Supporting Information for

Catalytic C(sp^3)—H Alkylation via an Iron Carbene Intermediate

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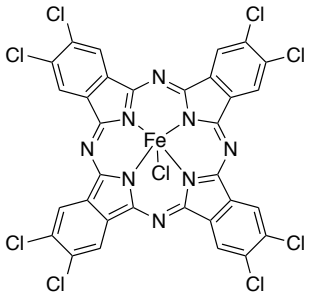
General Information

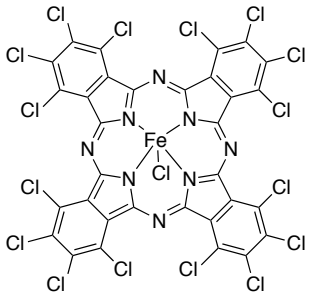
The following commercially obtained reagents were used as received: iron(III) phthalocyanine chloride ([FePc]Cl, Sigma-Aldrich), iron(II) phthalocyanine (FePc, Sigma-Aldrich), 5,10,15,20-Tetraphenyl-21*H*,23*H*-porphine iron(III) chloride (Fe(TPP)Cl, Sigma-Aldrich), 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine (PyBOX, Sigma-Aldrich), rhodium(II) acetate dimer ($Rh_2(OAc)_4$, Strem), and silver hexafluoroantimonate ($AgSbF_6$, Strem). Sodium tetrakis[3,5-bis(trifluoromethyl)phenyl]borate ($NaBAr^F{}_4$)[1], $MeSO_2N_3$[2], and $ClSO_2CH_2CO_2Et$[3] were prepared according to literature procedures and stored at rt in the glovebox, 4˚C, and -20˚C, respectively. All reactions were run in flame- or oven-dried glassware under an atmosphere of N_2 or Ar gas with dry solvents unless otherwise stated. All products were filtered through a glass wool plug prior to obtaining a final weight. Solid reagents were stored in a dessicator or glovebox, and anhydrous solvents were purified by passage through a bed of activated alumina immediately prior to use (Glass Countour, Laguna Beach, California). Chloroform-*d* was stored over 3Å molecular sieves. Thin-layer chromatography (TLC) was conducted with E. Merck silica gel 60 F254 pre-coated plates (0.25 mm) and visualized with UV and potassium permanganate stains. Flash chromatography was performed as described by Still [4] using American International ZEOprep 60 ECO silica gel (230-400 mesh). Achiral gas chromatographic (GC) analysis was performed on an Agilent 6890N Series instrument equipped with FID detectors using a HP-5 (5%-Phenyl)-methylpolysiloxane column (30m, 0.32mm, 0.25mm), and chiral GC analysis using a CycloSil-B column (30m, 0.25mm, 0.25mm). ^{1}H-NMR spectra were recorded on a Varian Inova-500 (500 MHz), Varian Unity-500 (500 MHz), Varian Unity Inova-400 (400 MHz), or Bruker (500Mz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 7.26 ppm). Data reported as: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, sxt = sextet, spt = septet, m = multiplet, br = broad, app = apparent; coupling constant(s) in Hz; integration. Proton-decoupled ^{13}C-NMR spectra were recorded on a Varian Unity-500 (126 MHz) or Bruker (126Mz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 77.16 ppm). Kinetic isotope effect analyses were recorded on a Varian Inova-600 (600 MHz) spectrometer. IR spectra were recorded by ATR on a Perkin Elmer Frontier FTIR and are reported in frequency of absorption (cm^{-1}). Optical rotations were measured using a 1 mL cell with a 50 mm path length on a Jasco P-1020 polarimeter. Optical rotations were obtained with a sodium lamp and are reported as follows: $[\alpha]_\lambda T^oC$ (*c* = g/100 mL, solvent). High-resolution mass spectra were obtained at the University of Illinois Mass Spectrometry Laboratory. Electrospray ionization (ESI) spectra were performed on a Waters Q-Tof Ultima spectrometer, and electron ionization (EI) and field desorption (FD) spectra were performed on a Micromass 70-VSE spectrometer.

Preparation of $[FeCl_8Pc]Cl$ and $[FeCl_{16}Pc]Cl$



$[FeCl_8Pc]Cl$: A flame-dried 25 mL round-bottom flask equipped with a stir bar and reflux condenser (the flask and stir bar should be free of trace metal impurities) was sequentially charged with 4,5-dichlorophthalonitrile (788 mg, 4.0 mmol, 4.0 equiv, TCI America), 1-hexanol (10 mL, freshly distilled over $MgSO_4$ and degassed), $FeCl_3$ (162 mg, 1.00 mmol, 1.0 equiv, Sigma-Aldrich) and DBU (597 µL, 4.0 mmol, 4.0 equiv, Sigma-Aldrich). The reaction flask was evacuated under vacuum and refilled with N_2 three times, then heated to 155ºC. The reaction mixture changed from brown to turquoise green over 10 min. After stirring at 155ºC overnight (12-15h), the heat was ceased and the condenser was removed. After cooling to room temperature, the reaction mixture was filtered. The remaining dark green solid was washed with 10 mL of 1 M HCl, 10 mL of water, and 10 mL of EtOH. The catalyst was dried under high vacuum overnight at 90˚C to afford 761 mg (0.867 mmol) of $[FeCl_8Pc]Cl$ as a dark green powder (87% yield).

UV-Vis (H_2SO_4, c = 13.4 µM, λ_{max} = nm, $\varepsilon = M^{-1}cm^{-1}$): 787 (ε = 49036), 305 (ε = 65655), 249 (ε = 107538).; IR (ATR, cm^{-1}) 2955.86, 2353.03, 1889.14, 1767.7, 1730.25, 1602.19, 1514.22, 1448.96, 1435.09, 1416.2, 1387.01, 1330.14, 1290.86, 1200.89, 1137.03, 1084.38, 1071.49, 962.86, 887.52, 849.38, 824.54, 799.97, 783.39, 749.16, 708.28, 679.24, 660.94, 593.17, 550, 504.; HRMS (EI) *m/z* = 839.77 (M-Cl).



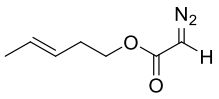
$[FeCl_{16}Pc]Cl$: A flame-dried 25 mL round-bottom flask equipped with a stir bar and reflux condenser (the flask and stir bar should be free of trace metal impurities) was sequentially charged with tetrachlorophthalonitrile (1.06 g, 4.0 mmol, 4.0 equiv, TCI America), 1-hexanol (10 mL, freshly distilled over $MgSO_4$ and degassed), $FeCl_3$ (162 mg, 1.00 mmol, 1.0 equiv, Sigma-Aldrich) and DBU (597 µL, 4.0 mmol, 4.0 equiv, Sigma-Aldrich). The reaction flask was evacuated under vacuum and refilled with N_2 three times, then heated to 155ºC. The reaction mixture changed from brown to turquoise green over 10 min. After stirring at 155ºC overnight (12-15h), the heat was ceased and the condenser was removed. After cooling to room temperature, the reaction mixture was filtered. The remaining dark green solid was washed with 10 mL of 1 M HCl, 10 mL of water, and 10 mL of EtOH. The catalyst was dried under high vacuum overnight at 90˚C to afford 638 mg (0.553 mmol) of $[FeCl_{16}Pc]Cl$ as a dark green powder (55% yield).

UV-Vis (H_2SO_4, c = 8.2 µM, λ_{max} = nm, $\varepsilon = M^{-1}cm^{-1}$): 797 (ε = 47290), 770 (ε = 47654), 317 (ε = 60784).; IR (ATR, cm^{-1}) 3258.78, 2323.85, 2162.01, 2050.37, 1980.01, 1736.67, 1561.44, 1501.78, 1391.32, 1302.37, 1273.49, 1212.62, 1156.98, 1091.91, 955.98, 856.36, 780.41, 770.26, 750.95, 647.41, 607.77, 511.89, 470.91.; HRMS (EI) *m/z* = 1111.46 (M-Cl).

Reaction Discovery: Traditional Diazo Sources

Preparation of diazoesters:

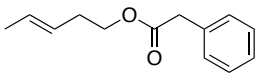
(*E*)-pent-3-en-1-yl 2-diazoacetate [1].



364 mg (2.14 mmol) of (*E*)-pent-3-en-1-yl 3-oxobutanoate was added to a 100 mL round-bottom flask, diluted with acetonitrile (21.4 mL, 0.1 M), and cooled to 0˚C. *p*ABSA (617 mg, 2.57 mmol, 1.2 equiv) and DBU (384 μL, 2.57 mmol, 1.2 equiv) were added and the reaction was allowed to warm to RT. After stirring for 12 hours, the reaction mixture was diluted with aq. NH_4Cl and extracted with diethyl ether. The organic extracts were combined, dried over $NaSO_4$, and filtered. After concentration, the crude yellow solid was triturated with a 50/50 mixture of pentane/ether, filtered, and the filtrate was concentrated. This crude mixture was then diluted with acetonitrile (10 mL) and cooled to 0˚C. 4% aq. KOH (40 mL) was added and the reaction mixture stirred vigorously until judged complete by TLC (1-5 h). The reaction mixture was extracted three times with diethyl ether, dried over $MgSO_4$, filtered and concentrated. Purification by flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 191 mg (1.24 mmol) of pure product as a yellow oil (58% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.53 (dqt, *J* = 15.4, 6.3, 1.3 Hz, 1H), 5.38 (dtq, *J* = 15.2, 6.8, 1.6 Hz, 1H), 4.73 (s, 1H), 4.15 (t, *J* = 6.9 Hz, 2H), 2.32 (qp, *J* = 6.8, 1.2 Hz, 2H), 1.66 (dq, *J* = 6.4, 1.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.80, 128.08, 126.12, 64.47, 46.17, 32.18, 18.00.; IR (ATR, cm^{-1}) 3121.57, 2961.99, 2919.82, 2858.19, 2105.68, 1686.59, 1453.75, 1394.35, 1354.67, 1337.81, 1237.29, 1179.08, 1155.09, 1080.75, 1025.21, 992.16, 965.71, 739.14, 553.27, 468.42.

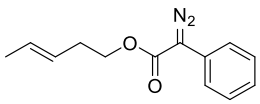
(*E*)-pent-3-en-1-yl 2-phenylacetate [S1].



Synthesized according to literature precedent[5]. (*E*)-pent-3-en-1-ol (689 mg, 8.0 mmol, 1.0 equiv), phenyl acetyl chloride (1.26 mL, 8.0 mmol, 1.0 equiv), pyridine (1.30 mL, 16 mmol, 2.0 equiv), DMAP (97.8 mg, 0.8 mmol, 0.1 equiv), and CH_2Cl_2 (16 mL, 0.5 M) were used. Flash column chromatography on silica using 0% → 5% ethyl acetate/hexanes as eluent gave 1.40 g (6.85 mmol) of pure product as a clear oil (86% yield).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.36 - 7.24 (m, 5H), 5.55 - 5.42 (m, 1H), 5.35 (dtq, J = 15.0, 6.6, 1.5 Hz, 1H), 4.10 (t, *J* = 6.8 Hz, 2H), 3.62 (s, 2H), 2.32-2.27 (m, 2H), 1.64 (dq, *J* = 6.4, 1.3 Hz, 3H).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.66, 134.27, 129.39, 128.63, 128.09, 127.14, 126.32, 64.56, 41.58, 32.05, 18.09.; HRMS (ESI) *m/z* calculated for $C_{13}H_{16}O_2Na$ $[M+Na]^+$: 227.1048, found 227.1051.

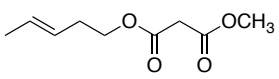
(*E*)-pent-3-en-1-yl 2-diazo-2-phenylacetate [2].



1.43 g (7.00 mmol) of (*E*)-pent-3-en-1-yl 2-phenylacetate **[S1]** was added to a 100 mL round-bottom flask, diluted with acetonitrile (70 mL, 0.1 M), and cooled to 0˚C. *p*ABSA (2.02 g, 8.40 mmol, 1.2 equiv) and DBU (1.57 mL, 10.5 mmol, 1.5 equiv) were added and the reaction was allowed to warm to RT. After stirring for 12 hours, the reaction mixture was diluted with aq. NH_4Cl and

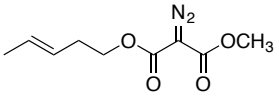
extracted with diethyl ether. The organic extracts were combined, dried over Na_2SO_4, and filtered. After concentration, the crude orange solid was triturated with a 50/50 mixture of pentane/ether, filtered, and the filtrate was concentrated. Flash column chromatography on silica using 5% diethyl ether/pentane as eluent gave 669 mg (2.91 mmol) of pure product as an orange oil (42% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.48 (m, 2H), 7.38 (m, 2H), 7.20-7.16 (m, 1H), 5.57 (dqt, *J* = 15.2, 6.3, 1.3 Hz, 1H), 5.42 (dtq, *J* = 15.3, 6.9, 1.5 Hz, 1H), 4.27 (t, *J* = 6.8 Hz, 2H), 2.38 (qp, *J* = 6.7, 1.2 Hz, 2H), 1.68 (dq, *J* = 6.4, 1.3 Hz, 3H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 165.27, 129.01, 128.36, 126.19, 125.85, 125.72, 124.05, 64.63, 32.31, 18.12; HRMS (ESI) *m/z* calculated for $C_{13}H_{15}O_2$ $[M+H-N_2]^+$: 203.1072, found 203.1073; IR (ATR, cm^{-1}) 3060.58, 3026.28, 2960.68, 2917.83, 2856.42, 2396.29, 2080.84, 1699.91, 1598.58, 1575.82, 1498.39, 1470.09, 1449.81, 1385.81, 1346.9, 1336.76, 1285.9, 1241.61, 1152.23, 1078.61, 1045.13, 1017.79, 997.6, 965.2, 904.04, 835.72, 752.9, 689.6, 666.78, 619.64, 555.99, 532.68, 493.98

(*E*)-methyl pent-3-en-1-yl malonate [S2].



1.08 g (8.0 mmol) of (*E*)-pent-3-en-1-ol, pyridine (1.21 mL, 16.0 mmol, 2 equiv), DMAP (97.7 mg, 0.8 mmol, 0.1 equiv) and CH_2Cl_2 were added to a flame-dried round-bottom flask and cooled to 0˚C. Methyl malonyl chloride (1.09 g, 8.0 mmol, 1 equiv) was added dropwise. The reaction mixture was slowly warmed to room temperature and stirred overnight. The reaction mixture was diluted with aq. NH_4Cl and extracted with CH_2Cl_2. The organic extracts were combined, dried over Na_2SO_4, and filtered. After concentration, the crude oil was purified by flash column chromatography on silica using 15% EtOAc/hexanes as eluent, giving 939 mg (5.0 mmol) of pure product as a clear oil (63% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.58-5.48 (m, 1H), 5.38 (dtq, *J* = 15.3, 6.9, 1.6 Hz, 1H), 4.14 (t, *J* = 6.9 Hz, 2H), 3.75 (s, 3H), 3.38 (s, 2H), 2.37-2.28 (m, 2H), 1.66 (dq, *J* = 6.4, 1.3 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 167.11, 166.58, 128.28, 125.99, 65.27, 52.59, 41.46, 31.85, 18.10.; HRMS (ESI) *m/z* calculated for $C_9H_{14}O_4Na$ $[M+Na]^+$: 209.0790, found 209.0786.

(*E*)-1-methyl 3-(pent-3-en-1-yl) 2-diazomalonate [3].



845 mg (4.54 mmol) of (*E*)-methyl pent-3-en-1-yl malonate [**S2**] was added to a 100 mL flame-dried round-bottom flask, diluted with acetonitrile (45 mL, 0.1 M), and cooled to 0˚C. *p*ABSA (1.31 mg, 5.44 mmol, 1.2 equiv) and DBU (815 μL, 5.44 mmol, 1.2 equiv) were added and the reaction was allowed to warm to RT. After stirring for 12 hours, the reaction mixture was diluted with aq. NH_4Cl and extracted with diethyl ether. The organic extracts were combined, dried over Na_2SO_4, and filtered. After concentration, the crude yellow solid was triturated with a 50/50 mixture of pentane/ether, filtered, and the filtrate was concentrated. Flash column chromatography on silica using 15% EtOAc/hexanes as eluent gave 812 mg (3.83 mmol) of pure product as a yellow oil (84% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.58 – 5.50 (m, 1H), 5.38 (dtq, *J* = 15.3, 6.9, 1.7 Hz, 1H), 4.22 (t, *J* = 6.9 Hz, 2H), 3.84 (s, 3H), 2.38-2.33 (m, *J* = 6.8, 1.2 Hz, 2H), 1.66 (dq, *J* = 6.4, 1.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.59, 160.70, 128.37, 125.76, 65.02, 52.48, 32.00, 17.96.; HRMS (ESI) *m/z* calculated for $C_9H_{12}N_2O_4Na$ $[M+Na]^+$: 235.0695, found 235.0698; IR (ATR,

cm^{-1}) 2957.88, 2857.93, 2132.69, 1759.74, 1733.99, 1692.34, 1436.48, 1387.26, 1319.59, 1270.24, 1202.92, 1181.13, 1080.91, 1014.16, 966.47, 830.65, 758.66, 732.78, 647.42, 543.79, 497.46.

General procedure for reaction discovery studies:
Into a 1 dram vial was added catalyst (0.040 mmol, 0.10 equiv) in a glovebox. The vial was sealed, taken out of the box, and the contents were added under a stream of nitrogen to a flame dried three neck 25 mL flask equipped with a stir bar, reflux condenser, glass stopper, and rubber septum. CH_2Cl_2 (5.0 mL, 0.08 M) was added and the flask was heated to reflux (~45˚C) for 10 minutes (4 hours for $FeCl_3$•pybox conditions[6]). Diazoester (0.40 mmol) was taken up with CH_2Cl_2 (3.0 mL, 0.13 M) in a glass syringe and added dropwise to the refluxing catalyst solution over the course 2-3 minutes (final reaction concentration is 0.05 M). After complete addition of diazoester, the reaction refluxed until judged complete by TLC (2-24 h). The reaction mixture was cooled, adsorbed onto SiO_2 or florisil, and applied directly to a silica column (75 mL SiO_2). Purification conditions are noted for individual entry.

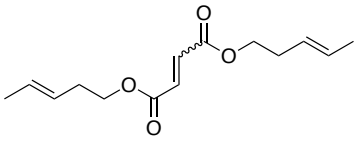
Acceptor diazoester:

$FeCl_3$•pybox Conditions: (*E*)-pent-3-en-1-yl 2-diazoacetate **[1]** (61.7 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (5.10 mg, 0.040 mmol, 0.10 equiv), 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine (14.8 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 5% → 10% → 15% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (61.6 mg, 0.399 mmol, 100% rsm). Run 2: (60.2 mg, 0.390 mmol, 98% rsm). **Average: 99% yield rsm.**

Fe(TPP)Cl Conditions: (E)-pent-3-en-1-yl 2-diazoacetate **[1]** (61.7 mg, 0.400 mmol, 1.0 equiv), Fe(TPP)Cl (28.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 5% → 10% → 15% EtOAc/hexanes as eluent gave recovered starting material (rsm) and dimer di(pent-3-en-1-yl) fumarate **[4]** as a mixture of olefin isomers.
Run 1: (47.9 mg, 0.190 mmol, 95% dimer), 0% rsm. Run 2: (45.9 mg, 0.182 mmol, 91% dimer), 0% rsm. **Average: 93% yield dimer [4], 0% rsm.**

[FePc]Cl Conditions: (*E*)-pent-3-en-1-yl 2-diazoacetate **[1]** (61.7 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 5% → 10% → 15% EtOAc/hexanes as eluent gave recovered starting material (rsm) and dimer di(pent-3-en-1-yl) fumarate **[4]** as a mixture of olefin isomers.
Run 1: (40.9 mg, 0.162 mmol, 81% dimer), 0% rsm. Run 2: (43.4 mg, 0.172 mmol, 86% dimer), 0% rsm. **Average: 84% yield dimer [4], 0% rsm.**

di(pent-3-en-1-yl) fumarate [4]



^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.23 (s, 2H), 5.54 (dqt, *J* = 15.4, 6.3, 1.3 Hz, 2H), 5.39 (dtq, *J* = 15.2, 6.8, 1.6 Hz, 2H), 4.18 (t, *J* = 7.0 Hz, 4H), 2.38-2.32 (m, 4H), 1.66 (dq, *J* = 6.4, 1.3 Hz, 6H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 165.32, 129.88, 128.24, 126.08, 65.05, 31.80, 18.12.; HRMS (ESI) *m/z* calculated for $C_{14}H_{20}O_4Na$ [M+Na]$^+$: 275.1259, found 275.1257.

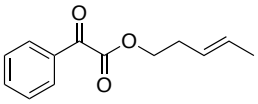
Donor/acceptor:

$FeCl_3$·pybox Conditions: (*E*)-pent-3-en-1-yl 2-diazo-2-phenylacetate **[2]** (92.1 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (5.10 mg, 0.040 mmol, 0.10 equiv), 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine (14.8 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (85.1 mg, 0.369 mmol, 92% rsm). Run 2: (80.1 mg, 0.348 mmol, 87% rsm). **Average: 90% rsm.**

Fe(TPP)Cl Conditions: (*E*)-pent-3-en-1-yl 2-diazo-2-phenylacetate **[2] (**92.1 mg, 0.400 mmol), FeTPPCl (28.2 mg, 0.040 mmol, 0.1 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Flash column chromatography on silica using 0% petroleum ether to 10% Et_2O/petroleum ether as eluent gave recovered starting material (rsm) and ketone (*E*)-pent-3-en-1-yl 2-oxo-2-phenylacetate **[5]**. Yields partially determined via NMR with mesitylene as an internal standard.
Run 1: (3.5 mg, 0.016 mmol, 4% ketone), (88.9 mg, 0.386 mmol, 97% rsm). Run 2: (2.8 mg, 0.013 mmol, 3% ketone), (85.9 mg, 0.373 mmol, 93% rsm). **Average: 4% yield ketone [5], 95% rsm**.

[FePc]Cl Conditions: (*E*)-pent-3-en-1-yl 2-diazo-2-phenylacetate **[2] (**92.1 mg, 0.400 mmol), FePcCl (24.2 mg, 0.040 mmol, 0.1 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Flash column chromatography on silica using 0% to 10% Et_2O/petroleum ether as eluent gave recovered starting material (rsm) and ketone (*E*)-pent-3-en-1-yl 2-oxo-2-phenylacetate **[5]**. Yields partially determined via NMR with mesitylene as an internal standard.
Run 1: (10.4 mg, 0.048 mmol, 12% ketone), (53.4 mg, 0.232 mmol, 58% rsm). Run 2: (10.4 mg, 0.048 mmol, 12% ketone), (53.4 mg, 0.232 mmol, 58% rsm). **Average: 12% yield ketone [5], 58% rsm**.

(*E*)-pent-3-en-1-yl 2-oxo-2-phenylacetate [5].



^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.68-7.64 (m, 1H), 7.51 (dd, *J* = 8.3, 7.4 Hz, 2H), 5.65 - 5.54 (m, 1H), 5.44 (dtq, *J* = 15.3, 6.8, 1.6 Hz, 1H), 4.40 (t, *J* = 6.8 Hz, 2H), 2.51 - 2.41 (m, 2H), 1.68 (dq, *J* = 6.4, 1.4 Hz, 3H).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.56, 164.01, 135.02, 132.61, 130.19, 129.00, 128.96, 125.69, 65.86, 32.01, 18.14. HRMS (ESI) *m/z* calculated for $C_{13}H_{14}O_3Na$ [M+Na]$^+$: 241.0841, found 241.0849.

Acceptor/acceptor:

$FeCl_3$·pybox Conditions: (*E*)-1-methyl 3-(pent-3-en-1-yl) 2-diazomalonate **[3]** (84.9 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (5.10 mg, 0.040 mmol, 0.10 equiv), 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine(14.8 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (81.9 mg, 0.386 mmol, 97% rsm). Run 2: (81.7 mg, 0.385 mmol, 96% rsm). **Average: 97% yield rsm.**

Fe(TPP)Cl Conditions: (*E*)-1-methyl 3-(pent-3-en-1-yl) 2-diazomalonate **[3]** (84.9 mg, 0.400 mmol, 1.0 equiv), Fe(TPP)Cl (28.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (78.7 mg, 0.371 mmol, 93% rsm). Run 2: (83.0 mg, 0.391 mmol, 98% rsm). **Average: 96% rsm.**

[FePc]Cl Conditions: (*E*)-1-methyl 3-(pent-3-en-1-yl) 2-diazomalonate **[3]** (84.9 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (78.5 mg, 0.370 mmol, 93% rsm). Run 2: (82.2 mg, 0.387 mmol, 97% rsm). **Average: 95% rsm.**

Acceptor/acceptor Sulfonate Ester:

$FeCl_3$·pybox Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (5.10 mg, 0.040 mmol, 0.10 equiv), 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine(14.8 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (105.6 mg, 0.382 mmol, 96% rsm). Run 2: (110.6 mg, 0.400 mmol, 100% rsm). **Average: 98% yield rsm.**

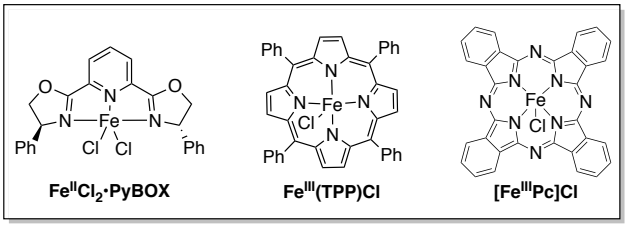
Fe(TPP)Cl Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), Fe(TPP)Cl (28.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave recovered starting material (rsm).
Run 1: (108.1 mg rsm, 0.391 mmol, 98%). Run 2: (108.0 mg rsm, 0.391 mmol, 98%). **Average: 0% yield, 98% rsm.**

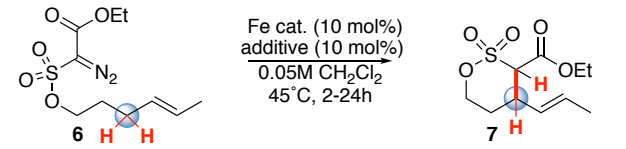
[FePc]Cl Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05

M) were used. Flash column chromatography on silica using 10% → 15% → 20% EtOAc/hexanes as eluent gave recovered starting material (rsm) and ethyl (*E*)-4-(prop-1-en-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide **[7]** as a mixture of diastereomers.
Run 1: (3.4 mg, 0.014 mmol, 3%), (107.1 mg rsm, 0.388 mmol, 97%). Run 2: (2.4 mg, 0.010 mmol, 2%), (107.0 mg rsm, 0.387 mmol, 97%). **Average: 3% yield [7], 97% rsm.**

Optimization of Fe-Catalyzed Intramolecular C—H Alkylation

Table S1:





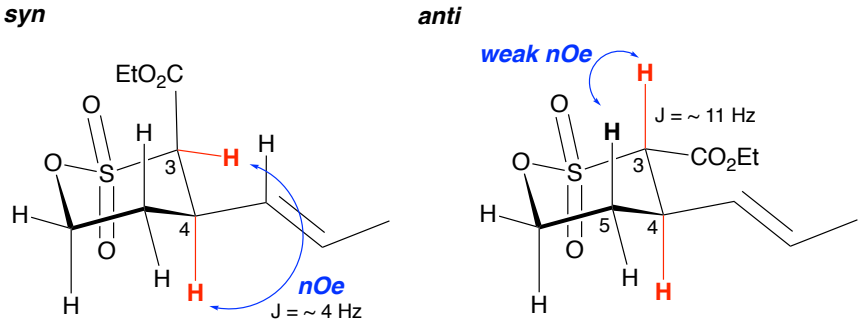
entry	catalyst	additive	% yield (% rsm)
1	[Fe^{III}Pc]Cl	–	3 (97)
2	[Fe^{III}Pc]Cl[a]	$AgSbF_6$	45 (0)
3	[Fe^{III}Pc]Cl[a]	$NaBAr^F_4$	48 (0)
4	[Fe^{III}Cl$_{16}$Pc]Cl	$NaBAr^F_4$	38 (0)
5	[Fe^{III}Cl$_8$Pc]Cl	$NaBAr^F_4$	25 (66)
6	[Fe^{III}Pc]Cl[a,b]	$NaBAr^F_4$	53 (0)
7	Fe^{III}(TPP)Cl	$NaBAr^F_4$	7 (93)
8	[Fe^{II}Pc]	–	0 (100)
9	–	$NaBAr^F_4$	0 (96)

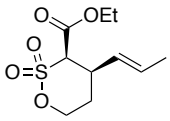
Reaction optimization. Reaction stirred for 24 hours after addition of diazoester unless otherwise noted. [a] Reaction stirred for 2 hours after dropwise addition of diazoester. [b] Diazo added via syringe pump over one hour. Pc = phthalocyanine; TPP = tetraphenylporphyrin; PyBOX = 2,6-bis[(4*R*)-4-phenyl-2-oxazolinyl]pyridine; $AgSbF_6$ = silver hexafluoroantimonate; $NaBAr^F_4$ = sodium tetrakis[3,5-bis(trifluoromethyl)phenyl]borate. Isolated yields are average of two runs. rsm denotes recovered starting material.

Ethyl (*E*)-4-(prop-1-en-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [7].

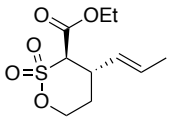
The relative stereochemistry of the δ-sultones was determined via coupling constant analysis, and confirmed by nOe NMR experiments (500 MHz, $CDCl_3$) in which the C3 and C4 protons for each diastereomer were irradiated (highlighted in red). Syn δ-sultones have a characteristic nOe between the C3 and C4 equatorial and pseudo-axial hydrogens. Conversely, the C3 and C4 hydrogens in the anti δ-sultones fall on opposite sides of the ring and therefore experience

no observable nOe. Instead, for anti allylic C—H alkylation products a weak nOe can be observed between the C3 pseudo-axial hydrogen and the pseudo-axial C5 hydrogen. All products described in this paper are designated with the same relative stereochemistry as **7** as determined by coupling constant analogy. The experimental data are consistent with previously reported studies in the literature [3, 7]. The syn and anti diastereomers were collected from initial purification of crude reaction as a mixture (d.r. 1:1-3:1 syn:anti) and subsequently purified for characterization. In most, but not all, cases, the syn diastereomer was less polar than the anti diastereomer.





Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.68 (dqd, *J* = 15.0, 6.5, 1.1 Hz, 1H), 5.33 (ddd, *J* = 15.3, 7.6, 1.7 Hz, 1H), 4.68-4.55 (m, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 4.05 (d, *J* = 4.7 Hz, 1H$_{C3}$), 3.28-3.18 (m, 1H), 2.61 (dtd, *J* = 14.5, 12.0, 5.6 Hz, 1H), 1.68 (ddd, *J* = 6.5, 1.7, 0.8 Hz, 3H), 1.64 (ddt, *J* = 14.6, 4.3, 2.4 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.11, 129.69, 127.73, 73.03, 64.90, 62.37, 40.59, 25.40, 17.99, 14.10.; HRMS (ESI) *m/z* calculated for $C_{10}H_{17}O_5S$ $[M+H]^+$: 249.0797, found 249.0795.



Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.68 (dqd, *J* = 13.9, 6.5, 0.9 Hz, 1H), 5.23 (ddd, *J* = 15.3, 8.3, 1.7 Hz, 1H), 4.70-4.61 (m, 1H), 4.52-4.44 (m, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.78 (d, *J* = 11.6 Hz, 1H$_{C3}$), 3.25 (dq, *J* = 11.4, 8.2 Hz, 1H), 1.92-1.84 (m, 2H), 1.66 (dd, *J* = 6.4, 1.4 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.35, 130.21, 127.93, 72.54, 68.19, 62.67, 41.70, 30.44, 17.98, 14.16.; HRMS (ESI) *m/z* calculated for $C_{10}H_{17}O_5S$ $[M+H]^+$: 249.0797, found 249.0796.

General procedure for optimization studies (entries 1-9):

Into a 1 dram vial was added catalyst (0.040 mmol, 0.10 equiv) in a glovebox. The vial was sealed, taken out of the box, and the contents were added under a stream of nitrogen to a flame dried three neck 25 mL flask equipped with a stir bar, reflux condenser, glass stopper, and rubber septum. CH_2Cl_2 (5.0 mL, 0.08 M) was added and the flask was heated to reflux (~45˚C) for 10 minutes. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **6** (110.5 mg, 0.400 mmol, 1.0 equiv) was taken up with CH_2Cl_2 (3.0 mL, 0.13 M) in a glass syringe and added dropwise to the refluxing catalyst solution over the course of 2-3 minutes (final reaction concentration is 0.05 M). After complete addition of diazosulfonate, the reaction refluxed until judged complete by TLC (2-24 h). The reaction mixture was cooled, adsorbed onto SiO_2 or florisil, and applied directly to a silica column (75 mL SiO_2) for purification to afford the desired

sultone product. The recovered starting material (rsm) and product were eluted with 10% → 15% → 20% EtOAc/hexanes and isolated separately. Any variation of these reaction conditions is noted for individual entries.

Entry 1. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (3.4 mg, 0.014 mmol, 3%), (107.1 mg rsm, 0.388 mmol, 97%). Run 2: (2.4 mg, 0.010 mmol, 2%), (107.0 mg rsm, 0.387 mmol, 97%). **Average: 3% yield, 97% rsm.**

Entry 2. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $AgSbF_6$ (13.7 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (45.5 mg, 0.183 mmol, 46%). Run 2: (44.4 mg, 0.179 mmol, 45%). **Average: 45% yield, 0% rsm.**

Entry 3. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (47.7 mg, 0.192 mmol, 48%). Run 2: (46.3 mg, 0.186 mmol, 47%). **Average: 48% yield, 0% rsm.**

Entry 4. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [$FeCl_{16}$Pc]Cl (46.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (35.1 mg, 0.141 mmol, 35%). Run 2: (39.6 mg, 0.159 mmol, 40%). **Average: 38% yield, 0% rsm.**

Entry 5. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [$FeCl_8$Pc]Cl (35.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (24.3 mg, 0.098 mmol, 25%), (73.7 mg rsm, 0.267 mmol, 67%). Run 2: (26.0 mg, 0.105 mmol, 26%), (72.6 mg rsm, 0.263 mmol, 66%). **Average: 25% yield, 66% rsm.**

Entry 6. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Solution of diazo was added via syringe pump over the course of an hour (see General procedure for [FePc]-catalyzed C—H alkylation).
Run 1: (53.4 mg, 0.215 mmol, 54%). Run 2: (51.9 mg, 0.209 mmol, 53%). **Average: 53% yield, 0% rsm.**

Entry 7. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), Fe(TPP)Cl (28.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.

Run 1: (7.5 mg, 0.030 mmol, 7%), (103.2 mg rsm, 0.373 mmol, 93%). Run 2: (6.3 mg, 0.025 mmol, 6%), (104.7 mg rsm, 0.379 mmol, 94%). **Average: 7% yield, 93% rsm.**

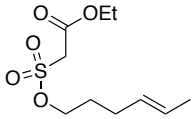
Entry 8. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), [$Fe^{II}Pc$] (22.7 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Run 1: (108.9 mg rsm, 0.394 mmol, 99%). Run 2: (110.2 mg RSM, 0.399 mmol, 100%). **Average: 0% yield, 100% rsm.**

Entry 9. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (105.7 mg rsm, 0.382 mmol, 96%). Run 2: (106.7 mg rsm, 0.386 mmol, 97%). **Average: 0% yield, 96% rsm.**

Preparation of Sulfonate Ester Starting Materials

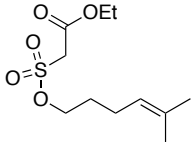
General procedure for preparation of sulfonate ester substrates:
A round-bottom flask equipped with a stir bar and rubber septum was charged with alcohol substrate (1 equiv), imidazole (1.2 equiv), and THF (0.4 M). Ethyl chlorosulfonylacetate (1.5 equiv) was dissolved in THF (0.4 M) and added slowly dropwise to the reaction over the course of 5 mins. The reaction was stirred at room temperature for 1-6 hours, or until complete consumption of starting material as monitored by TLC. The reaction was quenched with H_2O until the mixture turned clear (10-20 mL). The reaction mixture was partitioned between H_2O (15 mL) and CH_2Cl_2 (60 mL) and separated. The aqueous layer was then extracted with CH_2Cl_2 (2x20 mL). The organic layers were combined, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Purification by column chromatography on silica gel afforded the desired sulfonate ester.

Ethyl (*E*)-2-((hex-4-en-1-yloxy)sulfonyl)acetate [S3].



1.00 g (10.0 mmol) of (*E*)-hex-4-en-1-ol were used, along with imidazole (817 mg, 12.0 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (2.80 g, 15.0 mmol, 1.5 equiv), and THF (50 mL, 0.2 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 2.30 g (9.20 mmol) of pure product as a pale yellow oil (92% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.54-5.43 (m, 1H), 5.38 (dt, *J* = 15.2, 6.6 Hz, 1H), 4.33 (t, *J* = 6.5 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.07 (s, 2H), 2.11 (dt, *J* = 7.3, 6.6 Hz, 2H), 1.82 (p, *J* = 6.7 Hz, 2H), 1.65 (d, *J* = 6.3 Hz, 3H), 1.32 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.02, 129.02, 126.58, 71.77, 62.60, 54.67, 28.86, 28.14, 17.84, 13.90.; HRMS (ESI) *m/z* calculated for $C_{10}H_{18}O_5SNa$ $[M+Na]^+$: 273.0773, found 273.0771.

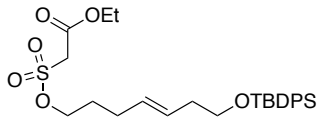
Ethyl 2-(((5-methylhex-4-en-1-yl)oxy)sulfonyl)acetate [S4].



0.57 g (5.0 mmol) of 5-methylhex-4-en-1-ol were used, along with imidazole (409 mg, 6.0 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.40 g, 7.5 mmol, 1.5 equiv), and THF (25 mL, 0.2 M). Flash column chromatography on silica using 15% EtOAc/hexanes as eluent gave 0.89 mg (3.40 mmol) of pure product as a clear

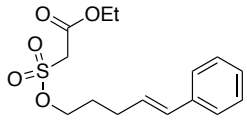
oil (68% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.08 (t, *J* = 7.2 Hz, 1H), 4.33 (t, *J* = 6.5 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.07 (s, 2H), 2.11 (dt, *J* = 7.3, 7.1 Hz, 2H), 1.80 (p, *J* = 6.8 Hz, 2H), 1.70 (s, 3H), 1.61 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) 162.14, 133.53, 122.37, 72.06, 62.84, 54.97, 29.41, 25.84, 23.85, 17.84, 14.10; HRMS (ESI) *m/z* calculated for $C_{11}H_{20}O_5SNa$ $[M+Na]^+$: 287.0929, found 287.0927.

Ethyl (*E*)-2-(((7-((*tert*-butyldiphenylsilyl)oxy)hept-4-en-1-yl)oxy)sulfonyl)acetate [S5].



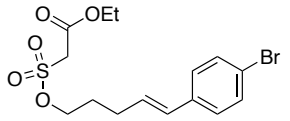
839 mg (2.30 mmol) of (*E*)-7-((*tert*-butyldiphenylsilyl)oxy)hept-4-en-1-ol were used, along with imidazole (191 mg, 2.80 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (653 mg, 3.50 mmol, 1.5 equiv), and THF (12 mL, 0.2 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 1.20 g (2.30 mmol) of pure product as a pale yellow oil (100% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ7.69-7.63 (m, 4H), 7.45-7.34 (m, 6H), 5.44 (m, 2H), 4.32 (t, *J* = 6.4 Hz, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 4.06 (s, 2H), 3.67 (t, *J* = 6.6 Hz, 2H), 2.25 (q, *J* = 6.4 Hz, 2H), 2.11 (q, *J* = 6.9 Hz, 2H), 1.81 (p, *J* = 6.5 Hz, 2H), 1.31 (t, *J* = 7.3 Hz, 3H), 1.04 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.07, 135.63, 134.03, 130.11, 129.64, 128.55, 127.68, 71.83, 63.82, 62.73, 54.85, 36.04, 28.96, 28.38, 26.93, 19.30, 14.04.; HRMS (ESI) *m/z* calculated for $C_{27}H_{39}O_6SiS$ $[M+H]^+$: 519.2237, found 519.2239.

Ethyl (*E*)-2-(((5-phenylpent-4-en-1-yl)oxy)sulfonyl)acetate [S6].



825 mg (5.10 mmol) of (*E*)-5-phenylpent-4-en-1-ol were used, along with imidazole (415 mg, 6.10 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.40 g, 7.70 mmol, 1.5 equiv), and THF (26 mL, 0.2 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 1.47 g (4.70 mmol) of pure product as a pale yellow oil (92% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.36-7.32 (m, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.24-7.19 (m, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.18 (dt, *J* = 15.9, 7.0 Hz, 1H), 4.40 (t, *J* = 6.4 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.09 (s, 2H), 2.36 (dt, *J* = 8.3, 7.1 Hz, 2H), 1.96 (p, *J* = 6.6 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 161.89, 137.15, 131.14, 131.11, 128.37, 128.24, 128.19, 127.00, 125.86, 71.48, 62.42, 54.46, 28.57, 28.47, 13.76.; HRMS (ESI) *m/z* calculated for $C_{15}H_{20}O_5SNa$ $[M+Na]^+$: 335.0929, found 335.0929.

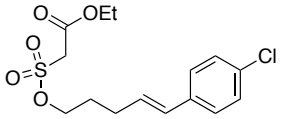
Ethyl (*E*)-2-(((5-(4-bromophenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate [S7].



735 mg (3.05 mmol) of (E)-hex-4-en-1-ol were used, along with imidazole (249 mg, 3.7 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (854 mg, 4.58 mmol, 1.5 equiv), and THF (15 mL, 0.2 M). Flash column chromatography on silica using 15% → 20% → 25% EtOAc/hexanes as eluent gave 950 mg (2.42 mmol) of pure product as a clear oil (79% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.17 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.39 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 2.35 (dt, *J* = 7.7, 6.8 Hz, 2H), 1.95 (p, *J* = 6.5 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.26, 136.47, 131.84, 130.54, 129.40, 127.84, 121.09, 71.80,

62.99, 55.08, 28.96, 28.94, 14.23; HRMS (ESI) *m/z* calculated for $C_{15}H_{19}BrO_5SNa$ $[M+Na]^+$: 413.0034; found 413.0026.

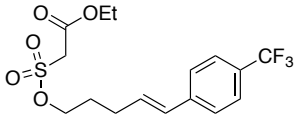
Ethyl (*E*)-2-(((5-(4-chlorophenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate [S8].



610 mg (3.10 mmol) of (*E*)-5-(4-chlorophenyl)pent-4-en-1-ol were used, along with imidazole (253 mg, 3.72 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (868 mg, 4.65 mmol, 1.5 equiv), and THF (16 mL, 0.2 M). Flash column chromatography on silica using 15% EtOAc/hexanes as eluent gave 846 mg (2.40 mmol) of pure product as a pale yellow oil (77% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.31- 7.23 (m, 4H), 6.40 (d, *J* = 15.9, 1H), 6.15 (dt, *J* = 15.6, 6.9 Hz, 1H), 4.39 (t, *J* = 6.4 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.09 (s, 2H), 2.36 (dt, *J* = 7.4, 7.0 Hz, 2H), 1.95 (p, *J* = 6.4 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.06, 135.87, 132.76, 130.29, 129.09, 128.70, 127.32, 71.60, 62.76, 54.89, 28.80, 28.72, 14.03; HRMS (ESI) *m/z* calculated for $C_{15}H_{19}O_5SClNa$ $[M+Na]^+$: 369.0530, found 369.0533.

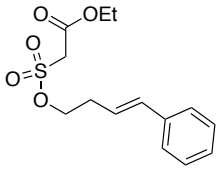
Ethyl(*E*)-2-(((5-(4-(trifluoromethyl)phenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate [S9].



890 mg (3.88 mmol) of (E)-hex-4-en-1-ol were used, along with imidazole (317 mg, 4.66 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.09 g, 5.82 mmol, 1.5 equiv), and THF (19 mL, 0.2 M). Flash column chromatography on silica using 25% → 30% → 33% EtOAc/hexanes as eluent gave 772 mg (2.03 mmol) of pure product as a clear oil (52% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ7.55 (d, *J* = 8.2 Hz, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.29 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.41 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.10 (s, 2H), 2.40 (dt, *J* = 7.3, 6.8 Hz, 2H), 1.97 (p, *J* = 6.5 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.27, 140.99, 131.36, 130.48, 129.20 (q, *J* = 32.4 Hz), 126.42, 125.72 (q, *J* = 3.8 Hz), 124.47 (q, *J* = 272.2 Hz), 71.75, 63.01, 55.08, 28.99, 28.88, 14.20. HRMS (ESI) m/z calculated for $C_{16}H_{19}F_3O_5SNa$ $[M+Na]^+$: 403.0803, found 403.0788.

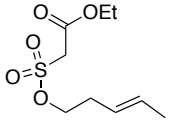
Ethyl (*E*)-2-(((4-phenylbut-3-en-1-yl)oxy)sulfonyl)acetate [S10].



764 mg (5.20 mmol) of (*E*)-4-phenylbut-3-en-1-ol were used, along with imidazole (422 mg, 6.20 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.46 g, 7.80 mmol, 1.5 equiv), and THF (26 mL, 0.2 M). Flash column chromatography on silica using 15% EtOAc/hexanes as eluent gave 1.38 g (4.60 mmol) of pure product as a pale yellow oil (88% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.37-7.33 (m, 2H), 7.33-7.28 (m, 2H), 7.25-7.21 (m, 1H), 6.52 (d, *J* = 15.8 Hz, 1H), 6.16 (dt, *J* = 15.9, 7.0 Hz, 1H), 4.45 (t, *J* = 6.7 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 4.10 (s, 2H), 2.69 (qd, *J* = 6.7, 1.5 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.07, 136.89, 133.68, 128.63, 127.63, 126.25, 123.66, 71.40, 62.78, 54.97, 32.90, 14.00.; HRMS (ESI) *m/z* calculated for $C_{14}H_{18}O_5SNa$ $[M+Na]^+$: 371.0773, found 321.0767.

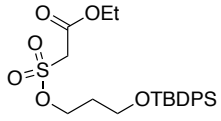
Ethyl (*E*)-2-((pent-3-en-1-yloxy)sulfonyl)acetate [S11].



431 mg (5.0 mmol) of (*E*)-pent-3-en-1-ol were used, along with imidazole (409 mg, 6.0 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.40 g, 7.50 mmol, 1.5 equiv), and THF (25 mL, 0.2 M). Flash column chromatography on silica using 10%

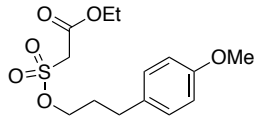
EtOAc/hexanes as eluent gave 807 mg (3.40 mmol) of pure product as a pale yellow oil (68% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.65-5.54 (m, 1H), 5.38 (dtq, *J* = 15.4, 6.9, 1.7 Hz, 1H), 4.32 (t, *J* = 6.8 Hz, 2H), 4.28 (q, *J* = 7.4 Hz, 2H), 4.08 (s, 2H), 2.45 (m, 2H), 1.68 (dq, *J* = 6.7, 1.4 Hz, 3H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.05, 129.40, 124.56, 71.83, 62.66, 54.82, 32.44, 17.98, 13.94.; HRMS (ESI) *m/z* calculated for $C_9H_{16}O_5SNa$ $[M+Na]^+$: 259.0616, found 259.0609.

Ethyl 2-((3-((*tert*-butyldiphenylsilyl)oxy)propoxy)sulfonyl)acetate [S12].



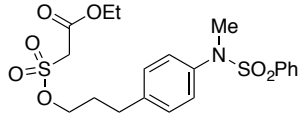
1.57 g (5.0 mmol) of 3-((*tert*-butyldiphenylsilyl)oxy)propan-1-ol were used, along with imidazole (409 mg, 6.0 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.4 g, 7.5 mmol, 1.5 equiv), and THF (25 mL, 0.2 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 1.16 g (2.50 mmol) of pure product as a sticky pale yellow oil (50% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.70-7.59 (m, 4H), 7.47-7.34 (m, 6H), 4.53 (t, *J* = 6.3 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 4.05 (s, 2H), 3.78 (t, *J* = 5.8 Hz, 2H), 1.98 (p, *J* = 6.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.05 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.03, 135.55, 133.39, 129.83, 127.81, 69.36, 62.69, 59.31, 54.75, 32.14, 26.87, 19.23, 14.01.; HRMS (ESI) *m/z* calculated for $C_{23}H_{33}O_6SiS$ $[M-H]^+$: 463.1611, found 463.1601.

Ethyl 2-((3-(4-methoxyphenyl)propoxy)sulfonyl)acetate [S13].



1.58 g (5.00 mmol) of 3-(4-methoxyphenyl)propan-1-ol were used, along with imidazole (408.5 mg, 6 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.40 g, 7.50 mmol, 1.5 equiv), and THF (25 mL, 0.2 M). Flash column chromatography on silica using 10% → 20% EtOAc/hexanes as eluent gave 855 mg (2.70 mmol) of pure product as a clear oil (53% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.09 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 4.31 (t, J = 6.3 Hz, 2H), 4.25 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 3.76 (s, 3H), 2.68 (t, J = 7.6 Hz, 2H), 2.03 (p, J = 7.6 Hz, 2H), 1.30 (t, J = 7.2, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.08, 158.15, 132.33, 129.45, 114.05, 71.54, 62.79, 55.33, 54.86, 31.04, 30.58, 14.05.; HRMS (ESI) *m/z* calculated for $C_{14}H_{20}O_6SNa$ $[M+Na]^+$: 339.0878, found 339.0872.

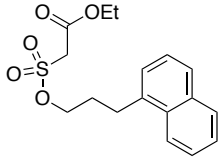
Ethyl 2-((3-(4-(*N*-methylphenylsulfonamido)phenyl)propoxy)sulfonyl)acetate [S14].



1.03 g (3.37 mmol) of *N*-(4-(3-hydroxypropyl)phenyl)-*N*-methylbenzenesulfonamide was used, along with imidazole (275.7 mg, 4.05 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (944.2 mg, 5.06 mmol, 1.5 equiv), and THF (17 mL, 0.2 M). Flash column chromatography on silica using 20% → 50% EtOAc/hexanes as eluent gave 802 mg (1.76 mmol) of pure product as a clear oil (52% yield).
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.62 - 7.51 (m, 3H), 7.69 - 7.43 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 4.34 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.09 (s, 2H), 3.15 (s, 3H), 2.74 (t, *J* = 7.6 Hz, 2H), 2.12 - 2.04 (m, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.13, 139.84, 139.74, 136.60, 132.90, 129.06, 128.89, 128.00, 126.97, 71.40,

62.93, 55.04, 38.32, 31.13, 30.74, 14.13.; HRMS (ESI) *m/z* calculated for $C_{20}H_{26}NO_7S_2$ $[M+H]^+$: 456.1151, found 456.1149.

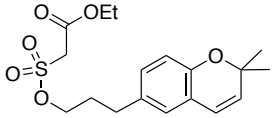
Ethyl 2-((3-(naphthalen-1-yl)propoxy)sulfonyl)acetate [S15].



175 mg (0.939 mmol) of 3-(naphthalen-1-yl)propan-1-ol were used, along with imidazole (76.7 mg, 1.13 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (263 mg, 1.41 mmol, 1.5 equiv), and THF (4.7 mL, 0.2 M). Flash column chromatography on silica using 10% → 25% EtOAc/hexanes as eluent gave 118 mg (0.351 mmol) of pure product as a clear oil (37% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.01 (d, J = 8.5 Hz, 1H), 7.87 (d, J = 7.5 Hz, 1H), 7.74 (d, J = 7.8 Hz, 1H), 7.55 - 7.47 (m, 2H), 7.41 (dd, *J* = 8.2, 7.0 Hz, 1H), 7.35 (dd, *J* = 7.1, 1.5 Hz, 1H), 4.42 (t, *J* = 6.2 Hz, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 4.11 (s, 2H), 3.23 (dd, *J* = 8.5, 6.8 Hz, 2H), 2.27 - 2.17 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.14, 136.44, 134.11, 131.76, 129.06, 127.32, 126.49, 126.24, 125.79, 125.69, 123.58, 71.82, 62.92, 55.07, 30.20, 28.84, 14.13.; HRMS (ESI) *m/z* calculated for $C_{17}H_{20}O_5SNa$ $[M+Na]^+$: 359.0929, found 359.0927.

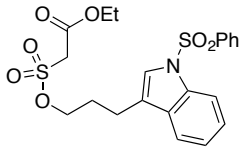
Ethyl 2-((3-(2,2-dimethyl-2*H*-chromen-6-yl)propoxy)sulfonyl)acetate [S16].



744 mg (3.41 mmol) of 3-(2,2-dimethyl-2*H*-chromen-6-yl)propan-1-ol was used, along with imidazole (279 mg, 4.10 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (955 mg, 5.12 mmol, 1.5 equiv), and THF (17 mL, 0.2 M). Flash column chromatography on silica using 20% → 50% EtOAc/hexanes as eluent gave 896 mg (2.43 mmol) of pure product as a clear oil (71% yield).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 6.91 (d, J = 8.4 Hz, 1H), 6.79 (s, 1H), 6.70 (d, *J* = 8.1 Hz, 1H), 6.28 (d, J = 9.6 Hz, 1H), 5.60 (d, *J* = 9.5 Hz, 1H), 4.33 (t, J = 6.4 Hz, 2H), 4.27 (q, J = 7.2 Hz, 2H), 4.07 (s, 2H), 2.64 (t, *J* = 7.5 Hz, 2H), 2.05 (p, J = 7.2 Hz, 2H), 1.42 (s, 6H), 1.32 (t, J = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.09, 151.45, 132.40, 131.11, 128.99, 126.32, 122.30, 121.36, 116.40, 76.21, 71.59, 62.82, 54.94, 31.00, 30.69, 28.06, 14.08.; HRMS (ESI) *m/z* calculated for $C_{18}H_{25}O_6S$ $[M+H]^+$: 369.1372, found 369.1364.

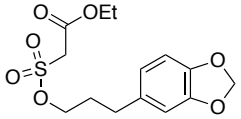
Ethyl 2-((3-(1-(phenylsulfonyl)-1H-indol-3-yl)propoxy)sulfonyl)acetate [S17].



2.30 g (7.28 mmol) of 3-(1-(phenylsulfonyl)-1H-indol-3-yl)propan-1-ol was used, along with imidazole (595 mg, 8.74 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (2.04 g, 10.9 mmol, 1.5 equiv), and THF (18 mL, 0.2 M). Flash column chromatography on silica using 10% → 30% EtOAc/hexanes as eluent gave 2.40 g (5.16 mmol) of pure product as a clear oil (71% yield). This compound was isolated with <5% of an unknown impurity that did not appear to affect reactivity.

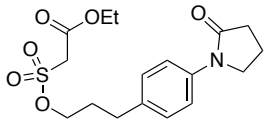
^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 7.2 Hz, 2H), 7.53 (t, J = 7.5 Hz, 1H), 7.49 - 7.38 (m, 4H), 7.33 (t, *J* = 8.0, 1H), 7.24 (m, 1H), 4.35 (t, *J* = 6.1 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.10 (s, 2H), 2.83 (t, J = 7.3 Hz, 2H), 2.14 (p, *J* = 7.5 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.13, 138.25, 135.54, 133.88, 130.71, 129.39, 126.87, 125.06, 123.38, 121.38, 119.46, 113.99, 71.20, 62.94, 55.03, 28.57, 20.86, 14.11. (one peak missing due to overlap); HRMS (ESI) *m/z* calculated for $C_{21}H_{24}NO_7S_2$ $[M+H]^+$: 446.0994, found 446.0993.

Ethyl 2-((3-(benzo[*d*][1,3]dioxol-5-yl)propoxy)sulfonyl)acetate [S18].



1.26 g (7.0 mmol) of 3-(benzo[*d*][1,3]dioxol-5-yl)propan-1-ol were used, along with imidazole (571 mg, 8.40 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.90 g, 10.5 mmol, 1.5 equiv), and THF (35 mL, 0.2 M). Flash column chromatography on silica using 20% EtOAc/hexanes as eluent gave 1.82 g (5.50 mmol) of pure product as a pale yellow oil (79% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.74 (d, *J* = 7.9 Hz, 1H), 6.68 (d, *J* = 1.5 Hz, 1H), 6.64 (dd, *J* = 7.9, 1.8 Hz, 1H), 5.93 (s, 2H), 4.33 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 2.68 (t, *J* = 7.5 Hz, 2H), 2.04 (p, *J* = 6.4 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.11, 147.89, 146.12, 134.13, 121.41, 108.95, 108.43, 101.00, 71.42, 62.87, 55.00, 31.31, 31.14, 14.11.; HRMS (ESI) *m/z* calculated for $C_{14}H_{18}O_7SNa$ $[M+Na]^+$: 353.0671, found 353.0672.

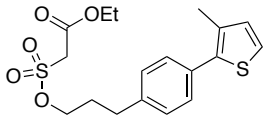


Ethyl 2-((3-(4-(2-oxopyrrolidin-1-yl)phenyl)propoxy)sulfonyl)acetate [S19].

414 mg (1.12 mmol) of 1-(4-(3-hydroxypropyl)phenyl)pyrrolidin-2-one was used, along with imidazole (91.1 mg, 1.34 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (312 mg, 1.67 mmol, 1.5 equiv), and THF (5.6 mL, 0.2 M). Flash column chromatography on silica using 30% → 70% EtOAc/hexanes as eluent gave 245 mg (0.771 mmol) of pure product as a clear oil (69% yield).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.6 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H), 4.34 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.08 (s, 2H), 3.85 (t, *J* = 7.0 Hz, 2H), 2.74 (t, J = 7.5 Hz, 2H), 2.61 (t, *J* = 8.1 Hz, 2H), 2.17 (p, J = 7.8 Hz, 2H), 2.08 (p, J = 7.0 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 174.25, 162.06, 137.75, 136.52, 128.87, 120.31, 71.44, 62.82, 54.86, 48.91, 32.75, 30.91, 30.84, 18.10, 14.06.; HRMS (ESI) *m/z* calculated for $C_{17}H_{24}O_6NS$ $[M+H]^+$: 370.1324, found 370.1325.

Ethyl 2-((3-(4-(3-methylthiophen-2-yl)phenyl)propoxy)sulfonyl)acetate [S20].



1.06 g (4.56 mmol) of 3-(4-(3-methylthiophen-2-yl)phenyl)propan-1-ol were used, along with imidazole (373 mg, 5.47 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.28 g, 6.84 mmol, 1.5 equiv), and THF (24.8 mL, 0.2 M). Flash column chromatography on silica using 10% → 20% EtOAc/hexanes as eluent gave 1.55 g (4.04 mmol) of pure product as a clear oil (89% yield).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 5.1 Hz, 1H), 6.97 (d, *J* = 5.1 Hz, 1H), 4.43 (t, *J* = 6.3 Hz, 2H), 4.33 (q, *J* = 7.1 Hz, 2H), 4.14 (s, 2H), 2.84 (t, J = 7.2 Hz, 2H), 2.37 (s, 3H), 2.17 (m, 2H), 1.37 (t, J = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.07, 139.33, 137.63, 133.08, 132.87, 131.19, 129.20, 128.68, 123.29, 71.51, 62.82, 54.88, 31.21, 30.74, 15.00, 14.05.; HRMS (ESI) *m/z* calculated for $C_{18}H_{23}O_5S_2$ $[M+H]^+$: 383.0987, found 383.0985.

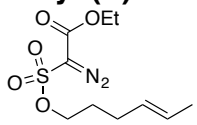
Preparation of Diazosulfonate Starting Materials

General procedure for diazo transfer:

A round-bottom flask equipped with a stir bar and rubber septum was charged with sulfonate ester (1 equiv) and THF (0.4 M) and cooled to -45˚C with a dry ice/acetonitrile bath. $MeSO_2N_3$

(2.2–3.0 equiv) was then added in one portion. DBU (1.5 equiv) was then added slowly dropwise over 2-5 minutes and the solution turned yellow. After the reaction was stirred for 1 hour at -45˚C, the cooling bath was removed and 15 mL of sat. aq. $(NH_4)_2SO_4$ was immediately added. The mixture was transferred to a separatory funnel with 20 mL of CH_2Cl_2. The aqueous layer was extracted with 3 x 10 mL CH_2Cl_2, and the combined organic extracts were washed with 2 x 10 mL 10% NaOH, 2 x 10 mL H_2O, and sat. aq. NaCl. The organic phase was dried over Na_2SO_4 or $MgSO_4$, filtered, and concentrated under reduced pressure. Purification by chromatography on silica gel gave the desired diazo product. Some diazoesters were difficult to separate from the sulfonamide byproduct ($MeSO_2NH_2$, singlet at 3.30 ppm). In these cases, the diazoester was washed 3x with 20% NaOH, 2x with H_2O, and 1x with brine after column chromatography. We found that this sulfonamide impurity (in trace quantities, <5%) did not affect the reactivity of the diazoesters. Following purification, the diazo compounds were stored at -20˚C under argon. The ^{13}C NMR signal for the diazo carbon was typically not detected; Novikov [7] and Du Bois [3] noticed similar results. *NOTE: Some diazosulfonate ester compounds exhibited suboptimal reactivity after storing for more than a month; repurification sometimes restored reactivity in these cases. Some diazosulfonate ester compounds decomposed during long periods under reduced pressure. These cases are noted below. WARNING: Although we have not experienced problems with the preparation and the handling of $MeSO_2N_3$ or diazosulfonate esters, sulfonyl azides and diazo compounds can be heat and/or shock sensitive and should be handled with appropriate caution.*

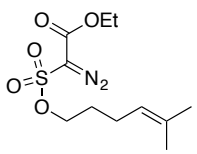
Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate [6].



2.30 g (9.20 mmol) of ethyl (*E*)-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[S3]** were used, along with $MeSO_2N_3$ (2.40 g, 20.2 mmol, 2.2 equiv), DBU (206 μL, 13.8 mmol, 1.5 equiv), and THF (23 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 1.98 g (7.17 mmol) of pure product as a yellow oil (78% yield).

^1H-NMR (500 MHz, $CDCl_3$) δ 5.53-5.43 (m, 1H), 5.37 (ddd, *J* = 15.2, 6.8, 5.1 Hz, 1H), 4.35 (t, *J* = 6.3 Hz, 2H), 4.33 (q, *J* = 7.2 Hz, 2H), 2.10 (dt, *J* = 7.7, 7.3 Hz, 2H), 1.81 (p, *J* = 6.7 Hz, 2H), 1.65 (dd, *J* = 6.2, 1.3 Hz, 3H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.62, 129.05, 126.88, 72.98, 62.80, 28.77, 28.29, 18.01, 14.39.; HRMS (ESI) *m/z* calculated for $C_{10}H_{16}O_5N_2SNa$ $[M+Na]^+$: 299.0678, found 299.0681; IR (ATR, cm^{-1}) 2970, 2940, 2856, 2130, 1721, 1465, 1447, 1373, 1285, 1224, 1178, 1083, 1005, 964, 929, 836, 765, 739, 614, 564, 541, 491.

Ethyl 2-diazo-2-(((5-methylhex-4-en-1-yl)oxy)sulfonyl)acetate [S21].

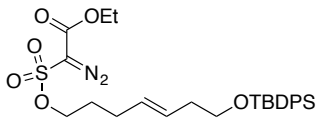


0.89 g (3.40 mmol) of ethyl 2-(((5-methylhex-4-en-1-yl)oxy)sulfonyl)acetate **[S4]** were used, along with $MeSO_2N_3$ (908 mg, 7.50 mmol, 2.2 equiv), DBU (762 μL, 5.10 mmol, 1.5 equiv), and THF (8.5 mL, 0.4 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 0.60 g (2.08 mmol) of pure product as a yellow oil (61% yield).

^1H-NMR (500 MHz, $CDCl_3$) δ 5.07 (t, *J* = 7.3 Hz, 1H), 4.34 (t, *J* = 6.6 Hz, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 2.10 (dt, *J* = 7.5, 7.0 Hz, 2H), 1.79 (p, *J* = 7.3 Hz, 2H), 1.69 (s, 3H), 1.61 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.58, 133.51, 122.26, 73.17, 62.76, 29.10,

25.77, 23.81, 17.77, 14.35; IR (ATR, cm^{-1}) 2970, 2918, 2130, 1722, 1447, 1373, 1285, 1224, 1178, 1083, 1007, 924, 879, 843, 777, 739, 615, 558, 495; HRMS (ESI) *m/z* calculated for $C_{11}H_{18}N_2O_5SNa$ $[M+Na]^+$: 313.0834, found 313.0831.

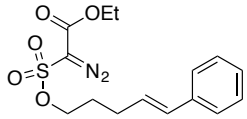
Ethyl (*E*)-2-(((7-((*tert*-butyldiphenylsilyl)oxy)hept-4-en-1-yl)oxy)sulfonyl)-2-diazoacetate [S22].



1.18 g (2.30 mmol) of ethyl (*E*)-2-(((7-((*tert*-butyldiphenylsilyl)oxy)hept-4-en-1-yl)oxy)sulfonyl)acetate **[S5]** were used, along with $MeSO_2N_3$ (618 mg, 5.10 mmol, 2.2 equiv), DBU (524 μL, 3.50 mmol, 1.5 equiv), and THF (5.75 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 879 mg (1.61 mmol) of pure product as a yellow oil (70% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.68-7.64 (m, 4H), 7.45-7.35 (m, 6H), 5.51-5.36 (m, 2H), 4.34 (t, *J* = 6.2 Hz, 2H), 4.31 (q, *J* = 7.3 Hz, 2H), 3.67 (t, *J* = 6.6 Hz, 2H), 2.25 (q, *J* = 6.9 Hz, 2H), 2.10 (dt, *J* = 7.7, 7.2 Hz, 2H), 1.80 (p, *J* = 6.5 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.04 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.59, 135.68, 134.06, 130.08, 129.68, 128.63, 127.71, 73.01, 63.84, 62.79, 36.08, 28.78, 28.44, 26.96, 19.34, 14.40.; HRMS (ESI) *m/z* calculated for $C_{27}H_{37}O_6N_2SiS$ $[M+H]^+$: 545.2142, found 545.2141; IR (ATR, cm^{-1}) 3071, 2931, 2857, 2130, 1723, 1589, 1472, 1376, 1288, 1225, 1179, 1091, 1006, 926, 822, 783.

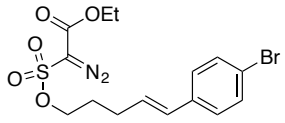
Ethyl (*E*)-2-diazo-2-(((5-phenylpent-4-en-1-yl)oxy)sulfonyl)acetate [S23].



1.49 g (4.70 mmol) of ethyl (*E*)-2-(((5-phenylpent-4-en-1-yl)oxy)sulfonyl)acetate **[S6]** were used, along with $MeSO_2N_3$ (1.25 g, 10.3 mmol, 2.2 equiv), DBU (106 μL, 7.10 mmol, 1.5 equiv), and THF (12 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 1.03 g (3.04 mmol) of pure product as a yellow oil (65% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.36-7.27 (m, 4H), 7.24- 7.19 (m, 1H), 6.43 (d, *J* = 15.8 Hz, 1H), 6.16 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.41 (t, *J* = 6.3 Hz, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 2.36 (q, *J* = 7.5 Hz, 2H), 1.95 (p, *J* = 6.3 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.56, 137.32, 131.59, 128.65, 128.24, 127.34, 126.12, 72.81, 62.82, 28.80, 28.67, 14.39.; HRMS (ESI) *m/z* calculated for $C_{15}H_{18}O_5N_2SNa$ $[M+H]^+$: 361.0834, found 361.0844; IR (ATR, cm^{-1}) 3024, 2997, 2970, 2946, 2513, 2127, 1715, 1598, 1576, 1492, 1471, 1447, 1372, 1287, 1226, 1177, 1155, 1079, 1007, 986, 962, 926, 848.

Ethyl (*E*)-2-(((5-(4-bromophenyl)pent-4-en-1-yl)oxy)sulfonyl)-2-diazoacetate [S24].

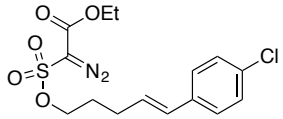


950 mg (2.42 mmol) of ethyl(*E*)-2-(((5-(4-bromophenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate **[S7]** were used, along with $MeSO_2N_3$ (640 mg, 5.3 mmol, 2.2 equiv), DBU (543 μL, 3.63 mmol, 1.5 equiv), and THF (6.1 mL, 0.4 M). Flash column chromatography on silica using 10% → 15% → 20% EtOAc/hexanes as eluent gave 614 mg (1.47 mmol) of pure product as a yellow oil (61% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ7.42 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 6.37 (d, *J* = 15.9 Hz, 1H), 6.16 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.41 (t, *J* = 6.3 Hz, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 2.34

(dt, *J* = 7.6, 7.0 Hz, 2H), 1.94 (p, *J* = 6.7 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ159.68, 136.43, 131.85, 130.61, 129.29, 127.83, 121.13, 72.89, 62.96, 28.94, 28.70, 14.53.; HRMS (ESI) *m/z* calculated for $C_{15}H_{18}N_2O_5SBr$ $[M+H]^+$: 417.0120, found 417.0113.; IR (ATR, cm^{-1}) 2982.7, 2131.1, 1717.0, 1587.7, 1487.6, 1466.0, 1445.4, 1373.3, 1285.6, 1223.1, 1177.1, 1085.4, 1073.1, 1007.4, 962.7, 924.3, 881.7, 838.1, 823.3, 738.2, 703.5, 613.8, 564.3, 537.3, 522.3, 500.5.

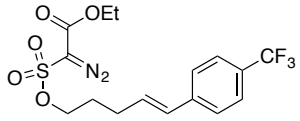
Ethyl (*E*)-2-(((5-(4-chlorophenyl)pent-4-en-1-yl)oxy)sulfonyl)-2-diazoacetate [S25].



846 mg (2.40 mmol) of ethyl (*E*)-2-(((5-(4-chlorophenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate **[S8]** were used, along with $MeSO_2N_3$ (641 mg, 5.3 mmol, 2.2 equiv), DBU (538 μL, 3.60 mmol, 1.5 equiv), and THF (6 mL, 0.4 M). Flash column chromatography on silica using 20% Et_2O/hexanes as eluent gave 633 mg (1.70 mmol) of pure product as a yellow oil (71% yield). This compound was isolated with <5% of an unknown impurity that did not appear to affect reactivity.

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.28-7.24 (m, 4H), 6.39 (dt, *J* = 15.8, 1.5 Hz, 1H), 6.14 (dt, *J* = 15.9, 7.0 Hz, 1H), 4.41 (t, *J* = 6.3 Hz, 2H), 4.31 (q, *J* = 7.0 Hz, 2H), 2.35 (dt, *J* = 7.9, 7.5 Hz, 2H), 1.94 (p, *J* = 6.5 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.41, 135.79, 132.70, 130.26, 128.98, 128.40, 127.27, 72.67, 62.72, 28.69, 28.46, 14.28.; HRMS (ESI) *m/z* calculated for $C_{15}H_{17}O_5N_2SCl$ $[M+H]^+$: 395.0444, found 395.0444; IR (ATR, cm^{-1}) 2970, 2940, 2131, 1718, 1593, 1490, 1465, 1373, 1285, 1224, 1177, 1088, 1011, 924, 839.

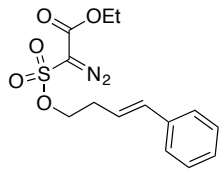
Ethyl (*E*)-2-diazo-2-(((5-(4-(trifluoromethyl)phenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate [S26].



772 mg (2.03 mmol) of ethyl(*E*)-2-(((5-(4-(trifluoromethyl)phenyl)pent-4-en-1-yl)oxy)sulfonyl)acetate **[S9]** were used, along with $MeSO_2N_3$ (541 mg, 4.47 mmol, 2.2 equiv), DBU (456 μL, 3.05 mmol, 1.5 equiv), and THF (5.1 mL, 0.4 M). Flash column chromatography on silica using 10% → 15% → 20% EtOAc/hexanes as eluent gave 412 mg (1.02 mmol) of pure product as a yellow oil (50% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 6.47 (d, *J* = 15.9 Hz, 1H), 6.28 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.42 (t, *J* = 6.2 Hz, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 2.39 (dt, *J* = 7.3, 7.0 Hz, 2H), 1.97 (p, *J* = 6.6 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.67, 140.95, 131.28, 130.53, 129.24 (q, *J* = 32.3 Hz), 126.42, 125.73 (q, *J* = 3.8 Hz), 124.46 (q, J = 273.42 Hz), 72.84, 62.96, 29.00, 28.62, 14.50. HRMS (ESI) m/z calc'd for $C_{16}H_{18}N_2O_5F_3S$ $[M+H]^+$: 407.0889, found 407.0899.; IR (ATR, neat, cm^{-1}) 2988.19, 2143.5, 1916.96, 1707.3, 1654.07, 1614.47, 1578.37, 1467.21, 1454.81, 1432.38, 1378.24, 1325.48, 1299.49, 1207.47, 1177.77, 1156.31, 1113.06, 1097.62, 1066.63, 1014.63, 1001.32, 975.17, 951.8, 924.77, 858.66, 843.86, 825.59, 799.6, 758.89, 741.11, 650.05, 615.73, 575.8, 534.59, 523.13, 509.54, 496.94

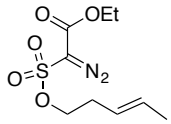
Ethyl (*E*)-2-diazo-2-(((4-phenylbut-3-en-1-yl)oxy)sulfonyl)acetate [S27].



1.38 g (4.60 mmol) of ethyl (*E*)-2-(((4-phenylbut-3-en-1-yl)oxy)sulfonyl)acetate **[S10]** were used, along with $MeSO_2N_3$ (1.22 g, 10.1

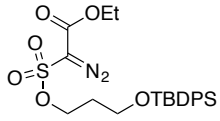
mmol, 2.2 equiv), DBU (1.00 mL, 6.90 mmol, 1.5 equiv), and THF (12 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 1.11 g (3.42 mmol) of pure product as a yellow oil (74% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.37-7.33 (m, 2H), 7.33-7.28 (m, 2H), 7.25-7.21 (m, 1H), 6.52 (dt, *J* = 15.9, 1.4 Hz, 1H), 6.15 (dt, *J* = 15.8, 7.0 Hz, 1H), 4.47 (t, *J* = 6.6 Hz, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.68 (qd, *J* = 6.7, 1.5 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.49, 136.88, 133.74, 128.65, 127.68, 126.27, 123.59, 72.69, 62.78, 32.66, 14.32.; HRMS (ESI) *m/z* calculated for $C_{14}H_{16}O_5N_2SNa$ $[M+Na]^+$: 347.0678, found 347.0672; IR (ATR, cm^{-1}) 3424, 3082, 3059, 3027, 2984, 2939, 2908, 2385, 2131, 1947, 1716, 1598, 1577, 1494, 1465, 1448, 1371, 1284, 1223, 1177, 1085, 1007, 961, 922, 852, 825, 738, 693.

Ethyl (*E*)-2-((pent-3-en-1-yloxy)sulfonyl)acetate [S28].



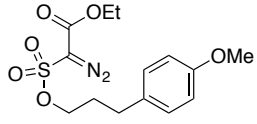
807 mg (3.40 mmol) of ethyl (*E*)-2-((pent-3-en-1-yloxy)sulfonyl)acetate **[S11]** were used, along with $MeSO_2N_3$ (908 mg, 7.50 mmol, 2.2 equiv), DBU (762 μL, 5.10 mmol, 1.5 equiv), and THF (8.50 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 777 mg (3.0 mmol) of pure product as a yellow oil (87% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.59 (dqt, *J* = 15.5, 6.4, 1.4 Hz, 1H), 5.38 (dtq, *J* = 15.3, 6.8, 1.7 Hz, 1H), 4.37-4.31 (m, 4H), 2.44 (qp, *J* = 6.8, 1.2 Hz, 2H), 1.67 (dq, *J* = 6.5, 1.4 Hz, 3H), 1.34 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.66, 129.66, 124.59, 73.14, 62.82, 32.36, 18.14, 14.43.; HRMS (ESI) *m/z* calculated for $C_9H_{14}O_5SNa$ $[M+Na]^+$: 285.0521, found 285.0515; IR (ATR, cm^{-1}) 3424, 2969, 2940, 2919, 2859, 2388, 2130, 1717, 1466, 1448, 1372, 1285, 1223, 1178, 1083, 1009, 961, 914, 842, 787, 739, 614, 560, 540, 491, 463.

Ethyl 2-((3-((*tert*-butyldiphenylsilyl)oxy)propoxy)sulfonyl)-2-diazoacetate [S29].



1.16 g (2.50 mmol) of ethyl 2-((3-((*tert*-butyldiphenylsilyl)oxy)propoxy) sulfonyl)acetate **[S12]** were used, along with $MeSO_2N_3$ (666 mg, 5.50 mmol, 2.2 equiv), DBU (561 μL, 3.80 mmol, 1.5 equiv), and THF (6.30 mL, 0.4 M). Flash column chromatography on silica using 2% EtOAc/hexanes as eluent gave 829 mg (1.69 mmol) of pure product as a pale yellow solid (68% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.67-7.62 (m, 4H), 7.49-7.33 (m, 6H), 4.54 (t, *J* = 6.3 Hz, 2H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.77 (t, *J* = 5.8 Hz, 2H), 1.97 (p, *J* = 6.1 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.05 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.54, 135.62, 133.47, 129.90, 127.88, 70.53, 62.79, 59.38, 32.03, 26.92, 19.31, 14.39.; HRMS (ESI) *m/z* calculated for $C_{23}H_{31}O_6N_2SiS$ $[M+H]^+$: 491.1672, found 491.1685; IR (ATR, cm^{-1}) 3071, 2998, 2962, 2938, 2891, 2860, 2136, 1728, 1588, 1471, 1428, 1419, 1373, 1281, 1224, 1179, 1159, 1102, 1090, 1068, 1010, 994.

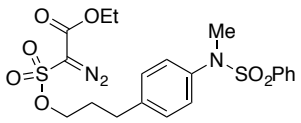
Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy)sulfonyl)acetate [30].



855 mg (2.70 mmol) of ethyl 2-((3-(4-methoxyphenyl)propoxy)-sulfonyl)acetate **[S13]** were used, along with $MeSO_2N_3$ (761 mg, 8.11 mmol, 3 equiv), DBU (605 μL, 4.06 mmol, 1.5 equiv), and THF (6.8 mL, 0.4 M). Flash column chromatography on silica using 10% → 20% EtOAc/hexanes as eluent gave 721 mg (2.11 mmol) of pure product as a yellow oil (78% yield).

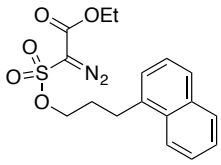
Note: the reactivity of this diazoester deteriorated after one week and could not be restored by additional purification.
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.4 Hz, 2H), 6.84 (d, J = 8.5 Hz, 2H), 4.38 - 4.28 (m, 4H), 3.79 (s, 3H), 2.69 (t, *J* = 7.5 Hz, 2H), 2.04 (p, J = 7.2 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.62, 158.25, 132.31, 129.52, 114.13, 72.66, 62.86, 55.42, 30.85, 30.66, 14.43.; HRMS (ESI) *m/z* calculated for $C_{14}H_{19}O_6N_2S$ $[M+H]^+$: 343.0964, found 343.0956; IR (ATR, cm^{-1}) 2938.6, 2837.84, 2132.38, 1717.58, 1612.33, 1584.04, 1512.66, 1465.72, 1373.33, 1285.65, 1244.79, 1224.31, 1176.21, 1084.54, 1033.1, 996.13, 830.05, 811.04, 738.66, 698.12, 614.07, 569.25, 554.06, 509.88.

Ethyl 2-diazo-2-((3-(4-(*N*-methylphenylsulfonamido)phenyl)propoxy)sulfonyl) acetate [S30].



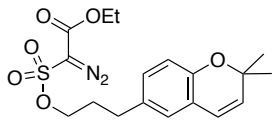
802 mg (1.76 mmol) of ethyl 2-((3-(4-(*N*-methylphenylsulfonamido)phenyl)propoxy)sulfonyl)acetate **[S14]** were used, along with $MeSO_2N_3$ (640.0 mg, 5.28 mmol, 3 equiv), DBU (390 *μ*L, 2.64 mmol, 1.5 equiv), and THF (4.4 mL, 0.4 M). Flash column chromatography on silica using 10% → 40% EtOAc/hexanes as eluent gave 639 mg (1.33 mmol) of pure product as a yellow oil (75% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.62 - 7.54 (m, 3H), 7.48 (dd, *J* = 8.3, 7.2, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 4.38 (t, J = 6.0 Hz, 2H), 4.36 (q J = 7.2, 2H), 3.18 (s, 3H), 2.73 (t, J = 7.5 Hz, 2H), 2.00 - 2.04 (m, 2H), 1.35 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.42, 139.74, 139.52, 136.50, 132.76, 128.91, 128.74, 127.86, 126.84, 72.42, 62.75, 38.18, 30.99, 30.38, 14.31. HRMS (ESI) *m/z* calculated for $C_{20}H_{23}O_7N_3S_2Na$ $[M+Na]^+$: 504.0875, found 504.0873; IR (ATR, cm^{-1}) 2924.36, 2852.36, 2133.81, 1717.16, 1608.82, 1584.78, 1508.07,1466.48, 1446.96, 1374.87, 1348.87, 1287.10, 1224.05, 1173.43, 1155.02, 1088.53, 1066.30, 1017.80, 1017.80, 998.66, 926.55, 870.22, 834.43, 722.39, 690.35, 665.65, 649.93, 615.06, 588.85, 572.72, 557.75, 497.97.

Ethyl 2-diazo-2-((3-(naphthalen-1-yl)propoxy)sulfonyl)acetate [S31].



118 mg (0.351 mmol) of ethyl 2-((3-(naphthalen-1-yl)propoxy)sulfonyl)acetate **[S15]** were used, along with $MeSO_2N_3$ (127.5 mg, 1.05 mmol, 3 equiv), DBU (78.6 *μ*L, 0.527 mmol, 1.5 equiv), and THF (0.9 mL, 0.4 M). Flash column chromatography on silica using 5% → 15% EtOAc/hexanes as eluent gave 86.8 mg (0.240 mmol) of pure product as a yellow oil (67% yield).
^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 7.9 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.56 - 7.46 (m, 2H), 7.40 (dd, *J* = 8.1, 7.0 Hz, 1H), 7.34 (d, J = 7.0 Hz, 1H), 4.43 (t, *J* = 6.1 Hz, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.22 (t, J = 7.4 Hz, 2H), 2.29 - 2.17 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.57, 136.33, 134.07, 131.71, 129.04, 127.31, 126.47, 126.22, 125.77, 125.64, 123.54, 72.93, 62.85, 29.86, 28.80, 14.40.; HRMS (ESI) *m/z* calculated for $C_{17}H_{19}O_5N_2S$ $[M+H]^+$: 363.1015, found 363.1008; IR (ATR, cm^{-1}) 2921.48, 2134.02, 1714.69, 1594.01, 1509.49, 1459.33, 1445.80, 1424.40, 1392.28, 1371.95, 1286.49, 1217.84, 1178.91, 1081.27, 1030.36, 1012.76, 989.32, 925.46, 912.74, 869.31, 854.39, 829.19, 817.01, 796.38, 779.72, 734.26, 648.01, 610.89, 561.82, 549.71, 533.15, 508.97, 496.86, 458.39.

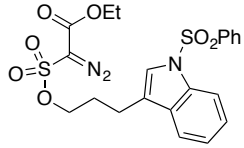
Ethyl 2-diazo-2-((3-(2,2-dimethyl-2*H*-chromen-6-yl)propoxy)sulfonyl)acetate [S32].



400. mg (1.09 mmol) of ethyl 2-((3-(2,2-dimethyl-2H-chromen-6-yl)propoxy)sulfonyl)acetate **[S16]** were used, along with $MeSO_2N_3$ (395 mg, 3.26 mmol, 3 equiv), DBU (243 μL, 1.63 mmol, 1.5 equiv), and THF (2.70 mL, 0.4 M). Flash column chromatography on silica using 5% → 15% EtOAc/hexanes as eluent gave 228 mg (0.578 mmol) of pure product as a yellow oil (53% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.93 (dd, *J* = 8.2, 2.2 Hz, 1H), 6.81 (d, *J* = 2.2 Hz, 1H), 6.73 (d, *J* = 8.1 Hz, 1H), 6.31 (d, *J* = 9.8 Hz, 1H), 5.64 (d, *J* = 9.8 Hz, 1H), 4.40 - 4.33 (m, 4H), 2.67 (t, *J* = 7.5 Hz, 2H), 2.07 (p, J = 6 Hz, 2H), 1.45 (s, 6H), 1.36 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.53, 151.43, 132.30, 131.11, 128.97, 126.30, 122.27, 121.36, 116.37, 76.20, 72.63, 62.81, 30.69, 30.64, 28.04, 14.38. HRMS (ESI) *m/z* calculated for $C_{18}H_{23}O_6N_2S$ $[M+H]^+$: 395.1277, found 395.1273; IR (ATR, cm^{-1}) 2976.12, 2925.41, 2131.58, 1719.37, 1638.37, 1489.48, 1464.88, 1372.12, 1286.63, 1261.45, 1211.27, 118.2, 1151.82, 1127.63, 1084.09, 1001.31, 958.9, 925.35, 923.03, 793.14, 768.19, 738.45, 717.48, 614.17, 584.89, 562.72, 535.86, 510.44, 486.44.

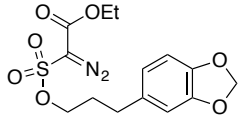
Ethyl 2-((3-(1-(phenylsulfonyl)-1H-indol-3-yl)propoxy)sulfonyl)acetate [S33].



2.30 g (7.28 mmol) of ethyl 2-((3-(1-(phenylsulfonyl)-1H-indol-3-yl)propoxy)sulfonyl)acetate **[S17]** was used, along with imidazole (595 mg, 8.74 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (2.04 g, 10.9 mmol, 1.5 equiv), and THF (18 mL, 0.2 M). Flash column chromatography on silica using 10% → 30% EtOAc/hexanes as eluent gave 2.40 g (5.16 mmol) of pure product as a clear oil (71% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.4 Hz, 1H), 7.90 - 7.85 (m, 2H), 7.56 - 7.50 (m, 1H), 7.45 (q, *J* = 8.7 Hz, 3H), 7.39 (s, 1H), 7.33 (t, J = 9.8 Hz, 1H), 7.25 - 7.21 (m, 1H), 4.38 (t, *J* = 6.0 Hz, 3H), 4.33 (q, *J* = 7.1 Hz, 3H), 2.82 (t, *J* = 7.4 Hz, 3H), 2.13 (p, *J* = 6.5 Hz, 3H), 1.31 (t, *J* = 7.1 Hz, 4H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.47, 138.30, 135.54, 133.85, 130.70, 129.36, 126.84, 125.04, 123.36, 123.33, 121.33, 119.42, 113.96, 72.44, 62.87, 28.36, 20.85, 14.37.; HRMS (ESI) *m/z* calculated for $C_{21}H_{21}NO_7S_2$ $[M+H]^+$: 514.0719, found 514.0769. IR (ATR, cm^{-1}) 2138.67, 1723.8, 1463.45. 1446.28, 1376.14, 1362.96, 1287.43, 1274.42, 1229.7, 1202.13, 1174.21, 1129.69, 1120.47, 1095.71, 1078.09, 1017.77, 970.87, 943.64, 869.67, 860.01, 821.95, 789.11, 766.62, 748.94, 737.16, 723.89, 685.23, 626.35, 602.94, 591.1, 578.54, 570.31, 549.73, 536.17, 501.85, 478.29.

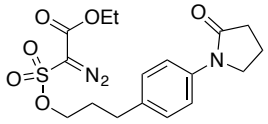
Ethyl 2-((3-(benzo[*d*][1,3]dioxol-5-yl)propoxy)sulfonyl)-2-diazoacetate [S34].



1.82 g (5.50 mmol) of ethyl 2-((3-(benzo[*d*][1,3]dioxol-5-yl)propoxy)sulfonyl)acetate **[S18]** were used, along with $MeSO_2N_3$ (1.47 g, 12.1 mmol, 2.2 equiv), DBU (1.23 mL, 8.3 mmol, 1.5 equiv), and THF (13.9 mL, 0.4 M). Flash column chromatography on silica using 5% → 10% EtOAc/hexanes as eluent gave 1.47 g (4.13 mmol) of pure product as a yellow oil (75% yield). *Note*: the reactivity of this diazoester deteriorated after one week and could not be restored by additional purification.

^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.73 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.4 Hz, 1H), 6.63 (dd, *J* = 7.9, 1.4 Hz, 1H), 5.93 (s, 2H), 4.38-4.29 (m, 4H), 2.67 (t, *J* = 7.5 Hz, 2H), 2.03 (p, *J* = 6.4 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.50, 147.85, 146.07, 134.00, 121.36, 108.90, 108.37, 100.97, 72.44, 62.81, 31.23, 30.79, 14.37.; HRMS (ESI) *m/z* calculated for $C_{14}H_{16}O_7N_2SNa$ $[M+Na]^+$: 379.0576, found 379.0574; IR (ATR, cm^{-1}) 2970, 2941, 2132, 1717, 1608, 1503, 1488, 1442, 1371, 1286, 1243, 1226, 1177, 1121, 1091, 1036, 999, 922, 838, 808, 770.

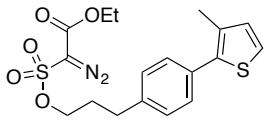
Ethyl 2-diazo-2-((3-(4-(2-oxopyrrolidin-1-yl)phenyl)propoxy)sulfonyl)acetate [S35].



649 mg (1.76 mmol) of ethyl 2-((3-(4-(2-oxopyrrolidin-1-yl)phenyl)propoxy)sulfonyl)acetate **[S19]** were used, along with $MeSO_2N_3$ (638 mg, 5.27 mmol, 3 equiv), DBU (390 μL, 2.64 mmol, 1.5 equiv), and THF (4.4 mL, 0.4 M). Flash column chromatography on silica using 30% → 60% EtOAc/hexanes as eluent gave 490 mg (1.24 mmol) of pure product as a yellow oil (70% yield). *Note*: this diazoester decomposes after twelve hours under vacuum at room temperature; for best results use immediately.

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.6 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 4.38 (t, *J* = 6.2 Hz, 2H), 4.37 (q, *J* = 7.2 Hz, 2H), 3.88 (t, *J* = 7.0 Hz, 2H), 2.76 (t, *J* = 7.6 Hz, 2H), 2.64 (t, *J* = 8.1 Hz, 2H), 2.20 (p, *J* = 7.3 Hz, 2H), 2.10 (p, *J* = 6.4 Hz, 2H), 1.36 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 174.22, 159.47, 137.79, 136.42, 128.85, 120.29, 72.50, 62.80, 48.89, 32.74, 30.91, 30.57, 18.09, 14.36. HRMS (ESI) *m/z* calculated for $C_{17}H_{22}O_6N_3S$ $[M+H]^+$: 396.1229, found 396.1221; IR (ATR, cm^{-1}) 2980.9, 2133.8, 1690.4, 1612.35, 1514.75, 1487.79, 1461.43, 1426.78, 1390.72, 1373.34, 1287.34, 1222.81, 1177.02, 1124.05, 1085.38, 1034.71, 996.62, 923.73, 834.88, 801.99, 738.04, 636.79, 613.70, 557.11, 541.11, 508.90.

Ethyl 2-diazo-2-((3-(4-(3-methylthiophen-2-yl)phenyl)propoxy)sulfonyl)acetate [S36].



1.55 g (4.04 mmol) of ethyl 2-((3-(4-(3-methylthiophen-2-yl)phenyl)propoxy)sulfonyl)acetate **[S20]** were used, along with $MeSO_2N_3$ (1.47 g, 12.1 mmol, 3 equiv), DBU (905 μL, 6.06 mmol, 1.5 equiv), and THF (10 mL, 0.4 M). Flash column chromatography on silica using 0% → 15% EtOAc/hexanes as eluent, and then a second purification using 2% → 15% Et_2O/petroleum ether as eluent gave 839 mg (2.05 mmol) of pure product as a yellow oil (51% yield).

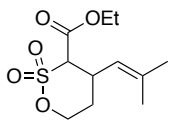
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 5.2 Hz, 1H), 6.92 (d, *J* = 5.0 Hz, 1H), 4.39 (t, *J* = 6.2 Hz, 2H), 4.33 (q, *J* = 7.2 Hz, 2H), 2.78 (t, *J* = 7.6 Hz, 2H), 2.32 (s, 3H), 2.11 (p, *J* = 6.4 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.56, 139.29, 137.70, 133.16, 133.04, 131.22, 129.30, 128.70, 123.35, 72.63, 62.85, 31.28, 30.55, 15.02, 14.41.; HRMS (ESI) *m/z* calculated for $C_{18}H_{21}O_5N_2S_2$ $[M+H]^+$: 409.0892, found 409.0883; IR (ATR, cm^{-1}) 2981.11, 2131.45, 1717.25, 1546.48, 1508.41, 1447.81, 1373.97, 1285.99, 1222.83, 1177.96, 1084.70, 997.25, 925.17, 833.91, 799.89, 736.71, 707.17, 614.44, 554.86, 535.08, 498.08.

Substrate Scope for Fe-Catalyzed Intramolecular C—H Alkylation

General procedure for [FePc]-catalyzed C—H alkylation:
Into a 1 dram vial was added [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv) and $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv) in a glovebox. The vial was sealed, taken out of the box, and the contents were added under a stream of nitrogen to a flame-dried three neck 25 mL flask equipped with a stir bar, reflux condenser, glass stopper, and rubber septum. CH_2Cl_2 (5.0 mL, 0.08 M) was added and the flask was heated to reflux (~45˚C) for 10 minutes. Diazosulfonate ester (0.40 mmol, 1 equiv) was taken up with CH_2Cl_2 (3.0 mL, 0.13 M) in a 5 mL glass syringe and added to the refluxing catalyst solution over the course of an hour (rate of 3 mL/h) via syringe pump (final reaction concentration is 0.05 M). After complete addition of diazosulfonate, the reaction refluxed until judged complete by TLC (2-12 h). The reaction mixture was cooled, adsorbed onto SiO_2 or florisil, and applied directly to a silica column for purification to afford the desired sultone product. Yields were calculated from a mixture of diastereomers. Subsequent purification afforded the separation of diastereomers for characterization. Relative stereochemistry was assigned *via* coupling constant analysis and analogy from **7**. Some substrates demonstrated improved yields with the addition of activated powdered 3Å molecular sieves. In these cases, the molecular sieves were added to the reaction flask at the same time as [FePc]Cl and $NaBAr^F_4$. Any further variation of these reaction conditions is noted for individual substrates.

General procedure for $Rh_2(OAc)_4$-mediated intramolecular C—H alkylation:
The following procedure was adapted from literature precedent [3, 7]. Into a 1 dram vial was added $Rh_2(OAc)_4$ (3.5 mg, 0.008 mmol, 0.02 equiv) in a glovebox. The vial was sealed, taken out of the box, and the contents were added under a stream of nitrogen to a flame dried three neck 25 mL flask equipped with a stir bar, reflux condenser, glass stopper, and rubber septum. CH_2Cl_2 (5.0 mL, 0.08 M) was added and the flask was heated to reflux (~45˚C). Diazosulfonate was taken up with CH_2Cl_2 (3.0 mL, 0.13 M) and added to the refluxing catalyst solution over the course of one minute. Transfer of the starting material was made quantitative with an additional 1 mL of CH_2Cl_2. After complete addition of diazosulfonate, the reaction refluxed until judged complete by TLC (1-12 h). The solution was cooled to RT, filtered through a small pad of celite with 30 mL of CH_2Cl_2, and concentrated under reduced pressure. Purification by silica gel chromatography afforded the desired sultone product.

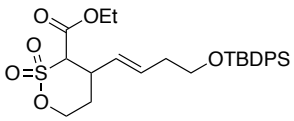
(±)-Ethyl 4-(2-methylprop-1-en-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [8].



Ethyl 2-diazo-2-(((5-methylhex-4-en-1-yl)oxy)sulfonyl)acetate **[S21]** (116.1 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 10% → 15% → 20% EtOAc in hexanes. Pure product was isolated as a white solid and a 2:1 syn:anti mixture of diastereomers. Subsequent purification using 5% → 10% → 15% EtOAc/hexanes allowed for separation of diastereomers.
Run 1: (63.5 mg, 0.242 mmol, 61%), 0% rsm. Run 2: (57.4 mg, 0.219 mmol, 55%), 0% rsm. Run 3: (57.8 mg, 0.220 mmol, 55%), 0% rsm. **Average: 57% yield ± 2.8, 0% rsm.**

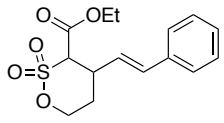
Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 4.96 (d, *J* = 9.1, 1H), 4.65 (td, *J* = 12.3, 2.6 Hz, 1H), 4.58 (ddd, *J* = 11.6, 5.1, 2.1 Hz, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.98 (d, *J* = 4.9 Hz, 1H), 3.44 (ddd, *J* = 12.0, 9.0, 4.6 Hz, 1H), 2.57 (dtd, *J* = 14.5, 12.3, 5.2 Hz, 1H), 1.71 (s, 3H), 1.70 (s, 3H), 1.61-1.50 (m, 1H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.40, 137.36, 121.37, 73.30, 64.50, 62.50, 36.85, 26.21, 25.92, 18.43, 14.25.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 4.84 (dd, *J* = 9.7, 1.4 Hz, 1H), 4.72-4.62 (m, 1H), 4.47 (ddd, *J* = 11.6, 4.4, 2.5 Hz, 1H), 4.30-4.18 (m, 2H), 3.74 (d, *J* = 11.4 Hz, 1H), 3.50 (qd, *J* = 10.1, 5.7 Hz, 1H), 1.84-1.75 (m, 2H), 1.70 (s, 3H), 1.69 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.56, 137.45, 121.76, 72.57, 68.37, 62.65, 38.12, 30.68, 25.90, 18.49, 14.13.
HRMS (ESI) *m/z* calculated for $C_{11}H_{18}O_5SNa$ $[M+Na]^+$: 285.0773, found 285.0773.

Ethyl (*E*)-4-(4-((*tert*-butyldiphenylsilyl)oxy)but-1-en-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [9].



Ethyl (*E*)-2-(((7-((*tert*-butyldiphenylsilyl)oxy)hept-4-en-1-yl)oxy)sulfonyl)-2-diazoacetate **[S22]** (108.9 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (4.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 10% EtOAc/hexanes. Pure product was isolated as a clear, thick oil and a 1.5:1 syn:anti mixture of diastereomers. Subsequent purification using 5% → 10% → 15% EtOAc/hexanes allowed for separation of diastereomers.
Run 1: (55.6 mg, 0.108 mmol, 54%), 0% rsm. Run 2: (57.4 mg, 0.111 mmol, 56%), 0% rsm. Run 3: (51.9 mg, 0.100 mmol, 50%), 0% rsm. **Average: 53% yield ± 2.2, 0% rsm.**
Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.67-7.61 (m, 4H), 7.46-7.35 (m, 6H), 5.68 (td, *J* = 15.2, 6.9 Hz, 1H), 5.35 (dd, *J* = 15.4, 7.6 Hz, 1H), 4.66-4.56 (m, 2H), 4.28-4.11 (m, 2H), 4.02 (d, *J* = 4.6 Hz, 1H), 3.67 (t, *J* = 6.4 Hz, 2H), 3.20 (ddt, *J* = 12.4, 8.5, 4.6 Hz, 1H), 2.60 (dtd, *J* = 14.6, 12.2, 5.5 Hz, 1H), 2.26 (q, *J* = 6.4 Hz, 2H), 1.63-1.56 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.04 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.09, 135.67, 133.87, 131.75, 129.82, 128.58, 127.80, 73.02, 64.85, 63.37, 62.44, 40.78, 35.89, 26.98, 25.46, 19.36, 14.14.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.70-7.60 (m, 4H), 7.46-7.34 (m, 6H), 5.67 (dt, *J* = 14.4, 6.9 Hz, 1H), 5.26 (dd, *J* = 15.4, 8.2 Hz, 1H), 4.68-4.58 (m, 1H), 4.47 (dt, *J* = 11.5, 3.6 Hz, 1H), 4.27-4.15 (m, 2H), 3.75 (d, *J* = 11.6 Hz, 1H), 3.64 (t, *J* = 6.5 Hz, 2H), 3.29-3.19 (m, 1H), 2.23 (q, *J* = 6.5 Hz, 2H), 1.94-1.78 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.04 (s, 9H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.25, 135.66, 133.83, 131.99, 129.82, 128.72, 127.89, 72.48, 68.11, 63.29, 62.73, 41.60, 35.86, 30.33, 26.96, 19.34, 14.16.; HRMS (ESI) *m/z* calculated for $C_{27}H_{37}O_6SSi$ $[M+H]^+$: 517.2080, found 517.2062.

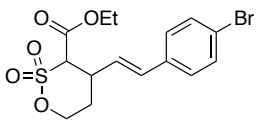
Ethyl (*E*)-4-styryl-1,2-oxathiane-3-carboxylate 2,2-dioxide [10].



Ethyl (*E*)-2-diazo-2-(((5-phenylpent-4-en-1-yl)oxy)sulfonyl)acetate **[S23]** (135.4 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), 100 mg activated powdered 3Å mol sieves, and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15%

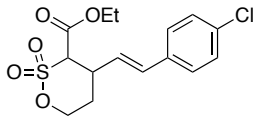
EtOAc/hexanes. Pure product was isolated as a white solid and a 1:1 syn:anti mixture of diastereomers. Subsequent purification using 15% → 20% → 25% EtOAc/hexanes allowed for separation of diastereomers. A trace impurity (<5%) was isolated with this product and was factored into yield calculations. The anti diastereomer was isolated with 12% of the syn.
Run 1: (89.1 mg, 0.287 mmol, 72%), 0% rsm. Run 2: (94.0 mg, 0.303 mmol, 75%), 0% rsm. Run 3: (87.3 mg, 0.281 mmol, 70%), 0% rsm. **Average: 73% yield ± 2.3, 0% rsm.**
Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.36-7.27 (m, 5H), 6.58 (dd, *J* = 15.9, 1.1 Hz, 1H), 6.06 (dd, *J* = 15.9, 7.8 Hz, 1H), 4.76-4.62 (m, 2H), 4.33-4.19 (m, 2H), 4.17 (d, *J* = 4.7 Hz, 1H), 3.51-3.41 (m, 1H), 2.77 (dtd, *J* = 14.5, 12.0, 5.5 Hz, 1H), 1.78 (ddt, *J* = 14.5, 4.3, 2.4 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.03, 135.99, 133.46, 128.79, 128.34, 126.51, 125.86, 72.92, 64.65, 62.54, 40.90, 25.40, 14.09.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.35-7.24 (m, 5H), 6.58 (dd, *J* = 15.7, 0.9 Hz, 1H), 5.94 (dd, *J* = 15.8, 8.5 Hz, 1H), 4.76-4.65 (m, 1H), 4.54 (ddd, *J* = 11.7, 4.2, 2.6 Hz, 1H), 4.30-4.19 (m, 2H), 3.91 (d, *J* = 11.5 Hz, 1H), 3.54-3.42 (m, 1H), 2.08-1.98 (m, 2H), 1.23 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.23, 135.97, 134.07, 128.81, 128.40, 126.55, 125.91, 72.45, 67.94, 62.88, 42.06, 30.41, 14.16.
HRMS (ESI) *m/z* calculated for $C_{15}H_{19}O_5S$ $[M+H]^+$: 311.0943, found 311.0953.

Ethyl (*E*)-4-(4-bromostyryl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [11].



Ethyl (*E*)-2-(((5-(4-bromophenyl)pent-4-en-1-yl)oxy)sulfonyl)-2-diazo-acetate **[S24]** (167 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), 100 mg activated powdered 3Å mol sieves, and CH_2Cl_2 (8.0 mL, 0.5 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 20%→25%→30% EtOAc/hexanes. Pure product was isolated as a yellow oil and a 1:1 syn:anti mixture of diastereomers. Subsequent purification using 10% → 20% → 25% → 30% EtOAc/hexanes allowed for separation of diastereomers. A trace impurity (<5%) was isolated with this product and was factored into yield calculations. The anti diastereomer was isolated with 10% of the syn.
Run 1: (101 mg, 0.260 mmol, 65%), 0% rsm. Run 2: (104 mg, 0.268 mmol, 67%), 0% rsm. Run 3: (106 mg, 0.272 mmol, 68%), 0% rsm. **Average: 67% yield ± 1.2, 0% rsm.**
Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.51 (d, *J* = 16.5 Hz, 1H), 6.07 (dd, *J* = 15.9, 7.8 Hz, 1H), 4.74-4.64 (m, 2H), 4.30-4.21 (m, 2H), 4.16 (d, *J* = 4.7 Hz, 1H), 3.45 (ddt, *J* = 12.1, 8.2, 4.6 Hz, 1H), 2.75 (dtd, *J* = 14.6, 11.8, 5.7 Hz, 1H), 1.82-1.73 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.01, 134.92, 132.41, 131.96, 128.06, 126.68, 122.27, 72.84, 64.54, 62.66, 40.90, 25.39, 14.16.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.51 (d, *J* = 15.7 Hz, 1H), 5.93 (dd, *J* = 15.8, 8.6 Hz, 1H), 4.77-4.65 (m, 1H), 4.54 (dt, *J* = 11.6, 3.4 Hz, 1H), 4.29-4.20 (m, 2H), 3.90 (d, *J* = 11.5 Hz, 1H), 3.54-3.42 (m, 1H), 2.06-1.97 (m, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ163.18, 134.86, 132.90, 131.90, 128.04, 126.68, 122.21, 72.43, 67.69, 62.88, 42.02, 30.21, 14.15.
HRMS (ESI) *m/z* calculated for $C_{15}H_{18}O_5SBr$ $[M+H]^+$: 389.0058, found 389.0045.

Ethyl (*E*)-4-(4-chlorostyryl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [12].



Ethyl (*E*)-2-(((5-(4-chlorophenyl)pent-4-en-1-yl)oxy)sulfonyl)-2-diazoacetate **[S25]** (149.1 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 20% EtOAc/hexanes. Pure product was isolated as a white solid and a 1.4:1 syn:anti mixture of diastereomers. Subsequent purification using 15% → 20% → 25% EtOAc/hexanes allowed for separation of diastereomers. A trace impurity (<5%) was isolated with this product and was factored into yield calculations. The syn diastereomer was isolated with 6% of the anti, and the anti diastereomer was isolated with 10% of the syn.

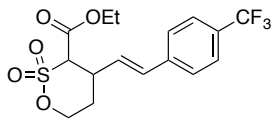
Run 1: (85.2 mg, 0.247 mmol, 62%), 0% rsm. Run 2: (78.5 mg, 0.228 mmol, 57%), 0% rsm. Run 3: (82.6 mg, 0.240 mmol, 60%), 0% rsm. **Average: 60% yield ± 2.0, 0% rsm.**

Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.33-7.23 (m, 4H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.05 (dd, *J* = 15.9, 7.8 Hz, 1H), 4.75-4.62 (m, 2H), 4.30-4.21 (m, 2H), 4.16 (d, *J* = 4.7 Hz, 1H), 3.51-3.41 (m, 1H), 2.75 (dtd, *J* = 14.5, 11.6, 5.4 Hz, 1H), 1.82-1.72 (m, 1H), 1.25 (td, *J* = 7.1, 1.0 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ164.00, 134.49, 134.05, 132.31, 128.99, 127.75, 126.56, 72.85, 64.58, 62.63, 40.87, 25.40, 14.14.

Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.45-7.10 (m, 4H), 6.53 (d, *J* = 15.7 Hz, 1H), 5.91 (dd, *J* = 15.8, 8.5 Hz, 1H), 4.76-4.67 (m, 1H), 4.54 (ddd, *J* = 11.6, 4.1, 2.6 Hz, 1H), 4.31-4.18 (m, 2H), 3.90 (d, *J* = 11.6 Hz, 1H), 3.47 (ddd, *J* = 16.7, 11.4, 8.3 Hz, 1H), 2.07-1.98 (m, 2H), 1.22 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.20, 134.43, 134.15, 132.92, 129.02, 127.77, 126.57, 72.38, 67.83, 62.93, 42.04, 30.35, 14.19.

HRMS (ESI) *m/z* calculated for $C_{15}H_{18}O_5SCl$ $[M+H]^+$: 345.0563, found 345.0561.

Ethyl (*E*)-4-(4-(trifluoromethyl)styryl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [13].



Ethyl(*E*)-2-diazo-2-(((5-(4-(trifluoromethyl)phenyl)pent-4-en-1-yl)oxy)-sulfonyl)acetate **[S26]** (163 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBArF_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), 100 mg activated powdered 3Å mol sieves, and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% EtOAc/hexanes. Pure product was isolated as a dark brown oil and a 1:1 syn:anti mixture of diastereomers. Subsequent purification using 15% EtOAc/hexanes allowed for separation of diastereomers. The syn diastereomer was isolated with 10% of the anti.

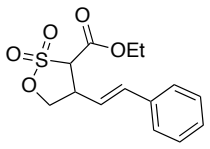
Run 1: (65.1 mg, 0.172 mmol, 43%), 0% rsm. Run 2: (65.1 mg, 0.172 mmol, 43%), 0% rsm. Run 3: (69.6 mg, 0.184 mmol, 46%), 0% rsm. **Average: 44% yield ± 1.7, 0% rsm.**

Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.2 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.18 (dd, *J* = 15.9, 7.7 Hz, 1H), 4.74-4.65 (m, 2H), 4.30-4.21 (m, 2H), 4.18 (d, *J* = 4.7 Hz, 1H), 3.50 (ddt, *J* = 11.7, 7.7, 3.9 Hz, 1H), 2.78 (dtd, *J* = 14.5, 11.7, 5.7 Hz, 1H), 1.83–1.76 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ164.09, 139.59, 132.38, 130.30 (q, J = 32.3 Hz), 128.79, 126.89, 125.93 (q, J = 3.8 Hz), 124.25 (q, J = 273.42 Hz), 72.93, 64.63, 62.83, 41.00, 25.50, 14.26.

Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.1 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 6.61 (d, *J* = 15.8 Hz, 1H), 6.05 (dd, *J* = 15.8, 8.5 Hz, 1H), 4.73 (td, *J* = 11.6, 3.8

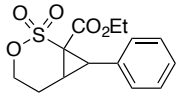
Hz, 1H), 4.55 (ddd, *J* = 11.4, 4.3, 2.1 Hz, 1H), 4.29-4.20 (m, 2H), 3.94 (d, *J* = 11.5 Hz, 1H), 3.51 (ddt, *J* = 15.9, 11.2, 5.2 Hz, 1H), 2.11 – 1.97 (m, 2H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.29, 139.54, 132.90, 130.31 (q, *J* = 32.3 Hz), 128.81, 126.88, 125.91 (q, *J* = 3.8 Hz), 124.25 (q, *J* = 272.16 Hz), 72.50, 67.80, 63.08, 42.11, 30.33, 14.27.
HRMS (ESI) m/z calculated for $C_{16}H_{18}O_5SF_3$ $[M+H]^+$: 379.0827, found 379.0825.

Ethyl (*E*)-4-styryl-1,2-oxathiolane-3-carboxylate 2,2-dioxide [14].



[FePc] Conditions: Ethyl (*E*)-2-diazo-2-(((4-phenylbut-3-en-1-yl)oxy)sulfonyl)acetate **[S27]** (129.7 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), 100 mg activated powdered 3Å mol sieves, and CH_2Cl_2 (8.0 mL, 0.05 M) were used. After the reaction was judged complete by TLC, the mixture was transferred to a 25 mL round-bottom flask, concentrated, and filtered through a celite plug with diethyl ether to remove the catalyst. This was then concentrated and analyzed by crude NMR which revealed that no cyclopropanated product was formed and the C—H insertion product was an 11:1 inseparable mixture of diastereomers. The product was purified via flash column chromatography on silica (75 mL SiO_2) using 10% EtOAc/hexanes. Pure C—H insertion product was isolated as a colorless oil. Relative stereochemistry was not assigned. Only characterization of the major diastereomer is reported.
Run 1: (72.4 mg, 0.244 mmol, 61%), 0% rsm. Run 2: (73.1 mg, 0.246 mmol, 62%), 0% rsm. Run 3: (70.0 mg, 0.236 mmol, 59%), 0% rsm. **Average: 61% yield C—H ins. ± 1.1, 0% rsm.**
$Rh_2(OAc)_4$ Conditions: The general procedure for $Rh_2(OAc)_4$ catalyzed C—H alkylation was followed. Ethyl (*E*)-2-diazo-2-(((4-phenylbut-3-en-1-yl)oxy)sulfonyl)acetate **[S27]** (137.3 mg, 0.400 mmol, 1.0 equiv), $Rh_2(OAc)_4$ (3.5 mg, 0.008 mmol, 0.02 equiv), and CH_2Cl_2 (8 mL, 0.05 M) were used. This was then concentrated and analyzed by crude NMR which revealed a 1:4 ratio of C—H insertion product to cyclopropanated product. The crude mixture was purified via flash column chromatography on silica (75 mL SiO_2) using 10% → 15% → 20% EtOAc/hexanes. Pure C—H insertion product was isolated as a colorless oil and pure cyclopropanated product (more polar) was isolated as a white solid.
Run 1: (16.5 mg, 0.056 mmol, 14% C—H ins.), (55.8 mg, 0.188 mmol, 47% cycloprop.), 0% rsm. Run 2: (14.2 mg, 0.048 mmol, 12% C—H ins.), (53.4 mg, 0.180 mmol, 45% cycloprop.), 0% rsm. Run 3: (11.8 mg, 0.040 mmol, 10% C—H ins.), (54.6 mg, 0.184 mmol, 46% cycloprop.), 0% rsm. **Average: 12% yield C—H ins. ± 1.6, 46% yield cycloprop. ± 0.9, 0% rsm.**
^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.39-7.27 (m, 5H), 6.68 (d, *J* = 15.7 Hz, 1H), 6.04 (dd, *J* = 15.7, 8.5 Hz, 1H), 4.66 (dd, *J* = 9.2, 7.4 Hz, 1H), 4.39 (dq, *J* = 10.8, 7.2 Hz, 1H), 4.30 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.22 (br. t, *J* = 9.0 Hz, 1H), 4.12 (dt, *J* = 16.5, 8.2 Hz, 1H), 4.05 (d, *J* = 8.9 Hz, 1H), 1.35 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.05, 135.97, 135.38, 128.81, 128.69, 126.64, 122.34, 71.08, 64.69, 63.47, 44.12, 13.99.; HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_5S$ $[M+H]^+$: 297.0797, found 297.0786.

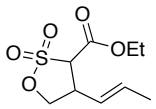
Ethyl 7-phenyl-3-oxa-2-thiabicyclo[4.1.0]heptane-1-carboxylate 2,2-dioxide [S37].



^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.43-7.12 (m, 5H), 4.75 (td, *J* = 11.6, 4.7 Hz, 1H), 4.45 (ddd, *J* = 11.9, 6.3, 2.5 Hz, 1H), 4.10-3.96 (m, 2H), 3.26 (d, *J* = 8.6 Hz, 1H), 3.12 (td, *J* = 8.5, 2.2 Hz, 1H), 2.41 (dddd, *J* = 14.6, 8.5, 4.8, 2.5 Hz, 1H), 2.31

(dddd, J = 14.8, 11.4, 6.3, 2.3 Hz, 1H), 0.98 (t, J = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ162.89, 132.22, 128.56, 128.52, 128.25, 71.15, 62.89, 49.86, 39.08, 27.96, 21.75, 13.80.; HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_5S$ $[M+H]^+$: 297.0797, found 297.0788.

Ethyl (*E*)-4-(prop-1-en-1-yl)-1,2-oxathiolane-3-carboxylate 2,2-dioxide [15].



[FePc] Conditions: Ethyl (*E*)-2-((pent-3-en-1-yloxy)sulfonyl)acetate **[S28]** (104.9 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), 100 mg activated powdered 3Å mol sieves, and CH_2Cl_2 (8.0 mL, 0.05 M) were used. After the reaction was judged complete by TLC, the mixture was transferred to a 25 mL round-bottom flask, concentrated, and filtered through a celite plug with diethyl ether to remove the catalyst. This was then concentrated and analyzed by crude NMR which revealed that no cyclopropanated product was formed and the C—H insertion product was a 10:1 inseparable mixture of diastereomers. The product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% EtOAc/hexanes. Pure C—H insertion product was isolated as a colorless oil. Relative stereochemistry was not assigned. Only characterization of the major diastereomer is reported.

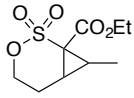
Run 1: (40.0 mg, 0.171 mmol, 43%), 0% rsm. Run 2: (39.8 mg, 0.170 mmol, 43%), 0% rsm. Run 3: (42.5 mg, 0.181 mmol, 45%), 0% rsm. **Average: 44% yield ± 1.3, 0% rsm.**

$Rh_2(OAc)_4$ Conditions: The general procedure for $Rh_2(OAc)_4$ catalyzed C—H alkylation was followed. Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[S28]** (137.3 mg, 0.400 mmol, 1.0 equiv), $Rh_2(OAc)_4$ (3.5 mg, 0.008 mmol, 0.02 equiv), and CH_2Cl_2 (8 mL, 0.05 M) were used. This was then concentrated and analyzed by crude NMR which revealed a 1:1 ratio of C—H insertion product to cyclopropanated product. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% → 25% EtOAc/hexanes. Pure C—H insertion product was isolated as a colorless oil and pure cyclopropanated product (more polar) was isolated as a white solid.

Run 1: (17.6 mg, 0.075 mmol, 19% C—H ins.), (23.6 mg, 0.101 mmol, 25% cycloprop.), 0% rsm. Run 2: (17.4 mg, 0.074 mmol, 19% C—H ins.), (29.9 mg, 0.127 mmol, 32% cycloprop.), 0% rsm. Run 3: (18.7 mg, 0.080 mmol, 20% C—H ins.), (29.2 mg, 0.124 mmol, 31% cycloprop.), 0% rsm. **Average: 19% yield C—H ins. ± 0.6, 29% yield cycloprop. ± 2.9, 0% rsm.**

^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.80 (dqd, J = 15.3, 6.6, 0.8 Hz, 1H), 5.34 (ddq, J = 15.2, 7.9, 1.7 Hz, 1H), 4.55 (dd, J = 9.1, 7.2 Hz, 1H), 4.37 (dq, J = 10.8, 7.1 Hz, 1H), 4.30 (dq, J = 10.6, 7.1 Hz, 1H), 4.12-4.05 (m, 1H), 3.94- 3.85 (m, 2H), 1.72 (dd, J = 6.3, 1.8 Hz, 3H), 1.34 (t, J = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ163.23, 132.79, 124.66, 71.35, 64.90, 63.45, 43.90, 18.08, 14.09.; HRMS (ESI) *m/z* calculated for $C_9H_{14}O_5SNa$ $[M+Na]^+$: 257.0460, found 257.0452.

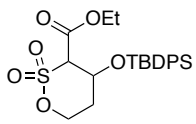
Ethyl 7-methyl-3-oxa-2-thiabicyclo[4.1.0]heptane-1-carboxylate 2,2-dioxide [S38].



^{1}H-NMR (500 MHz, $CDCl_3$) δ4.64 (ddd, J = 11.9, 11.2, 5.0 Hz, 1H), 4.42- 4.29 (m, 3H), 2.35 (td, J = 8.2, 2.3 Hz, 1H), 2.23 (dddd, J = 11.1, 10.3, 5.0, 2.7 Hz, 1H), 2.15 (dddd, J = 17.1, 11.2, 6.2, 2.3 Hz, 1H), 1.99 (dq, J = 8.4, 6.3 Hz, 1H), 1.36 (t, J = 7.1 Hz, 3H), 1.19 (d, J = 6.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ164.33, 70.76, 63.04, 47.84, 31.03,

30.05, 21.53, 14.17, 12.21.; HRMS (ESI) *m/z* calculated for $C_9H_{14}O_5SNa$ $[M+Na]^+$: 257.0460, found 257.0457.

Ethyl 4-((*tert*-butyldiphenylsilyl)oxy)-1,2-oxathiane-3-carboxylate 2,2-dioxide [16].

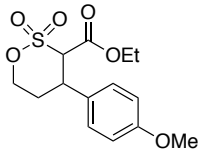


Ethyl 2-((3-((*tert*-butyldiphenylsilyl)oxy)propoxy)sulfonyl)-2-diazoacetate **[S29]** (98.1 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.025 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 5% → 10% → 15 → 20% EtOAc/hexanes. Pure product was isolated as a white solid and a 1:1 syn:anti mixture of diastereomers. The diastereomers were inseparable by column chromatography and were characterized as a mixture.

Run 1: (62.2 mg, 0.134 mmol, 67%), 0% rsm. Run 2: (64.3 mg, 0.139 mmol, 69%), 0% rsm. Run 3: (61.9 mg, 0.134 mmol, 67%), 0% rsm. **Average: 68% yield ± 1.2, 0% rsm.**

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.71-7.60 (m, 4H D_{maj} + 4H D_{min}), 7.51-7.44 (m, 2H D_{maj} + 2H D_{min}), 7.44-7.33 (m, 4H D_{maj} + 4H D_{min}), 4.62-4.56 (m, 1H D_{maj}), 4.48 (dt, *J* = 10.5, 5.0 Hz, 1H D_{min}), 4.43 (ddd, *J* = 11.9, 5.2, 2.5 Hz, 1H D_{min}), 4.38-4.30 (m, 1H D_{maj} + 1H D_{min}), 4.29-4.18 (m, 2H D_{maj} + 2H D_{min}), 4.18-4.09 (m, 1H D_{maj} + 1H D_{min}), 4.01 (d, *J* = 9.1 Hz, 1H D_{maj}), 2.75-2.64 (m, 1H D_{min}), 1.88 (h, *J* = 4.6, 4.1 Hz, 2H D_{maj}), 1.64 (ddt, *J* = 14.2, 4.7, 2.5 Hz, 1H D_{min}), 1.31 (dt, *J* = 8.3, 7.1 Hz, 3H D_{maj} + 3H D_{min}), 1.05 (s, 9H D_{min}), 1.01 (s, 9H D_{maj}).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.34, 163.04, 135.85, 135.83, 135.80, 135.74, 135.62, 132.98, 132.54, 132.14, 132.03, 130.64, 130.51, 130.46, 130.31, 129.83, 128.20, 128.11, 128.09, 127.99, 127.85, 70.17, 69.90, 68.99, 68.74, 68.43, 66.37, 63.00, 62.65, 33.01, 28.94, 26.78, 26.76, 19.35, 19.23, 14.07, 14.04.; HRMS (ESI) *m/z* calculated for $C_{23}H_{30}O_6SSiNa$ $[M+Na]^+$: 485.1430, found 485.1417.

Ethyl 4-(4-methoxyphenyl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [17].



Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy)sulfonyl)acetate **[30]** (137 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 10% → 25% EtOAc/hexanes. Pure product was isolated as a clear oil and a 1.1:1 syn:anti mixture of diastereomers. Subsequent purification using 10% → 25% EtOAc/hexanes allowed for separation of diastereomers. The anti diastereomer was isolated with 15% of the syn.

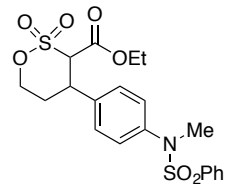
Run 1: (84.1 mg, 0.268 mmol, 67%), 0% rsm. Run 2: (75.0 mg, 0.239 mmol, 60%), 0% rsm. Run 3: (85.5 mg, 0.272 mmol, 68%), 0% rsm. **Average: 65% yield ± 3.6%, 0% rsm.**

Syn diastereomer (major): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.15 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 4.81 – 4.63 (m, 2H), 4.21 (d, J = 4.7 Hz, 1H), 4.18 – 3.93 (m, 2H), 3.87 - 3.80 (m, 1H), 3.79 (s, 3H), 3.16 (tdd, J = 13.9, 11.7, 6.6 Hz, 1H), 1.80 (dd, J = 13.2, 1.8 Hz, 1H), 1.05 (t, J = 7.2, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.11, 159.43, 129.94, 128.42, 114.45, 73.20, 66.51, 62.26, 55.41, 42.25, 24.34, 13.84.

Anti diastereomer (minor): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.14 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 4.74 (ddd, J = 13.4, 11.6, 2.2 Hz, 1H), 4.54 (ddd, J = 11.6, 4.9, 1.5 Hz, 1H), 4.17 (d, J = 12 Hz, 1H), 4.12 - 4.01 (m, 2H), 3.82 - 3.73 (m, 4H), 2.21 (dtd, J = 15.0, 13.0, 4.9 Hz,

1H), 1.99 (d, J = 14.8, 1H), 1.05 (t, J = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.95, 159.42, 130.63, 128.60, 128.46, 114.52, 72.65, 69.01, 62.67, 55.43, 43.68, 32.53, 13.89. HRMS (ESI) *m/z* calculated for $C_{14}H_{18}O_6SNa$ $[M+Na]^+$: 337.0721, found 337.0722.

Ethyl 4-(4-(*N*-methylphenylsulfonamido)phenyl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [18].

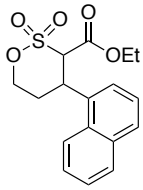


Ethyl 2-diazo-2-((3-(4-(*N*-methylphenylsulfonamido)phenyl) propoxy)sulfonyl) acetate **[S30]** (96.3 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F{}_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (4.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 10% → 30% EtOAc/hexanes. Pure product was isolated as a clear oil and a 1:1 syn:anti mixture of diastereomers. The diastereomers were inseparable by column chromatography and were characterized as a mixture.

Run 1: (65.7 mg, 0.145 mmol, 72%), 0% rsm. Run 2: (59.8 mg, 0.132 mmol, 66%), 0% rsm. Run 3: (55.7 mg, 0.123 mmol, 76% (scale: 76.9 mg, 0.160 mmol)), 0% rsm. **Average: 71% yield ± 4.1%, 0% rsm.**

Mixture of syn and anti diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (td, *J* = 6.5, 5.5, 3.6 Hz, 1H D_{syn} + 1H D_{anti}), 7.53 (ddd, *J* = 8.3, 4.7, 1.4 Hz, 2H D_{syn} + 2H D_{anti}), 7.47 (m, 2H D_{syn} + 2H D_{anti}), 7.21 (dd, *J* = 8.3, 5.3 Hz, 2H D_{syn} + 2H D_{anti}), 7.11 (dd, *J* = 8.4, 6.1 Hz, 2H D_{syn} + 2H D_{anti}), 4.82 - 4.71 (m, 1H D_{syn} + 2H D_{anti}), 4.59 (dd, J = 11.5, 4.5 Hz, 1H D_{syn}), 4.28 - 4.21 (m, 1H D_{syn}), 4.17 - 4.02 (m, 2H D_{syn} + 2H D_{anti}), 3.92 - 3.80 (m, 1H D_{syn} + 1H D_{anti}), 3.25 - 3.11 (m, 3H D_{syn} + 4H D_{anti}), 2.26 (dq, J = 13.5, 5.0 Hz, 1H D_{syn}), 2.04 (dd, *J* = 14.7, 3.7 Hz, 1H D_{anti}), 1.86 (dd, *J* = 14.5, 3.2 Hz, 1H D_{anti}), 1.09 (td, *J* = 7.1, 2.3 Hz, 3H D_{syn} + 3H D_{anti}). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.84, 162.73, 141.47, 141.37, 137.64, 136.91, 136.41, 133.05, 128.91, 128.02, 127.86, 127.82, 127.77, 127.75, 127.07, 127.06, 73.11, 72.66, 68.47, 66.11, 62.69, 62.37, 43.81, 42.37, 38.06, 32.15, 24.02, 13.87.; HRMS (ESI) *m/z* calculated for $C_{20}H_{24}NO_7S_2$ $[M+H]^+$: 454.0994, found 454.1003.

Ethyl 4-(naphthalen-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [19].



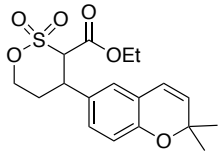
Ethyl 2-((3-(naphthalen-1-yl)propoxy)sulfonyl)acetate **[S31]** (72.4 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F{}_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (4.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 0.4% Et_2O, 19.6% DCM, 80% hexanes → 1.2% Et_2O, 58.8% CH_2Cl_2, 40% hexanes as eluent. Impure product was isolated as a clear oil and a 1.2:1 syn:anti mixture of diastereomers. Yield was determined with mesitylene as an internal standard. Subsequent purification using 0.4% Et_2O, 19.6% CH_2Cl_2, 80% hexanes → 1% Et_2O, 49% CH_2Cl_2, 50% hexanes as eluent allowed for separation of diastereomers. A trace impurity (<5%) was isolated with the anti product and was factored into yield calculations.

Run 1: (34.8 mg, 0.104 mmol, 52%), 0% rsm. Run 2: (34.8 mg, 0.104 mmol, 52%), 0% rsm. Run 3: (35.4 mg, 0.106 mmol, 53%), 0% rsm. **Average: 52% yield ± 0.5%, 0% rsm.**

Syn diastereomer (major): ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.5 Hz, 1H), 7.92 (dd, J = 8.1, 1.4 Hz, 1H), 7.83 (dd, J = 7.9, 1.4 Hz, 1H), 7.64 (ddd, J = 8.5, 6.8, 1.5 Hz, 1H), 7.56 (ddd,

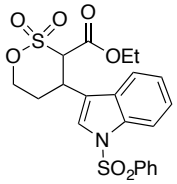
J = 8.1, 6.9, 1.1 Hz, 1H), 7.47 – 7.36 (m, 2H), 4.90 (ddd, J = 12.9, 11.5, 2.1 Hz, 1H), 4.81 (ddd, J = 11.5, 5.0, 1.5 Hz, 1H), 4.73 (dt, J = 13.1, 3.8 Hz, 1H), 4.47 (d, J = 4.4 Hz, 1H), 4.00 – 3.83 (m, 2H), 3.49 (dq, J = 16.5 6 Hz, 1H), 1.89 (d, J = 14.3 Hz, 1H), 0.86 (t, J = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.93, 134.15, 133.19, 130.46, 129.67, 129.09, 127.57, 126.32, 125.36, 124.31, 121.68, 73.53, 65.10, 62.18, 38.30, 24.31, 13.76.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.6 Hz, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.79 (d, *J* = 8.3 Hz, 1H), 7.60 (ddd, *J* = 8.5, 6.8, 1.4 Hz, 1H), 7.53 (ddd, *J* = 8.0, 6.8, 1.1 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 1H), 7.32 (dd, *J* = 7.1, 1.2 Hz, 1H), 4.89 (td, J = 11.6 Hz, 3.7 Hz, 1H), 4.77 (td, *J* = 11.8, 4.7 Hz, 1H), 4.58 (ddd, J = 12.0, 4.5, 2.0 Hz, 1H), 4.55 (d, J = 12.0 Hz, 1H), 4.01 - 3.88 (m, 2H), 2.27 - 2.13 (m, 2H), 0.85 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.83, 135.58, 134.18, 130.93, 129.22, 128.69, 127.02, 126.37, 125.43, 123.12, 122.57, 72.85, 68.70, 62.76, 38.24, 33.13, 13.66.
HRMS (ESI) *m/z* calculated for $C_{17}H_{19}O_5S$ $[M+H]^+$: 335.0953, found 335.0954.

Ethyl 4-(2,2-dimethyl-2*H*-chromen-6-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [20].



Ethyl 2-diazo-2-((3-(2,2-dimethyl-2*H*-chromen-6-yl)propoxy)sulfonyl)acetate **[S32]** (75.7 mg, 0.192 mmol, 1.0 equiv), [FePc]Cl (11.6 mg, 0.0192 mmol, 0.10 equiv), $NaBAr^F_4$ (17.0 mg, 0.0192 mmol, 0.10 equiv), and CH_2Cl_2 (3.80 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 10% → 30% EtOAc/hexanes. Pure product was isolated as a clear oil and a 1:1.2 syn:anti mixture of diastereomers. Subsequent purification with 12.5% → 27.5% Et_2O/hexanes separated the diastereomers. The anti diastereomer was isolated with 10% of the syn.
Run 1: (40.1 mg, 0.1094 mmol, 57%), 0% rsm. Run 2: (41.9 mg, 0.1143 mmol, 60%), 0% rsm. Run 3: (56.3 mg, 0.1536 mmol, 61% (scale: 92.7 mg, 0.253 mmol)), 0% rsm. **Average: 59% yield ± 1.7%, 0% rsm.**
Syn diastereomer (major): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.96 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.83 (d, *J* = 2.3 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 1H), 6.27 (d, J = 9.5 Hz, 1H), 5.64 (d, *J* = 9.8 Hz, 1H), 4.77 - 4.62 (m, 2H), 4.20 (d, *J* = 4.7 Hz, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 3.75 (dt, *J* = 13.4, 4.1 Hz, 1H), 3.14 (tdd, *J* = 13.9, 11.6, 6.5 Hz, 1H), 1.78 (ddt, J = 14.3, 3.4, 1.7 Hz, 1H), 1.41 (s, 3H), 1.42 (s, 3H), 1.06 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.20, 152.90, 131.71, 130.07, 127.89, 125.17, 121.98, 121.80, 116.95, 76.66, 73.19, 66.49, 62.31, 42.43, 28.10, 24.36, 13.91.
Anti diastereomer (minor): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.94 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.81 (d, *J* = 2.3 Hz, 1H), 6.72 (d, *J* = 8.3 Hz, 1H), 6.26 (d, J = 10 Hz, 1H), 5.63 (d, *J* = 9.8 Hz, 1H), 4.73 (ddd, *J* = 13.4, 11.6, 2.2 Hz, 1H), 4.53 (ddd, *J* = 11.6, 4.9, 1.5 Hz, 1H), 4.15 (d, *J* = 12.1 Hz, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.72 (td, *J* = 12.5, 3.7 Hz, 1H), 2.20 (dtd, *J* = 14.9, 13.0, 5.0 Hz, 1H), 2.03 - 1.96 (m, 1H), 1.41 (s, 6H), 1.05 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.97, 152.83, 131.64, 130.66, 127.95, 125.41, 121.94, 116.89, 76.72, 72.62, 68.96, 62.67, 43.76, 32.45, 28.13, 13.90.
HRMS (ESI) *m/z* calculated for $C_{18}H_{23}O_6S$ $[M+H]^+$: 367.1215, found 367.1220.

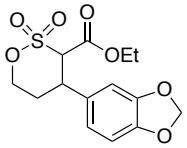
Ethyl 4-(1-(phenylsulfonyl)-1H-indol-3-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [21].



Ethyl 2-diazo-2-((3-(1-(phenylsulfonyl)-1H-indol-3-yl)propoxy)sulfonyl)acetate **[S33]** (196.6 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10

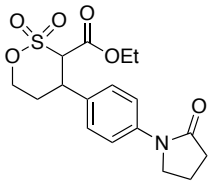
equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 5% → 25% EtOAc/hexanes. Pure product was isolated as a clear oil and a 1.1:1 syn:anti mixture of diastereomers. Diastereomers could not be separated.
Run 1: (118 mg, 0.255 mmol, 64%), 0% rsm. Run 2: (119 mg, 0.256 mmol, 64%), 0% rsm. Run 3: (116 mg, 0.250 mmol, 63%), 0% rsm. **Average: 64% yield ± 0.5%, 0% rsm.**
Mixture of syn and anti diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (dd, *J* = 13.0, 8.3 Hz, 1H D_{maj} + 1H D_{min}), 7.90 - 7.81 (m, 2H D_{maj} + 2H D_{min}), 7.61 - 7.52 (m, 2H D_{maj} + 2H D_{min}), 7.50 - 7.42 (m, 3H D_{min} + 3H D_{min}), 7.41 - 7.23 (m, 2H D_{maj} + 2H D_{min}), 4.88 –4.66 (m, 2H D_{maj} + 1H D_{min}), 4.57 (ddd, *J* = 11.8, 4.8, 1.6 Hz, 1H D_{min}), 4.37 (d, *J* = 4.6 Hz, 1H D_{maj}), 4.33 (d, *J* = 12.0 Hz, 1H D_{min}), 4.11 (ddd, *J* = 12.5, 8.2, 3.8 Hz, 1H D_{maj} + 1H D_{min}), 4.03 - 3.71 (m, 2H D_{maj} + 2H D_{min}), 3.19 (qd, *J* = 13.3, 5.2 Hz, 1H D_{maj}), 2.34 - 2.21 (m, 1H D_{min}), 2.19 - 2.11 (m, 1H D_{min}), 1.96 - 1.88 (m, 1H D_{maj}), 0.88 (t, *J* = 7.1 Hz, 3H D_{maj}), 0.80 (t, *J* = 7.1 Hz, 3H D_{min}). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.84, 162.73, 141.47, 141.37, 137.64, 136.91, 136.41, 133.05, 128.91, 128.02, 127.90, 127.86, 127.82, 127.77, 127.75, 127.07, 127.06, 73.11, 72.66, 68.47, 66.11, 62.69, 62.37, 43.81, 42.37, 38.06, 32.15, 24.02, 13.87. HRMS (ESI) *m/z* calculated for $C_{21}H_{22}NO_7S_2$ $[M+H]^+$: 464.0838, found 464.0829.

Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [22].



Ethyl 2-((3-(benzo[*d*][1,3]dioxol-5-yl)propoxy)sulfonyl)-2-diazoacetate **[S34]** (142.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8,0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% EtOAc/hexanes. Pure product was isolated as a white solid and a 2:1 syn:anti mixture of diastereomers. Subsequent purification using 15% → 20% → 25% EtOAc/hexanes allowed for separation of diastereomers.
Run 1: (71.1 mg, 0.217 mmol, 54%), 0% rsm. Run 2: (68.8 mg, 0.210 mmol, 52%), 0% rsm. Run 3: (70.6 mg, 0.215 mmol, 54%), 0% rsm. **Average: 53% yield ± 0.8, 0% rsm.**
Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.77 (d, *J* = 7.9 Hz, 1H), 6.74 – 6.68 (m, 2H), 5.96 (s, 2H), 4.76 – 4.66 (m, 2H), 4.19 (d, *J* = 4.6 Hz, 1H), 4.16 – 4.03 (m, 2H), 3.77 (dt, *J* = 13.3, 4.0 Hz, 1H), 3.13 (tdd, *J* = 13.8, 11.6, 6.4 Hz, 1H), 1.85 – 1.72 (m, 1H), 1.11 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ164.08, 148.26, 147.49, 131.73, 120.74, 108.76, 107.72, 101.43, 73.09, 66.52, 62.39, 42.75, 24.50, 13.91.; HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_7S$ $[M+H]^+$: 329.0695, found 329.0681.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.76 (d, *J* = 8.0 Hz, 1H), 6.71-6.67 (m, 2H), 5.96 (s, 2H), 4.73 (ddd, *J* = 12.9, 11.7, 2.2 Hz, 1H), 4.53 (ddd, *J* = 11.7, 4.9, 1.5 Hz, 1H), 4.18-4.06 (m, 3H), 3.75 (td, *J* = 12.4, 3.7 Hz, 1H), 2.18 (dtd, *J* = 14.8, 12.9, 4.9 Hz, 1H), 2.00 (ddt, *J* = 14.9, 3.6, 1.9 Hz, 1H), 1.11 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.85, 148.18, 147.43, 132.35, 120.90, 108.76, 107.77, 101.41, 72.58, 68.87, 62.71, 44.05, 32.50, 13.91.; HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_7S$ $[M+H]^+$: 329.0695, found 329.0688.

Ethyl 4-(4-(2-oxopyrrolidin-1-yl)phenyl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [23].

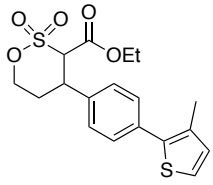


Ethyl 2-diazo-2-((3-(4-(2-oxopyrrolidin-1-yl)phenyl)propoxy)sulfonyl)acetate **[S35]** (75.3 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F{}_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (4.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 30% → 60% EtOAc/hexanes. Product was isolated as a clear oil and a 1.1:1 syn:anti mixture of diastereomers. The diastereomers were inseparable by column chromatography and were characterized as a mixture. Yield was determined via NMR with mesitylene as an internal standard.

Run 1: (29.2 mg, 0.080 mmol, 40%), 0% rsm. Run 2: (28.5 mg, 0.078 mmol, 39%), 0% rsm. Run 3: (29.2 mg, 0.080 mmol, 40%), 0% rsm. **Average: 40% yield ± 0.5%, 0% rsm.**

Mixture of syn and anti diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 8.7, 6.9 Hz, 2H D_{maj} + 2H D_{min}), 7.25 (dd, *J* = 8.5, 5.3 Hz, 2H D_{maj} + 2H D_{min}), 4.82 - 4.69 (m, 2H D_{maj} + 1H D_{min}), 4.56 (dd, J = 11.5, 4.0 Hz, 1H D_{min}), 4.26 (d, *J* = 3.8 Hz, 1H D_{maj}), 4.24 (d, *J* = 3.6 Hz, 1H D_{min}), 4.14 - 4.00 (m, 2H D_{maj} + 2H D_{min}), 3.92 - 3.78 (m, 3H D_{maj} + 3H D_{min}), 3.21 (dq, J = 14.0, 6.4 Hz, 1H D_{maj}), 2.63 (td, J = 8.1, 2.9 Hz, 2H D_{maj} + 2H D_{min}), 2.29-2.15 (m, 2H D_{maj} + 3H D_{min}), 2.00 (d, J = 14.5, 1H D_{min}), 1.82 (d, J = 14 Hz, 1H D_{maj}), 1.09 (t, J = 7.1 Hz, 3H D_{maj} + 3H D_{min}). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.47, 163.98, 162.80, 139.38, 139.28, 134.46, 133.67, 127.88, 127.76, 120.24, 120.17, 73.18, 72.66, 68.58, 66.25, 62.69, 62.36, 48.69, 48.66, 43.70, 42.39, 32.79, 32.34, 24.20, 18.01, 17.98, 13.85, 13.83. HRMS (ESI) *m/z* calculated for $C_{17}H_{22}NO_6S$ $[M+H]^+$: 368.1168, found 368.1167.

Ethyl 4-(4-(3-methylthiophen-2-yl)phenyl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [24].



Ethyl 2-diazo-2-((3-(4-(3-methylthiophen-2-yl)phenyl)propoxy)sulfonyl)acetate **[S36]** (81.7 mg, 0.2 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F{}_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.025 M) were used. Product was purified via flash column chromatography on silica using 0.4% Et_2O, 19.6% CH_2Cl_2, 80% hexanes → 1% Et_2O, 49% CH_2Cl_2, 50% hexanes as eluent. Pure product was isolated as a clear oil and a 1:1.3 syn:anti mixture of diastereomers. Subsequent purification using 0.4% Et_2O, 19.6% CH_2Cl_2, 80% hexanes → 1% Et_2O, 49% CH_2Cl_2, 50% hexanes as eluent allowed for separation of diastereomers. Yields were determined via NMR with mesitylene as an internal standard. The syn diastereomer was isolated with 10% of the anti, and the anti diastereomer was isolated with 5% syn.

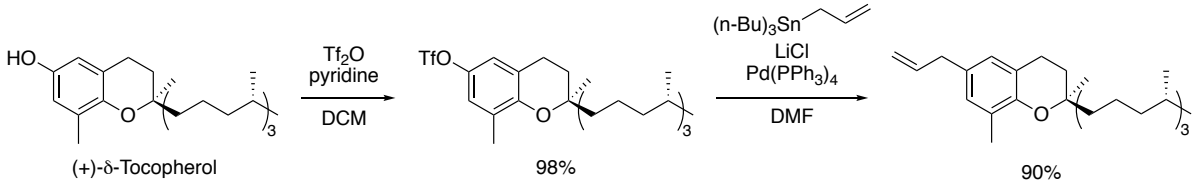
Run 1: (40.4 mg, 0.106 mmol, 53%), 0% rsm. Run 2: (37.9 mg, 0.099 mmol, 49%), 0% rsm. Run 3: (38.2 mg, 0.100 mmol, 52%), 0% rsm. **Average: 51% yield ± 1.7%, 0% rsm.**

Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 5.1 Hz, 1H), 6.93 (d, *J* = 5.1 Hz, 1H), 4.82 - 4.70 (m, 2H), 4.28 (d, *J* = 4.6 Hz, 1H), 4.07 (q, *J* = 7.1 Hz, 2H), 3.90 (dt, J = 13.5 Hz, 4 Hz, 1H), 3.32 - 3.17 (m, 1H), 2.31 (s, 3H), 1.86 (ddt, J = 14.7, 3.5, 1.7 Hz, 1H), 1.04 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.05, 136.92, 136.68, 134.97, 133.68, 131.40, 129.63, 127.53, 123.91, 73.18, 66.24, 62.39, 42.72, 24.11, 15.07, 13.87.

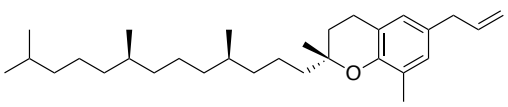
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.2 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.21 (d, *J* = 5.1 Hz, 1H), 6.92 (d, *J* = 5.1 Hz, 1H), 4.78 (ddd, J = 13.5, 11.5, 2.0

Hz, 1H), 4.57 (ddd, *J* = 12.4, 5.6, 2.2 Hz, 1H), 4.25 (d, *J* = 12.1 Hz, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 3.87 (td, *J* = 12.4, 3.7 Hz, 1H), 2.33 (s, 3H), 2.30 - 2.23 (m, 1H), 2.06 (d, J = 15 Hz, 1H), 1.04 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.90, 137.36, 136.95, 134.93, 133.66, 131.40, 129.65, 127.68, 123.88, 72.67, 68.65, 62.78, 44.16, 32.36, 15.09, 13.87.
HRMS (ESI) *m/z* calculated for $C_{18}H_{21}O_5S_2$ $[M+Na]^+$: 381.0830, found 381.0841.

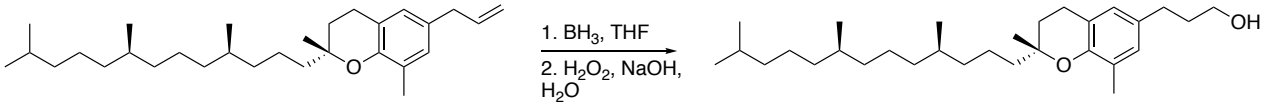
Diversification of Complex Molecules



(+)-(*R*)-6-allyl-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chromane [S39]



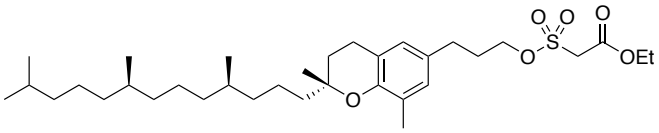
To an oven-dried round-bottom flask with a stir bar was added (+)-δ-tocopherol (3.2 g, 8.0 mmol, 1.0 equiv), CH_2Cl_2 (40 mL, 0.2 M), pyridine (1.94 mL, 12 mmol, 1.5 equiv). The reaction was cooled to 0 °C. Trifluoromethanesulfonic anhydride (1.62 mL, 9.6 mmol, 1.2 equiv) was added dropwise and the reaction was stirred at 0 °C for 30 min. After warming up to room temperature, the reaction was quenched with addition of cold water. The layers were separated, and the aqueous layer was extracted with CH_2Cl_2. The organic layers were combined and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography on silica using 100% hexanes as eluent. The characterization of the resulting compound matched the literature [8]. To an oven-dried round bottom flask equipped with condenser was added a stir bar, LiCl (477 mg, 11.25 mmol, 3.75 equiv), DMF (7.5 mL, 0.4 M), allyltributylstannane (1.16 mL, 3.75 mmol, 1.25 equiv), (+)-δ-tocopherol triflate (1.60 g, 3 mmol, 1 equiv) and $Pd(PPh_3)_4$ (104 mg, 0.09 mmol, 0.03 equiv). The reaction was heated to 100 °C and stirred for 12 hours. Upon completion, the reaction was cooled to room temperature, transferred into a separatory funnel with iced 10% NH_4OH solution (1:1 to DMF) and shake vigorously. Layers were separated, aqueous layer was extracted with ethyl acetate (30 mL x 3). The organic layers were combined and concentrated. The crude mixture was purified by flash column chromatography on silica using 100% petroleum ether as eluent to give (+)-allylated-δ-tocopherol derivative **[S39]** in 88% yield over two steps.
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.80 (d, *J* = 2.2 Hz, 1H), 6.73 (d, *J* = 2.1 Hz, 1H), 5.97 (ddt, *J* = 16.8, 10.0, 6.8 Hz, 1H), 5.13-4.95 (m, 2H), 3.28 (d, *J* = 6.8 Hz, 2H), 2.80-2.61 (m, 2H), 2.16 (s, 3H), 1.86-1.71 (m, 2H), 1.64-1.03 (m, 24H), 0.91-0.84 (m, 12H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.5, 138.5, 130.2, 128.6, 126.8, 126.3, 120.4, 115.2, 75.96, 40.3, 39.7, 39.5, 37.6, 37.6, 37.4, 33.0, 32.9, 31.4, 28.1, 25.0, 24.6, 24.4, 22.9, 22.8, 22.5, 21.1, 19.9, 19.8, 16.2. HRMS (ESI) *m/z* calculated for $C_{30}H_{50}O$ $[M]^+$: 426.3862, found 426.3851. $[\alpha]^{22}_D$ = +5.6 º (*c* = 1.05, $CHCl_3$)



(+)-3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)propan-1-ol [S40].

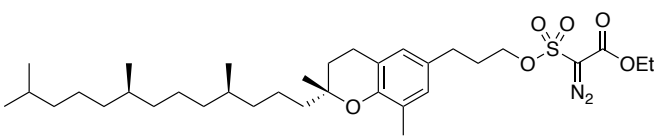
The following procedure was adapted from literature precedent [9]. A round-bottomed flask was flame-dried under vacuum, backfilled with nitrogen, and then charged with 1.28 g (3.0 mmol, 1 equiv) of (*R*)-6-allyl-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chromane [**S39**] and THF (8.6 mL, 0.35 M). The solution was cooled to 0°C in an ice bath. A 1.0 M BH_3 solution in THF (24 mL, 24 mmol, 8 equiv) was added dropwise. The reaction mixture was removed from the bath and allowed to stir at room temperature for one hour. The mixture was again cooled to 0°C and a 4 M solution of NaOH (2.70 mL) was added dropwise, followed by dropwise addition of 30% H_2O_2 (3.60 mL, 117 mmol, 39 equiv). The reaction was removed from the ice bath and allowed to stir for 40 minutes at room temperature. The mixture was then extracted three times with EtOAc. The combined organic phases were dried over magnesium sulfate and concentrated in vacuo. Flash column chromatography on silica gel with 5% → 13% EtOAc/hexanes as eluent yielded 644 mg (1.45 mmol) of pure product as a clear oil (48%).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.79 (d, *J* = 2.7 Hz, 1H), 6.73 (s, *J* = 2.2 Hz, 1H), 3.68 (t, J = 6.4 Hz, 2H), 2.77 – 2.67 (m, 2H), 2.57 (dd, J = 7.3, 7.9 Hz, 2H), 2.14 (s, 3H), 1.92 – 1.68 (m, 4H), 1.63 – 1.00 (m, 25H), 0.92 – 0.81 (m, 12H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 150.39, 131.89, 128.51, 126.67, 126.24, 120.40, 75.96, 62.72, 40.38, 39.53, 37.61, 37.57, 37.44, 34.70, 32.95, 32.85, 31.42, 31.40, 28.14, 24.96, 24.60, 24.41, 22.88, 22.79, 22.50, 21.15, 19.91, 19.82, 16.20.; $[\alpha]^{22}_D$=+3.80° (c=1.39, $CHCl_3$); HRMS (ESI) *m/z* calculated for $C_{30}H_{53}O_2$ $[M+H]^+$: 445.4046, found 445.4040.

(+)-Ethyl 2-((3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)propoxy)sulfonyl)acetate [S41].



Prepared according to the general procedure for preparation of sulfonate ester substrates. 644 mg (1.45 mmol) of 3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)propan-1-ol **[S40]** were used, along with imidazole (119 mg, 1.74 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (406 mg, 2.18 mmol, 1.5 equiv), and THF (7.2 mL, 0.2 M). Flash column chromatography on silica using 5% → 20% Et_2O/petroleum ether as eluent gave 427 mg (0.719 mmol) of pure product as a clear oil (50% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.80 (d, *J* = 2.2 Hz, 1H), 6.74 (d, *J* = 1.8 Hz, 1H), 4.38 (t, *J* = 6.4 Hz, 2H), 4.32 (q, *J* = 7.2 Hz, 2H), 4.11 (s, 2H), 2.79-2.69 (m, 2H), 2.64 (t, *J* = 7.0 Hz, 2H), 2.17 (s, 3H), 2.12 - 2.04 (m, 2H), 1.89 - 1.72 (m, 2H), 1.64 - 1.05 (m, 27H), 0.94 - 0.84 (m, 12H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.14, 150.67, 130.33, 128.49, 126.80, 126.42, 120.55, 76.05, 71.88, 62.86, 54.99, 40.40, 39.52, 37.60, 37.57, 37.43, 32.94, 32.85, 31.35, 31.18, 30.73, 28.13, 24.95, 24.60, 24.38, 22.88, 22.78, 22.46, 21.15, 19.90, 19.80, 16.18, 14.12.; $[\alpha]^{22}_D$=+3.21° (c=0.44, $CHCl_3$); HRMS (ESI) *m/z* calculated for $C_{34}H_{59}O_6S$ $[M+H]^+$: 595.4032, found 595.4036.

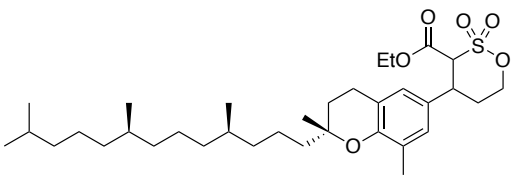
(+)-Ethyl 2-diazo-2-((3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)propoxy)sulfonyl)acetate [25].



Prepared according to the general procedure for diazo transfer. 427 mg (0.719 mmol) of ethyl 2-((3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-

4,8,12-trimethyltridecyl)chroman-6-yl)propoxy)sulfonyl)acetate **[S40]** were used, along with $MeSO_2N_3$ (261 mg, 2.16 mmol, 3 equiv), DBU (161 μL, 1.08 mmol, 1.5 equiv), and THF (1.8 mL, 0.4 M). Flash column chromatography on silica using 1% → 8% Et_2O/petroleum ether as eluent gave 280 mg (0.452 mmol) of pure product as a yellow oil (63% yield).
^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.75 (d, *J* = 2.9 Hz, 1H), 6.69 (d, *J* = 3.0 Hz, 1H), 4.35 (t, J = 6.3 Hz, 2H), 4.33 (q, J = 7.3 Hz, 2H), 2.76 - 2.65 (m, 2H), 2.60 (t, *J* = 7.5 Hz, 2H), 2.13 (s, 3H), 2.07 – 1.98 (m, 2H), 1.85 - 1.67 (m, 2H), 1.60 - 1.00 (m, 27H), 0.91 - 0.81 (m, 12H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.54, 150.60, 130.22, 128.42, 126.73, 126.31, 120.46, 75.95, 72.85, 62.74, 40.31, 39.45, 37.52, 37.49, 37.36, 32.86, 32.76, 31.29, 30.85, 30.64, 28.05, 24.88, 24.52, 24.31, 22.81, 22.72, 22.39, 21.06, 19.84, 19.75, 16.12, 14.35.; $[\alpha]^{22}_D$=+2.58° (c=1.99, $CHCl_3$); HRMS (ESI) *m/z* calculated for $C_{34}H_{56}O_6N_2SNa$ $[M+Na]^+$: 643.3757, found 643.3767; IR (ATR, cm^{-1}) 2925.67, 2867.31, 2130.74, 1723.96, 1598.56, 1480.78, 1464.51, 1376.38, 1288.42, 1222.30, 1180.52. 1151.78, 1092.88, 1006.25, 928.65, 835.62, 738.50, 615.85, 558.77, 538.19, 509.25.

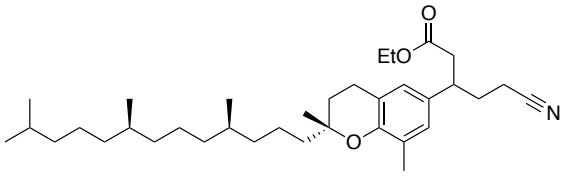
Ethyl 4-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [26].



Ethyl 2-diazo-2-((3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12 trimethyltridecyl)chroman-6-yl)propoxy)sulfonyl)acetate **[25]** (24.2 mg, 0.200 mmol, 1.0 equiv), [FePc]Cl (12.1 mg, 0.020 mmol, 0.10 equiv), $NaBAr^F_4$ (17.7 mg, 0.020 mmol, 0.10 equiv), and CH_2Cl_2 (4.0 mL, 0.05 M) were used. Product was purified via flash column chromatography on silica using 2% → 15% EtOAc/hexanes. Pure product was isolated as a yellow oil and a 1.4:1 syn:anti mixture of diastereomers. Subsequent purification using 5% → 20% Et_2O/hexanes allowed for separation of diastereomers. A trace impurity (<5%) was isolated with the syn product and was factored into yield calculations.
Run 1: (96.9 mg, 0.163 mmol, 82%), 0% rsm. Run 2: (85.2 mg, 0.144 mmol, 72%), 0% rsm. Run 3: (94.0 mg, 0.159 mmol, 79%), 0% rsm. **Average: 78% yield ± 4.2%, 0% rsm.**
Syn diastereomer (major): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.79 (d, *J* = 2.3 Hz, 1H), 6.74 (d, *J* = 2.4 Hz, 1H), 4.77 - 4.66 (m, 2H), 4.19 (d, *J* = 4.6 Hz, 1H), 4.14 - 4.00 (m, 2H), 3.71 (dt, *J* = 13.5, 4.1 Hz, 1H), 3.11 (dtd, J = 6.5 Hz, 11.6 Hz, 14.0 Hz, 1H), 2.80 - 2.60 (m, 2H), 2.12 (s, 3H), 1.86 - 1.68 (m, 4H), 1.55 - 0.97 (m, 26H), 0.91 - 0.77 (m, 12H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.32, 152.06, 128.00, 127.24, 127.19, 127.00, 125.73, 125.68, 120.96, 120.94, 76.44, 73.31, 66.67, 62.16, 42.57, 40.30, 40.23, 39.51, 37.60, 37.43, 32.94, 32.87, 32.83, 31.21, 28.13, 24.95, 24.59, 24.56, 24.32, 24.26, 22.88, 22.49, 21.12, 19.90, 19.78, 16.27, 13.91.
Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.78 (d, *J* = 2.3 Hz, 1H), 6.72 (d, *J* = 2.3 Hz, 1H), 4.72 (ddd, *J* = 13.4, 11.7, 2.2 Hz, 1H), 4.52 (ddd, *J* = 11.6, 4.9, 1.5 Hz, 1H), 4.15 (d, *J* = 12.0 Hz, 1H), 4.12 – 4.04 (m, 2H), 3.67 (td, *J* = 12.4, 3.6 Hz, 1H), 2.76 – 2.65 (m, 2H), 2.20 (dtd, *J* = 14.8, 12.9, 4.9 Hz, 1H), 2.12 (s, 3H), 2.01 - 1.94 (m, 1H), 1.82 – 1.70 (m, 2H), 1.59 - 0.99 (m, 27H), 0.87 – 0.83 (m, 12H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.10, 151.96, 128.59, 127.25, 127.20, 126.90, 125.99, 125.93, 120.91, 120.90, 76.42, 72.72, 69.11, 62.47,

43.79, 40.37, 40.30, 39.51, 37.60, 37.58, 37.42, 32.94, 32.85, 32.65, 31.17, 28.13, 24.94, 24.59, 24.32, 24.26, 22.87, 22.78, 22.44, 21.11, 19.89, 19.78, 16.26, 13.87.
HRMS (ESI) *m/z* calculated for $C_{34}H_{57}O_6S$ $[M+H]^+$: 593.3876, found 593.3865.

Ethyl 5-cyano-3-((R)-2,8-dimethyl-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman-6-yl)pentanoate [27].



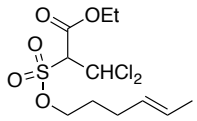
The following procedure was adapted from literature precedent[3]. Other examples of sultone eleaboration have been reported[10-13]. A round-bottomed flask was charged with ethyl 4-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide (93.9 mg, 0.159 mmol) and DMF (0.45 mL, 0.33 M). Sodium cyanide (8.60 mg, 0.174 mmol, 1.1 equiv) was added and the resulting mixture was heated to 100°C and stirred for three hours. The reaction mixture was then allowed to cool to room temperature and the DMF was removed in vacuo. The resulting brown oil was coevaporated three times with toluene and then allowed to stand under high vacuum for three hours at room temperature. The resulting brown oil was dissolved in toluene (0.63 mL, 0.25 M) and DMF (3.2 µL) and then oxalyl chloride (100.6 mg, 68 µL, 0.793 mmol, 5 equiv) were added. The resulting mixture was heated to 100°C and stirred for twenty minutes, and then allowed to cool to room temperature. The resulting suspension was filtered through a small pad of celite and the filter cake washed three times with toluene. The filtrate was concentrated in vacuo and the resulting brown oil was redissolved in THF (0.8 mL, 0.2 M). A separate round-bottomed flask was charged with zinc dust (104 mg, 1.59 mmol, 10 equiv), copper (II) acetate monohydrate (31.6 mg, 0.159 mmol, 1 equiv), and glacial acetic acid (0.8 mL). This mixture was stirred for one minute before the substrate solution in THF was added dropwise. The resulting reaction mixture was stirred at room temperature for 2.5 hours. The mixture was diluted with diethyl ether and filtered through a small pad of celite. The filter cake was washed three times with ether. The ether was removed in vacuo. Flash column chromatography on silica gel with 2% → 20% Et_2O/petroleum ether as eluent gave some pure product (19.5 mg, 0.037 mmol, 23%) as a clear oil and some product as a clear oil with minor impurities. The yield was determined via NMR with mesitylene as an internal standard.
Overall: 0.070 mmol, 44% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75 (s, 1H), 6.71 (s, 1H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.10 – 3.04 (m, 1H), 2.79 – 2.68 (m, 2H), 2.60 (dd, *J* = 7.5, 2.1 Hz, 2H), 2.33 - 2.03 (m, 7H), 1.90 – 1.74 (m, 3H), 1.65 - 1.03 (m, 26H), 0.91 – 0.87 (m, 12H). ^{13}C NMR (126 MHz, CDCl3) δ 171.93, 151.37, 131.18, 127.15, 127.10, 126.70, 126.69, 125.95, 125.90, 120.78, 119.74, 76.22, 60.59, 41.85, 40.64, 40.48, 39.52, 37.62, 37.58, 37.44, 32.95, 31.74, 31.25, 28.13, 24.95, 24.60, 24.37, 24.33, 22.87, 22.78, 22.50, 21.15, 19.90, 19.80, 16.31, 15.52, 14.30.; HRMS (ESI) *m/z* calculated for $C_{35}H_{58}NO_3$ $[M+H]^+$: 540.4417, found 540.4416.

Free Carbene Generation Study

Procedure for UV-light mediated diazo decomposition:
To a flame-dried quartz tube equipped with a rubber septum and stir bar was added **6** (55.3 mg, 0.20 mmol, 1 equiv) in CH_2Cl_2 (4.0 mL, 0.05M) under an inert atmosphere. Argon was bubbled

through the mixture for 10 minutes. The gas inlet was removed, the septum was wrapped in aluminum foil, and the reaction, while stirring, was irradiated with four 15W compact UV germicidal bulbs at room temperature. After 1 hour, the tube was removed and TLC analysis revealed complete consumption of starting material. The reaction mixture was then transferred to a 25 mL flask, concentrated and analyzed by crude NMR, which revealed the mixture was composed of predominantly one compound **[28]**. The product was purified via flash column chromatography on silica (40 mL SiO_2) using 5% → 10% → 15% EtOAc/hexanes. Pure product was isolated as a colorless oil. No other products were isolated off the column.

Ethyl (*E*)-3,3-dichloro-2-((hex-4-en-1-yloxy)sulfonyl)propanoate [28]



Run 1: (24.8 mg, 0.074 mmol, 37%), 0% rsm. Run 2: (25.7 mg, 0.078 mmol, 39%), 0% rsm. Run 3: (22.0 mg, 0.066 mmol, 33%), 0% rsm. **Average: 36% yield ± 2.5, 0% rsm.**

^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.49 (dt, *J* = 15.1, 6.3 Hz, 1H), 5.37 (ddd, *J* = 15.0, 6.6, 1.3 Hz, 1H), 4.59 (d, *J* = 11.9 Hz, 1H), 4.53 (td, *J* = 6.5, 1.2 Hz, 2H), 4.41 (m, 2H), 4.04 (d, *J* = 11.8 Hz, 1H), 2.11 (tt, *J* = 6.9, 6.0 Hz, 2H), 1.84 (dt, *J* = 13.4, 6.9 Hz, 2H), 1.66 (dq, *J* = 6.3, 1.4 Hz, 3H), 1.38 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 161.55, 128.91, 127.24, 85.20, 77.02, 65.18, 47.05, 29.50, 28.29, 18.16, 14.07.; HRMS (ESI) *m/z* calculated for $C_{11}H_{18}O_5SCl_2Na$ $[M+Na]^+$: 355.0150, found 355.0146.

Procedure for iron salt mediated diazo decomposition study:

Into a 1 dram vial was added iron salt (0.040 mmol, 0.10 equiv) in a glovebox. The vial was sealed, taken out of the box, and the contents were added under a stream of nitrogen to a flame dried three neck 25 mL flask equipped with a stir bar, reflux condenser, glass stopper, and rubber septum. CH_2Cl_2 (5.0 mL, 0.08 M) was added and the flask was heated to reflux (~45˚C) for 10 minutes. Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **6** (110.5 mg, 0.400 mmol, 1.0 equiv) was taken up with CH_2Cl_2 (3.0 mL, 0.13 M) in a glass syringe and added dropwise to the refluxing catalyst solution over the course of 2-3 minutes (final reaction concentration is 0.05 M). After complete addition of diazosulfonate, the reaction refluxed for 2 hours. The reaction mixture was cooled, adsorbed onto SiO_2 or florisil, and applied directly to a silica column (75 mL SiO_2) for purification to afford the desired sultone product. The recovered starting material (rsm) was eluted with 10% EtOAc/hexanes. No other compounds were isolated off the column.

$FeCl_3$ Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (6.5 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.

Run 1: (99.5 mg rsm, 0.360 mmol, 90%). Run 2: (103.5 mg rsm, 0.375 mmol, 94%). **Average: 0% yield, 92% rsm.**

$FeCl_2$ Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $FeCl_2$ (5.1 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.

Run 1: (109.3 mg rsm, 0.396 mmol, 99%). Run 2: (106.3 mg rsm, 0.385 mmol, 96%). **Average: 0% yield, 98% rsm.**

$FeCl_3$/$NaBAr^F_4$ Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $FeCl_3$ (6.5 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (108.8 mg rsm, 0.394 mmol, 98%). Run 2: (108.2 mg rsm, 0.391 mmol, 98%). **Average: 0% yield, 98% rsm.**

$FeCl_2$/$NaBAr^F_4$ Conditions: Ethyl (*E*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[6]** (110.5 mg, 0.400 mmol, 1.0 equiv), $FeCl_2$ (5.1 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used.
Run 1: (108.9 mg rsm, 0.394 mmol, 98%). Run 2: (110.5 mg rsm, 0.400 mmol, 100%). **Average: 0% yield, 99% rsm.**

Intramolecular Kinetic Isotope Effect Study

Method for KIE Determination: The column-purified product mixture **31** (*vide infra*, ca. 20 mg in 600 µL $CDCl_3$) was analyzed by ^{13}C-NMR (600 MHz instrument) [14-15]. $Cr(acac)_3$ (3.5-5.0 mg) was added directly to the solution in the NMR tube immediately prior to running the NMR study; this helps to significantly reduce delay times needed to obtain accurate integrations. The experiment was run under inverse-gated decoupling conditions without sample spinning. The following parameters were used for the experiment, listed as Varian commands:

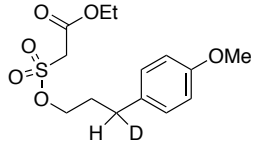
temp=23
dm='nny' (inverse-gated decoupling)
d1=5 (initial delay)
at=0.5 (acquisition time)
setsw(180,0) (spectral width, in ppm)
bs=64 (block size for FID)
nt=2944 (number of scans)
pw=7.0 (pulse width)
pw90=7.0 (90° pulse width)

The KIE was reported as the area of the deuterated peak over that of the non-deuterated peak for the benzylic carbon (~42 ppm). All diastereomeric peaks resolve in the ^{13}C NMR and the sample can be analyzed accurately as a mixture. However, the KIE was calculated off of the major (*syn*) diastereomer of the product due to better signal to noise ratios. Three NMR experiments were run for each catalyst and an average value was calculated with error reported as a standard deviation.

[FePc]Cl: $C—H/C—D = 5.0 \pm 0.1$
[$FeCl_8$Pc]Cl: $C—H/C—D = 4.8 \pm 0.1$
[$FeCl_{16}$Pc]Cl: $C—H/C—D = 4.5 \pm 0.1$

$Rh_2(OAc)_4$: *C—H/C—D* = 1.8 ± 0.1

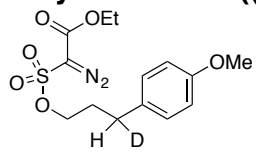
Ethyl 2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate [S41].



1.30 g (7.6 mmol) of 3-(4-methoxyphenyl)propan-3-*d*-1-ol [16] were used, along with imidazole (620 mg, 9.1 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (2.10 g, 11.3 mmol, 1.5 equiv), and THF (38 mL, 0.2 M). Flash column chromatography on silica using 20% EtOAc/hexanes as eluent gave 1.95 g (6.10 mmol) of pure product as a pale yellow oil (80% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.33 (t, *J* = 6.3 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 3.79 (s, 3H), 2.68 (t, *J* = 7.7 Hz, 1H), 2.05 (q, *J* = 6.7 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 161.96, 157.95, 132.17, 129.28, 113.85, 71.40, 62.56, 55.10, 54.54, 30.77, 30.03 (t, *J* = 19.8 Hz), 13.85.; HRMS (ESI) *m/z* calculated for $C_{14}H_{19}D_1O_6SNa$ $[M+Na]^+$: 340.0941, found 340.0933.

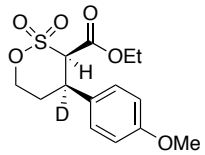
Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate [29].



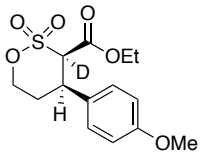
1.95 g (6.10 mmol) of ethyl 2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[S41]** were used, along with $MeSO_2N_3$ (1.63 g, 13.5 mmol, 2.2 equiv), DBU (1.4 mL, 9.20 mmol, 1.5 equiv), and THF (15 mL, 0.4 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 1.74 g (5.07 mmol) of pure product as a yellow oil (83% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.34 (t, *J* = 6.4 Hz, 2H), 4.33 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 2.67 (t, *J* = 7.6 Hz, 1H), 2.04 (q, *J* = 6.5 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.53, 158.17, 132.20, 129.45, 114.05, 72.60, 62.80, 55.34, 30.70, 30.24 (t, *J* = 19.7 Hz), 14.37.; HRMS (ESI) *m/z* calculated for $C_{14}H_{18}DO_6N_2S$ $[M+H]^+$: 344.1027, found 344.1020; IR (ATR, cm^{-1}) 2970, 2918, 2130, 1722, 1447, 1373, 1285, 1224, 1178, 1083, 1007, 924, 879, 843, 777, 739, 615, 558, 495.

Ethyl 4-(4-methoxyphenyl)-1,2-oxathiane-3-carboxylate-4-*d* 2,2-dioxide [31]:



major isomer



minor isomer

Product was isolated and characterized as a mixture of the two isomers as well as a minor amount of the non-deuterated product (formed from H-exchange at the acidified C3 site).

Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.16 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 4.78-4.67 (m, 2H), 4.21 (s, 1H), 4.13-3.98 (m, 2H), 3.80 (s, 3H), 3.18 (ddd, *J* = 18.5, 13.9, 7.4 Hz, 1H), 1.79 (dt, *J* = 14.4, 1.9 Hz, 1H), 1.07 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) 164.27, 159.57, 130.06, 130.04, 128.56, 114.59, 73.35, 73.33, 66.65 (non-deut), 66.58, 66.30 (t, J = 22.0 Hz), 62.44, 62.42, 55.56, 42.41 (non-deut), 42.31, 42.02 (t, J = 20.2 Hz), 24.47, 24.41, 14.00.; HRMS (ESI) *m/z* calculated for $C_{14}H_{18}DO_6S$ $[M+H]^+$: 316.0965, found 316.0968.

[FePc] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[29]** (137.3 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol,

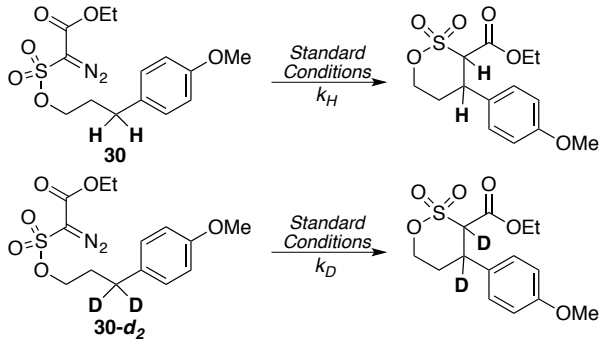
0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Flash column chromatography on silica (75 mL) using 10% → 15% → 20% → 25% EtOAc/hexanes gave the *syn* and *anti* diastereomers mostly separated. All of the fractions containing the *syn* product were collected, concentrated, and analyzed by ^{13}C NMR. This procedure was repeated three times.
Run 1: 4.94. Run 2: 5.05. Run 3: 5.06. **Average: 5.0 ± 0.1**

[$FeCl_8Pc$] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[29]** (206.0 mg, 0.600 mmol, 1.0 equiv), [$FeCl_8Pc$]Cl (52.8 mg, 0.060 mmol, 0.10 equiv), $NaBAr^F_4$ (53.2 mg, 0.060 mmol, 0.10 equiv), and CH_2Cl_2 (12.0 mL, 0.05 M) were used. The reaction was allowed to stir for 16 hours. Flash column chromatography on silica (75 mL) using 10% → 15% → 20% → 25% EtOAc/hexanes gave the *syn* and *anti* diastereomers mostly separated. All of the fractions containing the *syn* product were collected, concentrated, and analyzed by ^{13}C NMR. This procedure was repeated three times.
Run 1: 4.94. Run 2: 4.79. Run 3: 4.70. **Average: 4.8 ± 0.1**

[$FeCl_{16}Pc$] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[29]** (206.0 mg, 0.600 mmol, 1.0 equiv), [$FeCl_{16}Pc$]Cl (69.3 mg, 0.060 mmol, 0.10 equiv), $NaBAr^F_4$ (53.2 mg, 0.060 mmol, 0.10 equiv), and CH_2Cl_2 (12.0 mL, 0.05 M) were used. The reaction was allowed to stir for 16 hours. Flash column chromatography on silica (75 mL) using 10% → 15% → 20% → 25% EtOAc/hexanes gave the *syn* and *anti* diastereomers mostly separated. All of the fractions containing the *syn* product were collected, concentrated, and analyzed by ^{13}C NMR.
Run 1: 4.55. Run 2: 4.57. Run 3: 4.45. **Average: 4.5 ± 0.1**

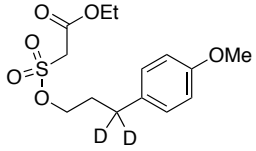
$Rh_2(OAc)_4$ Conditions:
The general procedure for $Rh_2(OAc)_4$ catalyzed C—H alkylation was followed. Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3-*d*)sulfonyl)acetate **[29]** (137.3 mg, 0.400 mmol, 1.0 equiv), $Rh_2(OAc)_4$ (3.5 mg, 0.008 mmol, 0.02 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. Flash column chromatography on silica (75 mL) using 10% → 15% → 20% → 25% EtOAc/hexanes gave the *syn* and *anti* diastereomers mostly separated. All of fractions containing the *syn* product were collected, concentrated, and analyzed by ^{13}C NMR. This procedure was repeated three times.
Run 1: 1.78. Run 2: 1.84. Run 3: 1.91. **Average: 1.8 ± 0.1**

Determination of Kinetic Isotope Effect via Initial Rates



General Procedure for Initial Rate Analysis: To a 1 dram flame-dried borosilicate vial containing a Teflon stir bar was added catalyst (0.010 mmol, 0.10 equiv) and $NaBAr^F_4$ (8.9 mg, 0.010 mmol, 0.10 equiv) in the glove box. The vial was removed and the contents were diluted with 1 mL of CH_2Cl_2, sealed with a Teflon septum cap, and heated to 40˚C for 10 minutes. **30** or **30-*d*$_2$** (34.2 mg, 0.2 mmol, 1.0 equiv) and internal standard nitrobenzene (0.04 mmol, 40 mol%), were dissolved in 1 mL of CH_2Cl_2 (400 μL, 0.5M) and added to the 1 dram vial in one portion. The septum cap was wrapped with Teflon tape. Aliquots (25 µL) were taken at the corresponding times from the reaction flask, and filtered through a silica pad with 500 µL of 1:1 isopropanol/hexanes for normal phase HPLC (Zorbax CN, 4.6 x 250 nm) analysis. The yield was determined by integration of the product peaks relative to the nitrobenzene internal standard and comparison to a standard curve. Initial rates were determined for formation of **17** by plotting the yield versus time. Rates are reported as the average of three runs, with the error denoted by standard deviation. Error for kinetic isotopes was calculated via propagation of the standard error of the mean for each set of rates.

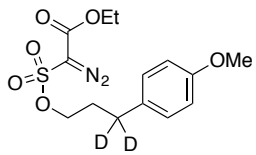
Ethyl 2-((3-(4-methoxyphenyl)propoxy-3,3-*d*$_2$)sulfonyl)acetate [S42].



1.71 g (10.2 mmol) of 3-(4-methoxyphenyl)propan-3,3-d_2-1-ol were used, along with imidazole (837.3 mg, 12.3 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (2.90 g, 15.3 mmol, 1.5 equiv), and THF (51 mL, 0.2 M). Flash column chromatography on silica using 10% → 20% EtOAc/hexanes as eluent gave 2.75 g (8.64 mmol) of pure product as a clear oil (85% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.11 (d, J = 8.6 Hz, 2H), 6.84 (d, J = 8.6 Hz, 2H), 4.33 (t, J = 6.3 Hz, 2H), 4.28 (q, J = 7.1 Hz, 2H), 4.07 (s, 2H), 3.79 (s, 3H), 2.04 (t, J = 6.3 Hz, 2H), 1.32 (t, J = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.13, 158.24, 132.31, 129.51, 114.13, 71.58, 62.89, 55.42, 55.00, 30.97, 14.12.; HRMS (ESI) *m/z* calculated for $C_{14}H_{19}D_2O_6S$ $[M+H]^+$: 319.1184, found 319.1188.

Ethyl 2-diazo-2-((3-(4-methoxyphenyl)propoxy-3,3-*d*$_2$)sulfonyl)acetate [30-*d*$_2$].



1.30 g (4.0 mmol) of ethyl 2-((3-(4-methoxyphenyl)propoxy-3,3-d_2)sulfonyl)acetate **[S42]** were used, along with $MeSO_2N_3$ (1.06 mg, 8.80 mmol, 2.2 equiv), DBU (895 μL, 6.0 mmol, 1.5 equiv), and THF (10 mL, 0.4 M). Flash column chromatography on silica using 10% → 20% EtOAc/hexanes as eluent gave 1.21 g (3.51 mmol) of pure product as a yellow oil (88% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.36-4.31 (m, 4H), 3.79 (s, 3H), 2.03 (t, *J* = 6.5 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.61, 158.26, 132.23, 129.50, 114.13, 72.65, 62.86, 55.42, 30.70, 14.43.; IR (ATR, cm^{-1}) 3424.56, 2984.44, 2959.73, 2937.88, 2837.83, 2543.3, 2132.08, 1887.91, 1717.93, 1611.77, 1581.55, 1513.07, 1465.1, 1444.66, 1372.35, 1286.84, 1244.11, 1224.78, 1176.61, 1089.82, 1032.45, 1008.39, 993.33, 960.88, 919.92, 826.41, 807.79, 786.49, 738.48, 699.17, 684.54, 612.94, 564.24, 552.45, 509.78, 468.23.; HRMS (ESI) *m/z* calculated for $C_{14}H_{17}D_2O_6N_2S$ $[M+H]^+$: 345.1089, found 345.1084.

[FePc]Cl: k_H = *0.86 % yield/min;* k_D = *0.29 % yield/min;* k_H/k_D = 3.0 ± 0.2
[FeCl$_{16}$Pc]Cl: k_H = *0.036 % yield/min;* k_D = *0.025 % yield/min;* k_H/k_D = 1.4 ± 0.1

Figure S1:

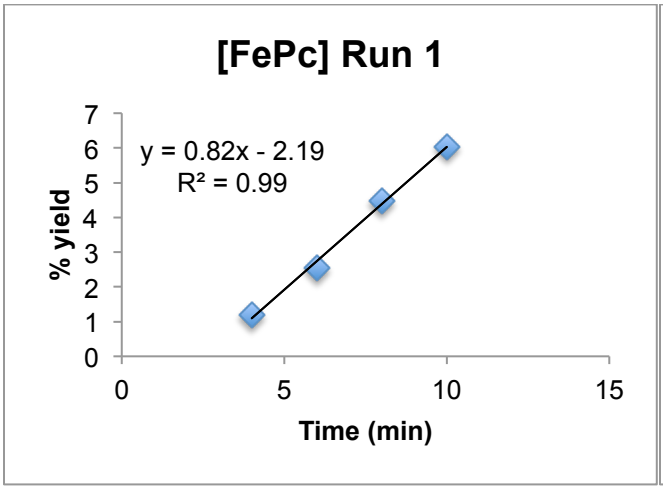


Figure S2:

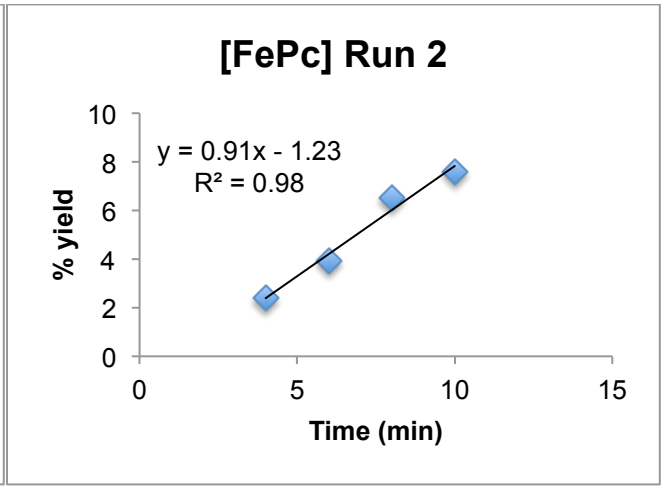


Figure S3:

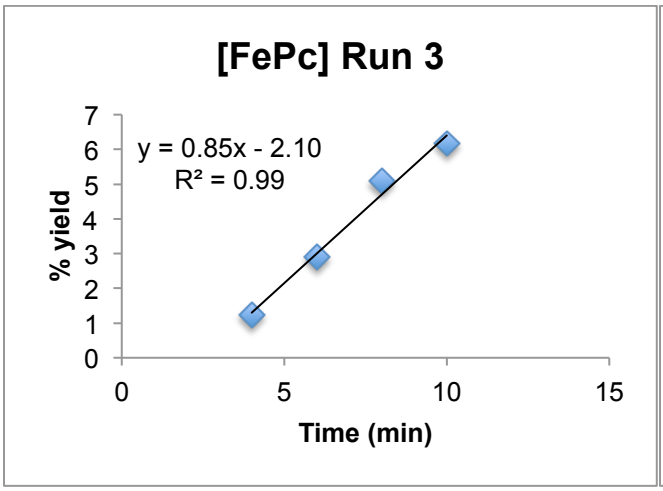


Figure S4:

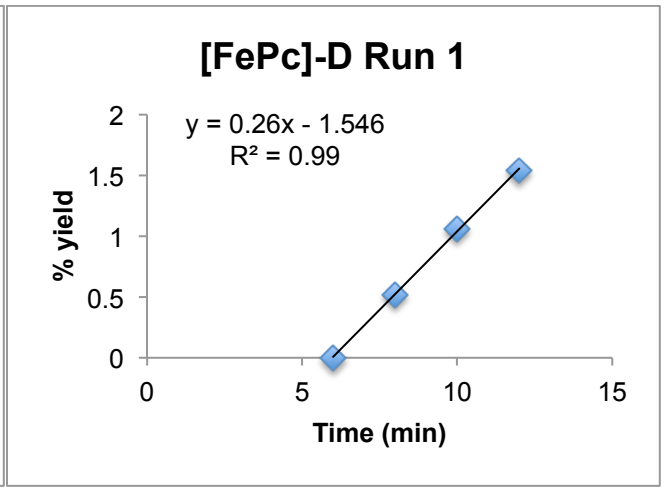


Figure S5:

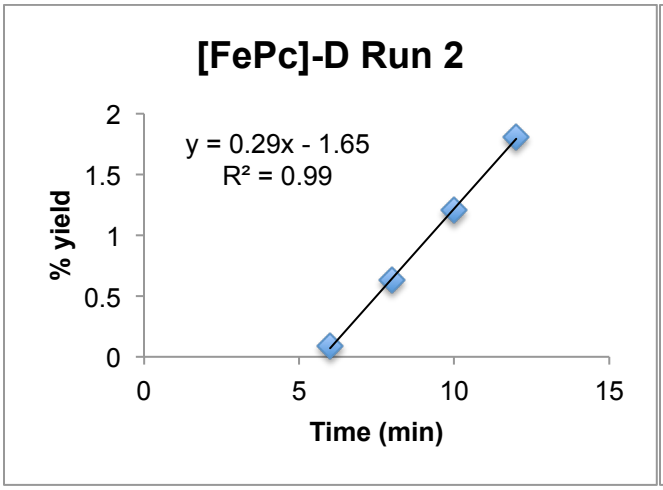


Figure S6:

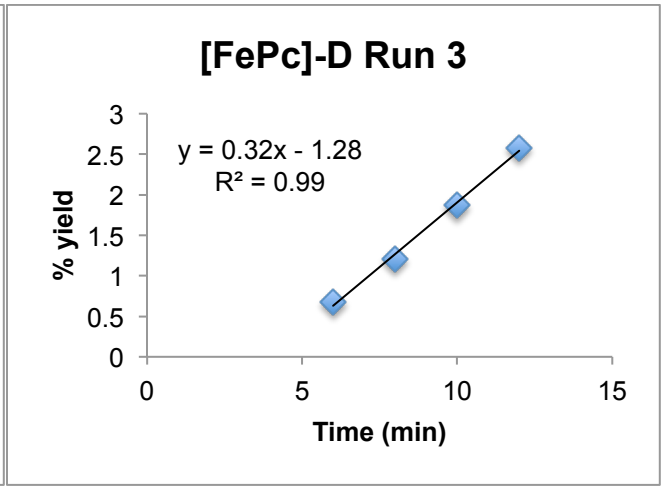


Figure S7:

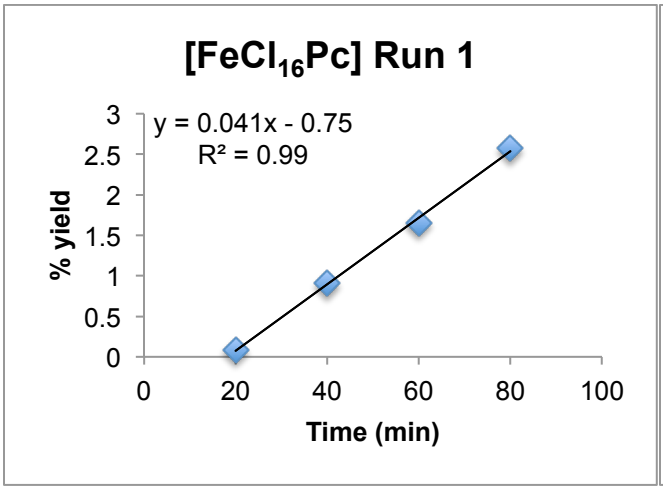


Figure S8:

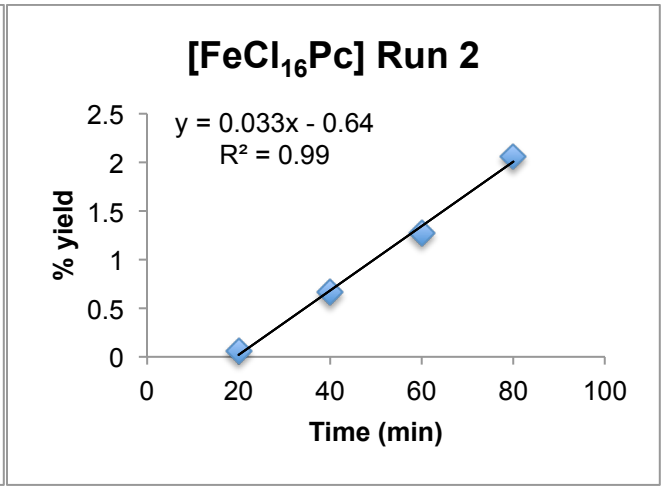


Figure S9:

Figure S10:

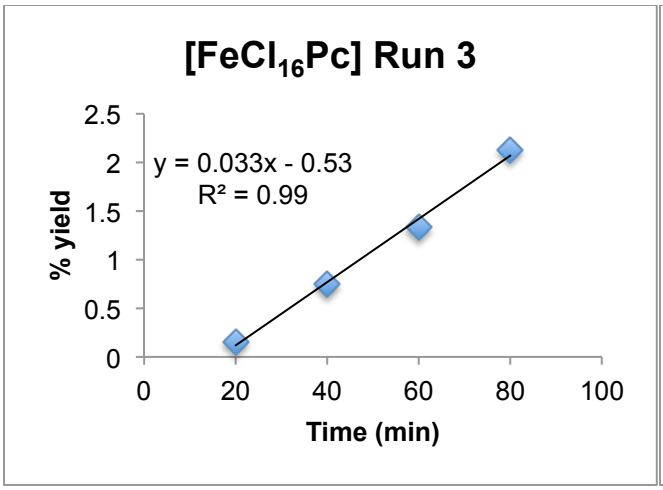


Figure S11:

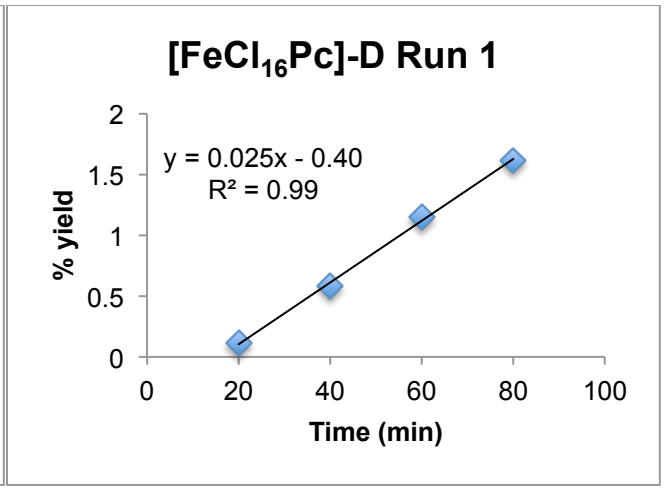
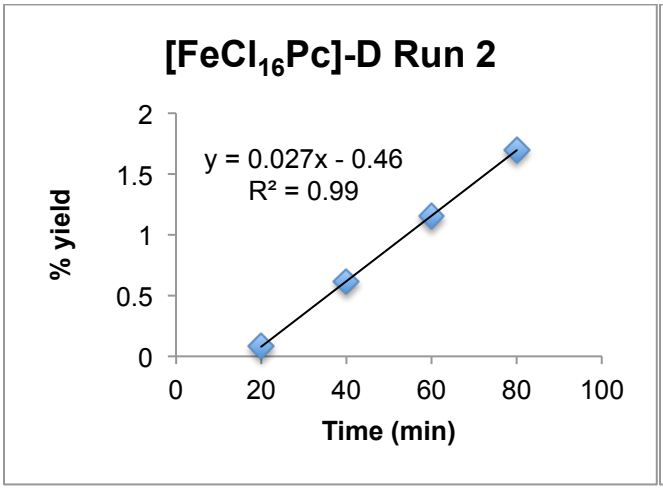
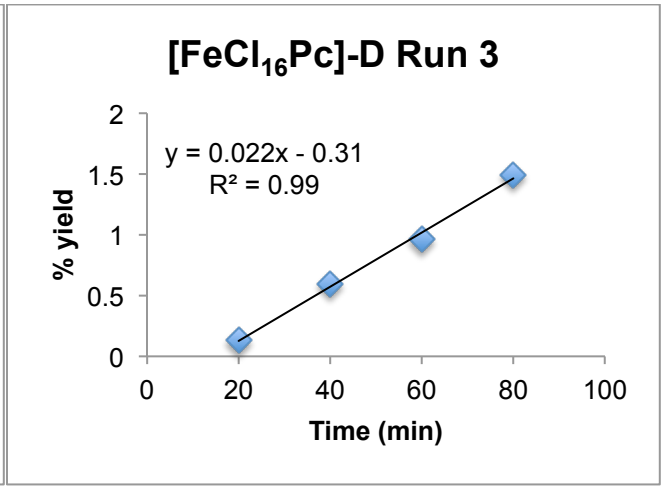


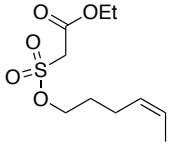
Figure S12:





Olefin Isomerization Study

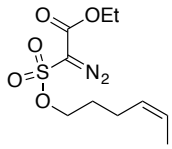
Ethyl (*Z*)-2-((hex-4-en-1-yloxy)sulfonyl)acetate [S43].



0.50 g (5.0 mmol) of (*Z*)-hex-4-en-1-ol were used, along with imidazole (409 mg, 6.0 mmol, 1.2 equiv), $ClSO_2CH_2CO_2Et$ (1.40 g, 7.5 mmol, 1.5 equiv), and THF (25 mL, 0.2 M). Flash column chromatography on silica using 10% EtOAc/hexanes as eluent gave 1.04 g (4.20 mmol) of pure product as a pale yellow oil (84% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.57-5.49 (m, 1H), 5.35 (dtq, *J* = 11.0, 7.4, 1.8 Hz, 1H), 4.34 (t, *J* = 6.5 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 2.18 (q, *J* = 7.3 Hz, 2H), 1.89-1.79 (p, *J* = 7.4 Hz, 2H), 1.62 (d, *J* = 6.9 Hz, 3H), 1.33 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 162.03, 128.17, 125.67, 71.80, 62.64, 54.75, 28.94, 22.56, 13.93, 12.74.; HRMS (ESI) *m/z* calculated for $C_{10}H_{18}O_5SNa$ $[M+Na]^+$: 273.0773, found 273.0771.

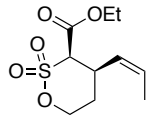
Ethyl (*Z*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate [32].



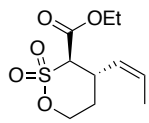
1.04 g (4.20 mmol) of ethyl (*Z*)-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[S43]** were used, along with $MeSO_2N_3$ (1.12 g, 9.20 mmol, 2.2 equiv), DBU (942 μL, 6.30 mmol, 1.5 equiv), and THF (11 mL, 0.4 M). Flash column chromatography on silica using 5% EtOAc/hexanes as eluent gave 957 mg (3.46 mmol) of pure product as a yellow oil (83% yield).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.58-5.49 (m, 1H), 5.34 (dtq, *J* = 9.0, 7.3, 3.6, Hz, 1H), 4.36 (t, *J* = 6.6 Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 2.17 (q, *J* = 7.4 Hz, 2H), 1.82 (p, *J* = 6.6 Hz, 2H), 1.61 (dd, *J* = 6.9, 0.9 Hz, 3H), 1.33 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 159.57, 128.16, 125.88, 73.05, 62.78, 28.82, 22.70, 14.37, 12.86.; HRMS (ESI) *m/z* calculated for $C_{10}H_{16}O_5N_2SNa$ $[M+Na]^+$: 299.0678, found 299.0675; IR (ATR, cm^{-1}) 2970, 2941, 2130, 1721, 1465, 1446, 1372, 1285, 1224, 1177, 1083, 1006, 991, 928, 836, 740, 704, 614.

Ethyl (*Z*)-4-(prop-1-en-1-yl)-1,2-oxathiane-3-carboxylate 2,2-dioxide [(*Z*)-7].



Syn diastereomer (major): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.69-5.63 (m, 1H), 5.25 (t, *J* = 9.9 Hz, 1H), 4.71-4.64 (m, 1H), 4.63-4.57 (m, 1H), 4.34-4.20 (m, 2H), 4.00 (d, *J* = 4.8 Hz, 1H), 3.57 (ddt, *J* = 13.4, 9.2, 4.5 Hz, 1H), 2.66-2.53 (m, 1H), 1.71 (d, *J* = 7.1 Hz, 3H), 1.63-1.54 (m, 1H), 1.31 (t, *J* = 7.2 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 164.16, 128.94, 126.84, 73.15, 64.11, 62.50, 35.43, 25.87, 14.09, 13.32.; HRMS (ESI) *m/z* calculated for $C_{10}H_{17}O_5S$ $[M+H]^+$: 249.0797, found 249.0797.



Anti diastereomer (minor): ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.64 (dq, *J* = 10.7, 6.9 Hz, 1H), 5.14-5.06 (m, 1H), 4.70 (td, *J* = 11.7, 3.5 Hz, 1H), 4.49 (ddd, *J* = 11.6, 4.3, 2.2 Hz, 1H), 4.25 (qd, *J* = 7.1, 2.1 Hz, 2H), 3.78 (d, *J* = 11.4 Hz, 1H), 3.64 (qd, *J* = 10.9, 4.7 Hz, 1H), 1.91-1.77 (m, 2H), 1.71 (dd, *J* = 6.9, 1.8 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H).; ^{13}C-NMR (126 MHz, $CDCl_3$) δ 163.42, 129.18, 127.25, 72.59, 68.00, 62.76, 36.74, 30.30, 14.10, 13.42.; HRMS (ESI) *m/z* calculated for $C_{10}H_{17}O_5S$ $[M+H]^+$: 249.0797, found 249.0797.

[FePc] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl (*Z*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[32]** (110.5 mg, 0.400 mmol, 1.0 equiv), [FePc]Cl (24.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8.0 mL, 0.05 M) were used. The reaction went to complete conversion. By GC analysis of the crude product, *Z/E* was **3.5:1** (this ratio was confirmed by subjecting column-purified *Z/E* mixtures of products to GC analysis). Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% EtOAc/hexanes. Pure product was isolated as a white solid and as a mixture of diastereomers. By GC analysis of column-purified product mixtures, *Z/E* was **3:1**.

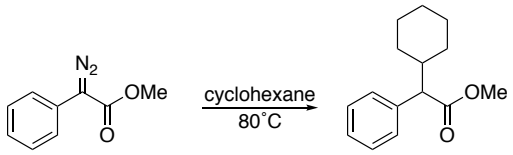
[$FeCl_8$Pc] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl (*Z*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[32]** (110.5 mg, 0.400 mmol, 1.0 equiv), [$FeCl_8$Pc]Cl (35.2 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F{}_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8 mL, 0.05 M) were used. After 16 h, the reaction did not go to

complete conversion; crude reaction samples containing unreacted diazosulfonate ester decomposed by GC analysis, producing unreliable data. Thus, the reaction was purified via flash column chromatography on silica (75 mL SiO_2) using 10→15% EtOAc/hexanes to remove unreacted starting material. Pure product was isolated as a white solid and as a mixture of diastereomers. By GC analysis of column-purified product mixtures, *Z/E* was **7:1**.

[$FeCl_{16}Pc$] Conditions: The general procedure for [FePc]-catalyzed C—H alkylation was followed. Ethyl (*Z*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[32]** (110.5 mg, 0.400 mmol, 1.0 equiv), [$FeCl_{16}Pc$]Cl (46.8 mg, 0.040 mmol, 0.10 equiv), $NaBAr^F_4$ (35.4 mg, 0.040 mmol, 0.10 equiv), and CH_2Cl_2 (8 mL, 0.05 M) were used. After 16 h, the reaction did not go to complete conversion; crude reaction samples containing unreacted diazosulfonate ester decomposed by GC analysis, producing unreliable data. Thus, the reaction was purified via flash column chromatography on silica (75 mL SiO_2) using 10→15% EtOAc/hexanes to remove unreacted starting material. Pure product was isolated as a white solid and as a mixture of diastereomers. By GC analysis of column-purified product mixtures, *Z/E* was **10:1**.

$Rh_2(OAc)_4$ Conditions:
The general procedure for $Rh_2(OAc)_4$ catalyzed C—H alkylation was followed. Ethyl (*Z*)-2-diazo-2-((hex-4-en-1-yloxy)sulfonyl)acetate **[32]** (110.5 mg, 0.400 mmol, 1.0 equiv), $Rh_2(OAc)_4$ (3.54 mg, 0.008 mmol, 0.02 equiv), and CH_2Cl_2 (8 mL, 0.05 M) were used. By GC analysis of the crude product, *Z/E* was **>20:1** (this ratio was confirmed by subjecting column-purified *Z/E* mixtures of products to GC analysis). Product was purified via flash column chromatography on silica (75 mL SiO_2) using 15% EtOAc/hexanes. Pure product was isolated as a white solid and as a mixture of diastereomers.

Thermal Decomposition of Diazo Esters



S44

The following procedure was adapted from a literature report[17] of an iron catalyzed C—H insertion reaction, wherein a 66% yield of C—H alkylation of cyclohexane was reported with the use of 2 mol% Fe(TPP)Cl at 80˚C. We conducted the reaction in the same fashion, but without the addition of catalyst.

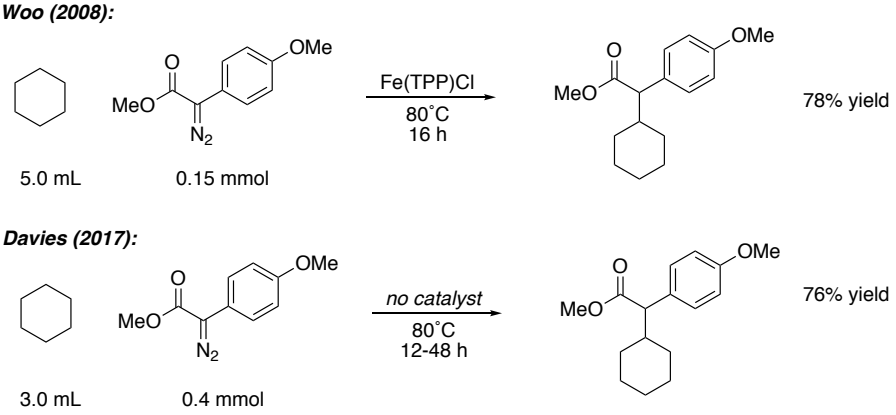
Procedure for metal-free C—H functionalization of cyclohexane:

Methyl 2-phenyldiazoacetate[18] (70.5 mg, 0.40 mmol) was added into a new, flame-dried flask equipped with a reflux condenser. Distilled and thoroughly degassed cyclohexane (11.7 mL) was then added and N_2 was bubbled through the solution for 10 minutes. The mixture stirred at 80˚C for 24 hours. The mixture was then cooled, concentrated, and purified via flash chromatography on silica using 10% EtOAc/hexanes. Due to co-elution with the diazoester starting material, the yield was determined via NMR with mesitylene as an internal standard. ^{1}H NMR spectrum matched literature values [19]. We also repeated this experiment with

glassware that had been washed with EDTA (to remove trace metals) and observed the same results.
Run 1: (43.7 mg, 0.188 mmol, 47% yield). Run 2: (29.7 mg, 0.128 mmol, 32% yield). Run 3: (40.9 mg, 0.176 mmol, 44% yield). **Average: 41% yield [S44] ± 6.5.**

These results are consistent with other reports in the literature where a nearly identical yield was achieved without catalyst (**Fig. S13**). According to Woo, the following transformation occurred in 78% yield under iron catalysis [17]. According to Davies, the same transformation occurred in 76% yield without the addition of catalyst [20].

Fig. S13



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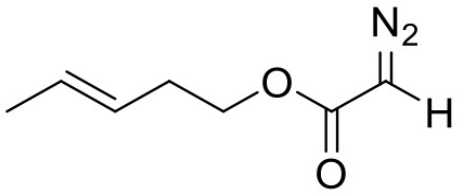

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Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	6.5000
Presaturation Frequency	
Acquisition Time	4.6645
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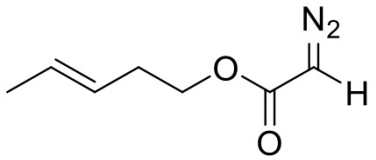
Spectral Width	7024.9
Lowest Frequency	-1022.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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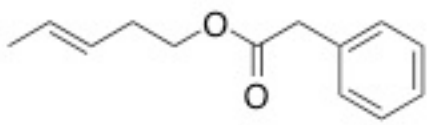
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	672
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	6.0000
Presaturation Frequency	
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	30
Relaxation Delay	10.0000
Pulse Width	12.0000
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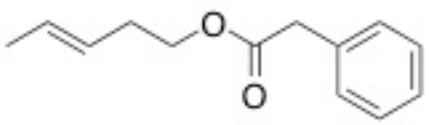
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
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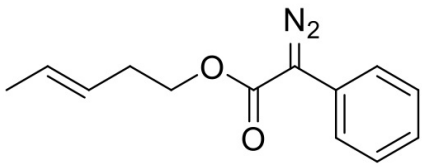
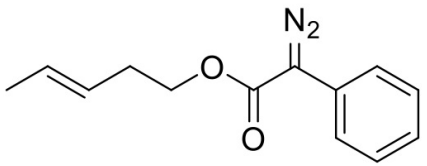
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Instrument	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	6.5000
Presaturation Frequency	
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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Owner	
Site	
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Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
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Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
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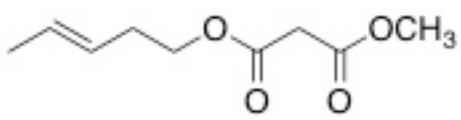

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Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
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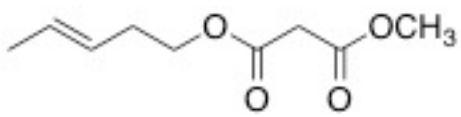
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Owner	
Site	
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Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	136
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Modification Date	2016-11-09T18:59:15
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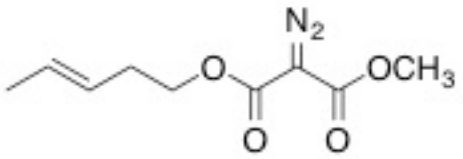
Lowest Frequency	-1276.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
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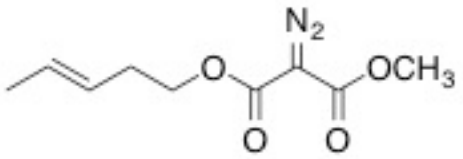
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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Experiment	1D
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Number of Scans	160
Receiver Gain	60
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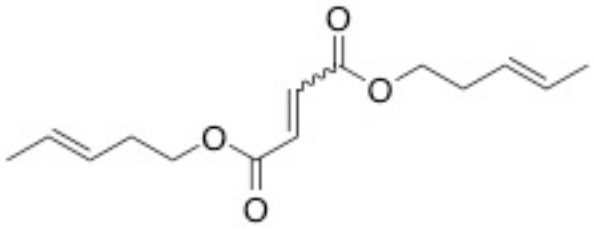
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Pulse Sequence	s2pul
Experiment	1D
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Number of Scans	16
Receiver Gain	52
Relaxation Delay	10.0000
Pulse Width	6.5000
Presaturation Frequency	
Acquisition Time	4.6645
Acquisition Date	2016-06-08T18:08:23
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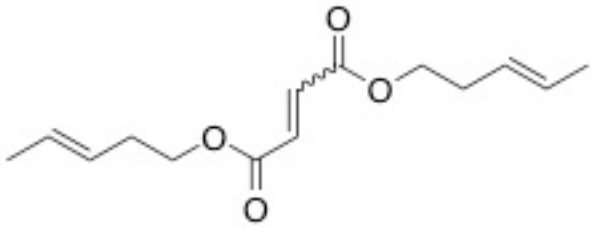
Spectral Width	7024.9
Lowest Frequency	-1021.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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Site	
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Author	
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Temperature	20.0
Pulse Sequence	s2pul
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Pulse Width	0.0000
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Class	
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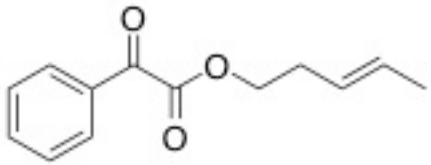
Lowest Frequency	-1276.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
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Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	8
Receiver Gain	30
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-11-14T10:30:29
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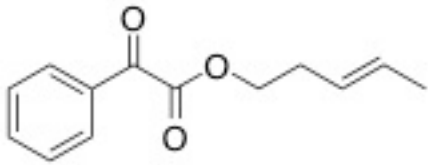

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-11-14T13:10:33
Modification Date	2016-11-14T13:10:32
Class	
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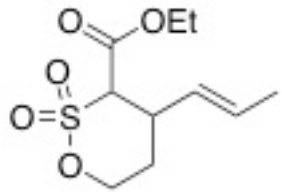
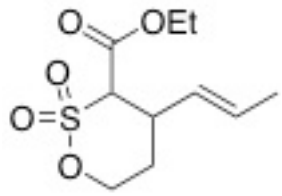
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



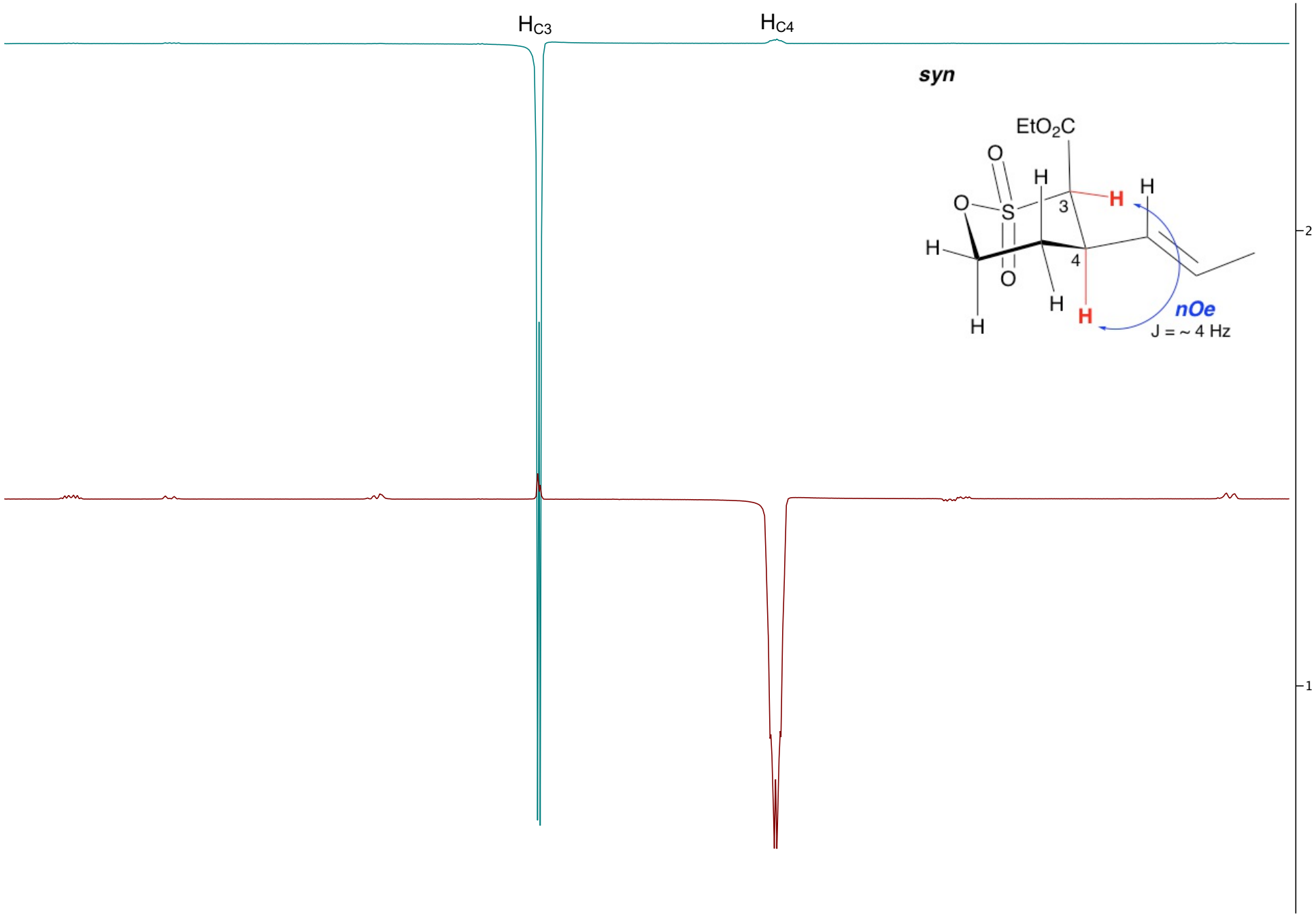
Syn diastereomer

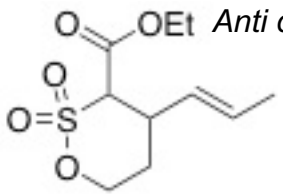
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2015-08-27T09:54:54
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	451
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-08-27T20:23:00
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1279.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536


H_C3
H_C4
syn
EtO2C
O
S
O
O
H
H
H
H
H
3
4
H
H
nOe
J = ~ 4 Hz
2
1
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)



Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
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Receiver Gain	56
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Pulse Width	0.0000
Modification Date	2015-08-27T10:02:00
Class	
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Spectral Width	8000.0
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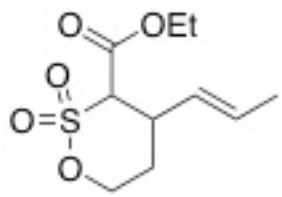
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Anti diastereomer

—134.43 Pc ligand

—123.68 Pc ligand

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	459
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-08-27T20:50:49
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Spectrometer Frequency	125.66
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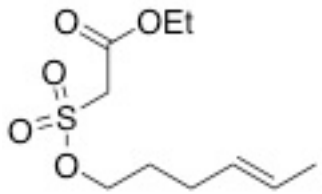
Lowest Frequency	-1277.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2015-11-16T11:15:42
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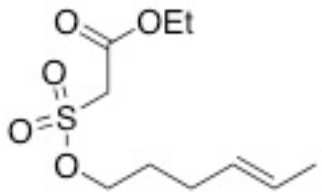
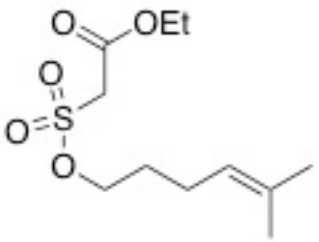
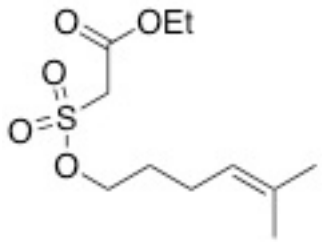
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



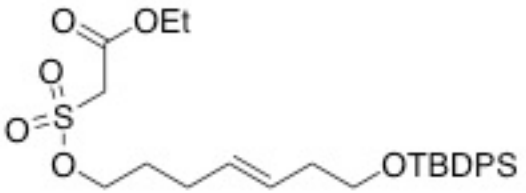
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	32
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Modification Date	2015-11-16T11:23:58
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1289.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



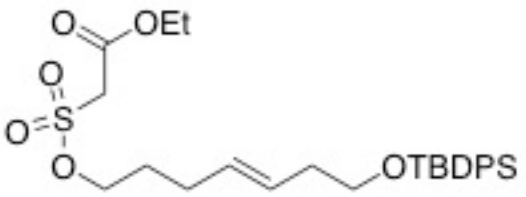
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2015-11-24T11:10:35
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



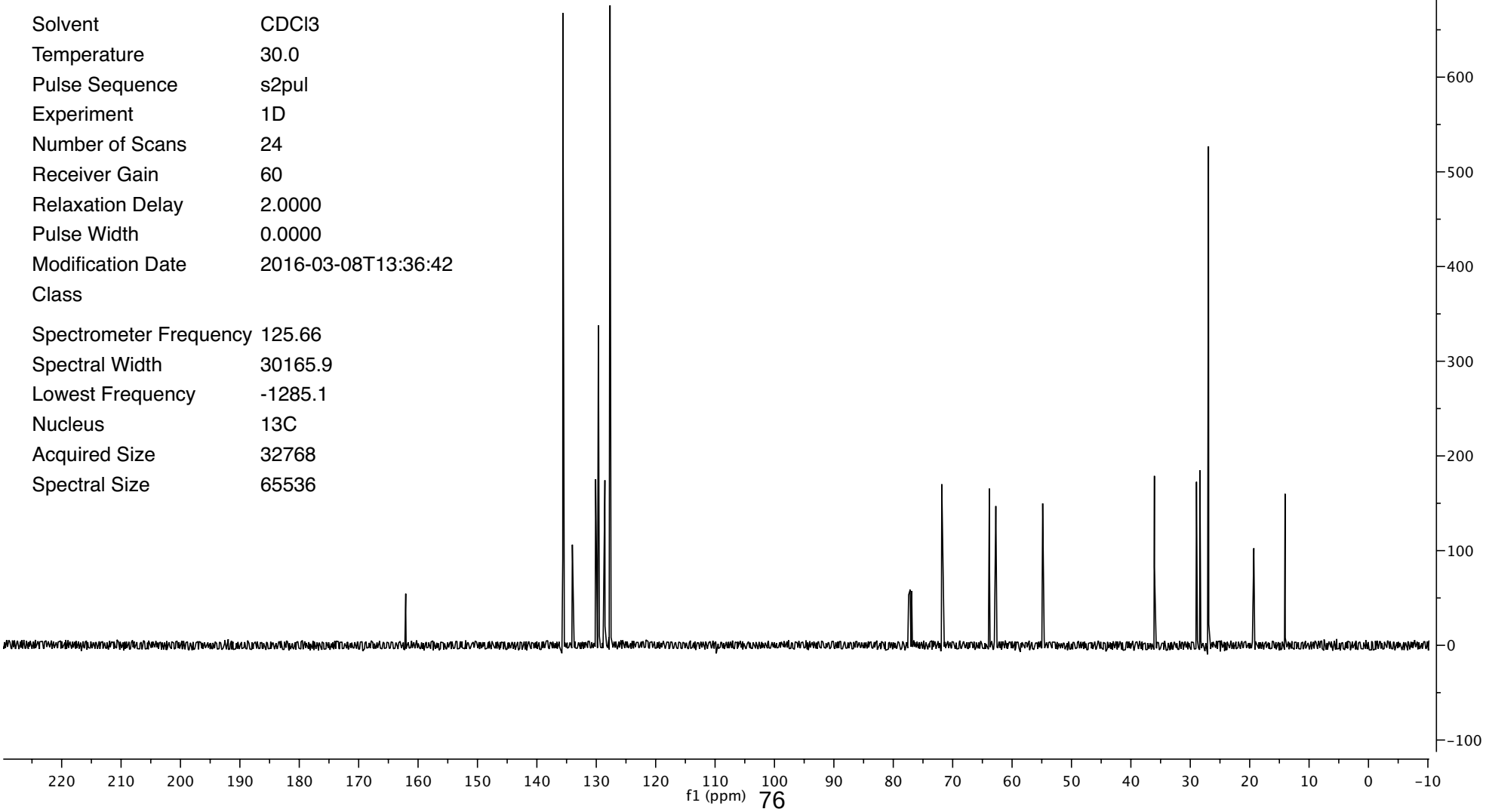
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	211
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-11-24T13:52:21
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1274.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

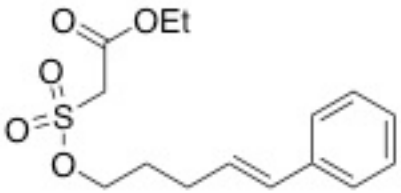


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-03-08T13:33:33
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

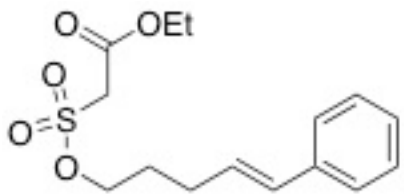


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	24
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-03-08T13:36:42
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1285.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

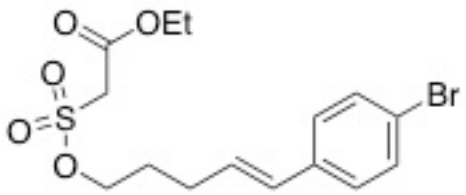




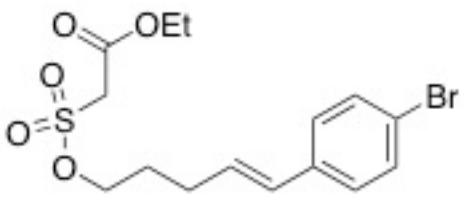
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	54
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-04-18T10:55:46
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1519.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



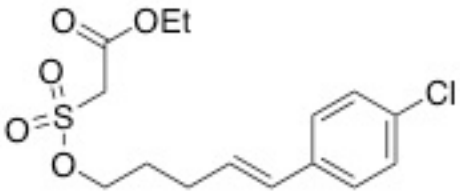
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	163
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-18T11:06:26
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2247.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



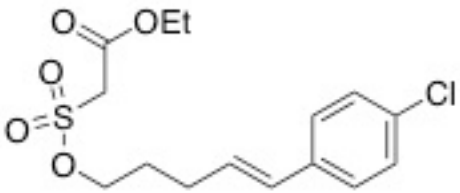
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	29.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



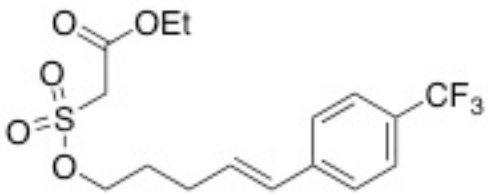
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	464
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



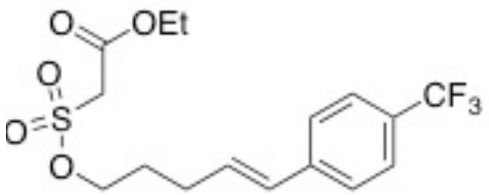
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	50
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-01-19T18:05:04
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



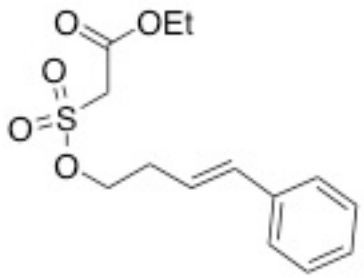
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	224
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-19T18:25:37
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2212.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



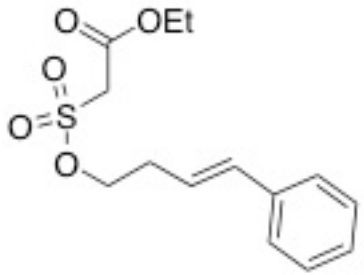
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



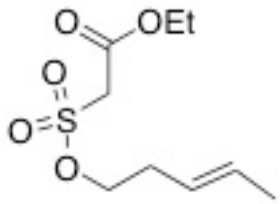
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	176
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



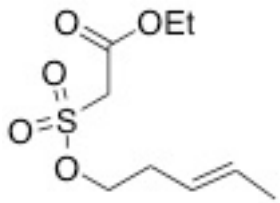
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-09-02T08:42:59
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



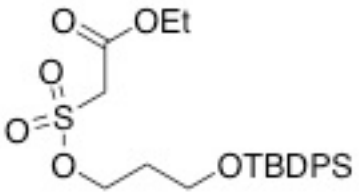
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	48
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-09-02T08:47:53
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



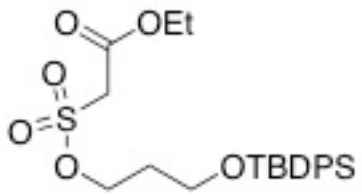
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-09-27T11:18:34
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



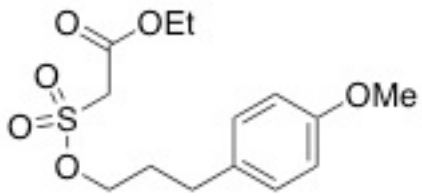
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	79
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-09-27T11:24:55
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1287.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



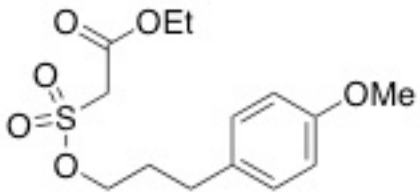
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-03-24T08:51:12
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1011.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



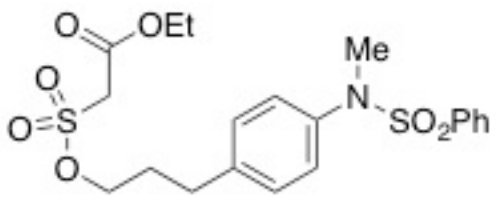
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	28
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-03-24T08:54:13
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1289.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



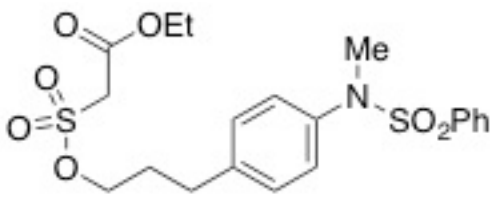
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	30
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-01-08T11:01:12
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2423.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



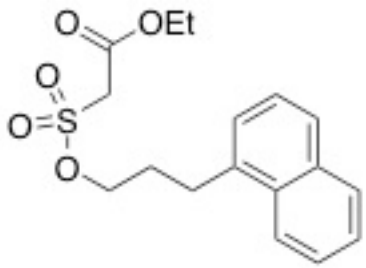
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	192
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-19T15:09:56
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1282.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



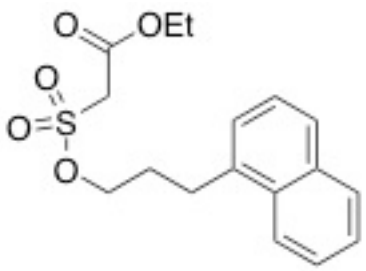
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	54
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-05-07T14:18:30
Class	
Spectrometer Frequency	399.74
Spectral Width	8000.0
Lowest Frequency	-2425.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



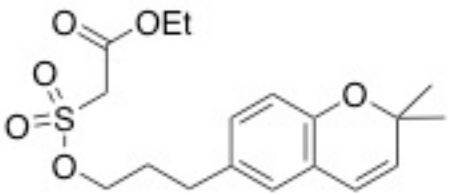
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	11532
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-30T07:52:34
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



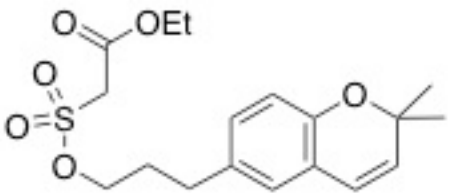
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	-20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	44
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-03-03T17:34:28
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1502.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



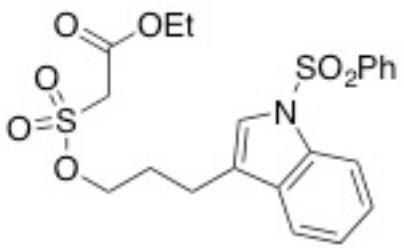
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	11808
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-07-02T07:52:38
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2188.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



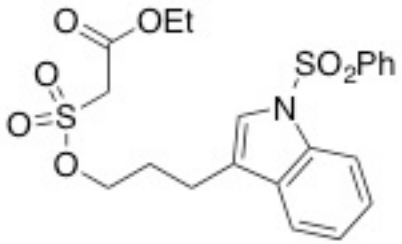
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-03-26T09:59:56
Class	
Spectrometer Frequency	399.74
Spectral Width	8000.0
Lowest Frequency	-2426.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



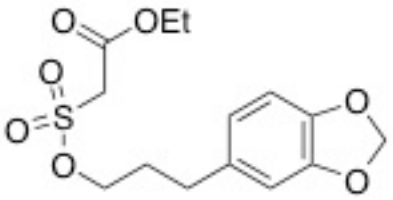
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	136
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-22T18:39:52
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2188.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



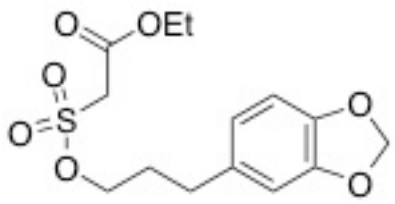
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	8
Receiver Gain	62
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-10-28T14:15:56
Modification Date	2016-10-28T14:15:56
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



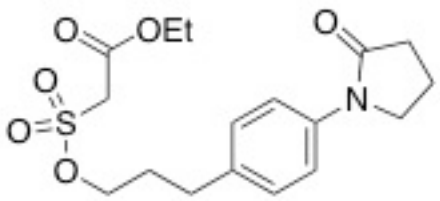
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-28T16:39:42
Modification Date	2016-10-28T16:39:42
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



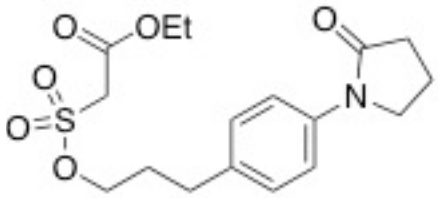
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2015-12-04T08:53:47
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



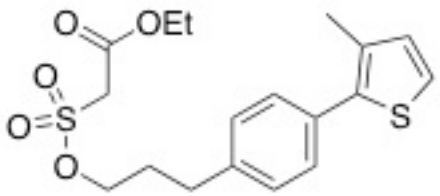
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	336
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-12-04T09:18:53
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2188.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



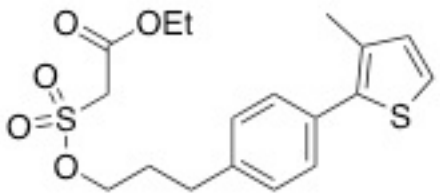
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-03-31T15:02:08
Class	
Spectrometer Frequency	399.74
Spectral Width	8000.0
Lowest Frequency	-2425.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



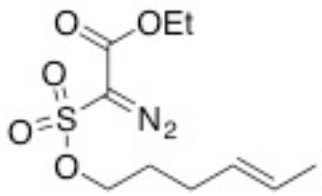
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	136
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-21T20:10:44
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



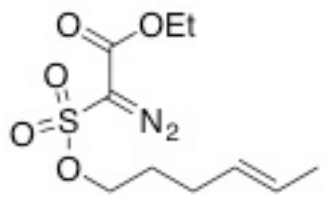
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-02-29T18:01:02
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



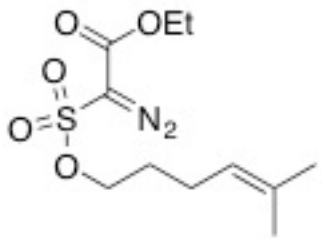
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	108
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-21T21:34:18
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



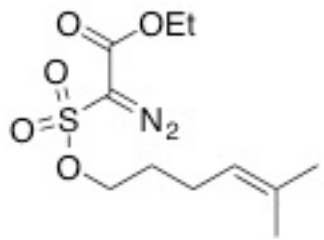
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	32
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2015-11-18T14:04:09
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



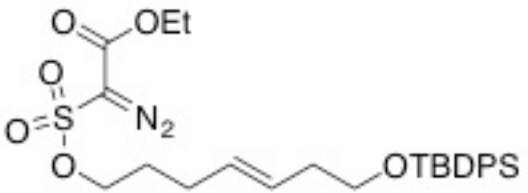
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	215
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-11-24T13:39:23
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1275.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



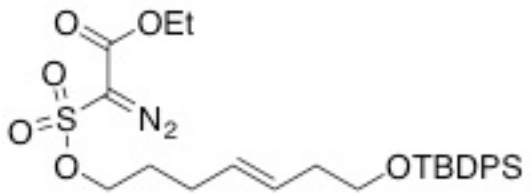
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	32
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2015-12-08T10:39:38
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



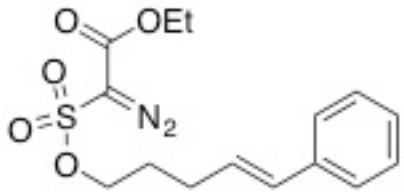
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	80
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2015-11-24T11:35:49
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1278.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



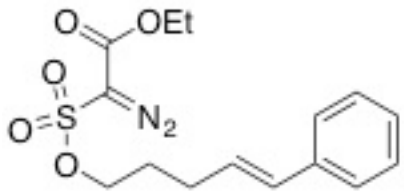
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	48
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-03-09T08:51:43
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



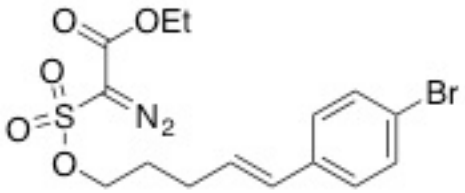
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	234
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-03-27T11:44:40
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



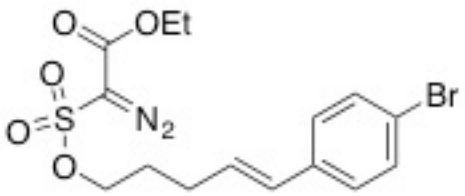
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-09T18:33:23
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



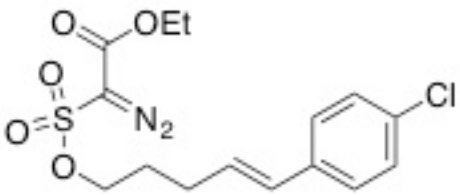
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	240
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-09T18:48:19
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



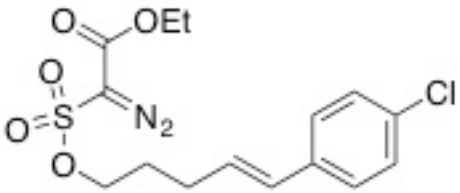
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



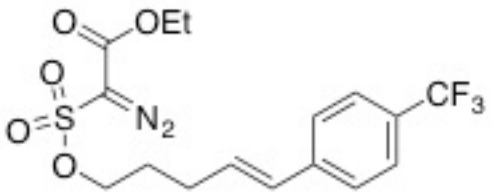
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	272
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



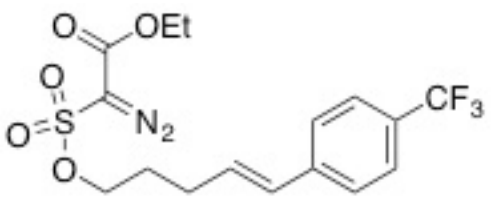
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	50
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-01-23T11:29:03
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1522.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



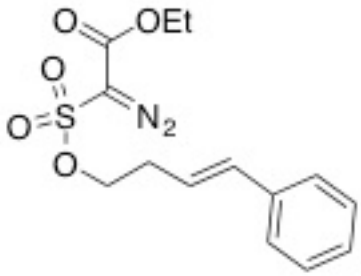
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	12
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-03-27T11:28:19
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



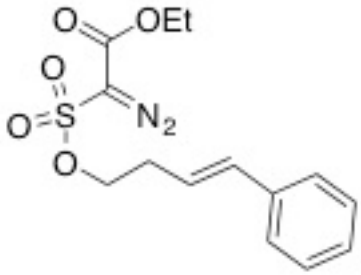
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



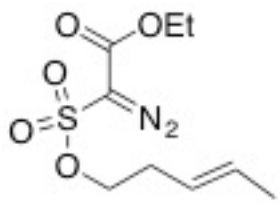
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	560
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-10-10T14:56:09
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

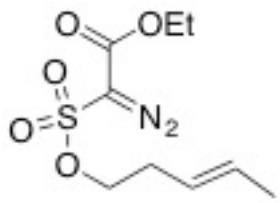


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	40
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-10-10T15:00:15
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1281.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

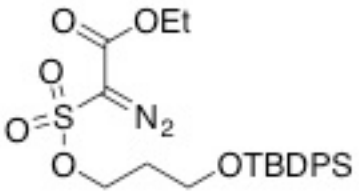


5.30 CH_2Cl_2

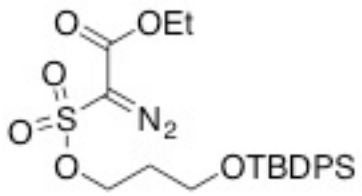
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	137
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-11-11T09:58:27
Modification Date	2016-11-11T09:58:27
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1956.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



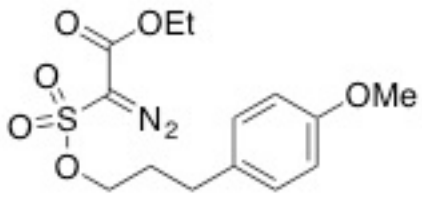
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	372
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-11-11T09:53:51
Modification Date	2016-11-11T09:53:51
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2282.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



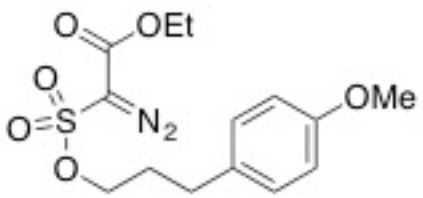
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-04-30T09:06:38
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1011.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



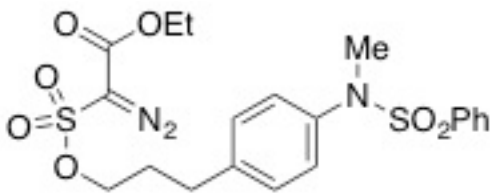
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	193
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-30T09:18:23
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1275.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



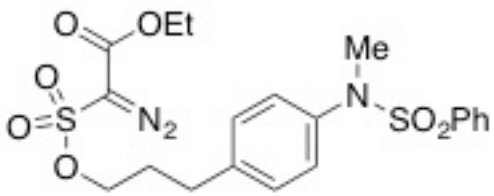
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	1.0000
Pulse Width	0.0000
Modification Date	2016-01-29T08:47:32
Class	
Spectrometer Frequency	399.74
Spectral Width	8000.0
Lowest Frequency	-2426.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



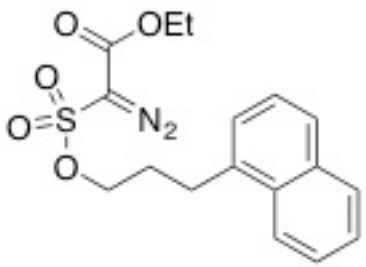
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-13T15:25:37
Modification Date	2016-10-13T15:25:36
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2282.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



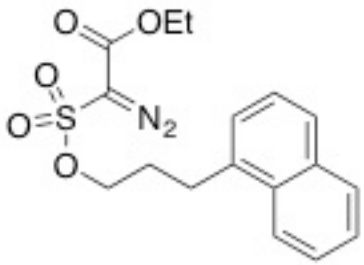
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	0
Receiver Gain	41
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-14T09:05:22
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



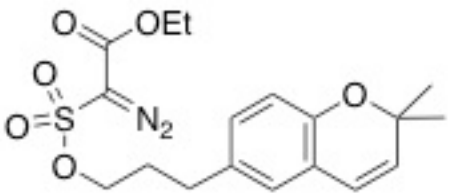
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	27.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	744
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-30T21:54:32
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2208.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



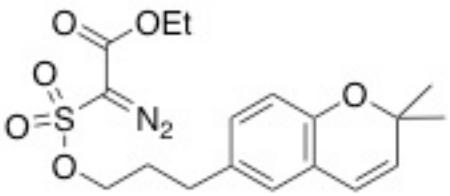
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	12
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-26T09:19:56
Class	
Spectrometer Frequency	399.74
Spectral Width	8000.0
Lowest Frequency	-2426.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



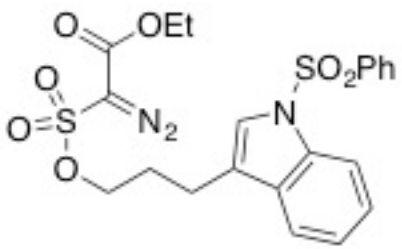
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	464
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-10-08T17:11:24
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1277.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



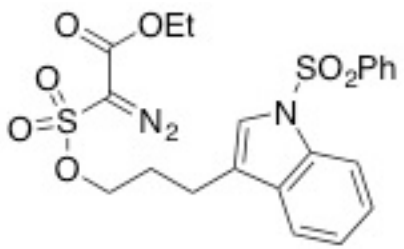
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	24
Receiver Gain	54
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-04-14T08:58:44
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1502.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



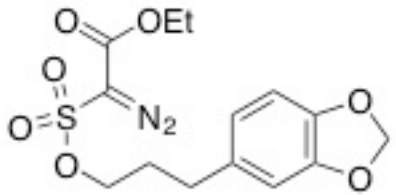
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	264
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-05-09T20:34:10
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1279.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



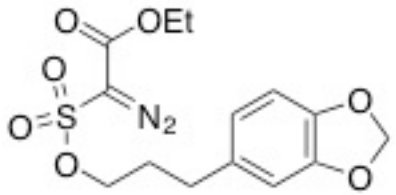
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	32
Receiver Gain	60
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-03-01T21:39:06
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



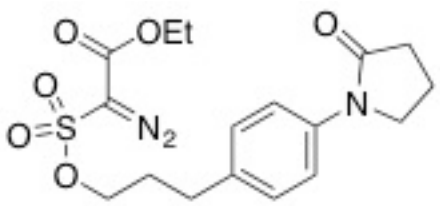
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	27.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	440
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-30T20:23:54
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2204.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



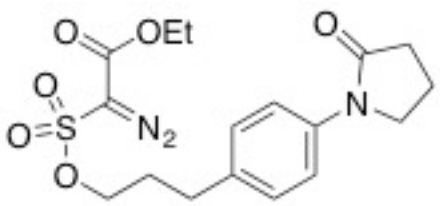
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-01-25T09:51:47
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



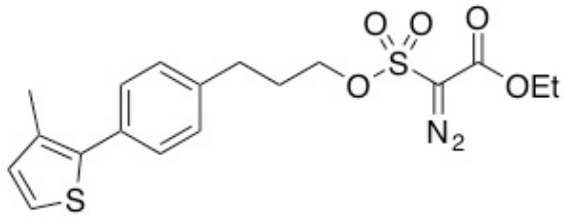
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	116
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-25T09:59:28
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



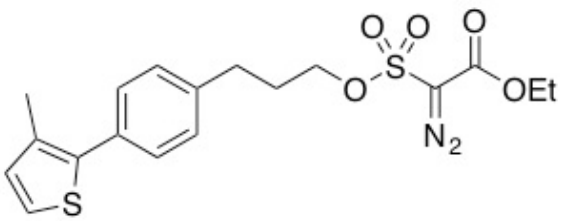
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-28T09:42:36
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1518.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



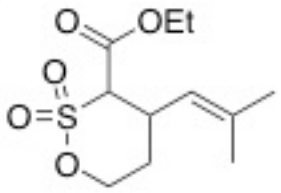
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	1284
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-07-03T21:06:42
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2211.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	32
Receiver Gain	60
Relaxation Delay	3.0000
Pulse Width	0.0000
Modification Date	2016-03-15T09:34:06
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

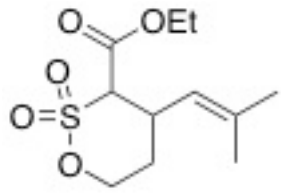


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	27.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	552
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-30T20:55:10
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2188.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



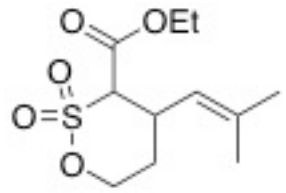
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	50
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-01-23T11:35:51
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



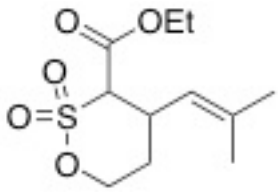
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	166
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-23T14:11:49
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	131072



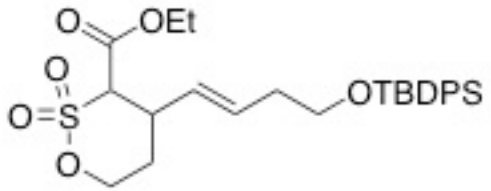
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	56
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-01-23T11:43:03
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



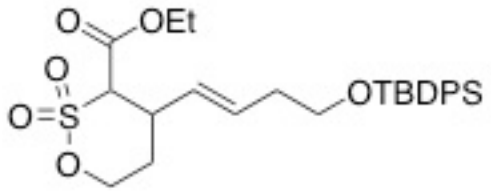
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	11666
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-02-17T07:55:07
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1271.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



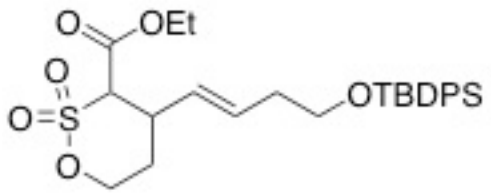
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	32
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-30T16:53:16
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



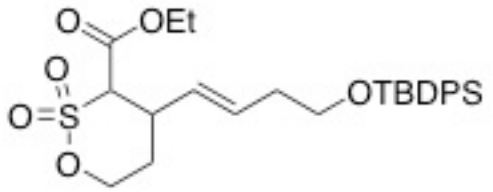
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	560
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-30T18:23:47
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2202.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



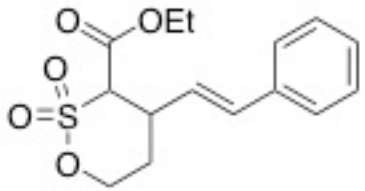
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-19T08:22:15
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



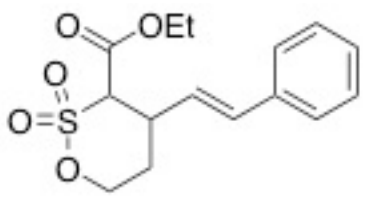
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	560
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-19T09:01:24
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1274.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



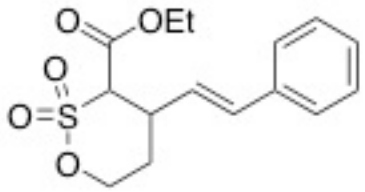
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-24T09:40:04
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



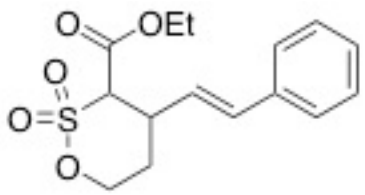
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	97
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-05-24T20:55:41
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1284.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



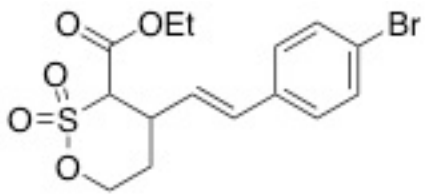
Anti diastereomer
(contains 12% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	48
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-24T09:46:10
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



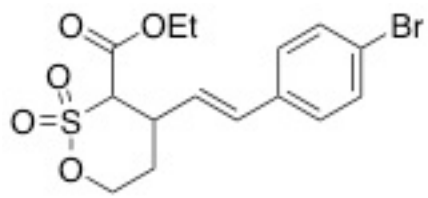
Anti diastereomer
(contains 12% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	84
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-05-24T21:02:26
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



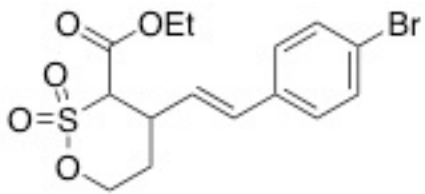
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-18T14:00:33
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1011.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



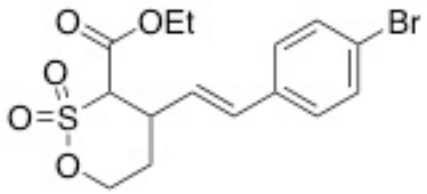
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	168
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-18T14:12:19
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



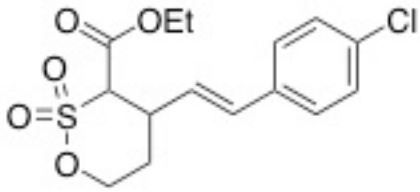
Anti diastereomer
(contains 10% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-18T10:30:16
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



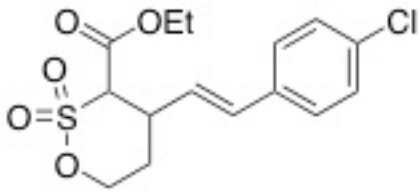
Anti diastereomer
(contains 12% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	150
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-18T14:22:28
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1280.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



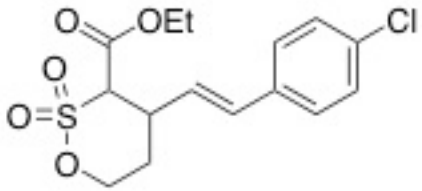
Syn diastereomer
(contains 6% anti)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-23T13:22:51
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



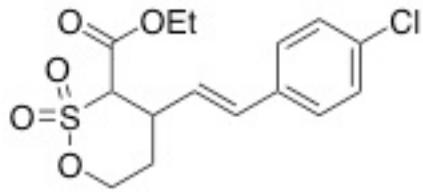
Syn diastereomer
(contains 6% anti)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	28
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-23T15:13:56
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1278.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



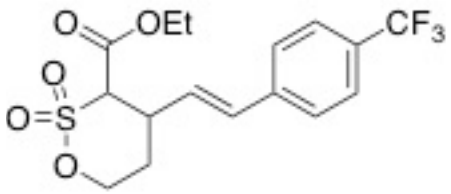
Anti diastereomer
(contains 10% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-23T13:29:28
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



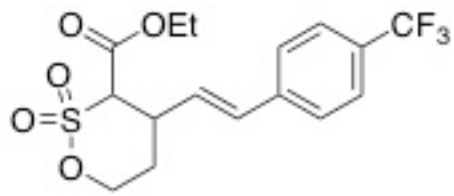
Anti diastereomer
(contains 10% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	692
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-04-23T15:51:51
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1274.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



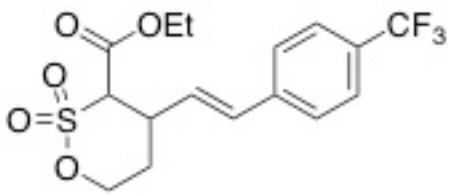
Syn diastereomer
(contains 10% anti)

Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



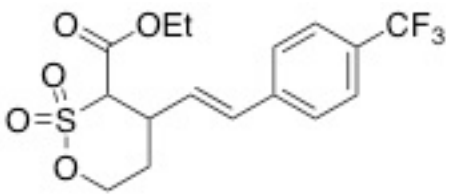
Syn diastereomer
(contains 10% anti)

Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	352
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



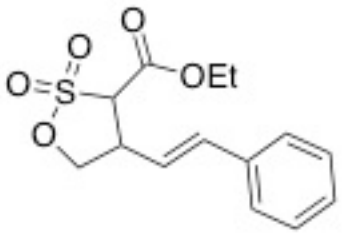
Anti diastereomer

Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	16
Receiver Gain	50
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	4.6645
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Anti diastereomer

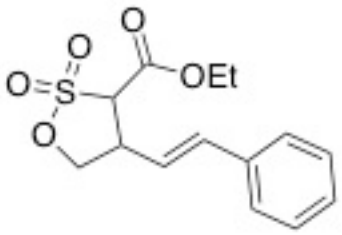
Parameter	Value
Origin	Varian
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Probe	QUAD
Number of Scans	320
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	1.0863
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Mixture of diastereomers

(d.r. calculated off of crude)

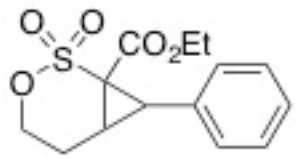
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	5.0000
Pulse Width	0.0000
Modification Date	2016-10-13T09:10:37
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



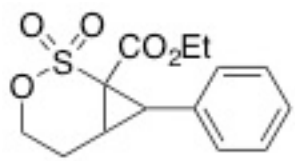
Mixture of diastereomers

(d.r. calculated off of crude)

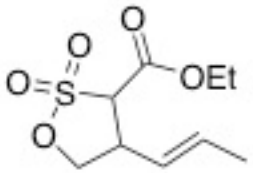
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	56
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-10-13T09:15:17
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1290.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-10-12T13:48:13
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



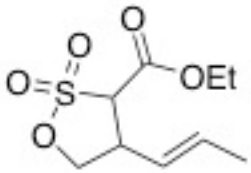
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	96
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-10-13T09:23:39
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1280.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Mixture of diastereomers

(d.r. calculated off of crude)

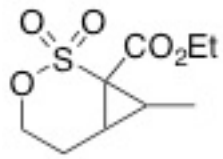
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	191
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-11-21T18:22:03
Modification Date	2016-11-21T18:22:03
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



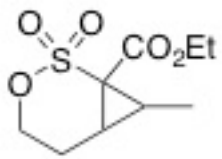
Mixture of diastereomers

(d.r. calculated off of crude)

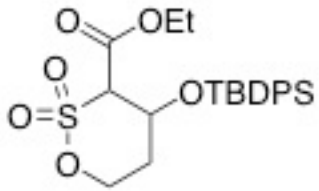
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	136
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-11-30T08:35:06
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1274.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	54
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-11-15T13:52:20
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1521.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

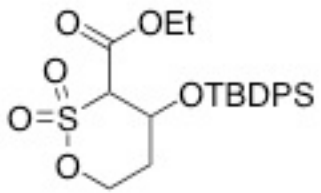


Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-11-15T18:21:55
Modification Date	2016-11-15T18:21:55
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



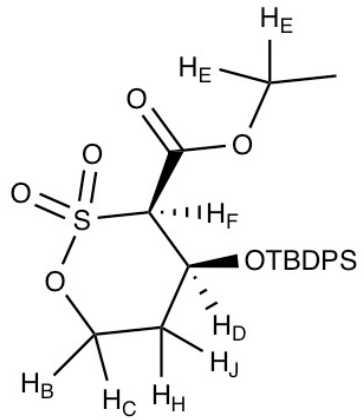
1:1 anti:syn
mix of diastereomers

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-14T08:32:16
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1011.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

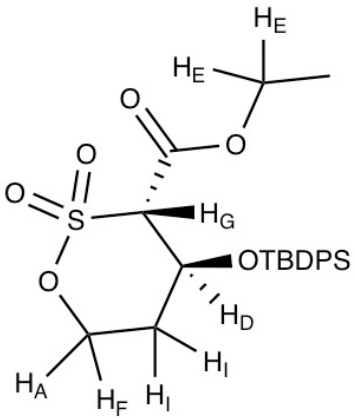


1:1 anti:syn
mix of diastereomers

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	240
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-17T19:39:29
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

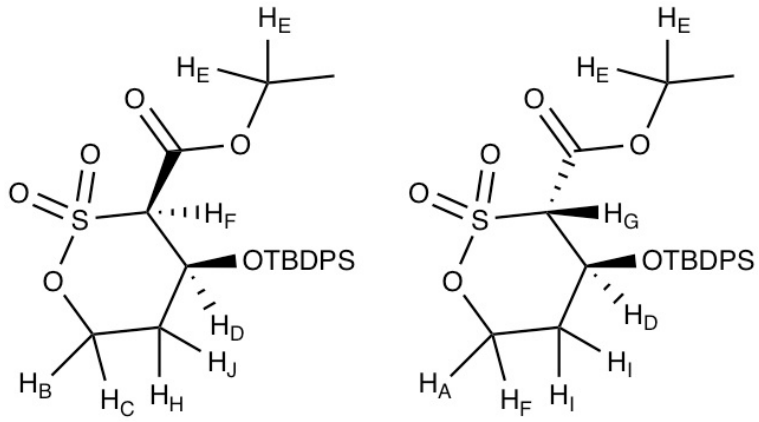

H_E
H_E
O
O
O
S
O
H_F
OTBDPS
H_D
H_J
H_B
H_C
H_H

syn


H_E
H_E
O
O
O
S
O
H_G
OTBDPS
H_D
H_I
H_A
H_F
H_I

anti

A
B
C
D
E
F
G
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
4.65
4.60
4.55
4.50
4.45
4.40
4.35
4.30
4.25
4.20
4.15
4.10
4.05
4.00
3.95
f1 (ppm)

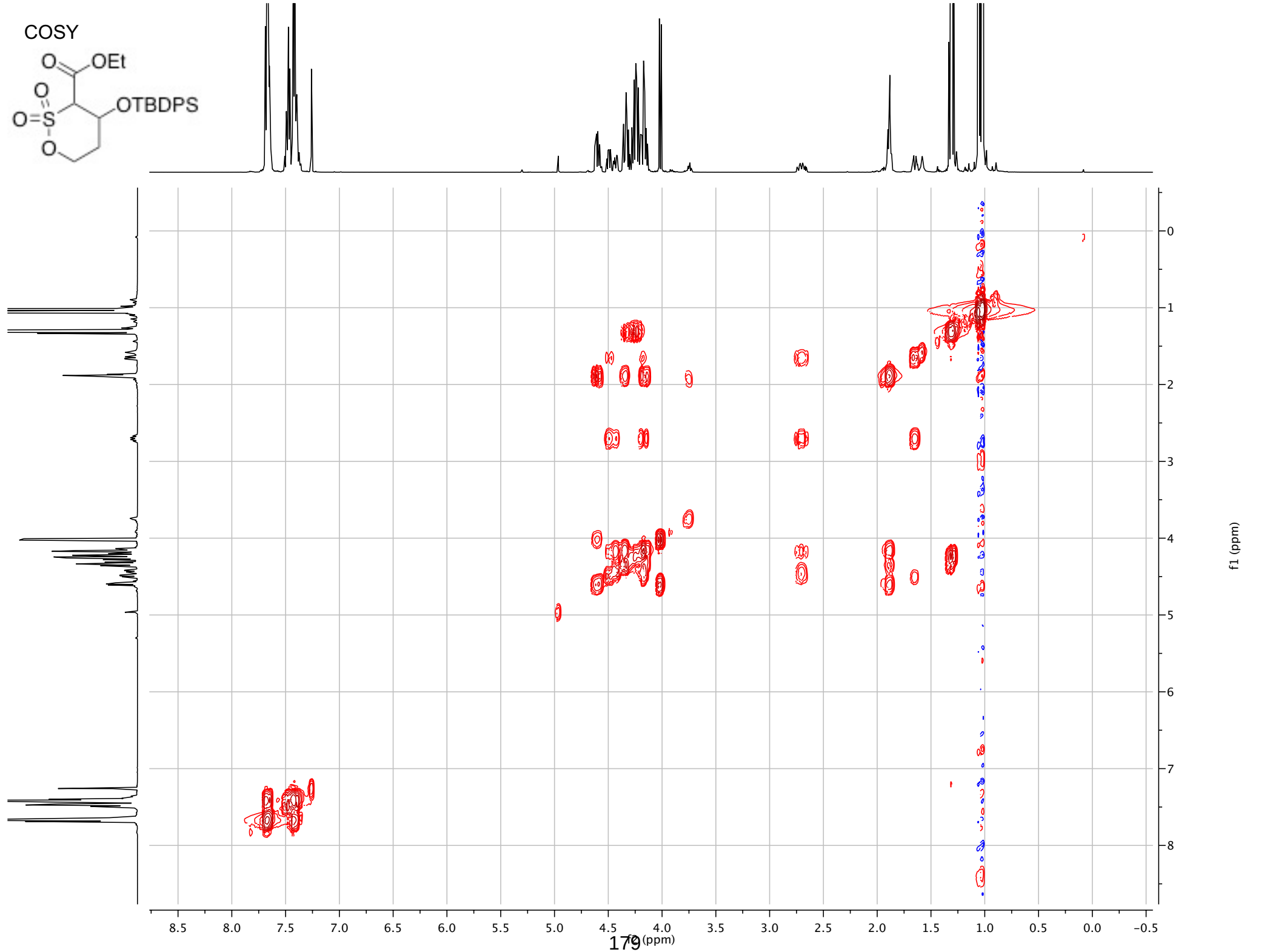

H_E
H_E
O
O
O
S
O
H_F
OTBDPS
H_D
H_J
H_B
H_C
H_H

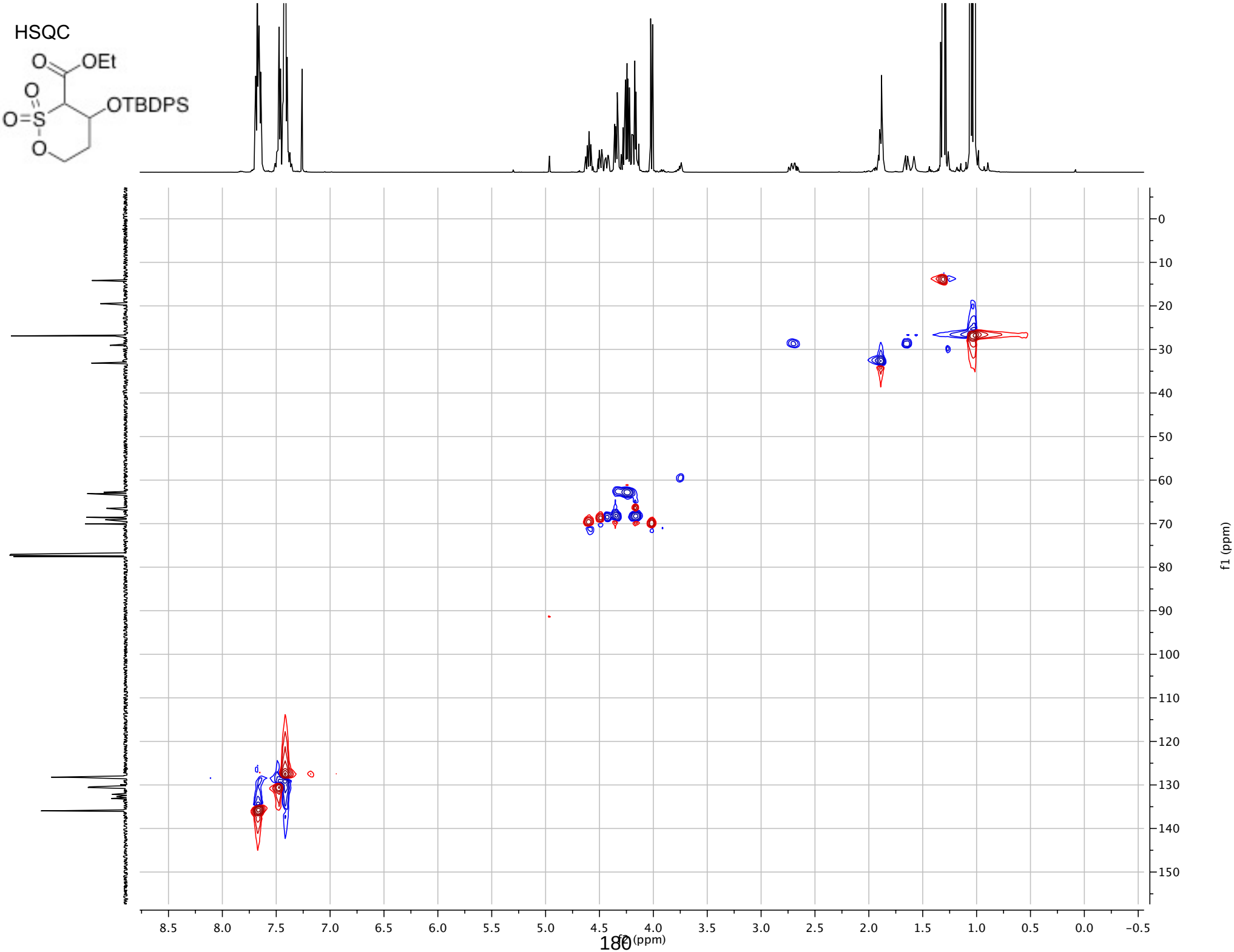
syn

H_E
H_E
O
O
O
S
O
H_G
OTBDPS
H_D
H_I
H_A
H_F
H_I

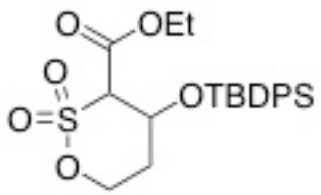
anti

H
I
J
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
2.80
2.75
2.70
2.65
2.60
2.55
2.50
2.45
2.40
2.35
2.30
2.25
2.20
2.15
2.10
2.05
2.00
1.95
1.90
1.85
1.80
1.75
1.70
1.65
1.60
f1 (ppm)

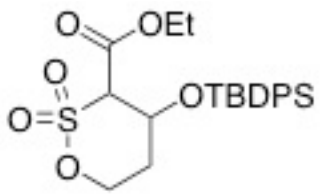

COSY
OEt
OTBDPS
f1 (ppm)
f2 (ppm)


HSQC
O
OEt
O
O=S
OTBDPS
O
f1 (ppm)
f2 (ppm)

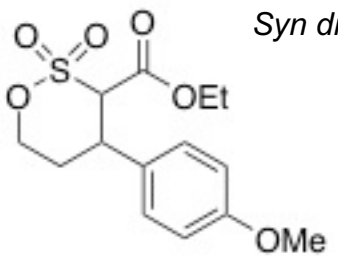
HMBC



TOCSY


O
OEt
O
O=S
OTBDPS
O

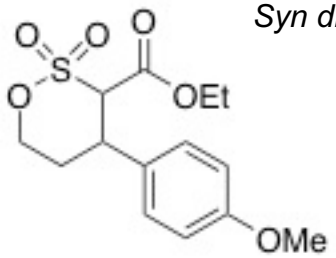
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f2 (ppm)
0
1
2
3
4
5
6
7
8
f1 (ppm)



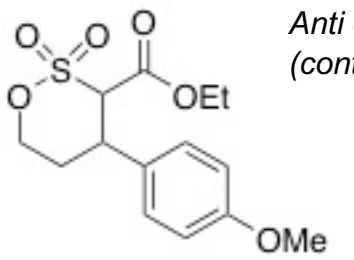
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-01-21T10:23:32
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Syn diastereomer

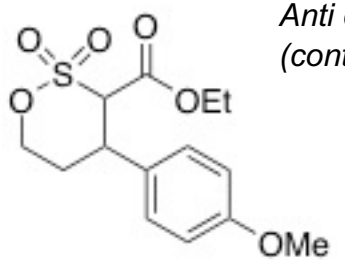


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	192
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-21T19:47:14
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1278.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



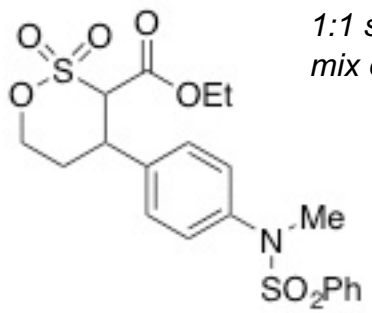
Anti diastereomer
(contains 15% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	52
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-01-21T10:28:28
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



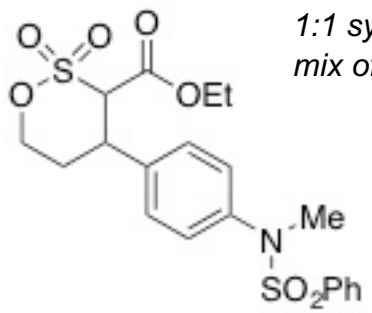
Anti diastereomer
(contains 15% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	432
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-01-21T20:13:48
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1273.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



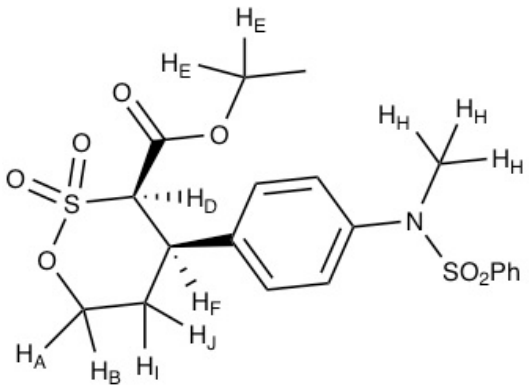
1:1 syn:anti
mix of diastereomers

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	4.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-08-15T20:07:32
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1518.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

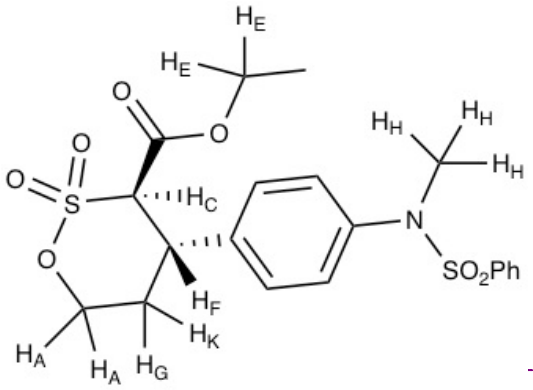


1:1 syn:anti
mix of diastereomers

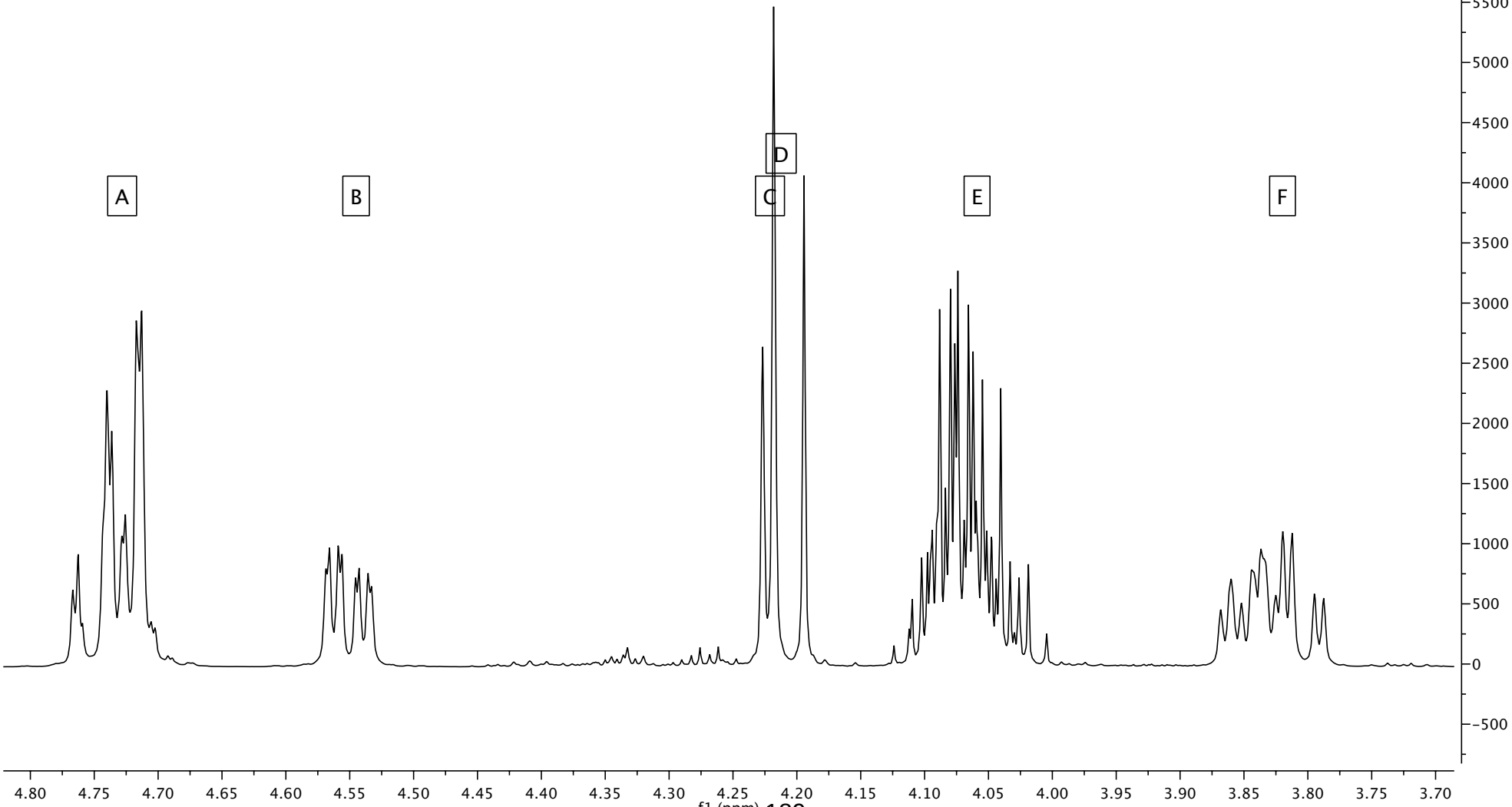
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	224
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-08-03T20:56:54
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2188.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

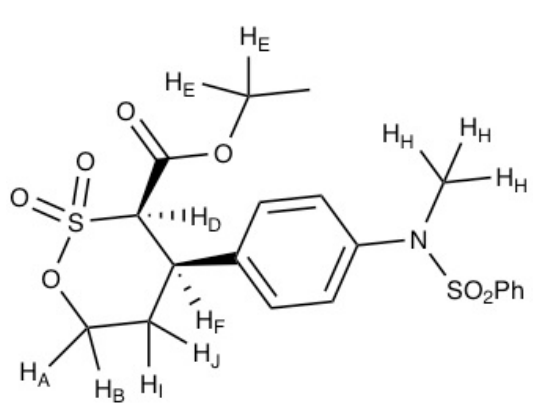

H_E
H_E
O
O
O
S
O
H_D
H_H
H_H
H_H
N
SO2Ph
H_F
H_J
H_A
H_B
H_I

syn

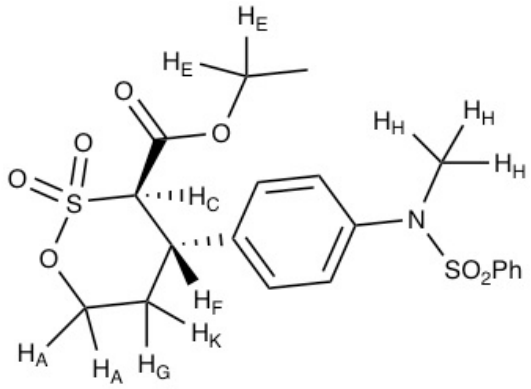

H_E
H_E
O
O
O
S
O
H_C
H_H
H_H
H_H
N
SO2Ph
H_F
H_K
H_A
H_A
H_G

anti


A
B
C
D
E
F
8500
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
4.80
4.75
4.70
4.65
4.60
4.55
4.50
4.45
4.40
4.35
4.30
4.25
4.20
4.15
4.10
4.05
4.00
3.95
3.90
3.85
3.80
3.75
3.70
f1 (ppm)



syn



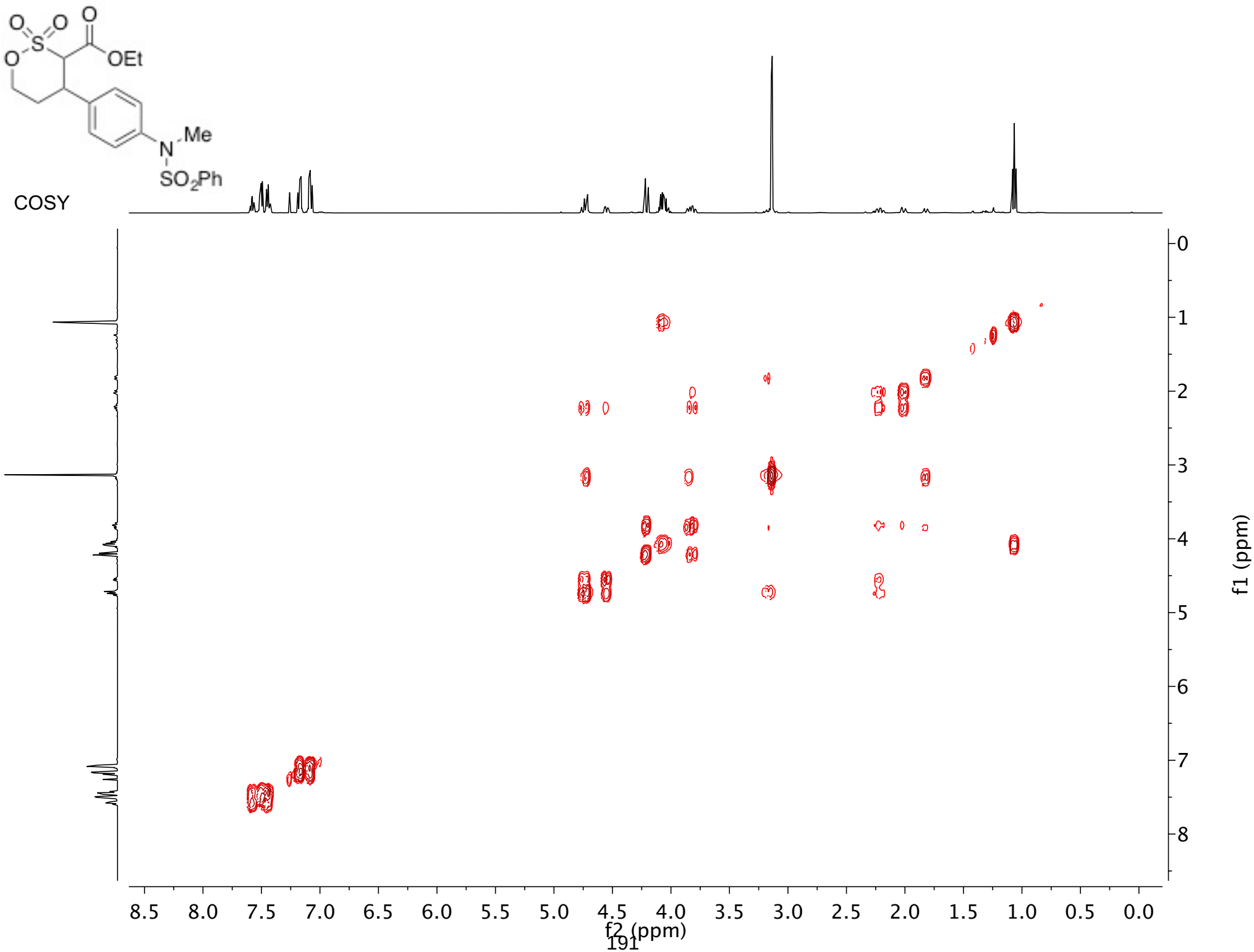
anti

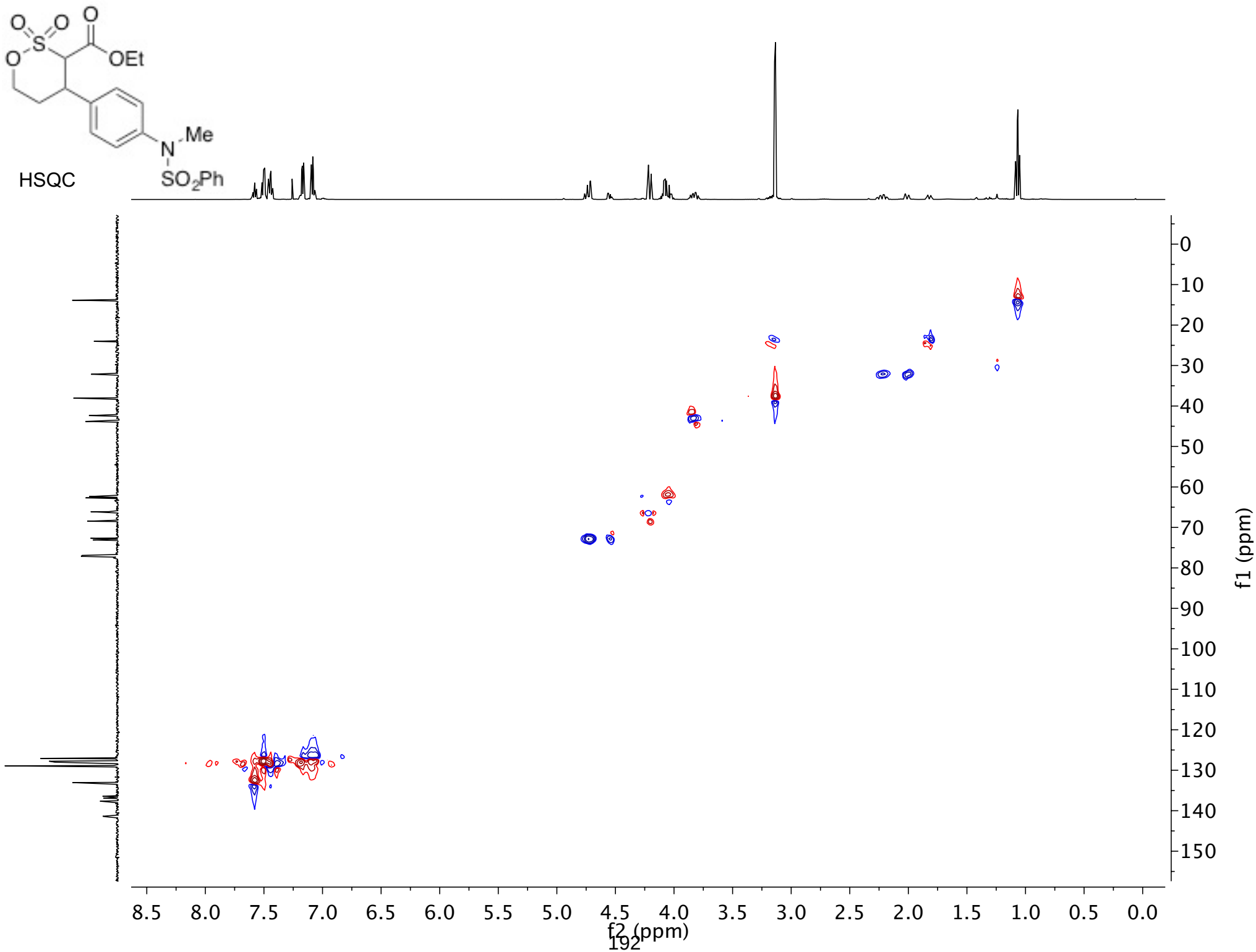
G H I J K

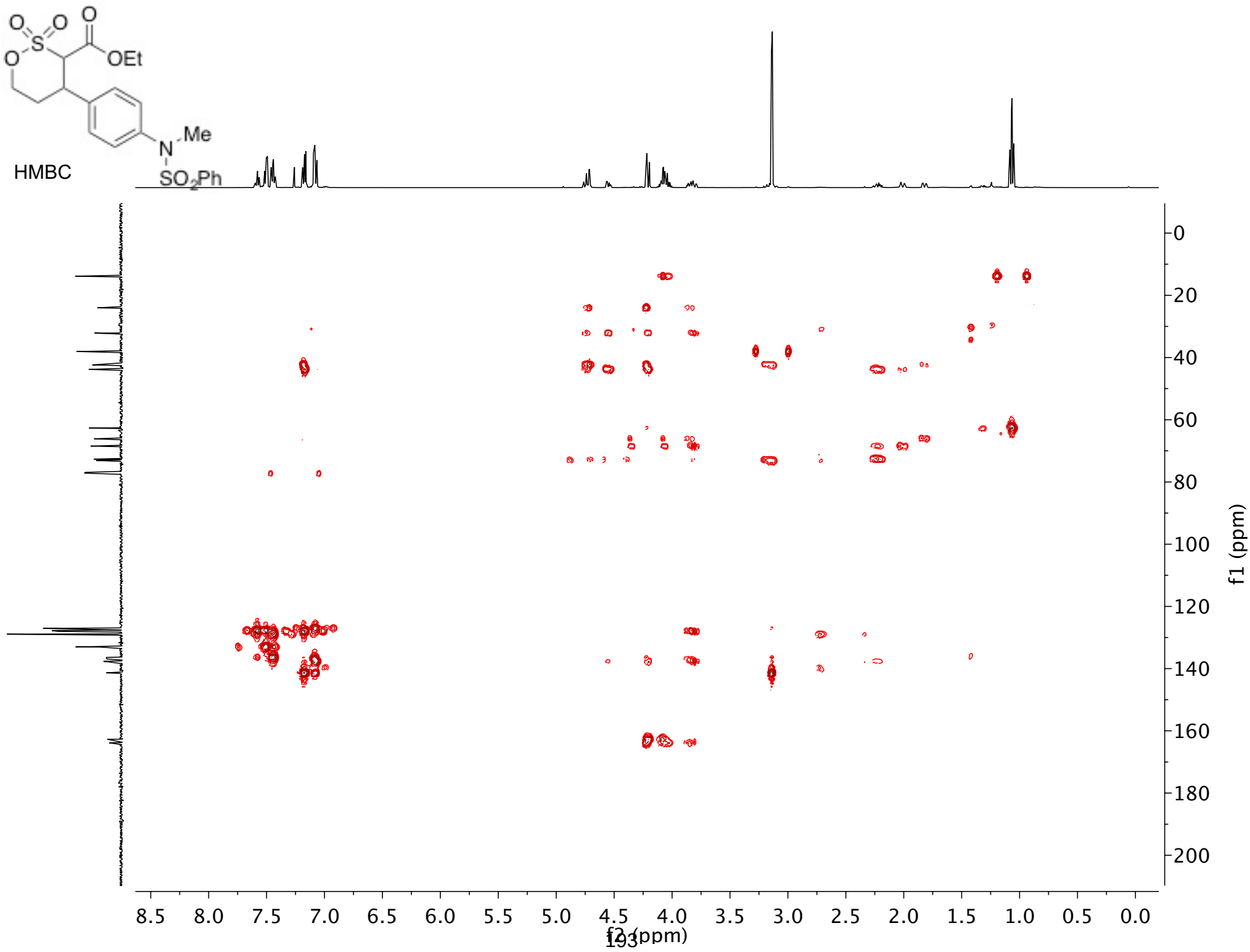
500 450 400 350 300 250 200 150 100 50 0

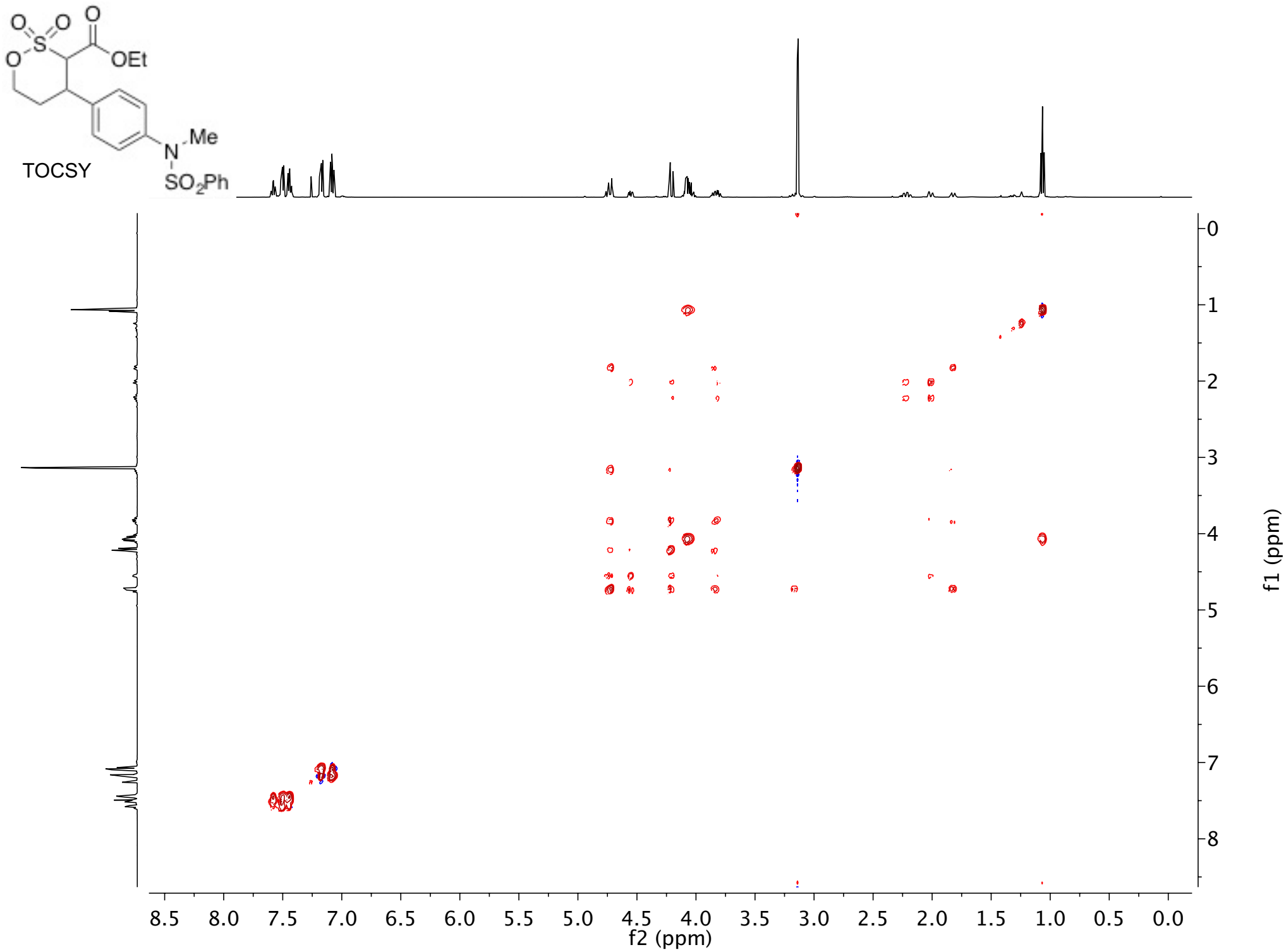
3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5

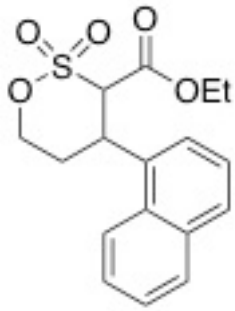
f1 (ppm)


COSY
O
O
S
O
O
OEt
N
Me
SO2Ph
f1 (ppm)
f2 (ppm)
0
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


HSQC
OEt
Me
N
SO2Ph
f1 (ppm)
f2 (ppm)

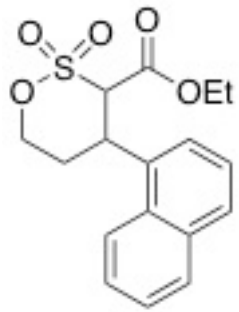

HMBC
OEt
Me
N
SO2Ph
f2 (ppm)
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
20
40
60
80
100
120
140
160
180
200


TOCSY
OEt
Me
N
SO2Ph
f2 (ppm)
f1 (ppm)



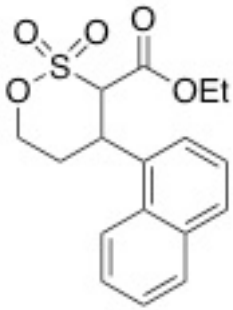
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	56
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-03-09T09:06:02
Class	
Spectrometer Frequency	399.95
Spectral Width	8000.0
Lowest Frequency	-2424.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



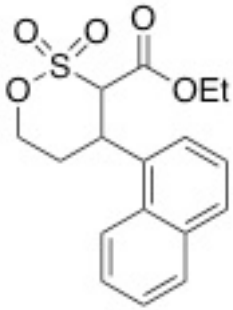
Syn diastereomer

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-31T11:53:30
Modification Date	2016-10-31T11:53:30
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2281.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



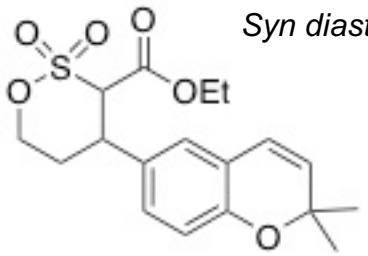
Anti diastereomer
(contains unknown impurity;
yield determined with internal standard)

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	8
Receiver Gain	107
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-10-25T15:36:17
Modification Date	2016-10-25T15:36:16
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



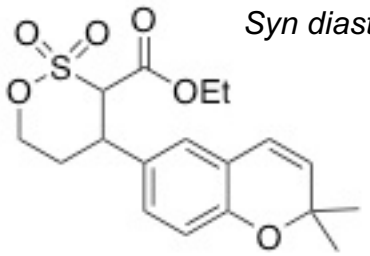
Anti diastereomer

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-25T15:57:40
Modification Date	2016-10-25T15:57:40
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2281.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



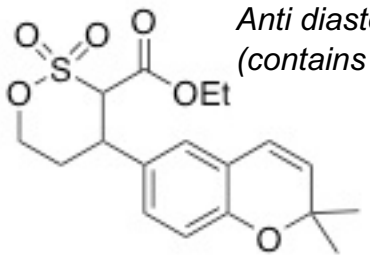
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	32
Receiver Gain	48
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-22T13:48:14
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



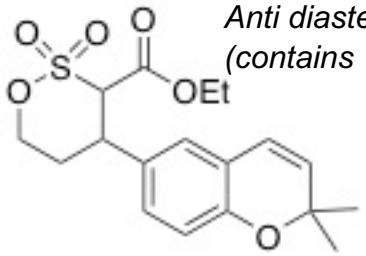
Syn diastereomer

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6650
Receiver Gain	191
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-18T02:15:23
Modification Date	2016-10-18T02:15:22
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2281.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



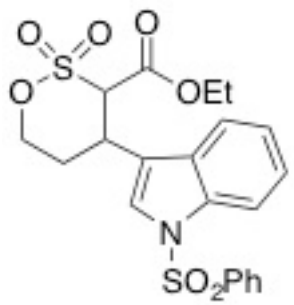
Anti diastereomer
(contains 10% syn)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	32
Receiver Gain	48
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-04-22T13:58:14
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



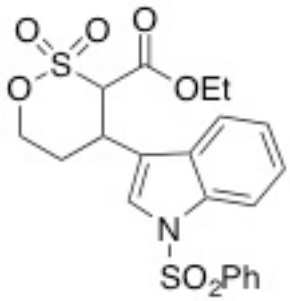
Anti diastereomer
(contains 10% syn)

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6650
Receiver Gain	191
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-19T06:55:05
Modification Date	2016-10-19T06:55:04
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2281.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



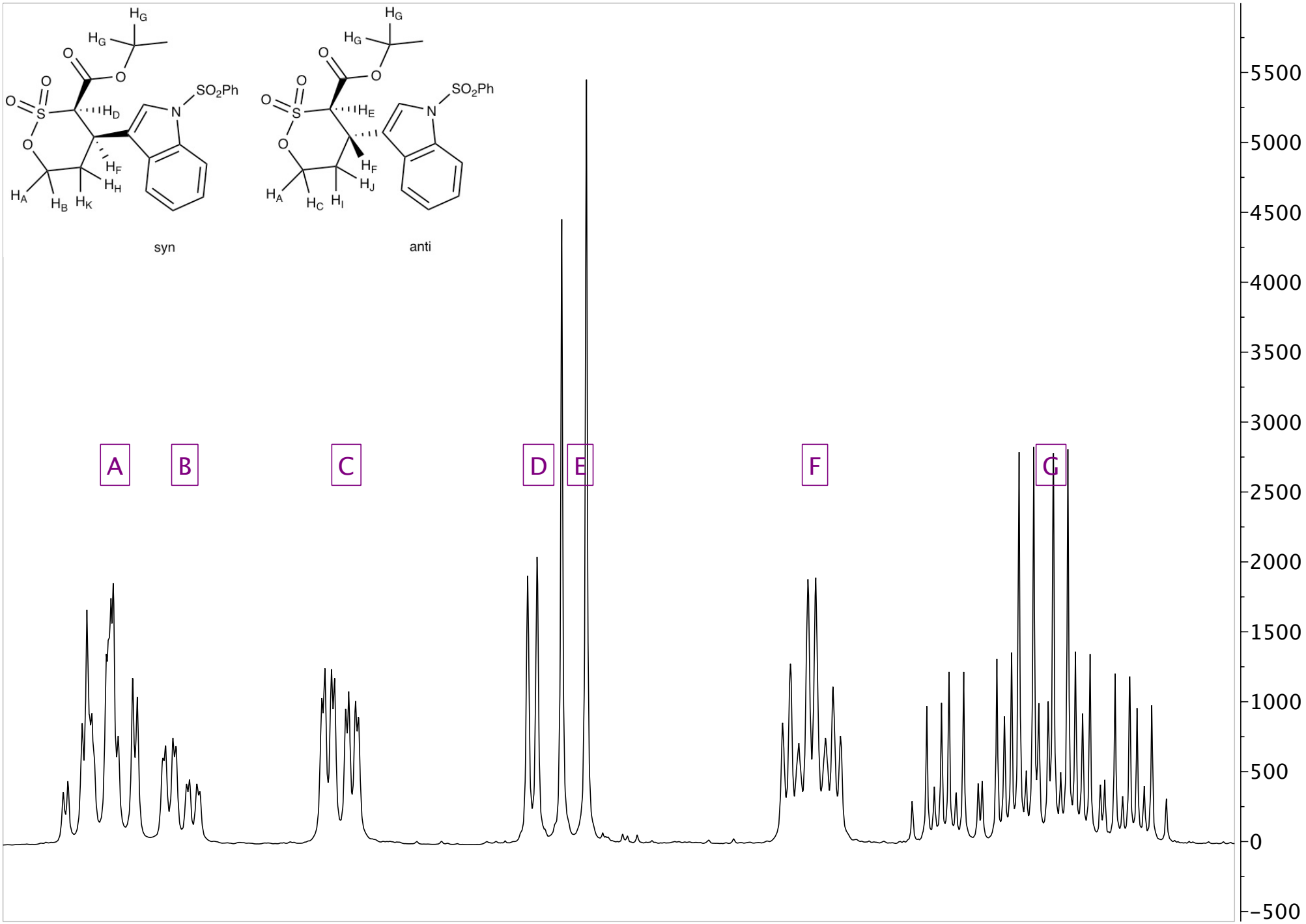
1:1 syn:anti
mix of diastereomers

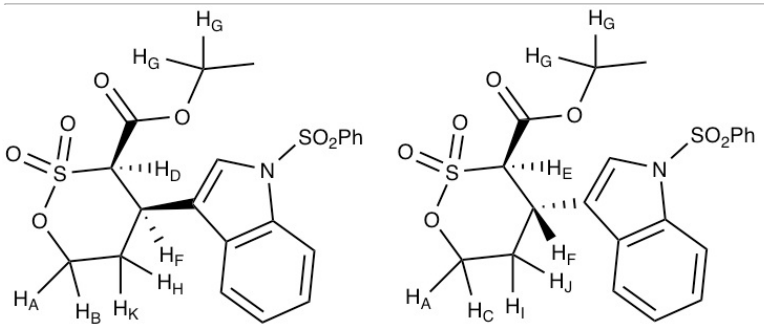
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	4.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-08-15T20:07:32
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest	-1518.7



1:1 syn:anti
mix of diastereomers

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	224
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-08-03T20:56:54
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2211.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

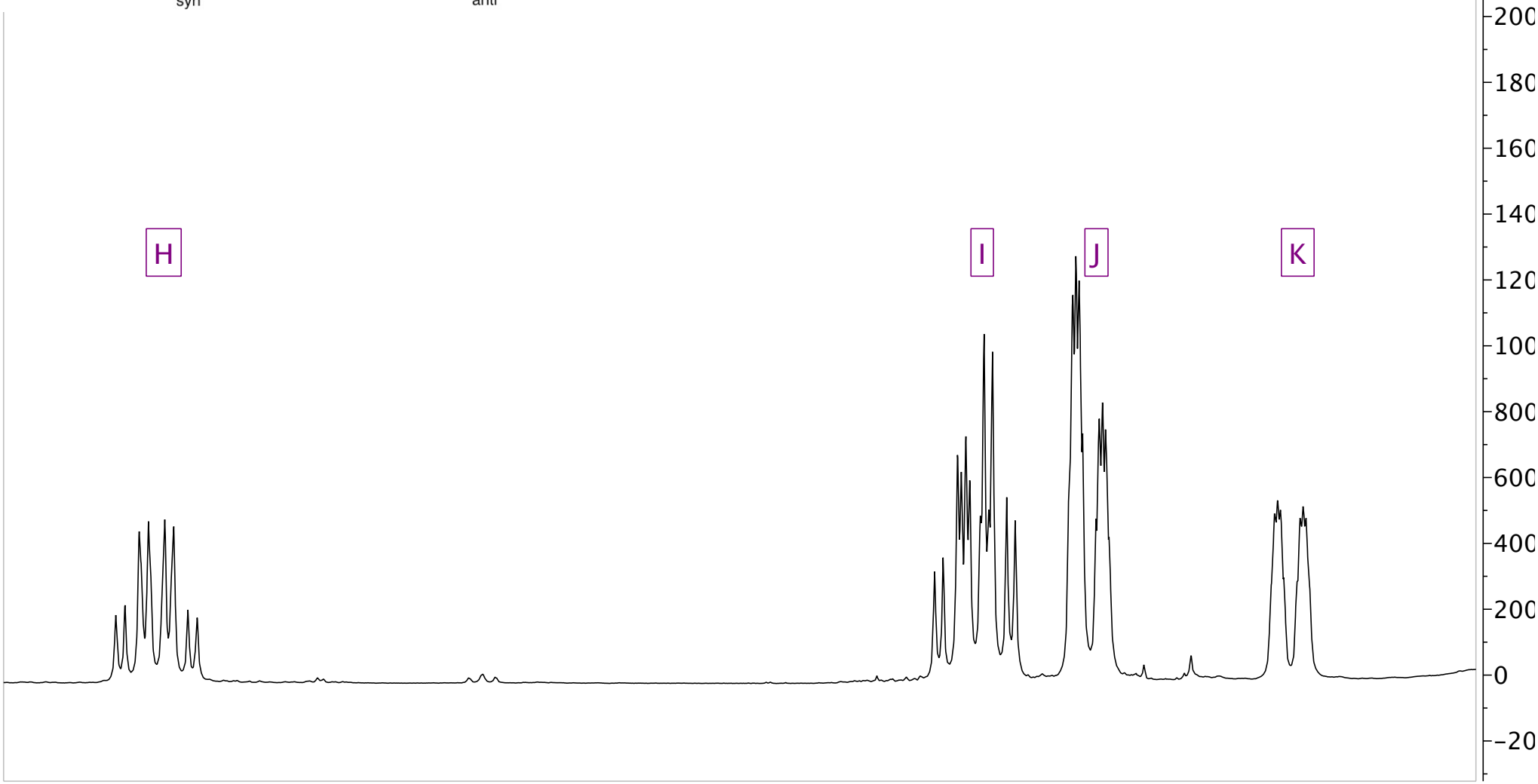

syn
anti
A
B
C
D
E
F
G
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
4.80
4.70
4.60
4.50
4.40
4.30
4.20
4.10
4.00
3.90
3.80
3.70
f1 (ppm)

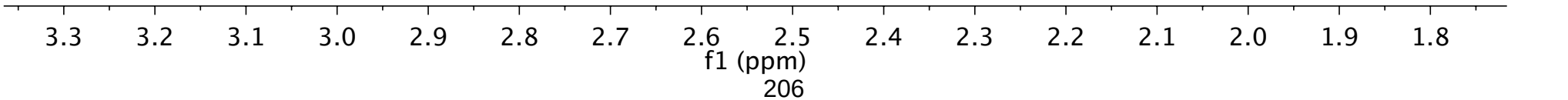

H_G
H_G
O
O
O
O
S
SO2Ph
N
H_D
H_F
H_H
H_A
H_B
H_K

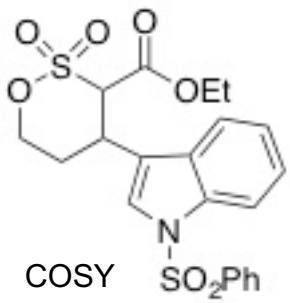
syn

H_G
H_G
O
O
O
O
S
SO2Ph
N
H_E
H_F
H_J
H_A
H_C
H_I

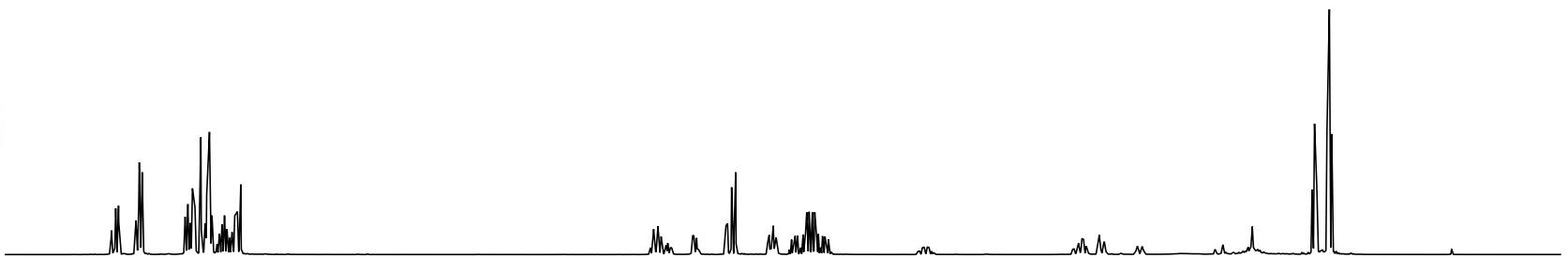
anti

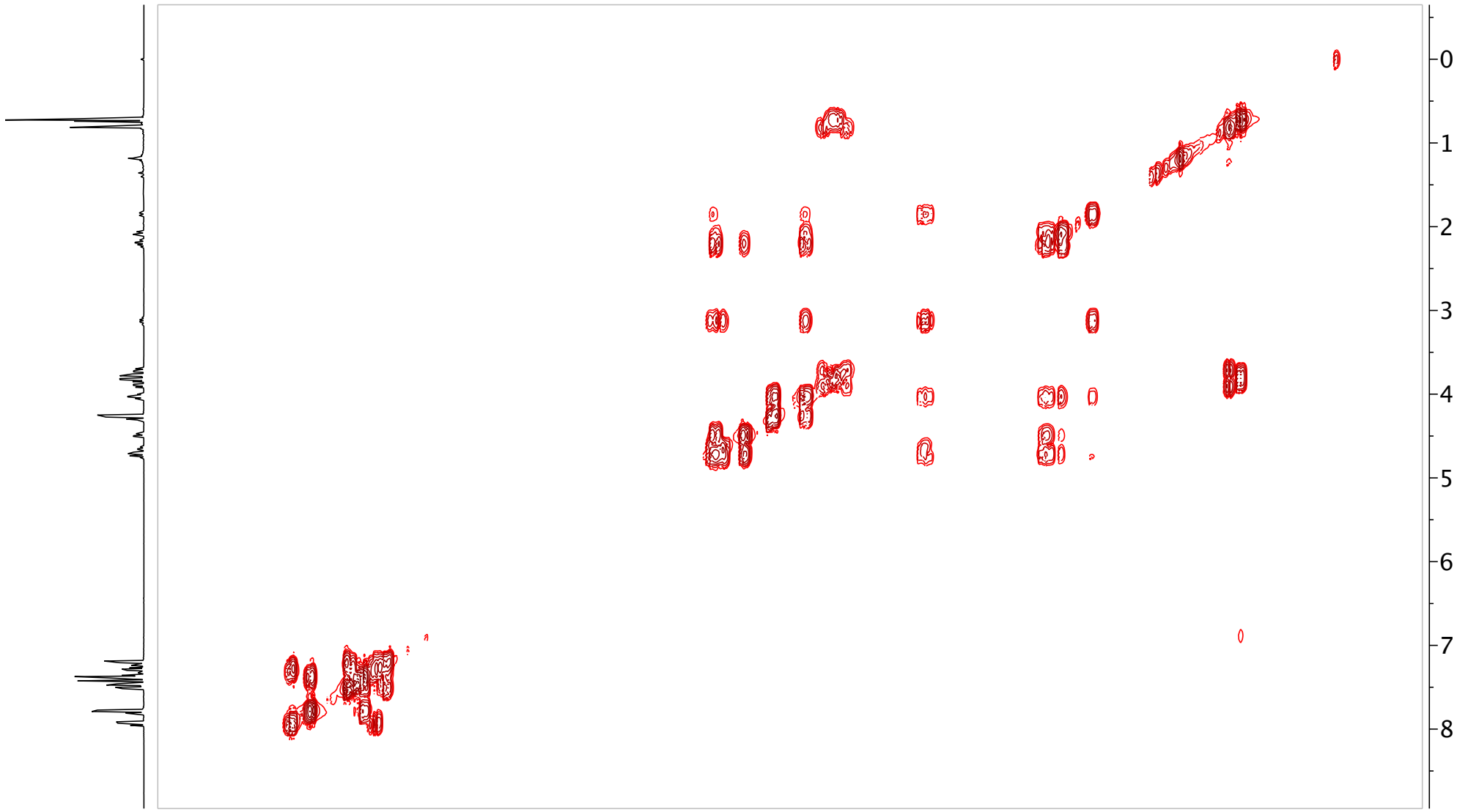

H
I
J
K

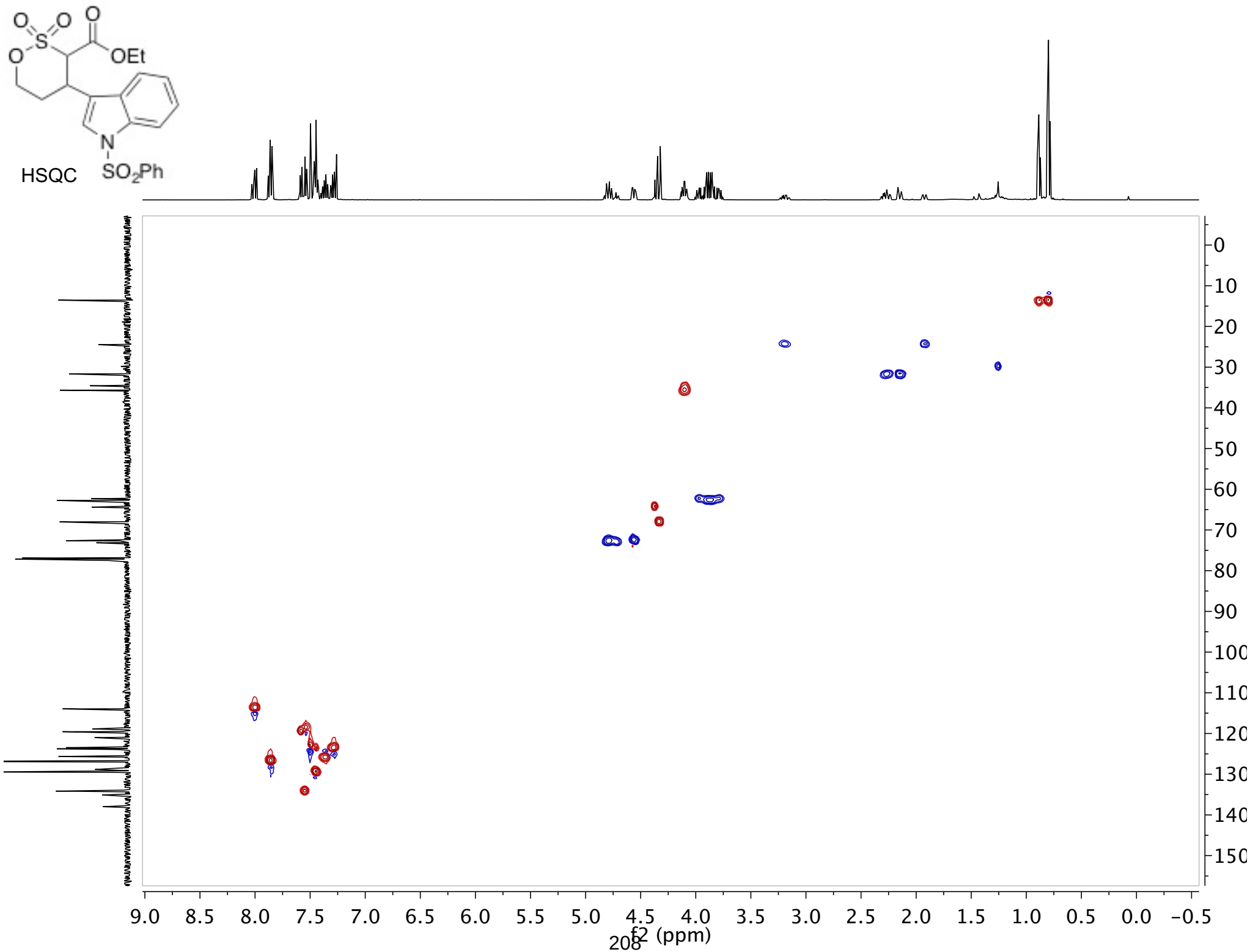

3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
f1 (ppm)

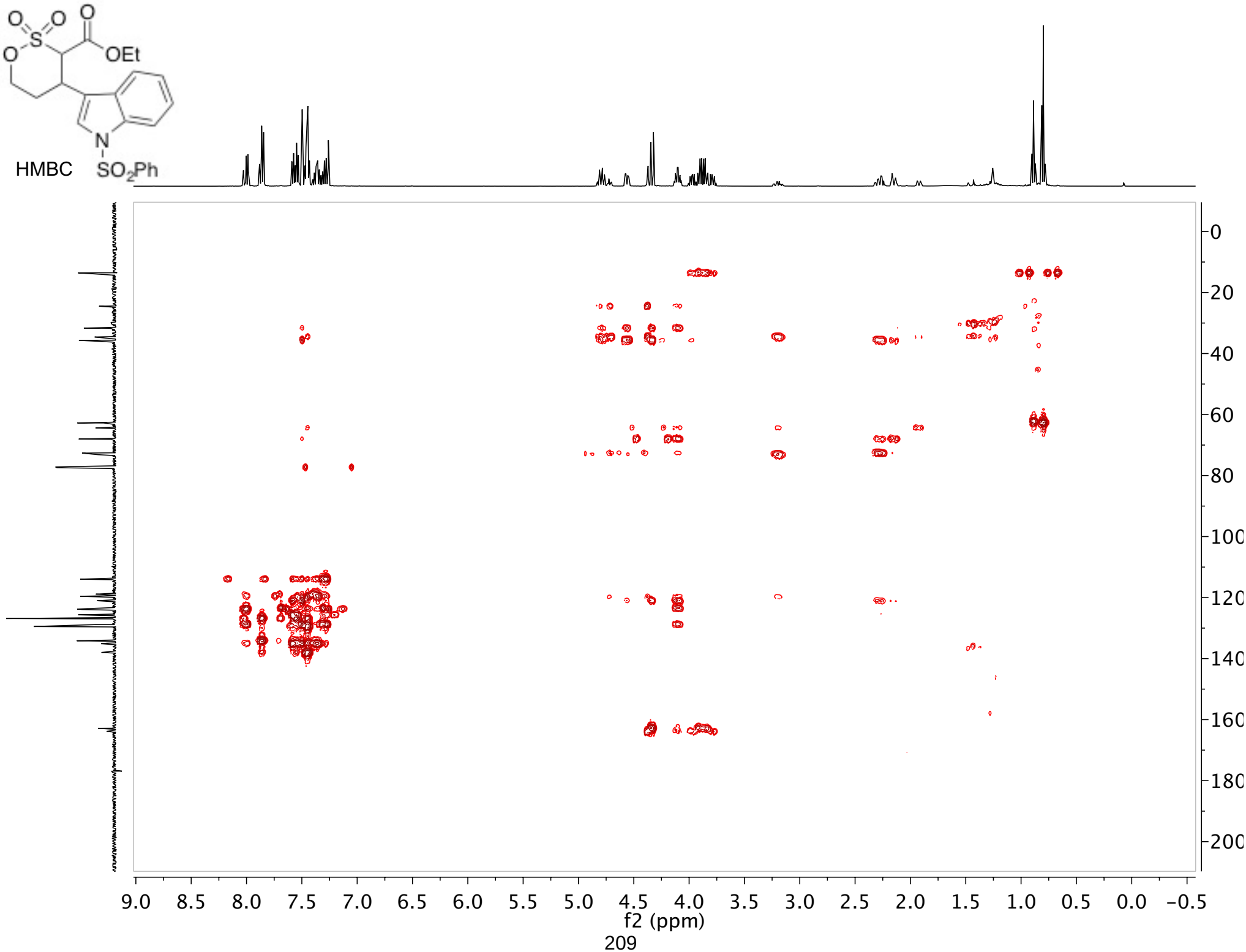

O
O
O
S
O
OEt
N
SO2Ph

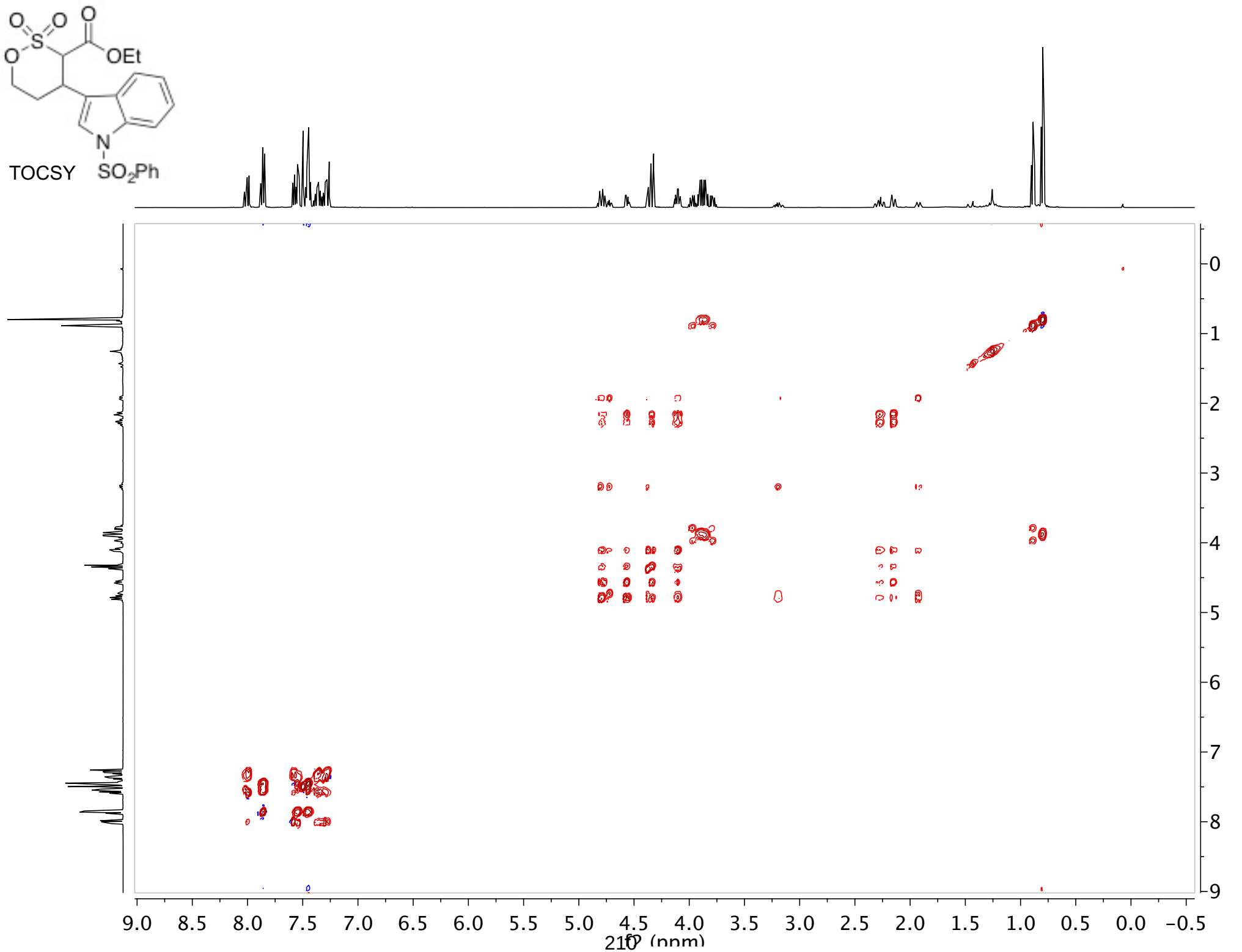
COSY

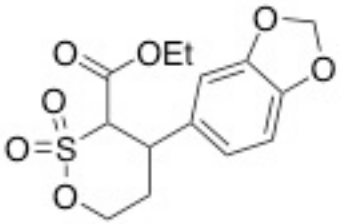



0
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f2 (ppm)


HSQC
OEt
N
SO2Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f2 (ppm)
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150


HMBC
SO2Ph
OEt
f2 (ppm)
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
0 20 40 60 80 100 120 140 160 180 200

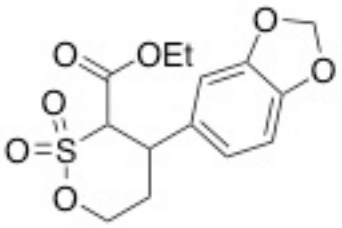

TOCSY
OEt
SO2Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f2 (ppm)
0 1 2 3 4 5 6 7 8 9



Syn diastereomer

7.89 Pc ligand
7.78 Pc ligand
2.05 EtOAc
1.54 H2O

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-07-04T09:24:17
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1519.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

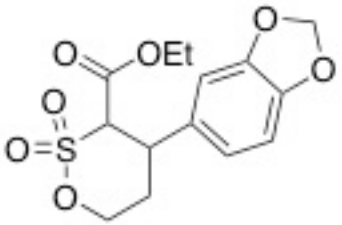


Syn diastereomer

—134.43 Pc ligand

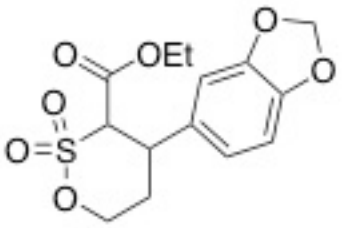
—123.69 Pc ligand

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	136
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-07-04T10:17:09
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



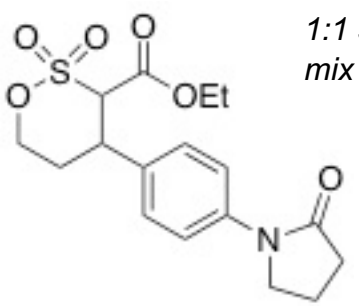
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-07-04T09:31:58
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1519.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Anti diastereomer

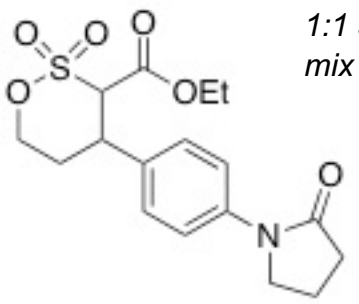
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	96
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-07-04T10:24:08
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



1:1 syn:anti
mix of diastereomers

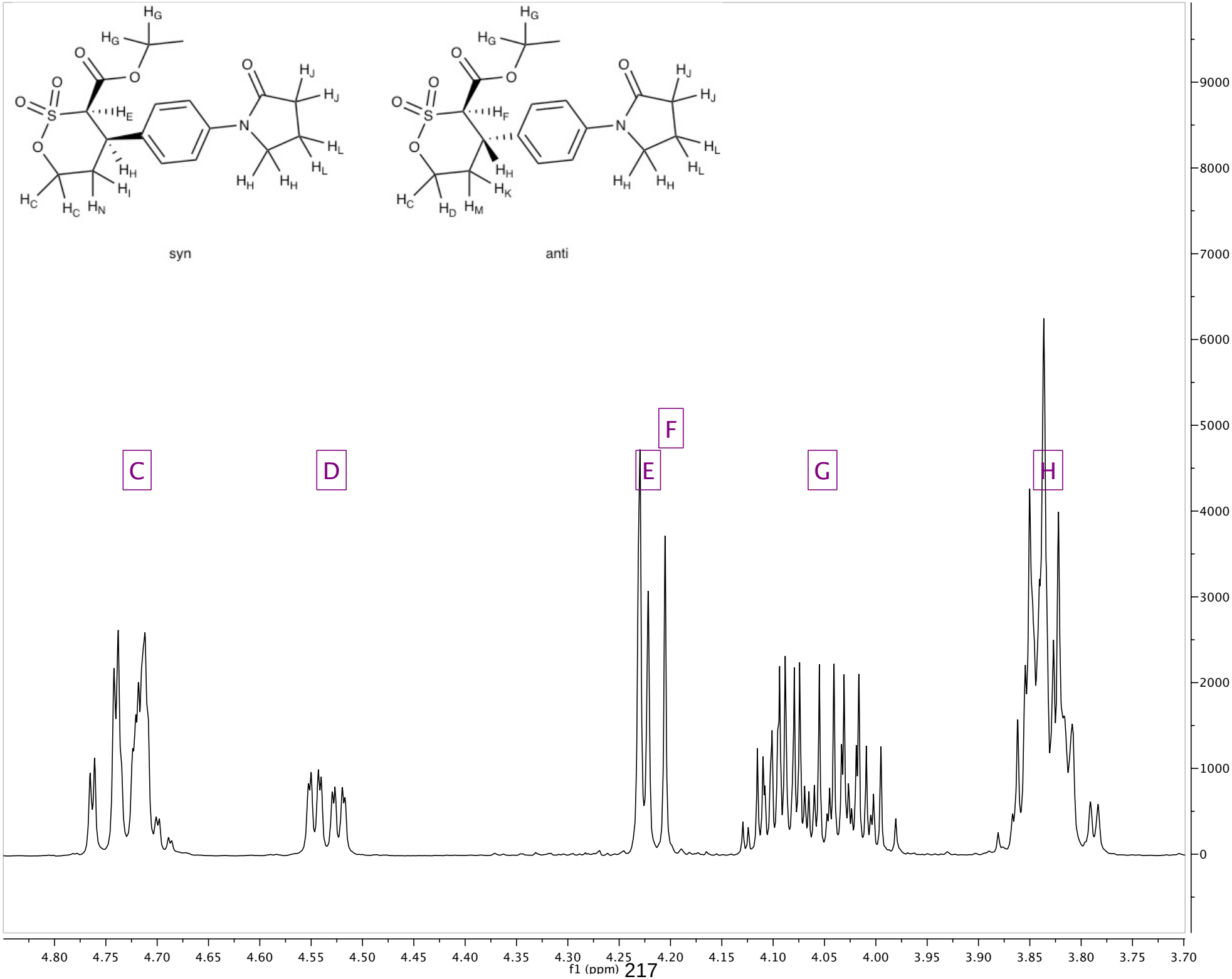
—5.28 CH_2Cl_2

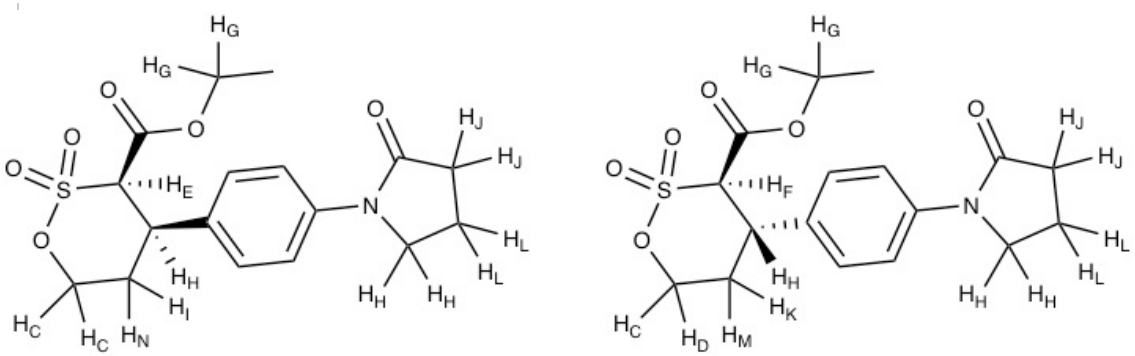
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	4.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-08-15T20:14:28
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1519.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



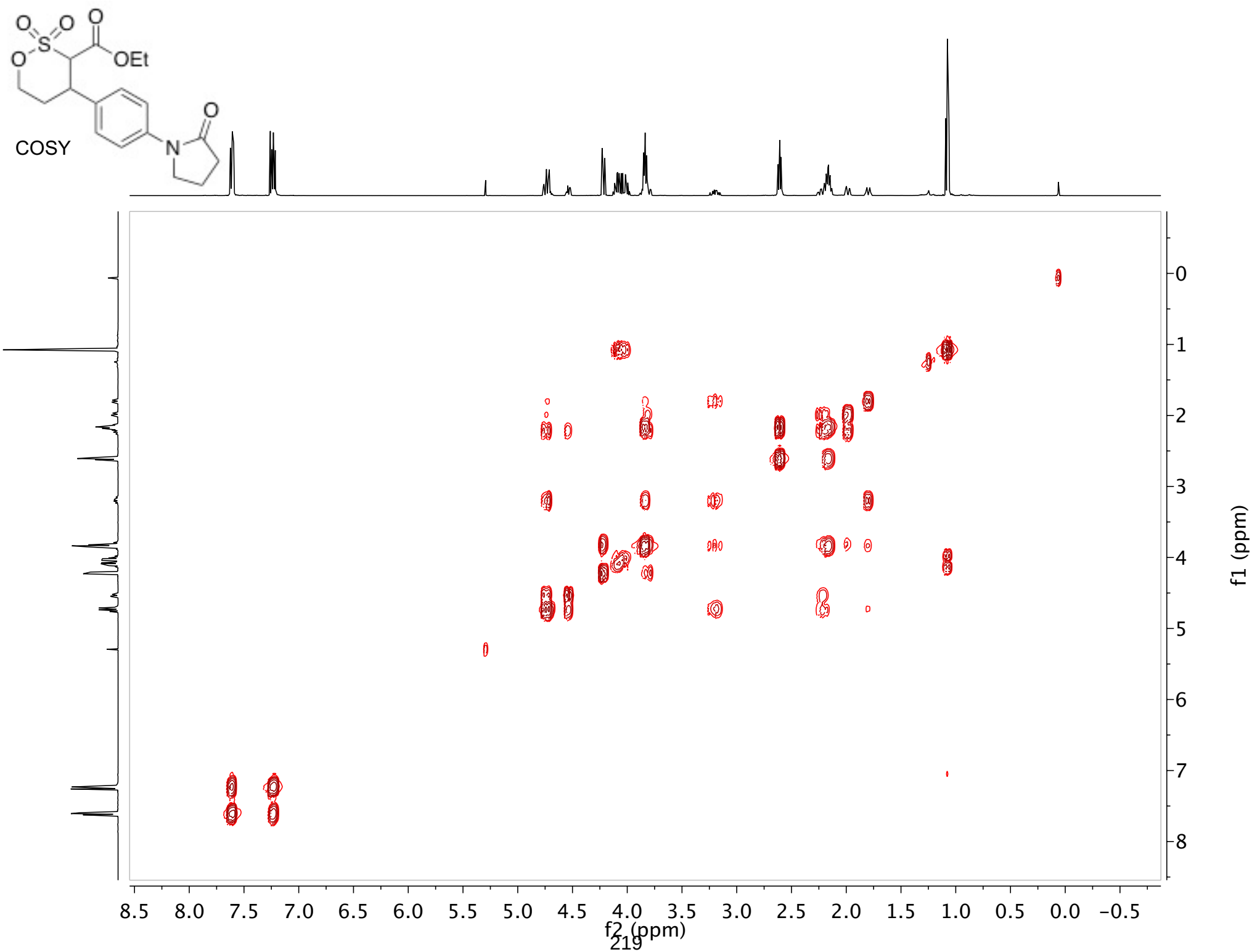
1:1 syn:anti
mix of diastereomers

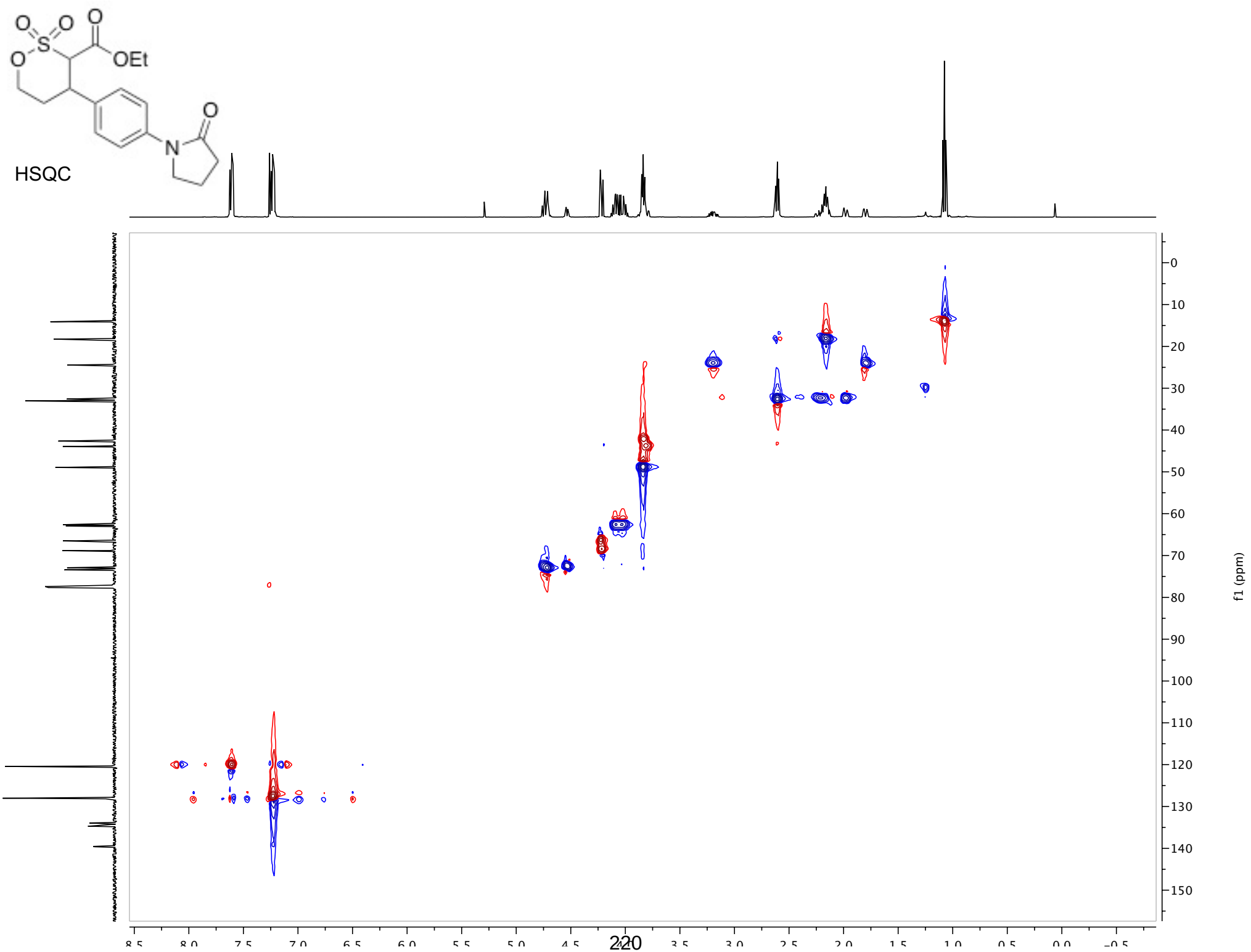
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	384
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-08-03T20:42:30
Class	
Spectrometer Frequency	125.60
Spectral Width	32000.0
Lowest Frequency	-2210.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

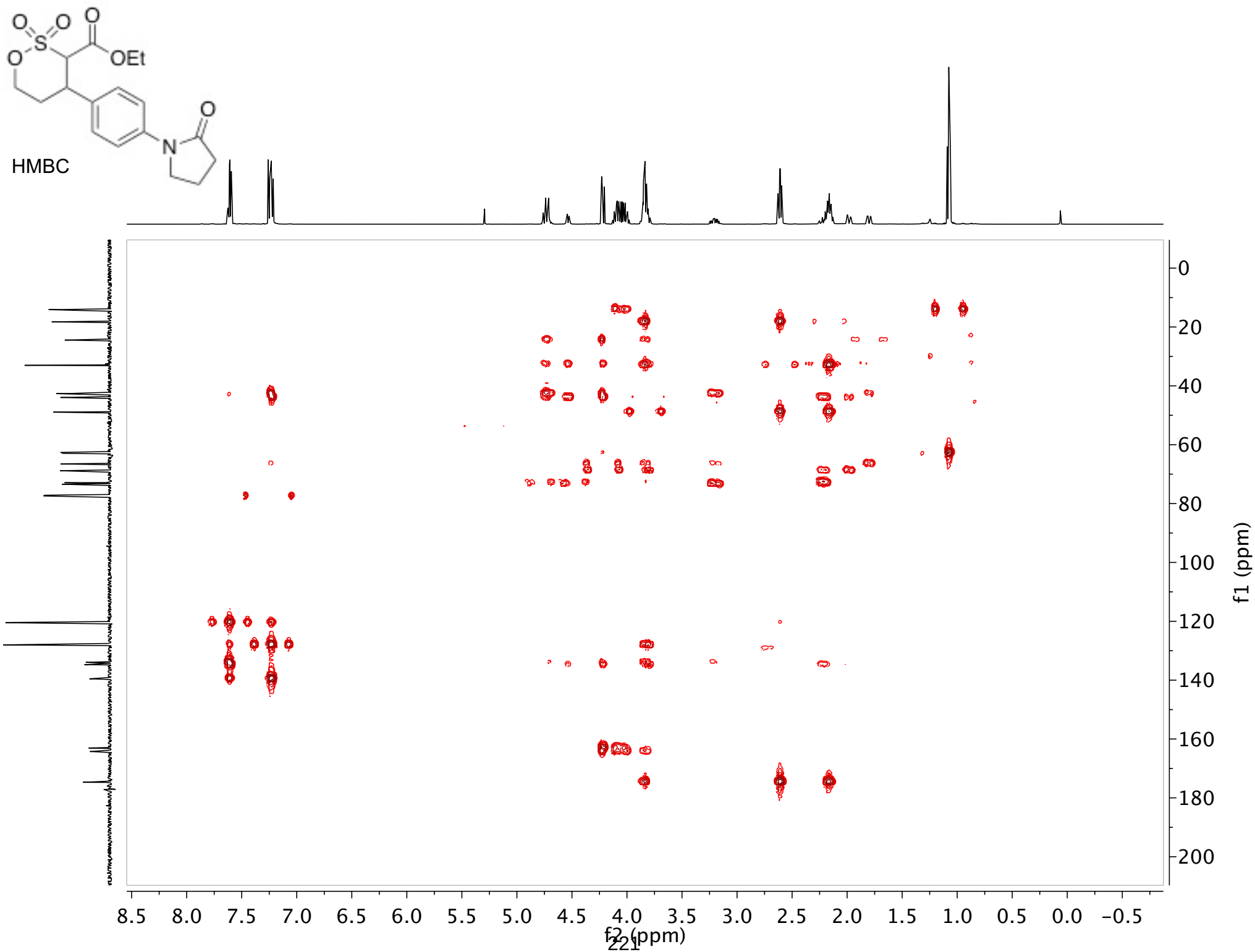

syn
anti
C
D
E
F
G
H
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
4.80
4.75
4.70
4.65
4.60
4.55
4.50
4.45
4.40
4.35
4.30
4.25
4.20
4.15
4.10
4.05
4.00
3.95
3.90
3.85
3.80
3.75
3.70
f1 (ppm)

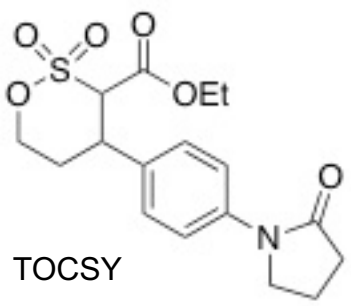

syn
anti

I
J
K
L
M
N
O
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
f1 (ppm)

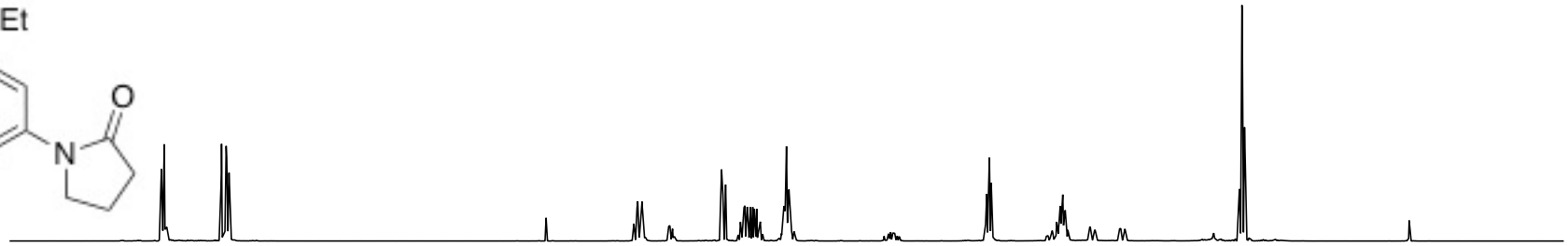

COSY
f2 (ppm)
f1 (ppm)
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
0 1 2 3 4 5 6 7 8

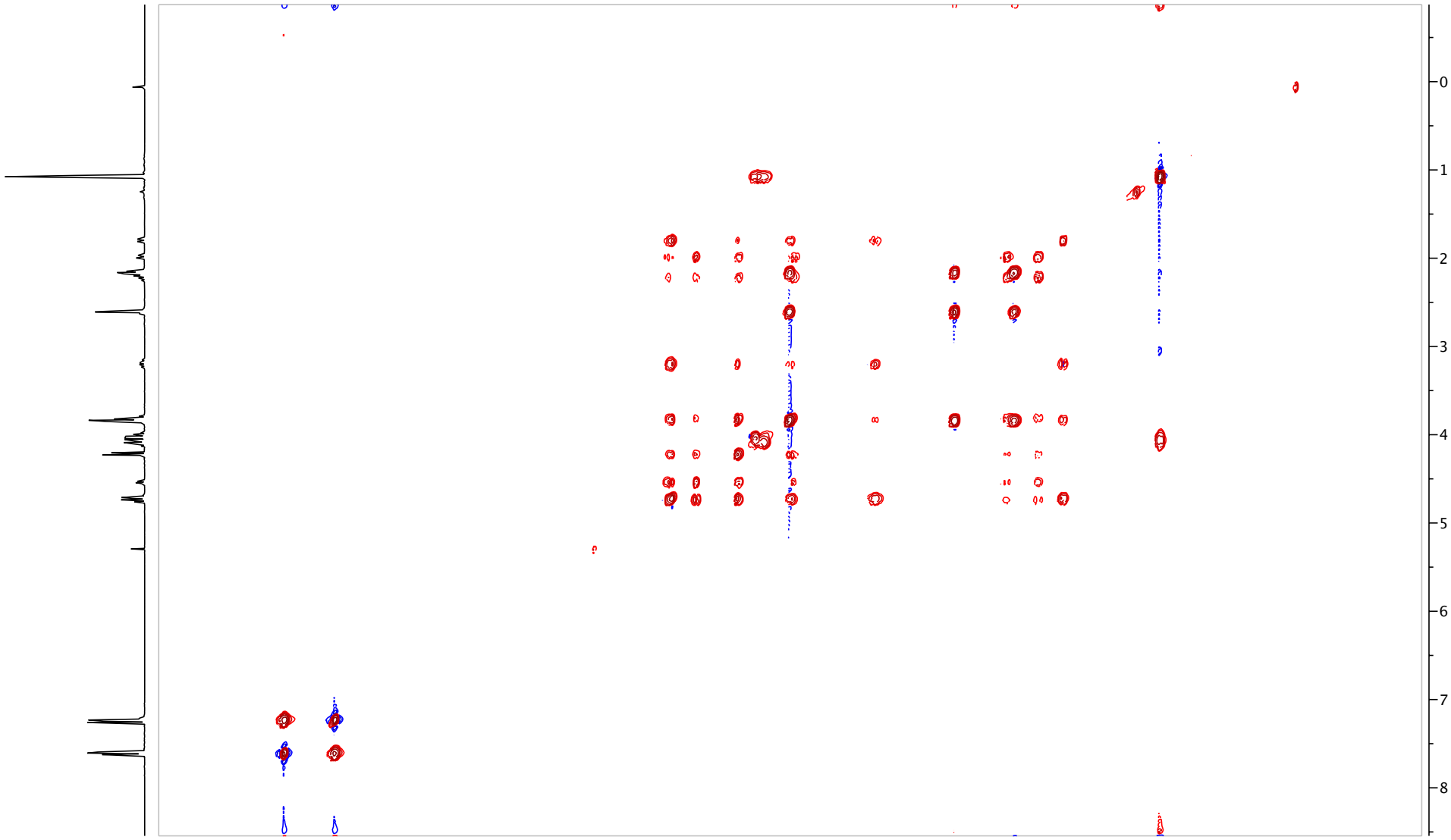

HSQC
f1 (ppm)

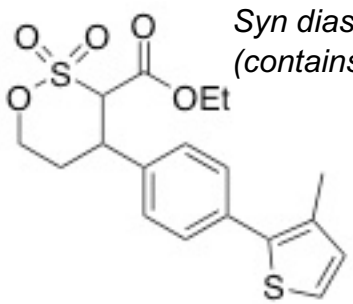

HMBC
f2 (ppm)
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
20
40
60
80
100
120
140
160
180
200


O
O
S
O
O
OEt
O
N

TOCSY

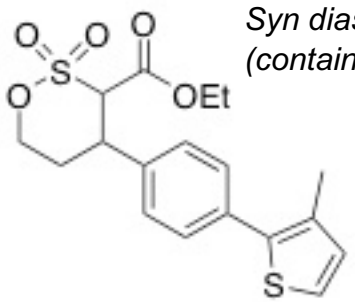



0
1
2
3
4
5
6
7
8
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f2 (ppm)



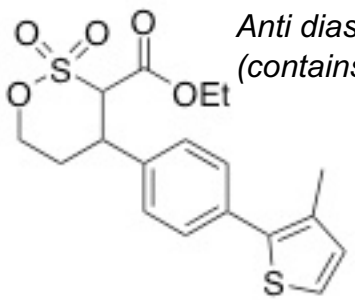
Syn diastereomer
(contains 10% anti)

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	128
Receiver Gain	123
Relaxation Delay	5.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-10-15T14:02:03
Modification Date	2016-10-15T14:02:02
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



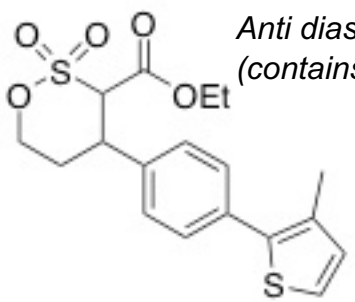
Syn diastereomer
(contains 10% anti)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	11432
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-08-10T07:51:08
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1273.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



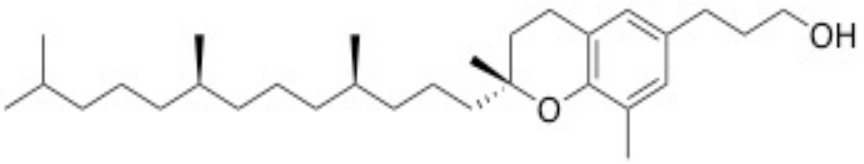
Anti diastereomer
(contains 5% syn)

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	86
Relaxation Delay	1.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-10-15T13:40:49
Modification Date	2016-10-15T13:40:48
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



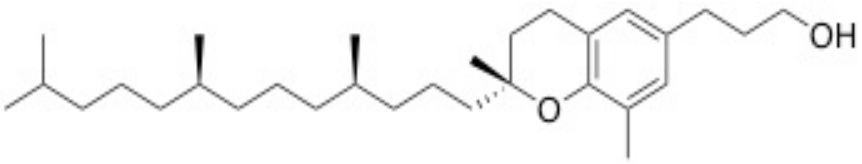
Anti diastereomer
(contains 5% syn)

Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-15T21:05:29
Modification Date	2016-10-15T21:05:28
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2282.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

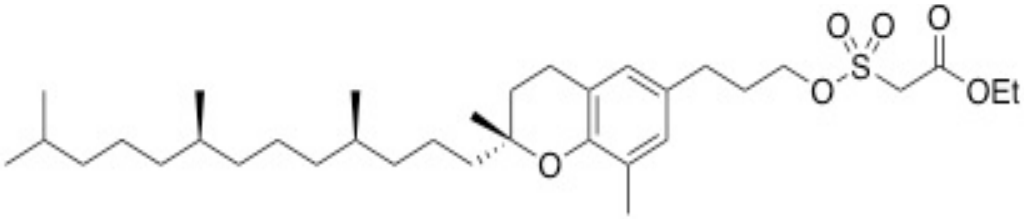


5.30 CH_2Cl_2

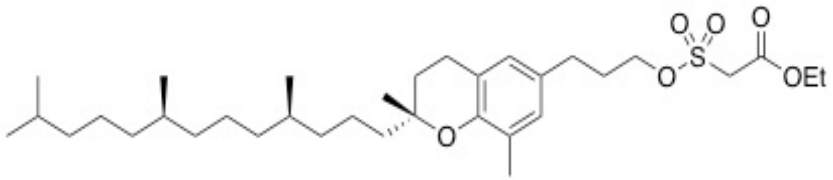
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	8
Receiver Gain	49
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2016-10-28T14:42:29
Modification Date	2016-10-28T14:42:28
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



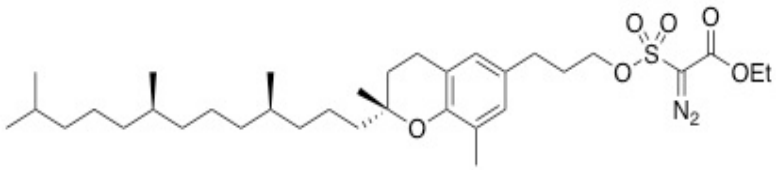
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	365
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-28T15:03:29
Modification Date	2016-10-28T15:03:28
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



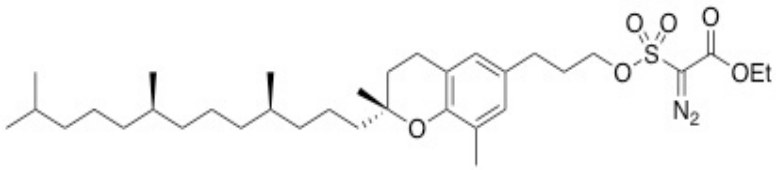
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	32
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-07-11T13:42:36
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1518.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



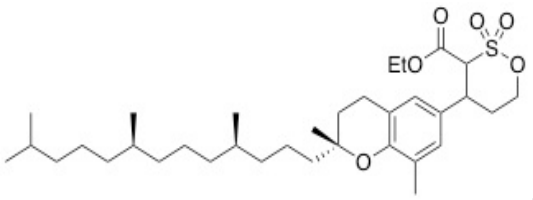
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	740
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-08-06T15:51:36
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	38
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-07-12T09:41:32
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

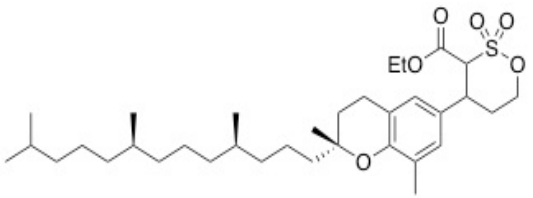


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	108
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-08-06T15:09:26
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



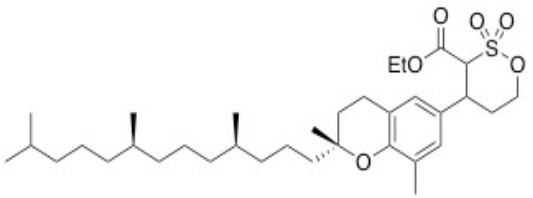
Syn diastereomer
(contains unknown impurity;
yield determined with internal standard)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	54
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-10-08T17:13:28
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



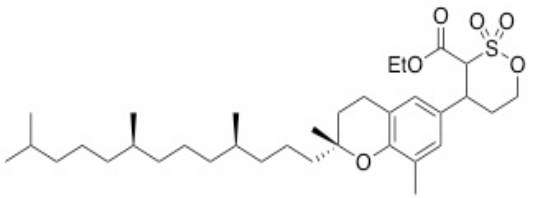
Syn diastereomer
(contains unknown impurity;
yield determined with internal standard)

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	20000
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-11-03T08:55:10
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



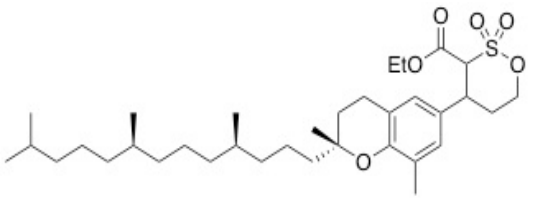
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	20000
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-11-03T08:55:10
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



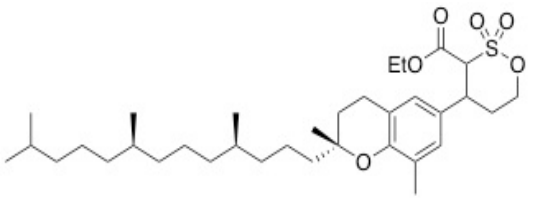
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	41
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-08-06T14:25:00
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1022.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



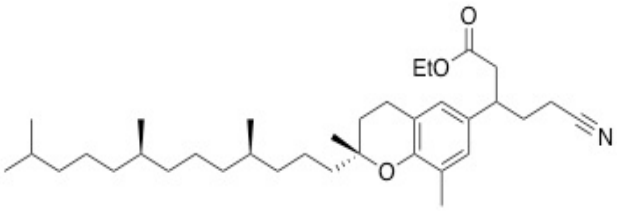
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	656
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-08-06T15:00:08
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



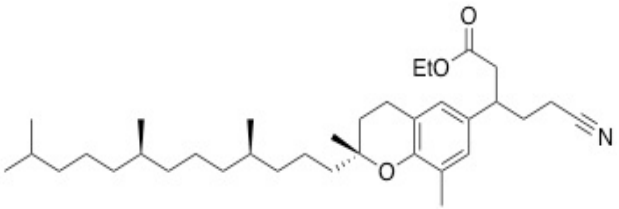
Anti diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	656
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-08-06T15:00:08
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

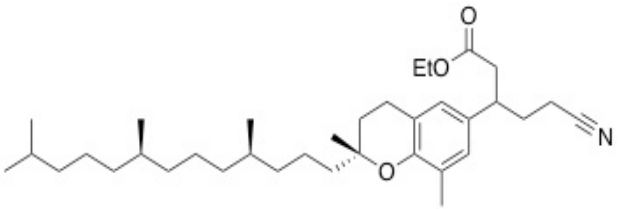


5.33 CH_2Cl_2

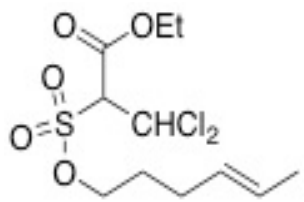
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	0.1
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	44
Relaxation Delay	10.0000
Pulse Width	0.0000
Acquisition Time	
Acquisition Date	
Modification Date	2016-10-21T10:14:14
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1502.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



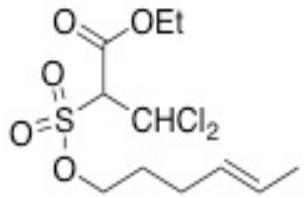
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	375
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-21T12:13:10
Modification Date	2016-10-21T12:13:10
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



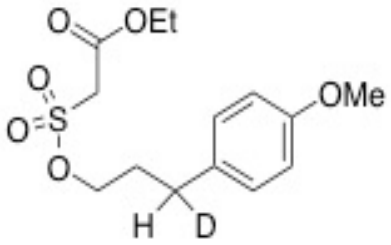
Parameter	Value
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	375
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2016-10-21T12:13:10
Modification Date	2016-10-21T12:13:10
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



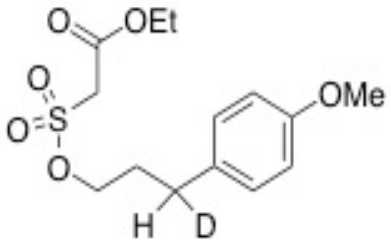
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	0.0000
Pulse Width	0.0000
Modification Date	2016-04-30T17:43:02
Class	
Spectrometer Frequency	499.43
Spectral Width	8000.0
Lowest Frequency	-1518.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



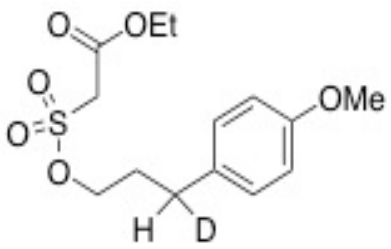
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	128
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-09T18:25:23
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



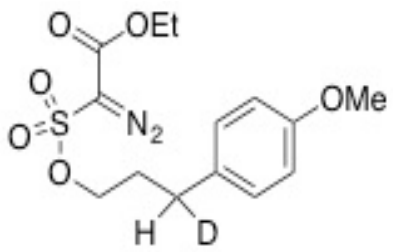
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-24T20:44:53
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



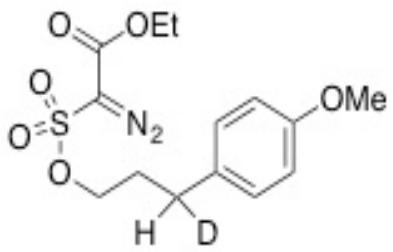
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-24T20:44:53
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



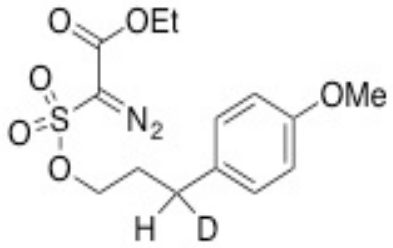
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	39
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-05-24T20:49:05
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1304.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



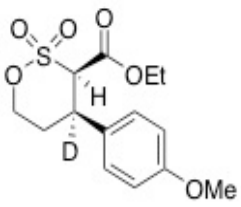
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-09T08:42:09
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



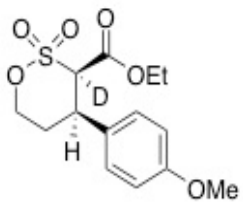
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-09T08:42:09
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1499.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	160
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-06-17T19:50:29
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1260.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

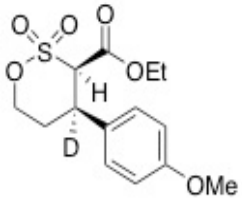


major isomer

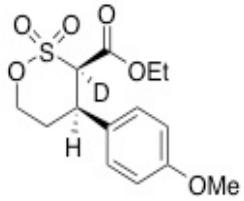


minor isomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	42
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-06-11T16:00:19
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1518.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

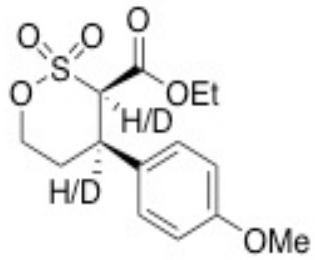


major isomer

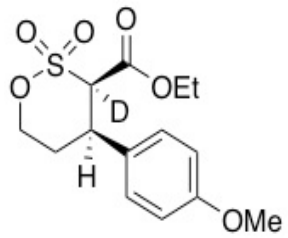


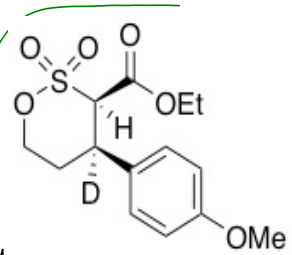
minor isomer

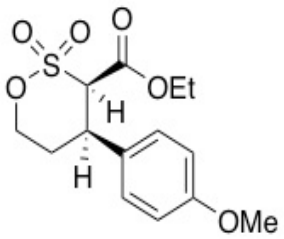
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	2944
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-06T07:52:30
Class	
Spectrometer Frequency	125.66
Spectral Width	22618.0
Lowest Frequency	-0.7
Nucleus	13C
Acquired Size	22618
Spectral Size	65536

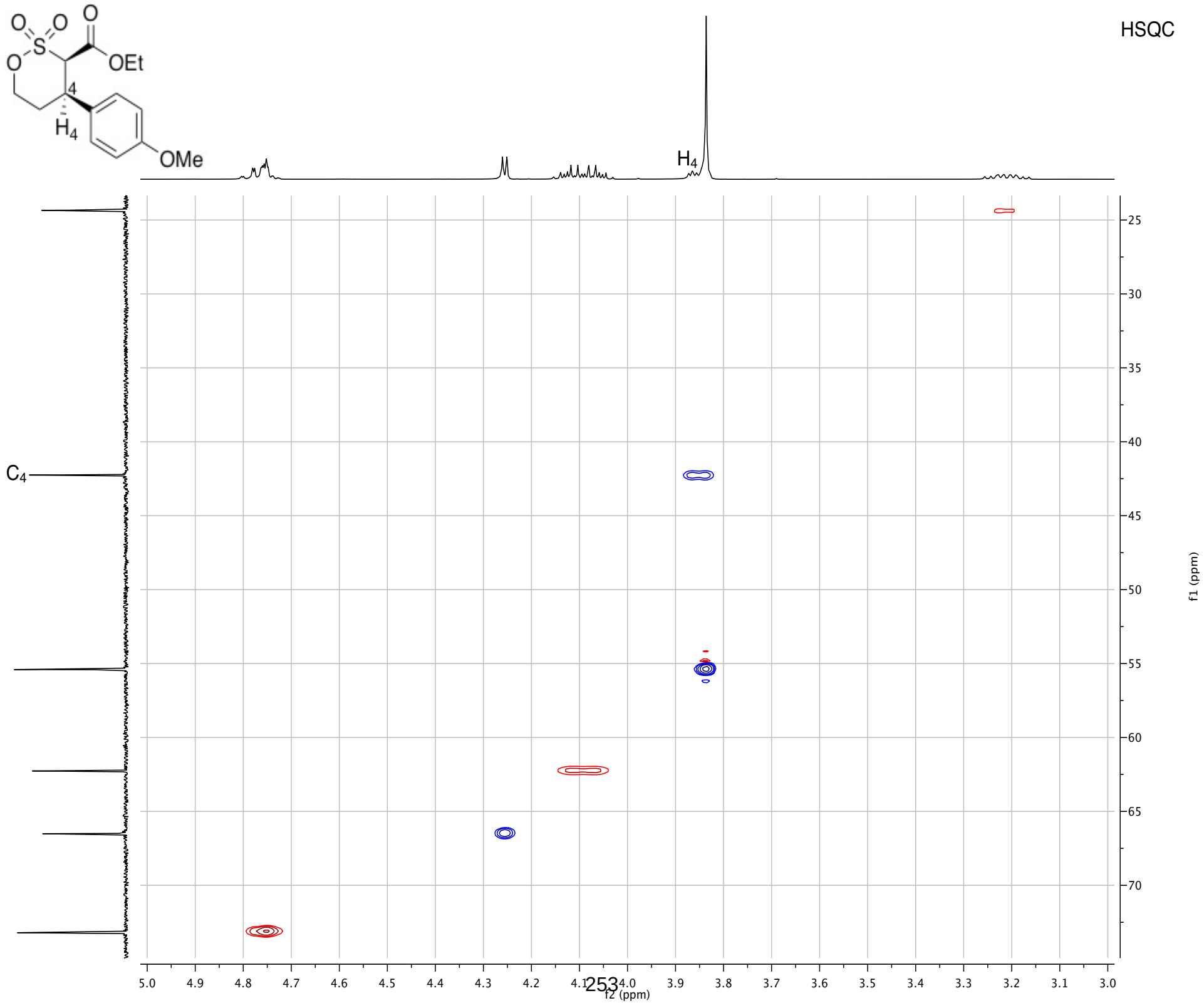


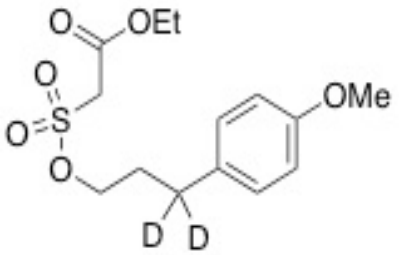
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	2944
Receiver Gain	60
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-05-06T07:52:30
Class	
Spectrometer Frequency	125.66
Spectral Width	22618.0
Lowest Frequency	-0.7
Nucleus	13C
Acquired Size	22618
Spectral Size	65536



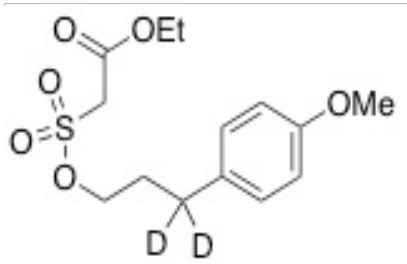




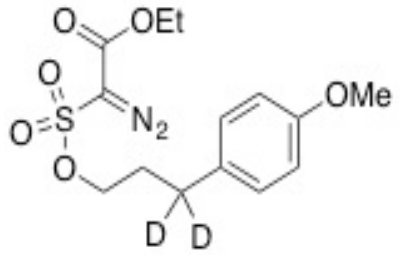

HSQC
OEt
OMe
4
H4
H4
C4
f2 (ppm)
f1 (ppm)
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
25
30
35
40
45
50
55
60
65
70



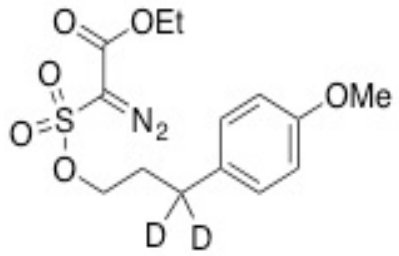
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	55
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2017-01-28T15:34:02
Modification Date	2017-01-28T15:34:02
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1922.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



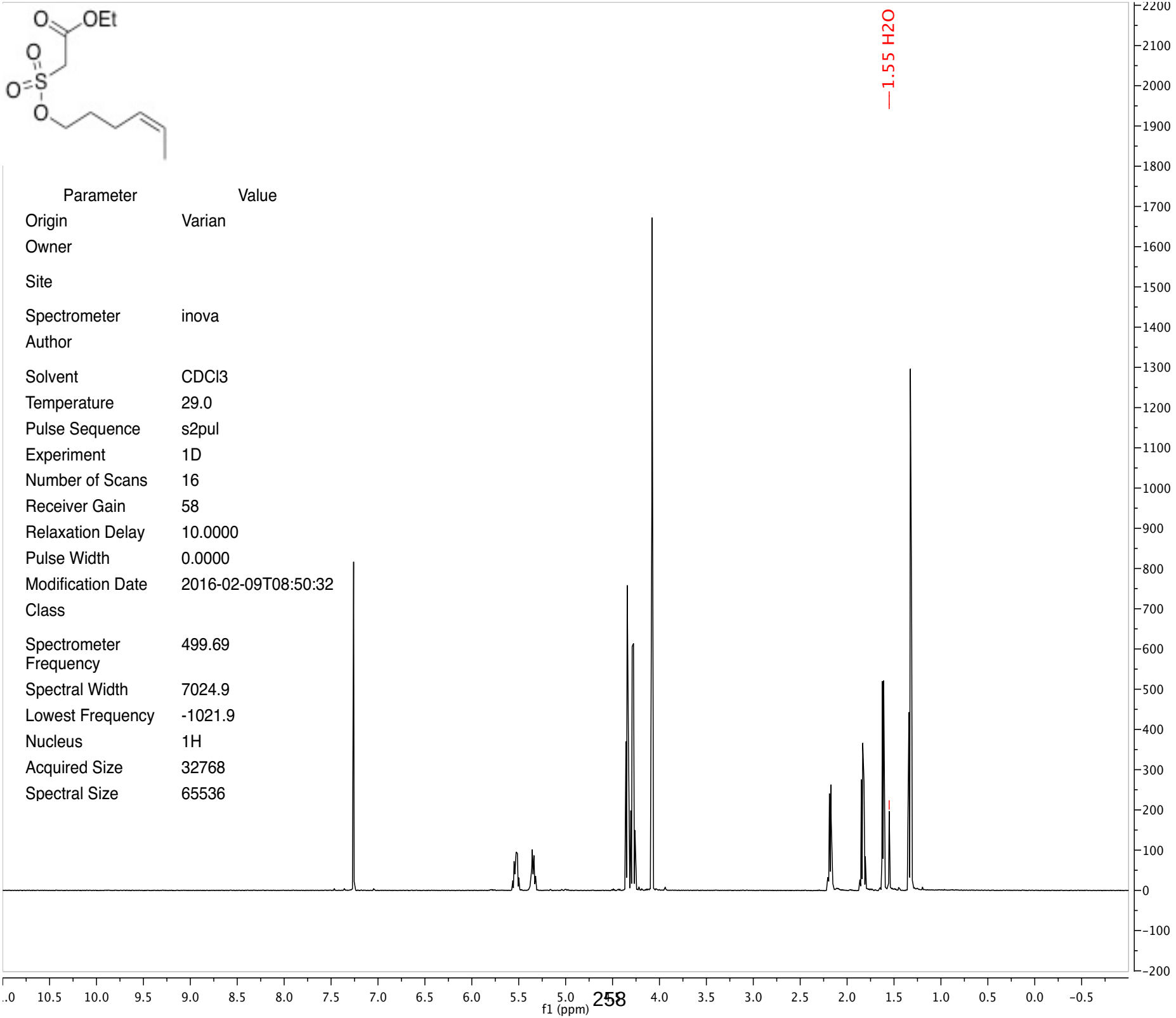
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2017-01-28T18:48:39
Modification Date	2017-01-28T18:48:39
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2282.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



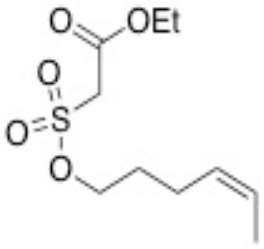
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	107
Relaxation Delay	10.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Acquisition Date	2017-01-28T15:40:30
Modification Date	2017-01-28T15:40:30
Class	
Spectrometer Frequency	500.35
Spectral Width	10000.0
Lowest Frequency	-1957.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



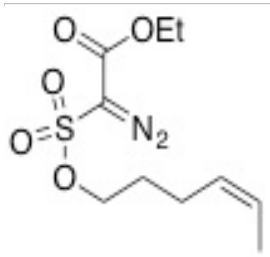
Parameter	Value
Comment	
Origin	Bruker BioSpin GmbH
Owner	user1d
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	191
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Acquisition Date	2017-01-28T20:33:23
Modification Date	2017-01-28T20:33:23
Class	
Spectrometer Frequency	125.83
Spectral Width	29761.9
Lowest Frequency	-2299.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



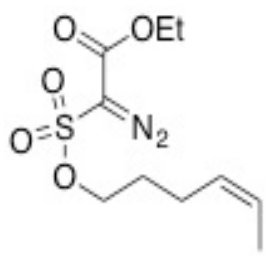
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	29.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	58
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-02-09T08:50:32
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



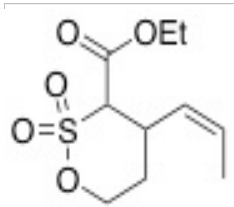
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	51
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-02-09T08:42:08
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1287.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	29.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	56
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-02-13T16:03:30
Class	
Spectrometer Frequency	499.69
Spectral Width	7024.9
Lowest Frequency	-1021.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

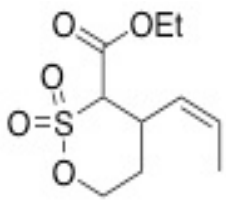


Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	270
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-02-13T16:18:59
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



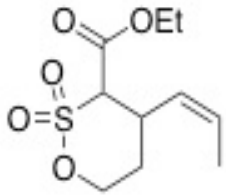
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	32
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-02-04T11:28:11
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



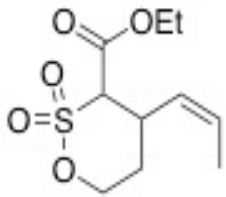
Syn diastereomer

Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	324
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-02-03T19:10:55
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1276.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



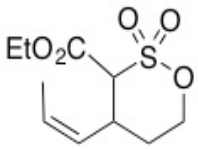
Anti diastereomer

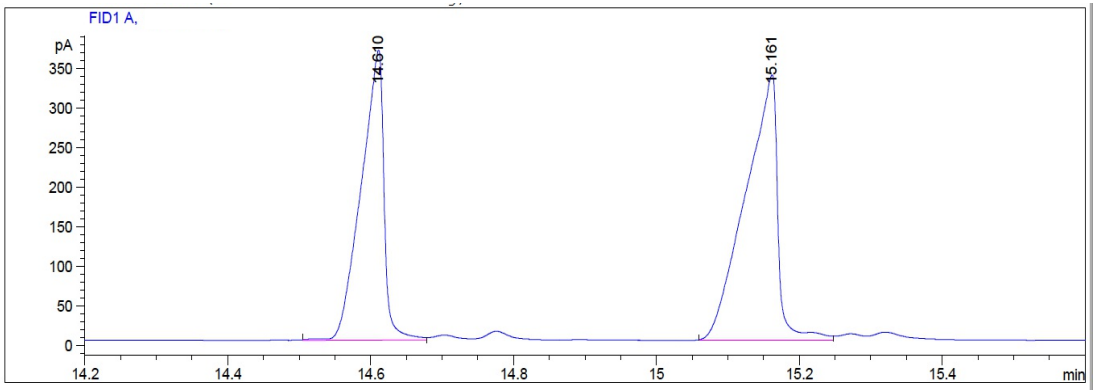
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	38
Relaxation Delay	10.0000
Pulse Width	0.0000
Modification Date	2016-02-04T11:41:41
Class	
Spectrometer Frequency	500.07
Spectral Width	8000.0
Lowest Frequency	-1520.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



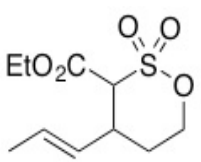
Anti diastereomer

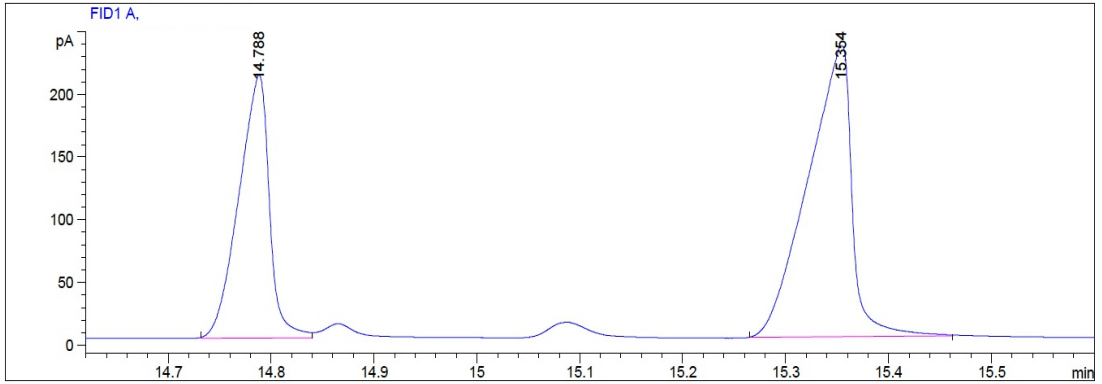
Parameter	Value
Origin	Varian
Owner	
Site	
Spectrometer	inova
Author	
Solvent	CDCl3
Temperature	22.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	322
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	0.0000
Modification Date	2016-02-03T19:30:10
Class	
Spectrometer Frequency	125.66
Spectral Width	30165.9
Lowest Frequency	-1274.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



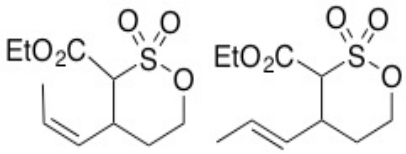


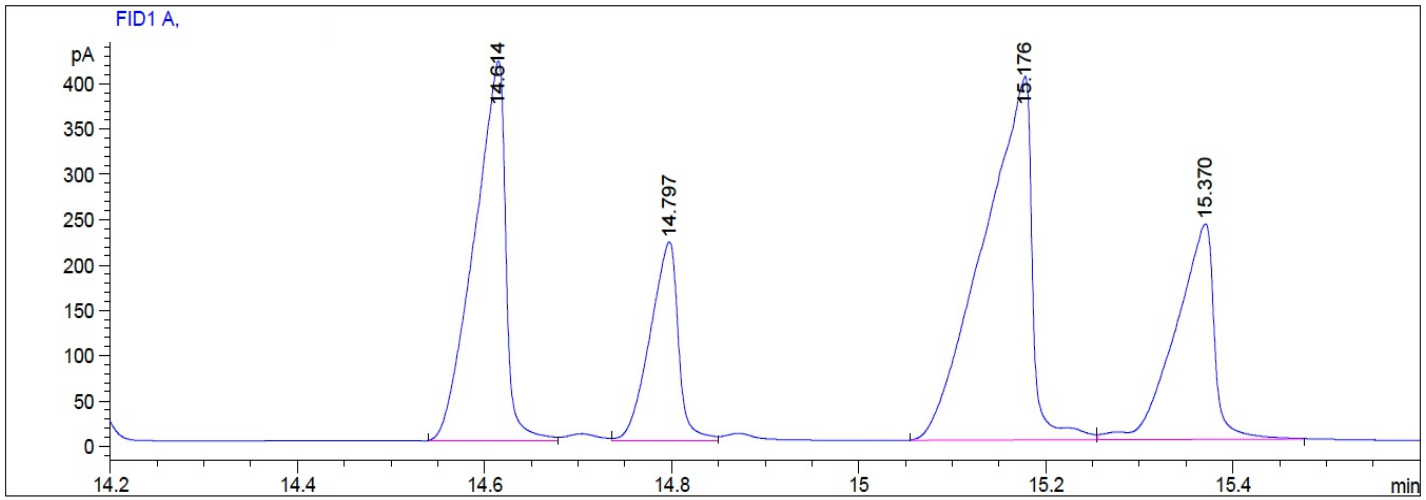

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  14.610 BV    0.0327  815.65625  361.64508 43.45029
   2  15.161 BV    0.0410 1061.56091  335.15915 56.54971
```





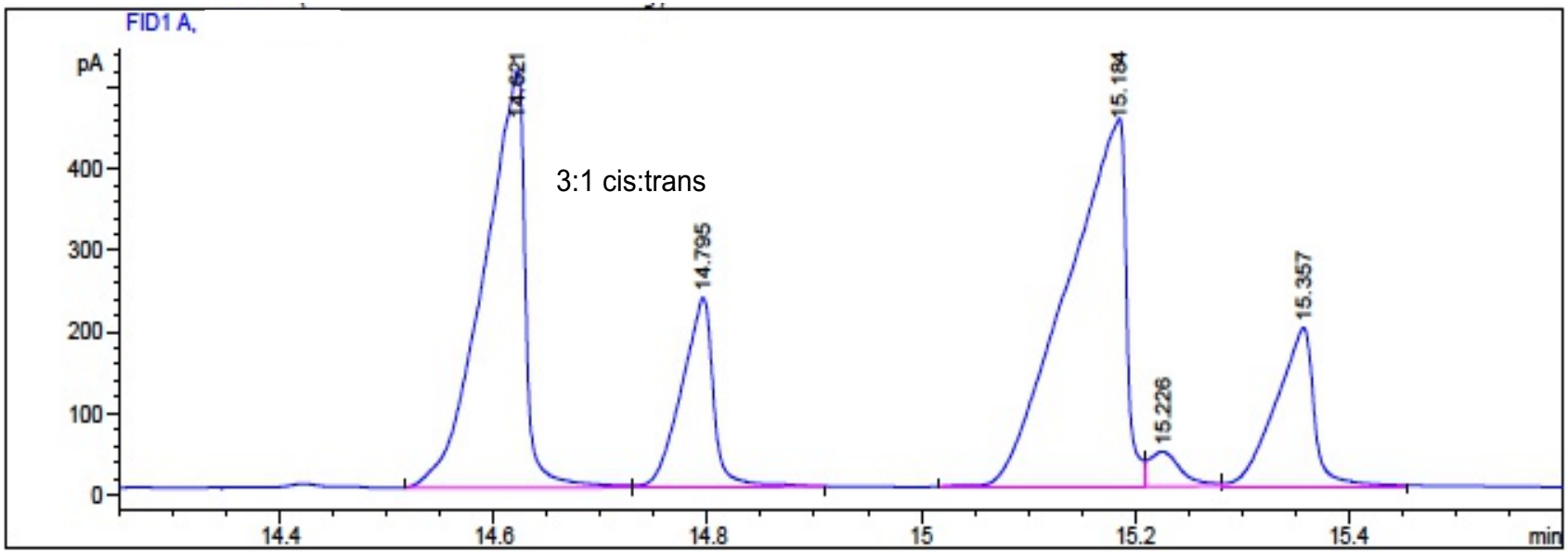

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  14.788 BV    0.0314  433.25272  209.27066 38.51358
   2  15.354 BB    0.0413  691.68225  231.35529 61.48642
```






```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  14.614 BV    0.0346  984.93317  418.73083 27.44760
   2  14.797 VV    0.0327  460.01556  217.36447 12.81947
   3  15.176 BV    0.0473 1456.88623  400.36572 40.59974
   4  15.370 VB    0.0411  686.57800  236.48363 19.13319
```

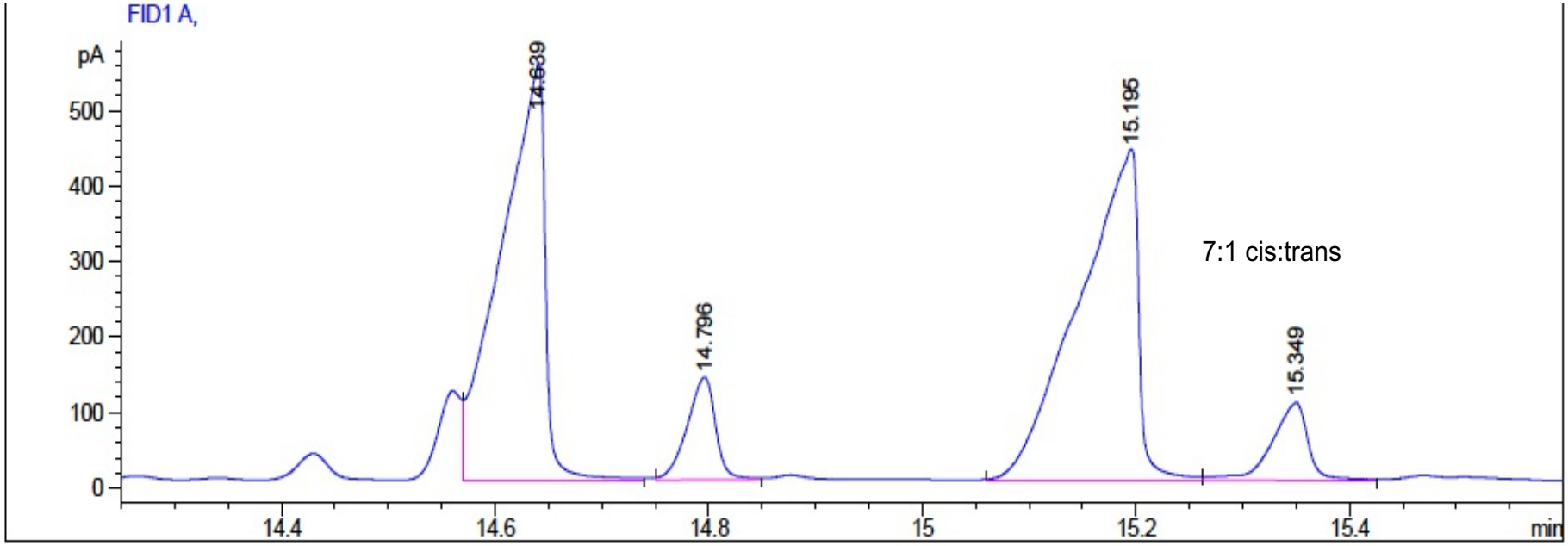
FePc




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  14.621 BV    0.0362 1394.29211  505.79163 33.22466
   2  14.795 VB    0.0301  488.75397  232.54837 11.64654
   3  15.184 BV    0.0517 1737.17041  455.29980 41.39512
   4  15.226 VV    0.0308   88.91061   42.59782  2.11866
   5  15.357 VB    0.0347  487.43134  195.26775 11.61503
```

3:1 cis:trans

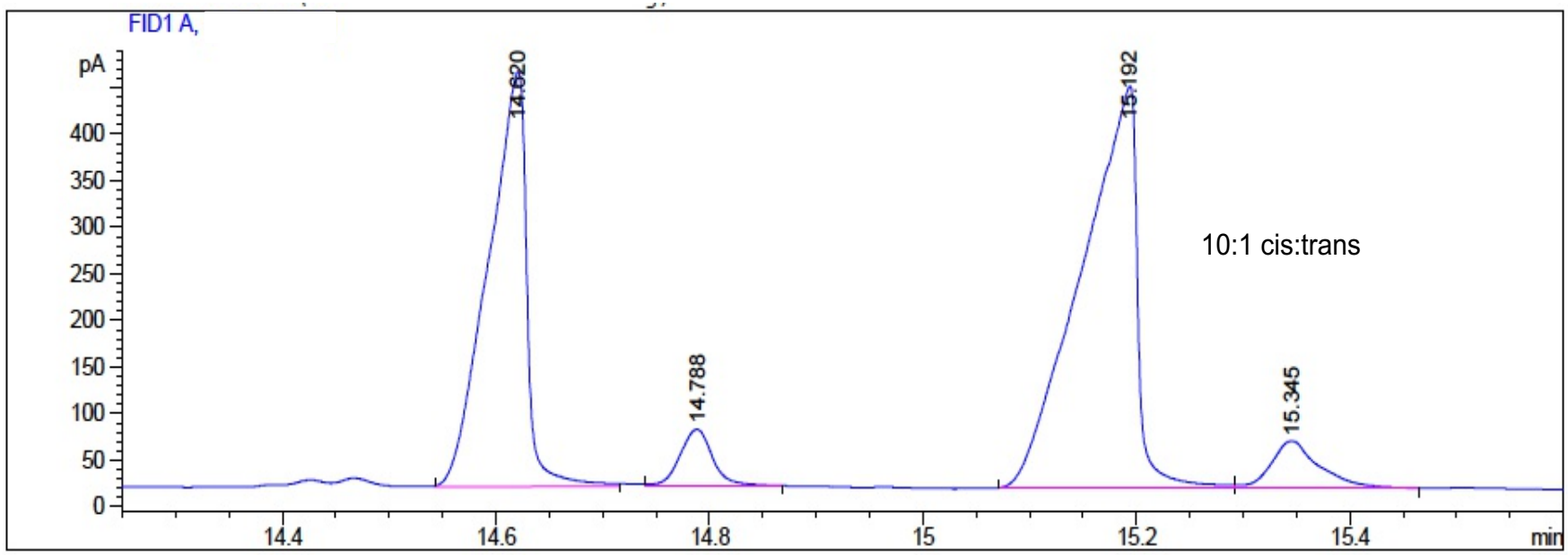
$FeCl_8Pc$




```
================================================================================
                              Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  14.639 VB    0.0388 1583.67676  543.25024 41.92070
   2  14.796 BV    0.0281  252.20367  135.67679  6.67595
   3  15.195 BV    0.0559 1694.44336  434.29486 44.85275
   4  15.349 VB    0.0366  247.46797  103.82152  6.55060
```

7:1 cis:trans

$FeCl_{16}Pc$




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  14.620 BB    0.0351 1122.50439  444.52274 37.48800
   2  14.788 BB    0.0342  133.54158   61.25869  4.45985
   3  15.192 PB    0.0490 1579.35278  432.41891 52.74525
   4  15.345 BP    0.0439  158.90454   50.52629  5.30690
```

10:1 cis:trans