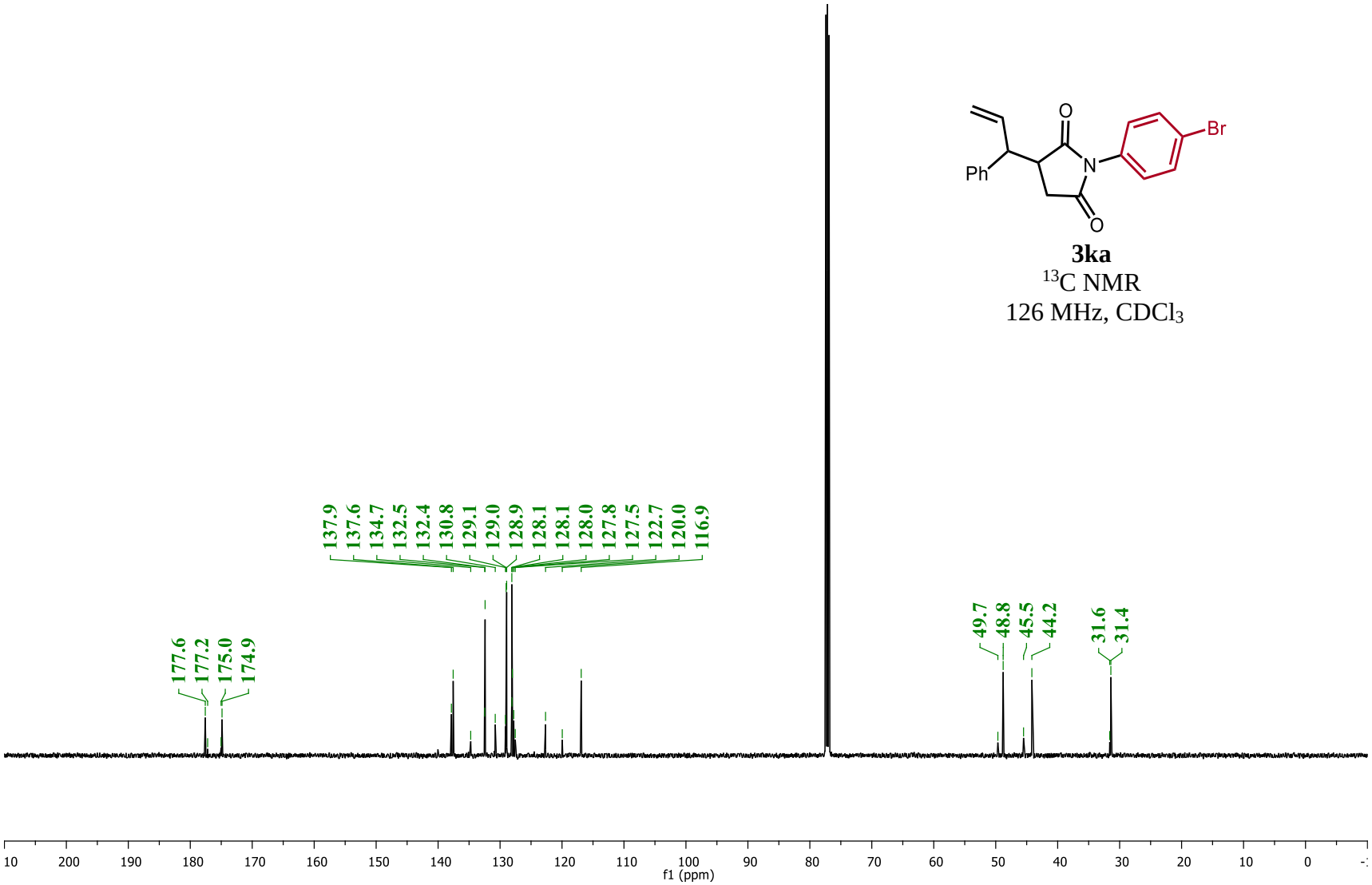


3ka

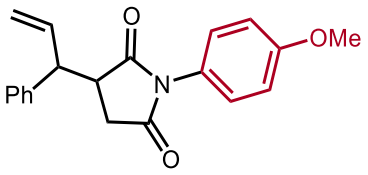
^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-22 C


O
Br
N
Ph
O
3ka
13C NMR
126 MHz, CDCl3
137.9
137.6
134.7
132.5
132.4
130.8
129.1
129.0
128.9
128.1
128.1
128.0
127.8
127.5
122.7
120.0
116.9
177.6
177.2
175.0
174.9
49.7
48.8
45.5
44.2
31.6
31.4
f1 (ppm)

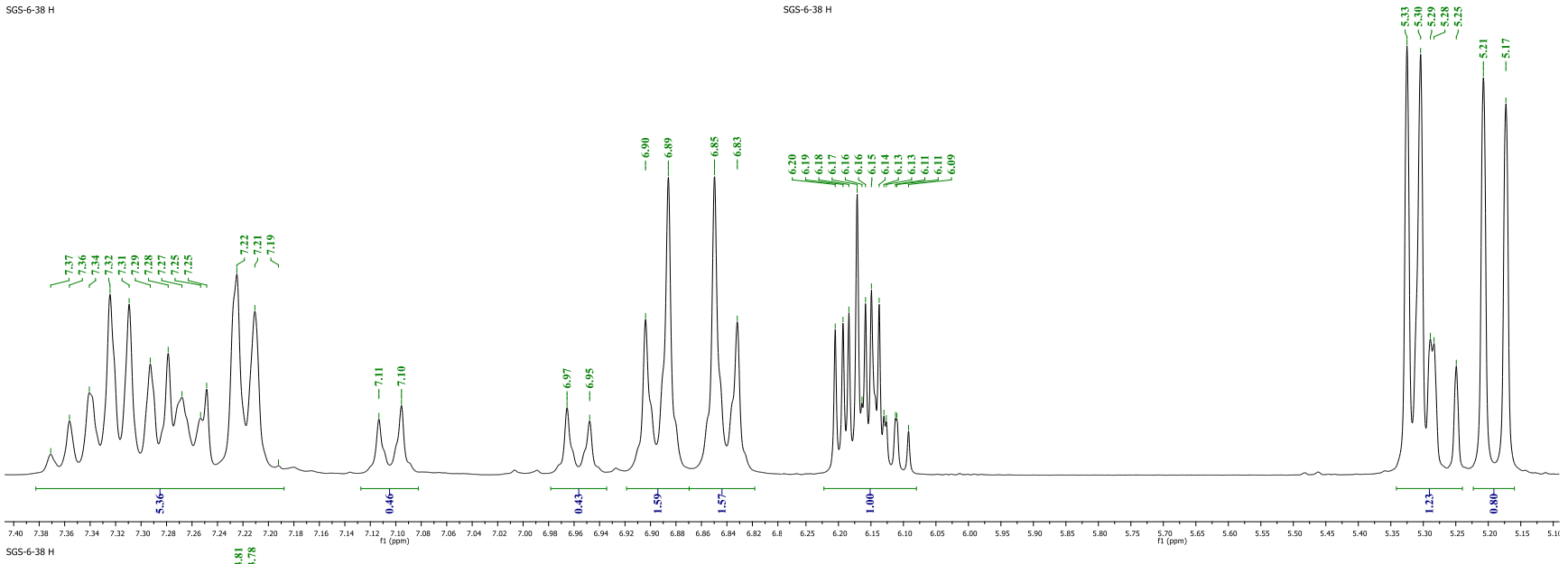
SGS-6-38 H

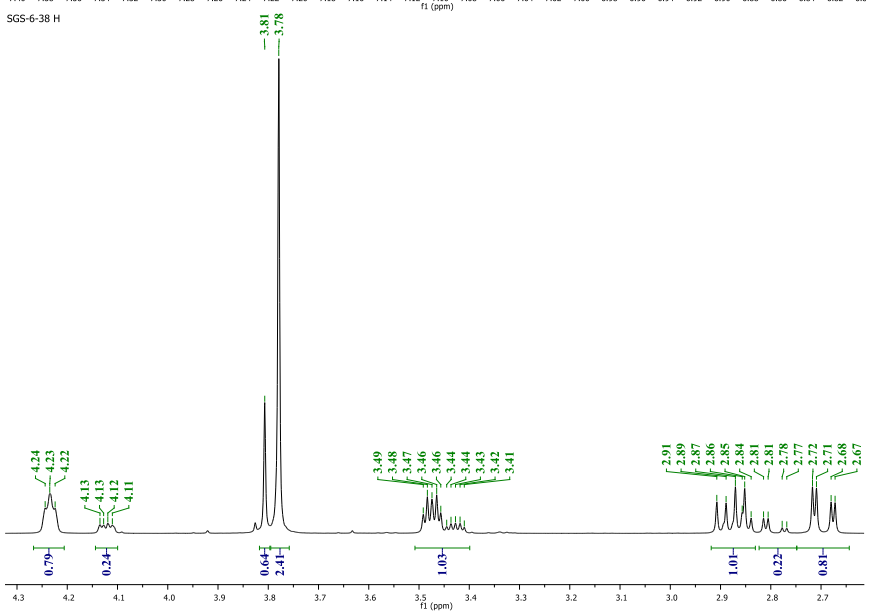


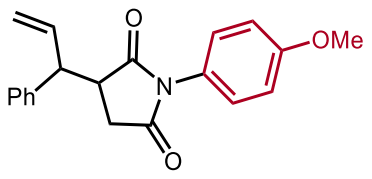
3la

^{1}H NMR

500 MHz, $CDCl_3$





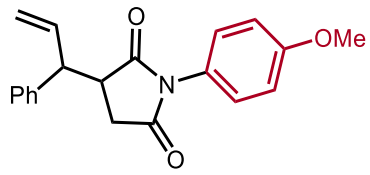


3la

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-38 C

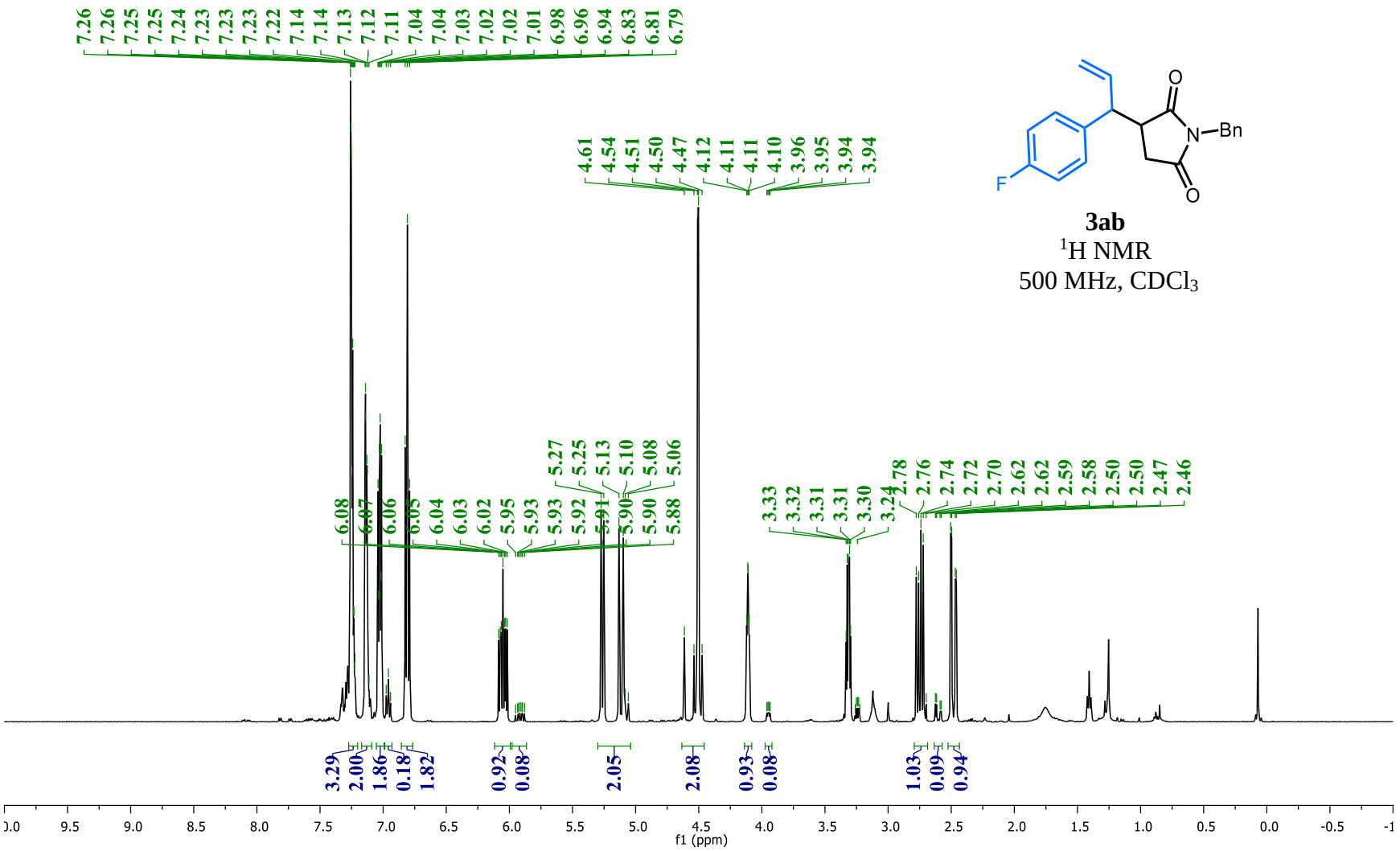


3la

^{13}C NMR

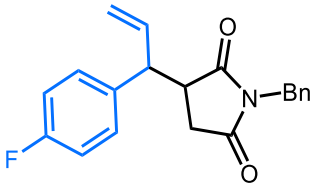
126 MHz, $CDCl_3$

SGS-6-56 H



3ab
^{1}H NMR
500 MHz, $CDCl_3$

SGS-6-56 C

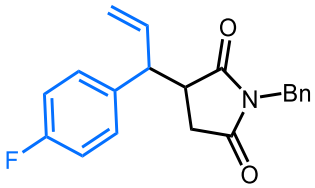


3ab

^{13}C NMR

126 MHz, $CDCl_3$

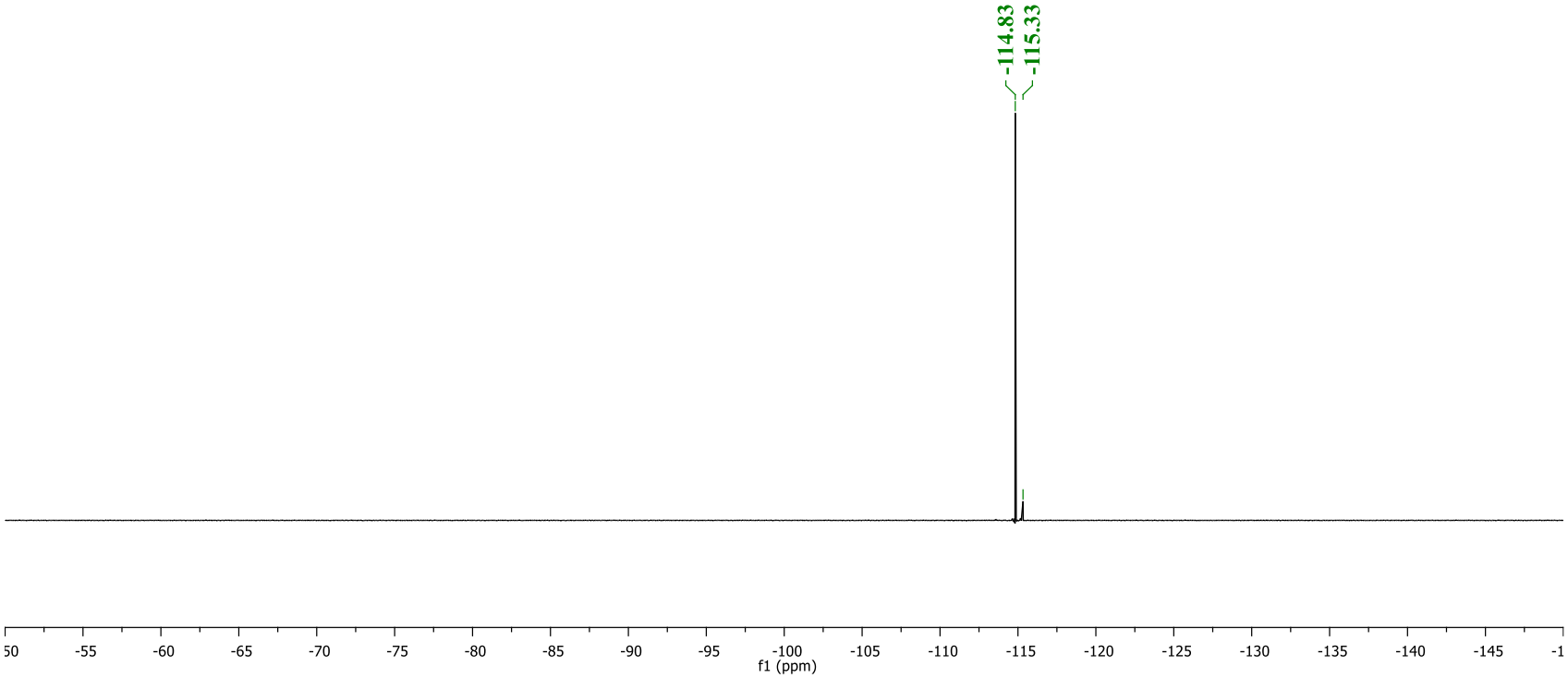
SGS-6-56 F



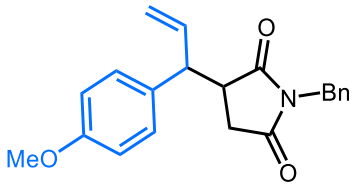
3ab

^{19}F NMR

471 MHz, $CDCl_3$



SGS-6-46 H

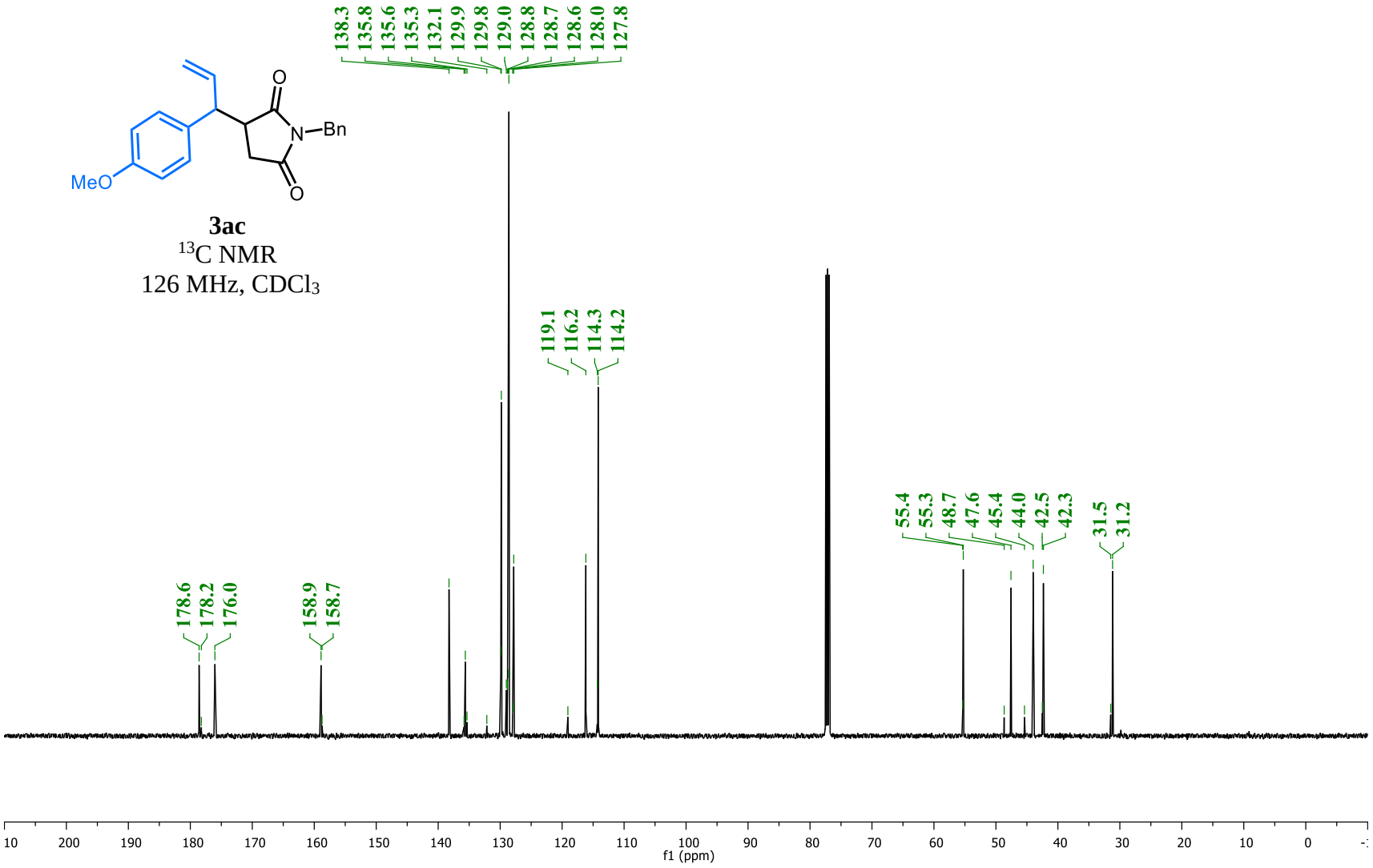


3ac

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-46 C

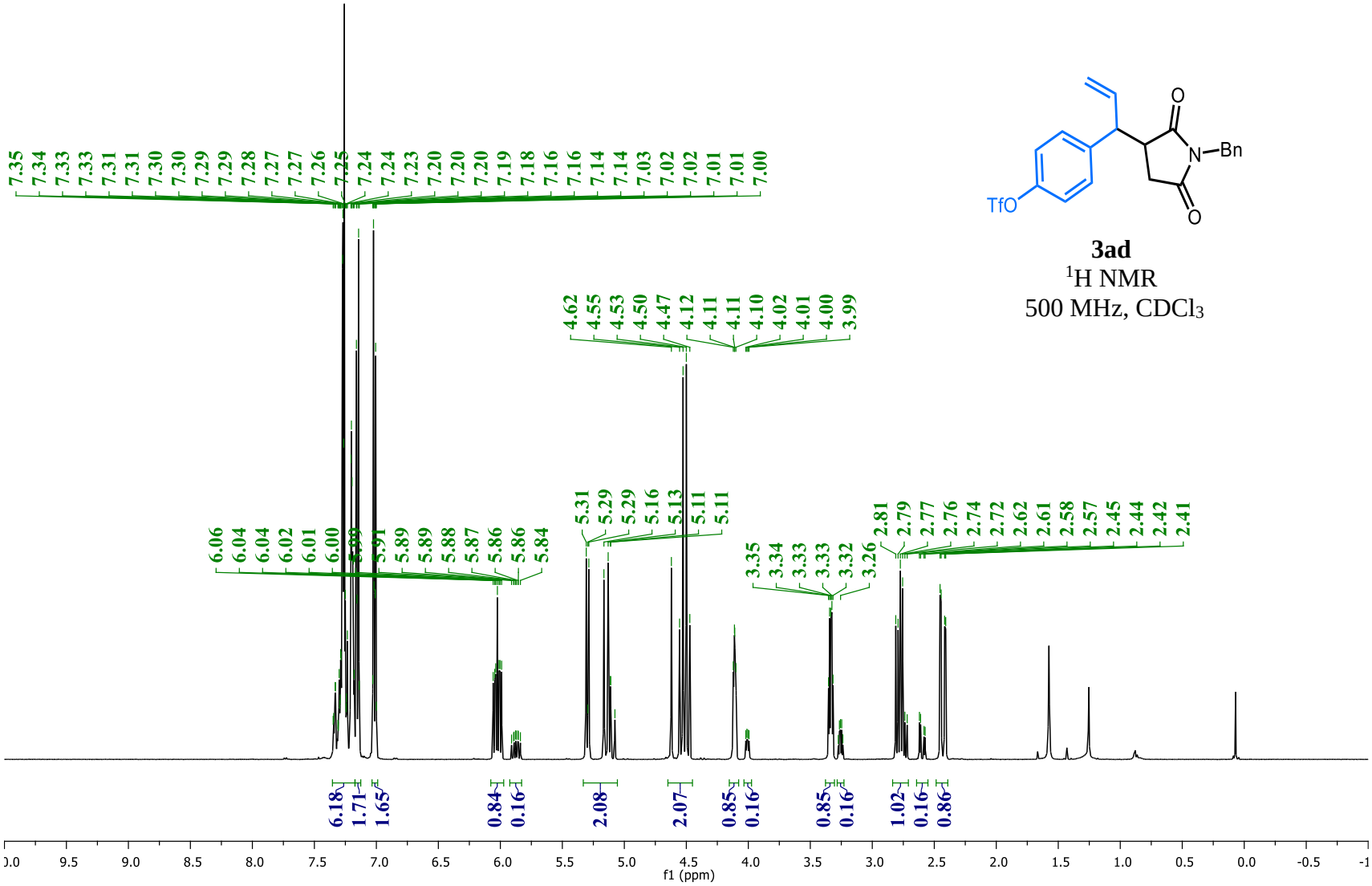


3ac

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-54 H

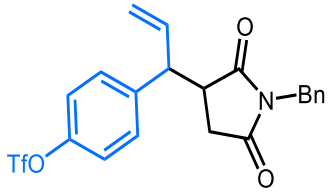


3ad

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-54 C

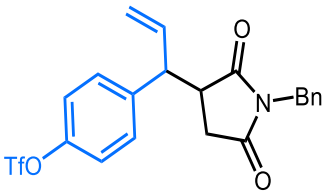


3ad

^{13}C NMR

126 MHz, $CDCl_3$

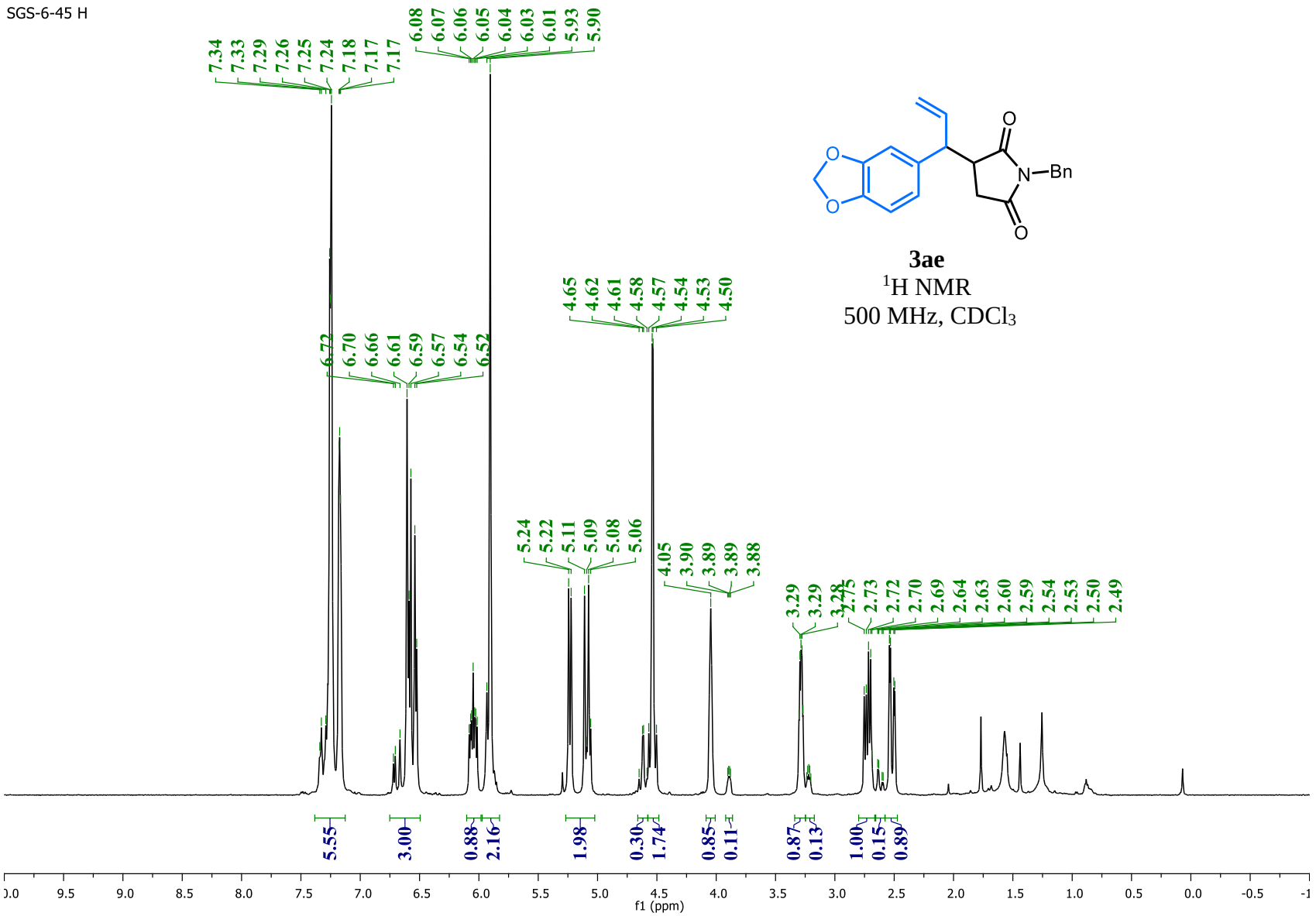
SGS-6-54 F



3ad

^{19}F NMR

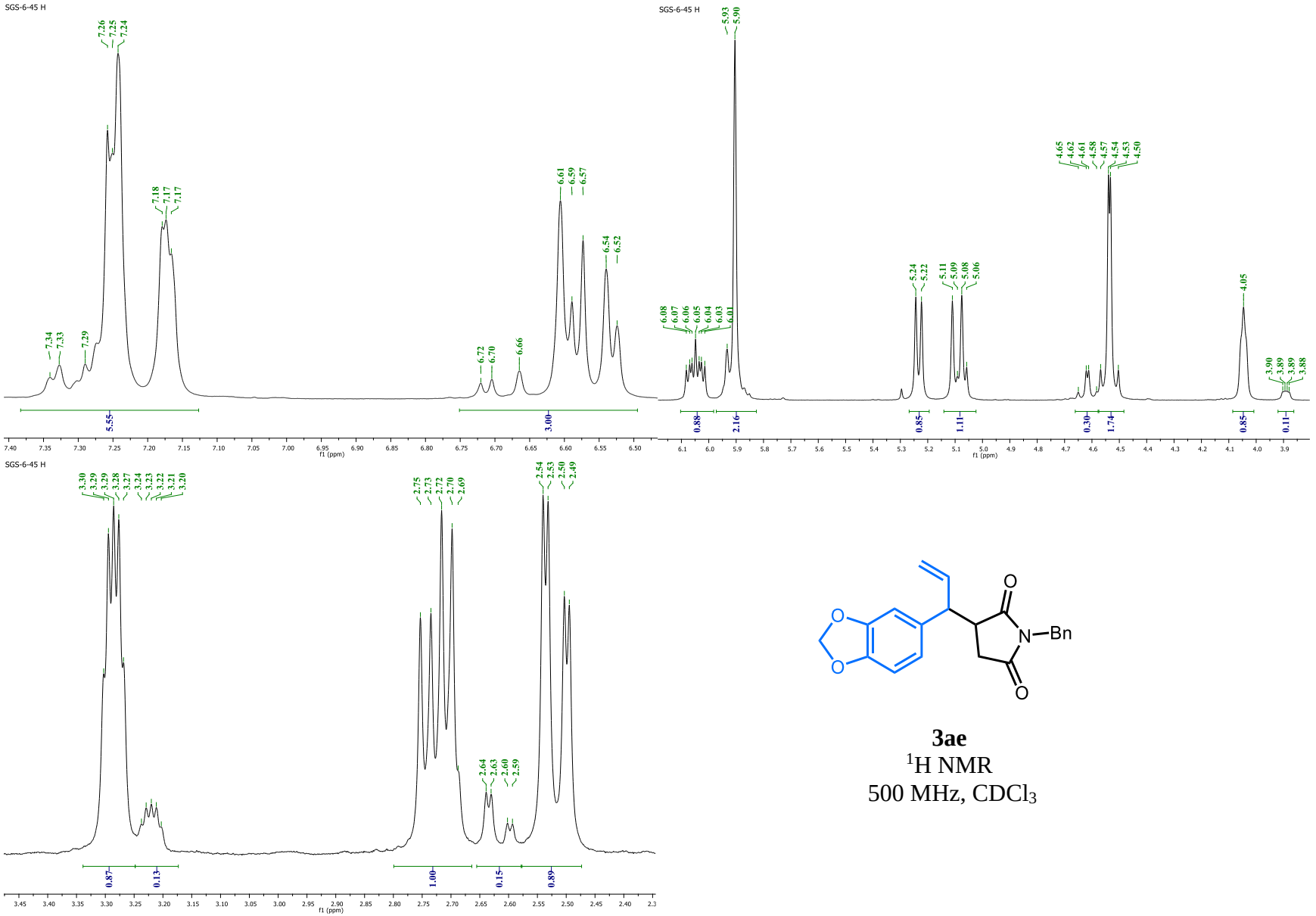
471 MHz, $CDCl_3$



3ae

^{1}H NMR

500 MHz, $CDCl_3$

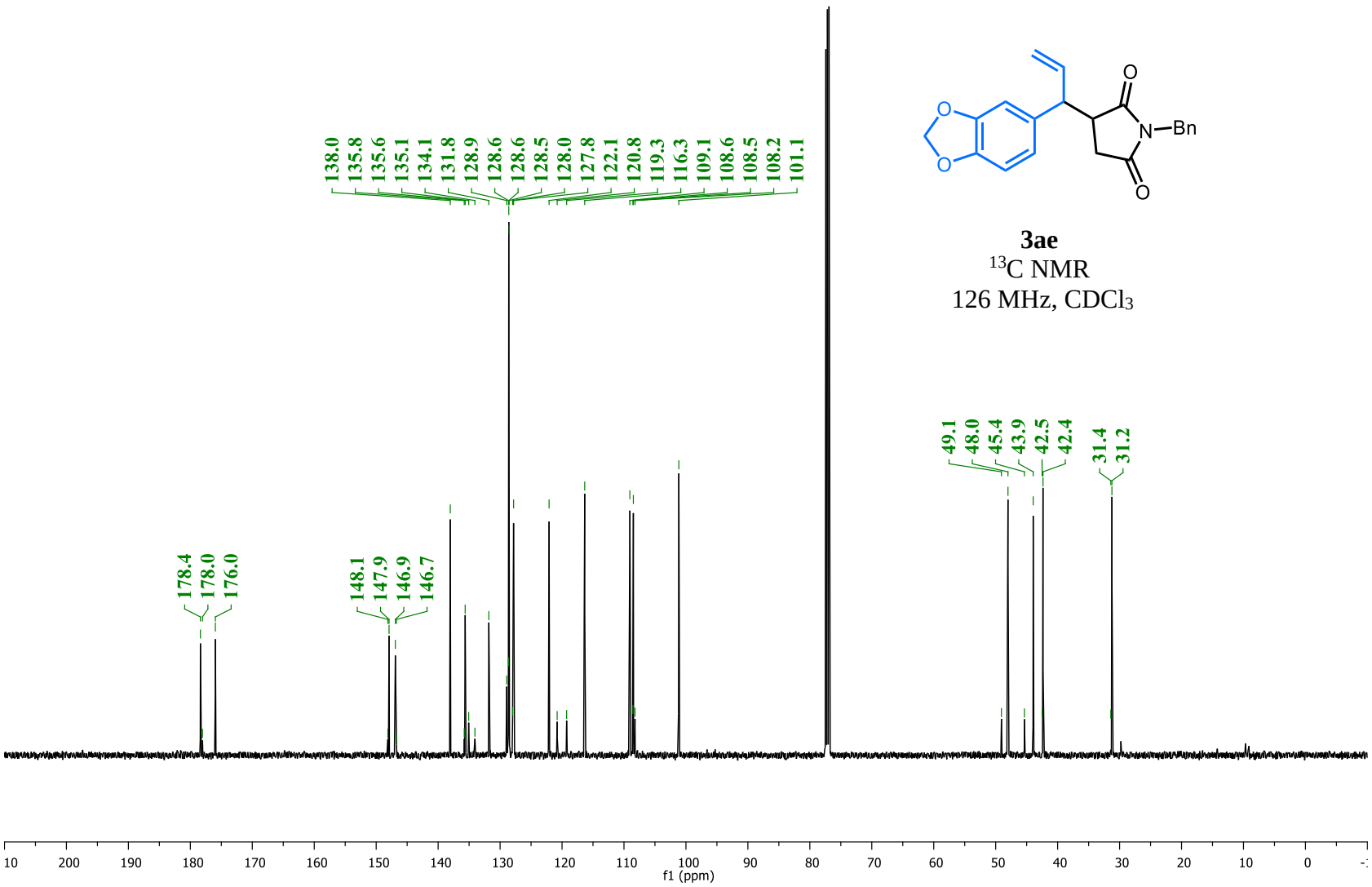


3ae

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-45 C

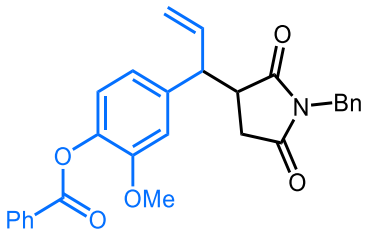


3ae

^{13}C NMR

126 MHz, $CDCl_3$

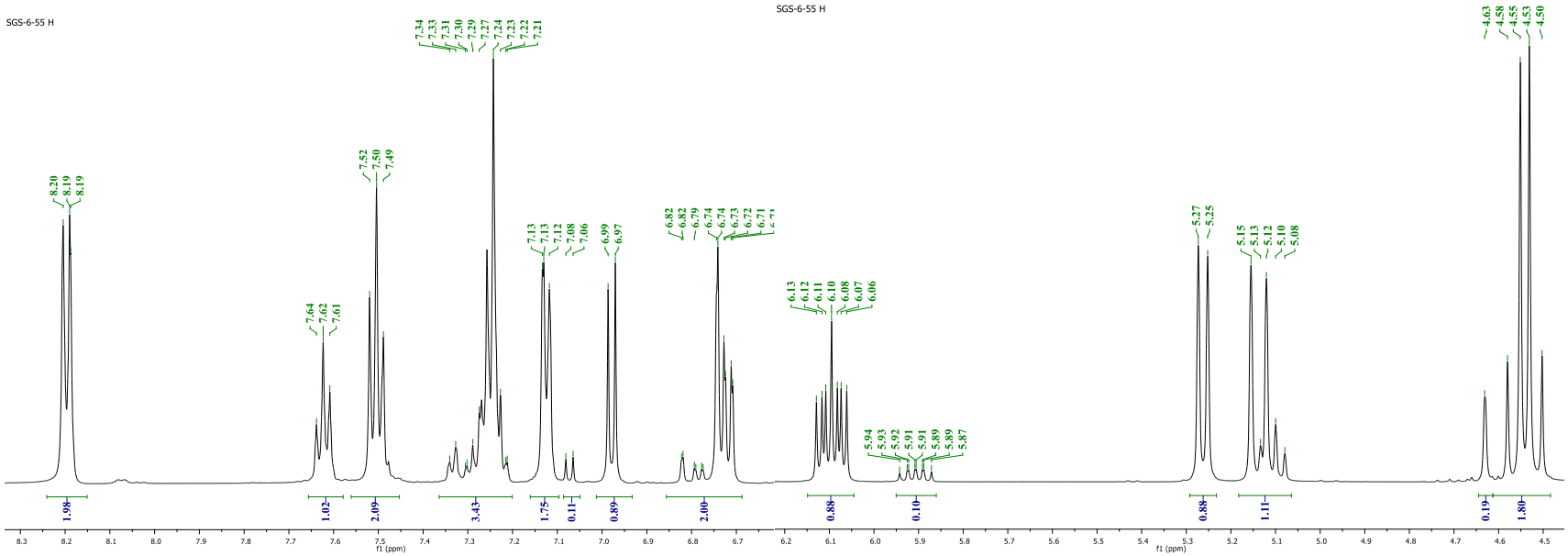
SGS-6-55 H

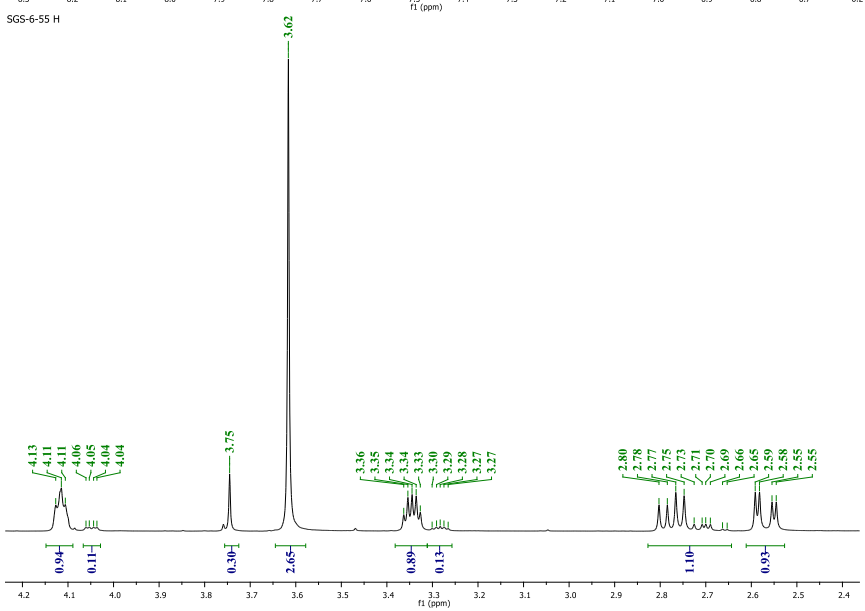


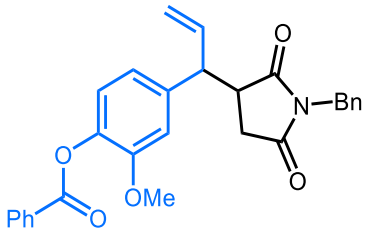
3af

^{1}H NMR

500 MHz, $CDCl_3$





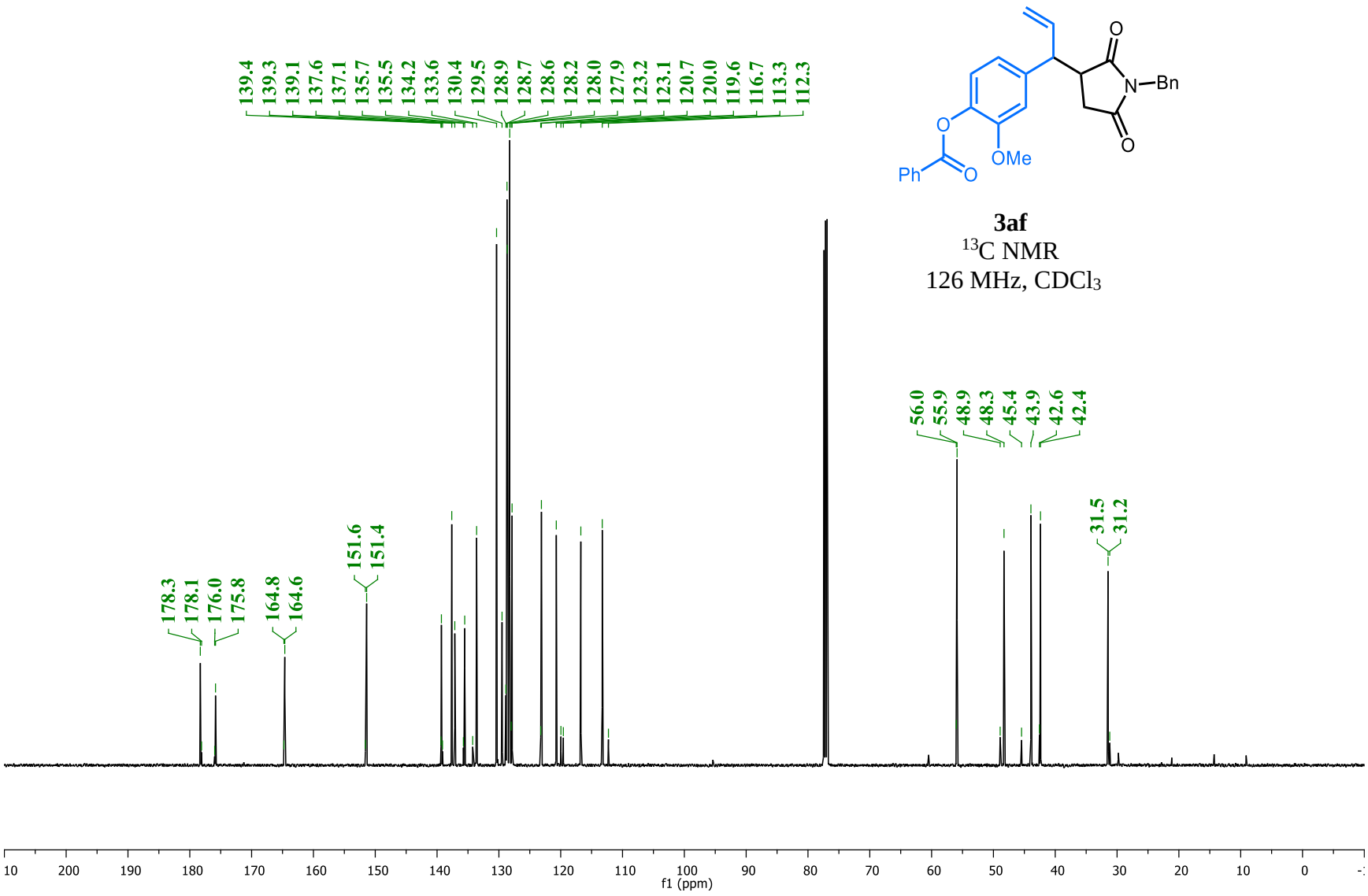


3af

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-55 C

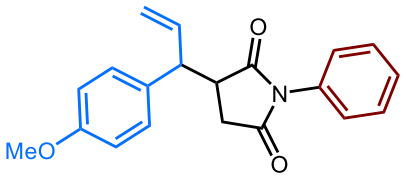


3af

^{13}C NMR

126 MHz, $CDCl_3$

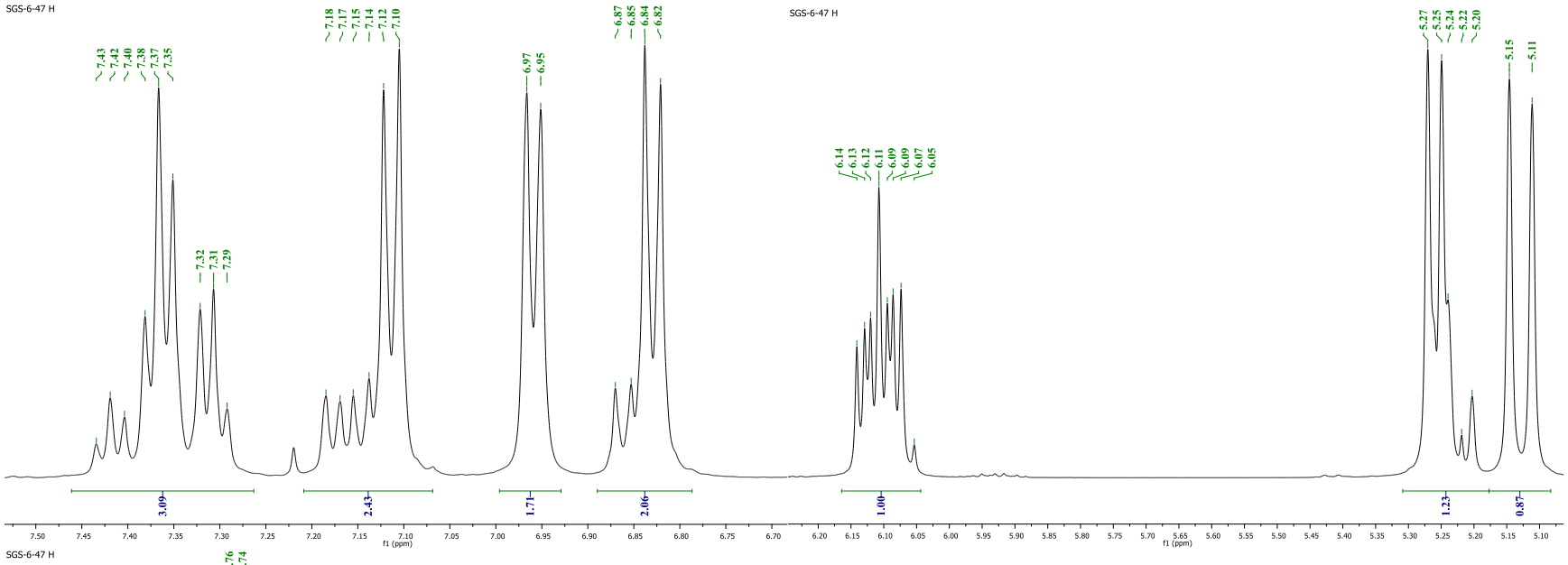
SGS-6-47 H

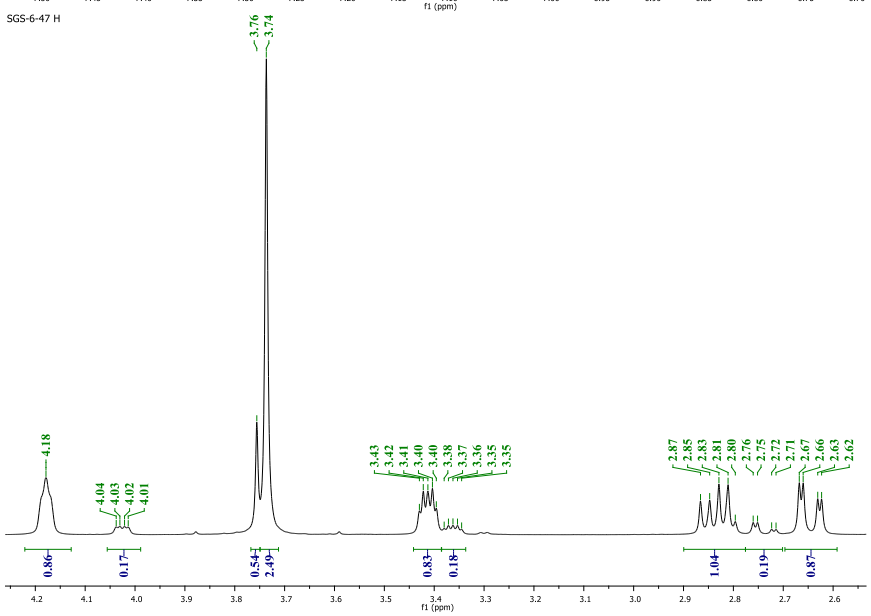


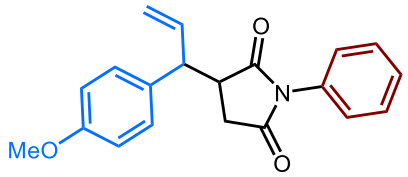
3fc

^{1}H NMR

500 MHz, $CDCl_3$





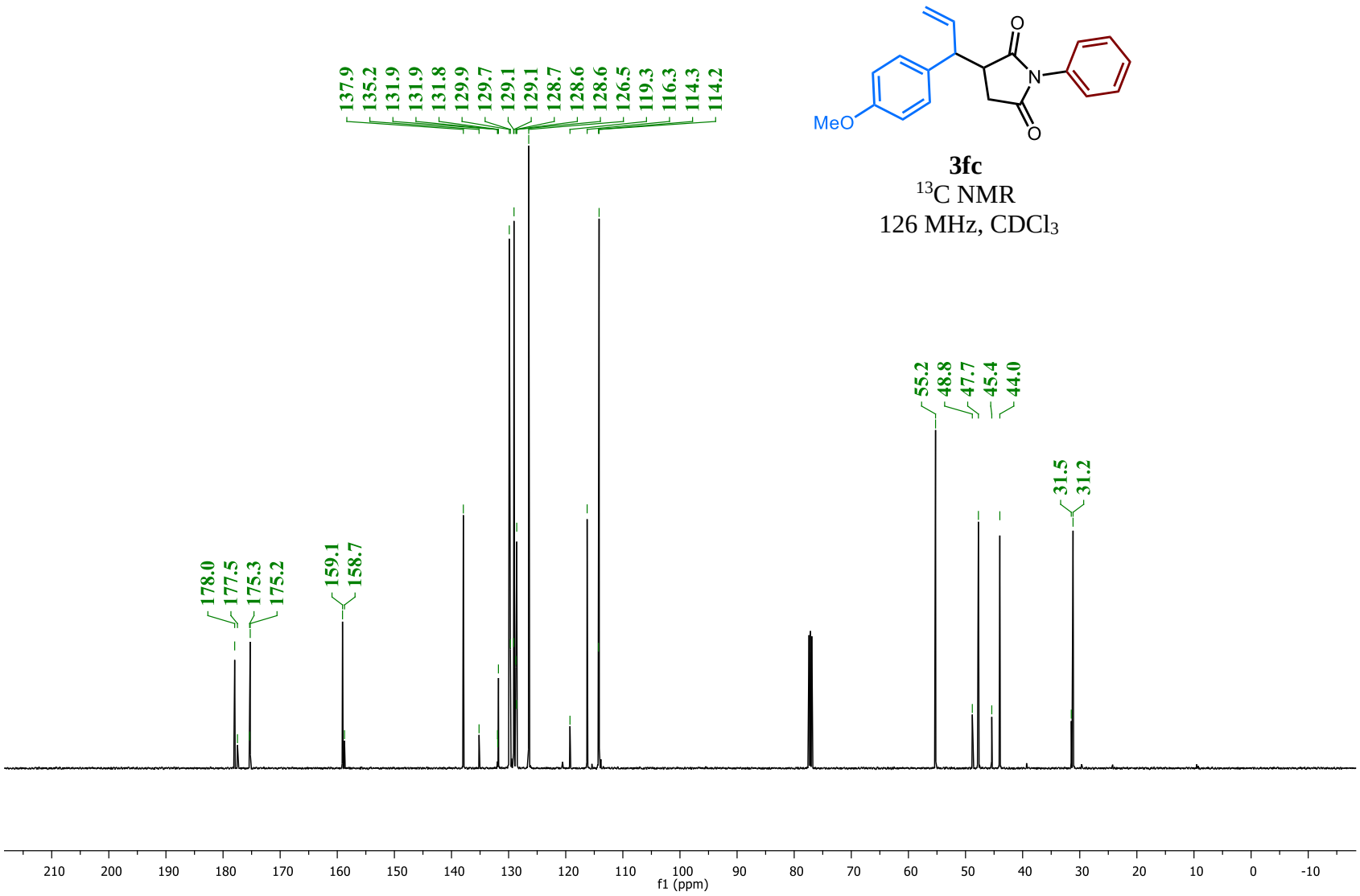


3fc

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-47 C

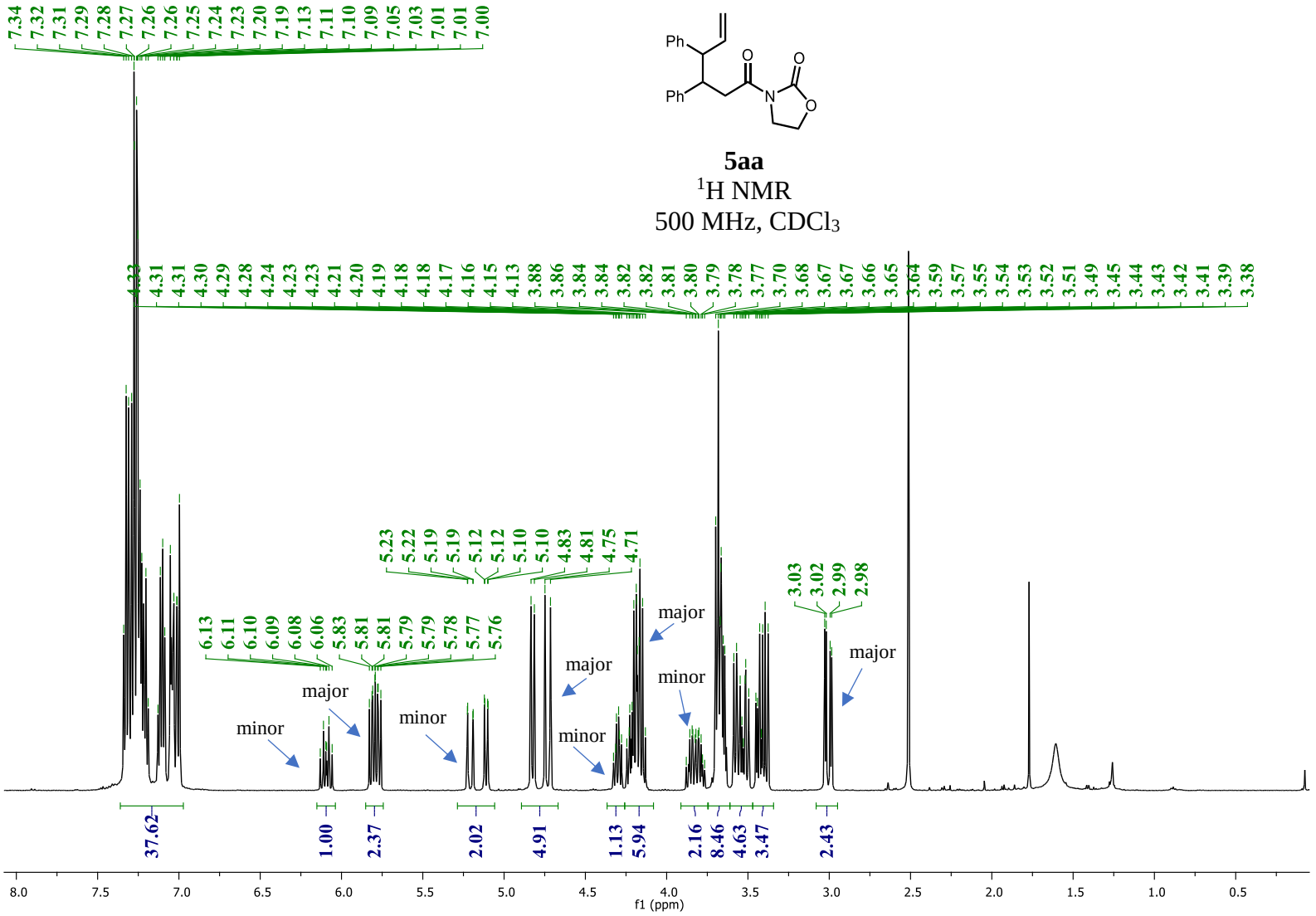


3fc

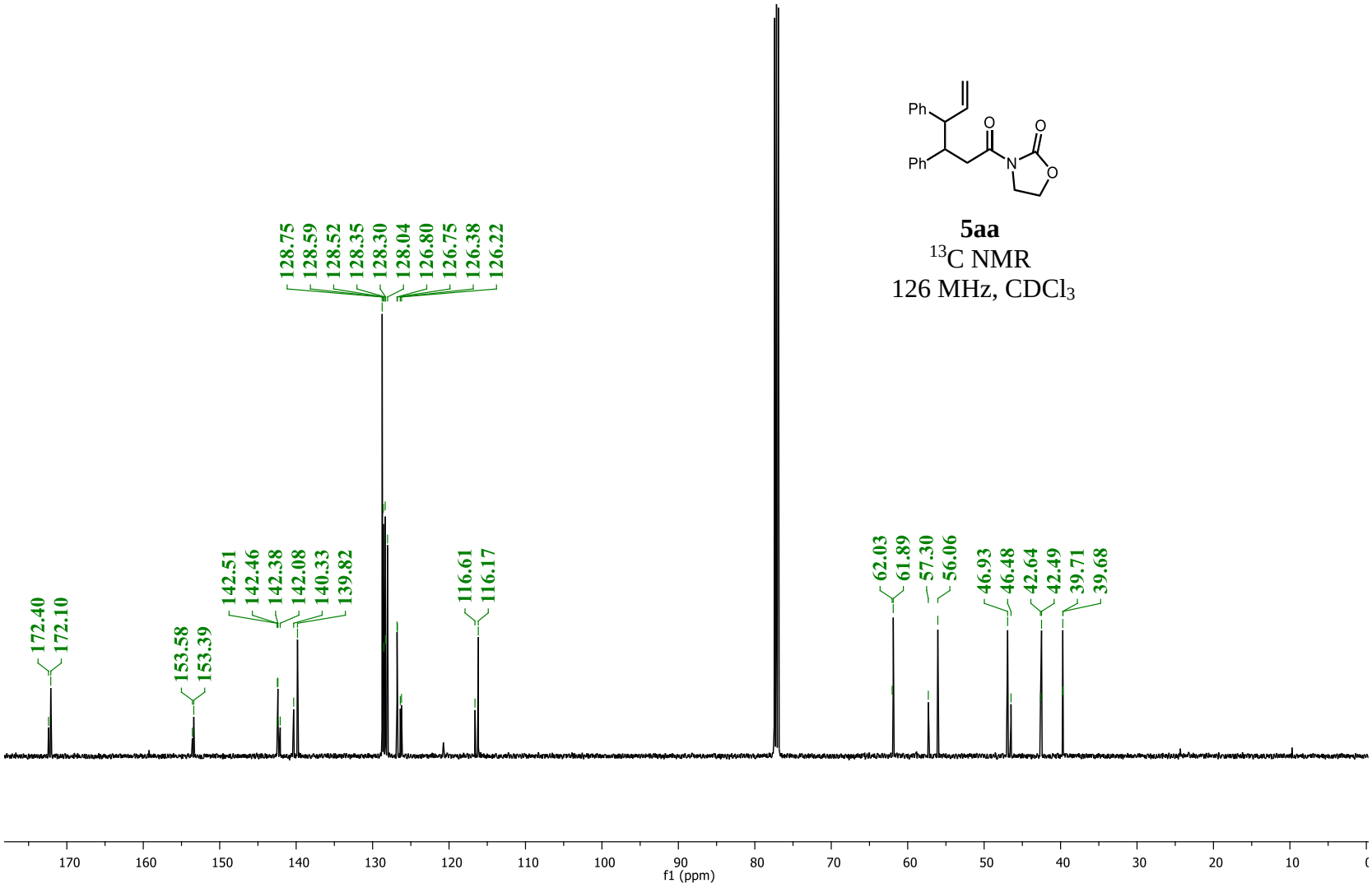
^{13}C NMR

126 MHz, $CDCl_3$

7.2 NMR spectra: Oxazolidinone Scope



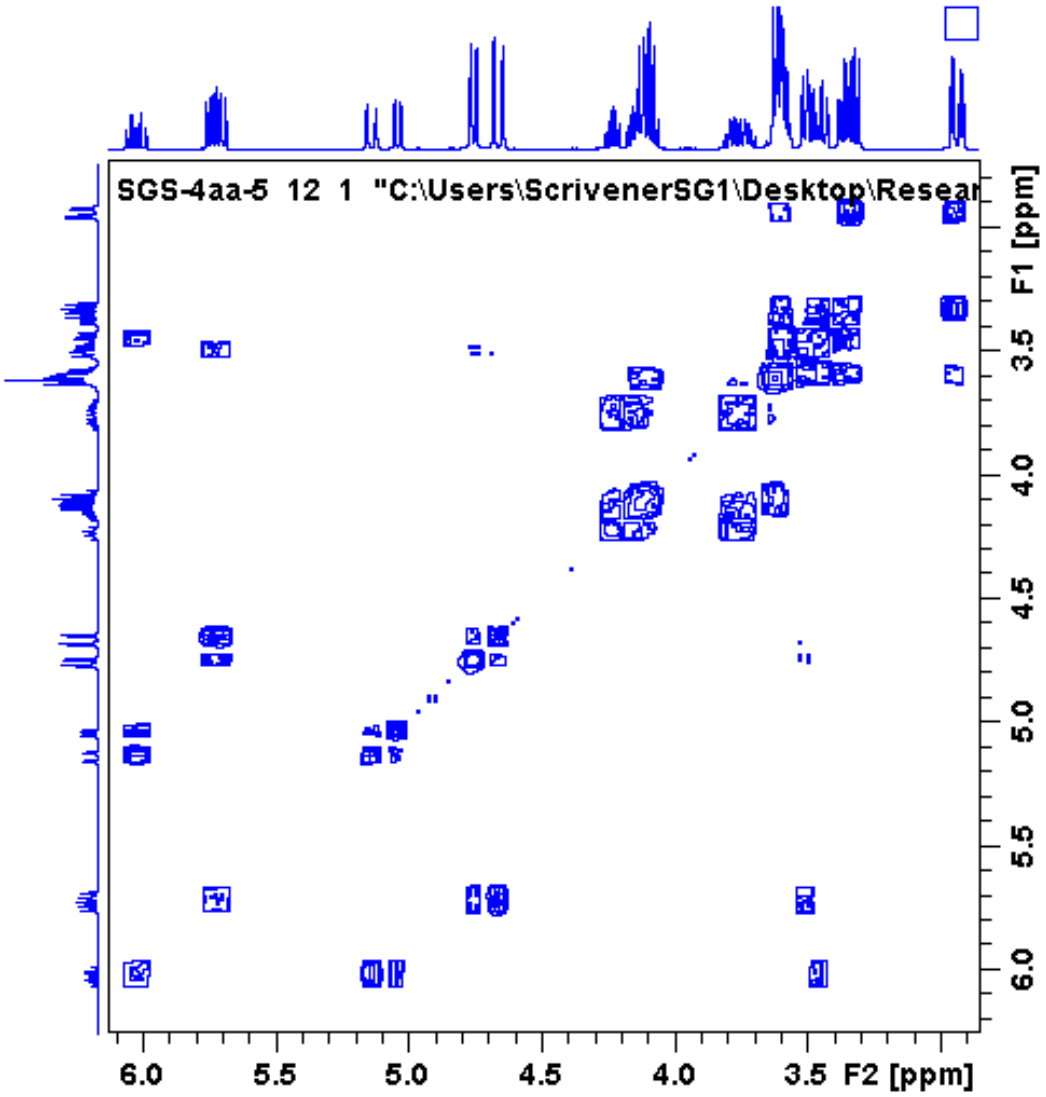
C

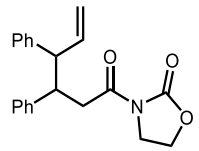


5aa

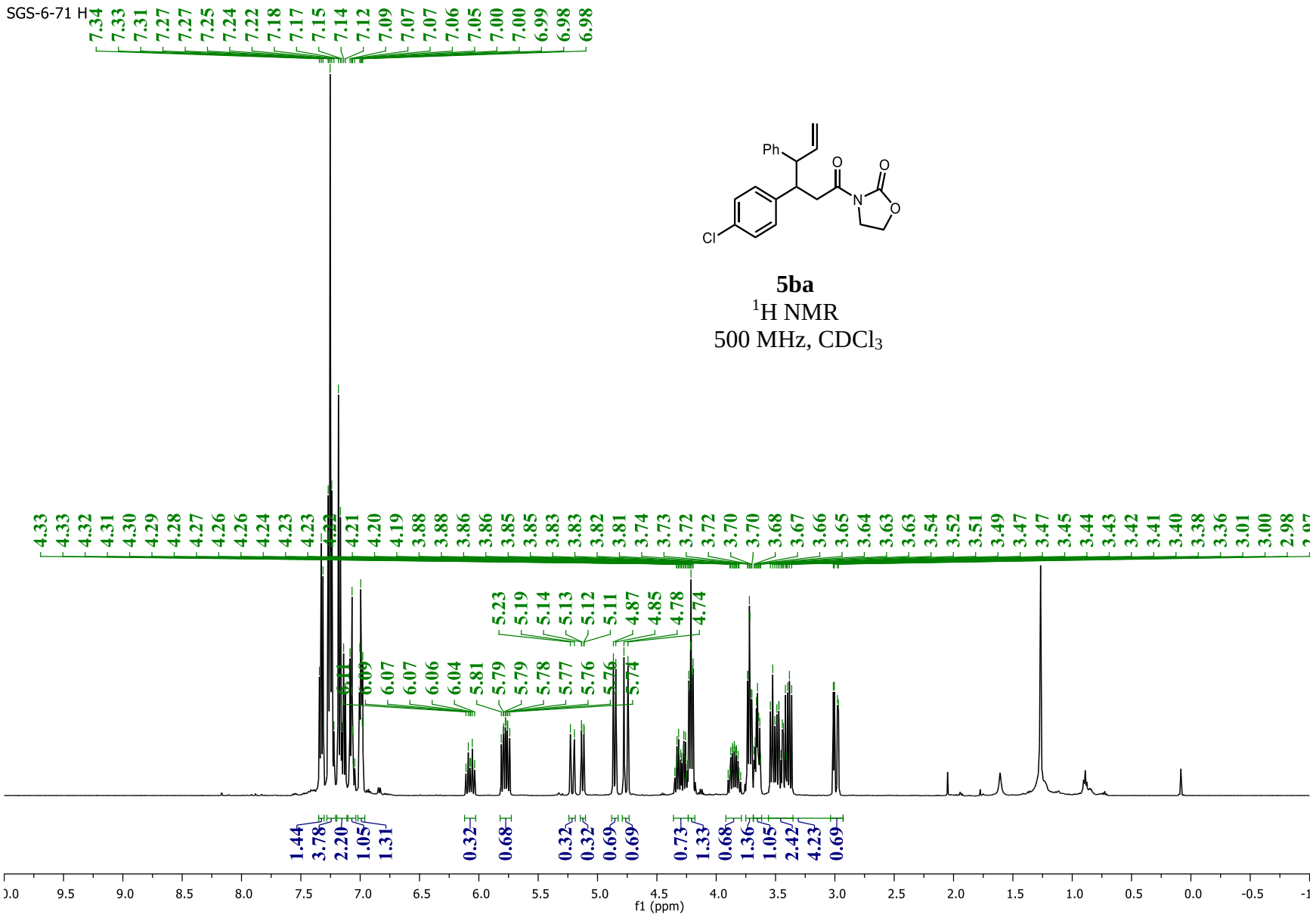
^{13}C NMR

126 MHz, $CDCl_3$





5aa
COSY NMR - aliphatic
500 MHz, $CDCl_3$

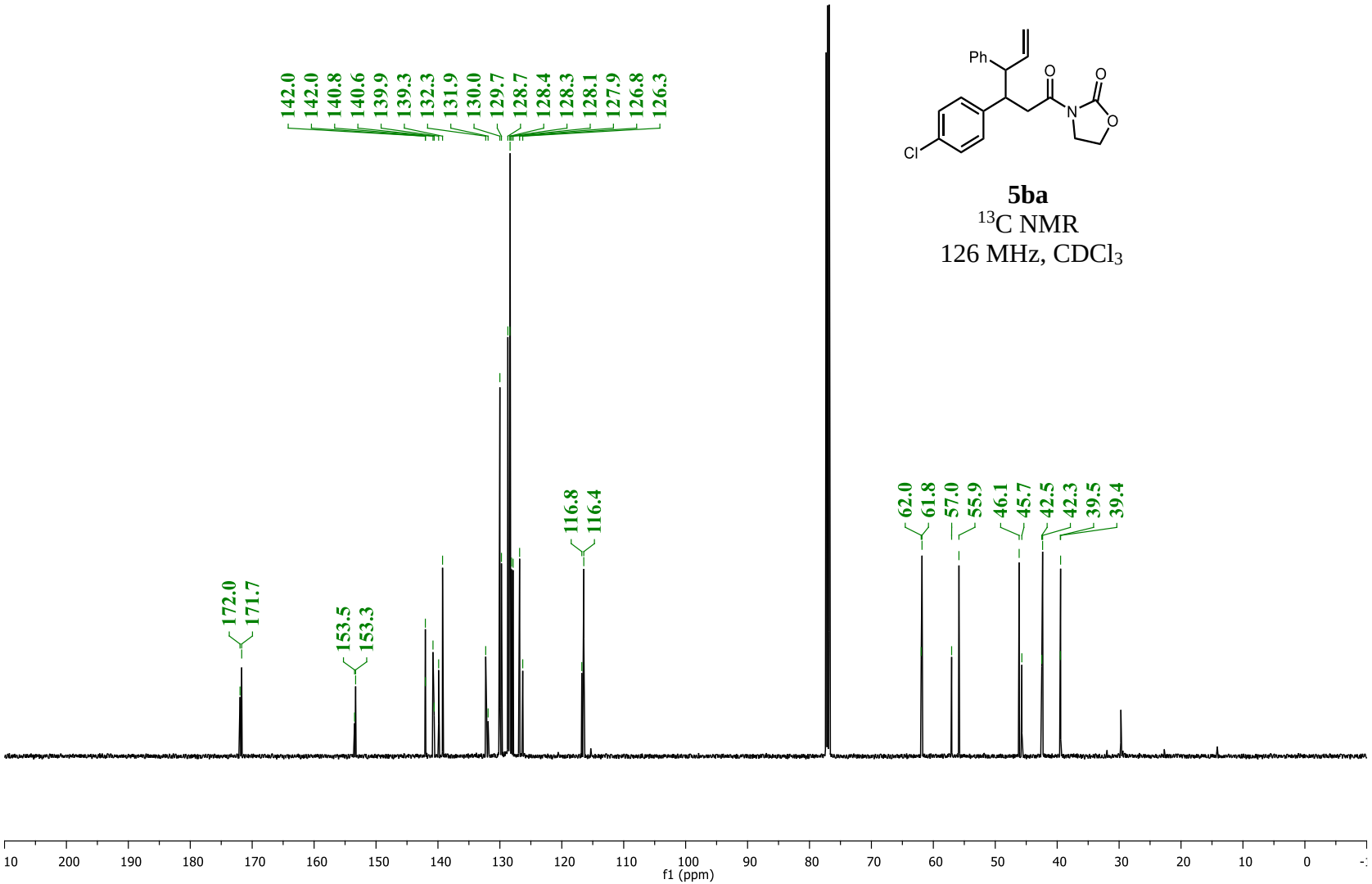


5ba

^{1}H NMR

500 MHz, $CDCl_3$

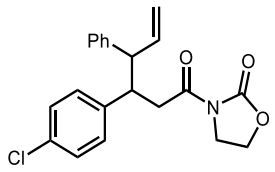
SGS-6-71 C



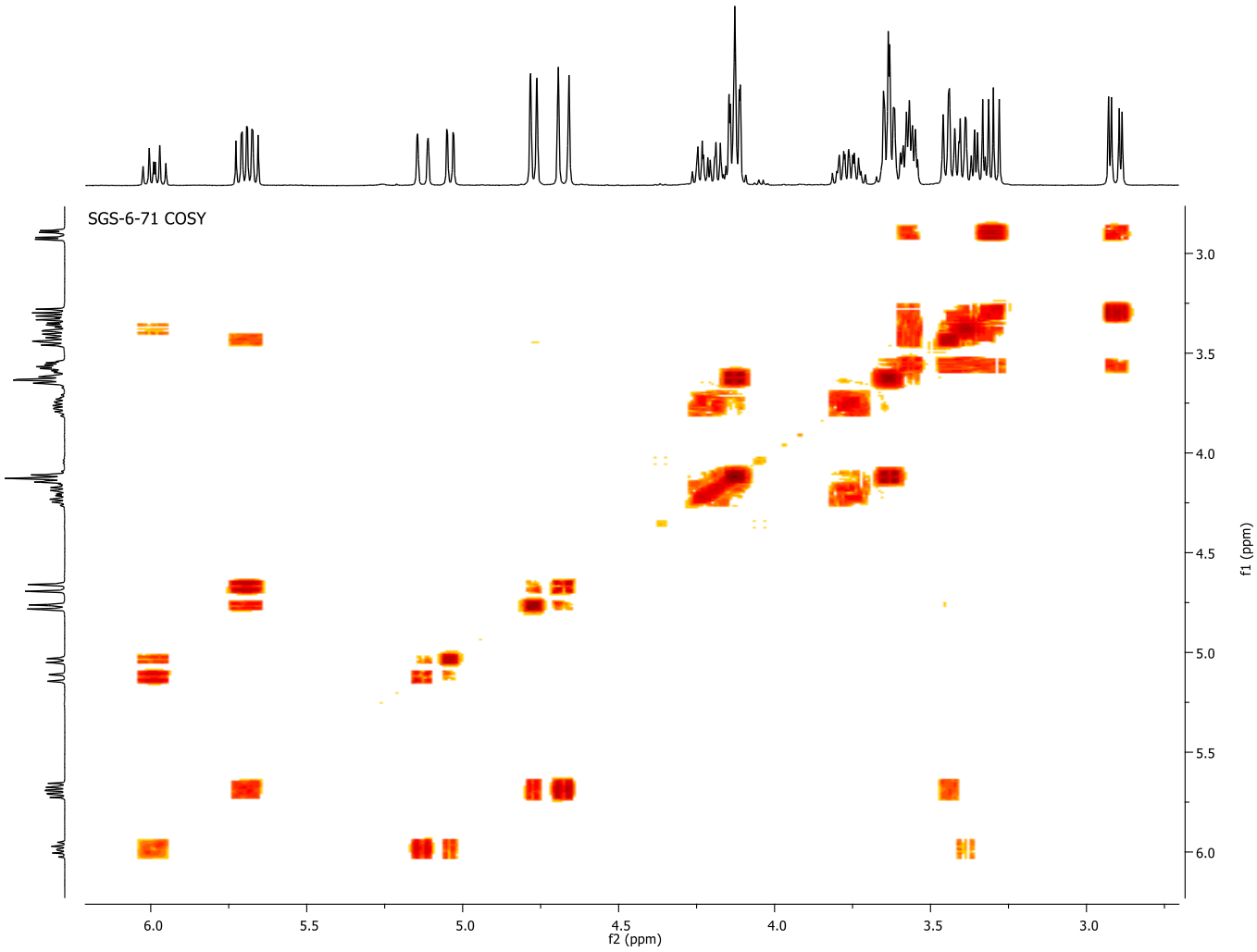
5ba

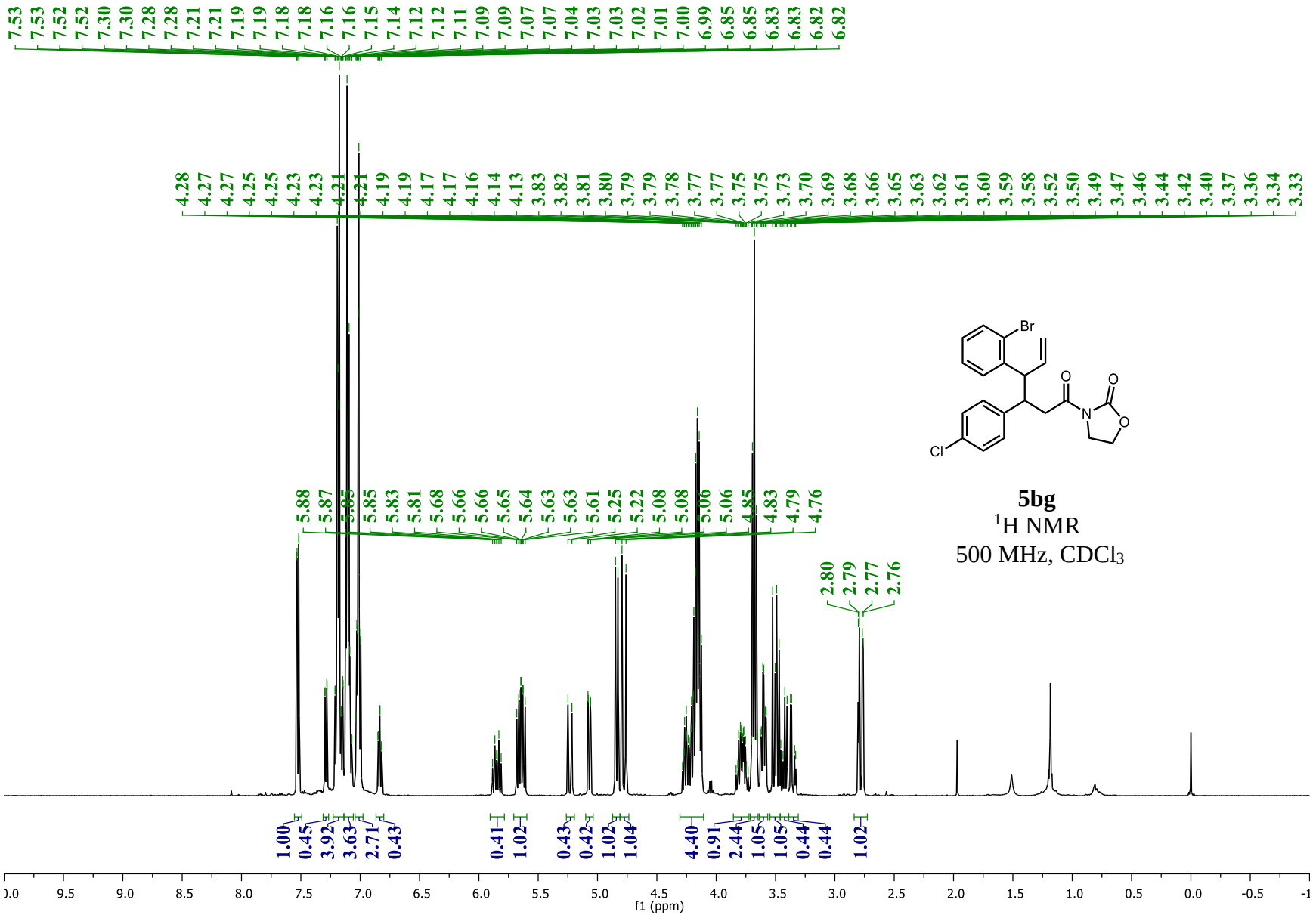
^{13}C NMR

126 MHz, $CDCl_3$



5ba
COSY NMR - aliphatic
500 MHz, $CDCl_3$

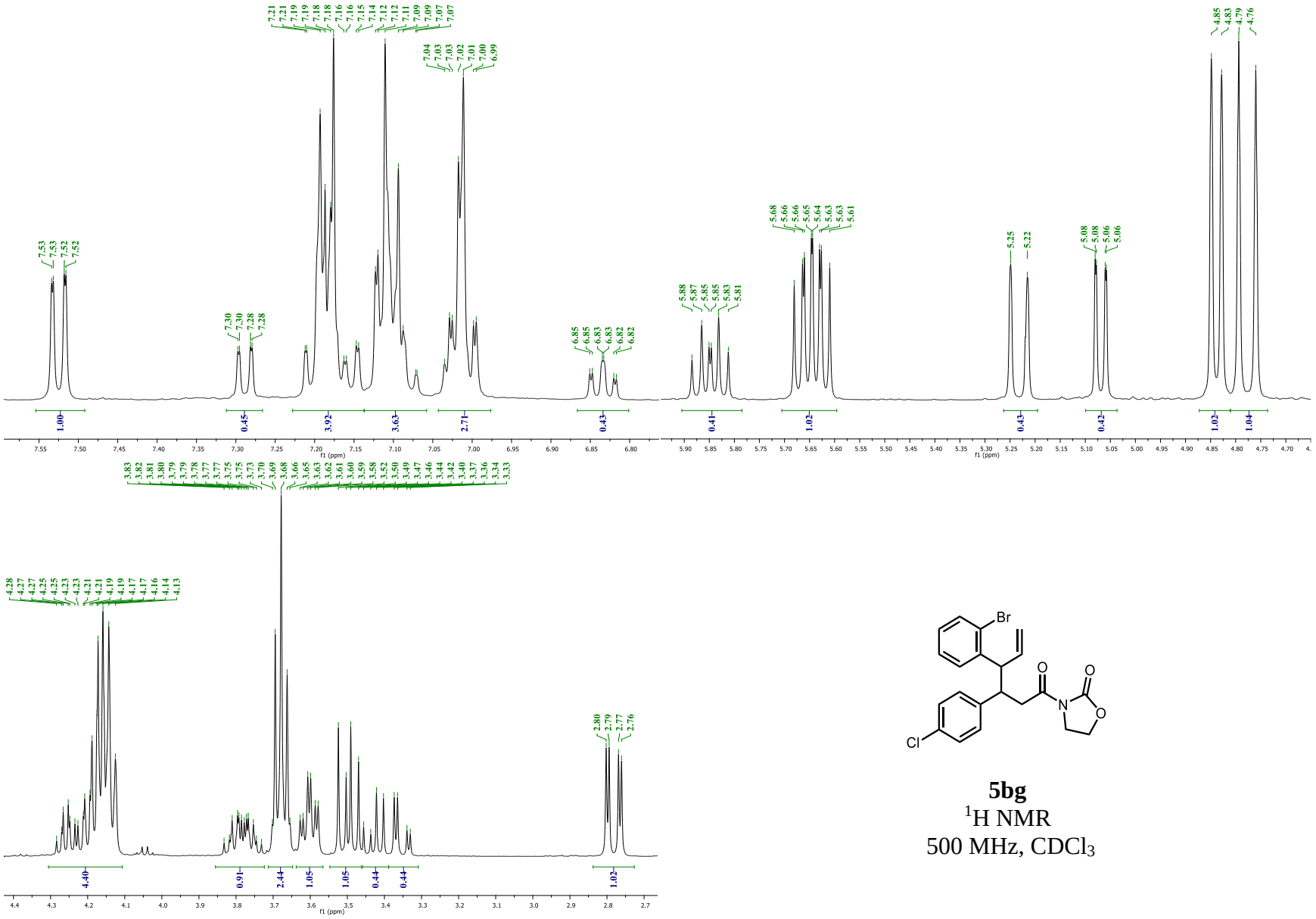




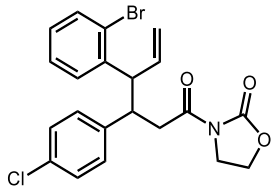
5bg

^{1}H NMR

500 MHz, $CDCl_3$



5bg
^{1}H NMR
500 MHz, $CDCl_3$

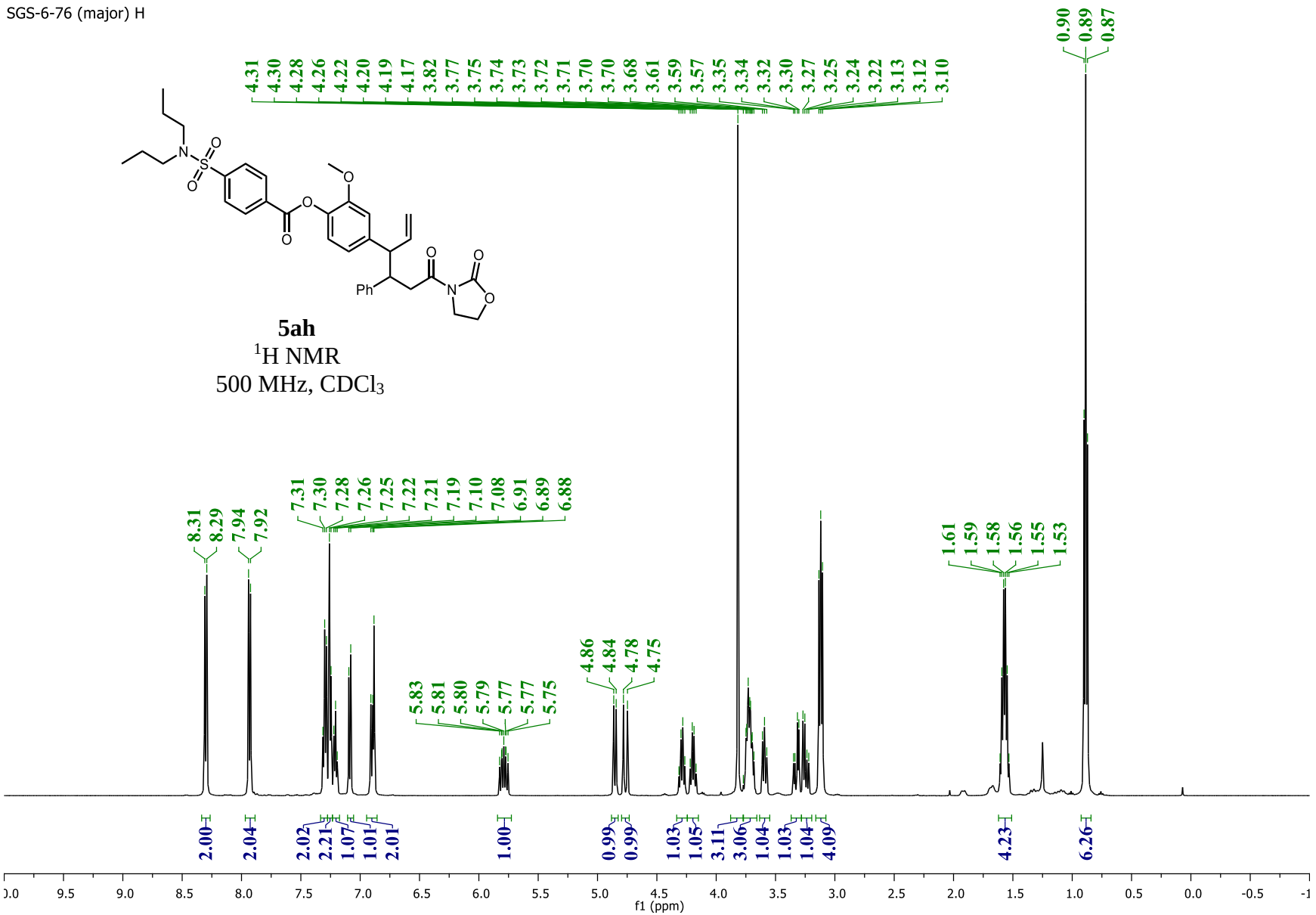


5bg

^{13}C NMR

126 MHz, $CDCl_3$

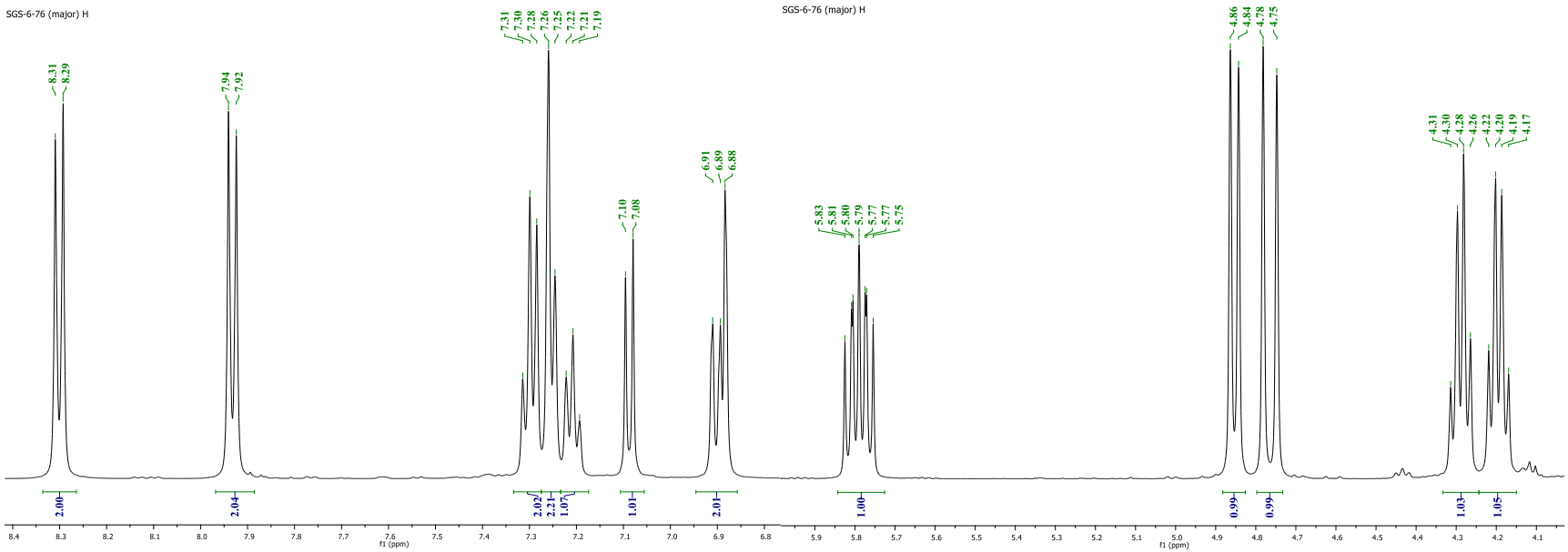
SGS-6-76 (major) H

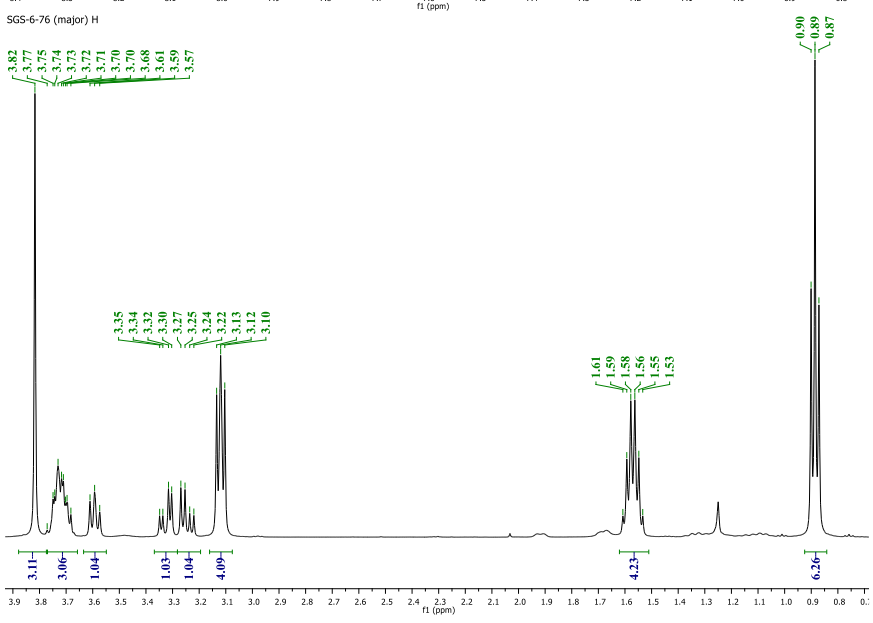


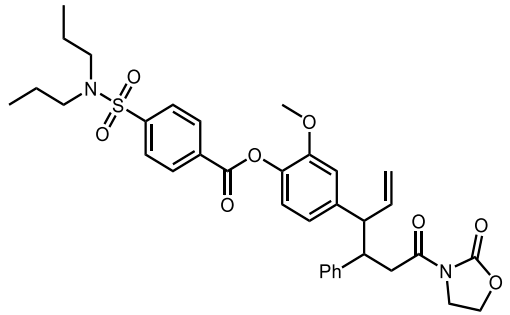
5ah

^{1}H NMR

500 MHz, $CDCl_3$

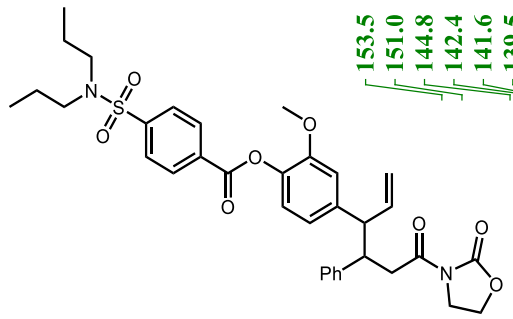






5ah
^{1}H NMR
500 MHz, $CDCl_3$

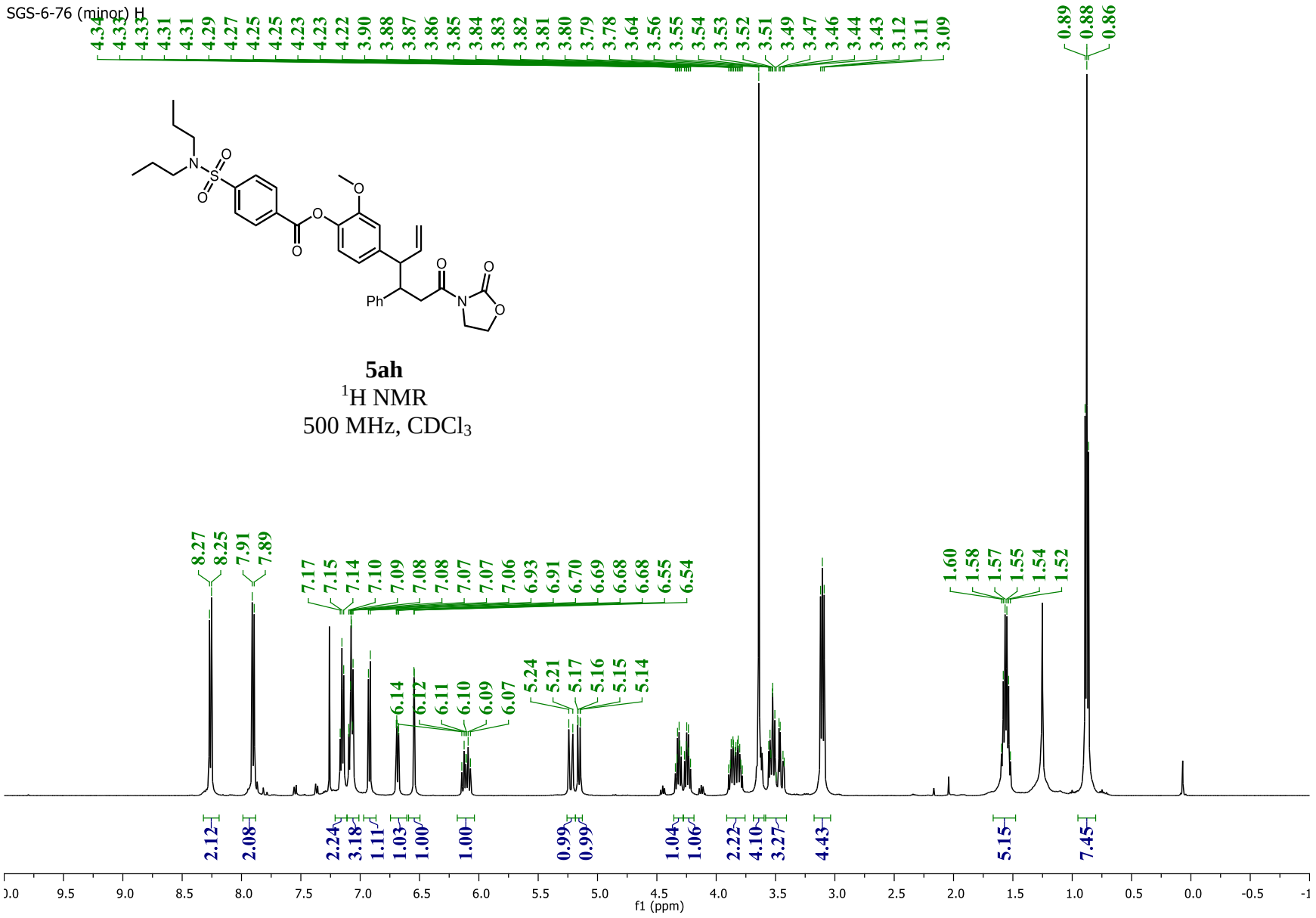
SGS-6-76 (major) C



5ah

^{13}C NMR

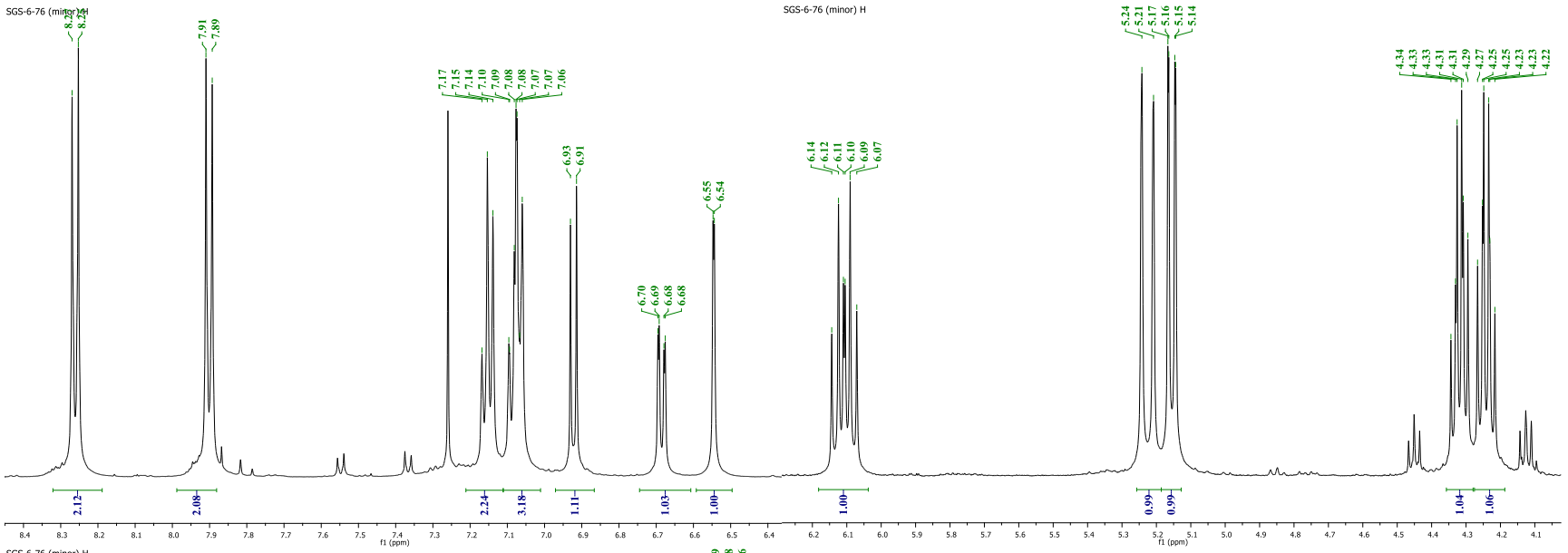
126 MHz, $CDCl_3$

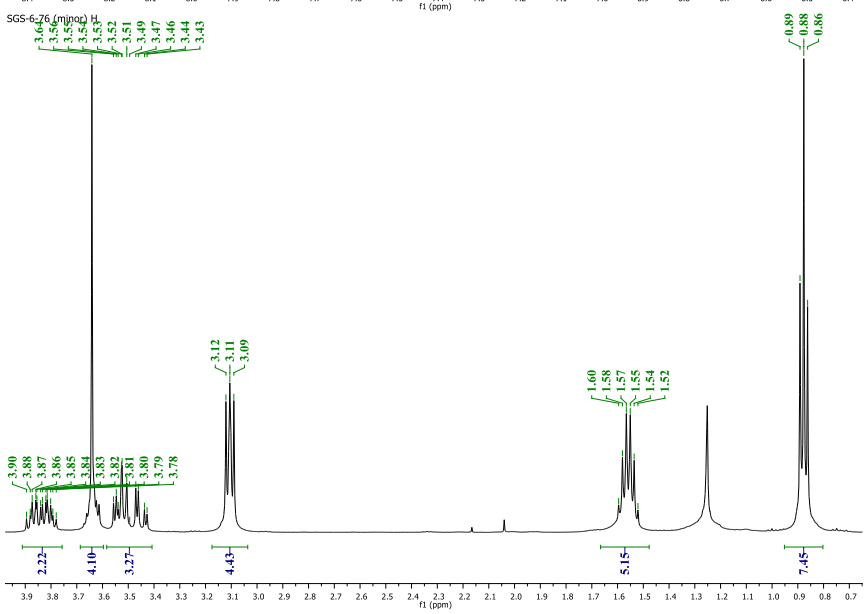


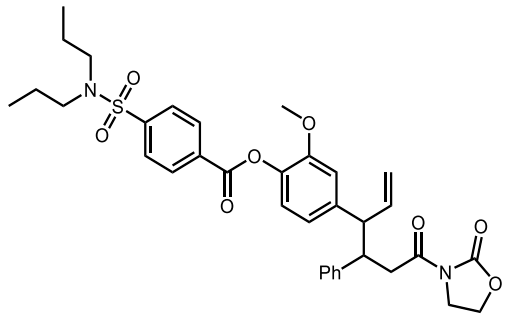
5ah

^{1}H NMR

500 MHz, $CDCl_3$





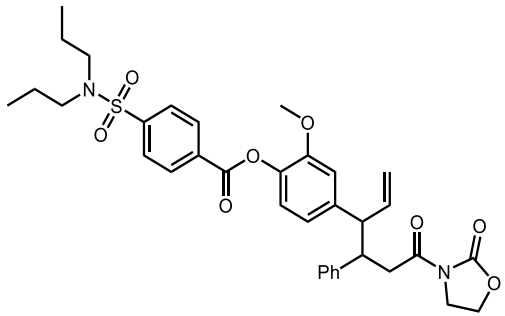


5ah

^{1}H NMR

500 MHz, $CDCl_3$

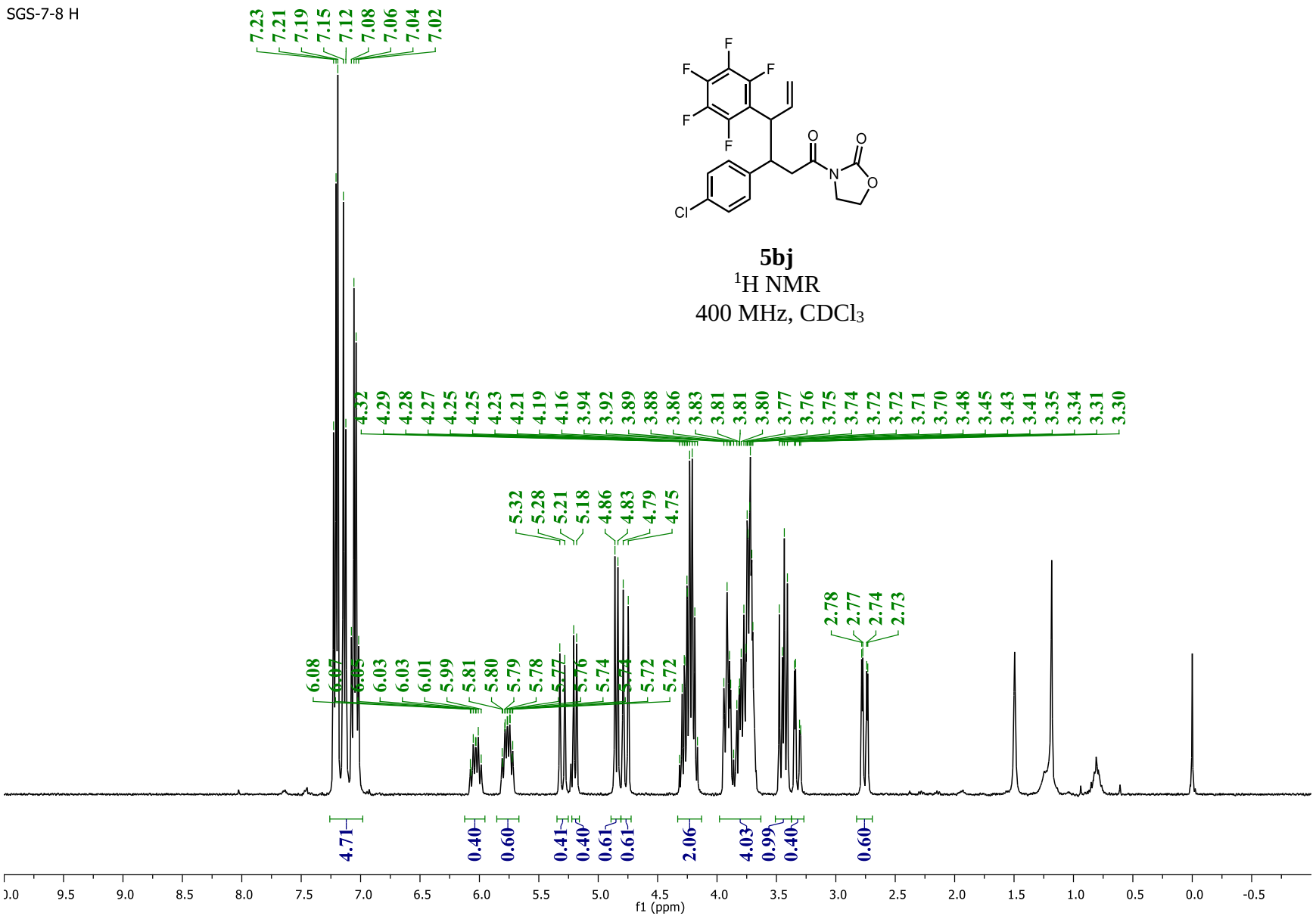
SGS-6-76 (minor) C

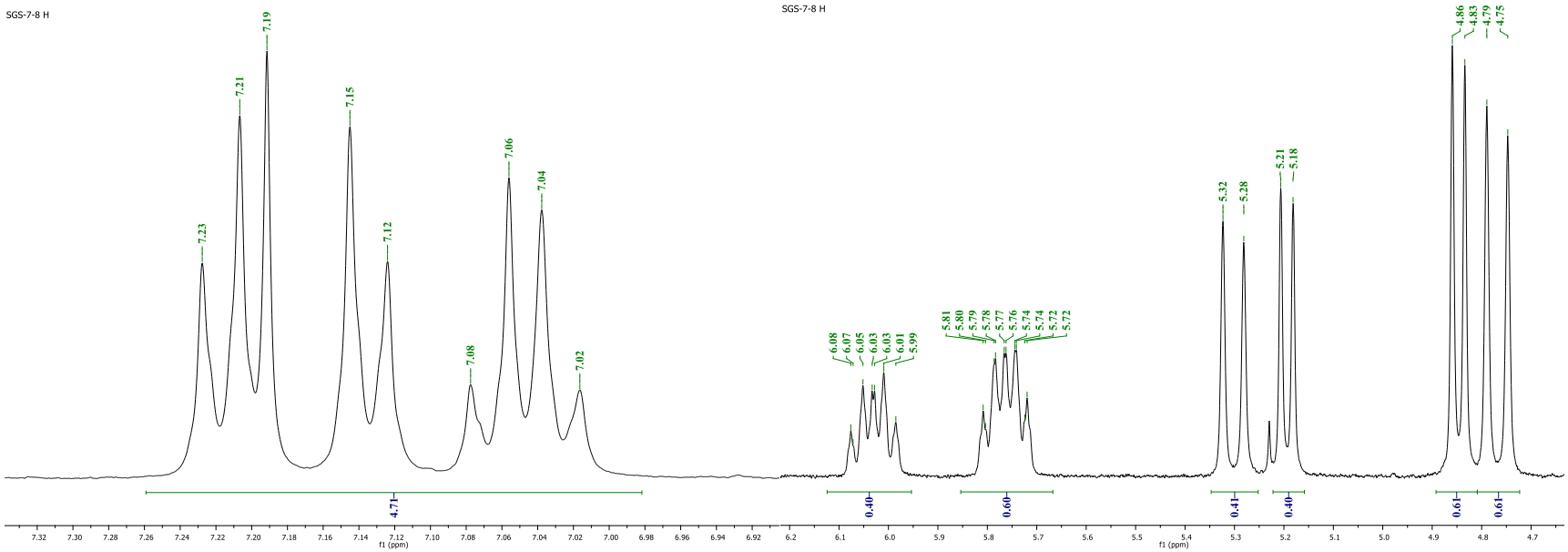


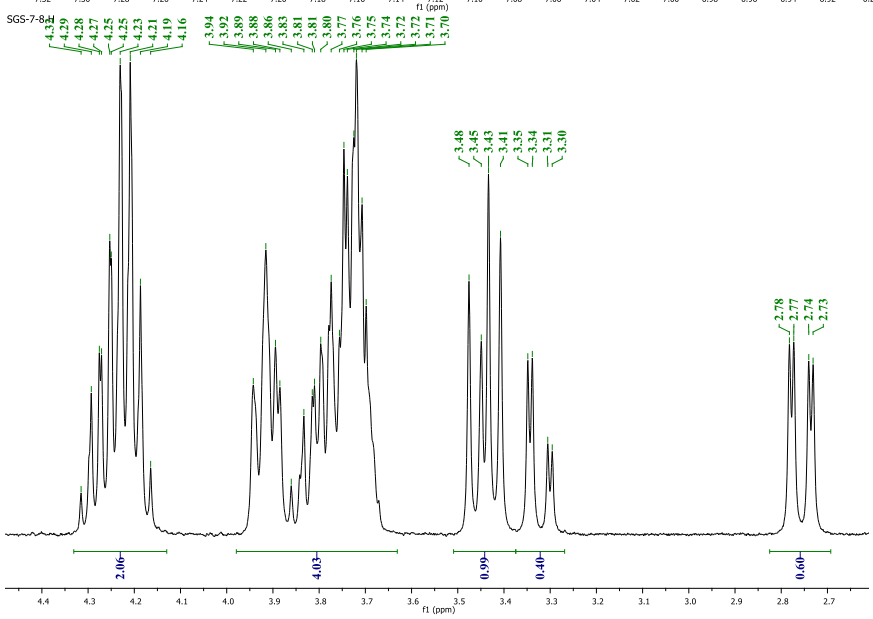
5ah

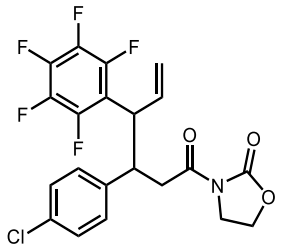
^{13}C NMR

126 MHz, $CDCl_3$


SGS-7-8 H
7.23
7.21
7.19
7.15
7.12
7.08
7.06
7.04
7.02
F
F
F
F
F
O
O
N
O
Cl
5bj
1H NMR
400 MHz, CDCl3
4.32
4.29
4.28
4.27
4.25
4.25
4.23
4.21
4.19
4.16
3.94
3.92
3.89
3.88
3.86
3.83
3.81
3.81
3.80
3.77
3.76
3.75
3.74
3.72
3.72
3.71
3.70
3.48
3.45
3.43
3.41
3.35
3.34
3.31
3.30
5.32
5.28
5.21
5.18
4.86
4.83
4.79
4.75
2.78
2.77
2.74
2.73
6.08
6.07
6.05
6.03
6.03
6.01
5.99
5.81
5.80
5.79
5.78
5.77
5.76
5.74
5.72
5.72
4.71
0.40
0.60
0.41
0.40
0.61
0.61
2.06
4.03
0.99
0.40
0.60
f1 (ppm)



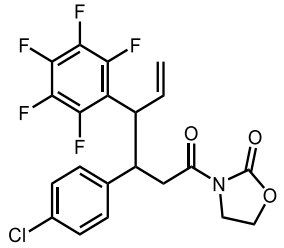




5bj

^{1}H NMR

400 MHz, $CDCl_3$



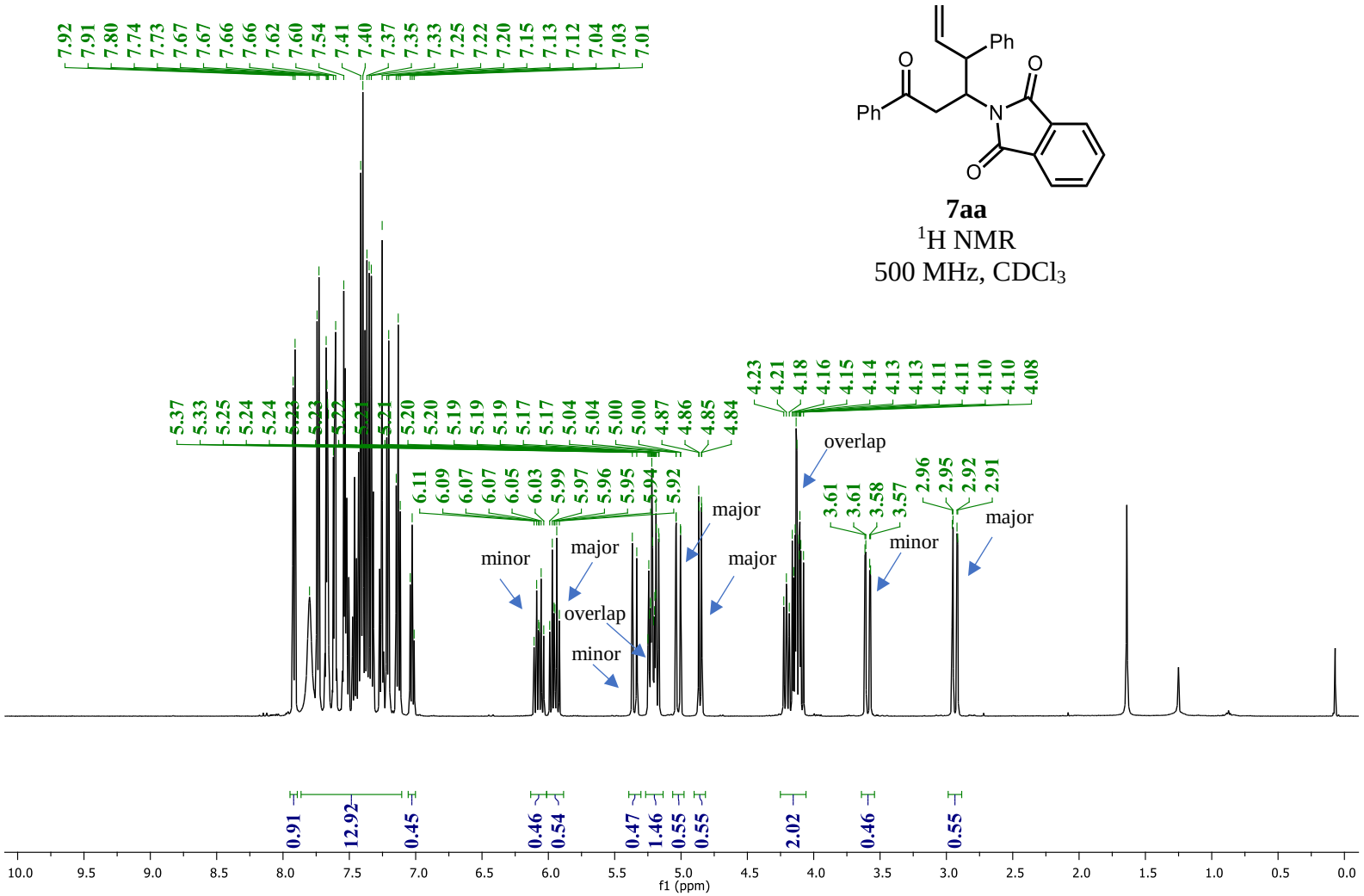
5bj

^{13}C NMR

126 MHz, $CDCl_3$

7.3 NMR spectra: Phthalimide Scope

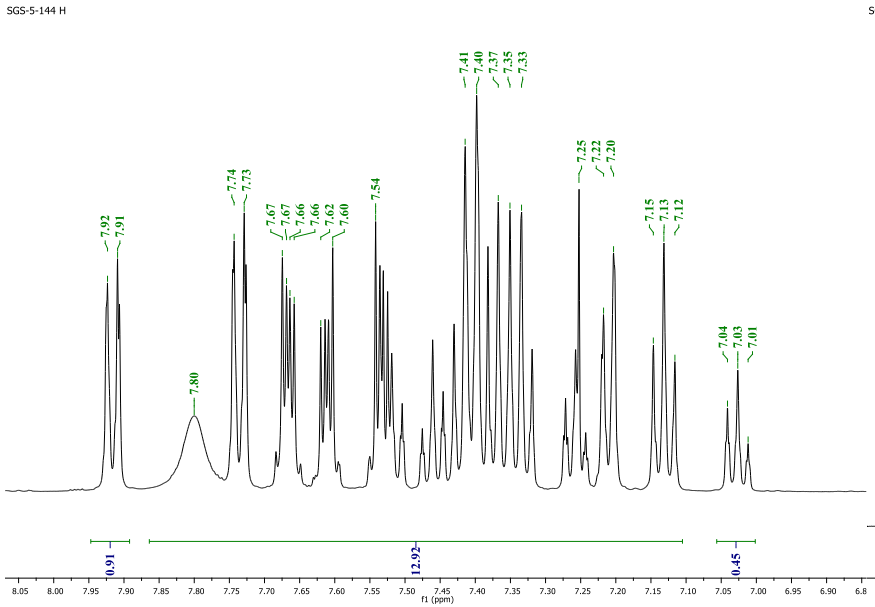
SGS-5-144 H

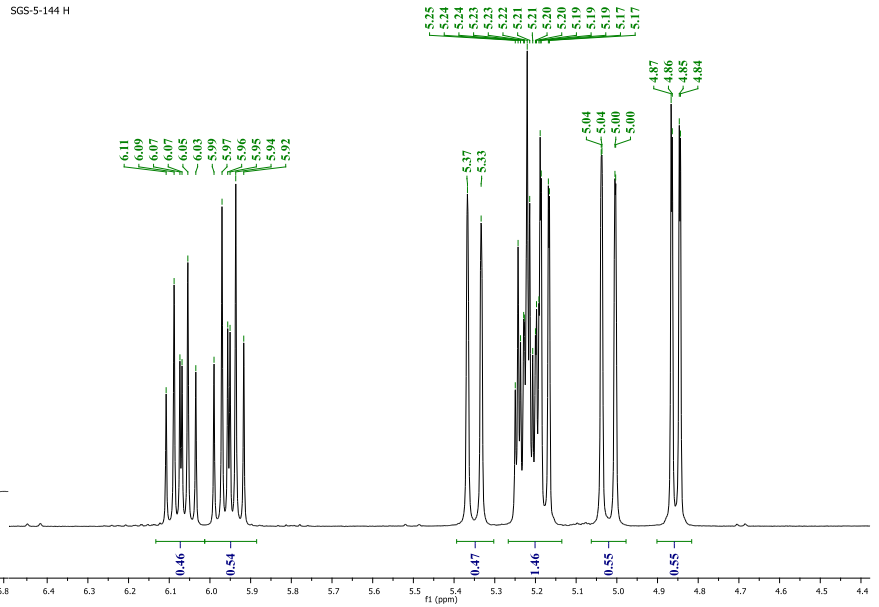


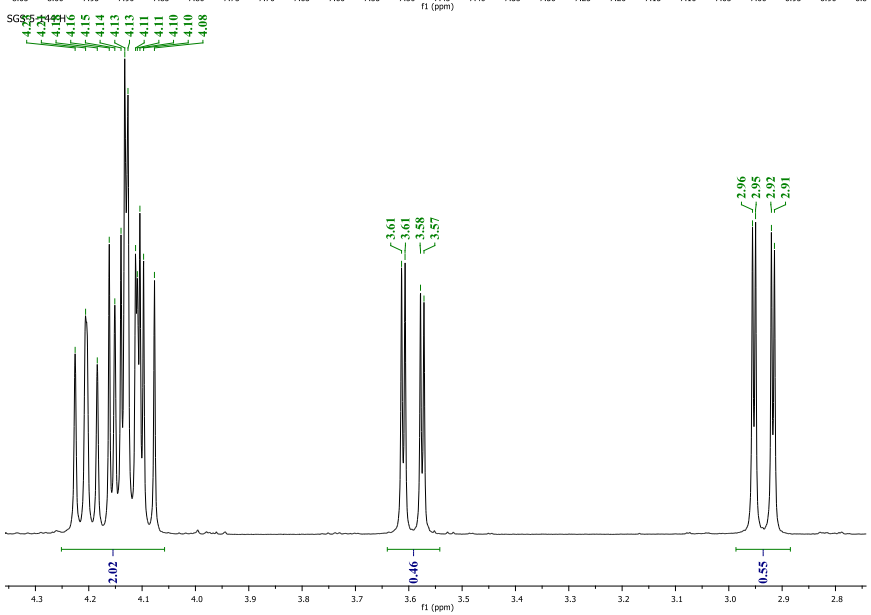
7aa

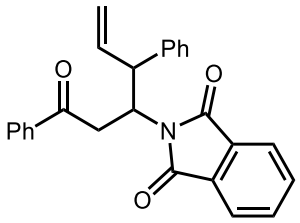
^{1}H NMR

500 MHz, $CDCl_3$







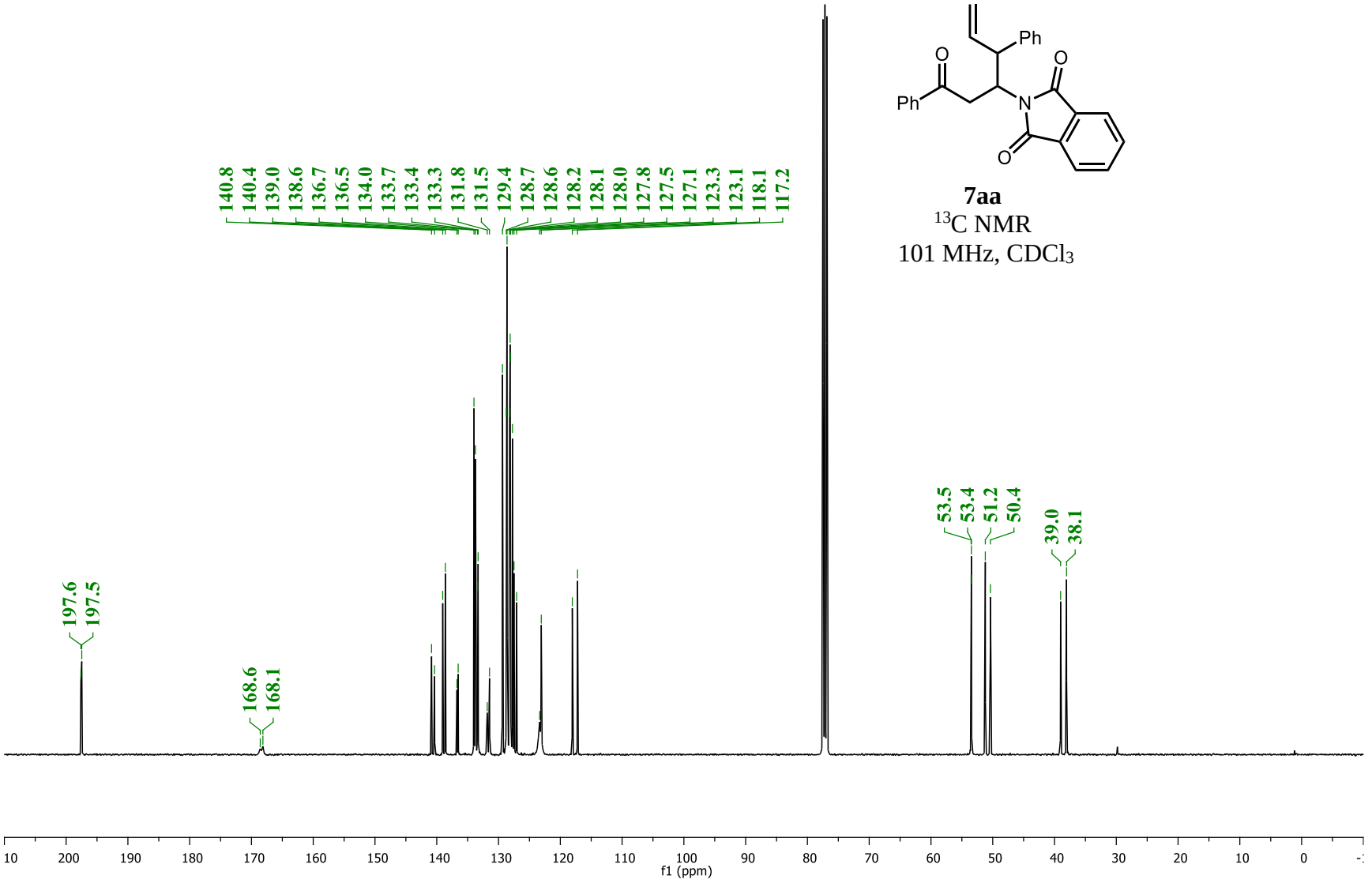


7aa

^{1}H NMR

500 MHz, $CDCl_3$

SGS-5-144 C

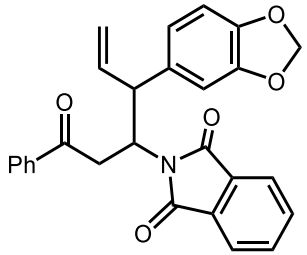


7aa

^{13}C NMR

101 MHz, $CDCl_3$

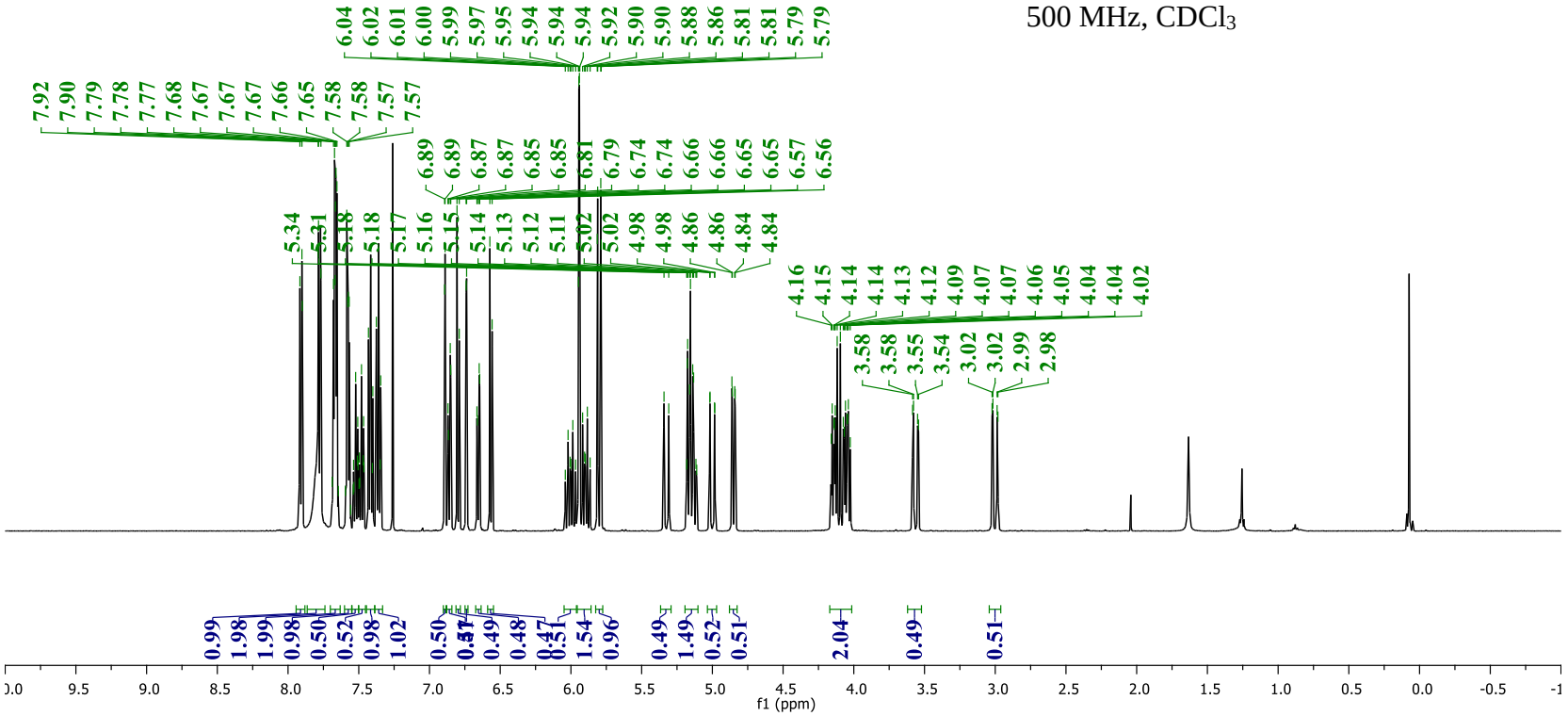
SGS-6-1 H

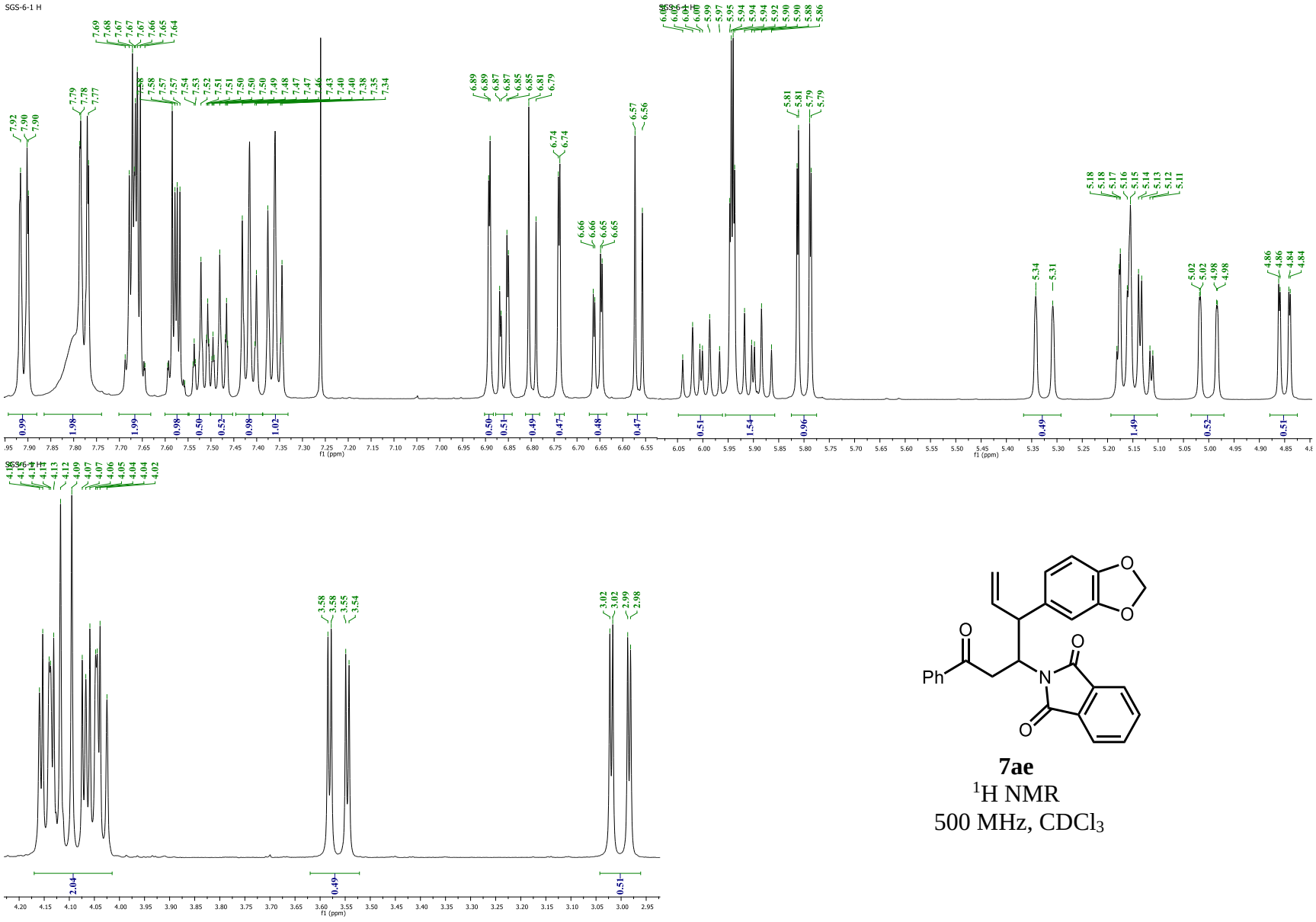


7ae

^{1}H NMR

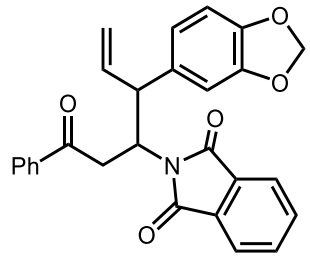
500 MHz, $CDCl_3$





7ae
^{1}H NMR
500 MHz, $CDCl_3$

SGS-6-1 C

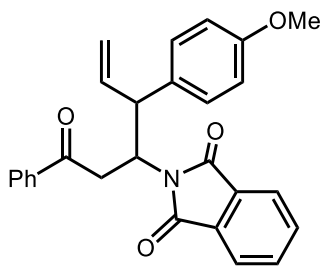


7ae

^{13}C NMR

126 MHz, $CDCl_3$

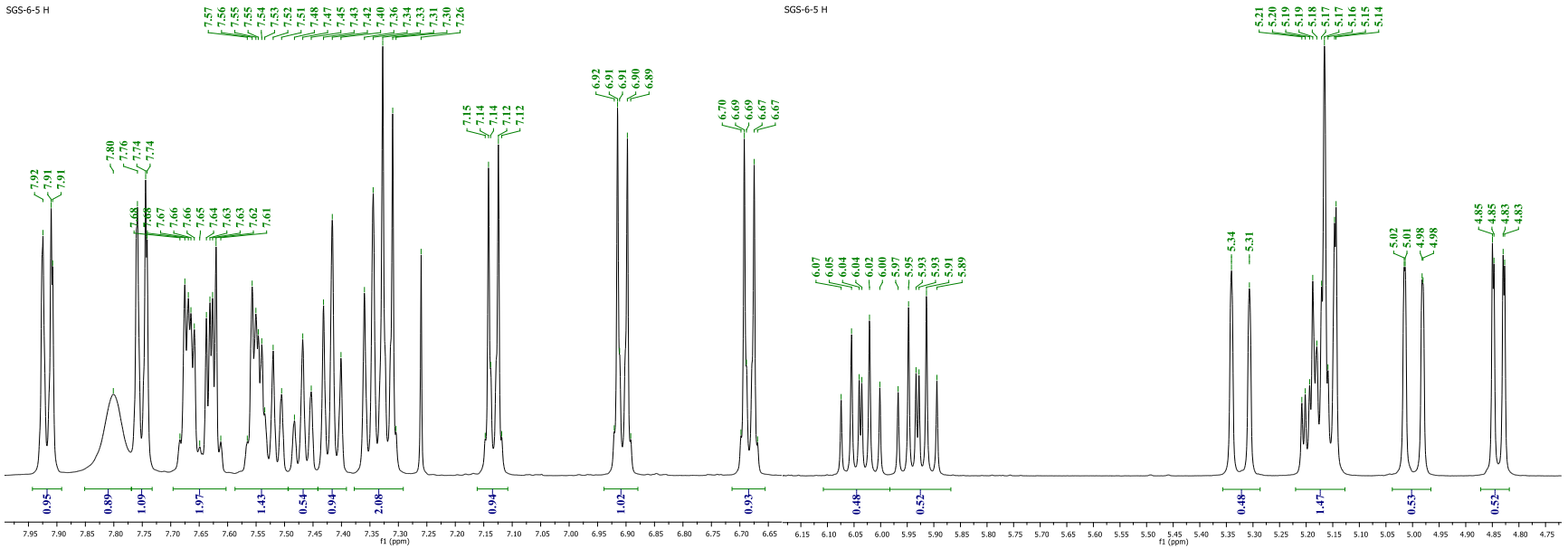
SGS-6-5 H

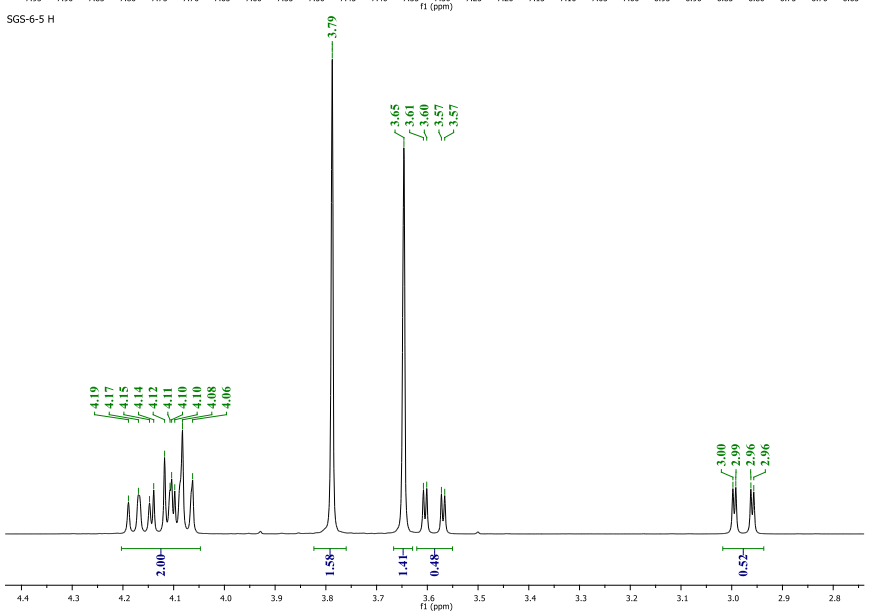


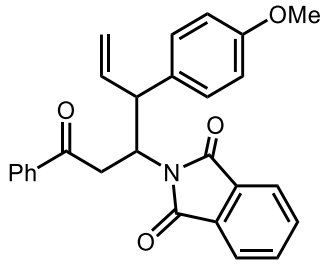
7ac

^{1}H NMR

500 MHz, $CDCl_3$





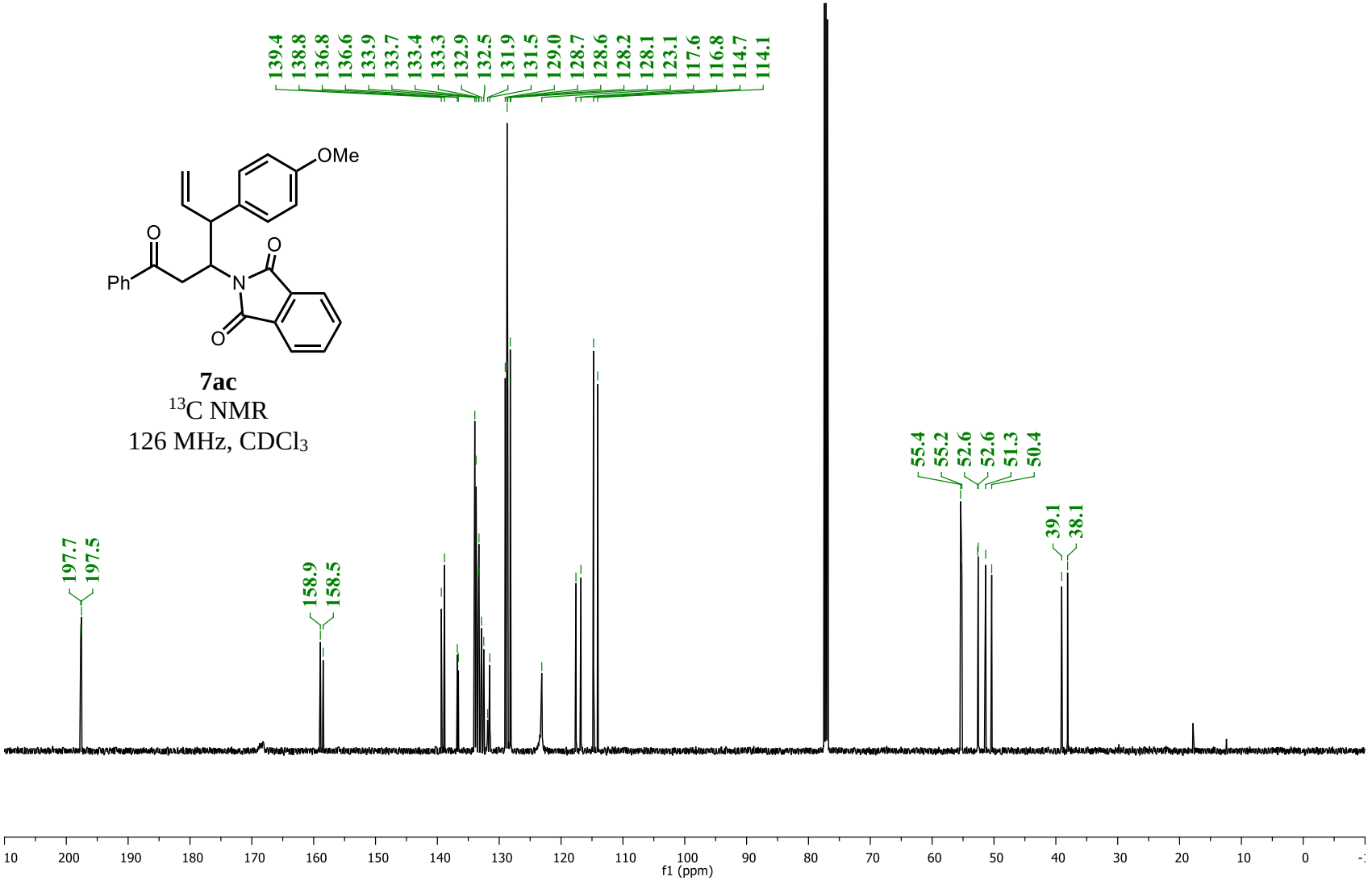


7ac

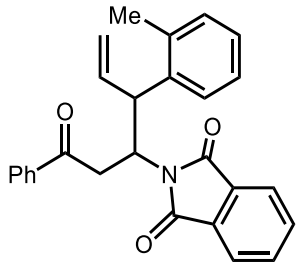
^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-5 C


139.4
138.8
136.8
136.6
133.9
133.7
133.4
133.3
132.9
132.5
131.9
131.5
129.0
128.7
128.6
128.2
128.1
123.1
117.6
116.8
114.7
114.1
OMe
O
O
Ph
N
O
7ac
13C NMR
126 MHz, CDCl3
55.4
55.2
52.6
52.6
51.3
50.4
39.1
38.1
197.7
197.5
158.9
158.5
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

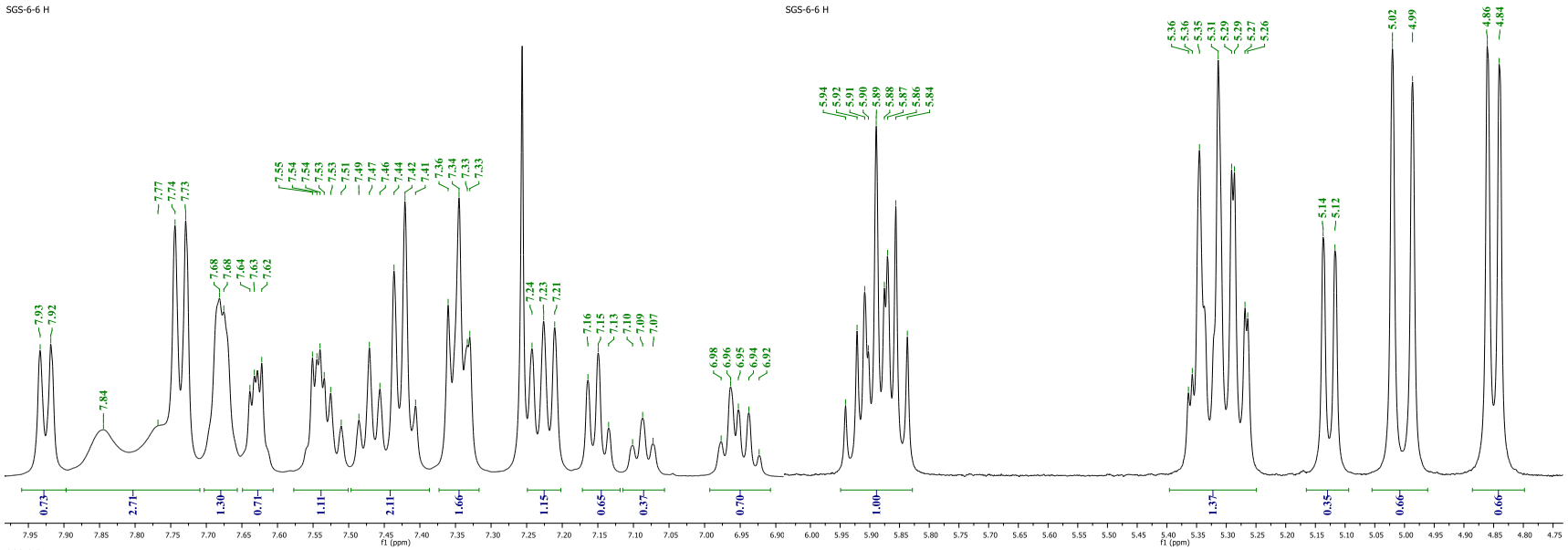
SGS-6-6 H

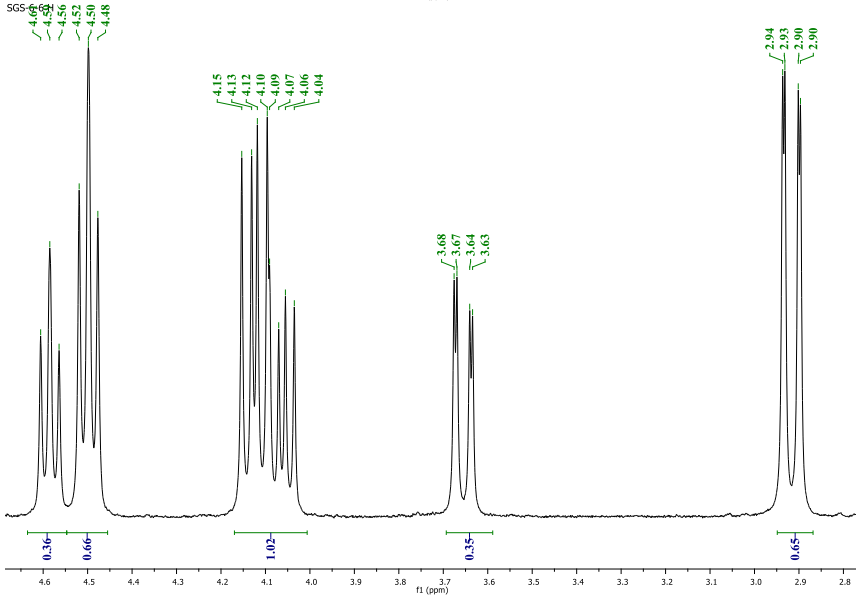


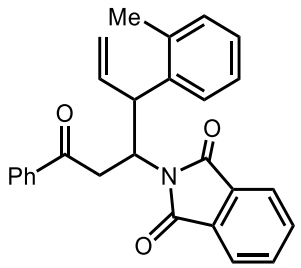
7ai

^{1}H NMR

500 MHz, $CDCl_3$

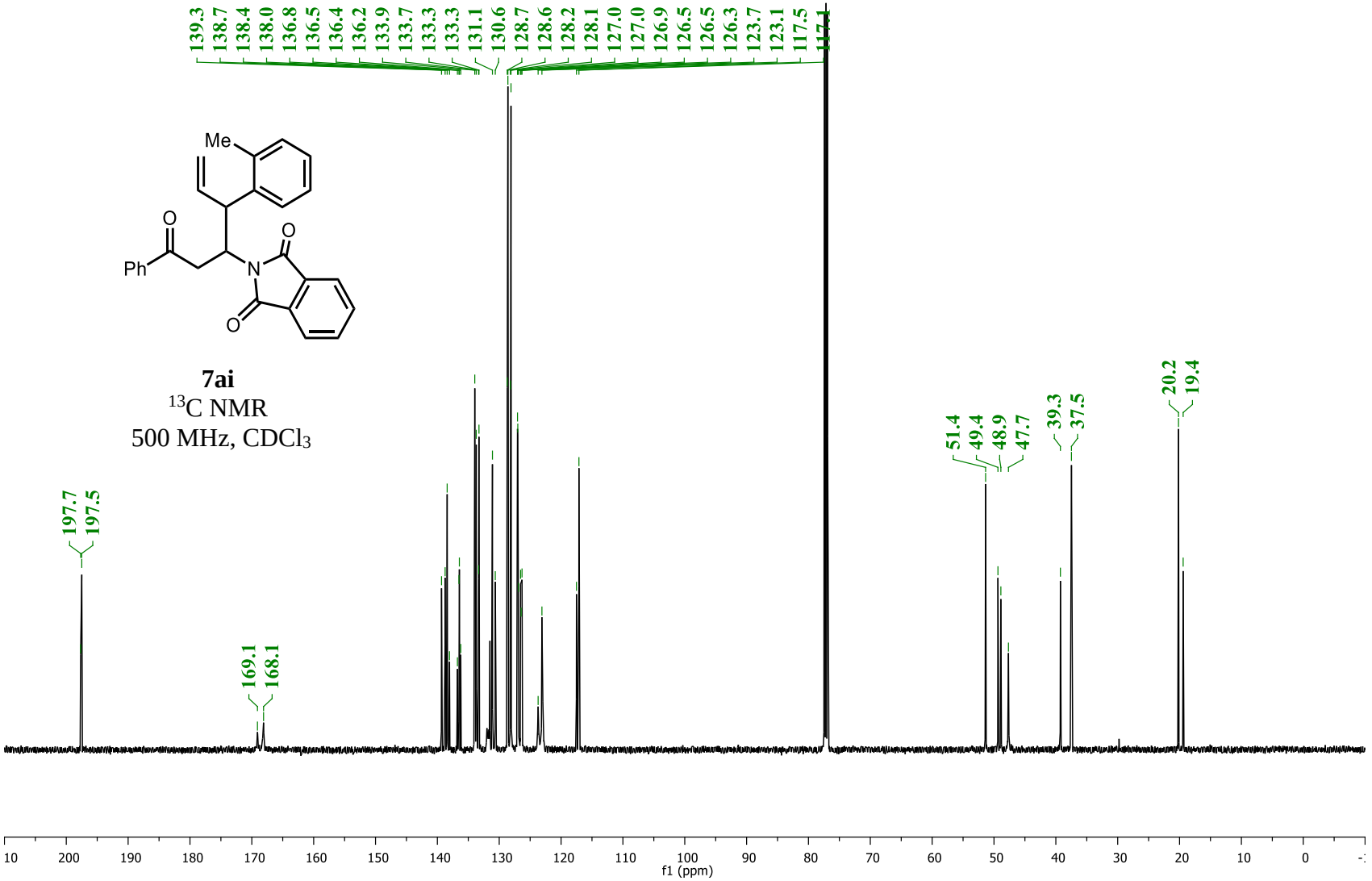




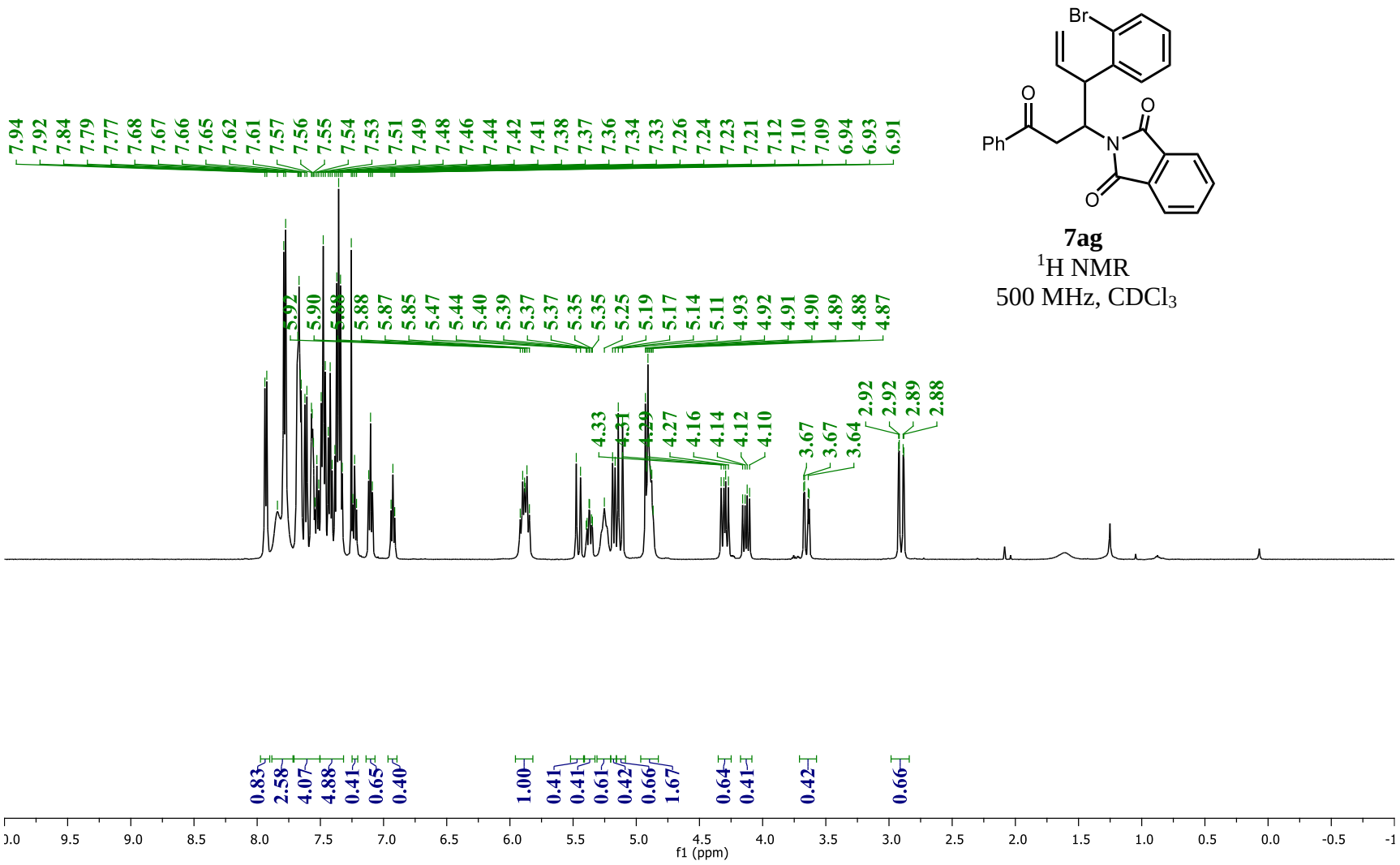


7ai
^{1}H NMR
500 MHz, $CDCl_3$

SGS-6-6 C


139.3
138.7
138.4
138.0
136.8
136.5
136.4
136.2
133.9
133.7
133.3
133.3
131.1
130.6
128.7
128.6
128.2
128.1
127.0
127.0
126.9
126.5
126.5
126.3
123.7
123.1
117.5
117.1
Me
O
Ph
N
O
O
7ai
13C NMR
500 MHz, CDCl3
197.7
197.5
169.1
168.1
51.4
49.4
48.9
47.7
39.3
37.5
20.2
19.4
f1 (ppm)

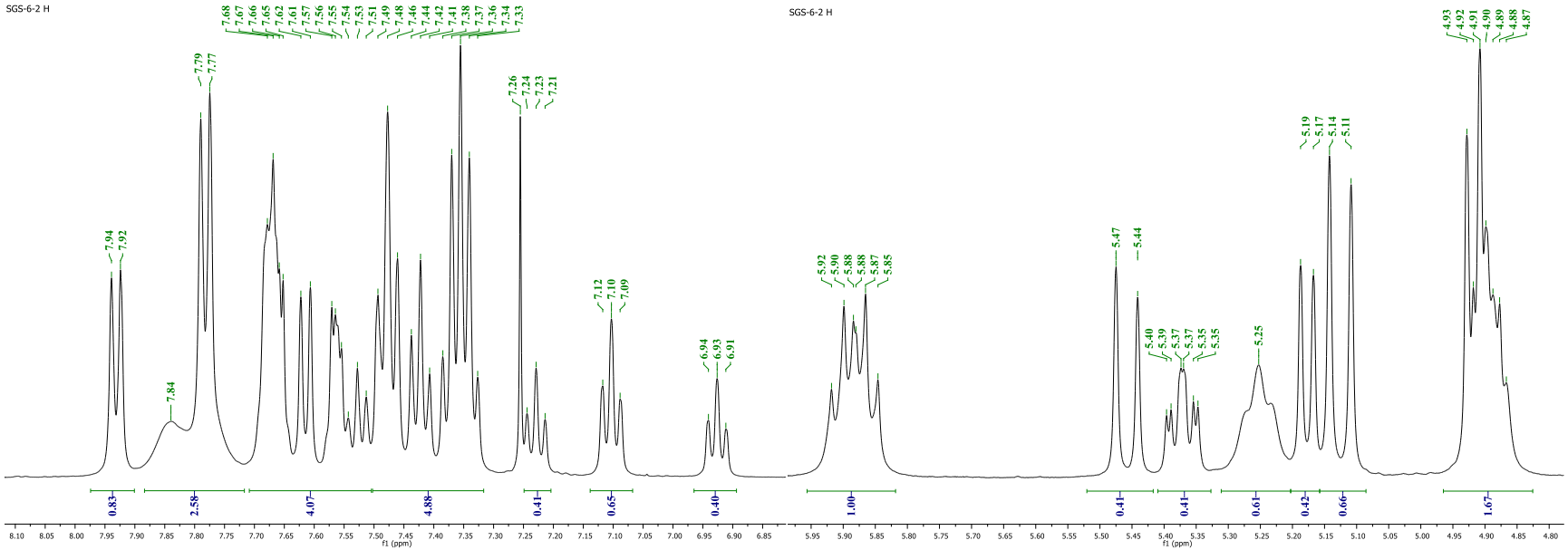
SGS-6-2 H

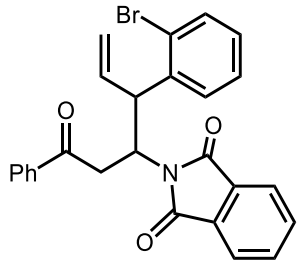


7ag

^{1}H NMR

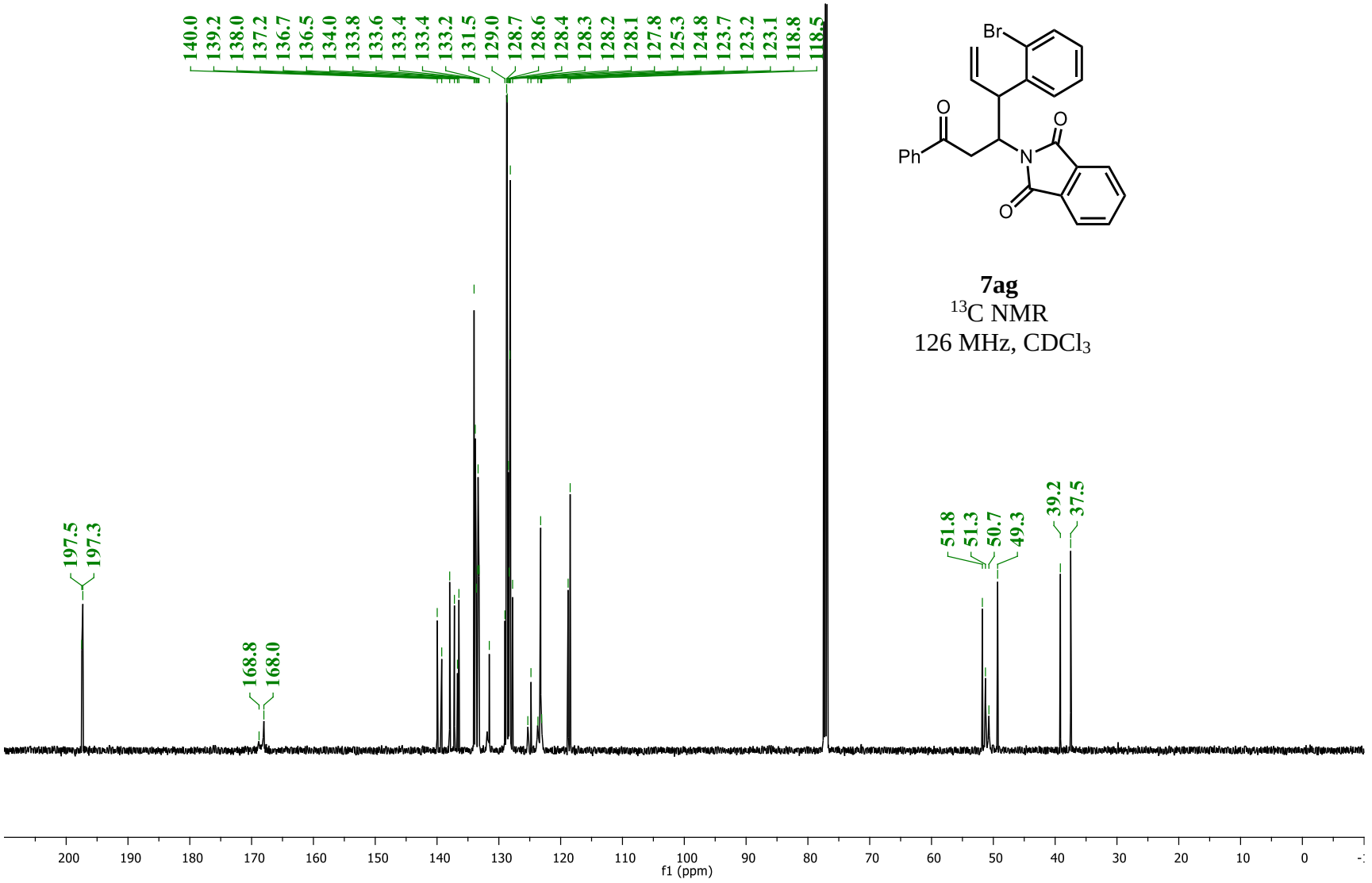
500 MHz, $CDCl_3$





7ag
^{1}H NMR
500 MHz, $CDCl_3$

SGS-6-2 C

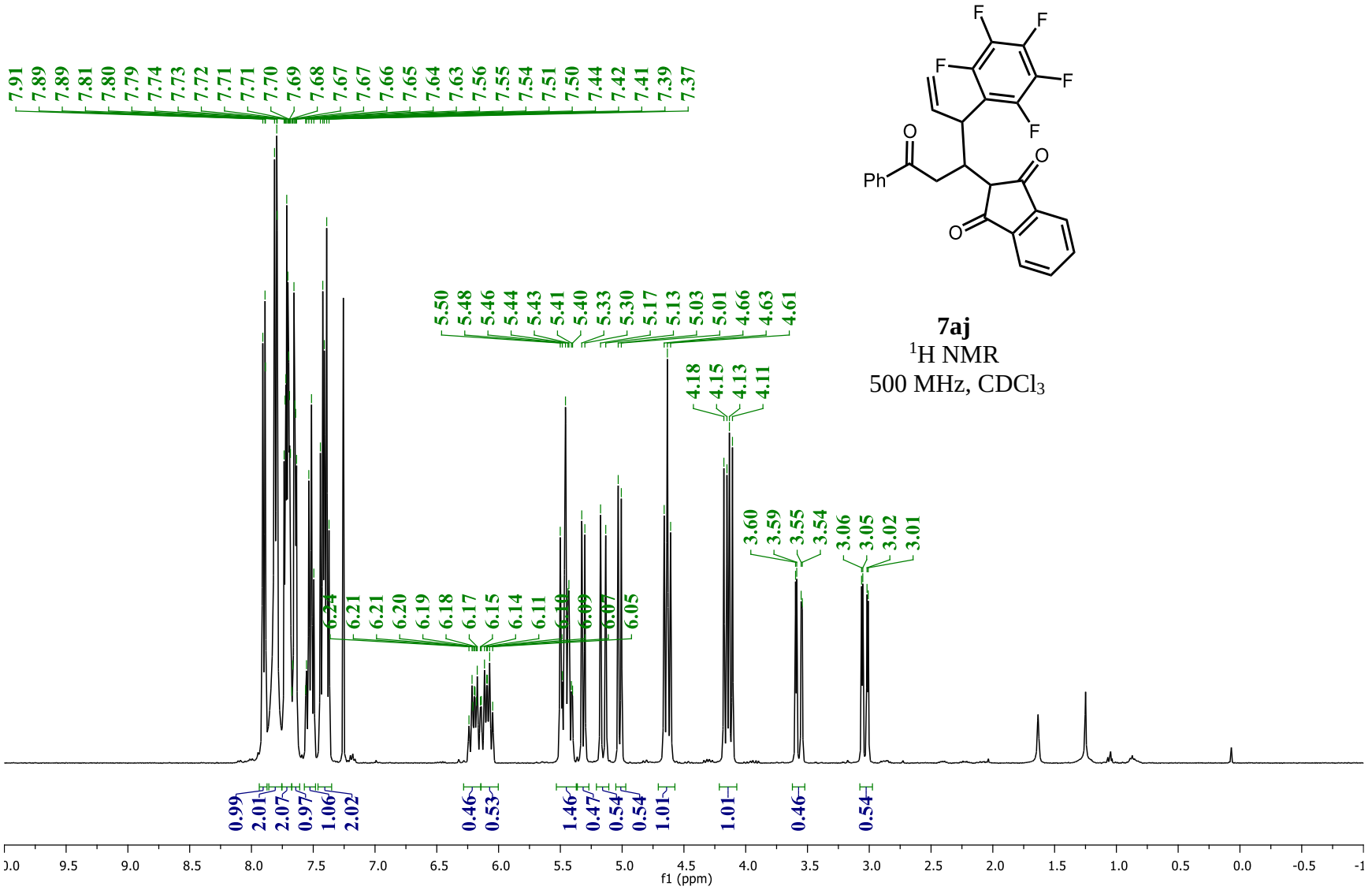


7ag

^{13}C NMR

126 MHz, $CDCl_3$

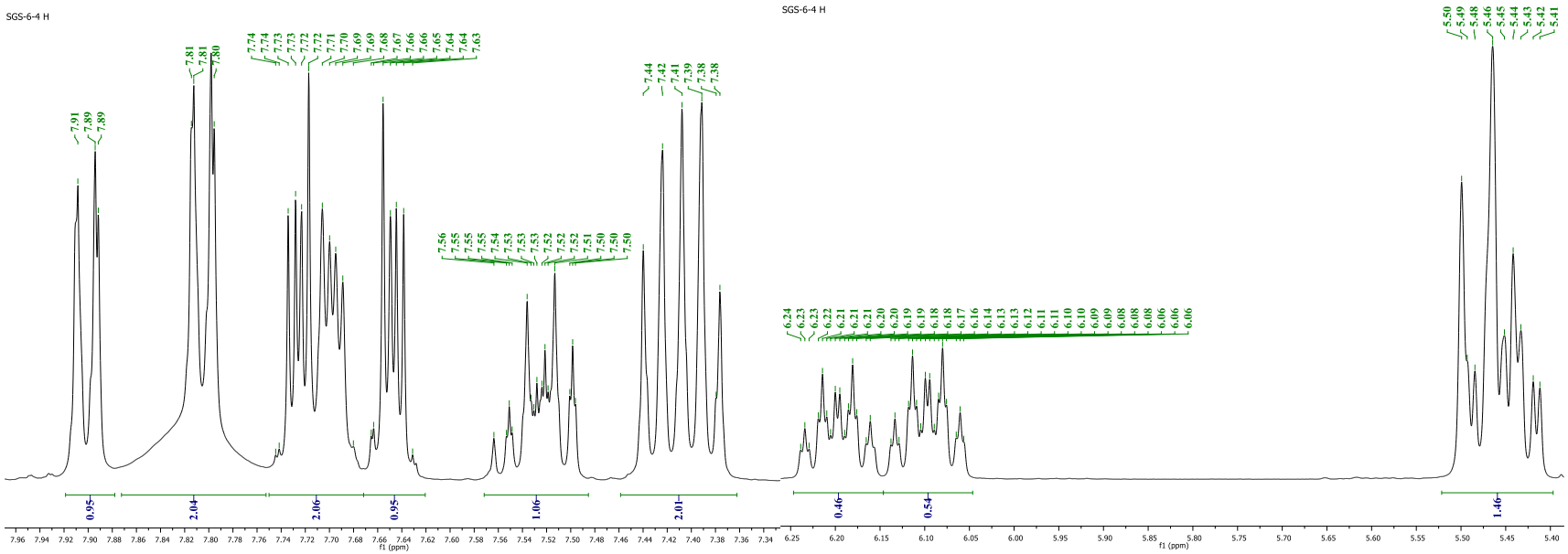
SGS-6-4 H

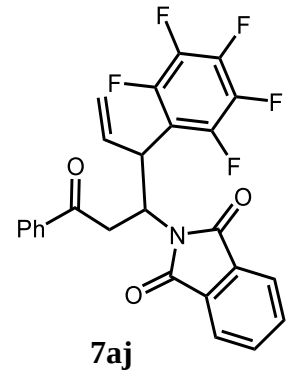


7aj

^{1}H NMR

500 MHz, $CDCl_3$

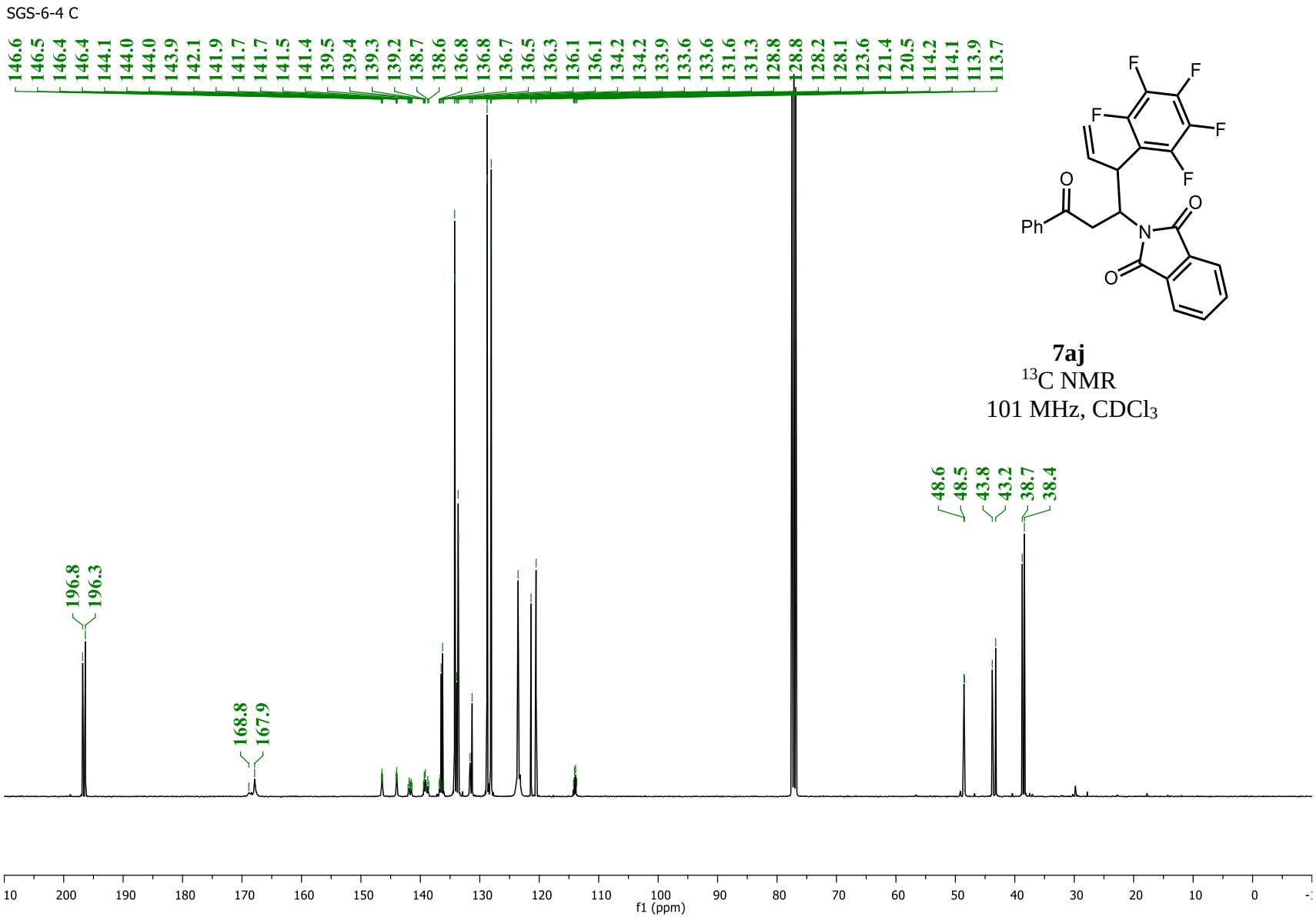




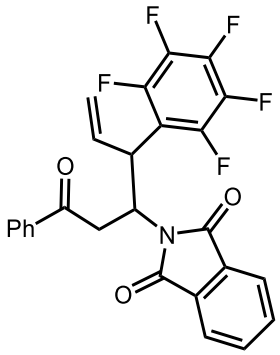
7aj

^{1}H NMR

500 MHz, $CDCl_3$


SGS-6-4 C
146.6
146.5
146.4
146.4
144.1
144.0
144.0
143.9
142.1
141.9
141.7
141.7
141.5
141.4
139.5
139.4
139.3
139.2
138.7
138.6
136.8
136.8
136.7
136.5
136.3
136.1
136.1
134.2
134.2
133.9
133.6
133.6
131.6
131.3
128.8
128.8
128.2
128.1
123.6
121.4
120.5
114.2
114.1
113.9
113.7
F
F
F
F
F
O
Ph
N
O
O
7aj
13C NMR
101 MHz, CDCl3
48.6
48.5
43.8
43.2
38.7
38.4
196.8
196.3
168.8
167.9
f1 (ppm)

SGS-6-4 C Zoom

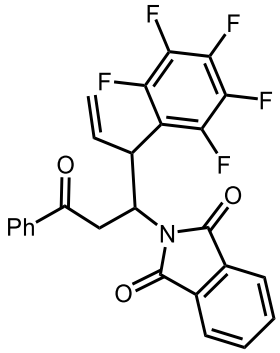


7aj

^{13}C NMR

101 MHz, $CDCl_3$

SGS-6-4 F

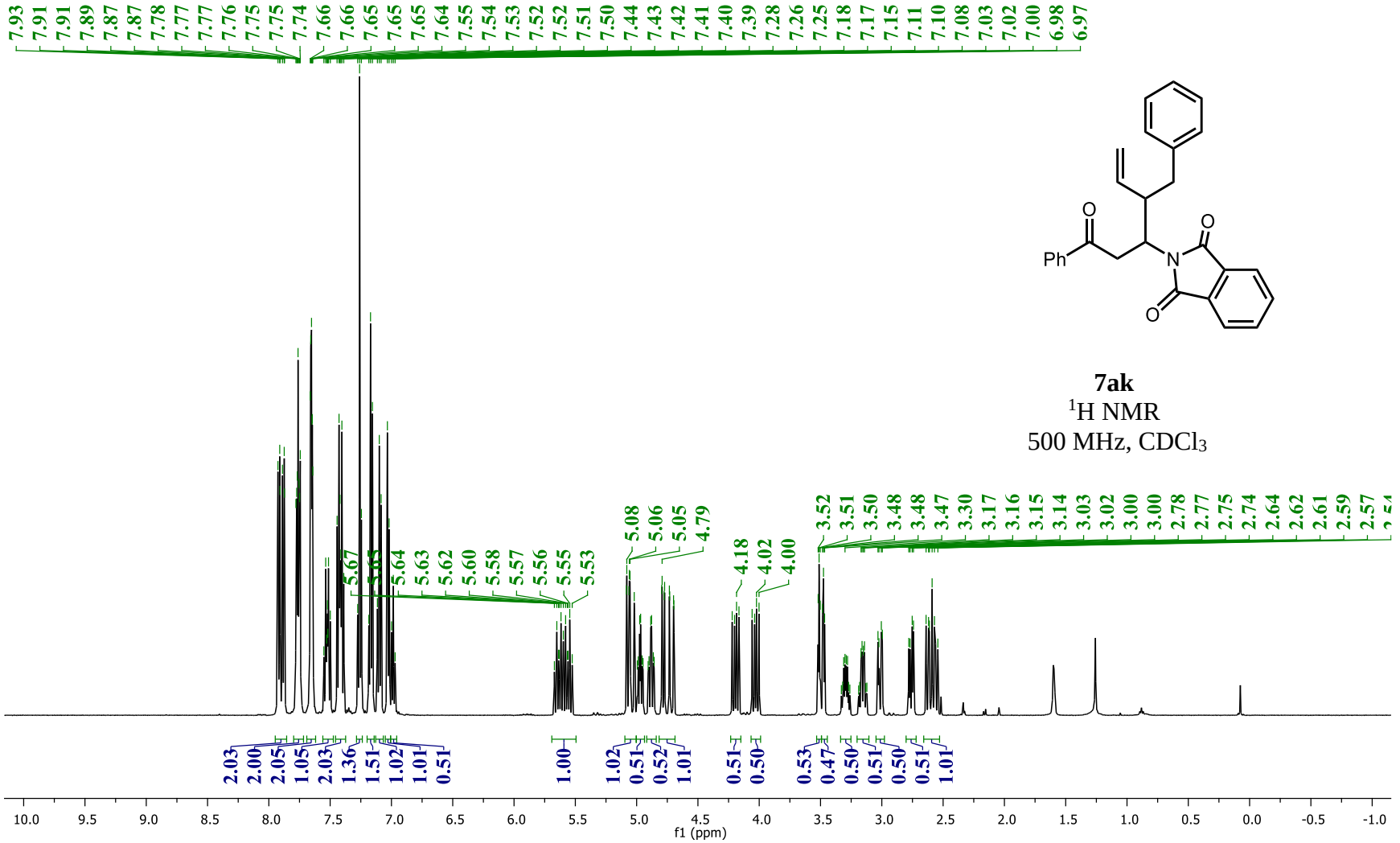


7aj

^{19}F NMR

471 MHz, $CDCl_3$

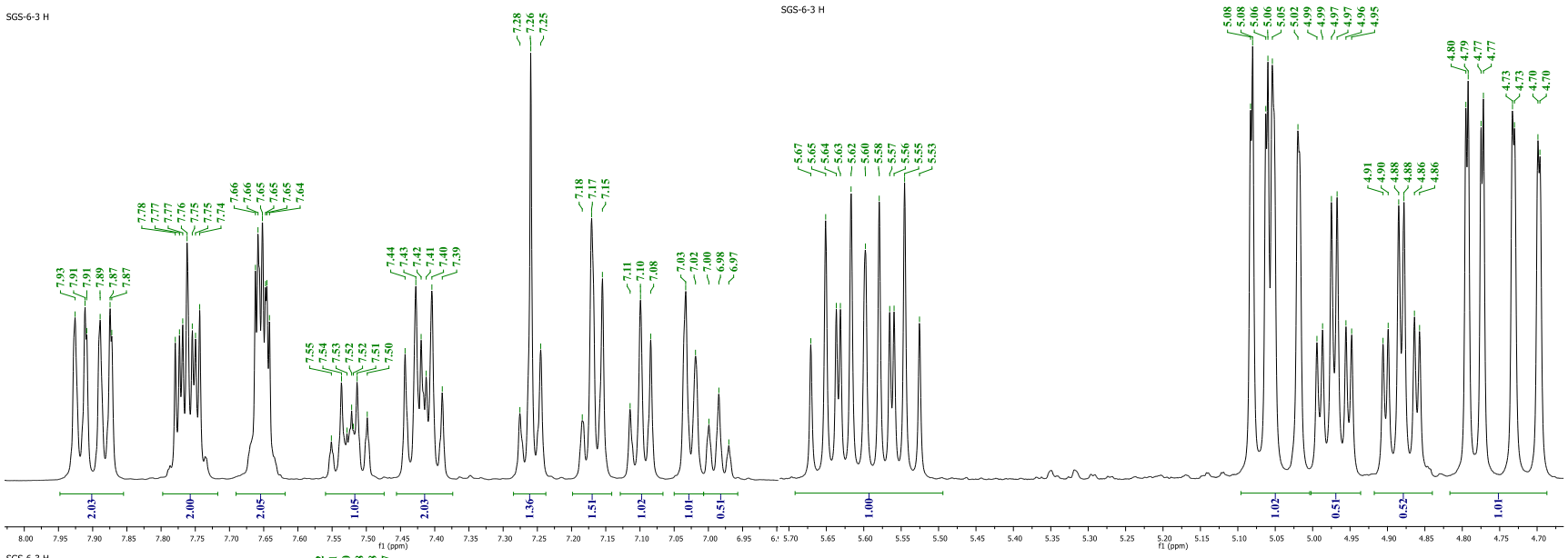
SGS-6-3 H

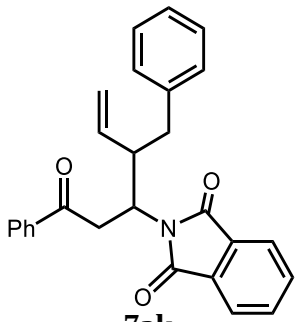


7ak

^{1}H NMR

500 MHz, $CDCl_3$



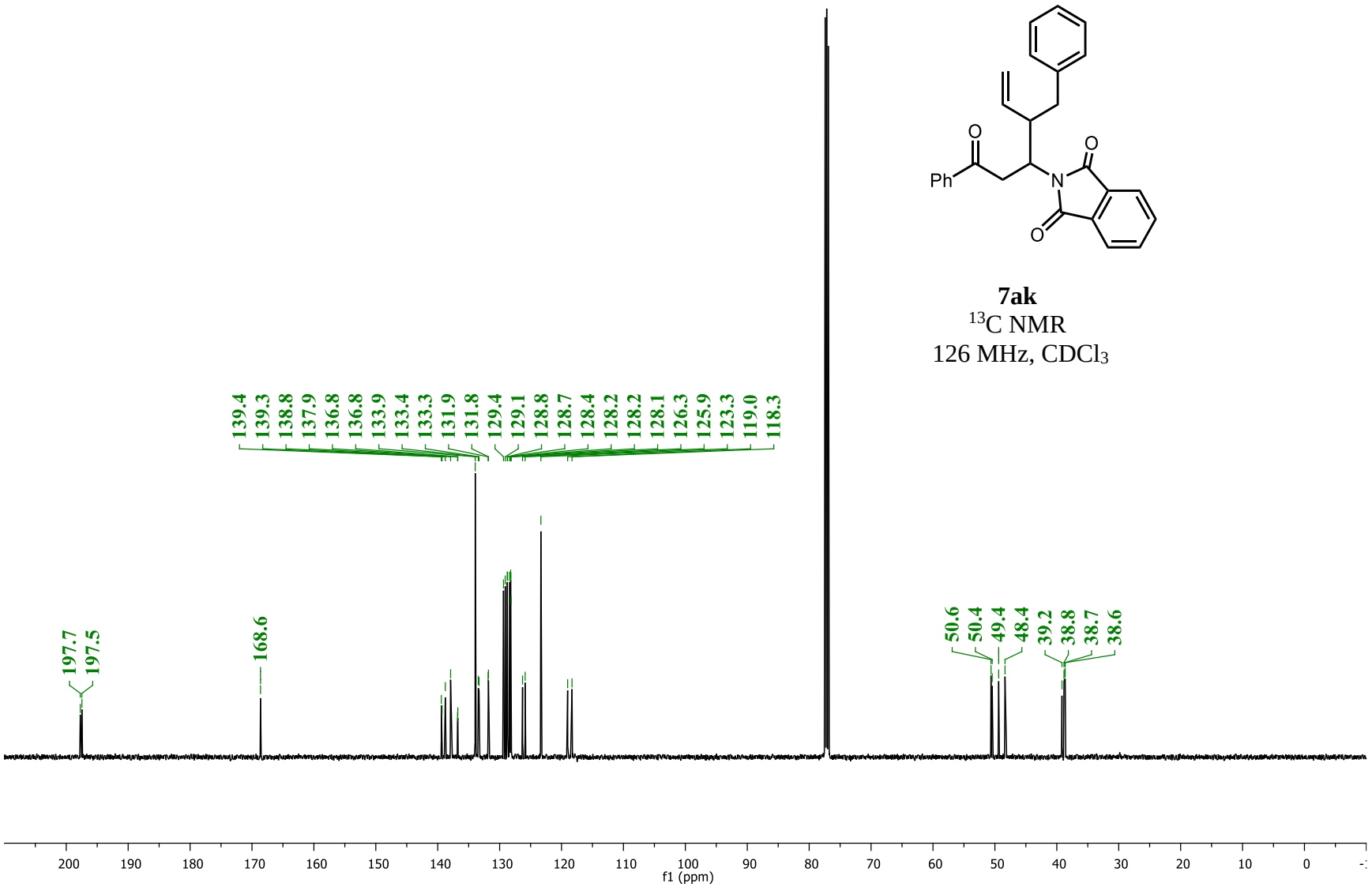


7ak

^{1}H NMR

500 MHz, $CDCl_3$

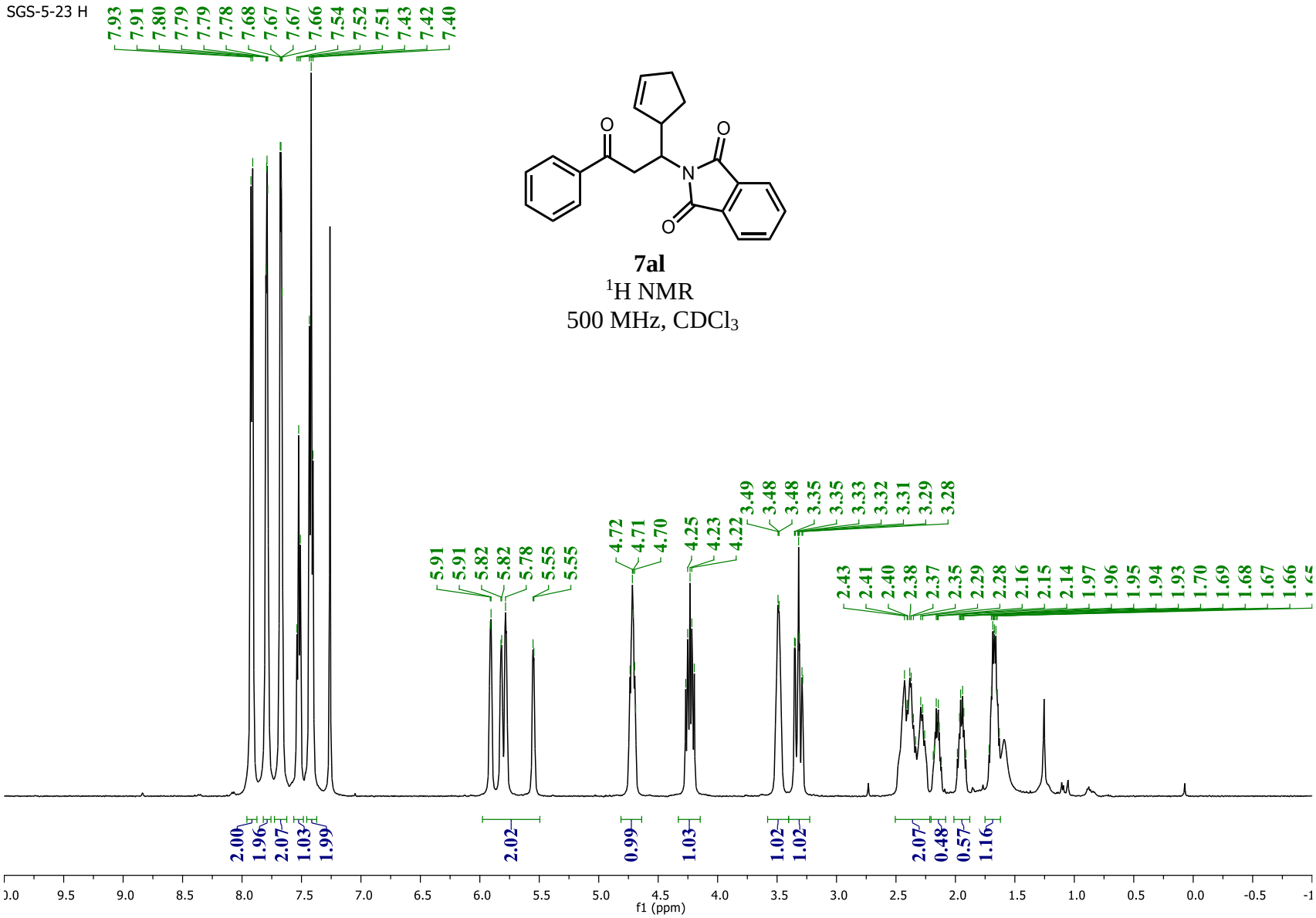
SGS-6-3 C



7ak

^{13}C NMR

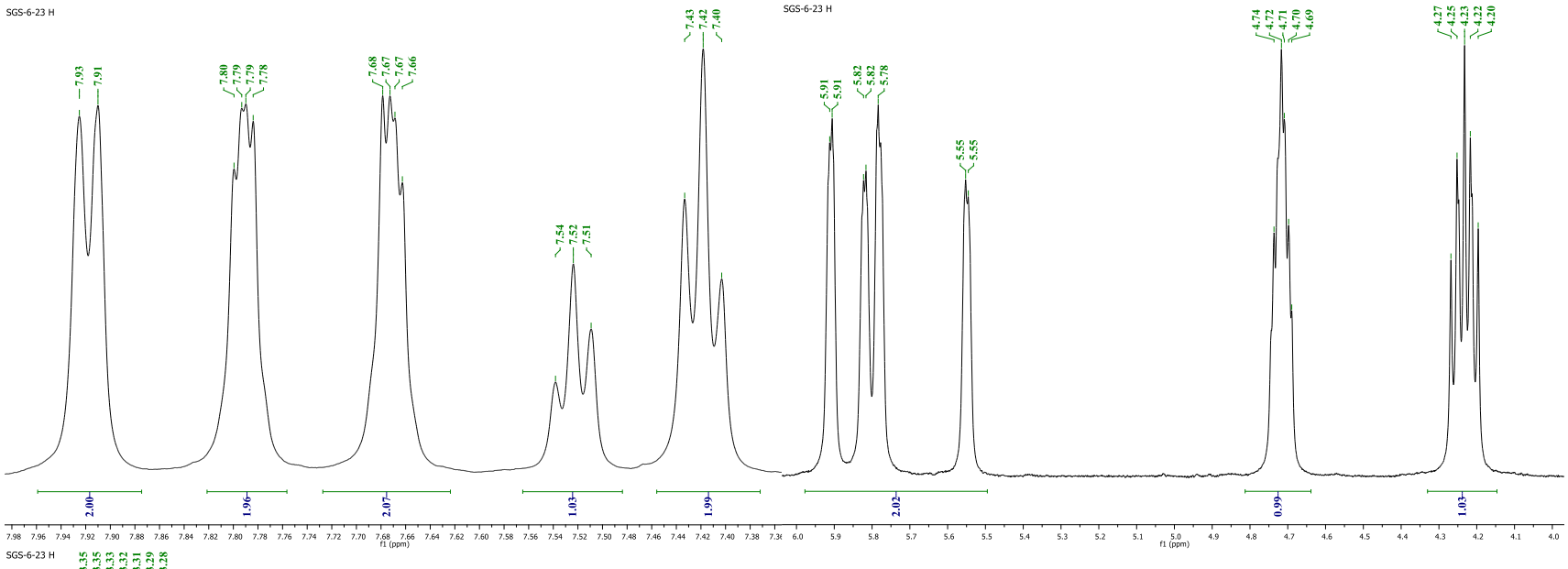
126 MHz, $CDCl_3$

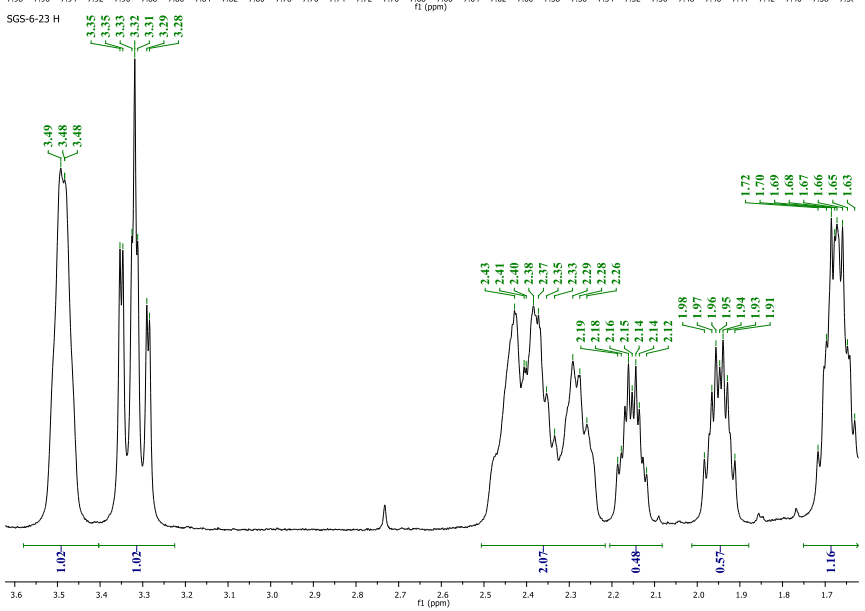


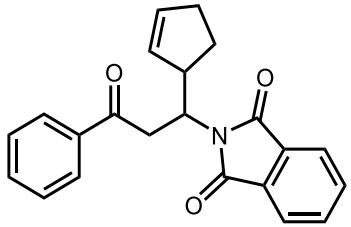
7al

^{1}H NMR

500 MHz, $CDCl_3$

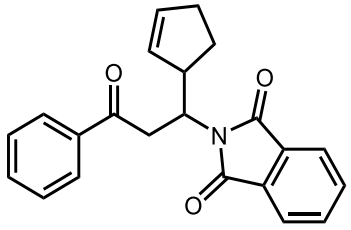






7al
^{1}H NMR
500 MHz, $CDCl_3$

SGS-5-23 C

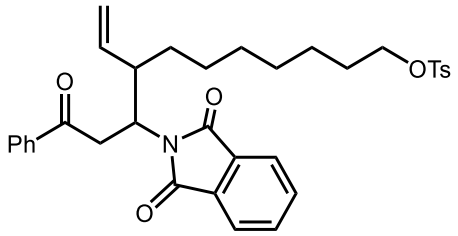


7al

^{13}C NMR

126 MHz, $CDCl_3$

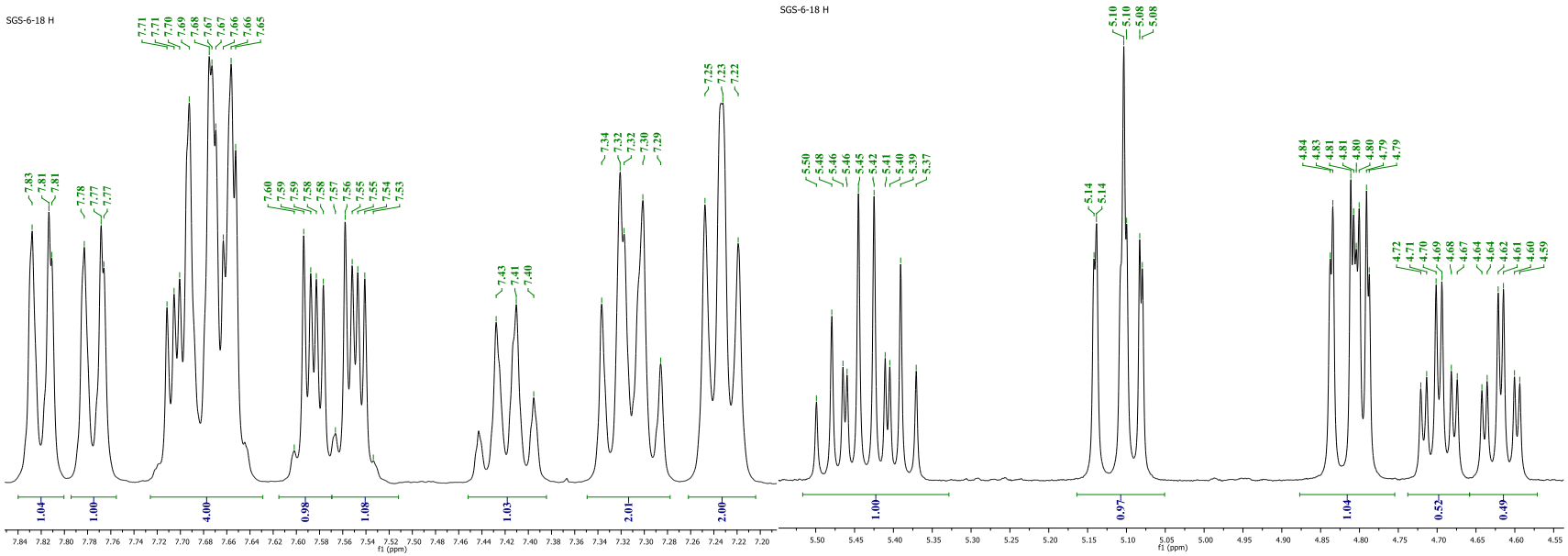
SGS-6-18 H

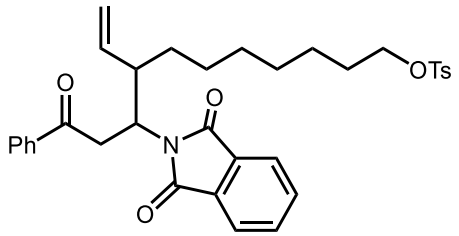


7am

^{1}H NMR

500 MHz, $CDCl_3$



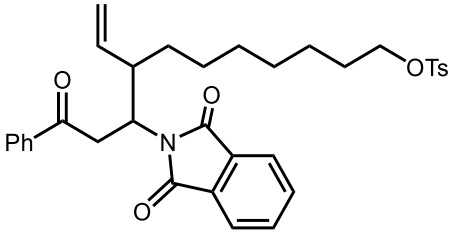


7am

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-18 C

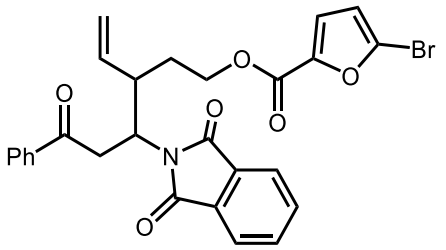


7am

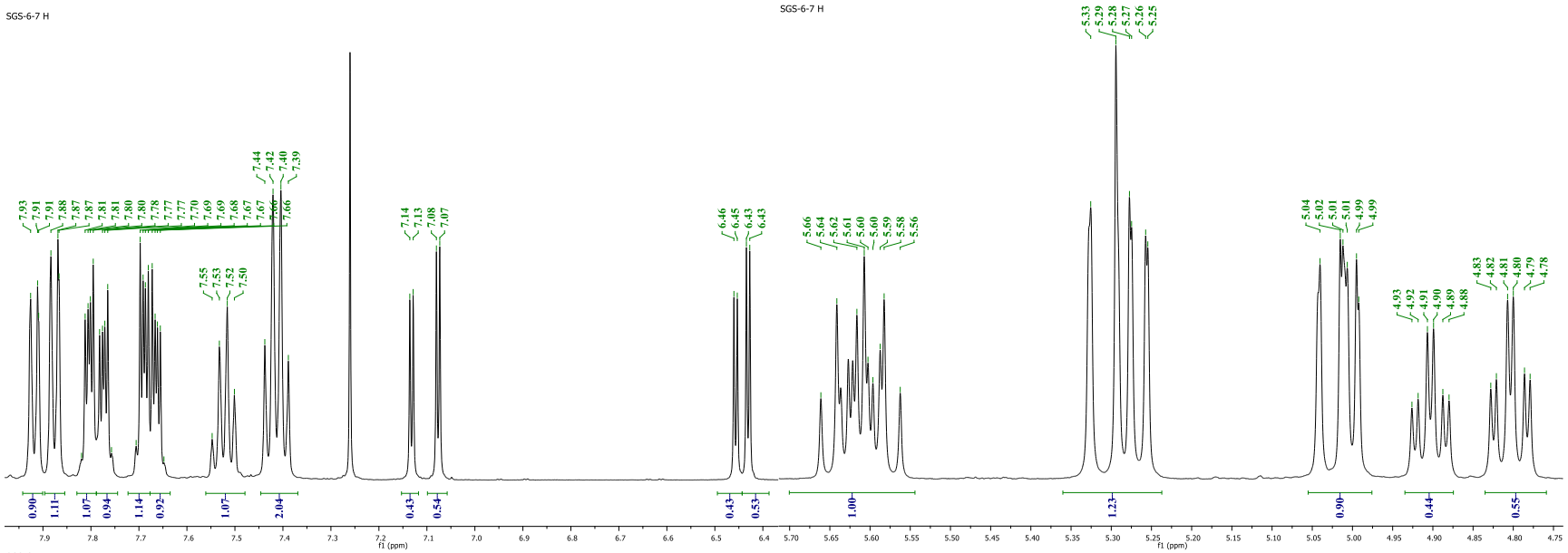
^{13}C NMR

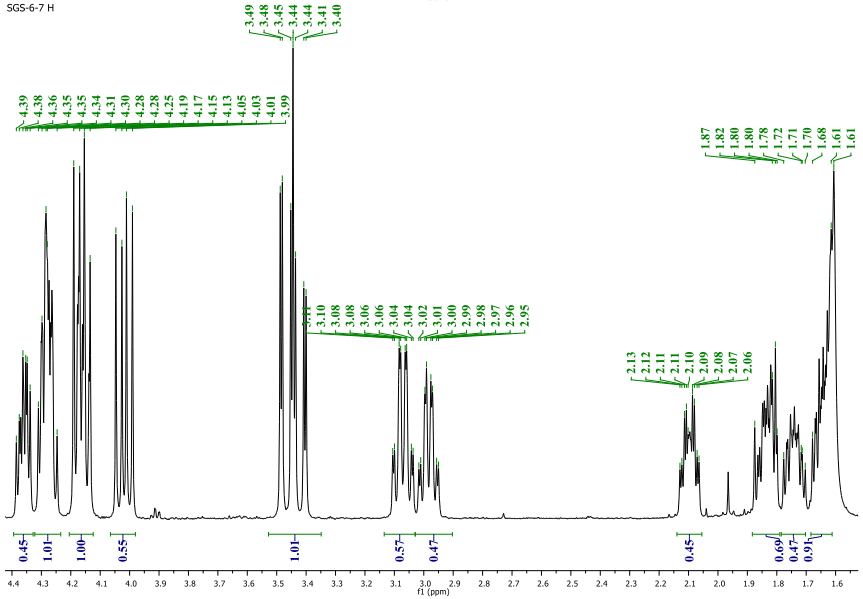
126 MHz, $CDCl_3$

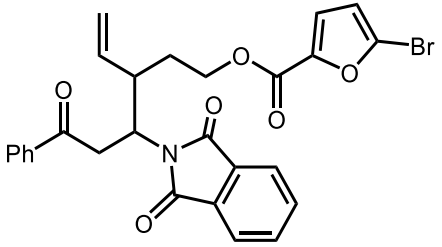
SGS-6-7 H



7an
^{1}H NMR
500 MHz, $CDCl_3$

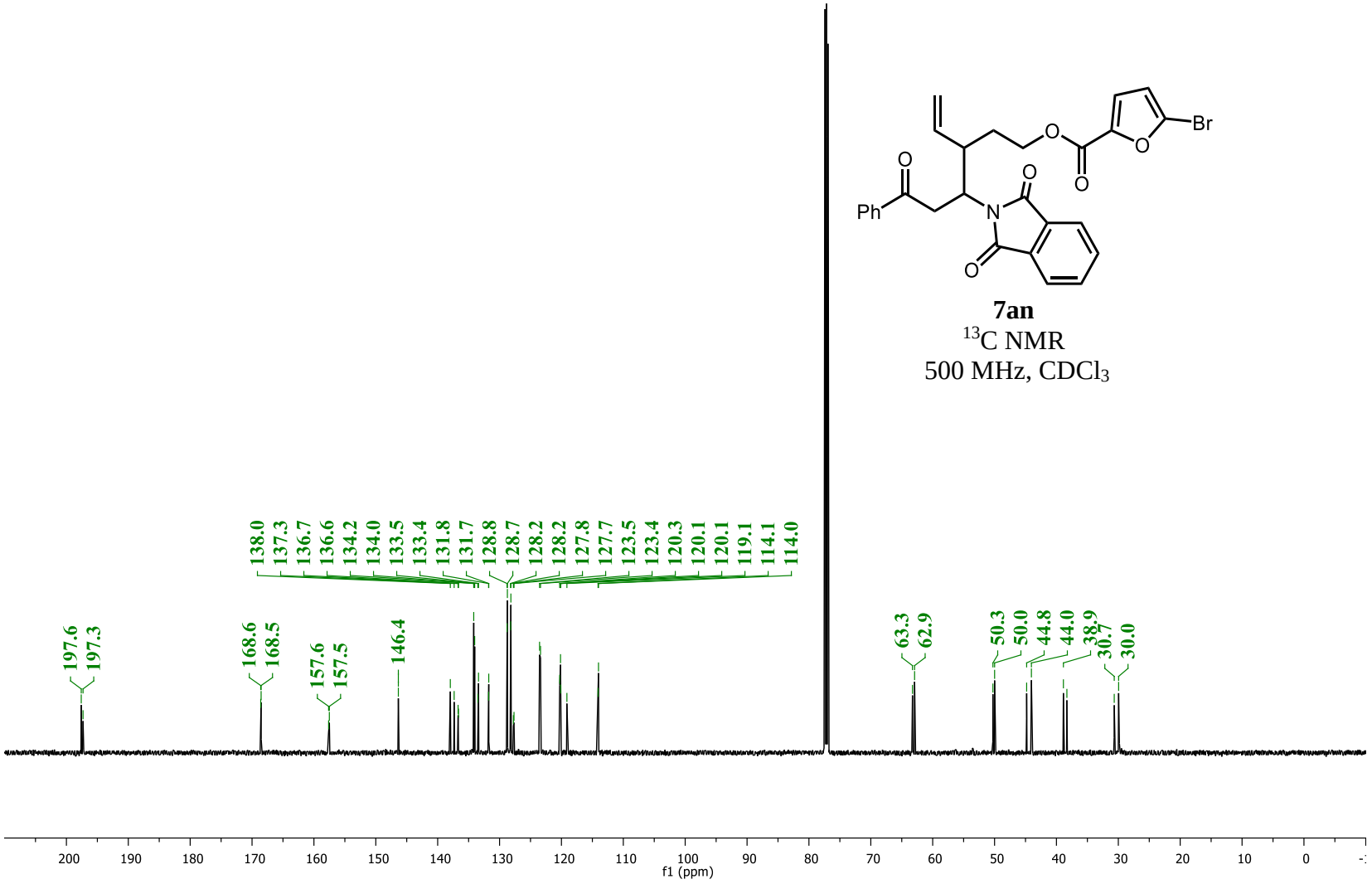






7an
^{1}H NMR
500 MHz, $CDCl_3$

SGS-6-7 C

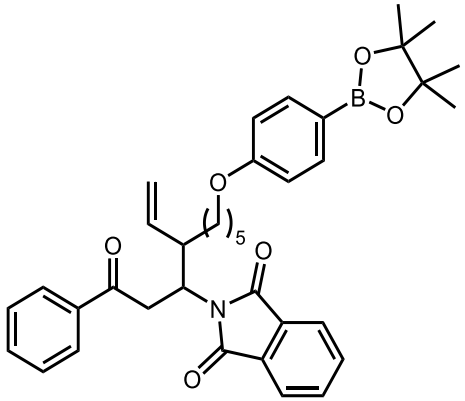


7an

^{13}C NMR

500 MHz, $CDCl_3$

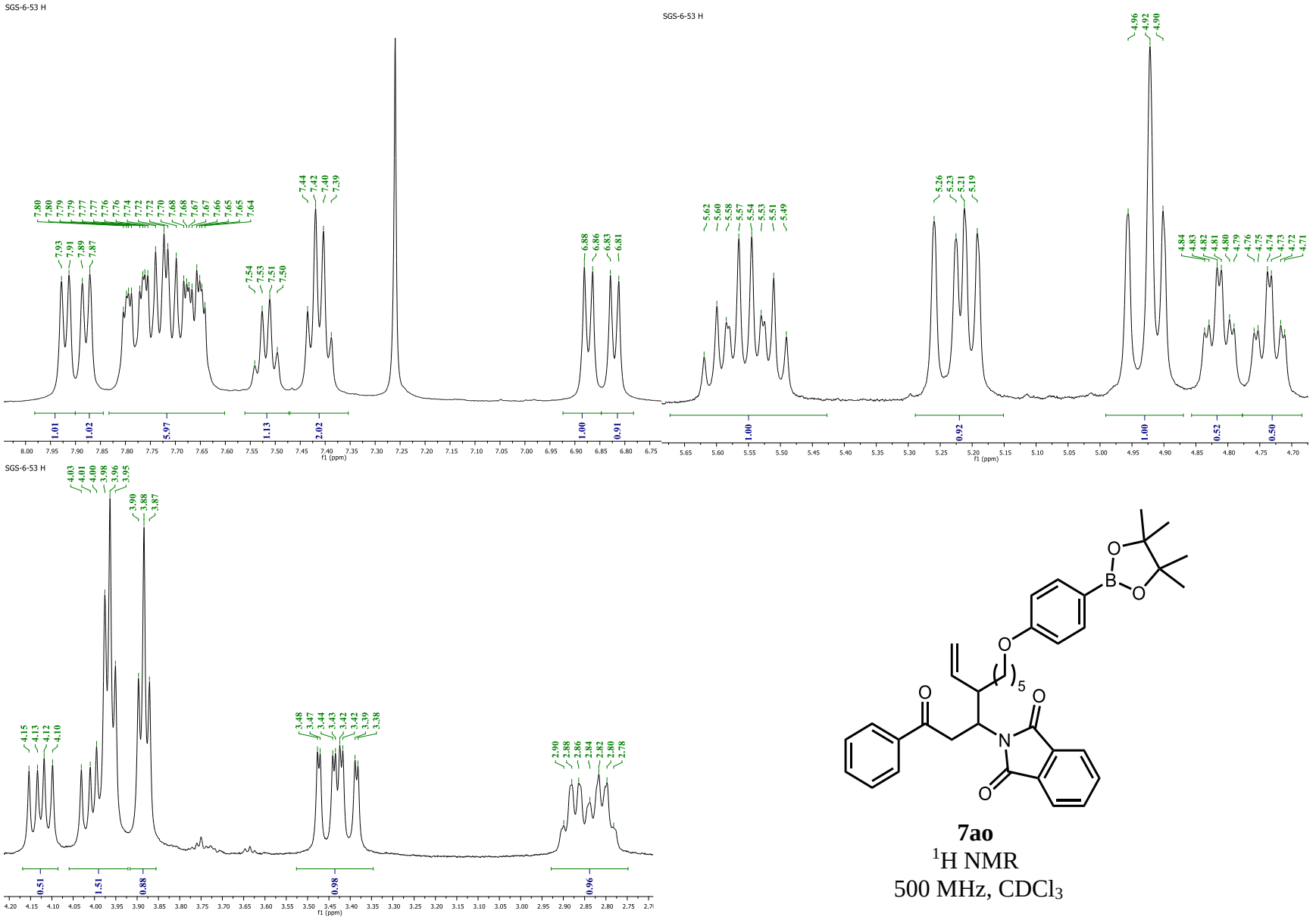
SGS-6-53 H



7ao

^{1}H NMR

500 MHz, $CDCl_3$

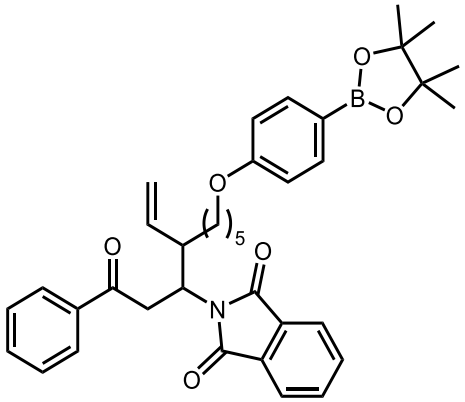


7ao

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-53 C

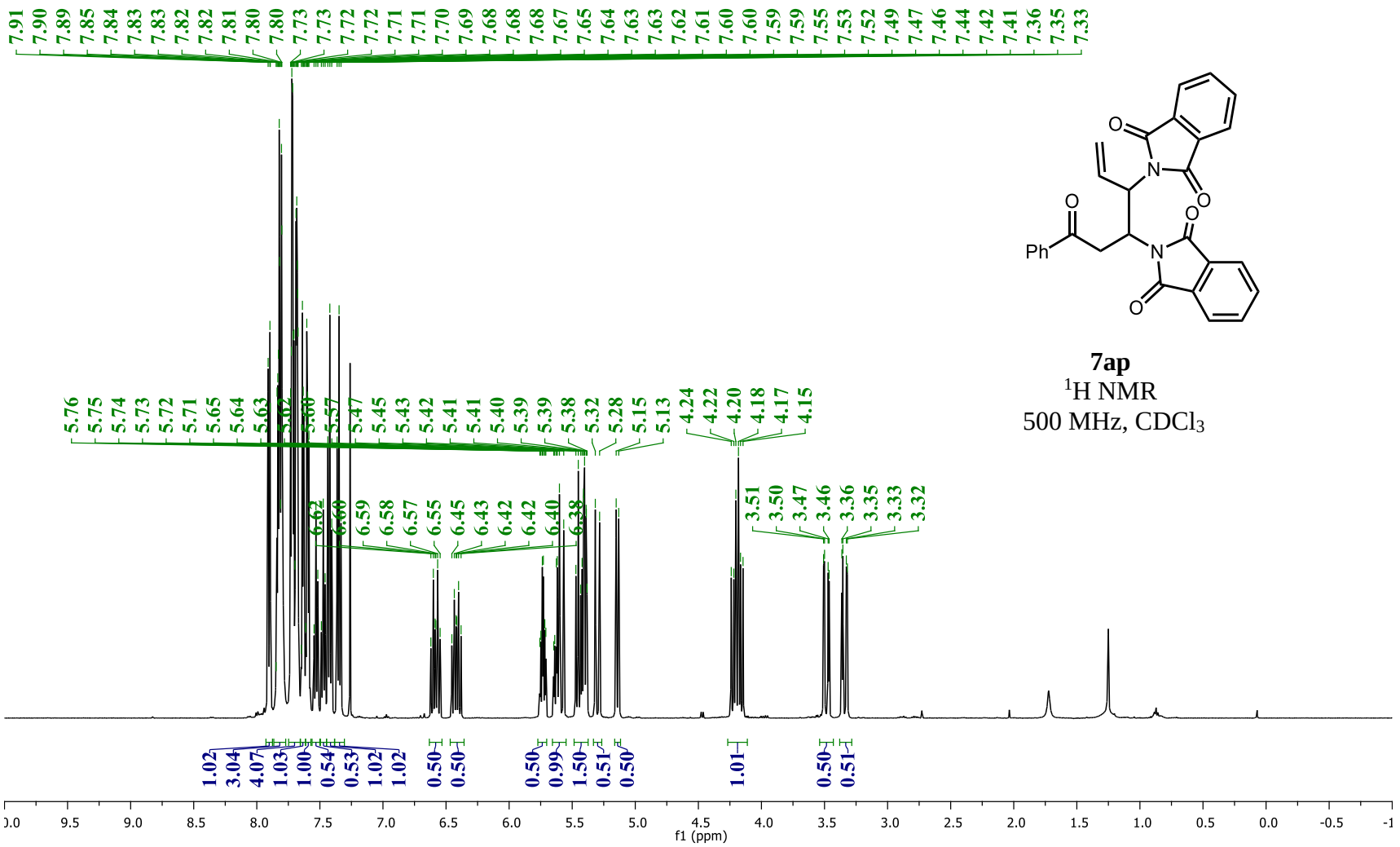


7ao

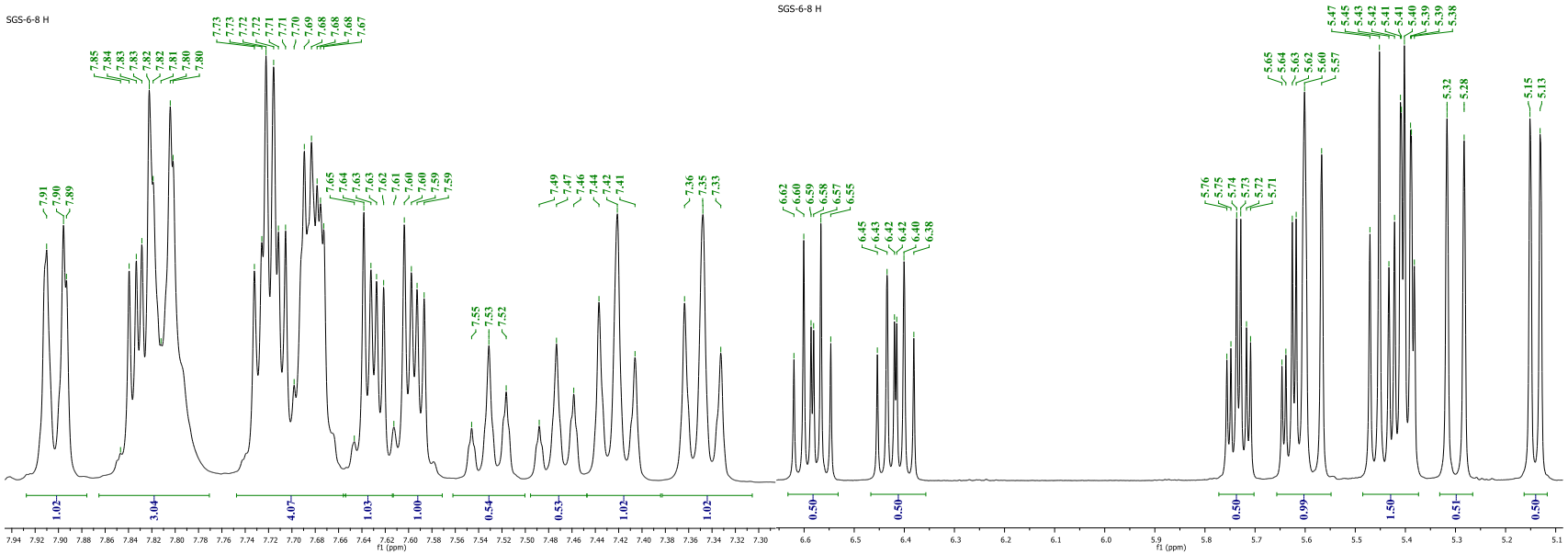
^{13}C NMR

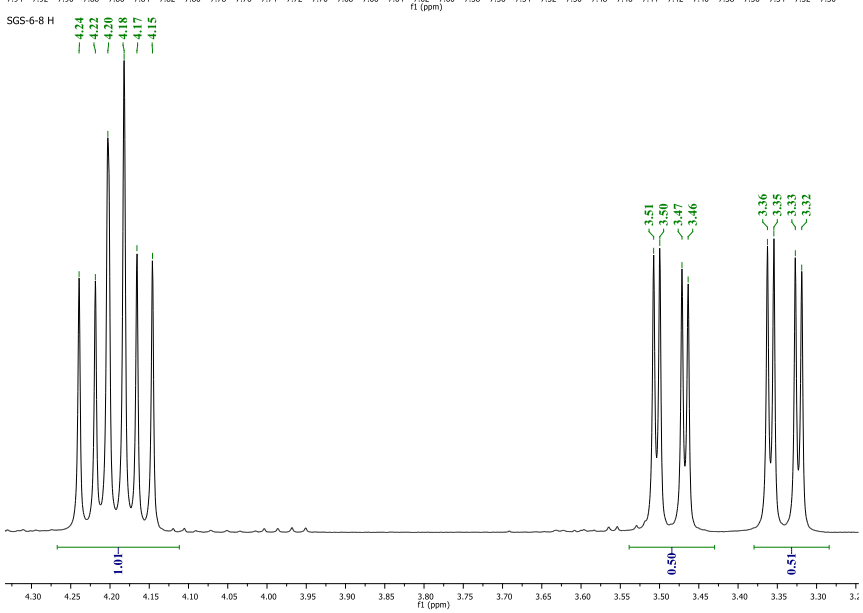
126 MHz, $CDCl_3$

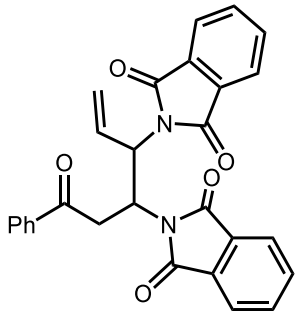
SGS-6-8 H



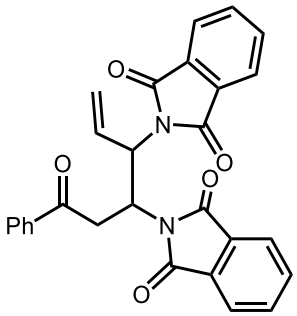
7ap
^{1}H NMR
500 MHz, $CDCl_3$



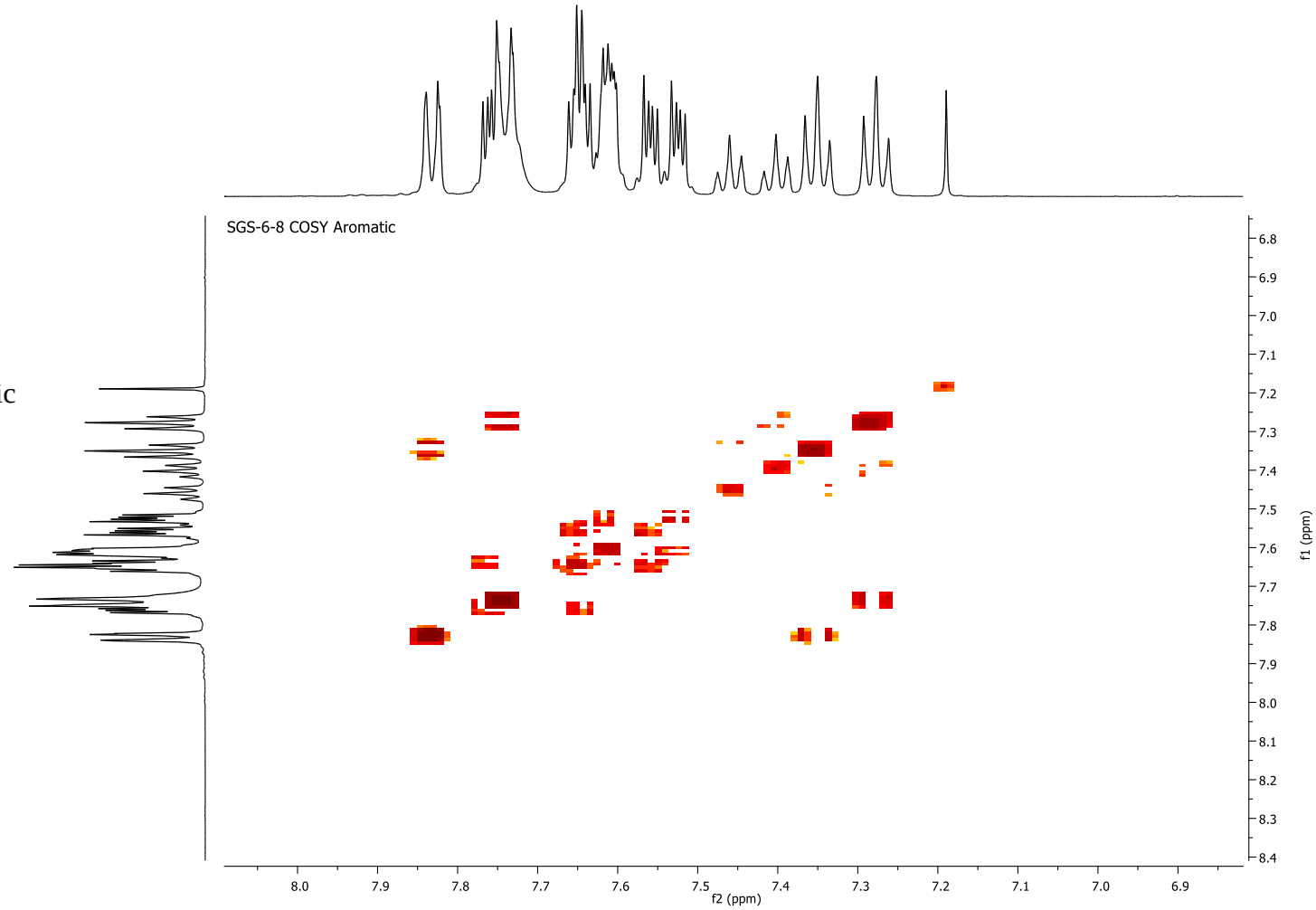


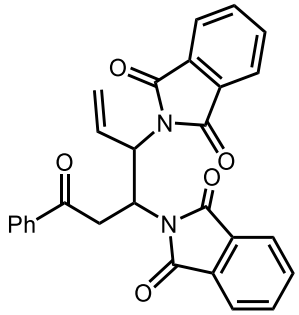


7ap
^{1}H NMR
500 MHz, $CDCl_3$

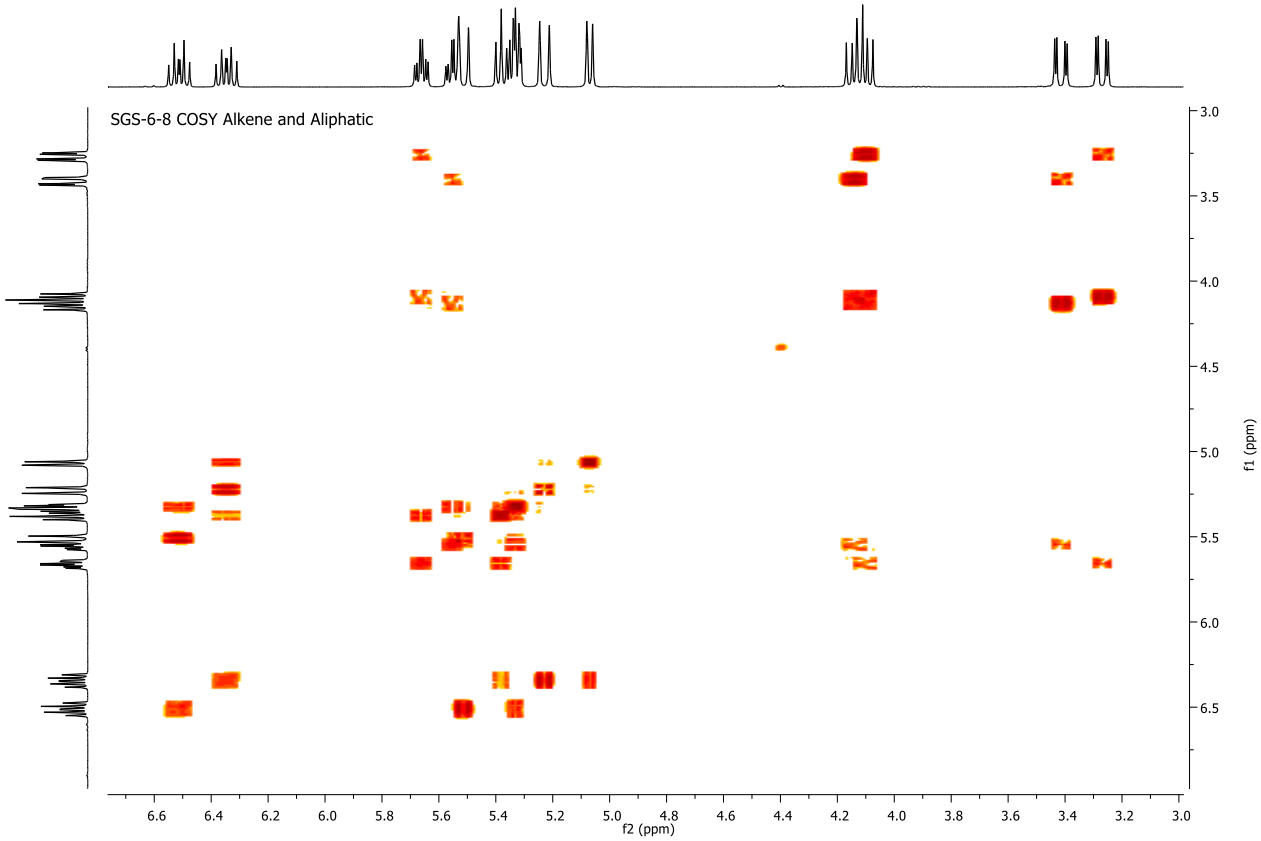


7ap
COSY NMR - aromatic
500 MHz, $CDCl_3$

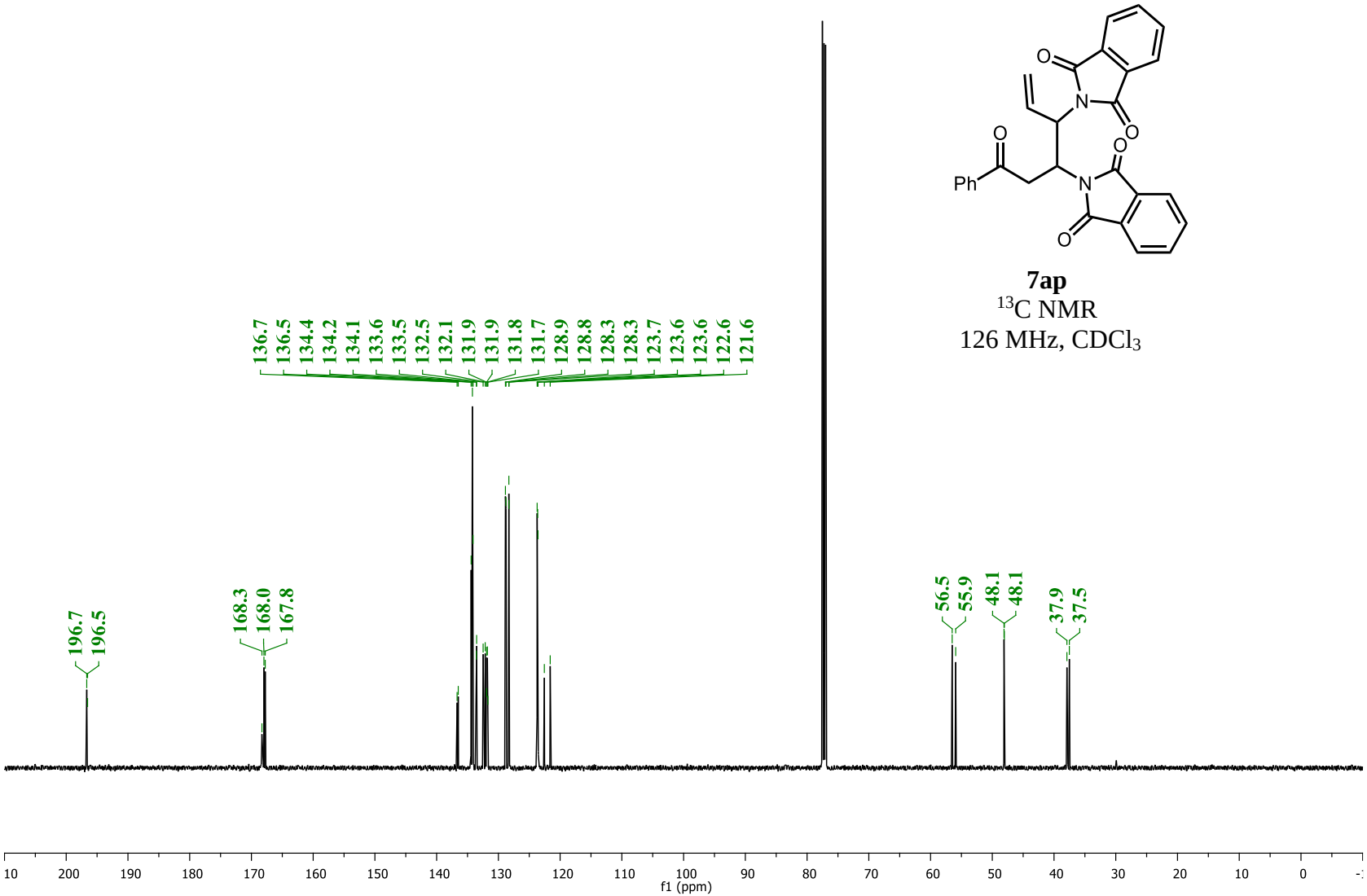




7ap
COSY NMR - aliphatic
500 MHz, $CDCl_3$



SGS-6-8 C

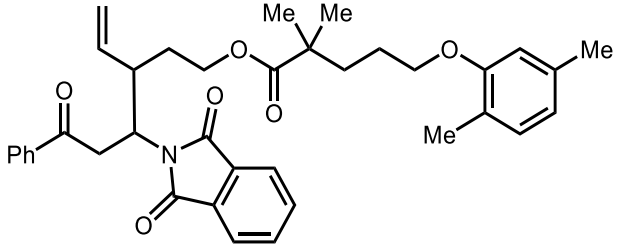


7ap

^{13}C NMR

126 MHz, $CDCl_3$

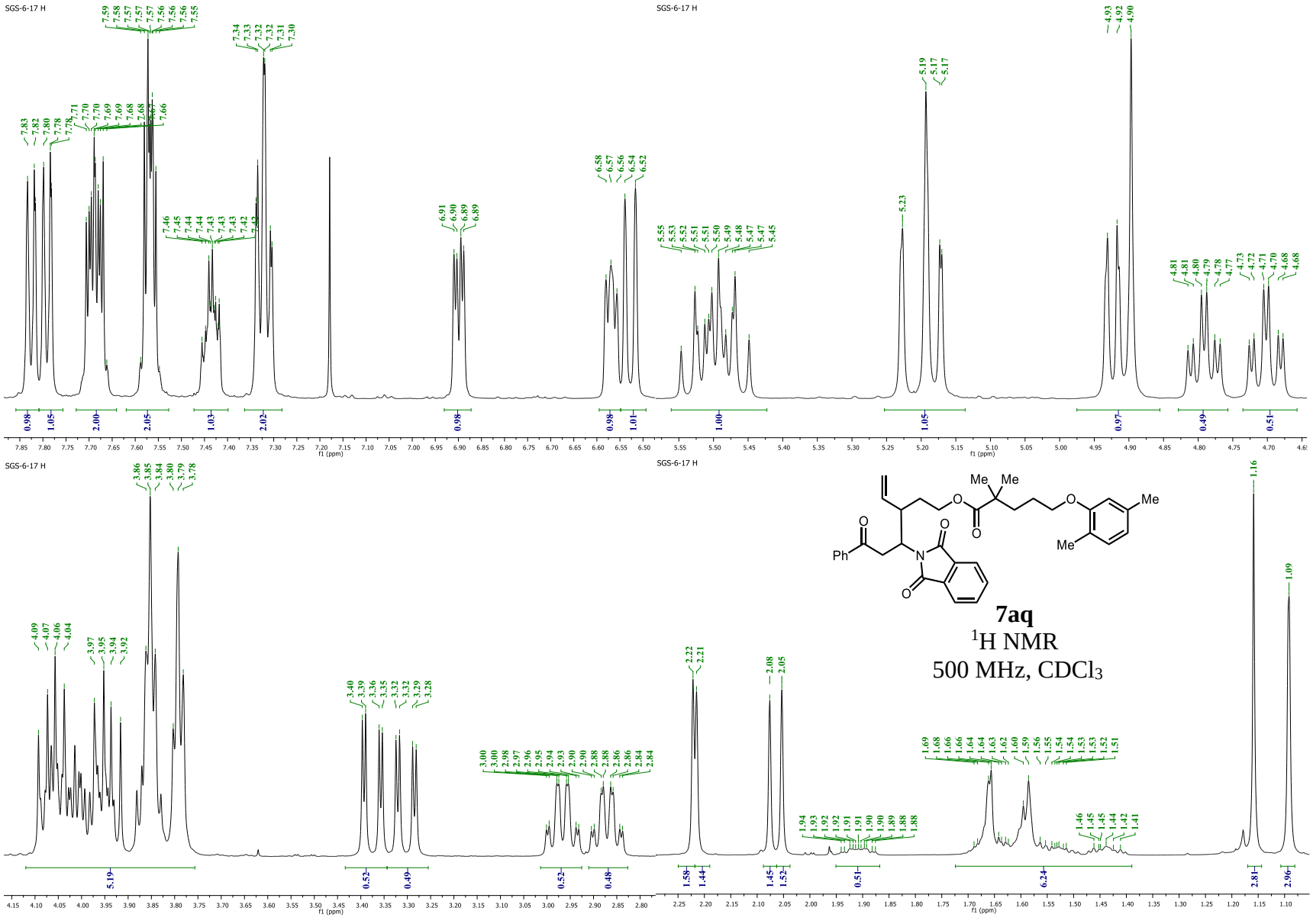
SGS-6-17 H

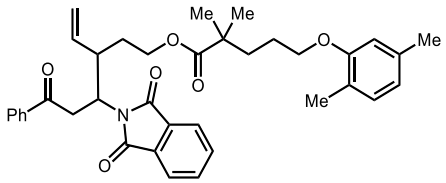


7aq

^{1}H NMR

500 MHz, $CDCl_3$

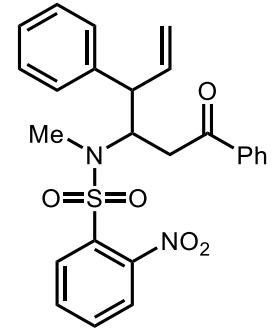

SGS-6-17 H
SGS-6-17 H
SGS-6-17 H
SGS-6-17 H
Me
Me
Me
Me
O
O
O
O
O
O
N
Ph
7aq
1H NMR
500 MHz, CDCl3



7aq
COSY NMR - aliphatic
500 MHz, $CDCl_3$

7.4 NMR spectra: Sulfonamide Scope

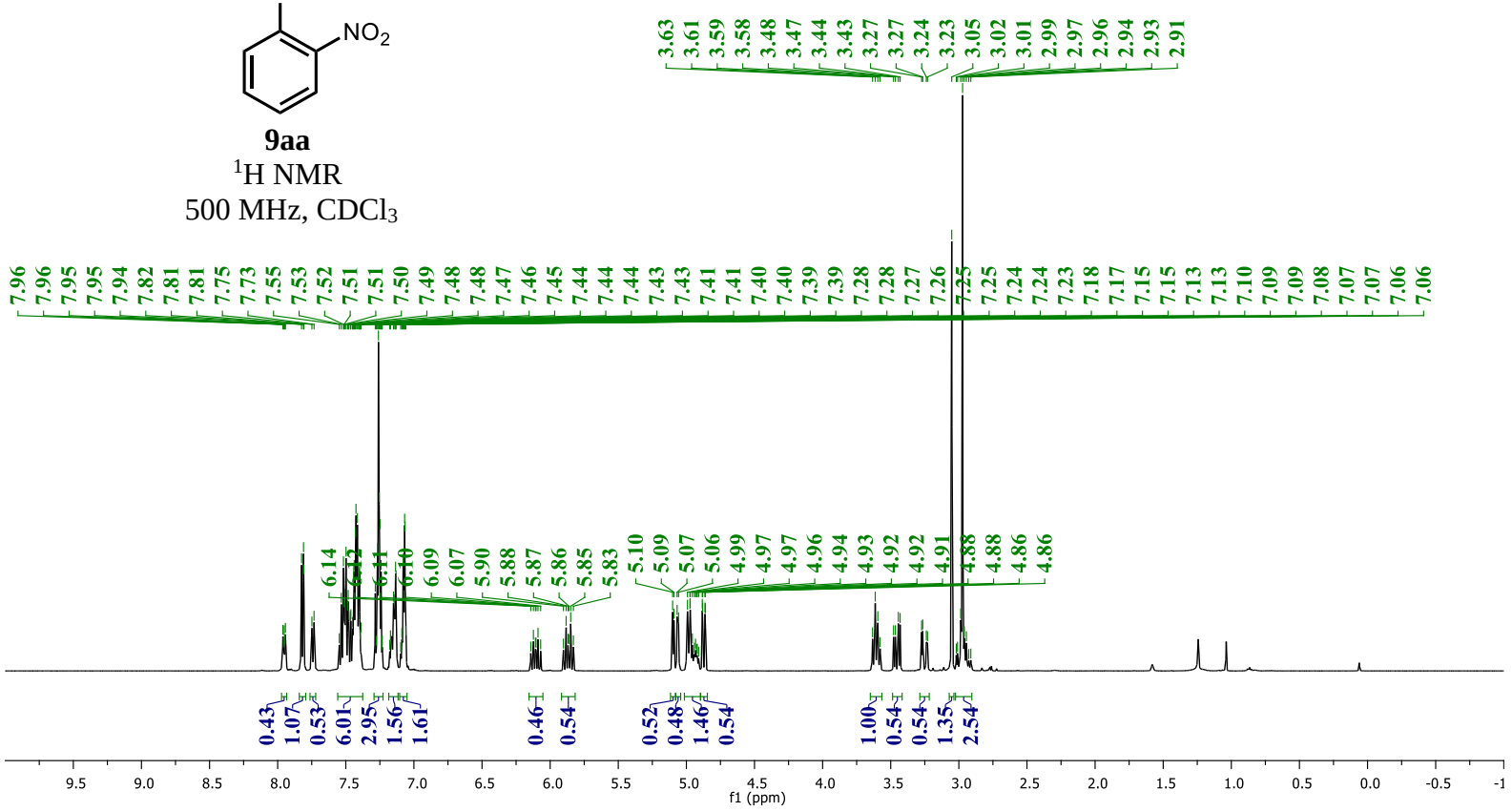
SGS-5-184 H

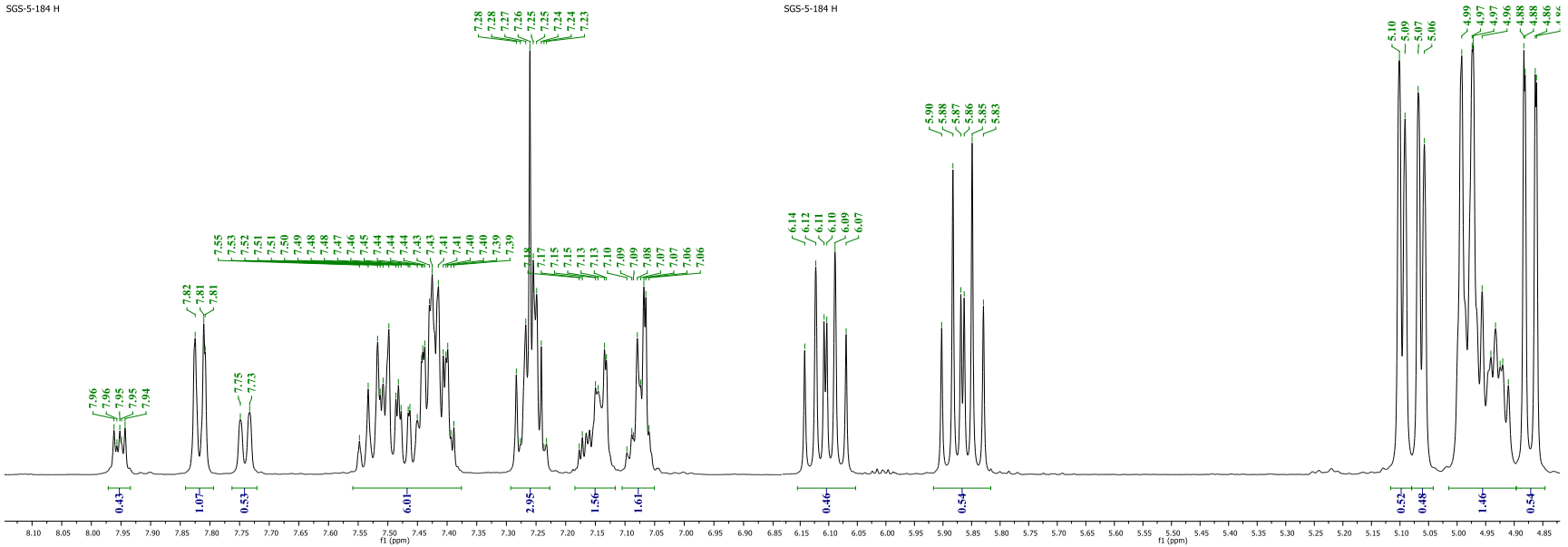


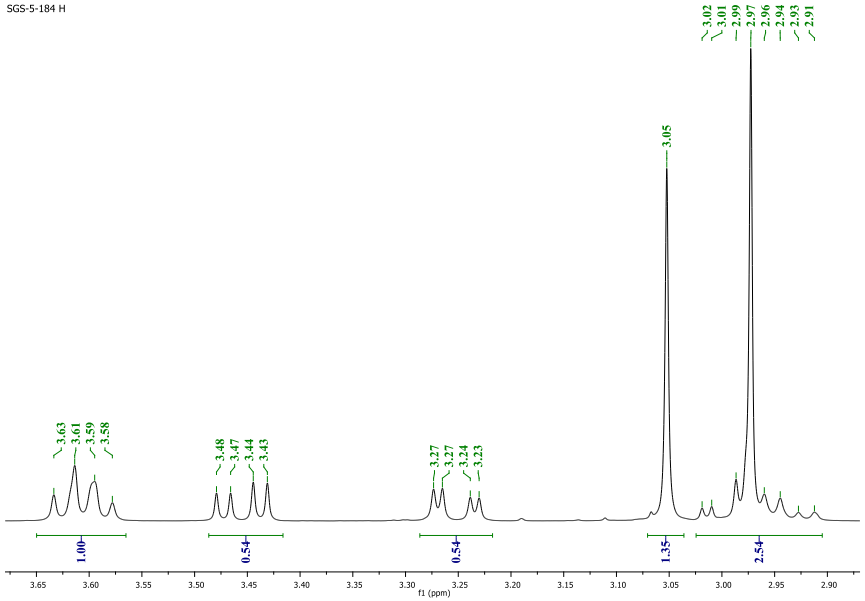
9aa

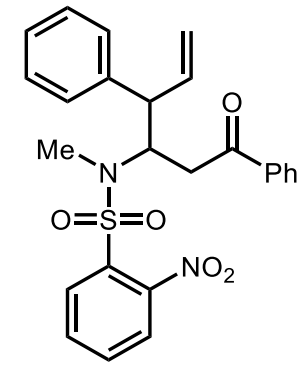
^{1}H NMR

500 MHz, $CDCl_3$



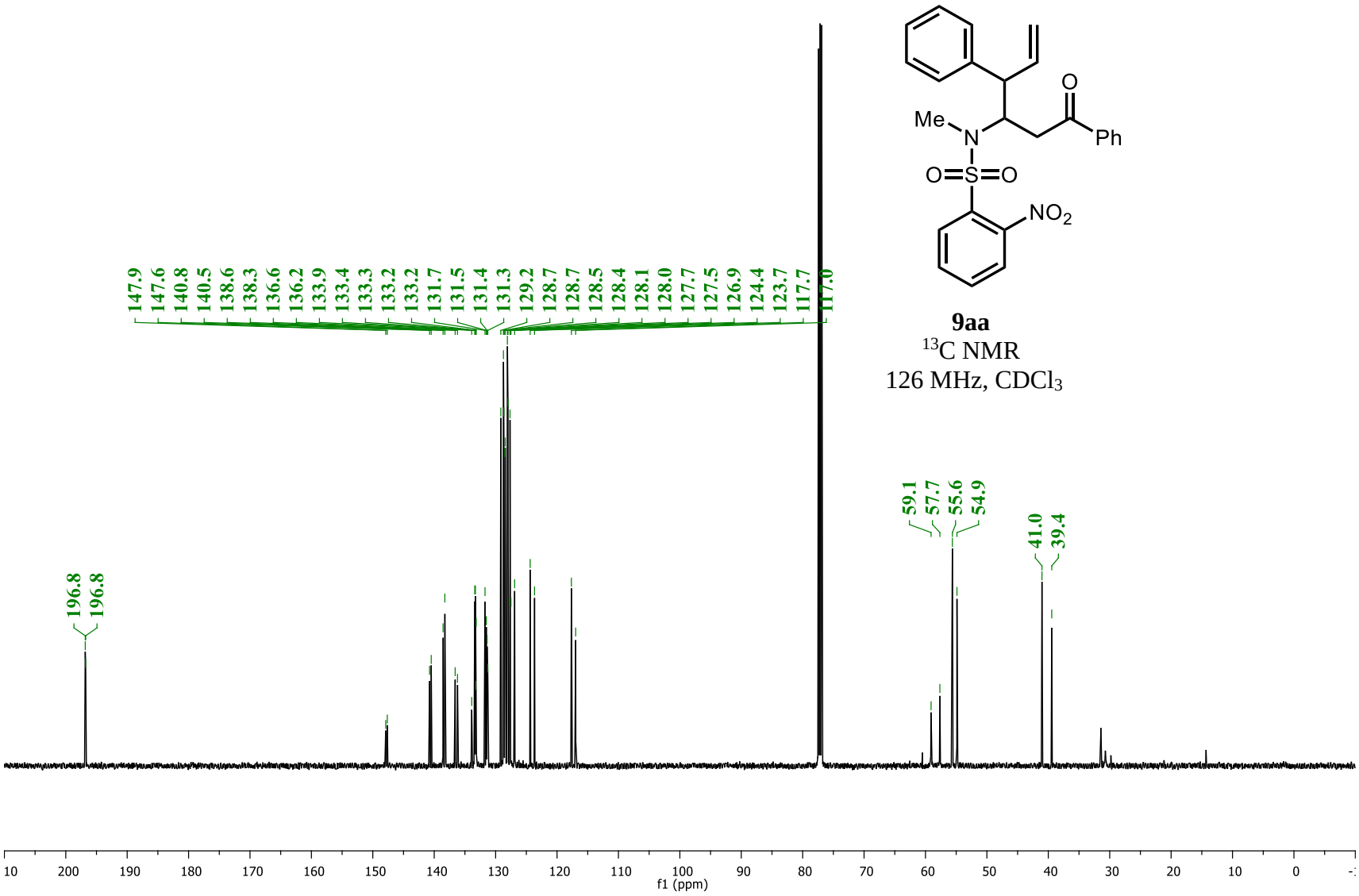






9aa
^{1}H NMR
500 MHz, $CDCl_3$

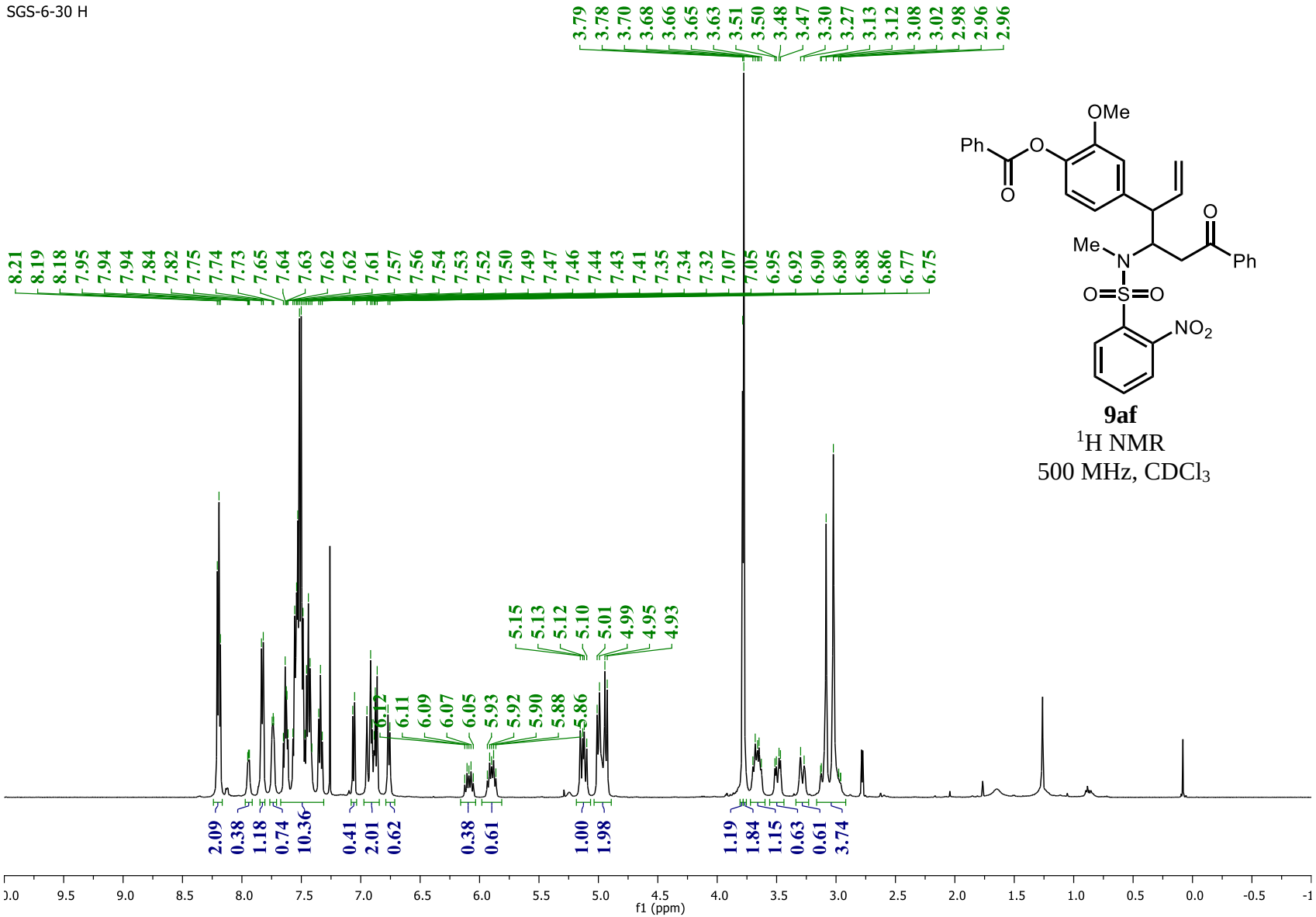
SGS-5-184 C



9aa

^{13}C NMR

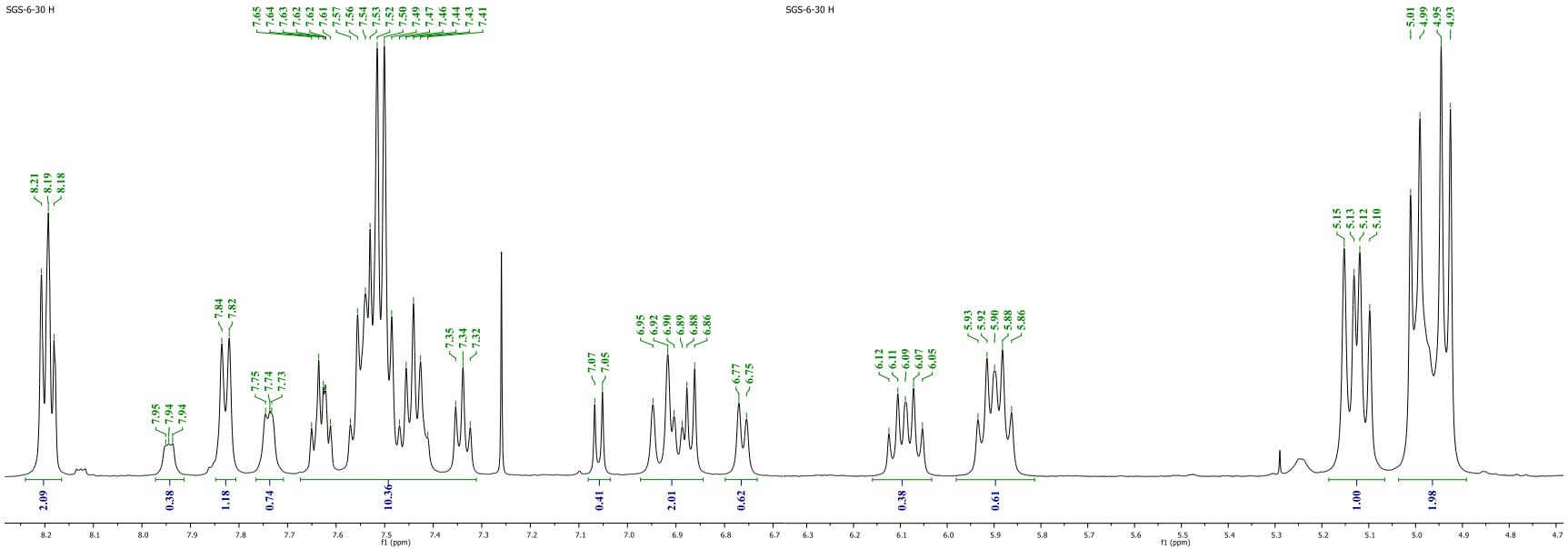
126 MHz, $CDCl_3$

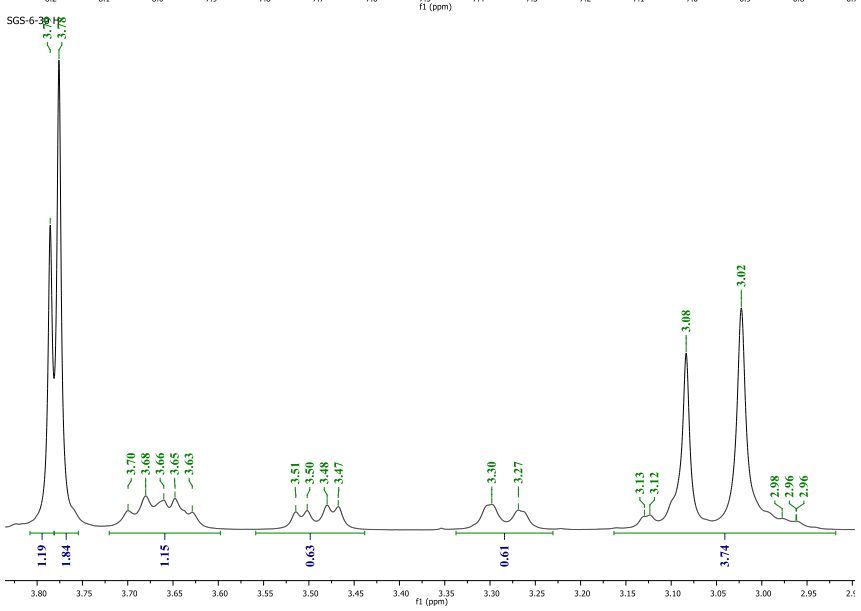


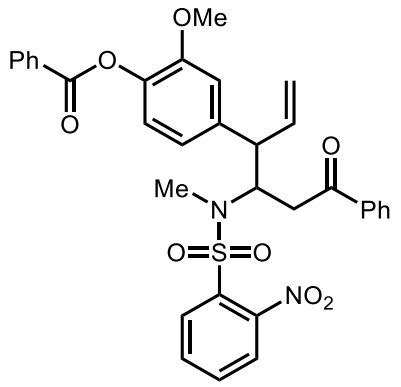
9af

^{1}H NMR

500 MHz, $CDCl_3$





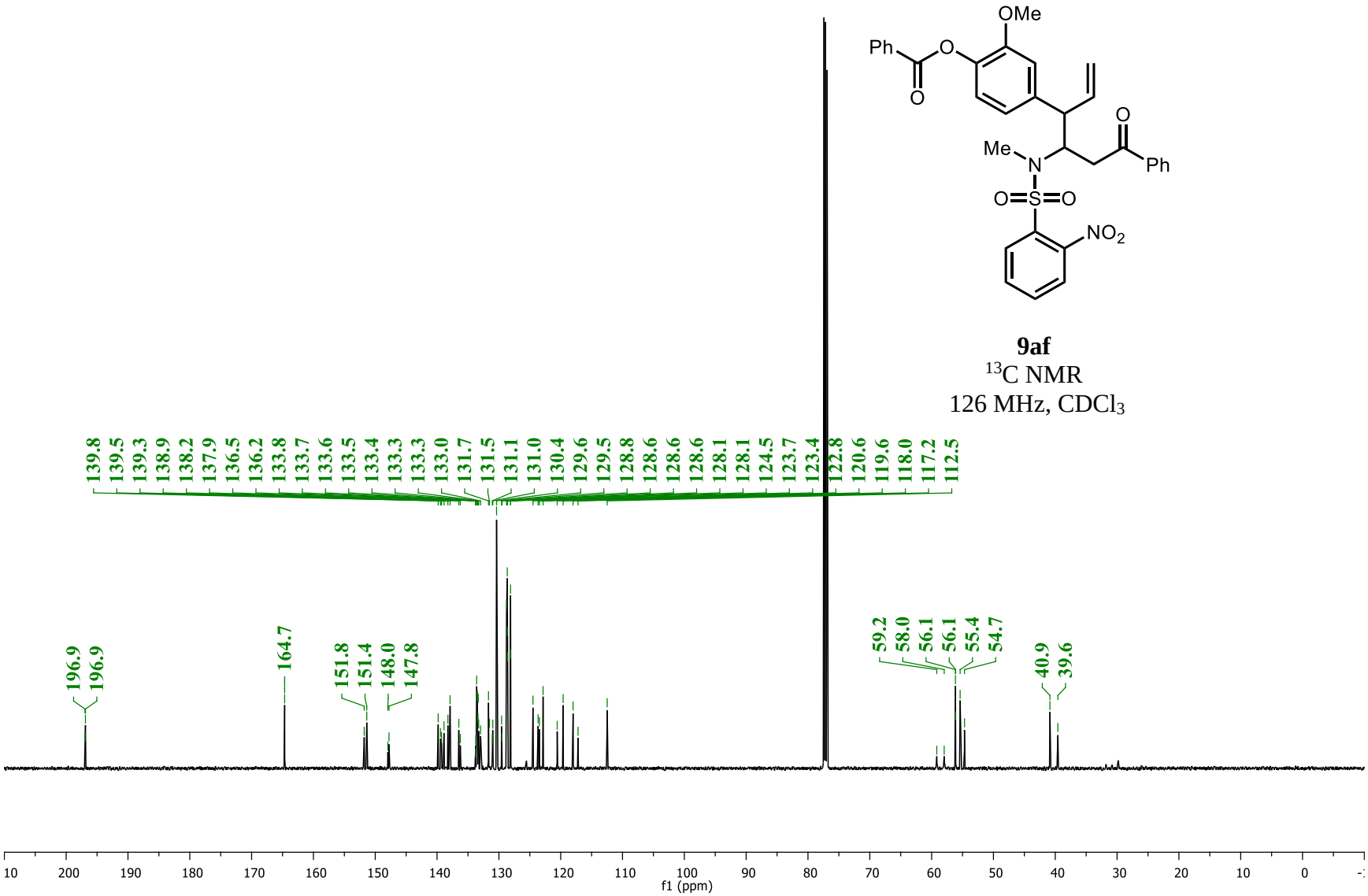


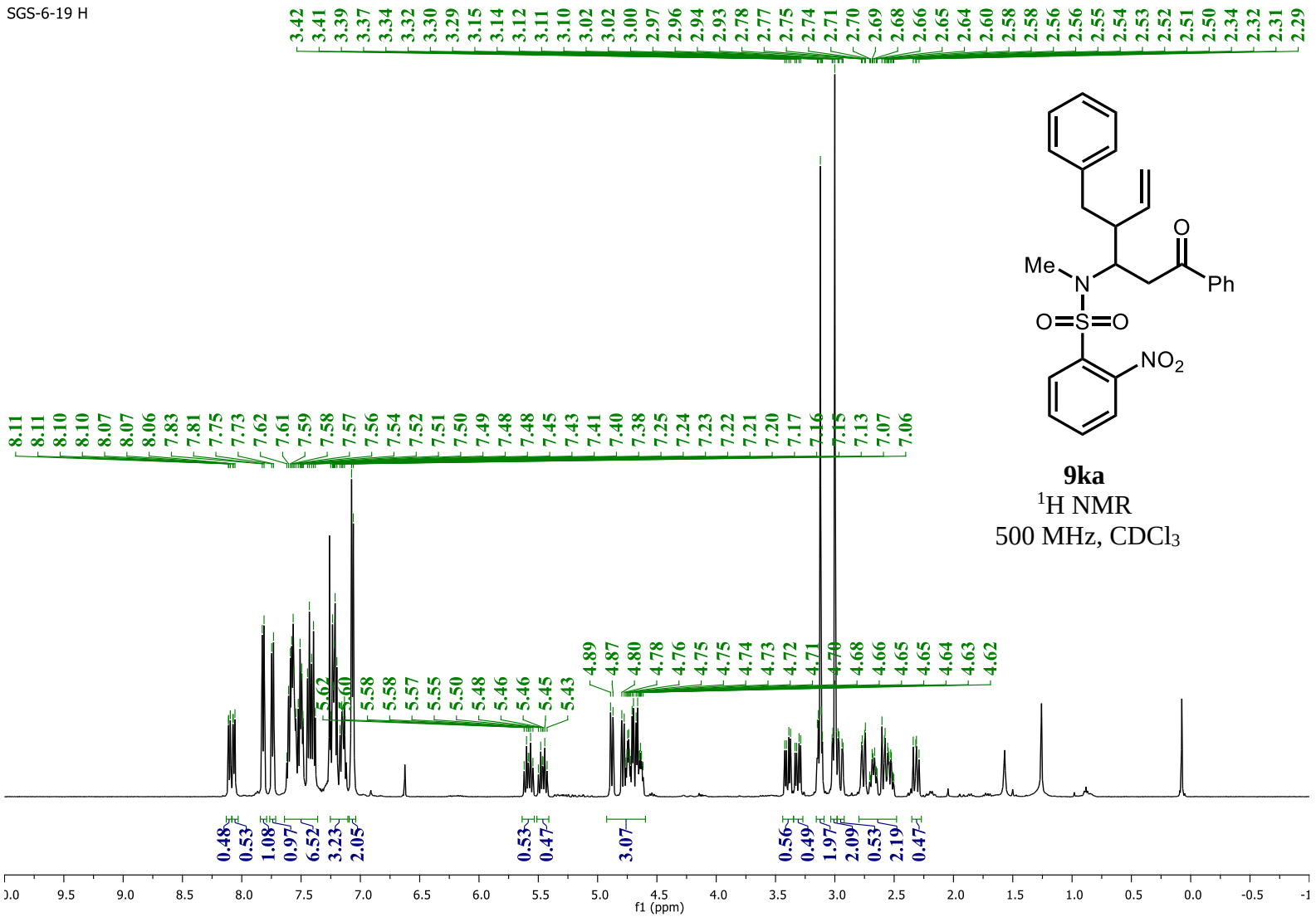
9af

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-30 C


OMe
Ph
O
O
O
Me
N
Ph
O=S=O
NO2
9af
13C NMR
126 MHz, CDCl3
139.8
139.5
139.3
138.9
138.2
137.9
136.5
136.2
133.8
133.7
133.6
133.5
133.4
133.3
133.3
133.0
131.7
131.5
131.1
131.0
130.4
129.6
129.5
128.8
128.6
128.6
128.6
128.1
128.1
124.5
123.7
123.4
122.8
120.6
119.6
118.0
117.2
112.5
196.9
196.9
164.7
151.8
151.4
148.0
147.8
59.2
58.0
56.1
56.1
55.4
54.7
40.9
39.6
f1 (ppm)

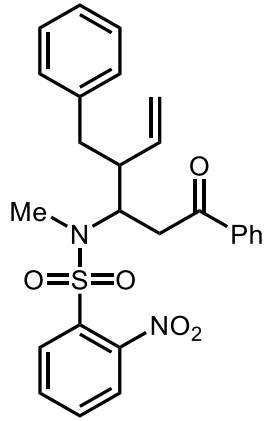


9ka

^{1}H NMR

500 MHz, $CDCl_3$

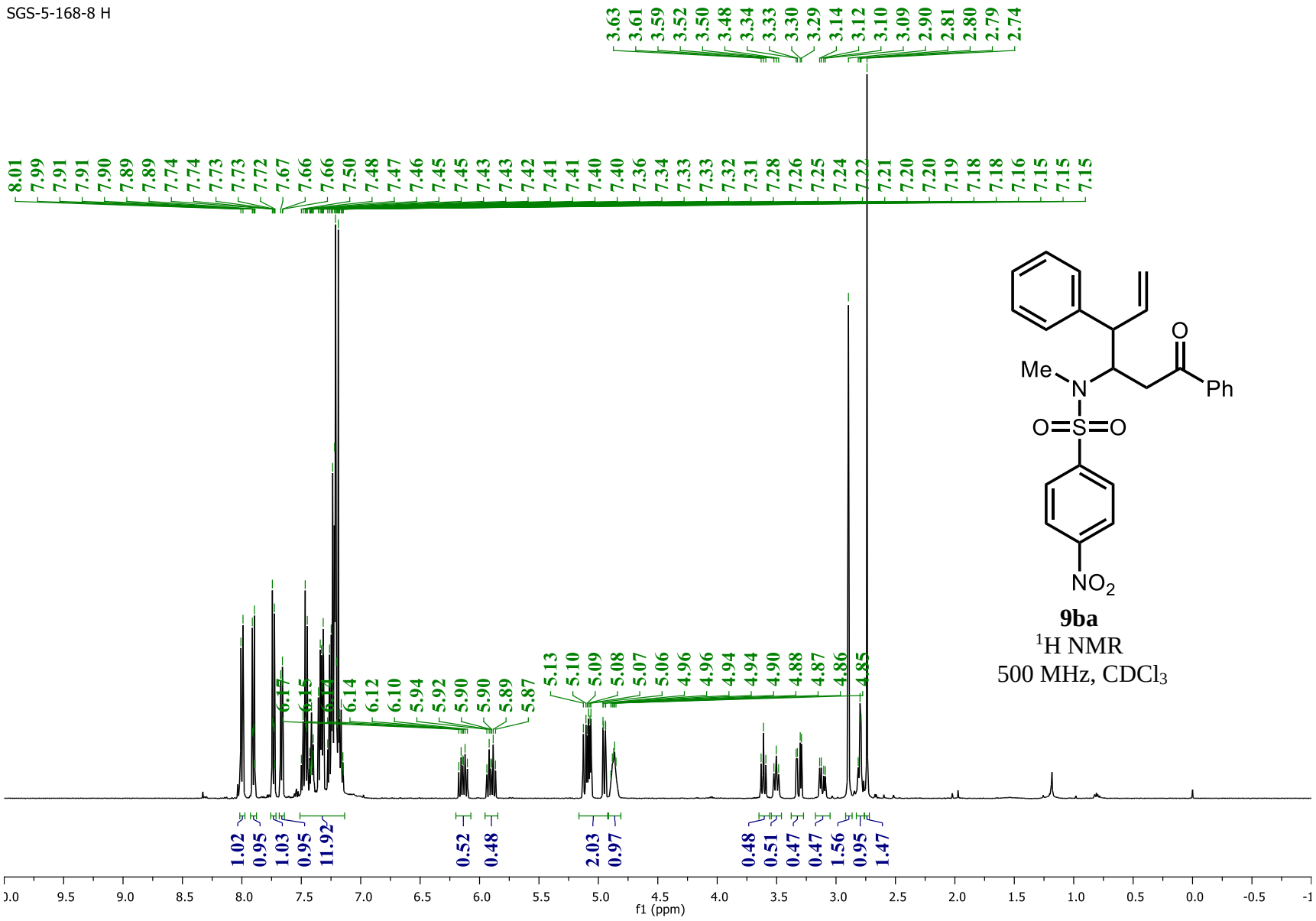
SGS-6-19 C



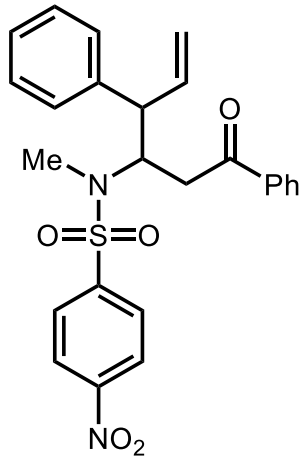
9ka

^{13}C NMR

126 MHz, $CDCl_3$


SGS-5-168-8 H
3.63 3.61 3.59 3.52 3.50 3.48 3.34 3.33 3.30 3.29 3.14 3.12 3.10 3.09 2.90 2.81 2.80 2.79 2.74
8.01 7.99 7.91 7.91 7.90 7.89 7.89 7.74 7.74 7.73 7.73 7.72 7.67 7.66 7.66 7.50 7.48 7.47 7.46 7.45 7.45 7.43 7.43 7.42 7.41 7.41 7.40 7.40 7.36 7.34 7.33 7.33 7.32 7.31 7.28 7.26 7.25 7.24 7.22 7.21 7.20 7.20 7.19 7.18 7.18 7.16 7.15 7.15 7.15
6.17 6.15 6.14 6.12 6.10 5.94 5.92 5.90 5.90 5.89 5.87
5.13 5.10 5.09 5.08 5.07 5.06 4.96 4.96 4.94 4.94 4.90 4.88 4.87 4.86 4.85
1.02 0.95 1.03 0.95 11.92 0.52 0.48 2.03 0.97 0.48 0.51 0.47 0.47 1.56 0.95 1.47
f1 (ppm)
Me
N
O
Ph
O=S=O
NO2
9ba
1H NMR
500 MHz, CDCl3

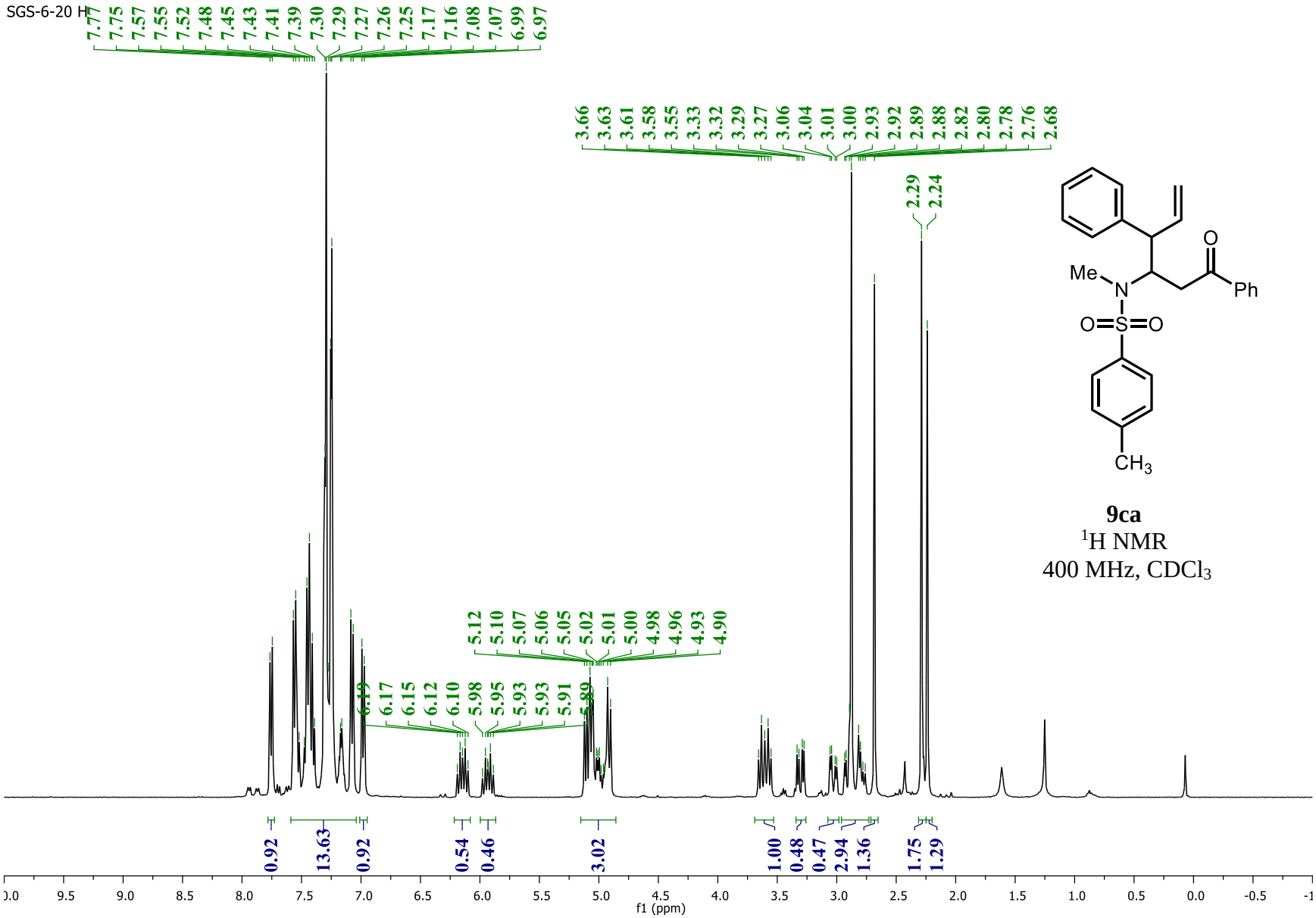
SGS-5-168-8 C



9ba

^{13}C NMR

126 MHz, $CDCl_3$

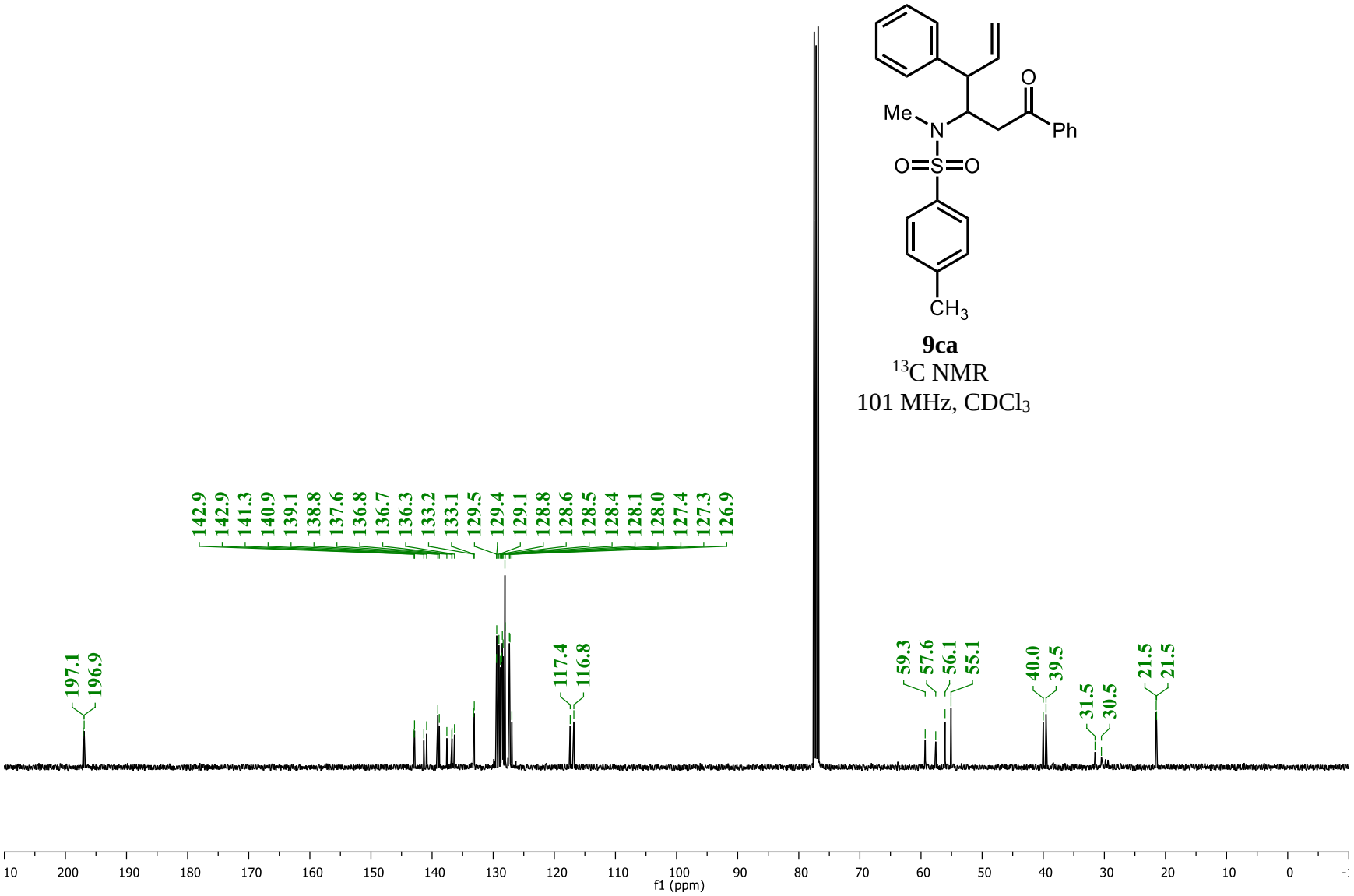


9ca

^{1}H NMR

400 MHz, $CDCl_3$

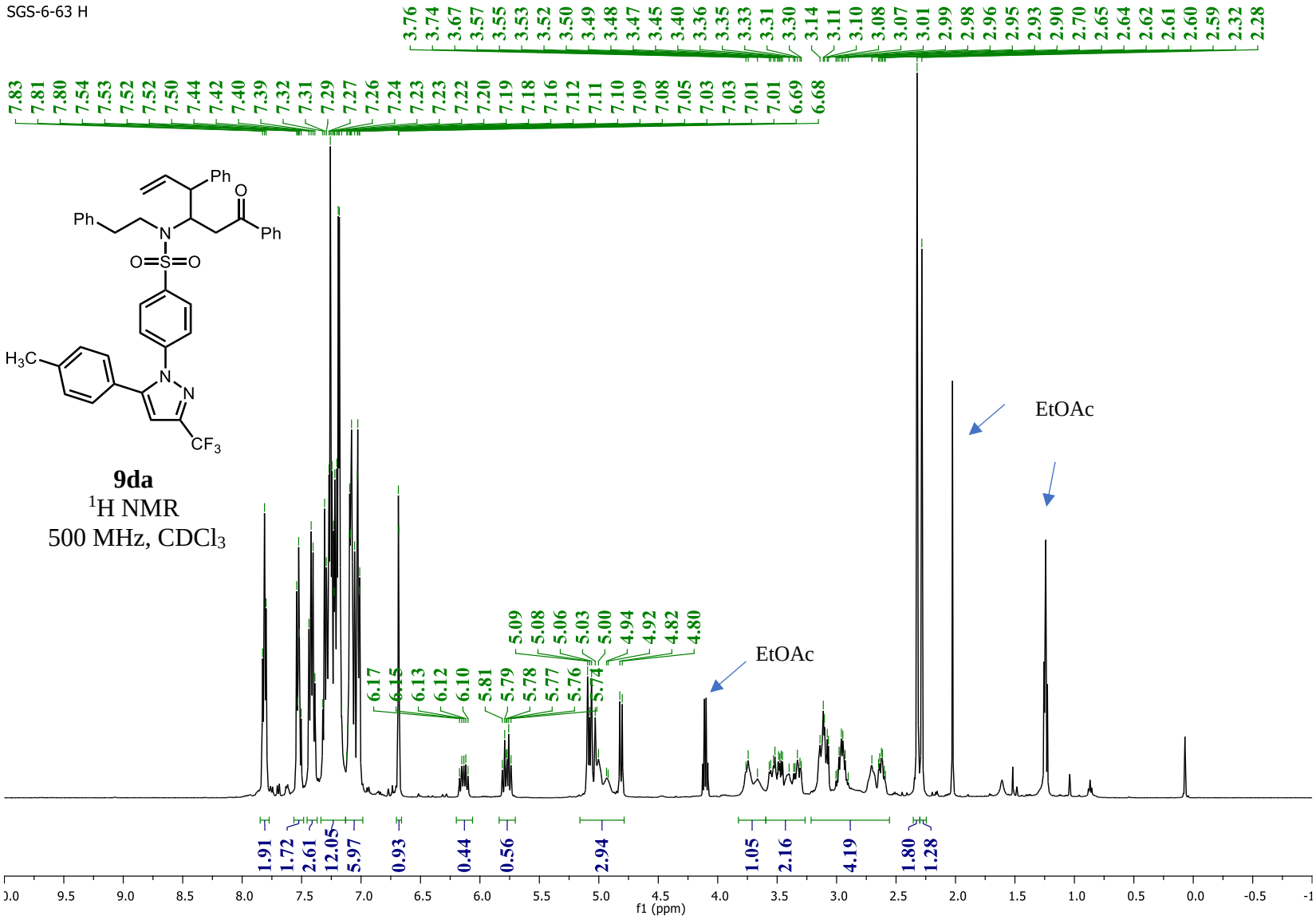
SGS-6-20 C

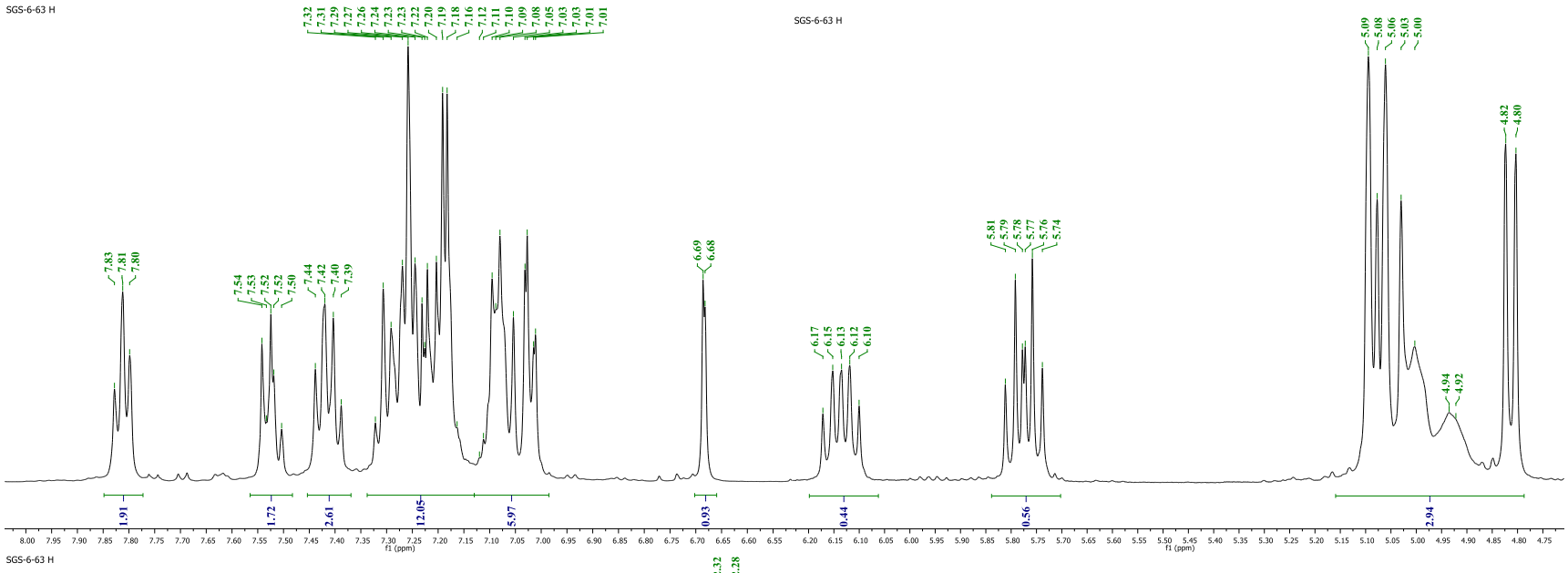


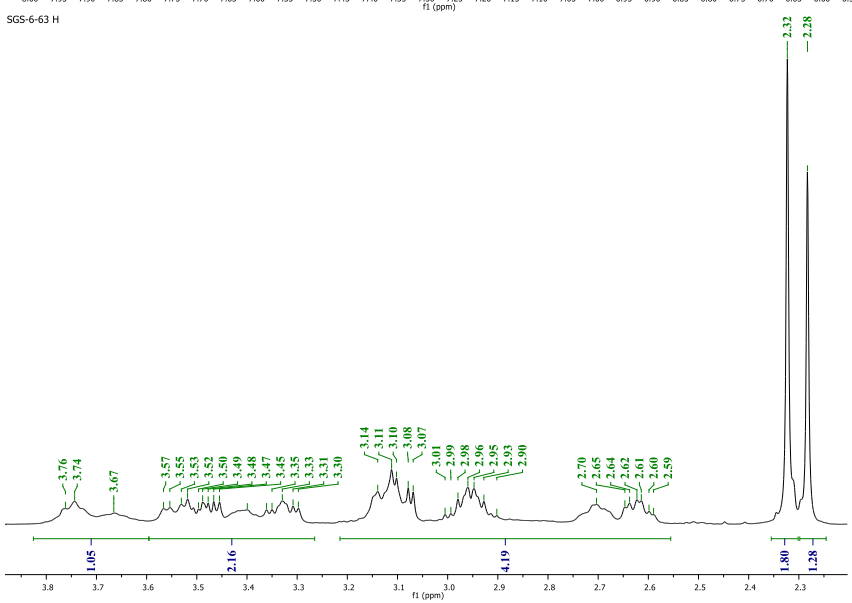
9ca

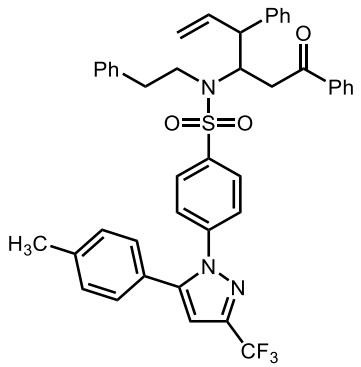
^{13}C NMR

101 MHz, $CDCl_3$


SGS-6-63 H
9da
1H NMR
500 MHz, CDCl3
EtOAc
EtOAc
f1 (ppm)





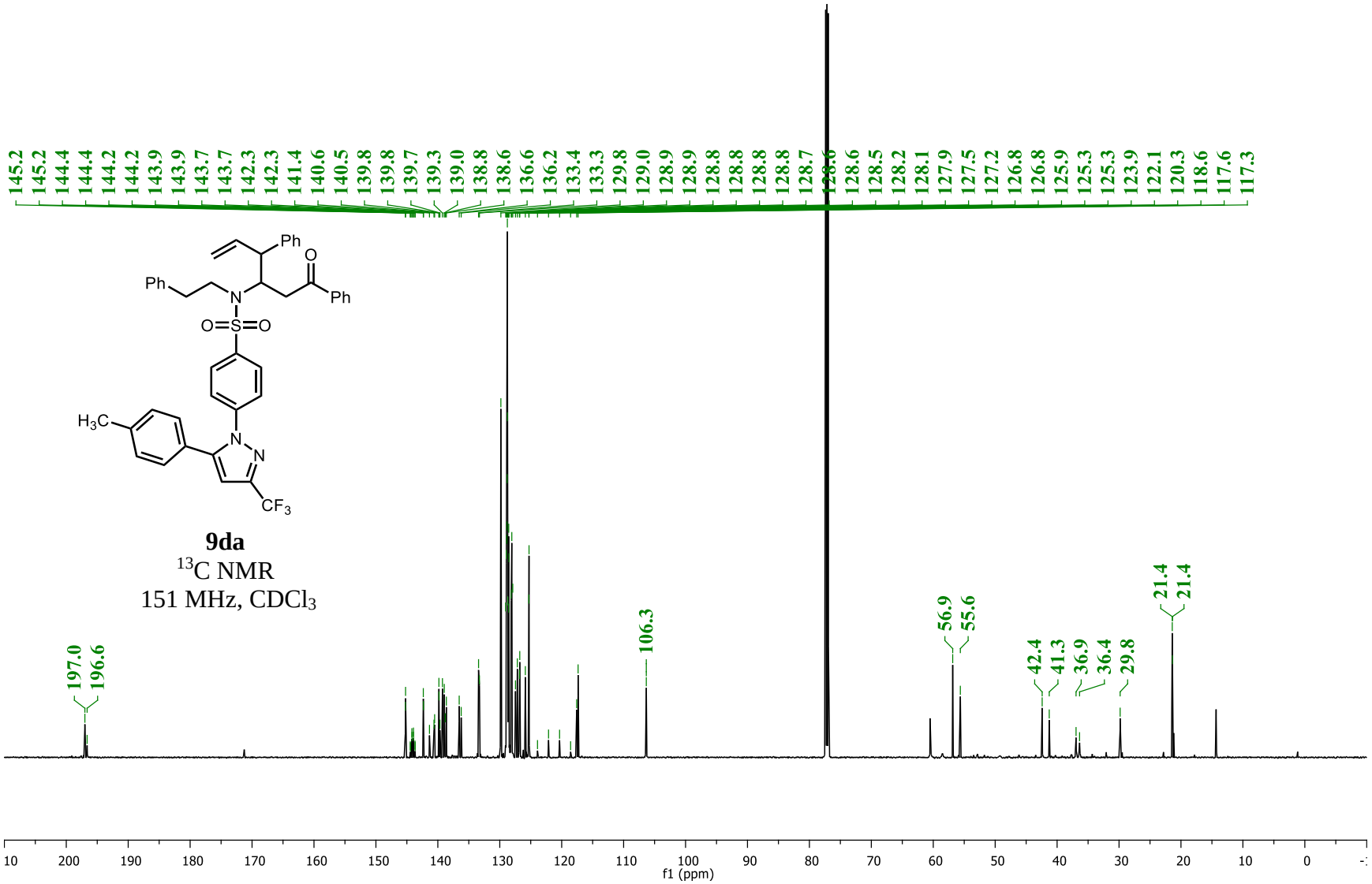


9da

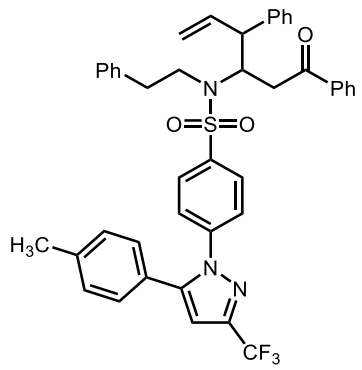
^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-63 C


145.2
145.2
144.4
144.4
144.2
144.2
143.9
143.9
143.7
143.7
142.3
142.3
141.4
140.6
140.5
139.8
139.8
139.7
139.3
139.0
138.8
138.6
136.6
136.2
133.4
133.3
129.8
129.0
128.9
128.9
128.8
128.8
128.8
128.8
128.7
128.6
128.6
128.5
128.2
128.1
127.9
127.5
127.2
126.8
126.8
125.9
125.3
125.3
123.9
122.1
120.3
118.6
117.6
117.3
Ph
O
Ph
N
Ph
O=S=O
H3C
N
N
CF3
9da
13C NMR
151 MHz, CDCl3
197.0
196.6
106.3
56.9
55.6
42.4
41.3
36.9
36.4
29.8
21.4
21.4
f1 (ppm)

SGS-6-63 F



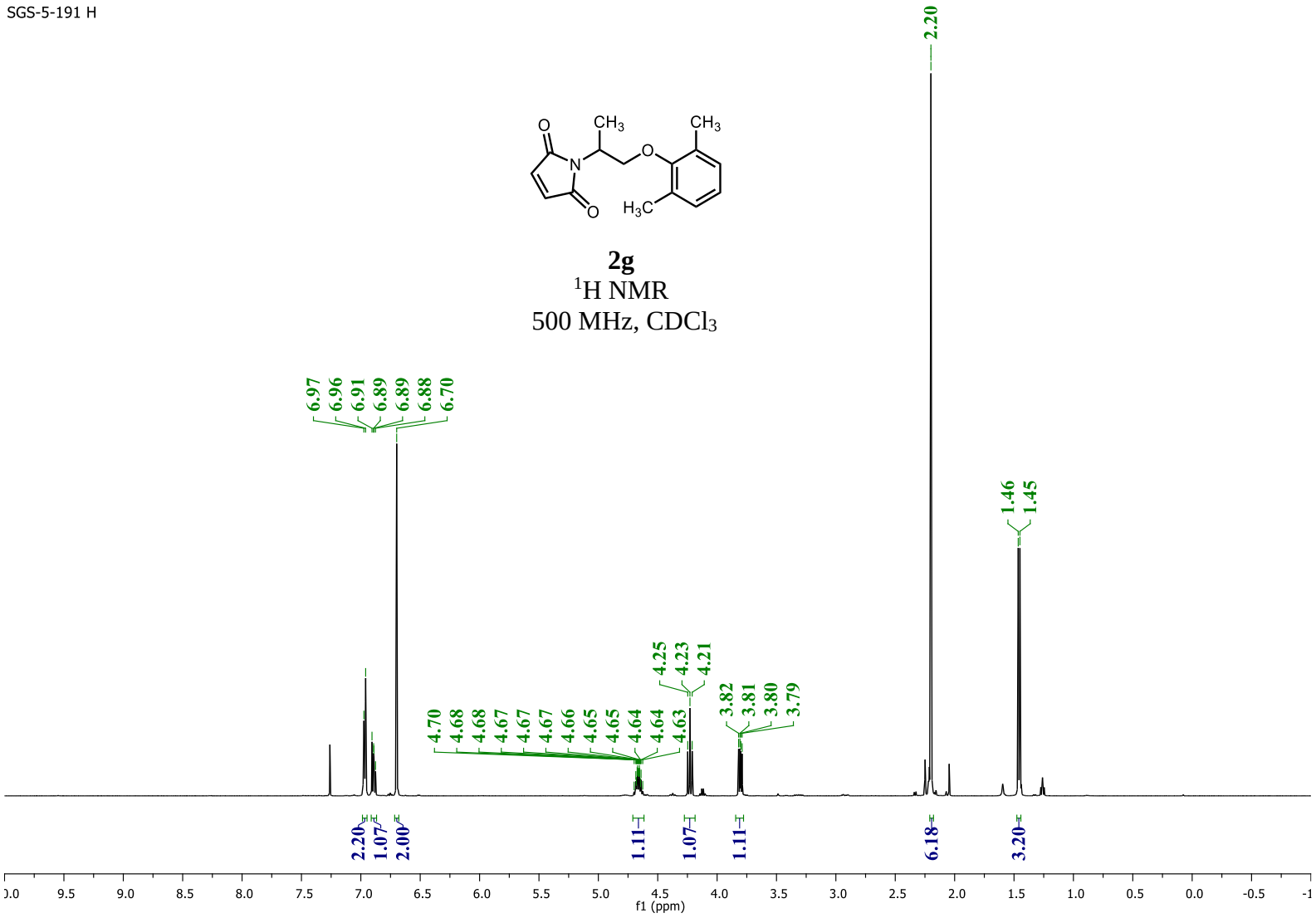
9da

^{19}F NMR

471 MHz, $CDCl_3$

7.5 NMR spectra of starting materials

SGS-5-191 H

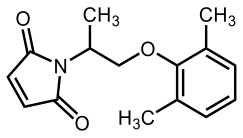


2g

^{1}H NMR

500 MHz, $CDCl_3$

SGS-5-191 C

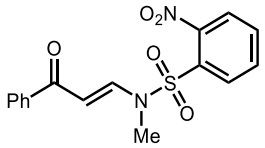


2g

^{13}C NMR

126 MHz, $CDCl_3$

SGS-5-181 H

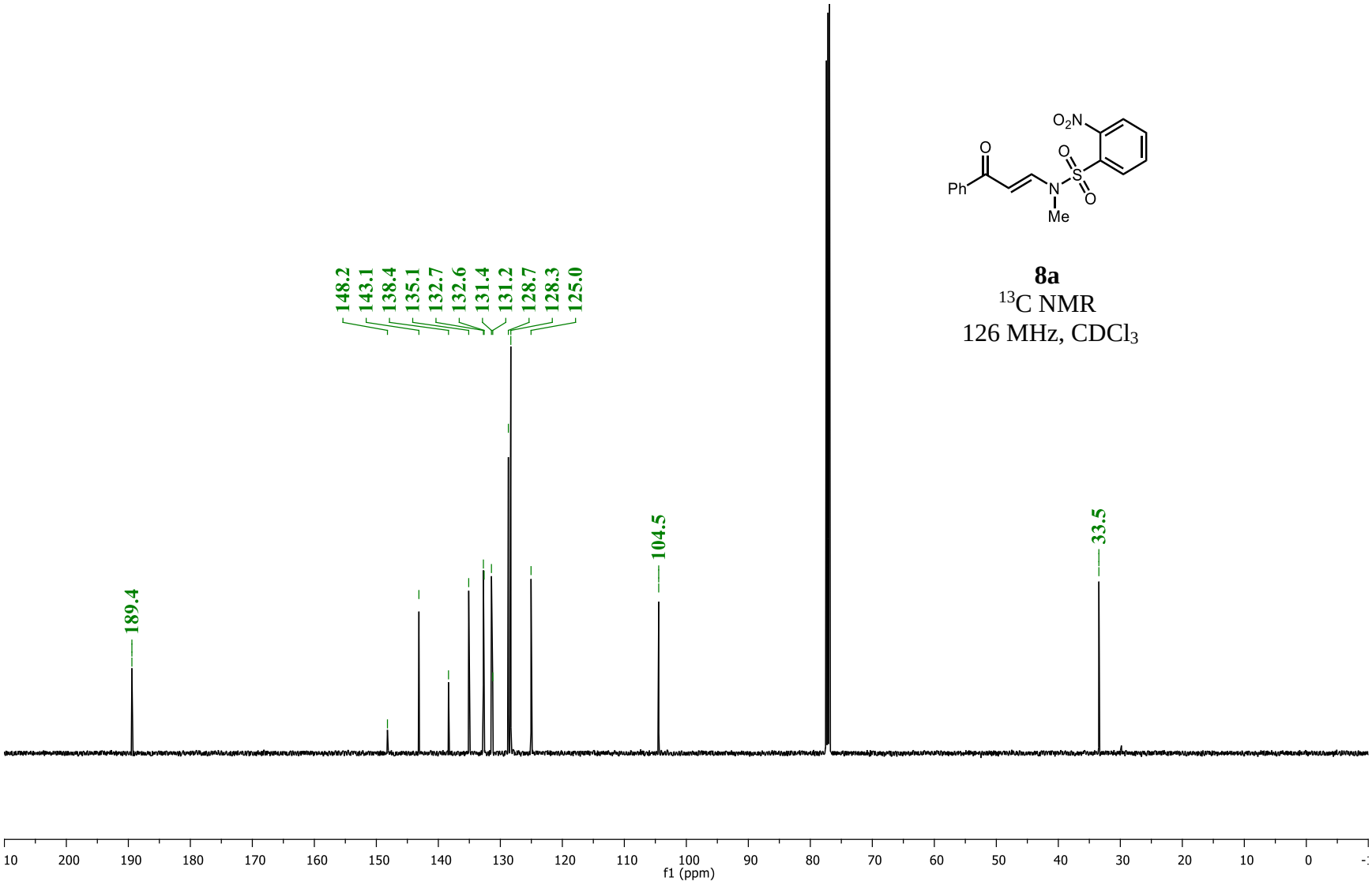


8a

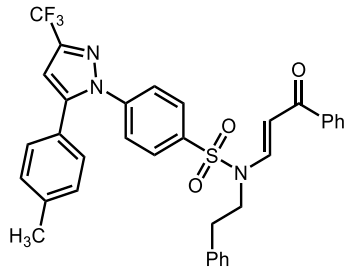
^{1}H NMR

500 MHz, $CDCl_3$

SGS-5-181 C


148.2
143.1
138.4
135.1
132.7
132.6
131.4
131.2
128.7
128.3
125.0
189.4
104.5
33.5
O2N
O
O
S
O
Ph
N
Me
8a
13C NMR
126 MHz, CDCl3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

SGS-5-183 H

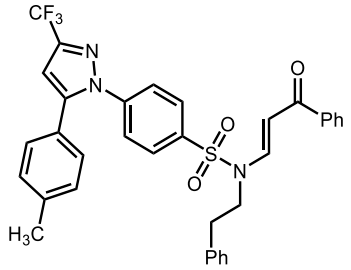


8d

^{1}H NMR

500 MHz, $CDCl_3$

SGS-5-183 F

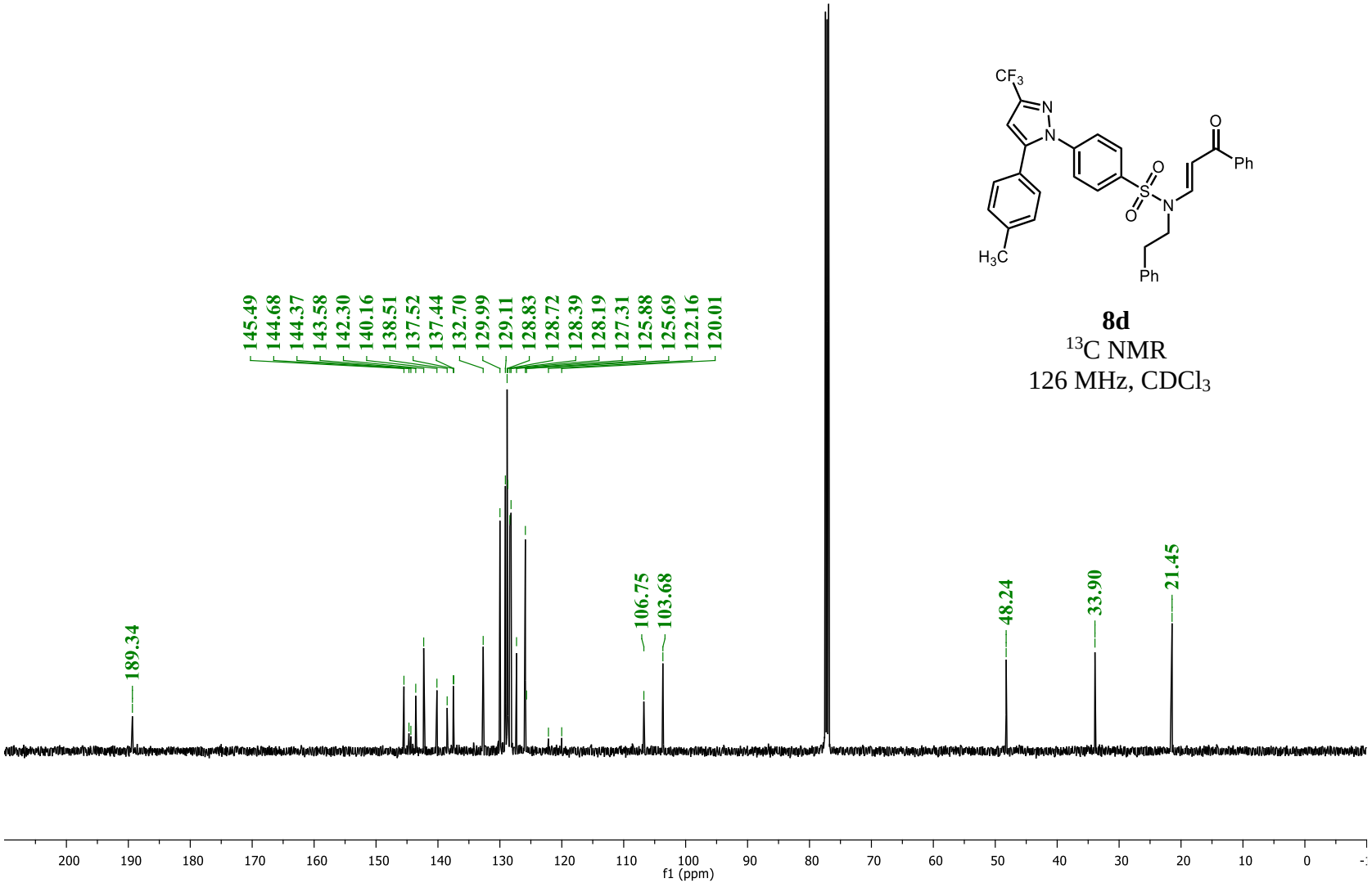


8d

^{19}F NMR

471 MHz, $CDCl_3$

SGS-5-183 C

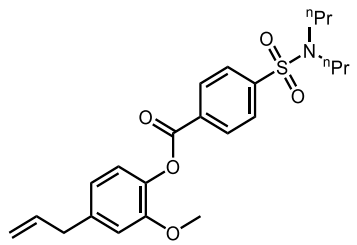


8d

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-62 H



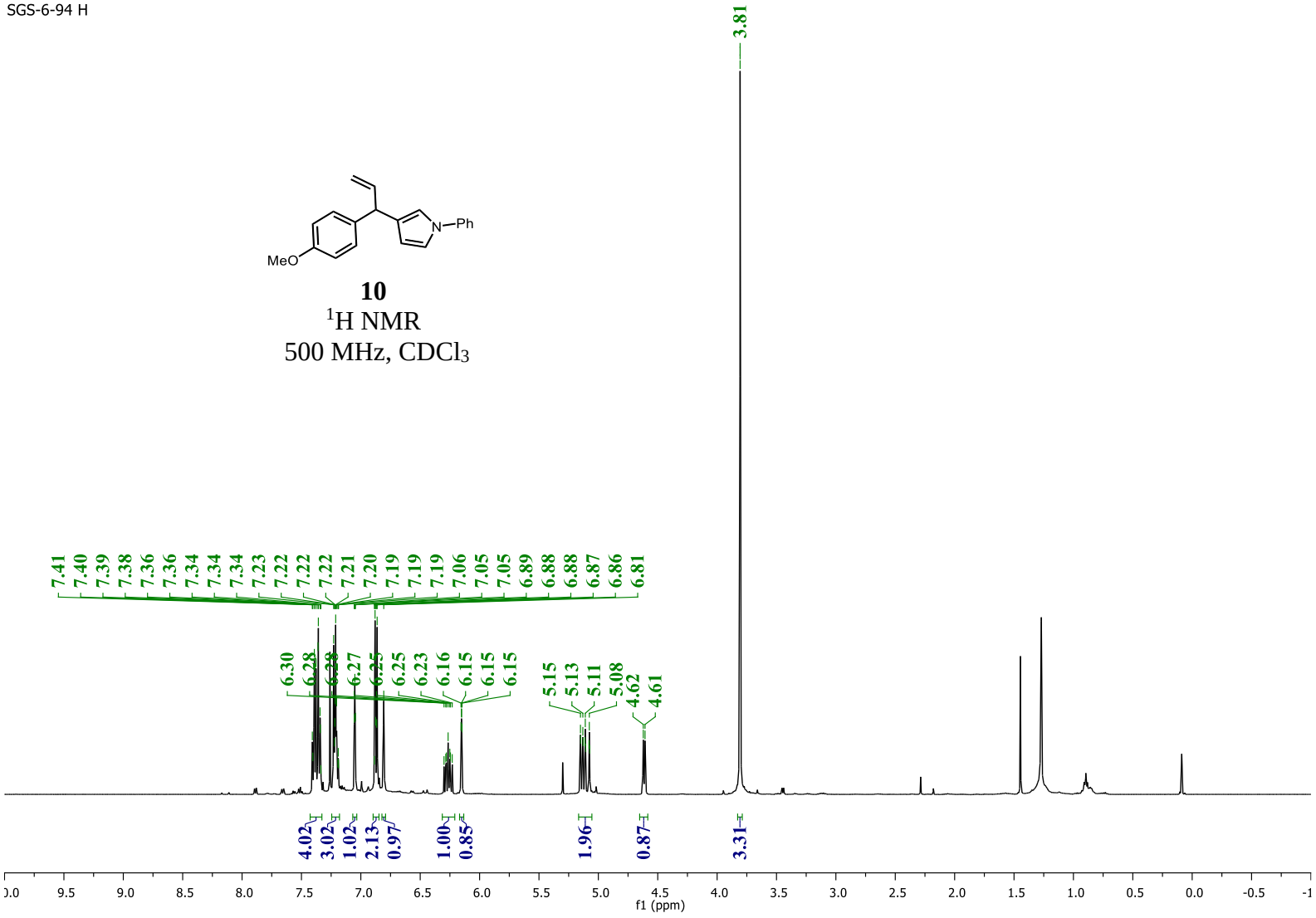
5ah

^{1}H NMR

400 MHz, $CDCl_3$

7.6 NMR spectra of synthetic application of products

SGS-6-94 H

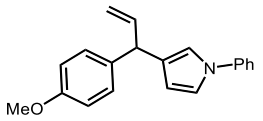


10

^{1}H NMR

500 MHz, $CDCl_3$

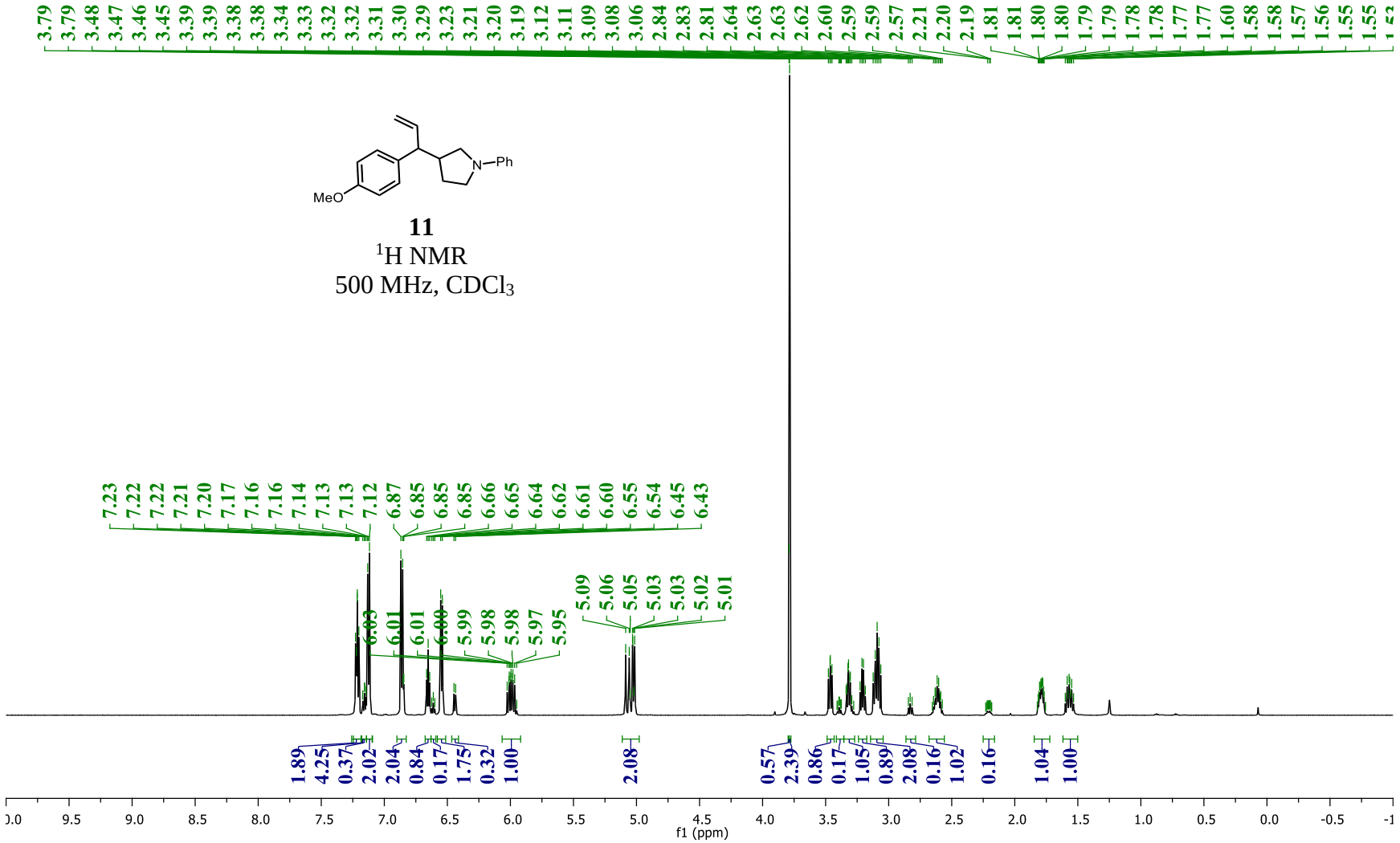
SGS-6-94 C



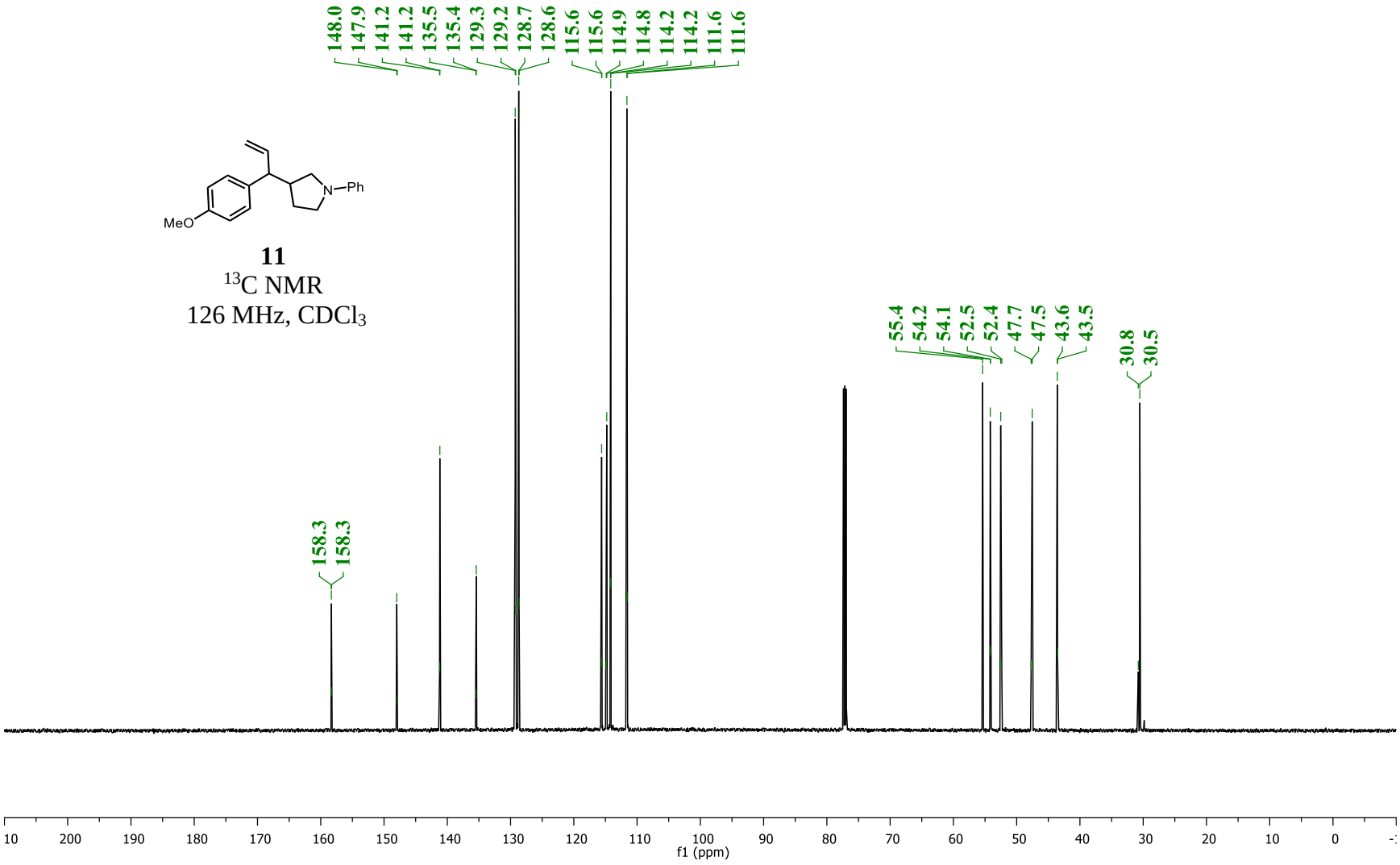
10

^{13}C NMR

126 MHz, $CDCl_3$


MeO
N—Ph
11
1H NMR
500 MHz, CDCl3
f1 (ppm)

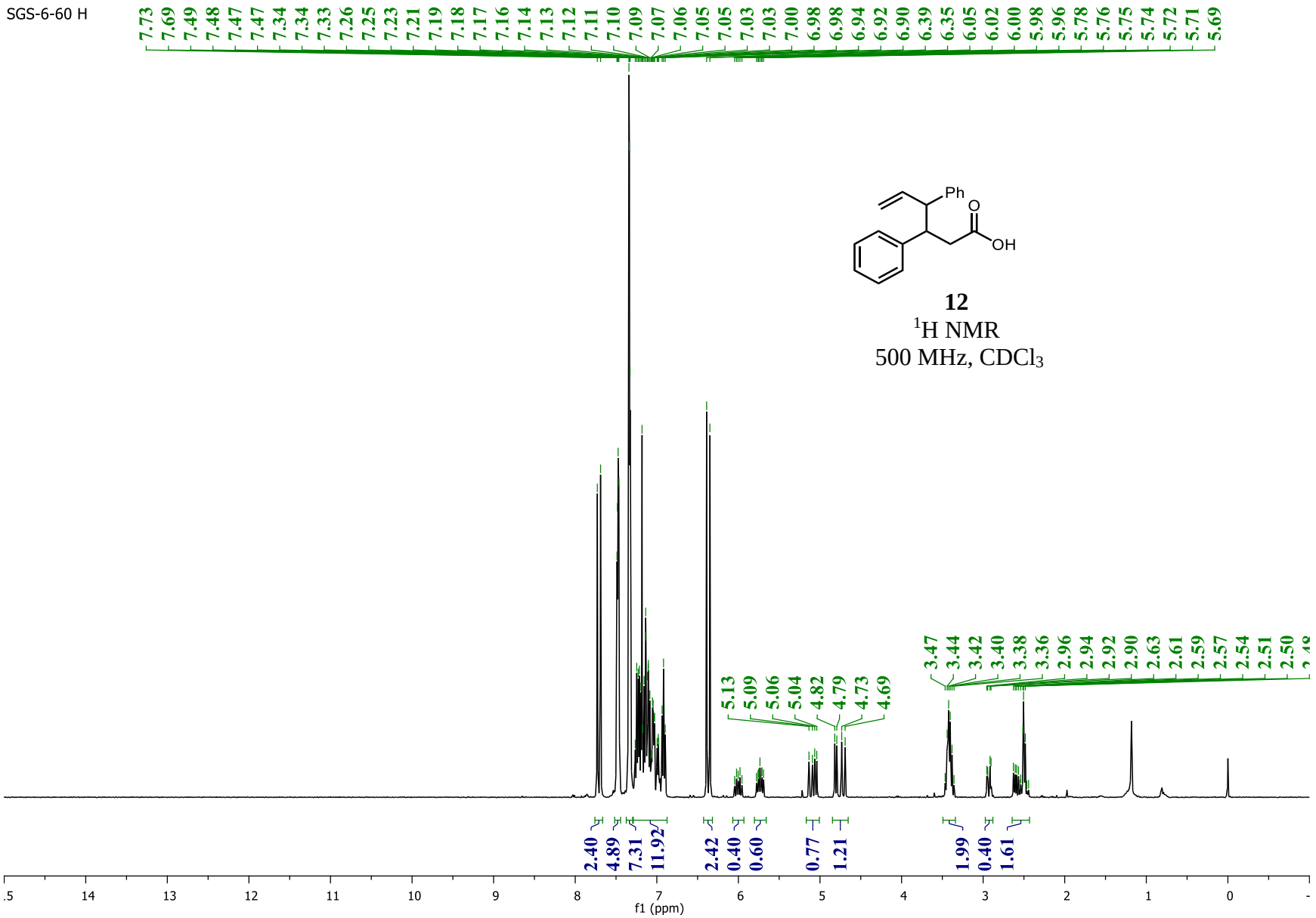
SGS-6-67 C



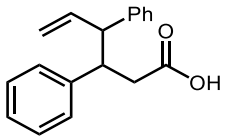
11

^{13}C NMR

126 MHz, $CDCl_3$


SGS-6-60 H
7.73 7.69 7.49 7.48 7.47 7.47 7.34 7.34 7.33 7.26 7.25 7.23 7.21 7.19 7.18 7.17 7.16 7.14 7.13 7.12 7.11 7.10 7.09 7.07 7.06 7.05 7.05 7.03 7.03 7.00 6.98 6.98 6.94 6.92 6.90 6.39 6.35 6.05 6.02 6.00 5.98 5.96 5.78 5.76 5.75 5.74 5.72 5.71 5.69
Ph
O
OH
12
1H NMR
500 MHz, CDCl3
5.13 5.09 5.06 5.04 4.82 4.79 4.73 4.69
3.47 3.44 3.42 3.40 3.38 3.36 2.96 2.94 2.92 2.90 2.63 2.61 2.59 2.57 2.54 2.51 2.50
2.40 4.89 7.31 11.92 2.42 0.40 0.60 0.77 1.21 1.99 0.40 1.61
f1 (ppm)

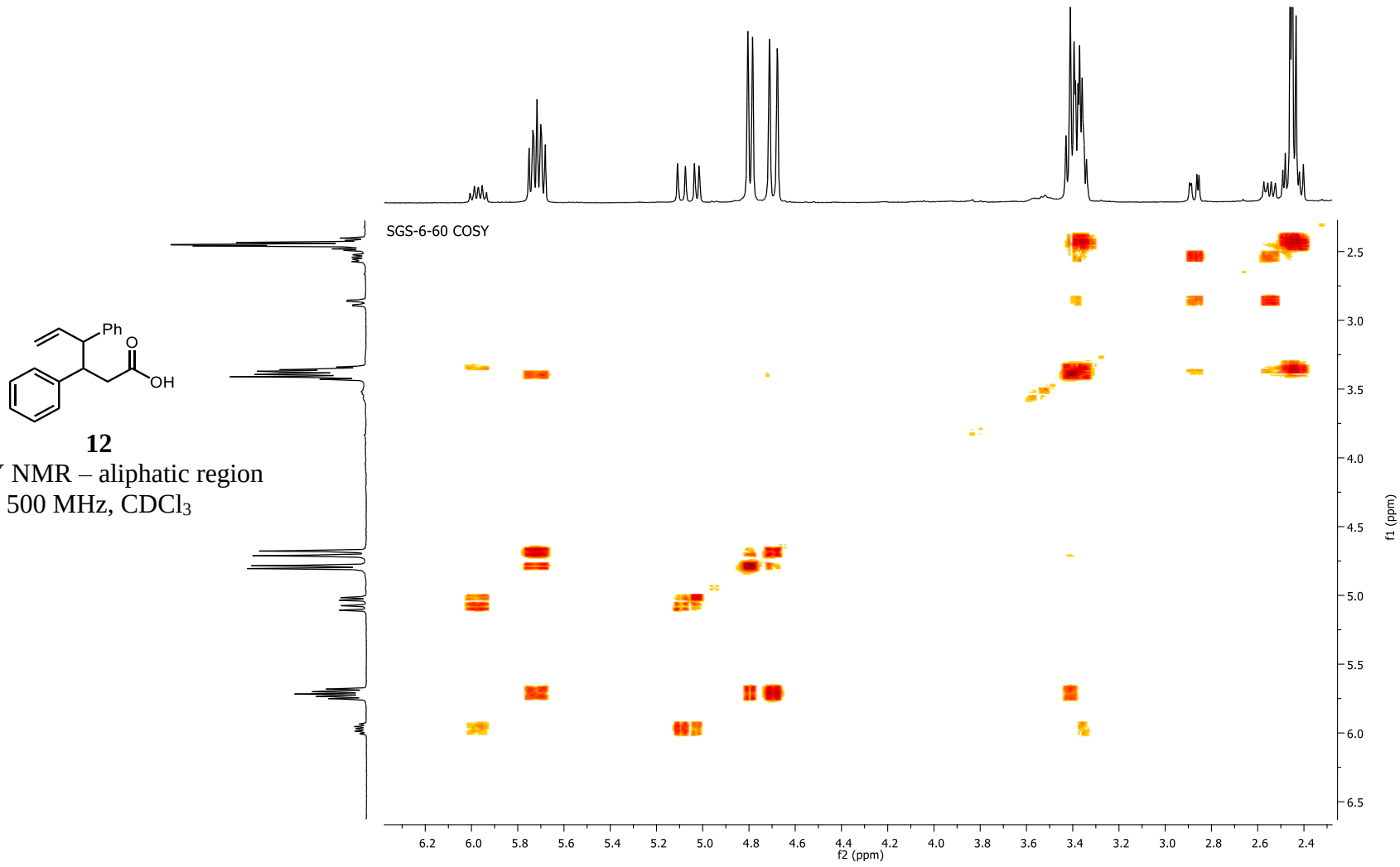
SGS-6-60 C



12

^{13}C NMR

126 MHz, $CDCl_3$

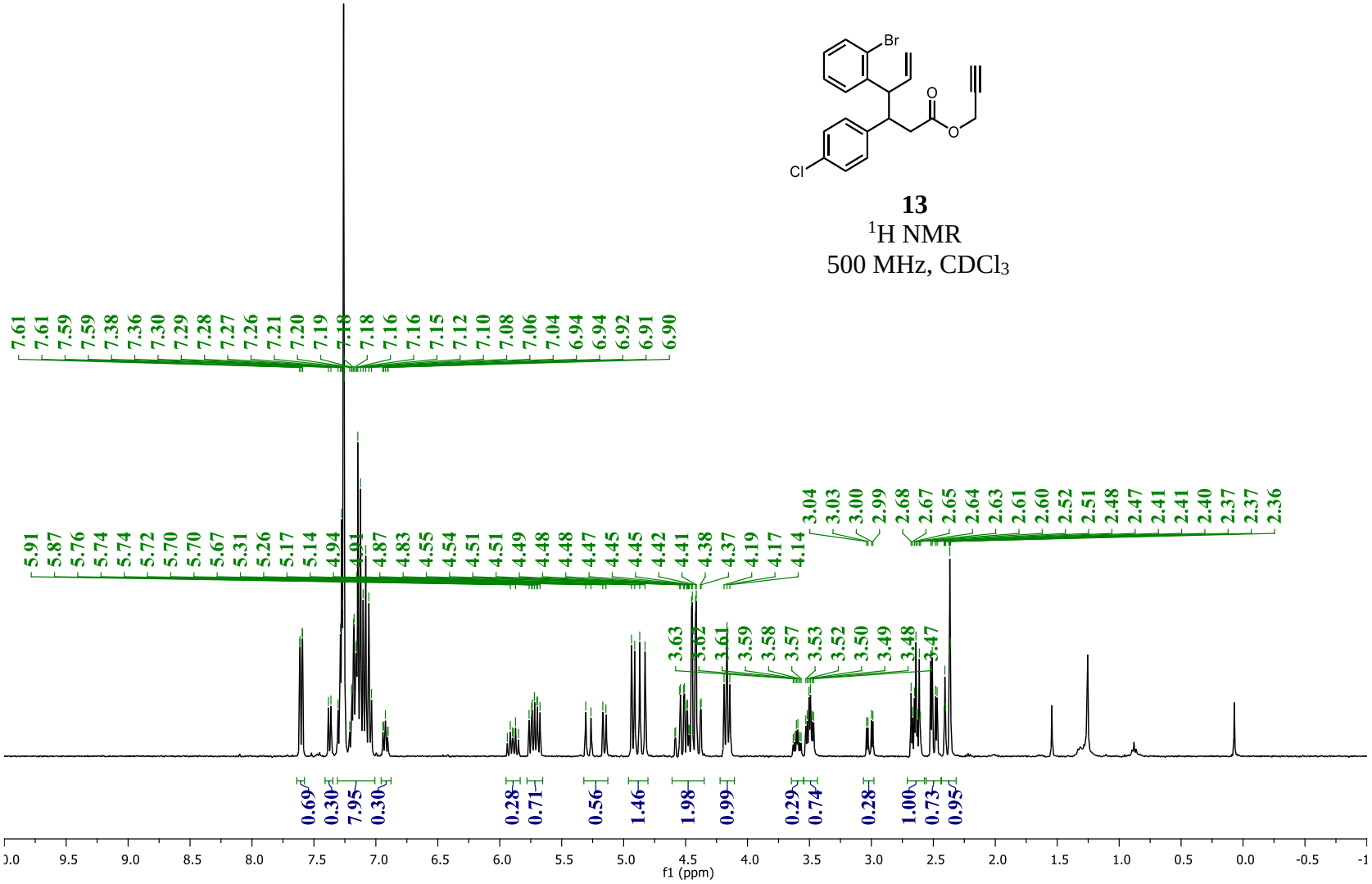


12

COSY NMR – aliphatic region

500 MHz, $CDCl_3$

SGS-7-4 H

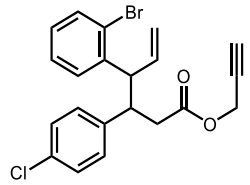


13

^{1}H NMR

500 MHz, $CDCl_3$

SGS-7-4 C

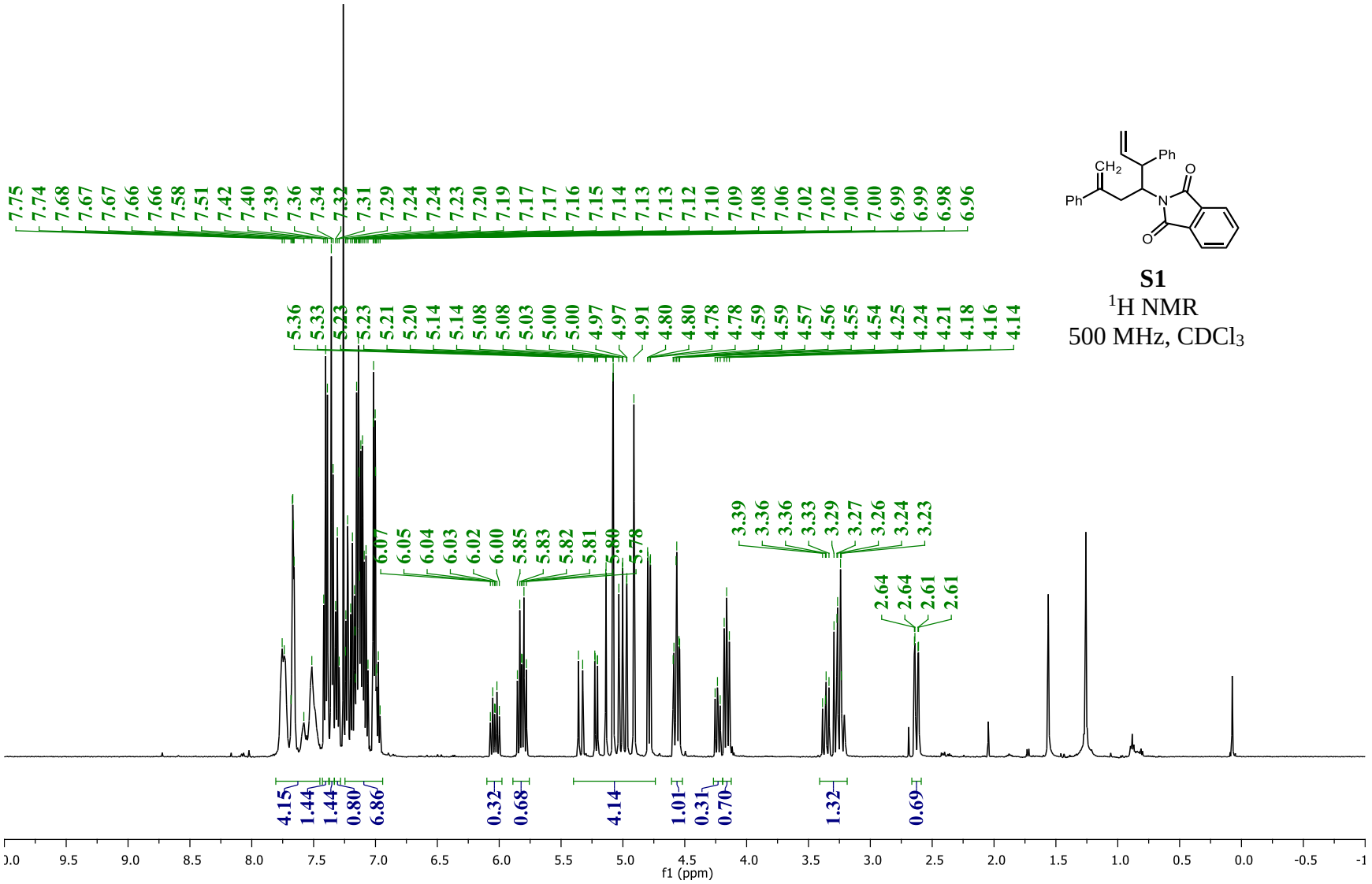


13

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-79 H

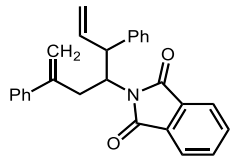


S1

^{1}H NMR

500 MHz, $CDCl_3$

SGS-6-79 C

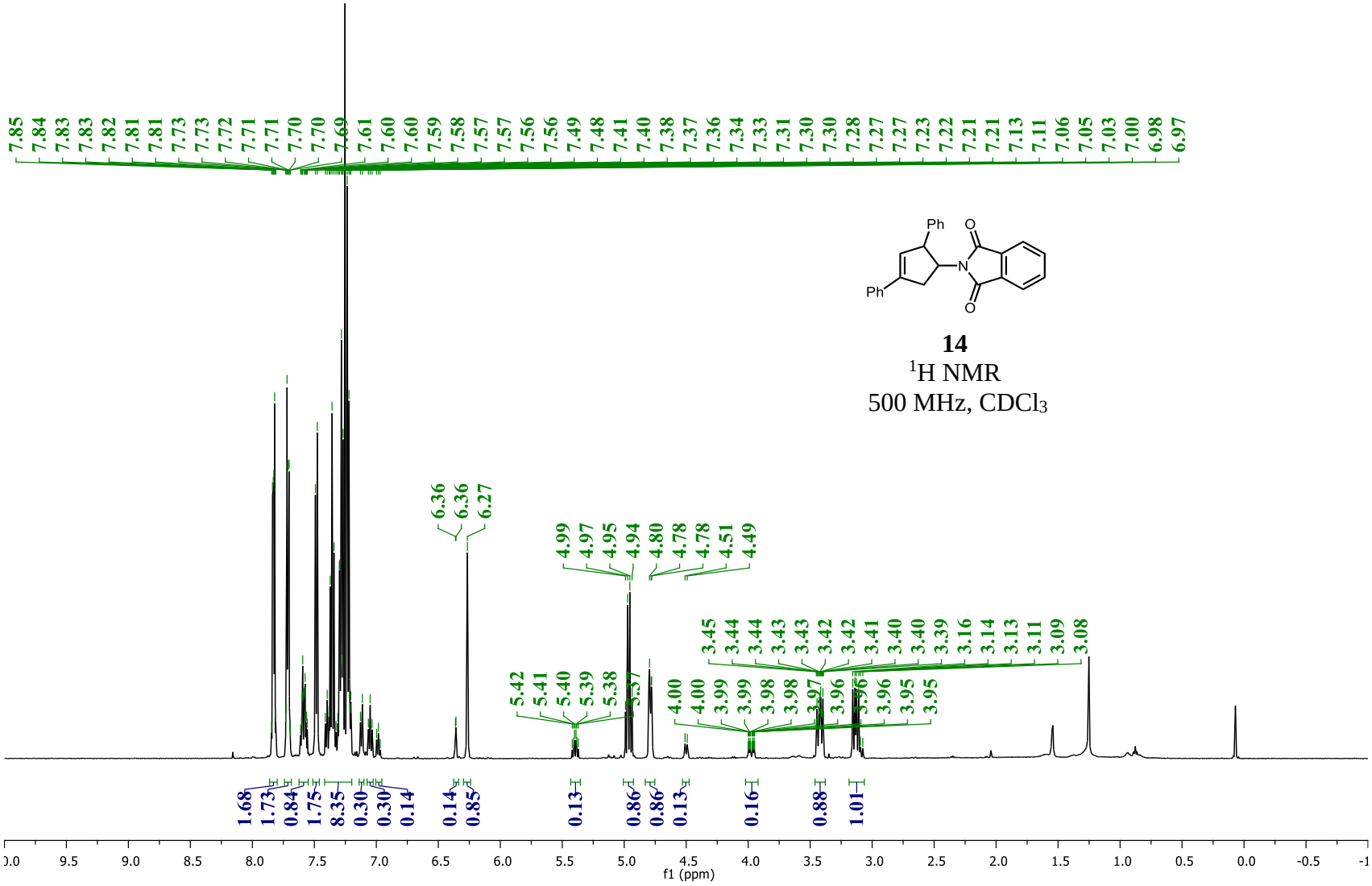


S1

^{13}C NMR

126 MHz, $CDCl_3$

SGS-6-90 H

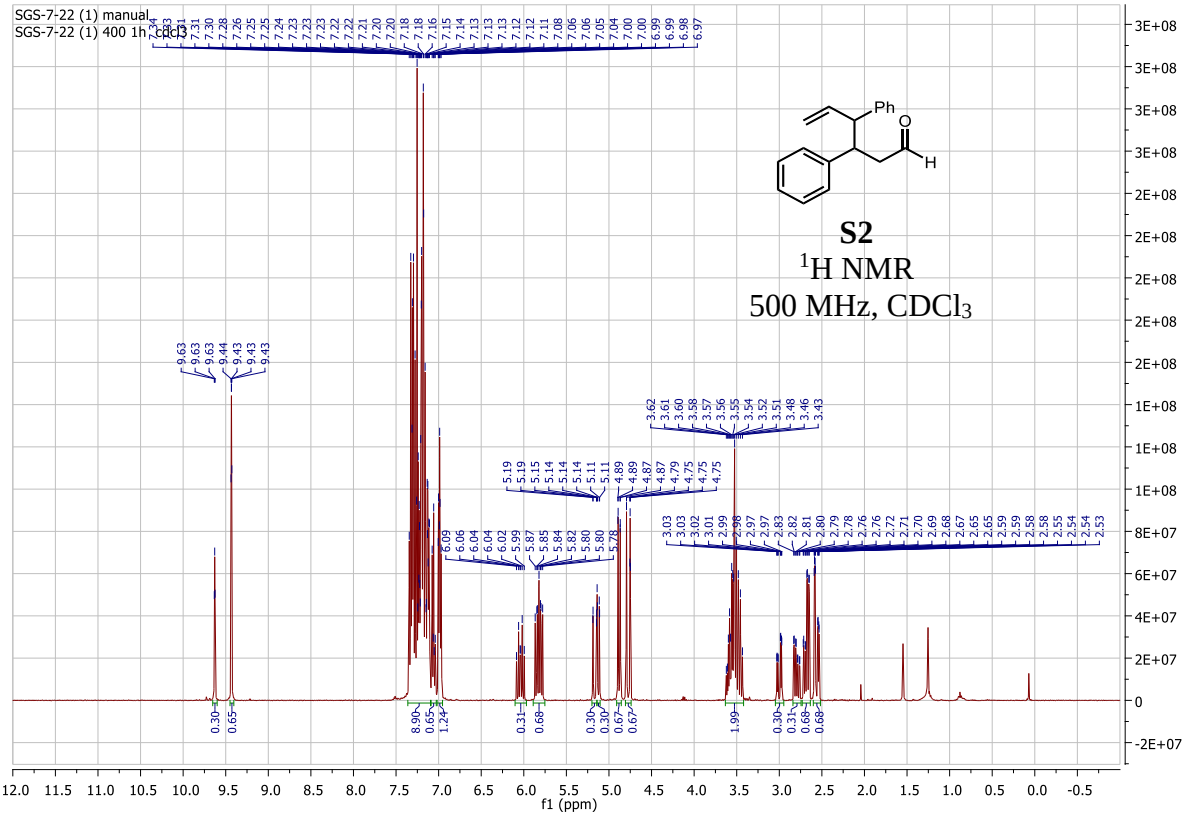


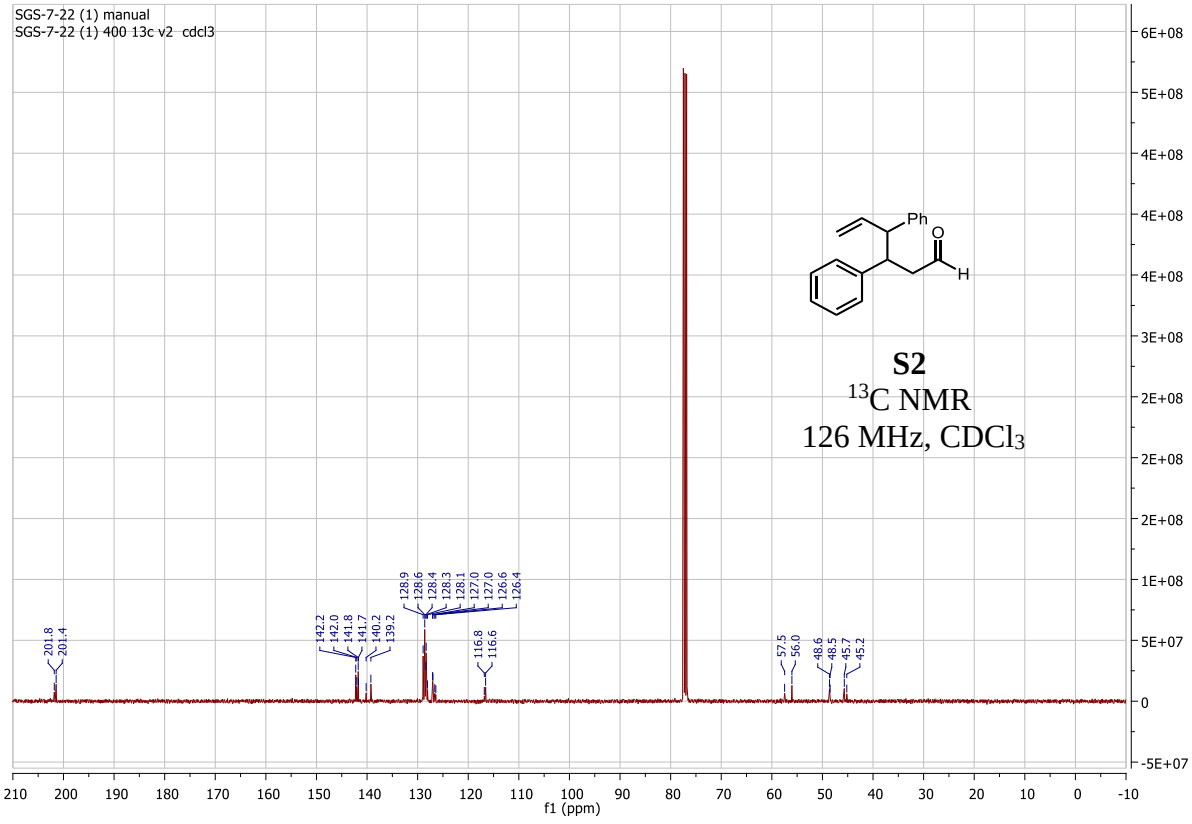
14

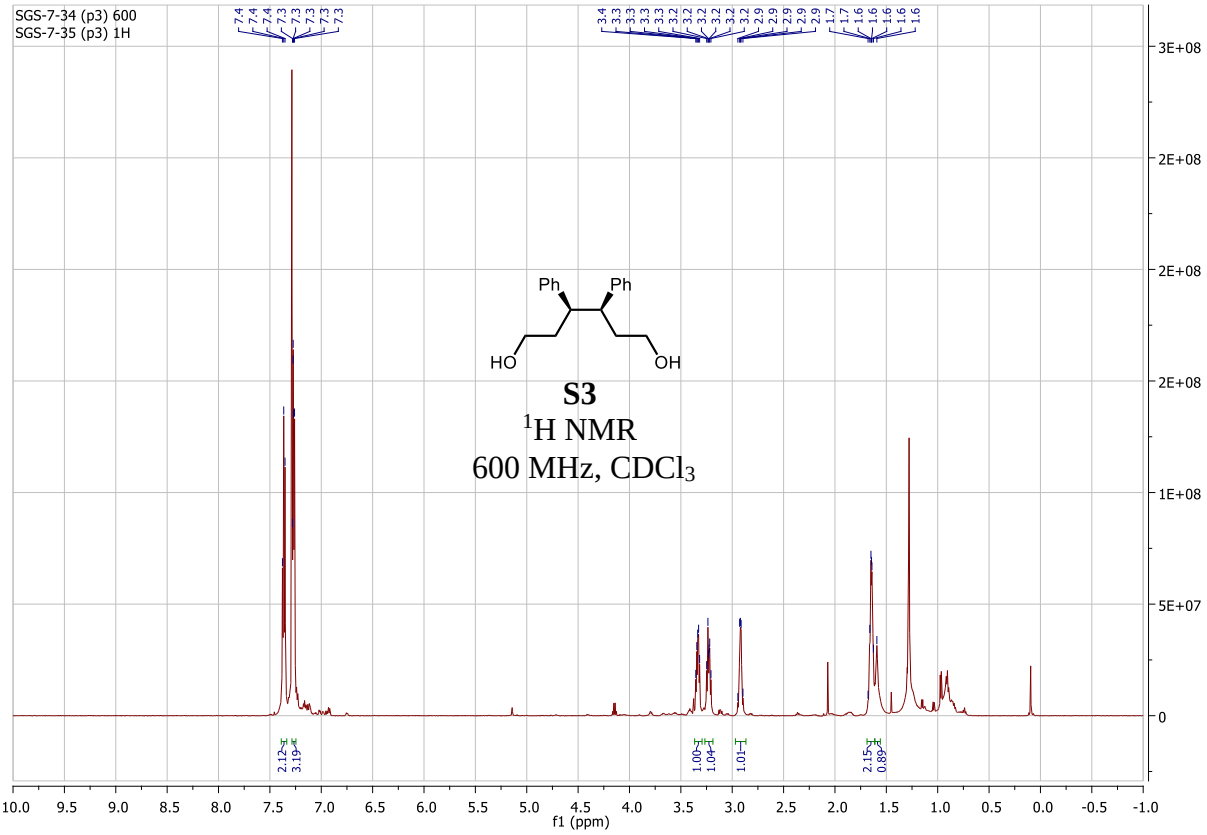
^{1}H NMR

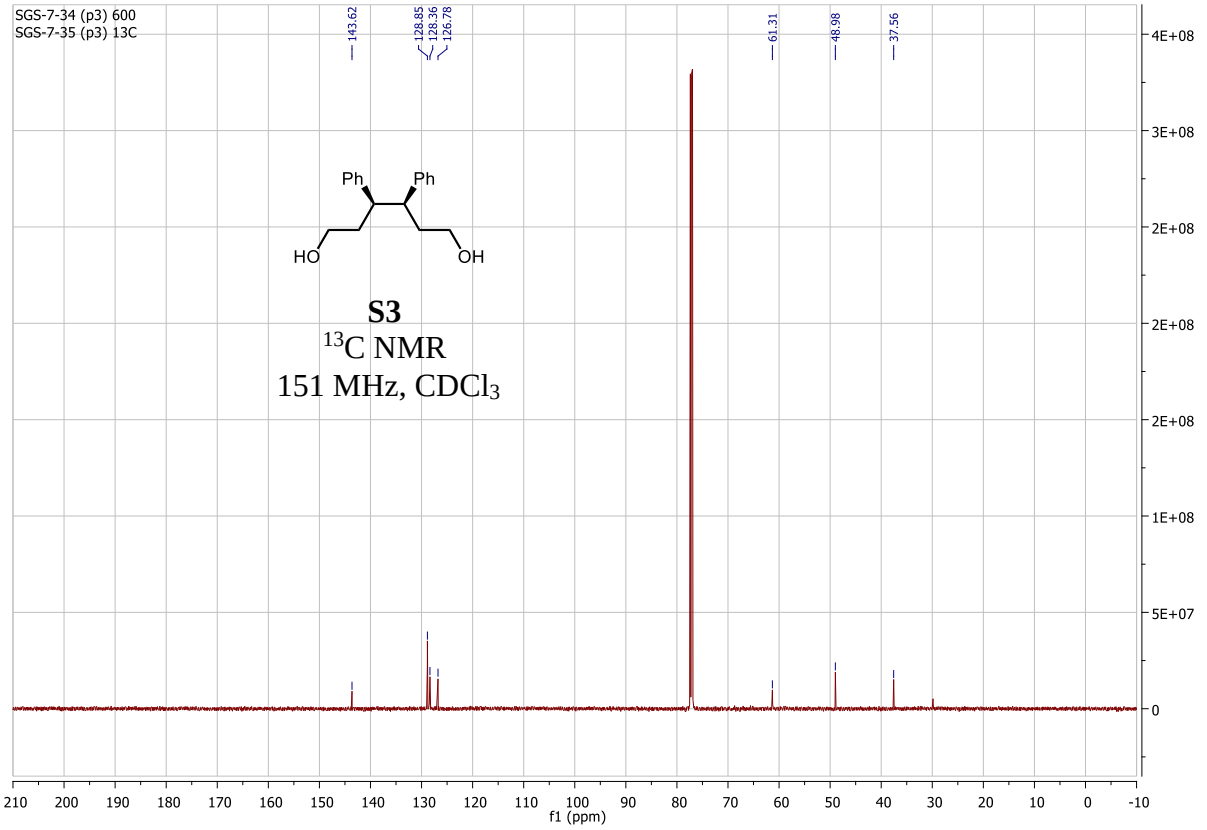
500 MHz, $CDCl_3$

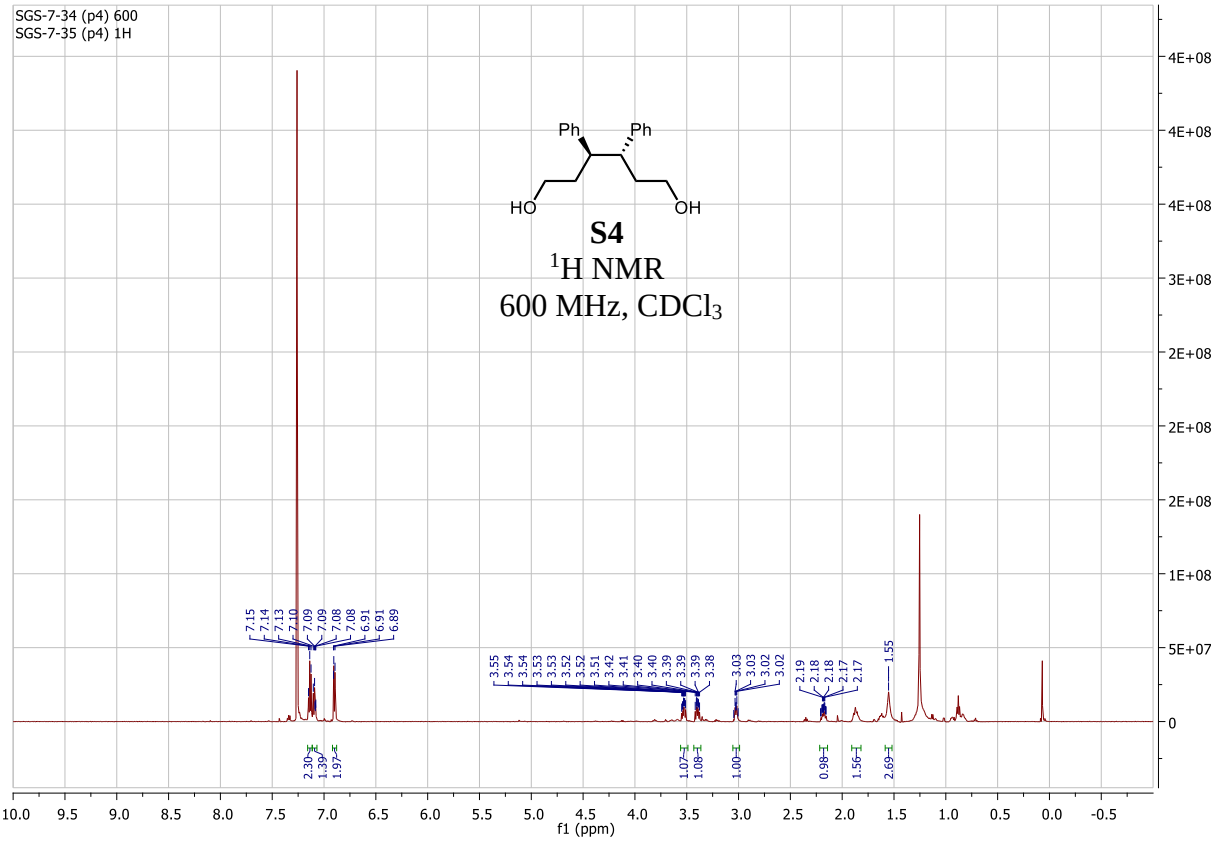
7.7 Derivatization for N-Acyl oxazolidinone diastereomer assignments

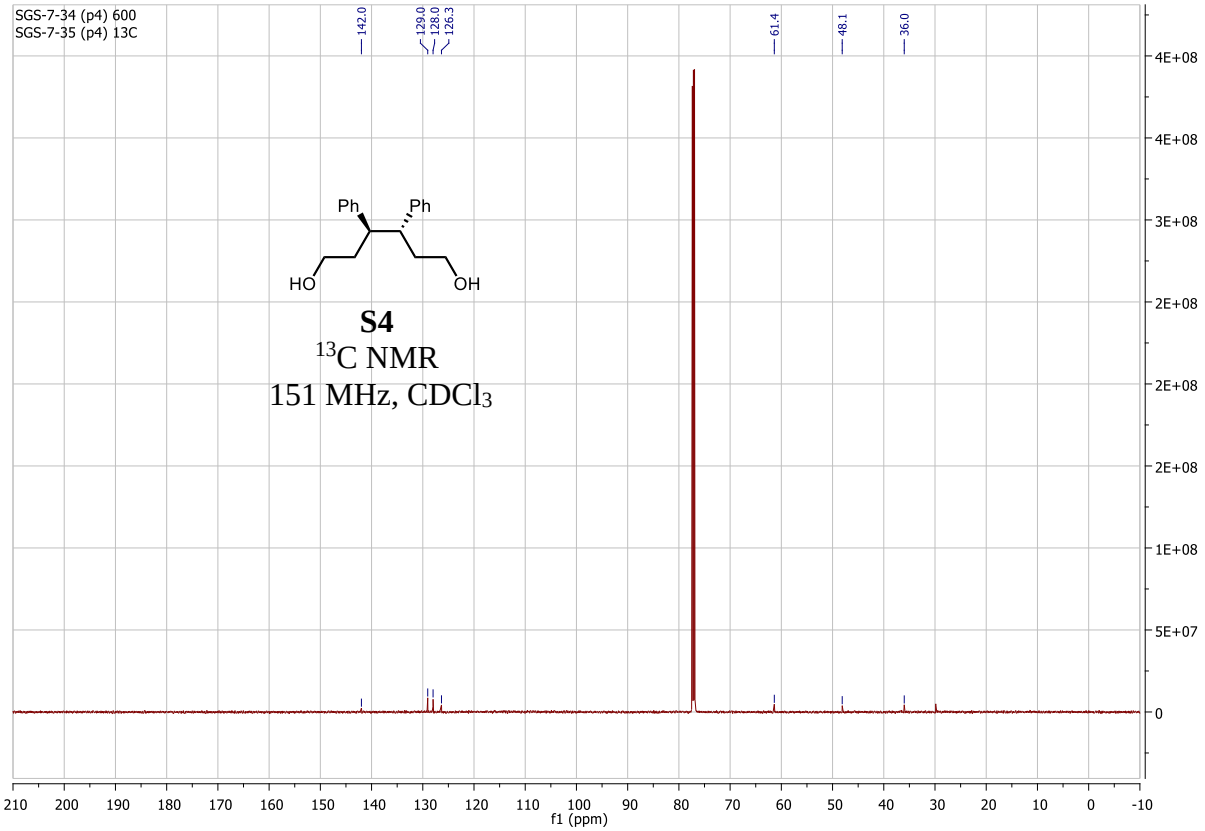





SGS-7-34 (p3) 600
SGS-7-35 (p3) 1H
Ph
Ph
HO
OH
S3
1H NMR
600 MHz, CDCl3
f1 (ppm)


SGS-7-34 (p3) 600
SGS-7-35 (p3) 13C
143.62
128.85
128.36
126.78
61.31
48.98
37.56
Ph
Ph
HO
OH
S3
13C NMR
151 MHz, CDCl3
f1 (ppm)


SGS-7-34 (p4) 600
SGS-7-35 (p4) 1H
Ph
Ph
HO
OH
S4
1H NMR
600 MHz, CDCl3
f1 (ppm)


SGS-7-34 (p4) 600
SGS-7-35 (p4) 13C
142.0
129.0
128.0
126.3
61.4
48.1
36.0
Ph
Ph
HO
OH
S4
13C NMR
151 MHz, CDCl3
f1 (ppm)

8. References

1. Y. Wang, J. Zhu, A. C. Durham, H. Lindberg and Y.-M. Wang, *J. Am. Chem. Soc.*, 2019, **141**, 19594-19599.
2. G. Xu, Y. Yang, Y. Yang, G. Song, S. Li, J. Zhang, W. Yang, L.-L. Wang, Z. Weng and Z. Zuo, *Eur. J. Med. Chem.*, 2020, **191**, 112115.
3. Y. He, W. Li, H. Zhou, G. Zeng, Z. Chen, J.-Y. Ge, N. Lv and J. Chen, *Adv. Synth. Catal.*, 2022, **364**, 3730-3735.
4. X. Li, X. Zhang, F. Zhang, X. Luo and H. Luo, *Adv. Synth. Catal.*, 2022, **364**, 1683-1688.
5. N. M. Nascimento-Júnior, T. C. F. Mendes, D. M. Leal, C. M. N. Corrêa, R. T. Sudo, G. Zapata-Sudo, E. J. Barreiro and C. A. M. Fraga, *Bioorg. Med. Chem. Lett.,* 2010, **20**, 74-77.
6. L. Zhao, Y. Ma, W. Duan, F. He, J. Chen and C. Song, *Org. Lett.*, 2012, **14**, 5780-5783.
7. J.-F. Wang, X. Meng, C.-H. Zhang, C.-M. Yu and B. Mao, *Org. Lett.*, 2020, **22**, 7427-7432.
8. X. Liang, X. Huang, M. Xiong, K. Shen and Y. Pan, *Chem. Commun.*, 2018, **54**, 8403-8406.
9. A. Gómez-Palomino and J. Cornella, *Angew. Chem. Int. Ed.*, 2019, **58**, 18235-18239.
10. S. G. Scrivener, Y. Wang and Y.-M. Wang, *Org. Lett.*, 2023, **25**, 1420-1424.
11. X. Qi, P. Chen and G. Liu, *Angew. Chem. Int. Ed.*, 2017, **56**, 9517-9521.
12. S.-L. Liu, Y. Li, J.-R. Guo, G.-C. Yang, X.-H. Li, J.-F. Gong and M.-P. Song, *Org. Lett.*, 2017, **19**, 4042-4045.
13. L. M. Rice, E. E. Reid and C. H. Grogan, *J. Org. Chem.*, 1954, **19**, 884-893.
14. G. L. Beutner, B. M. Cohen, A. J. DelMonte, D. D. Dixon, K. J. Fraunhoffer, A. W. Glace, E. Lo, J. M. Stevens, D. Vanyo and C. Wilbert, *Org. Process Res. Dev.*, 2019, **23**, 1378-1385.
15. J. M. Stevens, A. C. Parra-Rivera, D. D. Dixon, G. L. Beutner, A. J. DelMonte, D. E. Frantz, J. M. Janey, J. Paulson and M. R. Talley, *J. Org. Chem.*, 2018, **83**, 14245-14261.
16. Z. Zhao, L. Racicot and G. K. Murphy, *Angew. Chem. Int. Ed.*, 2017, **56**, 11620-11623.
17. A. K. Chatterjee, T.-L. Choi, D. P. Sanders and R. H. Grubbs, *J. Am. Chem. Soc.*, 2003, **125**, 11360-11370.