Supporting Information

Copper Catalyzed C(sp^3)-H Bond Alkylation via Photoinduced Ligand to Metal Charge Transfer

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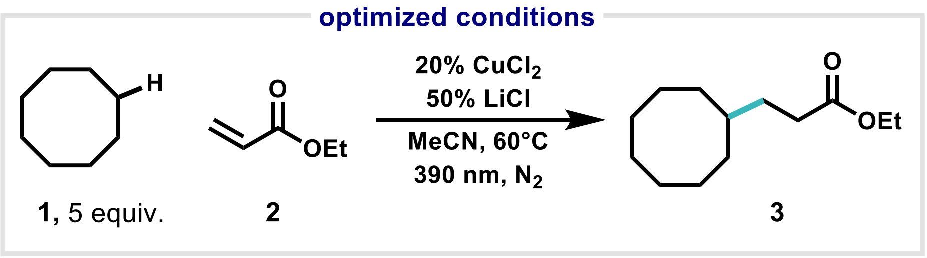
Materials and Methods:

Unless otherwise noted, all reactions were performed in oven-dried glassware and carried out under an atmosphere of nitrogen with magnetic stirring. All photochemical reactions were run in 1.0 dram vials fitted with Teflon caps under irradiation from a PR-160 Kessil 40W LED lamp with Teflon stir-bars under vigorous magnetic stirring. All photochemical reactions were set-up in a nitrogen glovebox, though can also be performed with suitable Schlenk-line techniques. All column chromatography was performed using a Teledyne Isco Combiflash using CombiFlash pre-packed columns outfitted with an ELSD detector. As most of the compounds listed do not exhibit an UV trace, ELSD was integral to the separation of product while thin layer chromatography was performed on SiliCycle® 250 μm 60 Å plates. Visualization was accomplished with 254 nm UV light, Seebach's stain, or I_2.

^{1}H NMR spectra were recorded on Bruker 400 or 500 MHz spectrometers at ambient temperature. Chemical shift is reported in parts per million (ppm) from $CDCl_3$ (7.26 ppm) with multiplicity (s = singlet, bs = broad singlet, d = doublet, t = triplet, q = quartet, and m = multiplet) and coupling constants (Hz). ^{13}C NMR was recorded on Bruker 500 or 400 MHz spectrometers (126 MHz) at ambient temperature. Chemical shifts are reported in ppm from $CDCl_3$ (77.2 ppm). Mass spectra were recorded on an Agilent 7890B GC System 5977B MSD GCMS with an EI ionization method. High resolution mass spectra (HRMS) were obtained from the Columbia University Chemistry Department Mass Spectrometry Facility on a Waters XEVO G2XS QToF mass spectrometer equipped with a UPC2 SFC inlet and a LockSpray source with one of the following three probes: electrospray ionization (ESI) probe, atmospheric pressure chemical ionization (APCI) probe, or atmospheric pressure solids analysis probe (ASAP). Infrared spectra were collected on a Perkin Elmer Spectrum Two FT-IR Spectrometer.

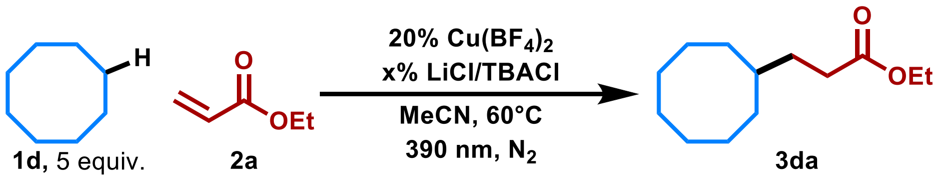
Unless otherwise mentioned, all starting materials were obtained from commercial sources including Millipore-Sigma, TCI, and Alfa-Aesar. Anhydrous $CuCl_2$, anhydrous LiCl, and anhydrous acetonitrile were obtained from Millipore-Sigma.

Table S1. Extended Optimization Studies:



Entry	Deviation from Standard Conditions	Yield 3 (%)
1	none	93
2	3 equiv. cyclooctane	73
3	0% $CuCl_2$	0
4	0% LiCl	79
5	in the dark	0
6	440 nm LED	66
7	30°C	45
8	under air	55
9	with 10% Cs_2CO_3	22
10	Irradiated 1 hour, then in the dark	14
11	3 mmol scale (10x)	86
12	1 equiv. cyclooctane	44

Table S2. Identifying the Catalytically Active Species: Conditions correspond to experiments conducted by Mereshchenko *et. al.*[1] Photochemical studies conducted by these authors reveal that both $CuCl^+$ and $CuCl_3^-$ are capable of photoreduction at varying wavelengths with quantum yields of ~6.0% and ~1.5 % respectively.



Entry	% TBACl	Catalyst Formed In-situ	Yield 3da (%, GC)
1	**10**	**$CuCl^+$**	0
2	**60**	**$CuCl_3^-$**	78
3	**200**	**$CuCl_4^{2-}$**	0

Figure S1. Absorbance spectrum of 60 μM $CuCl_2$ and 150 μM solution of LiCl in MeCN. This is 1000x dilution from catalytic conditions. The spectrum matches the spectrum produced by Mereshchenko *et. al.*[1]

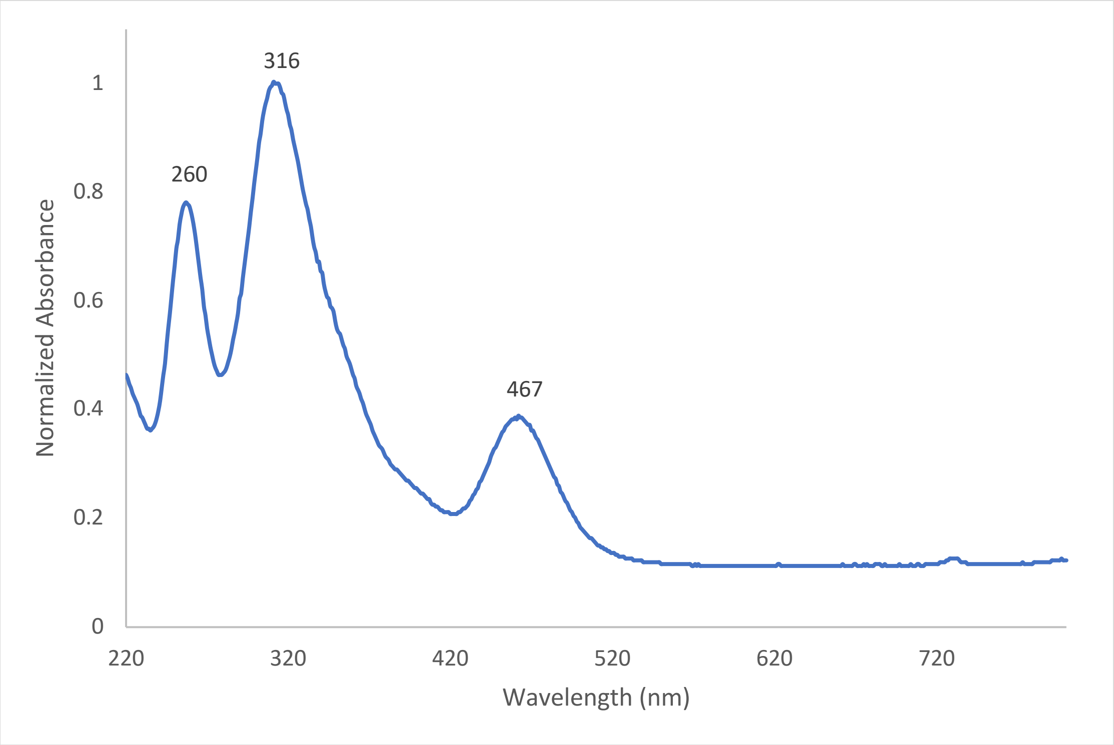


Figure S2. Absorbance spectrum of 60 μM $CuCl_2$ in MeCN. This is 1000x dilution from catalytic conditions.

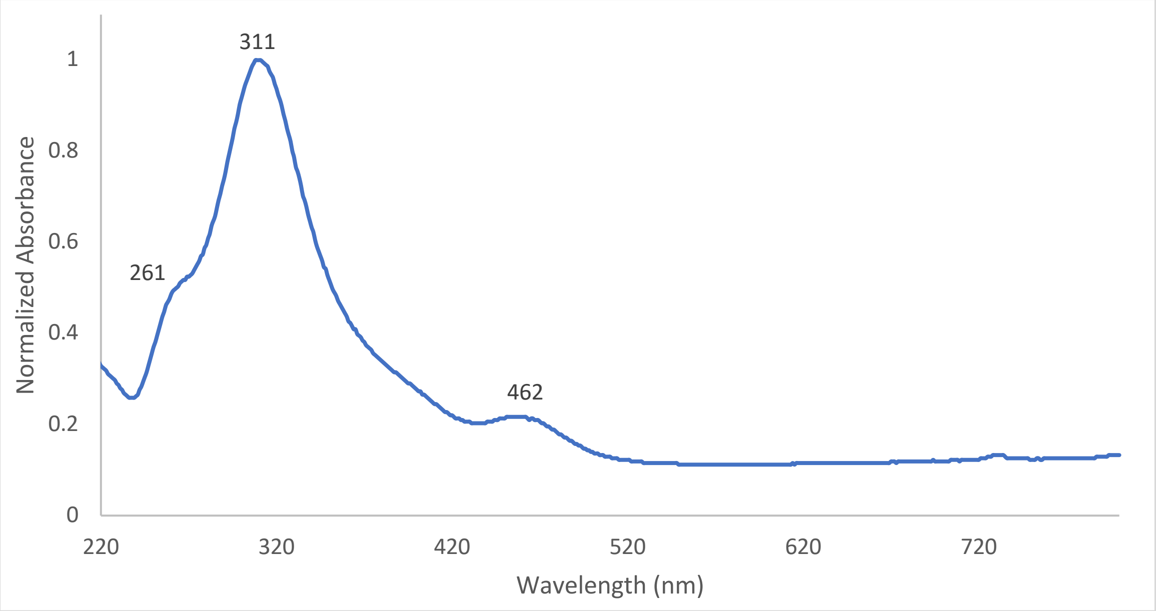


Figure S3. Absorbance spectrum of 60 μM $CuCl_2$ and 150 μM solution of LiCl in MeCN after 2 hours of irradiation under 390 nm LED.

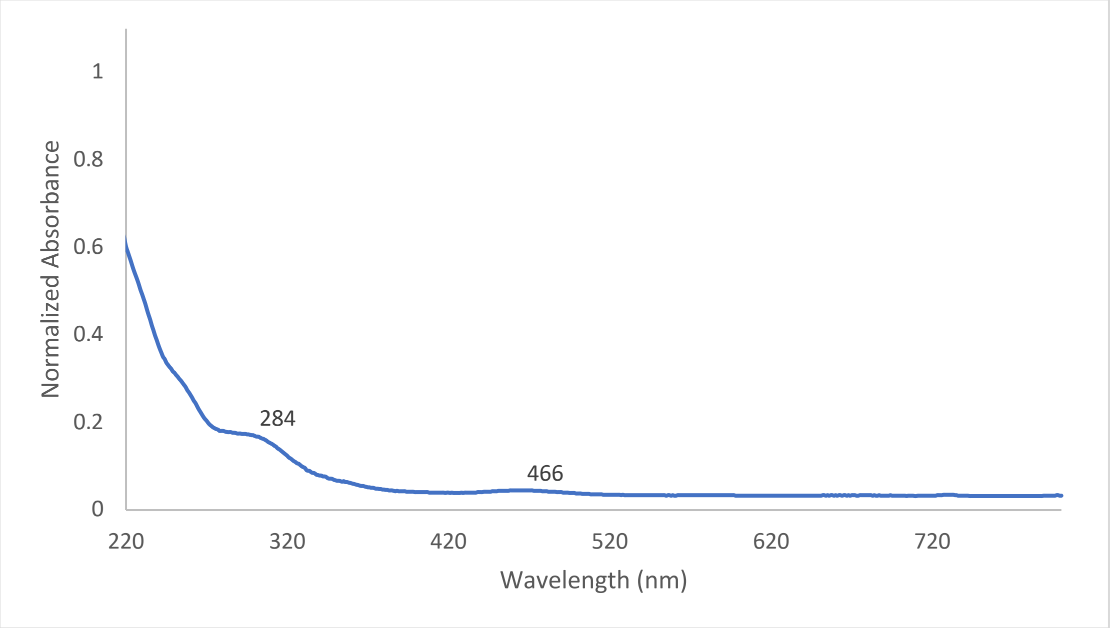


Figure S4. Absorbance spectrum of reaction mixture after 6 hours irradiation at 1000x dilution under N_2.

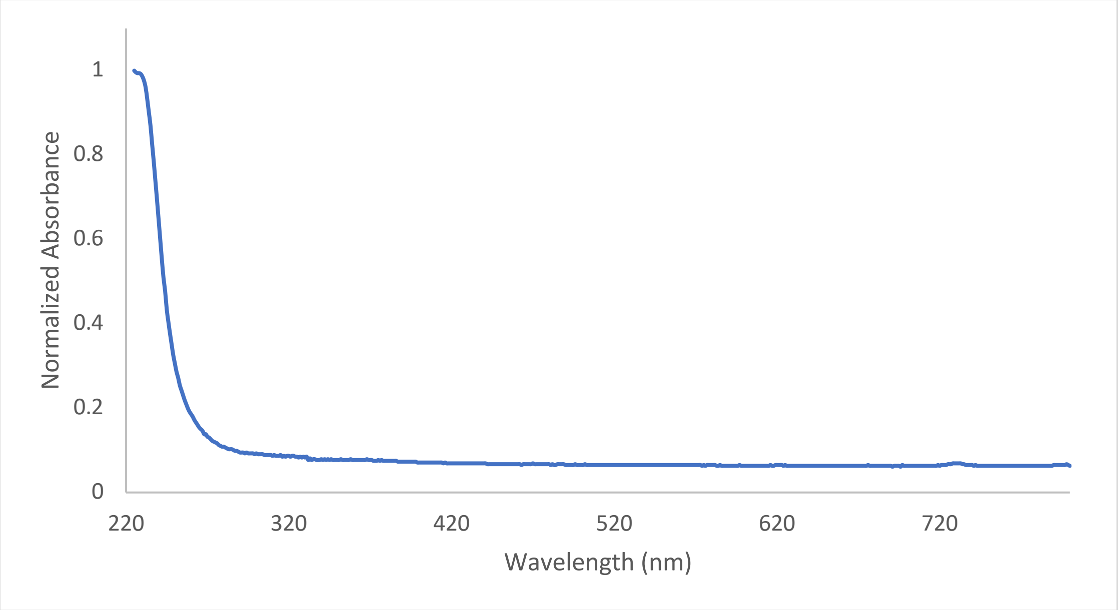


Figure S5. Absorbance spectrum of reaction mixture after stirring open to air for 2 hours at 1000x dilution.

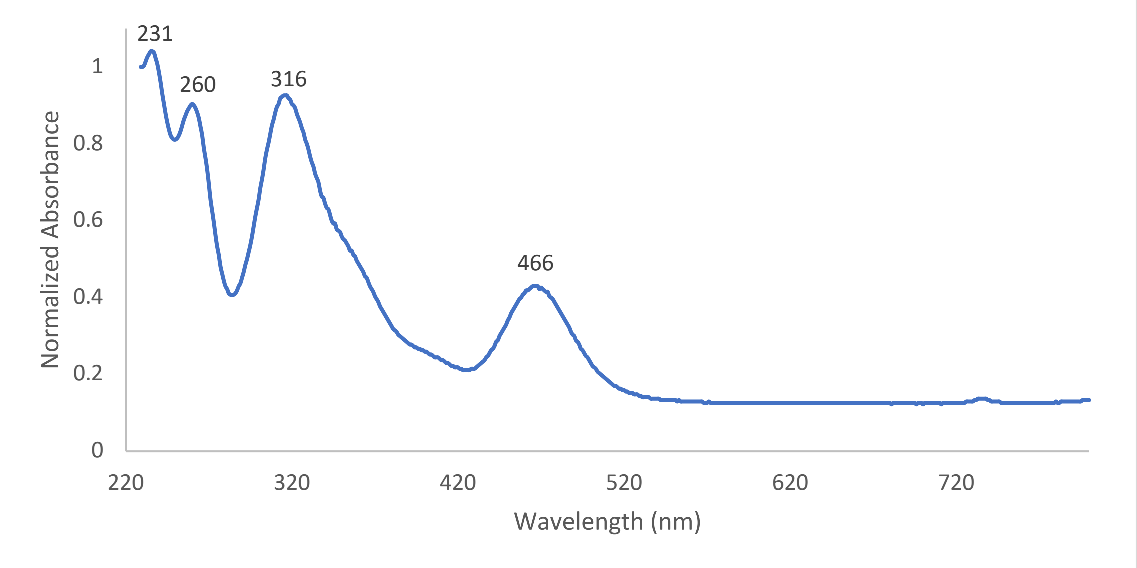


Figure S6. Image of Chlorocuprate solutions:

Left: 60 mM $CuCl_2$ in MeCN

Right: 60 mM $CuCl_2$ and 150 mM solution of LiCl

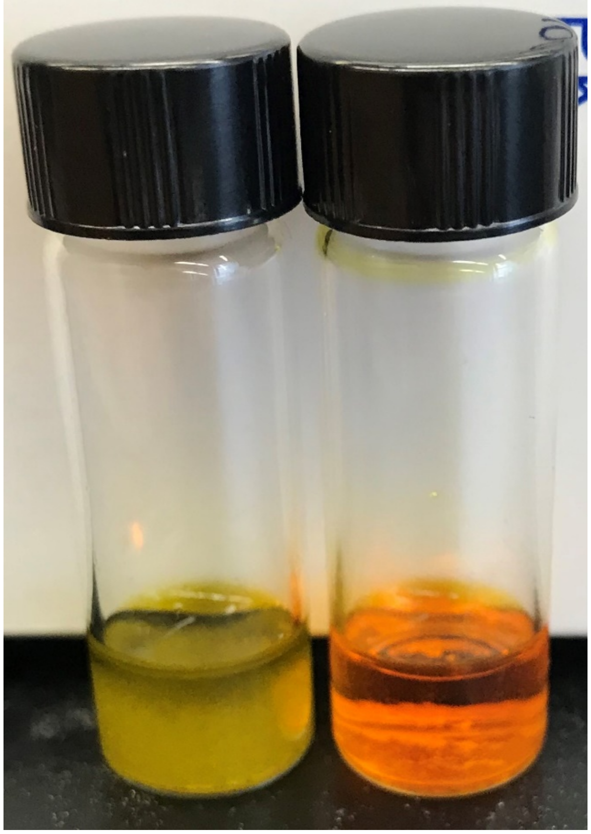
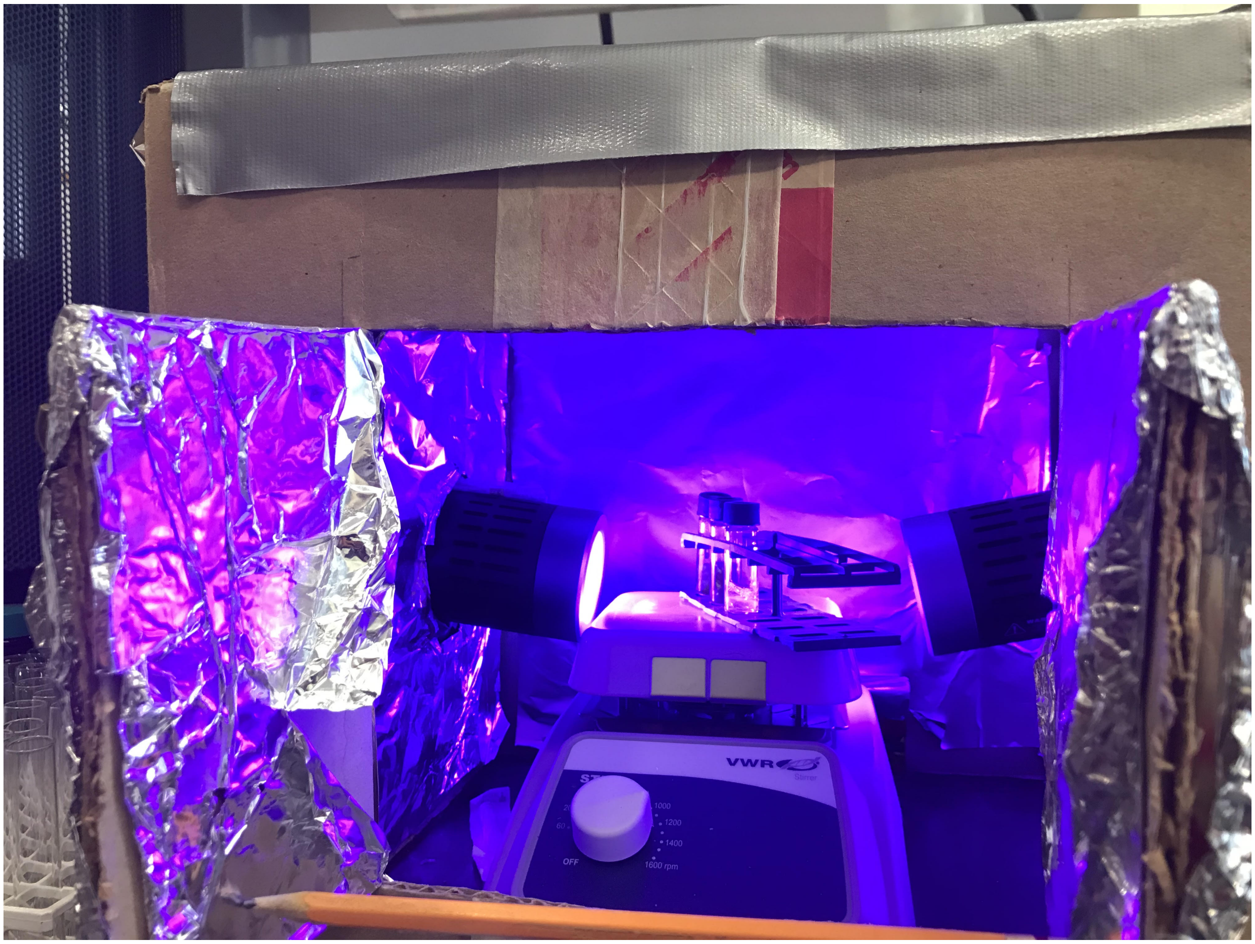
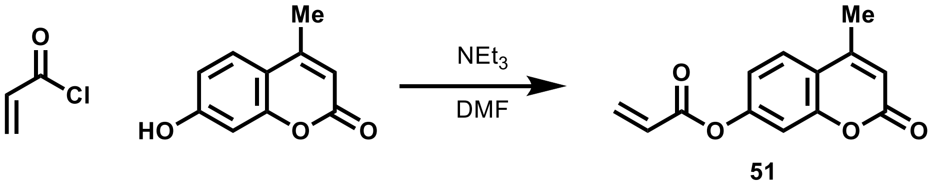


Figure S7. Image of Photoreactor:



Starting Material Synthesis and characterization Data:



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Procedure adapted from reference 2.

To a solution of 7-hydroxy-4-methyl-2H-chromen-2-one (1.00 mmol) in DMF (5.0 mL) was added NEt_3 (180 uL, 1.30 mmol. The mixture was cooled in an ice bath after which acryloyl chloride (117 uL, 1.30 mmol) in DMF (1.0 mL) was added dropwise. The solution was allowed to warm to room temperature and stirred vigorously for 12 hours. Aq. NH_4Cl (5 mL) was then added followed by 10 mL of ethyl acetate. The organic layer was separated and the aqueous layer was extracted twice with ethyl acetate. (10 mL) The combined organic extractions were dried with $MgSO_4$ filtered and concentrated by rotary evaporation. The product was isolated by column chromatography using hexanes:ethyl acetate as the eluent.

1 - 4-methyl-2-oxo-2H-chromen-7-yl acrylate

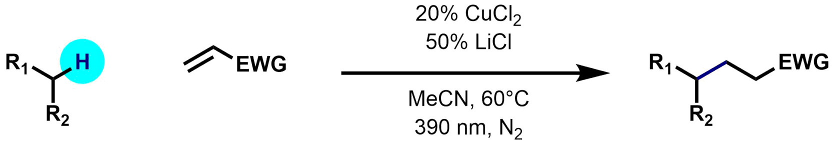
Yield 90%

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 (d, J = 8.6 Hz, 1H), 7.17 (d, J = 2.2 Hz, 1H), 7.13 (dd, J = 8.6, 2.3 Hz, 1H), 6.65 (dd, J = 17.3, 1.1 Hz, 1H), 6.33 (dd, J = 17.3, 10.5 Hz, 1H), 6.27 (d, J = 1.3 Hz, 1H), 6.08 (dd, J = 10.5, 1.2 Hz, 1H), 2.44 (d, J = 1.2 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.94, 160.60, 154.36, 153.14, 152.00, 133.75, 127.49, 125.53, 118.17, 118.05, 114.73, 110.59, 18.86.

HRMS (ASAP+) [$C_{13}H_{11}O_4$] m/z calculated [M+H] 231.07, found 231.0680

Standard Reaction Conditions:



A) To an oven-dried 1- dram vial, $CuCl_2$ and LiCl were added. The vial was charged with a stir bar and transferred to a glovebox, where the solids were backfilled with an inert atmosphere. In the glovebox anhydrous acetonitrile was added (1 mL, 0.3 M) followed by the alkane (5 equiv.) and electron deficient olefin and sealed tightly. The vial was then placed ~3 inches from a 390 nm Kessil lamp to be irradiated and stirred for 36 hours at 60°C. Upon completion, silica was added to the reaction and then concentrated in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

B) Identical to standard reaction conditions A but with 50% $CuCl_2$ and 1.25 equiv. of LiCl.

C) Identical to standard reaction conditions A but with only 3 equivalents of alkane.

D) Identical to standard reaction conditions A but with 1 equivalent of alkane and 1.1 equivalents of electron deficient olefin.

E) To an oven-dried 20- mL scintillation vial, $CuCl_2$ and LiCl were added. The vial was charged with a stir bar and transferred to a glovebox, where the solids were backfilled with an inert atmosphere. In the glovebox anhydrous acetonitrile was added (10 mL, 0.3 M) followed by the alkane (5 equiv.) and electron deficient olefin and sealed tightly. The vial was then placed ~3 inches from a 390 nm Kessil lamp to be irradiated and stirred for 36 hours at 60°C. Upon completion, silica was added to the reaction and then concentrated in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

Characterization Data of Products:

4 -ethyl 3-cyclopentylpropanoate
Prepared using standard reaction conditions A from cyclopentane.
Elutes with 5% EtOAc in Hexanes.
Yield 90% (45.8 mg)
^{1}H (500 MHz, Chloroform-*d*) δ 4.12 (q, *J* = 7.1 Hz, 2H), 2.30 (t, *J* = 7.8 Hz, 2H), 1.82 – 1.70 (m, 3H), 1.67 – 1.56 (m, 4H), 1.55 – 1.46 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.15 – 1.03 (m, 2H).
^{13}C NMR (126 MHz, Chloroform-d) δ 174.23, 60.35, 39.89, 33.92, 32.58, 31.36, 25.30, 14.44.
LRMS (EI) [$C_{10}H_{18}O_2$] m/z calculated 170.13, found 170.1
IR (film) ν_{max} 2948, 2866, 1735, 1178, 1036

5 -ethyl 3-cyclohexylpropanoate
Prepared using standard reaction conditions A from cyclohexane.
Elutes with 5% EtOAc in Hexanes.
Yield 92% (51.2 mg)
^{1}H NMR (400 MHz, CDCl3) δ 4.12 (q, J = 7.1 Hz, 2H), 2.29 (t, J = 7.9 Hz, 2H), 1.77 – 1.44 (m, 7H), 1.34 – 1.07 (m, 7H), 0.89 (q, J = 11.2, 10.6 Hz, 2H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.42, 60.35, 37.43, 33.15, 32.55, 32.16, 26.72, 26.41, 14.43.
LRMS (EI) [$C_{11}H_{20}O_2$] m/z calculated 184.15, found 184.1
IR (film) ν_{max} 2922, 2851, 1734, 1449, 1176, 1162, 943

6-ethyl 3-cycloheptylpropanoate
Prepared using standard reaction conditions A from cycloheptane.
Elutes with 5% EtOAc in Hexanes.
Yield 89% (52.7 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 2.29 (t, J = 7.9 Hz, 2H), 1.78 – 1.32 (m, 13H), 1.33 – 1.06 (m, 5H)
^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.38, 60.34, 38.97, 34.38, 33.18, 32.67, 28.66, 26.51, 14.43.
LRMS (EI) [$C_{12}H_{22}O_2$] m/z calculated 198.16, found 198.2
IR (film) ν_{max} 2920, 2853, 1735, 1458, 1177, 1035, 824

3 -ethyl 3-cyclooctylpropanoate
Prepared using standard reaction conditions A from cyclooctane.
Elutes with 5% EtOAc in Hexanes.
Yield 93% (59.0 mg)
Also prepared using standard reaction conditions D from cyclooctane.
Yield 44% (27.9 mg)
Also prepared using standard reaction conditions E from cyclooctane.
Yield 83% (528.2 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 2.29 (t, J = 7.9 Hz, 2H), 1.74 – 1.35 (m, 15H), 1.31-1.18 (m, 5H).
^{13}C NMR (101 MHz, Chloroform-d) δ 174.37, 60.33, 37.01, 33.24, 32.74, 32.19, 27.40, 26.46, 25.57, 14.44.
LRMS (EI) [$C_{13}H_{24}O_2$] m/z calculated 212.18, found 212.2
IR (film) ν_{max} 2917, 2854, 1735, 1447, 1158, 1030, 857

7-ethyl 4-methylheptanoate (major isomer)
Prepared using standard reaction conditions A from n-pentane.
Elutes with 5% EtOAc in Hexanes.
Mix of regioisomers: 1:1.7:1 (a:b:c)
Yield 95% (49.2 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.11 (qd, J = 7.1, 0.8 Hz, 2H), 2.36 – 2.18 (m, 2H), 1.73 – 1.04 (m, 11H), 0.95 – 0.76 (m, 5H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.35, 60.34, 39.12, 32.34, 32.09, 25.26, 20.18, 19.42, 14.42, 10.94.
LRMS (EI) [$C_{10}H_{20}O_2$] m/z calculated 172.15, found 172.1
IR (film) ν_{max} 2924, 2854, 1738, 1461, 718

8 -ethyl 4-ethoxypentanoate
Prepared using standard reaction conditions A from diethyl ether.
Elutes with 10% EtOAc in Hexanes.
Yield 71% (37.2 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 3.54 (dq, J = 9.0, 7.0 Hz, 1H), 3.48 – 3.27 (m, 2H), 2.50 – 2.28 (m, 2H), 1.87 – 1.70 (m, 2H), 1.25 (t, J = 7.2 Hz, 3H), 1.20 – 1.12 (m, 6H).
^{13}C NMR (126 MHz, Chloroform-d) δ 174.01, 74.21, 63.90, 60.39, 31.82, 30.61, 19.82, 15.70, 14.41.
LRMS (EI) [$C_9H_{18}O_3$] m/z calculated 174.13, found 174.1
IR (film) ν_{max} 2923, 2854, 1733, 1461, 1273, 1123, 961, 742

9 -ethyl 3-(tetrahydrofuran-2-yl)propanoate
Prepared using standard reaction conditions A from tetrahydrofuran.
Elutes with 10% EtOAc in Hexanes.
Yield 77% (39.9 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 3.83 (dddd, J = 10.9, 7.2, 5.5, 2.4 Hz, 2H), 3.70 (td, J = 7.9, 6.2 Hz, 1H), 2.50 – 2.31 (m, 2H), 1.98 (dddd, J = 11.7, 8.5, 6.5, 5.0 Hz, 1H), 1.94 – 1.77 (m, 3H), 1.46 (ddt, J = 12.0, 8.7, 7.4 Hz, 1H), 1.24 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.77, 78.36, 67.86, 60.46, 31.35, 30.90, 25.88, 14.39.
LRMS (EI) [$C_9H_{16}O_3$] m/z calculated 172.11, found 172.1
IR (film) ν_{max} 3388, 2930, 2854, 1730, 1712, 1172, 1025, 754

10 -ethyl 3-(tetrahydro-2H-pyran-2-yl)propanoate
Prepared using standard reaction conditions A from tetrahydropyran.
Elutes with 10% EtOAc in Hexanes.
Yield 61% (34.1 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 3.95 (ddt, J = 11.5, 4.1, 1.8 Hz, 1H), 3.38 (td, J = 11.6, 2.5 Hz, 1H), 3.24 (dddd, J = 10.9, 7.3, 5.1, 2.1 Hz, 1H), 2.52 – 2.30 (m, 2H), 1.87 – 1.70 (m, 3H), 1.62 – 1.44 (m, 4H), 1.31 – 1.22 (m, 4H).
^{13}C NMR (126 MHz, Chloroform-d) δ 174.01, 68.63, 60.39, 31.99, 31.70, 30.60, 26.27, 23.64, 14.42.
LRMS (EI) [$C_{10}H_{18}O_3$] m/z calculated 186.13, found 186.1
IR (film) ν_{max} 3454, 2930, 2872, 1728, 1664, 1183, 1157, 1080, 1027

11 -ethyl 3-(1,4-dioxan-2-yl)propanoate
Prepared using standard reaction conditions A from 1,4 dioxane.
Elutes with 15% EtOAc in Hexanes.
Yield 83% (46.7 mg)
Also prepared using standard reaction conditions D from 1,4 dioxane.
Yield 60% (33.9 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.13 (q, J = 7.1 Hz, 2H), 3.81 – 3.63 (m, 4H), 3.63 – 3.51 (m, 2H), 3.28 (dd, J = 11.5, 9.9 Hz, 1H), 2.55 – 2.31 (m, 2H), 1.78 – 1.62 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.47, 74.54, 71.24, 66.93, 66.67, 60.60, 30.02, 26.82, 14.40.
LRMS (EI) [$C_9H_{16}O_4$] m/z calculated 188.10, found 188.1
IR (film) ν_{max} 3452, 2980, 2934, 1729, 1632, 1403, 1181, 1025, 590

12 -ethyl 4-phenylbutanoate
Prepared using reaction conditions B from toluene.
Elutes with 5% EtOAc in Hexanes.
Yield 69% (39.7 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 7.32 – 7.26 (m, 2H), 7.19 (td, J = 7.3, 6.8, 1.5 Hz, 3H), 4.12 (q, J = 7.1 Hz, 2H), 2.71 – 2.60 (m, 2H), 2.32 (t, J = 7.5 Hz, 2H), 1.96 (p, J = 7.6 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.71, 141.64, 128.69, 128.57, 126.15, 60.46, 35.34, 33.88, 26.74, 14.45.
LRMS (EI) [$C_{12}H_{16}O_2$] m/z calculated 192.12, found 192.1
IR (film) ν_{max} 2921, 2958, 2853, 1731, 1174, 1119, 898, 875, 621

13 -ethyl 3-(2,3-dihydro-1H-inden-1-yl)propanoate

Prepared using standard reaction conditions B from indane.
Elutes with 5% EtOAc in Hexanes.
Yield 72% (47.1 mg)
Prepared using standard reaction conditions D from indane.
Yield 47% (30.8 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 7.25 – 7.10 (m, 4H), 4.15 (q, J = 7.1 Hz, 2H), 3.24 – 2.76 (m, 3H), 2.53 – 2.12 (m, 4H), 1.93 – 1.61 (m, 2H), 1.27 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.92, 146.69, 144.15, 126.68, 126.30, 124.69, 123.86, 60.51, 44.34, 32.64, 31.94, 31.50, 30.13, 14.44.
LRMS (EI) [$C_{14}H_{18}O_2$] m/z calculated 218.13, found 218.1
IR (film) ν_{max} 2959, 2929, 2870, 1733, 1453, 1163, 700

14 -ethyl 4-phenylpentanoate
Prepared using standard reaction conditions A from ethylbenzene.
Elutes with 5% EtOAc in Hexanes.
Mix of regioisomers 5:1 (2:1 isolated) ^{1}H NMR shifts are not resolved. In the cases in which the signals overlap, the minor signal integrations are subtracted from overlapping signals for reported shifts. ^{13}C NMR shifts completely resolve allowing for major regioisomer to be selected.
Yield 68% (41.9 mg)
^{1}H NMR (major regioisomer) (500 MHz, Chloroform-*d*) δ 7.33 – 7.25 (m, 2H), 7.22 – 7.15 (m, 3H), 4.21 – 4.00 (m, 2H), 2.71 (dq, J = 15.6, 6.9 Hz, 1H), 2.26 – 2.11 (m, 2H), 1.99 – 1.83 (m, 2H), 1.36 – 1.18 (m, 6H).
^{13}C NMR (major regioisomer) (126 MHz, Chloroform-d) δ 173.90, 146.49, 128.63, 127.21, 126.36, 60.40, 39.62, 33.41, 32.78, 22.33, 14.40.
LRMS (EI) [$C_{13}H_{18}O_2$] m/z calculated 206.13, found 206.1
IR (film) ν_{max} 2979, 2930, 1731, 1453, 1177, 1142, 1030, 745, 699

15 -ethyl 4-(3,5-dimethylphenyl)butanoate
Prepared using standard reaction conditions B from mesitylene.
Elutes with 5% EtOAc in Hexanes.
Yield 69% (45.5 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 6.84 (s, 1H), 6.81 (s, 1H), 4.14 (q, J = 7.2 Hz, 2H), 2.64 – 2.52 (m, 2H), 2.39 – 2.26 (m, 8H), 1.94 (p, J = 7.6 Hz, 2H), 1.26 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.77, 141.55, 138.00, 127.75, 126.51, 60.40, 35.18, 33.95, 26.77, 21.43, 14.43.
LRMS (EI) [$C_{14}H_{20}O_2$] m/z calculated 220.15, found 220.1
IR (film) ν_{max} 2920, 1733, 1606, 1459, 1373, 1241, 1178, 1144, 842, 703

16 -ethyl 4-phenoxybutanoate
Prepared using standard reaction conditions B from anisole.
Elutes with 10% EtOAc in Hexanes.
Yield 58% (33.2 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 7.32 – 7.26 (m, 2H), 6.94 (tt, J = 7.3, 1.1 Hz, 1H), 6.89 (dt, J = 7.9, 1.0 Hz, 2H), 4.15 (q, J = 7.1 Hz, 2H), 4.01 (t, J = 6.1 Hz, 2H), 2.52 (t, J = 7.3 Hz, 2H), 2.11 (tt, J = 7.4, 6.1 Hz, 2H), 1.26 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.45, 159.00, 129.63, 120.89, 114.65, 66.77, 60.62, 31.02, 24.85, 14.42.
LRMS (EI) [$C_{12}H_{16}O_3$] m/z calculated 208.11, found 208.1
IR (film) ν_{max} 2925, 2856, 1732, 1496, 1243, 1172, 753, 691

17 -ethyl 3-(3-oxocyclohexyl)propanoate
Prepared using standard reaction conditions A from cyclohexanone.
Elutes with 15% EtOAc in Hexanes.
Yield 72% (42.7 mg)
Prepared using standard reaction conditions D from cyclohexanone.
Yield 56% (6:1 rr)
7:1 regioselectivity, major regioisomer selected (isolated fraction 11:1 rr)

^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (qd, J = 7.1, 1.5 Hz, 2H), 2.42 (ddd, J = 13.8, 4.0, 2.0 Hz, 1H), 2.39 – 2.20 (m, 4H), 2.10 – 1.96 (m, 2H), 1.95 – 1.88 (m, 1H), 1.85 – 1.58 (m, 4H), 1.41 – 1.29 (m, 1H), 1.25 (td, J = 7.2, 1.5 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 211.36, 173.51, 60.62, 47.91, 41.53, 38.67, 31.80, 31.64, 31.17, 25.26, 14.40.
LRMS (EI) [$C_{11}H_{18}O_3$] m/z calculated 198.13, found 198.1
IR (film) $ν_{max}$ 2931, 2865, 1729, 1710, 1176, 1155, 1033, 863

18 -ethyl 4-methyl-6-oxononanoate
Prepared using standard reaction conditions A from 4-heptanone.
Elutes with 15% EtOAc in Hexanes.
Yield 78% (50.3 mg)
6:1 regioselectivity, major regioisomer selected
^{1}H NMR (400 MHz, Chloroform-d) δ 4.11 (q, J = 7.2 Hz, 2H), 2.43 – 2.18 (m, 7H), 2.10 – 1.95 (m, 1H), 1.71 – 1.42 (m, 5H), 1.24 (t, J = 7.1 Hz, 3H), 0.98 – 0.83 (m, 6H).
^{13}C NMR (101 MHz, Chloroform-d) δ 210.72, 173.81, 60.50, 50.02, 45.47, 32.22, 31.95, 28.84, 19.64, 17.37, 14.40, 13.91.
LRMS (EI) [$C_{12}H_{22}O_3$] m/z calculated 214.16, found 214.2
IR (film) $ν_{max}$ 2960, 2934, 2875, 1732, 1711, 1372, 1176, 1032

19 -1-ethyl 7-methyl 4-(2-methoxy-2-oxoethyl)heptanedioate
Prepared using standard reaction conditions A from dimethyl adipate.
Elutes with 25% EtOAc in Hexanes.
Yield 65% (53.8 mg)
6:1 regioselectivity, major regioisomer selected
^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 3.67 (d, J = 2.3 Hz, 6H), 2.44 – 2.22 (m, 6H), 2.03 – 1.84 (m, 1H), 1.77 – 1.56 (m, 4H), 1.25 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.92, 173.47, 173.12, 60.61, 51.81 (d, J = 2.8 Hz), 38.33, 34.20, 31.66, 31.42, 28.75, 14.39.
HRMS (ESI+) [$C_{13}H_{22}O_6$] m/z calculated [M+Na] 297.13, found 297.1329
IR (film) $ν_{max}$ 2952, 1729, 1436, 1253, 1151, 1018

20 -ethyl 4-acetoxypentanoate
Prepared using standard reaction conditions A from ethyl acetate.
Elutes with 15% EtOAc in Hexanes.
Yield 58% (32.5 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.92 (h, J = 6.3 Hz, 1H), 4.13 (q, J = 7.1 Hz, 2H), 2.47 – 2.23 (m, 2H), 2.03 (s, 3H), 1.96 – 1.81 (m, 2H), 1.37 – 1.16 (m, 6H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.23, 170.82, 70.21, 60.67, 31.11, 30.58, 20.07, 14.40.
LRMS (ESI+) [$C_9H_{16}O_4$] m/z calculated [M+Na] 211.09, found 211.0972
IR (film) $ν_{max}$ 2981, 2934, 1734, 1373, 1241, 1180

21 -ethyl 3-(4-oxotetrahydro-2H-pyran-2-yl)propanoate
Prepared using standard reaction conditions A from tetrahydro-4H-pyran-4-one.
Elutes with 25% EtOAc in Hexanes.
Yield 58% (34.6 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.27 (ddd, J = 11.5, 7.4, 1.5 Hz, 1H), 4.14 (q, J = 7.1 Hz, 2H), 3.73 – 3.56 (m, 2H), 2.64 – 2.24 (m, 6H), 1.99 – 1.80 (m, 2H), 1.26 (t, J = 7.2 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 206.69, 173.29, 66.67, 60.66, 48.39, 42.33, 31.47, 30.30, 14.41.
LRMS (EI) [$C_{10}H_{16}O_4$] m/z calculated 200.10, found 200.0
IR (film) $ν_{max}$ 2975, 2931, 2857, 1719, 1276, 1174, 1156, 1087, 862

22 -ethyl 4-(N-methylformamido)butanoate
Prepared using standard reaction conditions B from dimethylformamide.
Elutes with 25% EtOAc in Hexanes.
Yield 42% (22.0 mg)

^{1}H NMR (400 MHz, Chloroform-d) δ 8.05 (s, 1H), 4.14 (qd, J = 7.1, 3.2 Hz, 2H), 3.33 (dt, J = 31.0, 7.1 Hz, 2H), 2.90 (d, J = 32.6 Hz, 3H), 2.31 (dt, J = 9.8, 7.3 Hz, 2Hz), 1.88 (h, J = 7.2 Hz, 2H), 1.26 (td, J = 7.1, 2.5 Hz).
^{13}C NMR (126 MHz, Chloroform-d) δ 172.94 (d, J = 51.2 Hz), 162.78 (d, J = 3.3 Hz), 60.77 (d, J = 25.9 Hz), 48.88, 43.67, 34.74, 31.55, 30.83, 29.62, 23.33, 22.13, 14.38.
LRMS (EI) [$C_8H_{15}NO_3$] m/z calculated 173.11, found 173.1
IR (film) ν_{max} 2927, 1775, 1730, 1162, 1069, 1026, 917

23 -ethyl 4-(N-methylacetamido)butanoate
Prepared using standard reaction conditions B from dimethylacetamide.
Elutes with 25% EtOAc in Hexanes.
Yield 59% (33.4 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.14 (dq, J = 9.1, 7.1 Hz, 2H), 3.36 (ddd, J = 29.1, 8.1, 6.5 Hz, 2H), 2.95 (d, J = 30.4 Hz, 3H), 2.32 (q, J = 7.1 Hz, 2H), 2.08 (d, J = 12.6 Hz, 3H), 2.01 – 1.79 (m, 2H), 1.26 (td, J = 7.2, 5.4 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 173.13 (d, J = 70.3 Hz), 170.75 (d, J = 16.0 Hz), 60.73 (d, J = 34.7 Hz), 50.06, 46.90, 36.29, 33.32, 31.67, 31.05, 23.55, 22.71, 22.04, 21.35, 14.40.33.32, 31.67, 31.05, 23.55, 22.71, 22.04, 21.35, 14.40.
LRMS (EI) [$C_9H_{17}NO_3$] m/z calculated 187.12, found 187.1
IR (film) ν_{max} 2966, 2927, 2856, 1719, 1372, 1254, 1174, 1156, 1087

24 -ethyl 4-oxo-4-phenylbutanoate
Prepared using standard reaction conditions C from benzaldehyde.
Elutes with 10% EtOAc in Hexanes.
Yield 94% (57.9 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 8.02 – 7.96 (m, 2H), 7.61 – 7.54 (m, 1H), 7.51 – 7.43 (m, 2H), 4.16 (q, J = 7.2 Hz, 2H), 3.32 (t, J = 6.7 Hz, 2H), 2.76 (t, J = 6.7 Hz, 2H), 1.27 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 198.34, 173.11, 136.78, 133.39, 128.80, 128.23, 60.85, 33.58, 28.48, 14.39.
LRMS (EI) [$C_{12}H_{14}O_3$] m/z calculated 206.09, found 206.1
IR (film) ν_{max} 2926, 2855, 1731, 1689, 1243, 1172, 754, 691

25 -ethyl 3-(triphenylsilyl)propanoate
Prepared using standard reaction conditions C from triphenylsilane.
Elutes with 5% EtOAc in Hexanes.
Yield 82% (88.5 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 7.56 – 7.49 (m, 5H), 7.45 – 7.39 (m, 3H), 7.37 (ddt, J = 8.3, 6.7, 1.3 Hz, 5H), 4.05 (q, J = 7.1 Hz, 2H), 2.48 – 2.36 (m, 2H), 1.77 – 1.66 (m, 2H), 1.20 (t, J = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, Chloroform-d) δ 174.84, 135.78, 134.35, 129.83, 128.18, 60.65, 29.11, 14.37, 8.44.
LRMS (EI) [$C_{12}H_{24}O_2Si$] m/z calculated 360.15, found 360.1
IR (film) ν_{max} 3068, 2924, 2853, 2360, 1735, 1428, 1112, 701

26 -3-methyl-6-oxo-6-phenoxyhexanoic acid
Prepared using standard reaction conditions A from butanoic acid and ethyl acrylate.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 60%, 2:1 rr (33.7 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.22 – 4.03 (m, 2H), 2.45 – 2.12 (m, 4H), 2.00 (dq, J = 13.7, 6.8 Hz, 1H), 1.80 – 1.49 (m, 3H), 1.47 – 1.34 (m, 1H), 1.25 (td, J = 7.1, 1.2 Hz, 3H), 0.99 (d, J = 6.7 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-d) δ 178.33, 173.71, 60.60, 41.24, 34.28, 32.15, 31.65, 29.88, 19.50, 14.40.
HRMS (ASAP-) [$C_9H_{15}O_4$] m/z calculated [M-H] 187.0970, found 187.0966
IR (film) ν_{max} 2935, 1734, 1708, 1375, 1182, 1094, 1032

27 -7-ethoxy-3-methyl-7-oxoheptanoic acid
Prepared using standard reaction conditions A from 3-methylbutanoic acid and ethyl acrylate.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 73%, 3:1 rr (44.1 mg)

^{1}H NMR (400 MHz, Chloroform-d, major selected) δ 4.13 (q, J = 7.1 Hz, 2H), 2.44 – 2.12 (m, 4H), 1.99 (dt, J = 13.5, 6.7 Hz, 1H), 1.78 – 1.54 (m, 2H), 1.39 (ddt, J = 13.4, 11.0, 5.6 Hz, 1H), 1.31 – 1.18 (m, 5H), 0.99 (d, J = 6.6 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-d) δ 178.26, 173.80, 60.47, 36.15, 34.52, 30.10, 27.07, 22.51, 19.73, 14.43.
HRMS (ASAP-) [$C_{10}H_{17}O_4$] m/z calculated [M-H] 201.112, found 201.1126
IR (film) ν_{max} 2963, 1733, 1706, 1371, 1265, 1186, 935, 738

28 -7-ethoxy-3,3-dimethyl-7-oxoheptanoic acid
Prepared using standard reaction conditions A from 3,3-dimethylbutanoic acid and ethyl acrylate.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 59% (38.2 mg)
^{1}H NMR (400 MHz, Chloroform-d) δ 4.13 (q, J = 7.1 Hz, 2H), 2.28 (t, J = 7.4 Hz, 2H), 2.24 (s, 2H), 1.68 – 1.57 (m, 2H), 1.41 – 1.30 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H), 1.03 (s, 6H).
^{13}C NMR (101 MHz, Chloroform-d) δ 177.88, 173.87, 60.47, 45.73, 41.60, 34.98, 33.31, 27.33, 19.92, 14.43.
HRMS (ASAP-) [$C_{11}H_{19}O_4$] m/z calculated [M-H] 215.1283, found 214.128
IR (film) ν_{max} 2924, 2855, 1704, 1191, 1162, 1133, 690

29 -3-(3-ethoxy-3-oxopropyl)cyclopentane-1-carboxylic acid
Prepared using standard reaction conditions A from cyclopentanecarboxylic acid and ethyl acrylate.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 57%, 1:1 dr (36.5 mg)
^{1}H NMR (500 MHz, Chloroform-d) δ 4.12 (q, J = 7.1 Hz, 2H), 2.95 – 2.73 (m, 1H), 2.31 (td, J = 7.7, 3.9 Hz, 2H), 2.19 – 2.06 (m, 1H), 2.05 – 1.77 (m, 4H), 1.77 – 1.58 (m, 2H), 1.55 – 1.38 (m, 1H), 1.25 (td, J = 7.1, 2.2 Hz, 4H).
^{13}C NMR (126 MHz, Chloroform-d, major selected) δ 182.55, 173.90, 60.51, 42.82, 39.30, 35.74, 33.62, 32.77, 30.78, 29.70, 14.42.
HRMS (ASAP-) [$C_{11}H_{17}O_4$] m/z calculated [M-H] 213.1127, found 213.1127
IR (film) ν_{max} 2945, 1733, 1701, 1450, 1374, 1181, 940

30 -6-ethoxy-2,2,3-trimethyl-6-oxohexanoic acid
Prepared using standard reaction conditions A from 2,2-dimethylbutanoic acid and ethyl acrylate.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 62%, 2:1 rr (39.0 mg)
^{1}H NMR (400 MHz, Chloroform-d, major selected) δ 4.12 (qd, J = 7.1, 2.4 Hz, 2H), 2.48 – 2.14 (m, 2H), 1.88 – 1.73 (m, 1H), 1.68 – 1.44 (m, 2H), 1.25 (td, J = 7.2, 2.9 Hz, 3H), 1.14 (d, J = 8.5 Hz, 6H), 0.89 (d, J = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-d) δ 184.17, 173.84, 60.54, 46.15, 39.48, 33.20, 25.13, 21.88, 21.64, 14.42.
HRMS (ASAP-) [$C_{11}H_{19}O_4$] m/z calculated [M-H] 215.1283, found 215.1285
IR (film) ν_{max} 2976, 2939, 1734, 1697, 1463, 1372, 1180, 937, 857

31 -methyl 3-cyclooctylpropanoate
Prepared using standard reaction conditions A from methyl acrylate and cyclooctane.
Elutes with 5% EtOAc in Hexanes.
Yield 96% (57.3 mg)
^{1}H NMR (400 MHz, CDCl3) δ 3.66 (s, 3H), 2.34 – 2.28 (m, 2H), 1.71 – 1.35 (m, 15H), 1.32 – 1.20 (m, 2H).
^{13}C NMR (101 MHz, CDCl3) δ 174.79, 51.64, 37.05, 33.20, 32.47, 32.17, 27.43, 26.46, 25.56.
LRMS (EI) [$C_{12}H_{22}O_2$] m/z calculated 198.16, found 198.1
IR (film) ν_{max} 2918, 2853, 1736, 1446, 1032, 857

32 -phenyl 3-cyclooctylpropanoate
Prepared using standard reaction conditions A from phenyl acrylate and cyclooctane.
Elutes with 5% EtOAc in Hexanes.
Yield 90% (70.0 mg)
^{1}H NMR (400 MHz, CDCl3) δ 7.42 – 7.33 (m, 2H), 7.25 – 7.19 (m, 1H), 7.12 – 7.05 (m, 2H), 2.61 – 2.52 (m, 2H), 1.75 – 1.27 (m, 6H).
^{13}C NMR (101 MHz, CDCl3) δ 172.73, 150.99, 129.55, 125.84, 121.74, 37.04, 33.19, 32.78, 32.24, 27.40, 26.48, 25.59.
LRMS (EI) [$C_{17}H_{24}O_2$] m/z calculated 260.18, found 260.1

IR (film) ν_{max} 2918, 2854, 1758, 1492, 1193, 1134, 749, 689

33 -methyl 3-cyclooctyl-2-methylpropanoate
Prepared using standard reaction conditions A from methyl methacrylate and cyclooctane.
Elutes with 5% EtOAc in Hexanes.
Yield 64% (40.8 mg)
^{1}H NMR (500 MHz, CDCl3) δ 3.66 (s, 3H), 2.52 (dq, J = 13.6, 7.0 Hz, 1H), 1.67 – 1.36 (m, 14H), 1.29 – 1.17 (m, 3H), 1.12 (d, J = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, CDCl3) δ 172.73, 150.99, 129.55, 125.84, 121.74, 37.04, 33.19, 32.78, 32.24, 27.40, 26.48, 25.59.
LRMS (EI) [$C_{13}H_{24}O_2$] m/z calculated 212.18, found 212.2
IR (film) ν_{max} 2921, 2853, 1738, 1460, 1196, 1164

34 -diisopropyl 2-(cyclooctylmethyl)malonate
Elutes with 15% EtOAc in Hexanes.
Prepared using standard reaction conditions A from diisopropyl 2-methylenemalonate and cyclooctane.
Yield 48% (44.9 mg)
^{1}H NMR (500 MHz, CDCl3) δ 5.05 (hept, J = 6.2 Hz, 2H), 3.35 (t, J = 7.7 Hz, 1H), 1.77 (t, J = 7.4 Hz, 2H), 1.67 – 1.37 (m, 13H), 1.32 – 1.26 (m, 2H), 1.23 (dd, J = 6.3, 3.2 Hz, 12H).
^{13}C NMR (126 MHz, CDCl3) δ 169.57, 68.75, 50.79, 36.70, 35.08, 32.14, 27.32, 26.47, 25.46, 21.85, 21.78.
LRMS (EI) [$C_{18}H_{32}O_4$] m/z calculated 312.23, found 312.0
IR (film) ν_{max} 2980, 2921, 2856, 1728, 1246, 1100

35 -3-cyclooctylpropanoic acid
Prepared using standard reaction conditions A from acrylic acid and cyclooctane.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 83% (45.9 mg)
^{1}H NMR (500 MHz, CDCl3) δ 2.47 – 2.23 (m, 2H), 1.70 – 1.37 (m, 15H), 1.33 – 1.21 (m, 2H).
^{13}C NMR (126 MHz, CDCl3) δ 180.45, 36.96, 32.88, 32.37, 32.13, 27.41, 26.43, 25.52.
LRMS (EI) [$C_{11}H_{20}O_2$] m/z calculated 184.15, found 184.1
IR (film) ν_{max} 2919, 2853, 1708, 1446, 1272, 933

36-4-methyl-2-oxo-2H-chromen-6-yl 3-cyclooctylpropanoate
Prepared using standard reaction conditions A from 4-methyl-2-oxo-2H-chromen-6-yl acrylate and cyclooctane.
Elutes with 10% EtOAc in Hexanes.
Yield 86% (88.1 mg)
^{1}H NMR (500 MHz, CDCl3) δ 7.60 (d, J = 8.6 Hz, 1H), 7.10 (d, J = 2.2 Hz, 1H), 7.06 (dd, J = 8.6, 2.3 Hz, 1H), 6.27 (q, J = 1.3 Hz, 1H), 2.67 – 2.54 (m, 2H), 2.43 (d, J = 1.3 Hz, 3H), 1.74 – 1.65 (m, 8H), 1.53 – 1.42 (m, 5H), 1.36 – 1.23 (m, 4H).
^{13}C NMR (126 MHz, CDCl3) δ 172.15, 160.75, 154.39, 153.41, 152.12, 125.52, 118.32, 117.95, 114.66, 110.65, 37.03, 33.02, 32.75, 32.21, 27.39, 26.47, 25.57, 18.92.
LRMS (EI) [$C_{21}H_{26}O_4$] m/z calculated 342.18, found 342.2
IR (film) ν_{max} 2917, 2853, 1757, 1729, 1612, 1261, 1127, 855

37 -(2-(phenylsulfonyl)ethyl)cyclooctane
Prepared using standard reaction conditions B from phenyl vinyl sulfone and cyclooctane.
Elutes with 10% EtOAc in Hexanes.
Yield 77% (65.1 mg)
^{1}H NMR (500 MHz, CDCl3) δ 7.90 (dd, J = 8.4, 1.3 Hz, 2H), 7.68 – 7.62 (m, 1H), 7.59 – 7.53 (m, 2H), 3.15 – 3.02 (m, 2H), 1.65 – 1.48 (m, 10H), 1.46 – 1.32 (m, 5H), 1.28 – 1.17 (m, 2H).
^{13}C NMR (126 MHz, CDCl3) δ 139.45, 133.76, 129.42, 128.20, 55.02, 36.46, 32.14, 30.45, 27.16, 26.31, 25.38.
LRMS (EI) [$C_{16}H_{24}O_2S$] m/z calculated 280.15 found 280.1
IR (film) ν_{max} 2919, 2855, 1495, 1335, 1145, 762, 687

38 -5-((2-cyclooctylethyl)sulfonyl)-1-phenyl-1H-tetrazole

Prepared using standard reaction conditions B from diisopropyl 1-phenyl-5-(vinylsulfonyl)-1H-tetrazole and cyclooctane.
Elutes with 20% EtOAc in Hexanes.
Yield 73% (76.2 mg)
^{1}H NMR (500 MHz, CDCl3) δ 7.71 – 7.66 (m, 2H), 7.64 – 7.57 (m, 3H), 3.77 – 3.71 (m, 2H), 1.88 – 1.79 (m, 2H), 1.71 – 1.54 (m, 8H), 1.47 (d, J = 8.4 Hz, 5H), 1.39 – 1.29 (m, 2H).
^{13}C NMR (126 MHz, CDCl3) δ 153.66, 133.22, 131.62, 129.88, 125.25, 54.82, 36.54, 31.99, 29.58, 27.18, 26.29, 25.34.
HRMS (ASAP+) [$C_{17}H_{24}N_4O_2S$] m/z calculated [M+H] 349.17, found 349.1698
IR (film) ν_{max} 2917, 2852, 1497, 1336, 1148, 761, 688

39 -diisopropyl 1-cyclooctylhydrazine-1,2-dicarboxylate
Prepared using standard reaction conditions B from diisopropyl azodicarboxylate and cyclooctane.
Elutes with 25% EtOAc in Hexanes.
Yield 72% (67.7 mg)
^{1}H NMR (400 MHz, CDCl3) δ 6.10 (d, J = 56.3 Hz, 1H), 4.93 (dp, J = 12.6, 6.3 Hz, 2H), 4.22 (s, 1H), 1.89 – 1.41 (m, 15H), 1.24 (t, J = 6.8 Hz, 12H).
^{13}C NMR (126 MHz, CDCl3) δ 156.00 (d, J = 185.4 Hz), 69.82 (d, J = 33.3 Hz), 57.78, 31.13, 26.81, 26.21, 24.67, 22.22 (d, J = 18.0 Hz).
LRMS (EI) [$C_{16}H_{30}N_2O_4$] m/z calculated 314.22, found 314.2
IR (film) ν_{max} 3291, 2979, 2922, 2855, 1729, 1702, 1682, 1107, 760

40 -3-cyclooctylcyclopentan-1-one
Prepared using standard reaction conditions A from cyclopent-2-en-1-one and cyclooctane.
Elutes with 5% EtOAc in Hexanes.
Yield 65% (38.0 mg)
^{1}H NMR (400 MHz, CDCl3) δ 2.42 – 2.23 (m, 2H), 2.23 – 2.07 (m, 2H), 2.05 – 1.92 (m, 1H), 1.89 – 1.76 (m, 1H), 1.77 – 1.58 (m, 7H), 1.54 – 1.40 (m, 7H), 1.38 – 1.24 (m, 2H).
^{13}C NMR (101 MHz, CDCl3) δ 220.18, 44.15, 43.73, 42.98, 39.31, 30.89, 29.95, 28.12, 27.52, 27.44, 26.54, 25.70, 25.59.
LRMS (EI) [$C_{13}H_{22}O$] m/z calculated 194.17, found 194.2
IR (film) ν_{max} 2915, 2854, 1740, 1470, 1446, 1162, 503

41 -3-cyclooctyldihydrofuran-2,5-dione
Prepared using standard reaction conditions A from furan-2,5-dione and cyclooctane.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 76% (47.9 mg)
^{1}H NMR (400 MHz, CDCl3, major selected) δ 3.11 (ddd, J = 10.3, 6.2, 4.3 Hz, 1H), 2.96 (dd, J = 18.9, 10.0 Hz, 1H), 2.73 (dd, J = 18.9, 6.2 Hz, 1H), 2.27 (dtd, J = 11.3, 7.5, 3.9 Hz, 1H), 1.77 – 1.31 (m, 14H).
^{13}C NMR (101 MHz, CDCl3) δ 173.38, 170.60, 48.18, 38.07, 32.04, 31.21, 29.42, 26.55, 26.46, 26.36, 26.04, 25.36.
LRMS (EI) [$C_{12}H_{18}O_3$] m/z calculated 210.13, found 210.1
IR (film) ν_{max} 2919, 2853, 1860, 1777, 1470, 1220, 1061, 917, 640, 422

42 -3-cyclooctyl-1-methylpyrrolidine-2,5-dione
Prepared using standard reaction conditions A from 1-methyl-1H-pyrrole-2,5-dione and cyclooctane.
Elutes with 20% EtOAc in Hexanes.
Yield 87% (58.0 mg)
^{1}H NMR (400 MHz, CDCl3, major selected) δ 2.97 (s, 3H), 2.81 (dt, J = 8.9, 4.3 Hz, 1H), 2.67 (dd, J = 18.2, 9.2 Hz, 1H), 2.44 (dd, J = 18.3, 4.7 Hz, 1H), 2.33 – 2.22 (m, 1H), 1.72 – 1.24 (m, 14H).
^{13}C NMR (101 MHz, CDCl3) δ 179.86, 177.40, 47.40, 37.58, 32.35, 31.13, 28.68, 26.62, 26.54, 26.13, 25.54, 24.86.
LRMS (EI) [$C_{13}H_{21}NO_2$] m/z calculated 223.16, found 223.2
IR (film) ν_{max} 2917, 2851, 1774, 1694, 1434, 1382, 1278, 1119, 953, 699, 606

43 -3-cyclooctyl-2-methylcyclopentan-1-one
Prepared using standard reaction conditions A from 2-methylcyclopent-2-en-1-one and cyclooctane.
Elutes with 10% EtOAc in Hexanes.

Yield 83% >20:1 dr (51.6 mg)
Major diastereomer assigned by literature precedent.[3]
^{1}H NMR (400 MHz, CDCl3) δ 2.40 – 2.00 (m, 2H), 2.00 – 1.83 (m, 2H), 1.80 – 1.24 (m, 17H), 1.08 (d, J = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, CDCl3) δ 221.93, 51.79, 47.37, 38.32, 37.43, 32.87, 27.79, 27.16, 27.05, 26.89, 26.59, 25.90, 22.86, 13.85.
LRMS (EI) [$C_{14}H_{24}O$] m/z calculated 208.18, found 208.2
IR (film) ν_{max} 2916, 2852, 1739, 1448, 1168, 953, 821

44 -endo-3-(cyclooctylmethyl)bicyclo[2.2.1]heptan-2-one
Prepared using standard reaction conditions A from 3-methylenebicyclo[2.2.1]heptan-2-one and cyclooctane.
Elutes with 10% EtOAc in Hexanes.
Yield 72% 10:1 dr (51.1 mg)
Major diastereomer assigned by similarity to literature precedent.[4]
^{1}H NMR (400 MHz, CDCl3, major selected) δ 2.59 (d, J = 4.7 Hz, 2H), 2.06 (dd, J = 10.4, 4.5 Hz, 1H), 1.86 – 1.74 (m, 1H), 1.68 – 1.36 (m, 19H), 1.33 – 1.18 (m, 2H), 1.15 – 1.05 (m, 1H).
^{13}C NMR (101 MHz, CDCl3) δ 220.97, 51.93, 50.77, 38.71, 37.31, 35.50, 34.32, 33.59, 30.60, 27.61, 27.46, 26.43, 25.55, 25.53, 25.47, 21.51.
LRMS (EI) [$C_{16}H_{26}O$] m/z calculated 234.20, found 234.2
IR (film) ν_{max} 2915, 2852, 1742, 1446, 1068, 915, 489

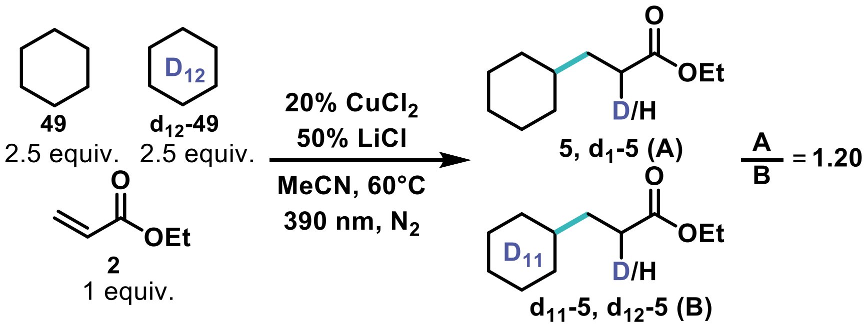
45 -endo-3-phenethylbicyclo[2.2.1]heptan-2-one
Prepared using standard reaction conditions A from 3-methylenebicyclo[2.2.1]heptan-2-one and toluene.
Elutes with 10% EtOAc in Hexanes.
Yield 70% 20:1 dr (45.3 mg)
Major diastereomer assigned by literature precedent.[5]
^{1}H NMR (400 MHz, CDCl3, major selected) δ 7.27 (d, J = 7.6 Hz, 2H), 7.20 (d, J = 7.4 Hz, 3H), 2.80 – 2.51 (m, 4H), 2.15 – 1.95 (m, 2H), 1.89 – 1.75 (m, 1H), 1.73 – 1.48 (m, 5H), 1.48 – 1.36 (m, 1H).
^{13}C NMR (101 MHz, CDCl3) δ 220.00, 141.78, 128.58, 126.14, 52.95, 50.75, 38.48, 37.19, 34.24, 28.26, 25.51, 21.38.
LRMS (EI) [$C_{15}H_{18}O$] m/z calculated 214.14, found 214.1
IR (film) ν_{max} 3026, 2958, 2875, 1740, 1454, 914, 750, 700, 487

46 -1-(2-cyclooctylcyclohexyl)ethan-1-one
Prepared using standard reaction conditions B from 1-cyclohexylethan-1-one and cyclooctane.
Elutes with 10% EtOAc in Hexanes.
Yield 57% 15:1 dr (40.4 mg)
Major diastereomer assigned by similarity to literature precedent.[6]
^{1}H NMR (400 MHz, CDCl3, major selected) δ 2.44 (td, J = 11.3, 3.4 Hz, 1H), 2.12 (s, 3H), 1.85 – 1.59 (m, 7H), 1.54 – 1.12 (m, 16H), 1.01 (qd, J = 12.5, 3.1 Hz, 1H).
^{13}C NMR (101 MHz, CDCl3) δ 213.76, 55.28, 47.08, 38.92, 33.93, 30.36, 28.90, 27.49, 27.08, 27.04, 26.74, 26.57, 26.46, 26.34, 26.12, 25.02.
LRMS (EI) [$C_{16}H_{28}O$] m/z calculated 236.21, found 236.2
IR (film) ν_{max} 2919, 2853, 1707, 1446, 1352, 1159, 606, 561

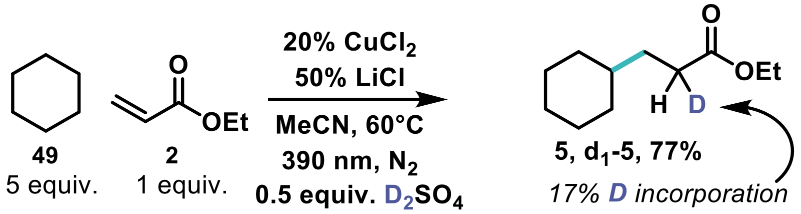
47 -2-cyclooctylcyclopentane-1-carboxylic acid
Prepared using standard reaction conditions A from cyclopent-1-ene-1-carboxylic acid and cyclooctane.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 75% >20:1 dr (50.5 mg)
Major diastereomer assigned by analogy.
^{1}H NMR (500 MHz, CDCl3, major selected) δ 2.93 (td, J = 7.3, 2.1 Hz, 1H), 1.99 – 1.76 (m, 6H), 1.75 – 1.51 (m, 9H), 1.49 – 1.30 (m, 6H), 1.30 – 1.18 (m, 1H).
^{13}C NMR (126 MHz, CDCl3) δ 182.47, 51.59, 46.39, 38.91, 31.49, 31.20, 30.08, 29.27, 27.81, 27.47, 26.66, 25.76, 25.01, 23.72.
HRMS (ASAP-) [$C_{14}H_{23}O_2$] m/z calculated 223.1698, found 223.17

IR (film) ν_{max} 2920, 2854, 1699, 1447, 1231, 932

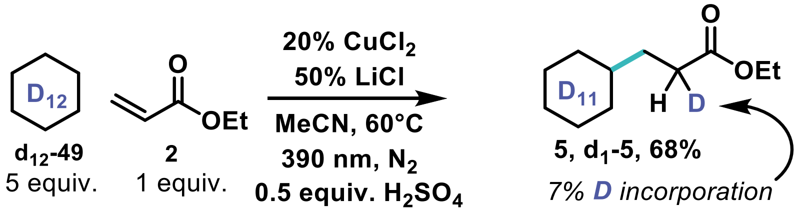
48 -3-cyclooctyl-3,4-dimethyldihydrofuran-2,5-dione
Prepared using standard reaction conditions A from 3,4-dimethylfuran-2,5-dione and cyclooctane.
Elutes with 10% EtOAc and .1% acetic acid in Hexanes.
Yield 67% (48.2 mg)
Major diastereomer assigned by analogy.
^{1}H NMR (400 MHz, CDCl3, major selected) δ 2.86 (q, J = 7.3 Hz, 1H), 1.85 (t, J = 9.2 Hz, 1H), 1.79 – 1.67 (m, 3H), 1.61 – 1.39 (m, 13H), 1.33 (d, J = 7.3 Hz, 4H).
^{13}C NMR (101 MHz, CDCl3) δ 173.71, 52.86, 46.57, 42.00, 29.61, 28.59, 27.08, 26.41, 25.66, 21.93, 8.56.LRMS
LRMS (EI) $[C_{13}H_{22}O_1]$ m/z (-CO_2) calculated 194.17, found 194.2
IR (film) ν_{max} 2921, 2853, 1842, 1775, 1461, 1220, 948, 922, 755, 540



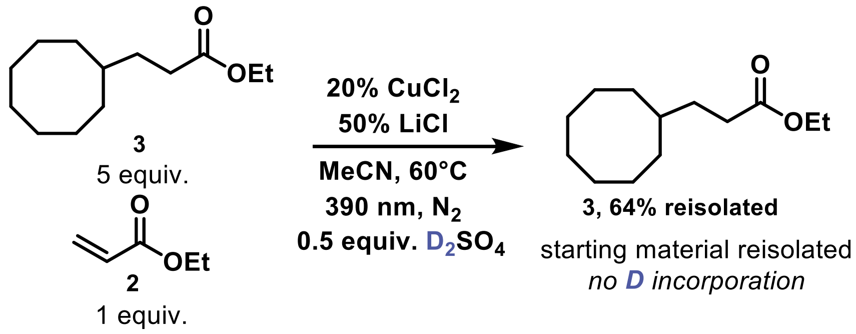
5, d_1-5, d_{11}-5, d_{12}-5 – isotopically mixed - ethyl 3-cyclohexylpropanoate
Prepared using standard reaction conditions A from a 1:1 mixture of cyclohexane and cyclohexane-d_{12} (5 equiv. alkane total). However, the reaction was quenched after 6 hours instead of the typical 36 hour reaction time. Analysis of the reaction mixture by GC-MS revealed resolution of two isotopologues identified as mixtures A and B. The ratios of these mixtures were determined via calibration of prepared mixtures of A and B. A and B were independently synthesized from cyclohexane and cyclohexane-d_{12} respectively.



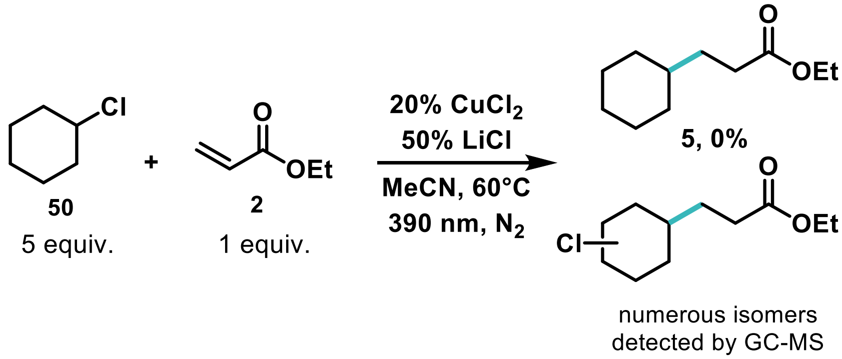
5, d_1-5, – isotopically mixed - ethyl 3-cyclohexylpropanoate
Prepared using standard reaction conditions A from cyclohexane with the addition 0.5 equivalents of D_2SO_4. Deuterium incorporation was measured via proton NMR and confirmed by mass spectrometry.



5, d_1-5, – isotopically mixed - ethyl 3-cyclohexylpropanoate
Prepared using standard reaction conditions A from cyclohexane-d_{12} with the addition 0.5 equivalents of H_2SO_4. Deuterium incorporation was measured via proton NMR and confirmed by mass spectrometry.



3 – ethyl 3-cyclooctylpropanoate
Reisolated after material subjected to standard reaction conditions A with the addition of 0.5 equivalents of D_2SO_4. Despite addition of D_2SO_4, there is no evidence of deuterium incorporation into the starting material by ^{1}H NMR, ^{2}H NMR, or mass spectrometry.



5 – ethyl 3-cyclohexylpropanoate
Subjecting chlorocyclohexane to standard reaction conditions A showed no evidence for the synthesis of product **5.** GC-MS analysis of the reaction mixture revealed numerous signals with parent masses corresponding to the C-H alkylation of chlorocyclohexane.

References:

[1] Mereshchenko, A. S.; Olshin, P. K.; Karimov, A. M.; Skripkin, M. Y.; Burkov, K. A.; Tveryanovich, Y. S.; Tarnovsky, A. N. *Chem. Phys. Lett.* **2014**, *615*, 105–110.

[2] Schultze, C.; Schmidt, B. *Beilstein J. Org. Chem.* **2018**, *14*, 2991–2998.

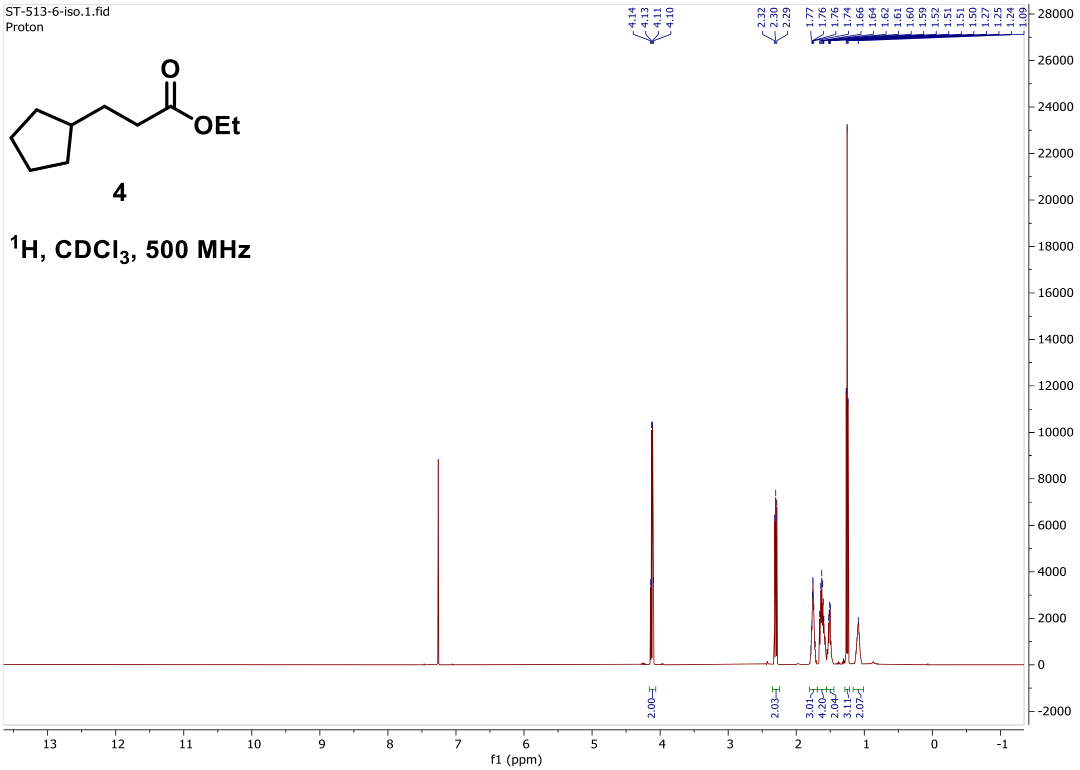
[3] Germain, N.; Guénée, L.; Mauduit, M.; Alexakis, A., *Organic Letters* **2014,** *16*, 118-121.

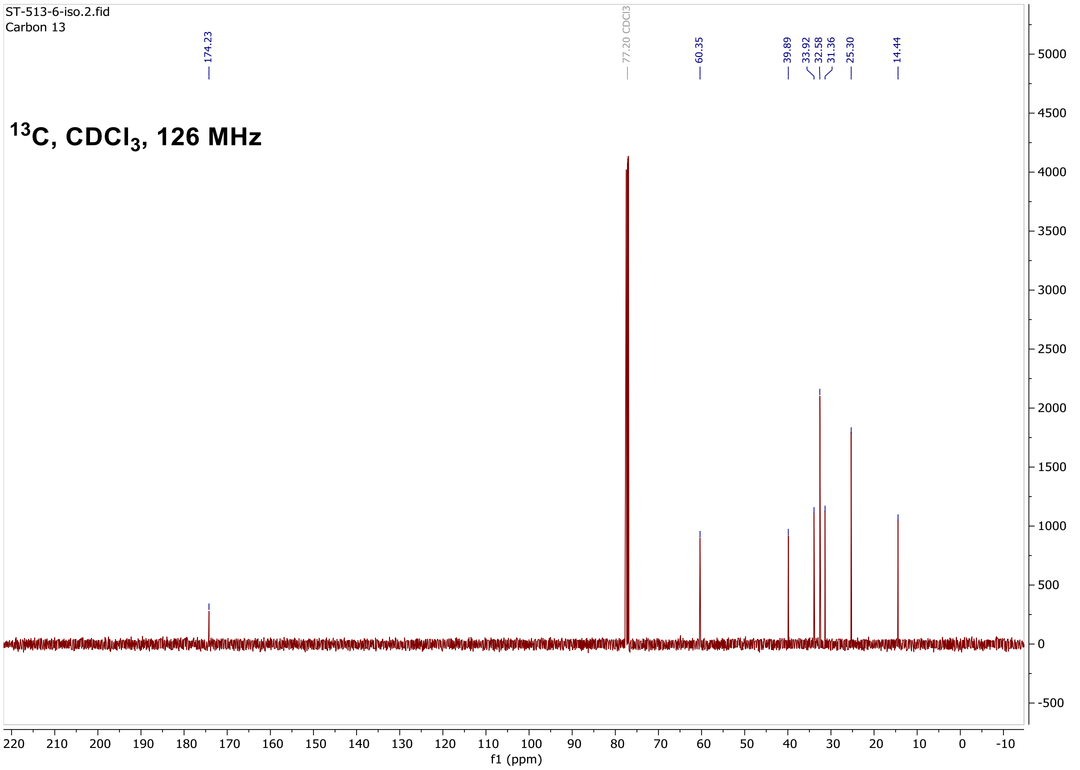
[4] Dondi, D.; Cardarelli, A. M.; Fagnoni, M.; Albini, A., *Tetrahedron* **2006,** *62*, 5527-5535.

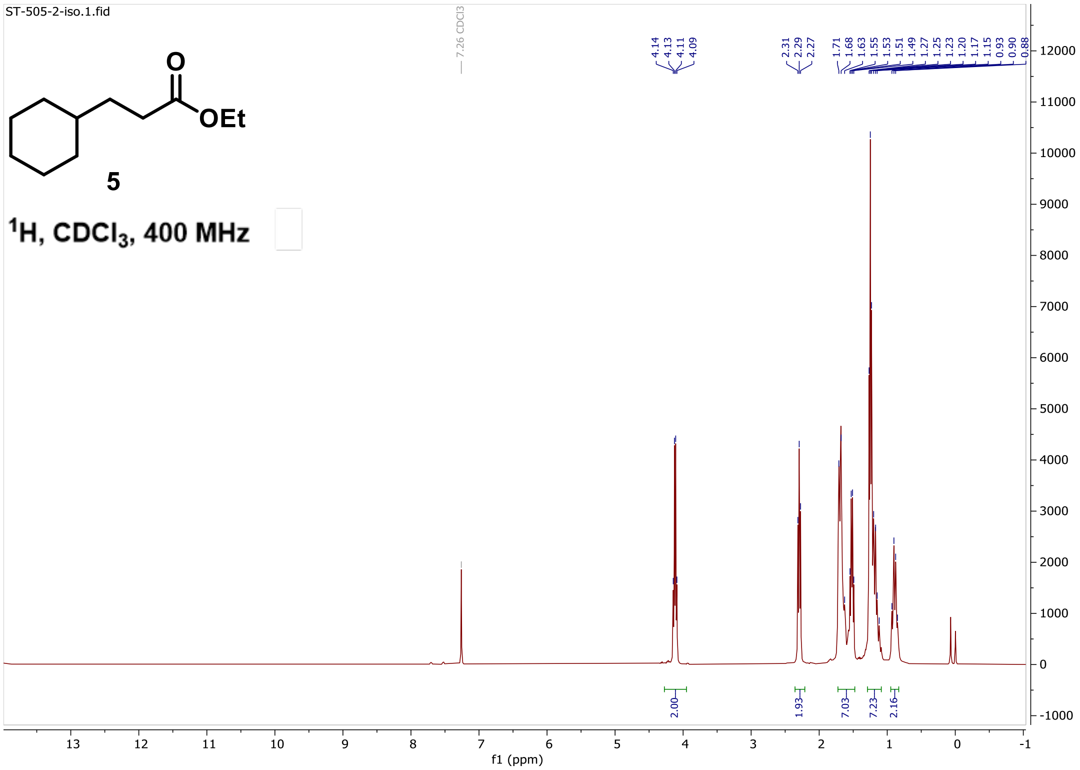
[5] Qrareya, H.; Ravelli, D.; Fagnoni, M.; Albini, A., *Advanced Synthesis & Catalysis* **2013,** *355*, 2891-2899.

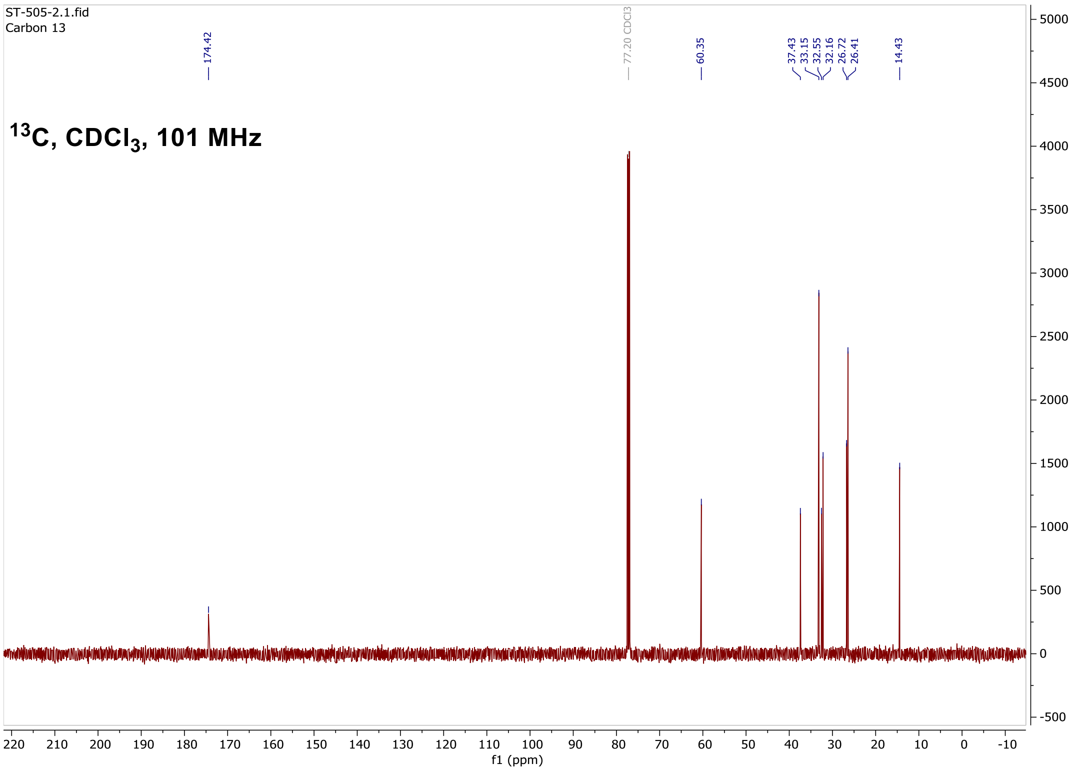
[6] Mizutani, H.; Degrado, S. J.; Hoveyda, A. H., *J. Am. Chem. Soc.* **2002,** *124*, 779-781.

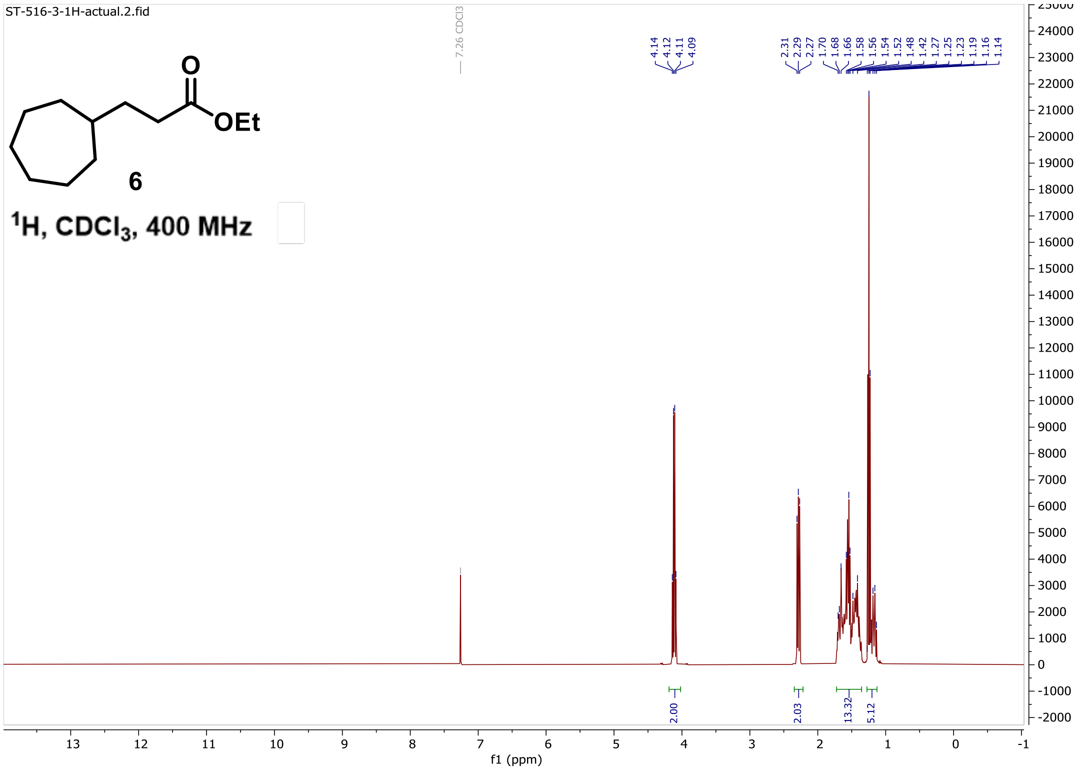
NMR Spectra:

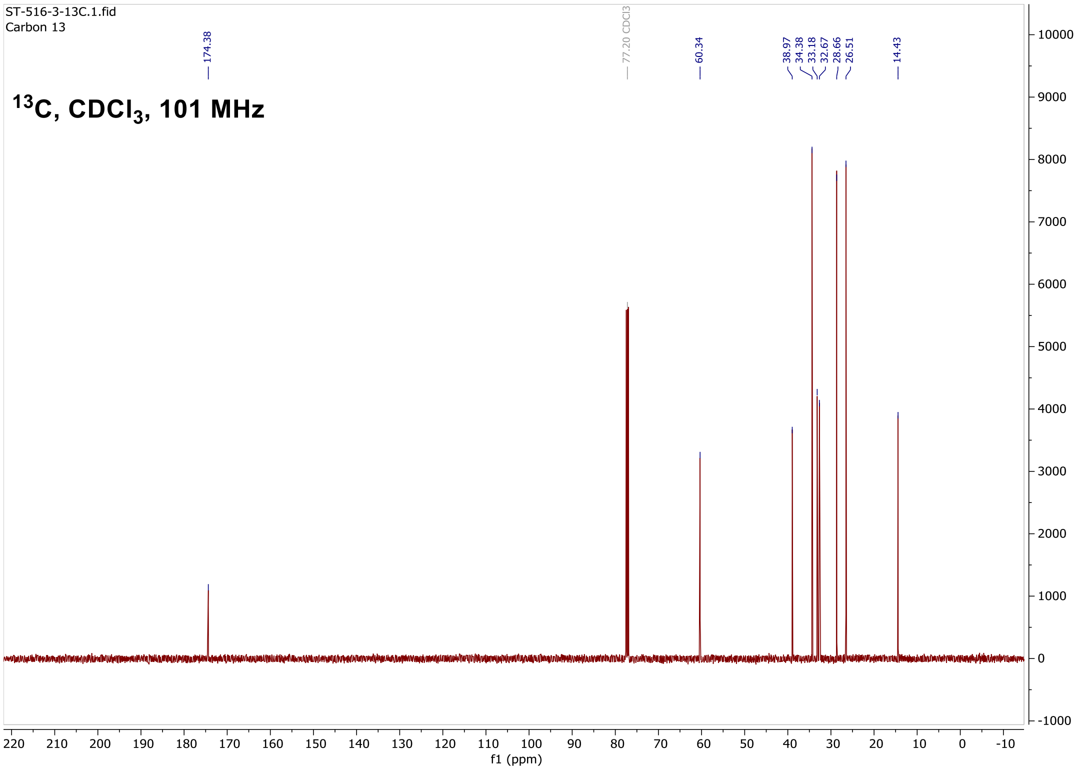

ST-513-6-iso.1.fid
Proton
4.14
4.13
4.11
4.10
2.32
2.30
2.29
1.77
1.76
1.76
1.74
1.66
1.64
1.62
1.61
1.60
1.59
1.52
1.51
1.51
1.50
1.27
1.25
1.24
1.09
O
OEt
4
^{1}H, CDCl$_3$, 500 MHz
2.00
2.03
3.01
4.20
2.04
3.11
2.07
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

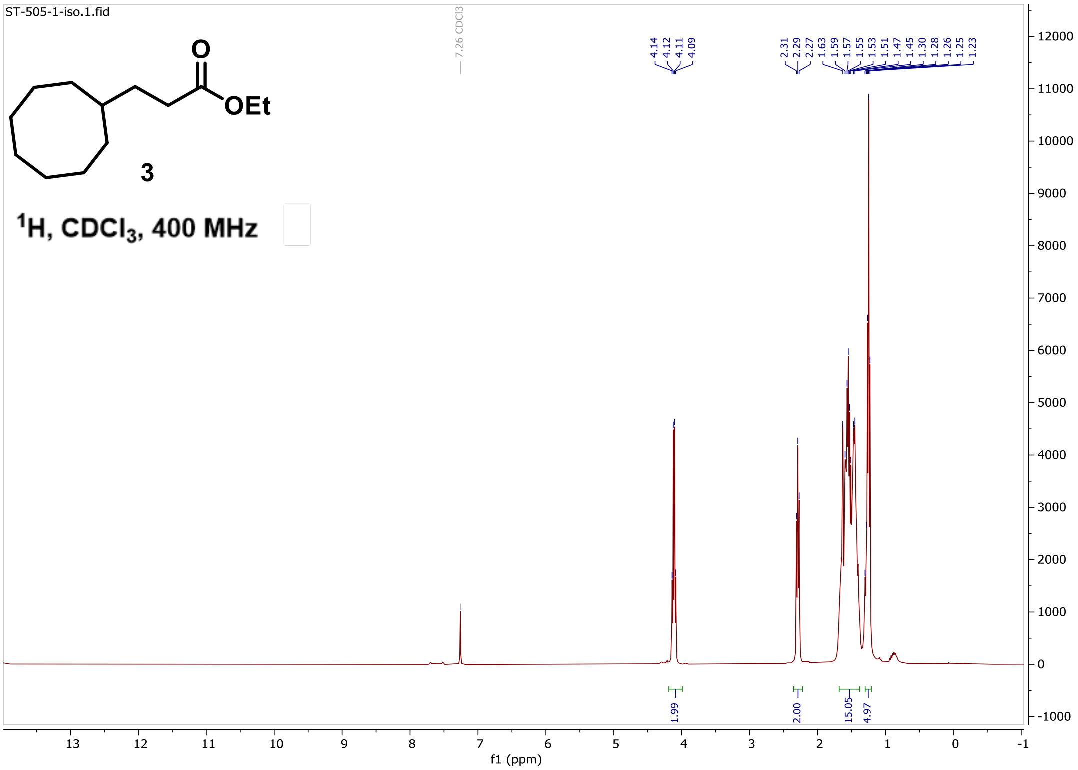

ST-513-6-iso.2.fid
Carbon 13
174.23
77.20 CDCl3
60.35
39.89
33.92
32.58
31.36
25.30
14.44
^{13}C, CDCl$_3$, 126 MHz
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

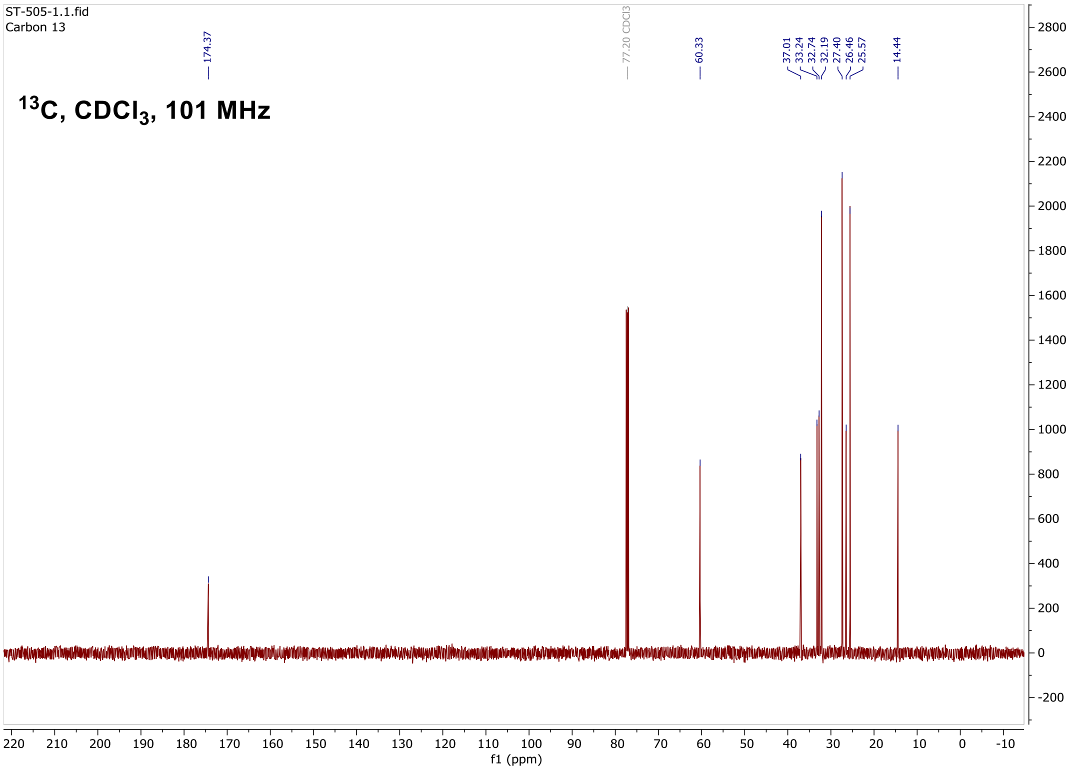

ST-505-2-iso.1.fid
O
OEt
5
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
4.14
4.13
4.11
4.09
2.31
2.29
2.27
1.71
1.68
1.63
1.55
1.53
1.51
1.49
1.27
1.25
1.23
1.20
1.17
1.15
0.93
0.90
0.88
2.00
1.93
7.03
7.23
2.16
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-505-2.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
174.42
77.20 CDCl3
60.35
37.43
33.15
32.55
32.16
26.72
26.41
14.43
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


ST-516-3-1H-actual.2.fid
O
OEt
6
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
4.14
4.12
4.11
4.09
2.31
2.29
2.27
1.70
1.68
1.66
1.58
1.56
1.54
1.52
1.48
1.42
1.27
1.25
1.23
1.19
1.16
1.14
2.00
2.03
13.32
5.12
25000
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-516-3-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
174.38
77.20 CDCl3
60.34
38.97
34.38
33.18
32.67
28.66
26.51
14.43
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


ST-505-1-iso.1.fid
O
OEt
3
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
4.14
4.12
4.11
4.09
2.31
2.29
2.27
1.63
1.59
1.57
1.55
1.53
1.51
1.47
1.45
1.30
1.28
1.26
1.25
1.23
1.99
2.00
15.05
4.97
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)
12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000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Carbon 13
174.37
77.20 CDCl3
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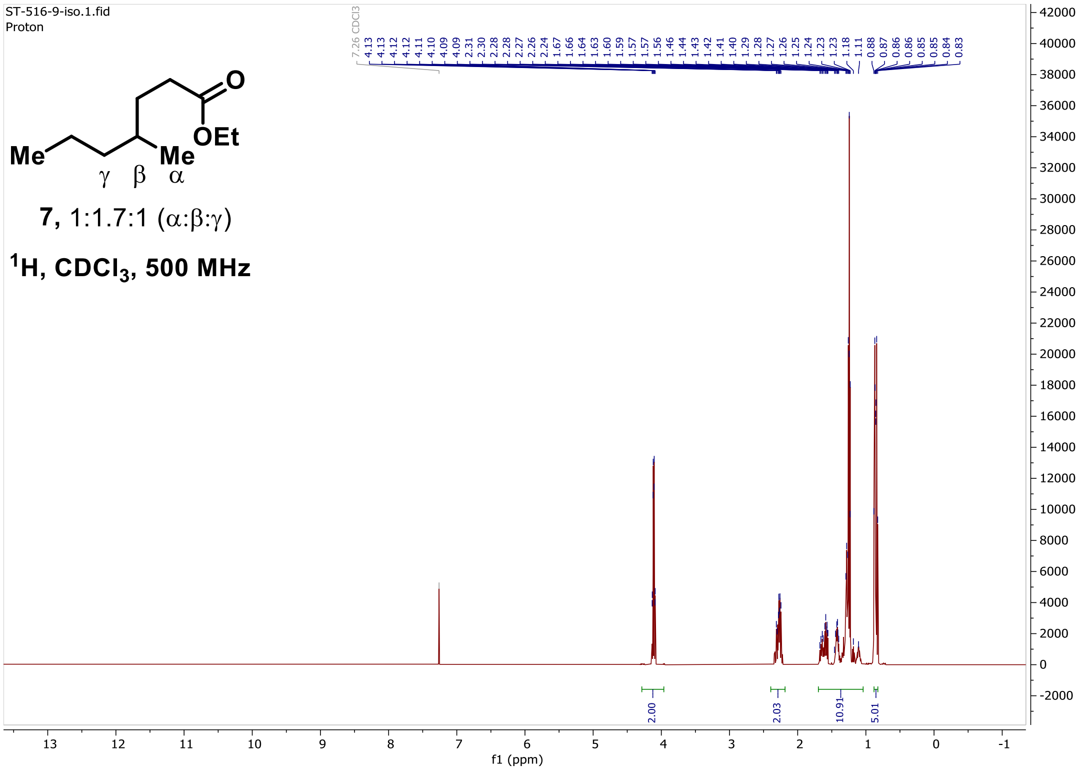
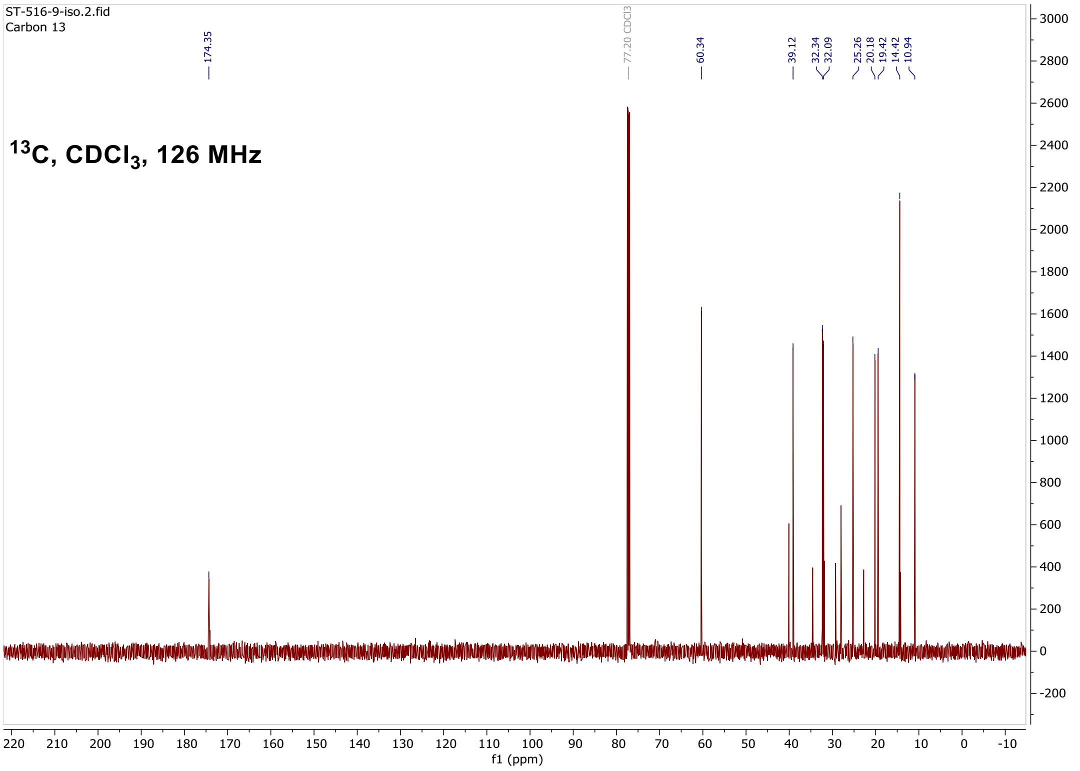
37.01
33.24
32.74
32.19
27.40
26.46
25.57
14.44
^{13}C, CDCl$_3$, 101 MHz
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200


ST-516-9-iso.1.fid
Proton
Me
Me
O
OEt
γ β α
7, 1:1.7:1 (α:β:γ)
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
4.13
4.13
4.12
4.12
4.11
4.10
4.09
4.09
2.31
2.30
2.28
2.28
2.27
2.26
2.24
1.67
1.66
1.64
1.63
1.60
1.59
1.57
1.57
1.56
1.46
1.44
1.43
1.42
1.41
1.40
1.29
1.28
1.27
1.26
1.25
1.24
1.23
1.23
1.18
1.11
0.88
0.87
0.86
0.86
0.85
0.85
0.84
0.83
2.00
2.03
10.91
5.01
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)
42000
40000
38000
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000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Carbon 13
^{13}C, CDCl$_3$, 126 MHz
174.35
77.20 CDCl3
60.34
39.12
32.34
32.09
25.26
20.18
19.42
14.42
10.94
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
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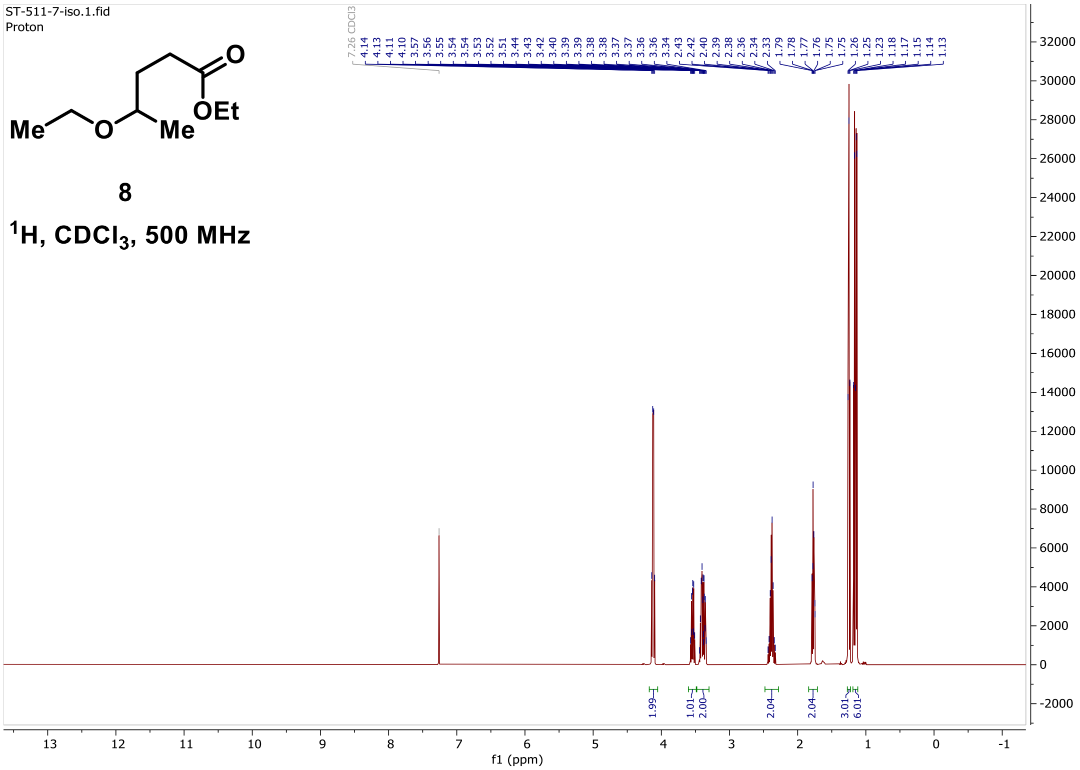
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200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Proton
Me
O
Me
O
OEt
8
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
4.14
4.13
4.11
4.10
3.57
3.56
3.55
3.54
3.54
3.53
3.52
3.51
3.44
3.43
3.42
3.40
3.39
3.39
3.38
3.38
3.37
3.37
3.36
3.36
3.34
2.43
2.42
2.40
2.39
2.38
2.36
2.34
2.33
1.79
1.78
1.77
1.76
1.75
1.75
1.26
1.25
1.23
1.18
1.17
1.15
1.14
1.13
1.99
1.01
2.00
2.04
2.04
3.01
6.01
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
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8
7
6
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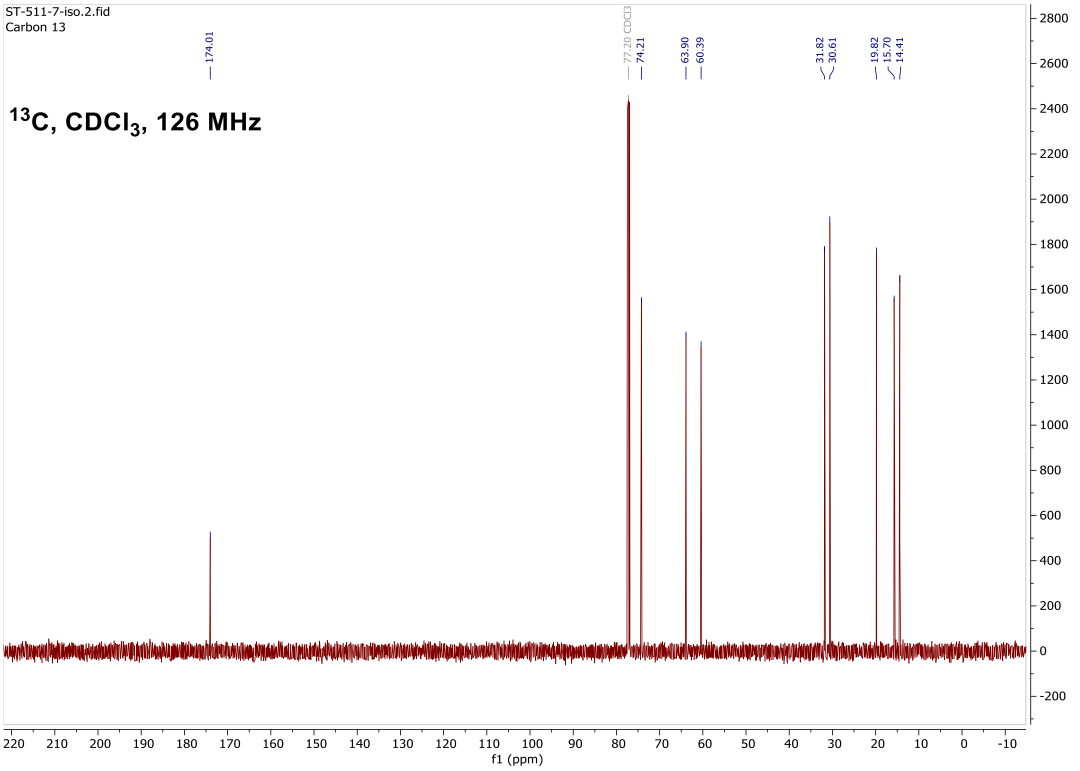
4
3
2
1
0
-1
f1 (ppm)


ST-511-7-iso.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
174.01
77.20 CDCl3
74.21
63.90
60.39
31.82
30.61
19.82
15.70
14.41
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220
210
200
190
180
170
160
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140
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120
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100
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80
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60
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40
30
20
10
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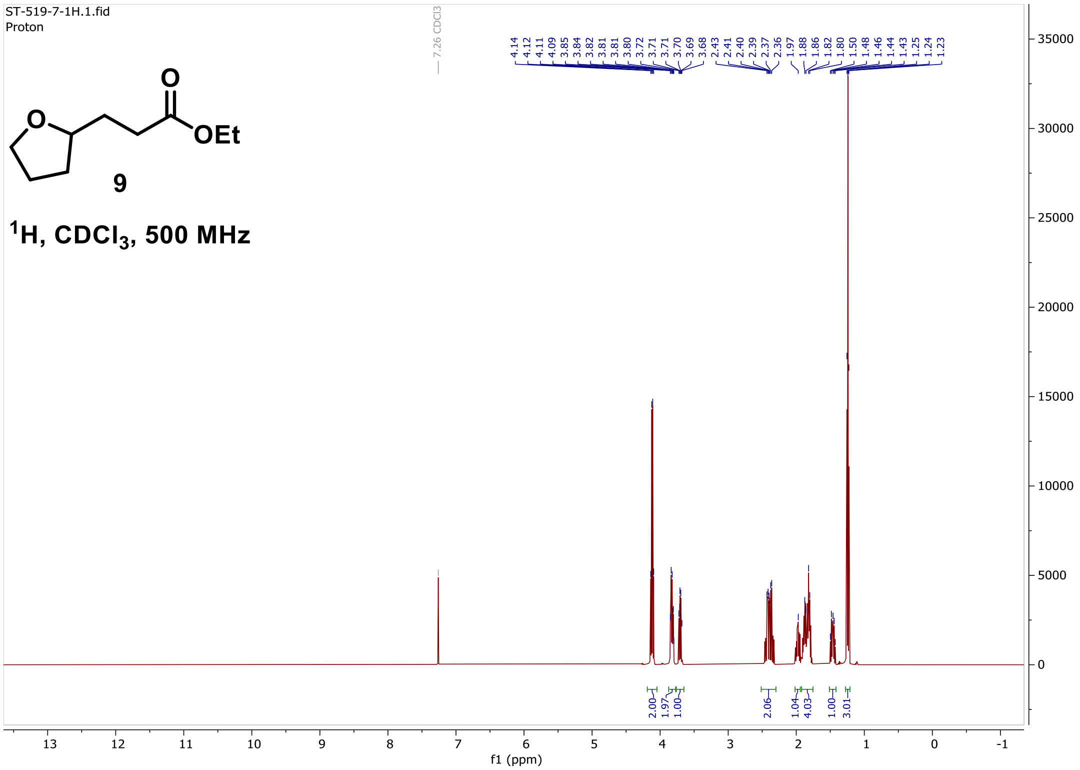
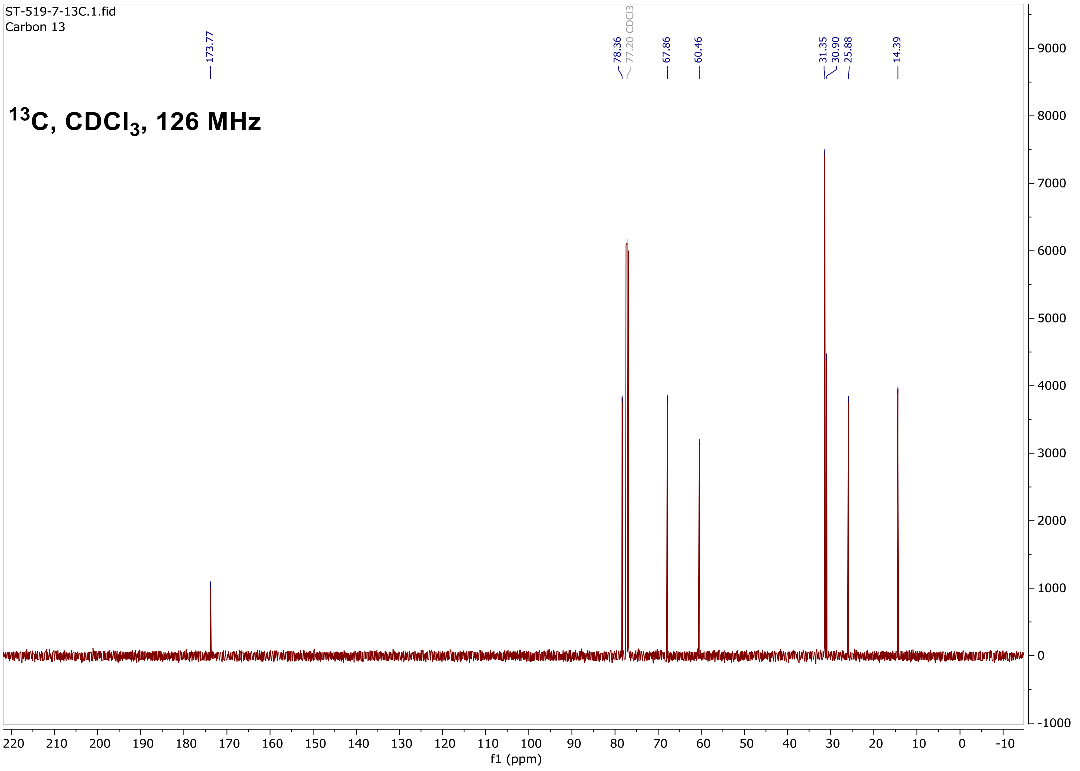
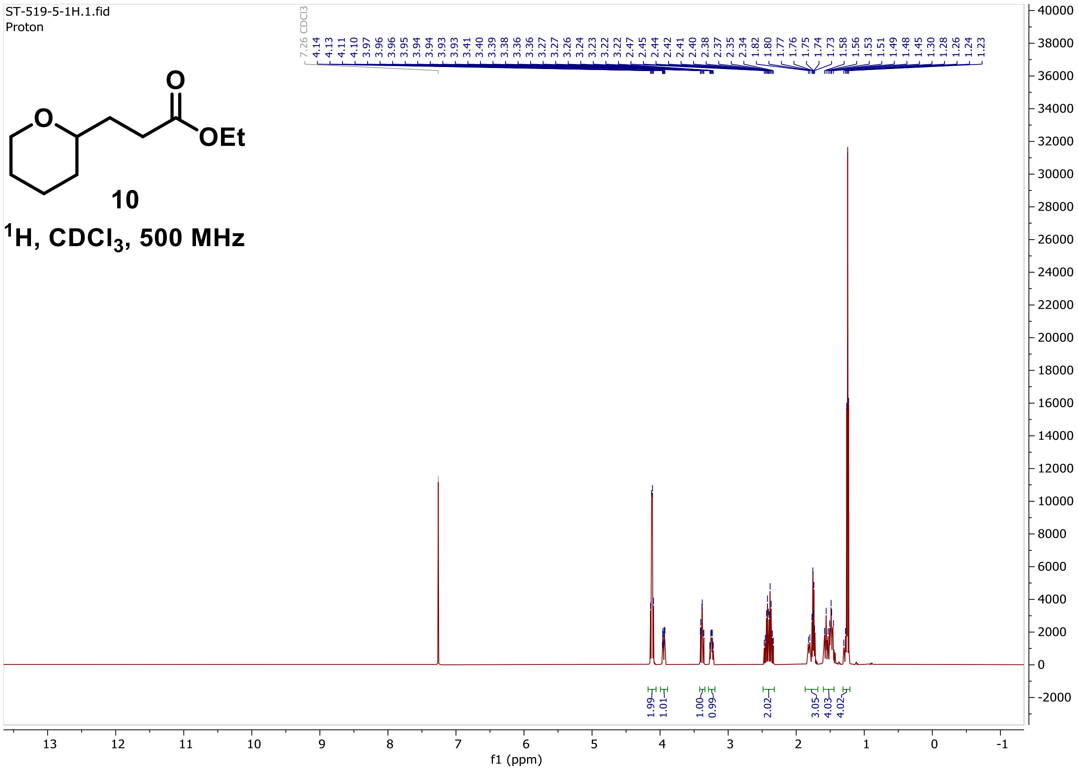
f1 (ppm)

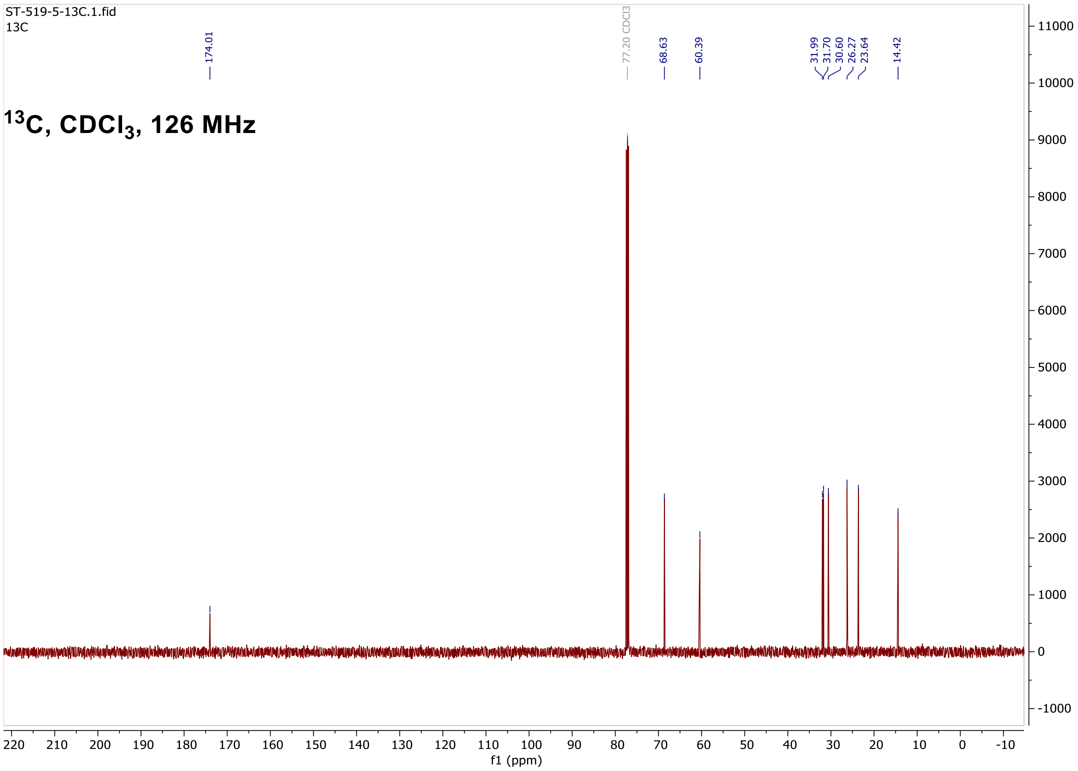

ST-519-7-1H.1.fid
Proton
O
OEt
9
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
4.14 4.12 4.11 4.09 3.85 3.84 3.82 3.81 3.81 3.80 3.72 3.71 3.71 3.70 3.69 3.68 2.43 2.41 2.40 2.39 2.37 2.36 1.97 1.88 1.86 1.82 1.80 1.50 1.48 1.46 1.44 1.43 1.25 1.24 1.23
2.00 1.97 1.00 2.06 1.04 4.03 1.00 3.01
35000 30000 25000 20000 15000 10000 5000 0
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


ST-519-7-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
173.77
78.36
77.20 CDCl3
67.86
60.46
31.35
30.90
25.88
14.39
9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

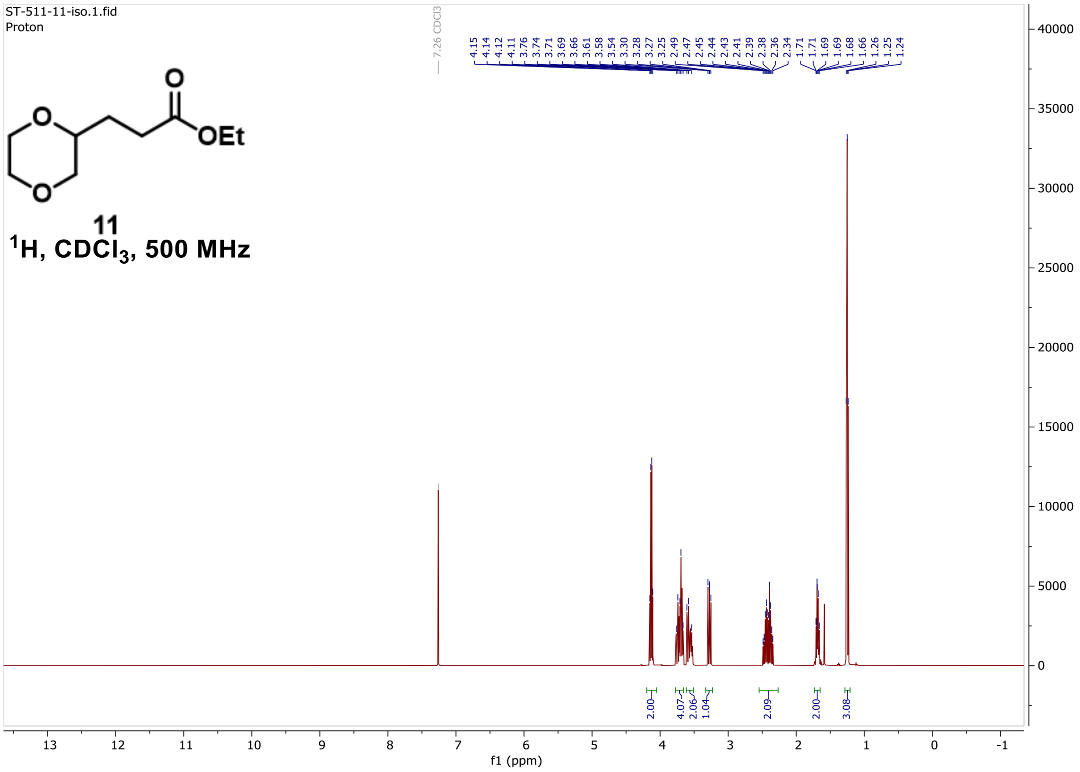


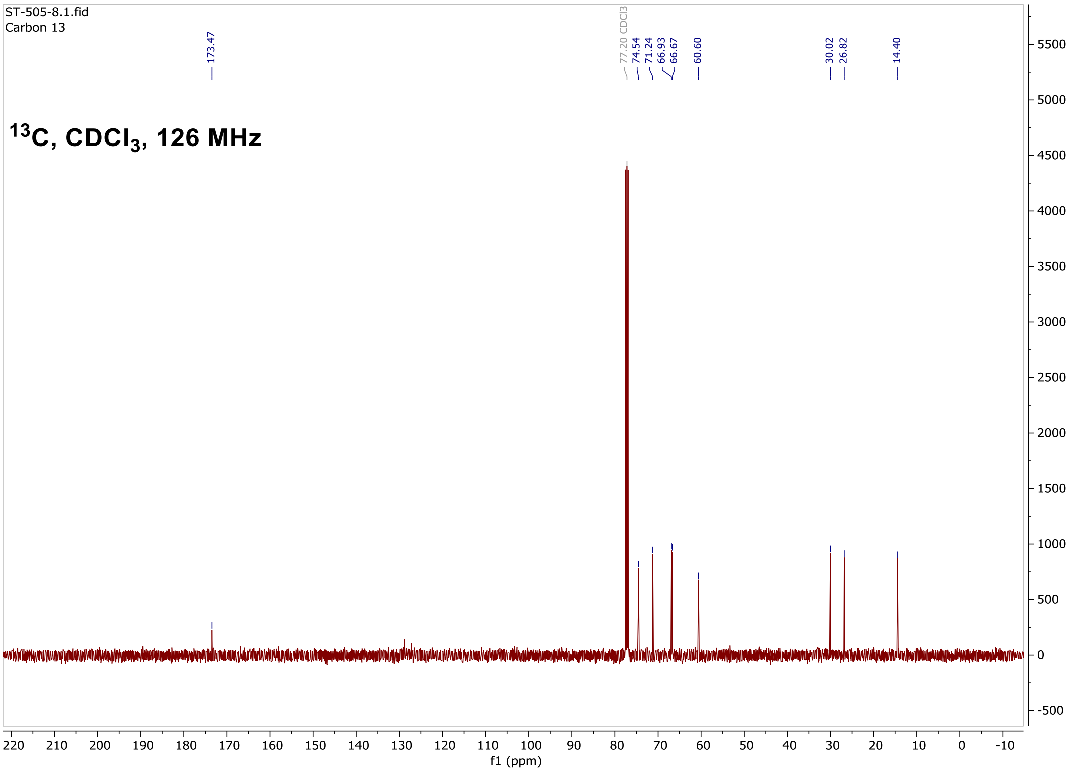
10

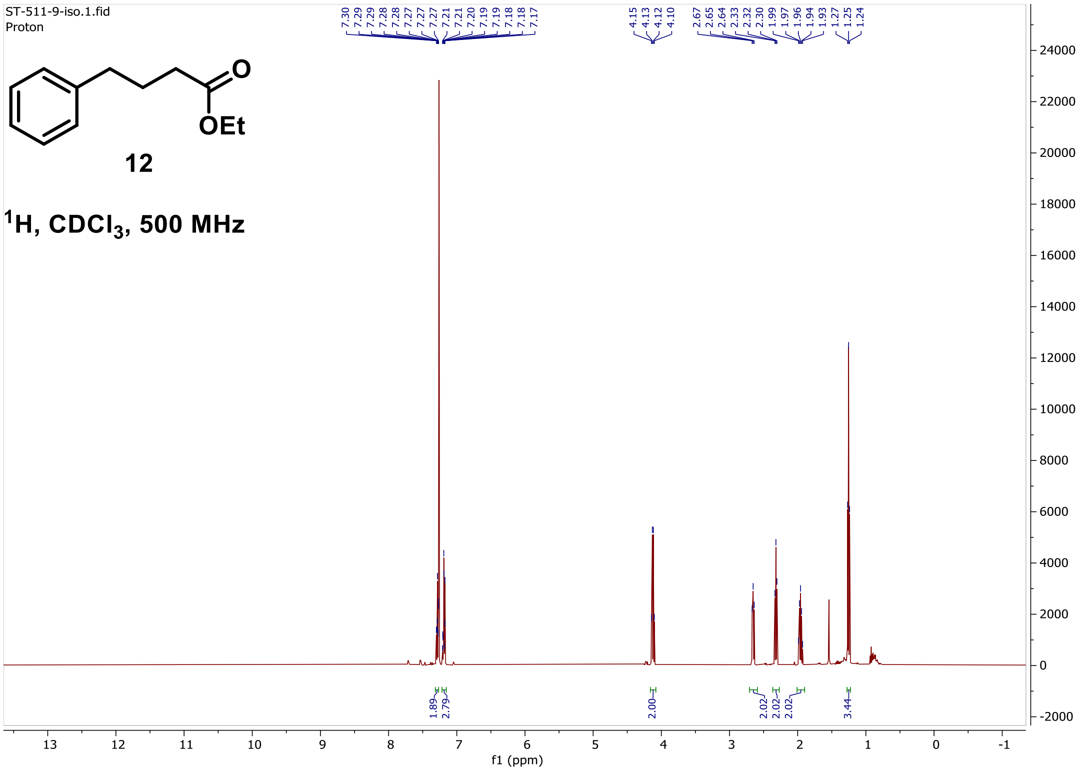
^{1}H, CDCl$_3$, 500 MHz



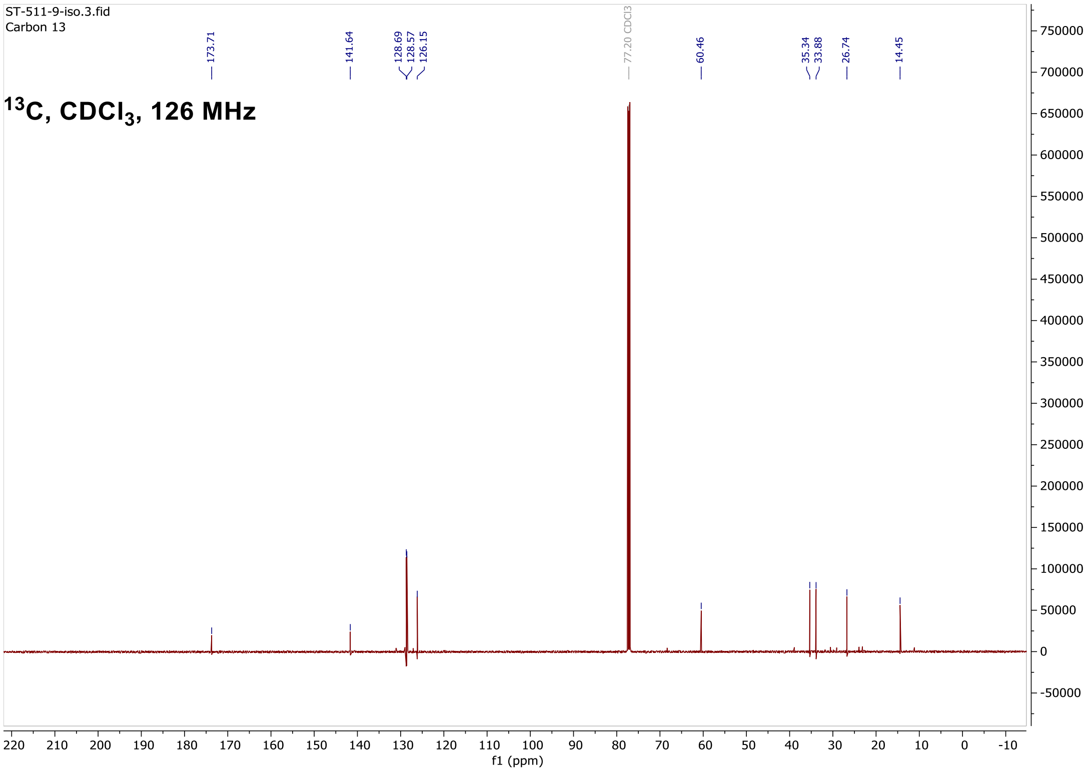
^{13}C, CDCl$_3$, 126 MHz


ST-511-11-iso.1.fid
Proton
11
^{1}H, CDCl$_3$, 500 MHz
O
OEt
7.26 CDCl3
4.15 4.14 4.12 4.11 3.76 3.74 3.71 3.69 3.66 3.61 3.58 3.54 3.30 3.28 3.27 3.25 2.49 2.47 2.45 2.44 2.43 2.41 2.39 2.38 2.36 2.34 1.71 1.71 1.69 1.69 1.68 1.66 1.26 1.25 1.24
2.00 4.07 2.06 1.04 2.09 2.00 3.08
f1 (ppm)
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
40000 35000 30000 25000 20000 15000 10000 5000 0

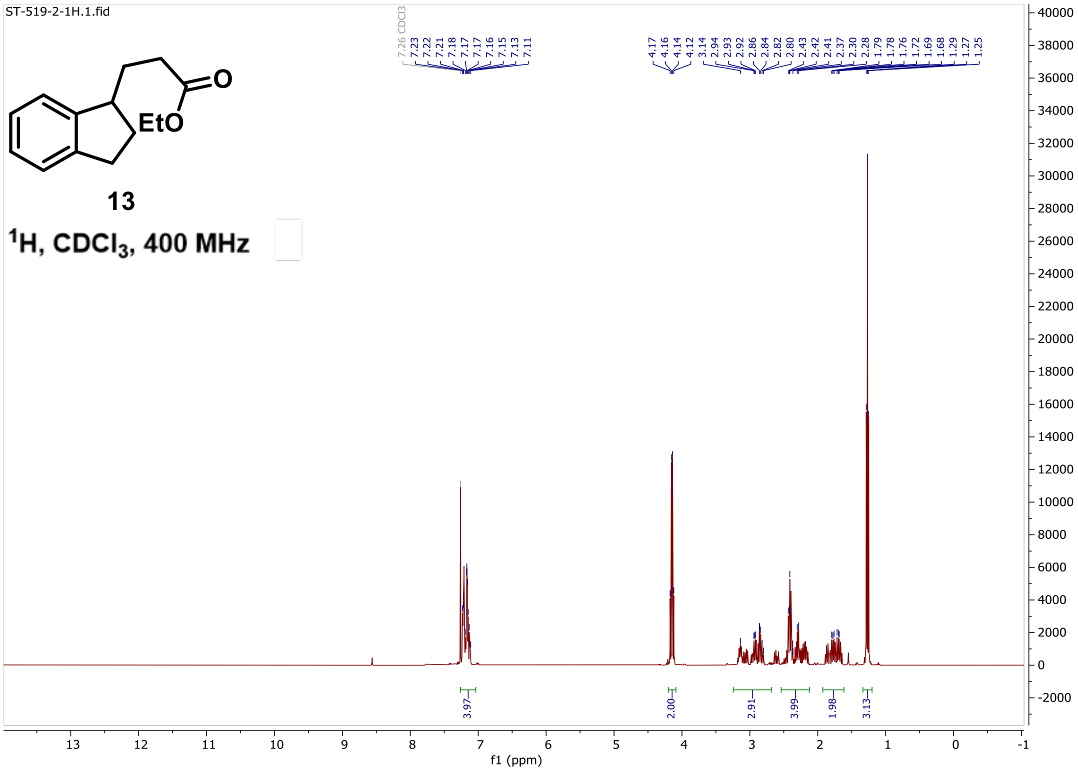

ST-505-8.1.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
173.47
77.20 CDCl3
74.54 71.24 66.93 66.67 60.60
30.02 26.82
14.40
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

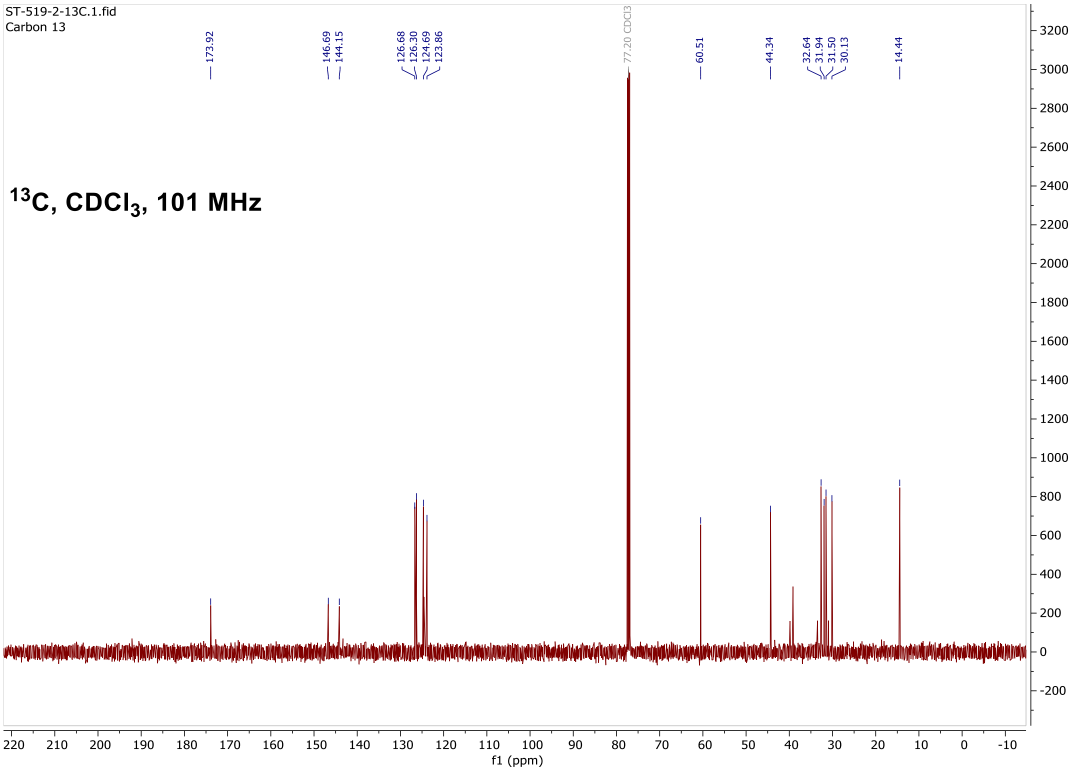


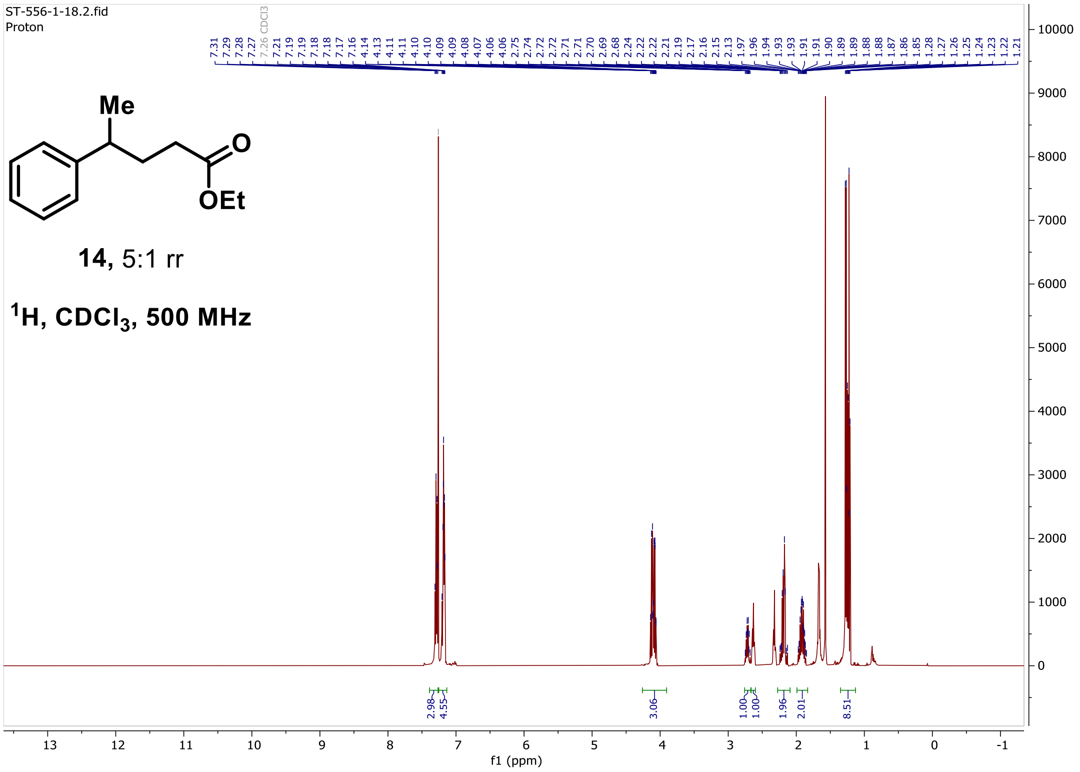
ST-511-9-iso.1.fid
Proton
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.21
7.21
7.20
7.19
7.19
7.18
7.18
7.17
4.15
4.13
4.12
4.10
2.67
2.65
2.64
2.33
2.32
2.30
1.99
1.97
1.96
1.94
1.93
1.27
1.25
1.24
O
OEt
12
¹H, CDCl₃, 500 MHz
1.89
2.79
2.00
2.02
2.02
2.02
3.44
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

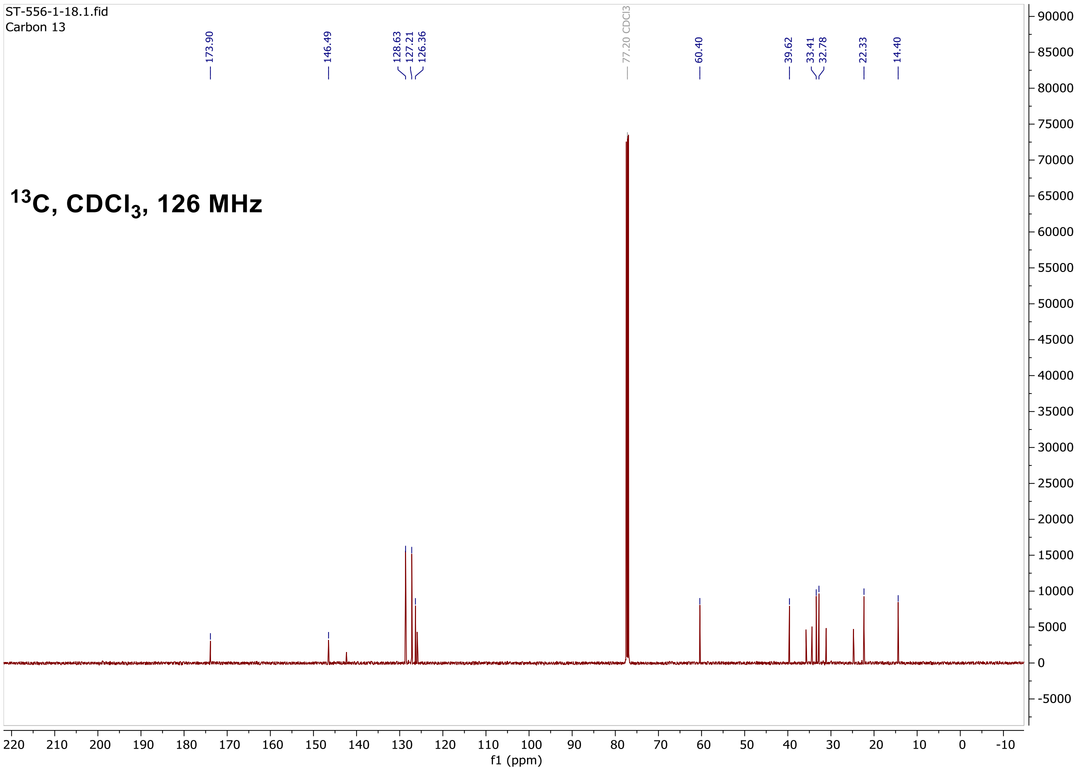


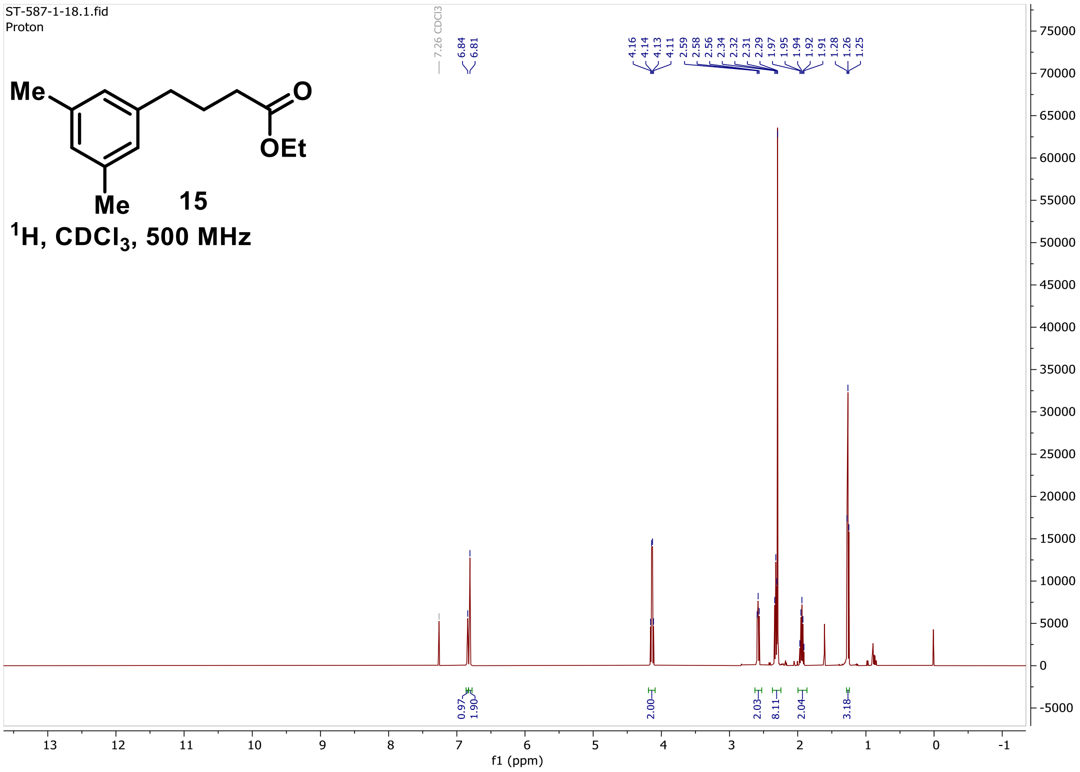
ST-511-9-iso.3.fid
Carbon 13
173.71
141.64
128.69
128.57
126.15
77.20 CDCl3
60.46
35.34
33.88
26.74
14.45
¹³C, CDCl₃, 126 MHz
750000
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

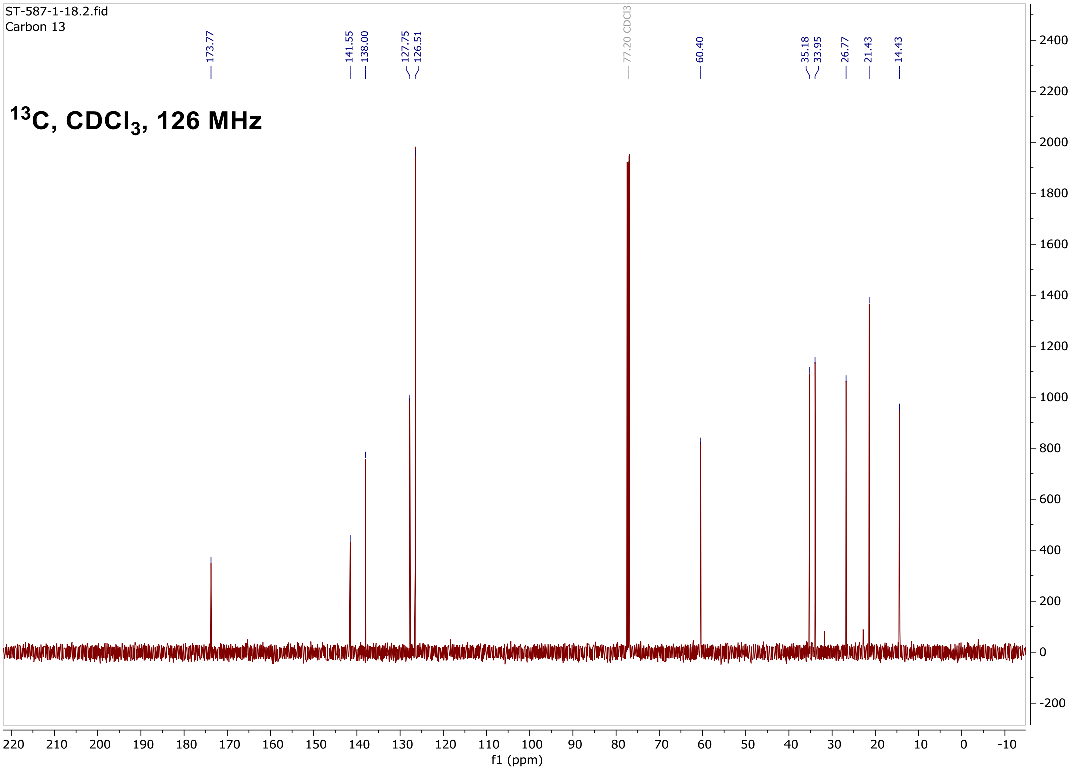

ST-519-2-1H.1.fid
EtO
O
13
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
7.23
7.22
7.21
7.18
7.17
7.17
7.16
7.15
7.13
7.11
4.17
4.16
4.14
4.12
3.14
2.94
2.93
2.92
2.86
2.84
2.82
2.80
2.43
2.42
2.41
2.37
2.30
2.28
1.79
1.78
1.76
1.72
1.69
1.68
1.29
1.27
1.25
3.97
2.00
2.91
3.99
1.98
3.13
40000
38000
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

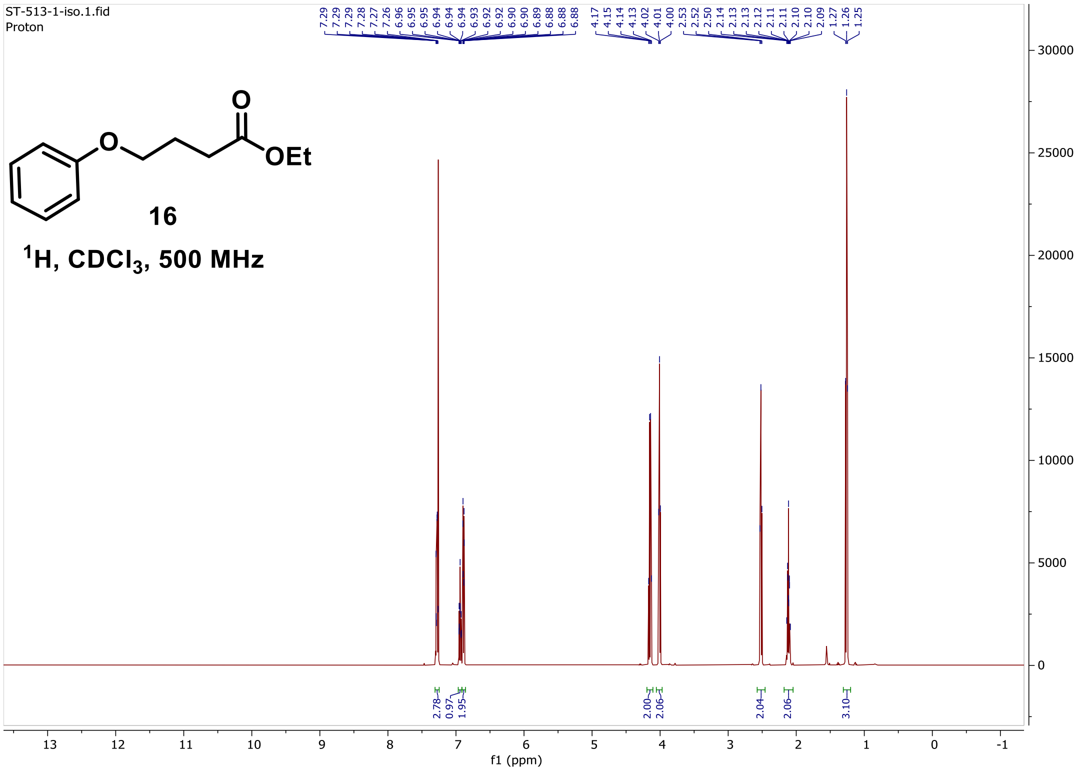

ST-519-2-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
173.92
146.69
144.15
126.68
126.30
124.69
123.86
77.20 CDCl3
60.51
44.34
32.64
31.94
31.50
30.13
14.44
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

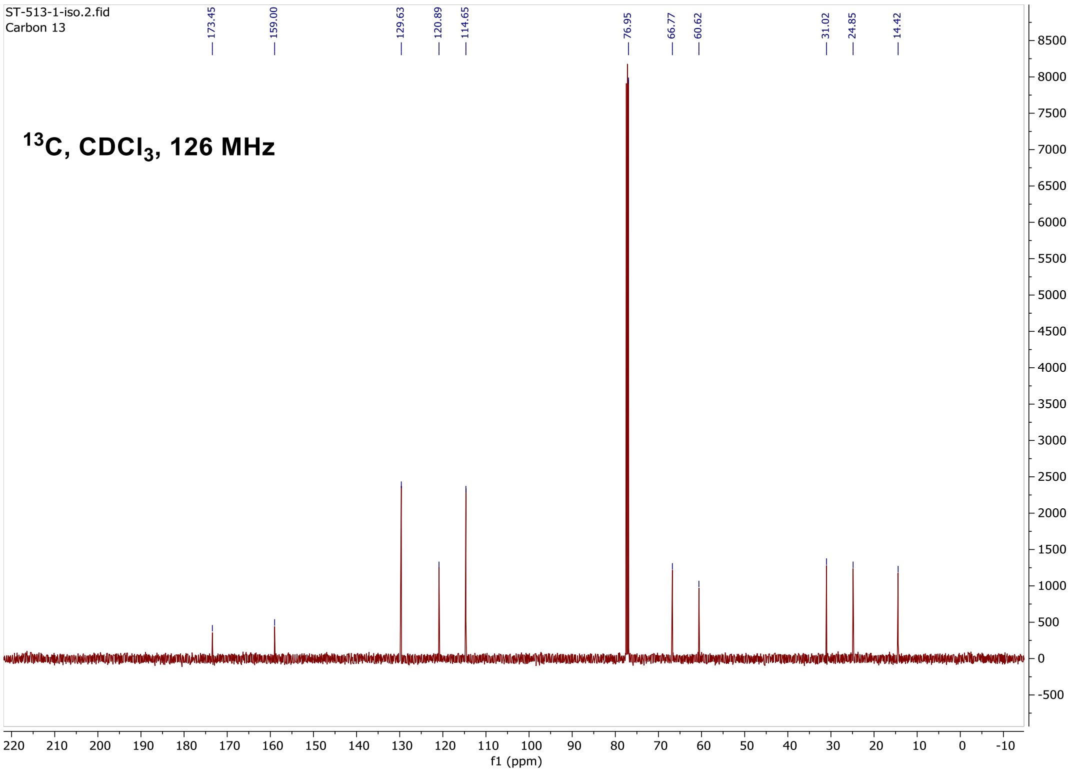

ST-556-1-18.2.fid
Proton
7.31
7.29
7.28
7.27
7.26 CDCl3
7.21
7.19
7.19
7.18
7.18
7.17
7.16
4.14
4.13
4.11
4.11
4.10
4.10
4.09
4.09
4.08
4.07
4.06
4.06
2.75
2.74
2.72
2.72
2.71
2.71
2.70
2.69
2.68
2.24
2.22
2.22
2.21
2.19
2.17
2.16
2.15
2.13
1.97
1.96
1.94
1.93
1.93
1.91
1.91
1.90
1.89
1.89
1.88
1.88
1.87
1.86
1.85
1.28
1.27
1.26
1.25
1.24
1.23
1.22
1.21
Me
O
OEt
14, 5:1 rr
^{1}H, CDCl$_3$, 500 MHz
2.98
4.55
3.06
1.00
1.00
1.96
2.01
8.51
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0

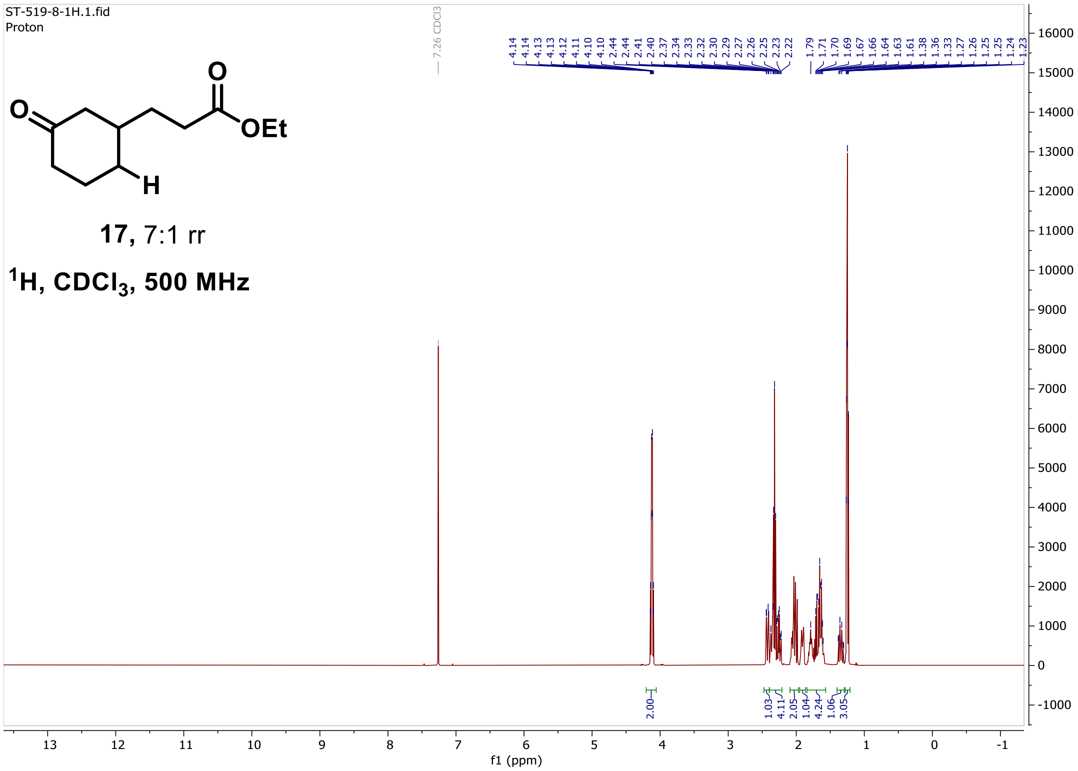

ST-556-1-18.1.fid
Carbon 13
173.90
146.49
128.63
127.21
126.36
77.20 CDCl3
60.40
39.62
33.41
32.78
22.33
14.40
^{13}C, CDCl$_3$, 126 MHz
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
90000
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000


ST-587-1-18.1.fid
Proton
Me
Me
O
OEt
15
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
6.84
6.81
4.16
4.14
4.13
4.11
2.59
2.58
2.56
2.34
2.32
2.31
2.29
1.97
1.95
1.94
1.92
1.91
1.28
1.26
1.25
0.97
1.90
2.00
2.03
8.11
2.04
3.18
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


ST-587-1-18.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
173.77
141.55
138.00
127.75
126.51
77.20 CDCl3
60.40
35.18
33.95
26.77
21.43
14.43
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

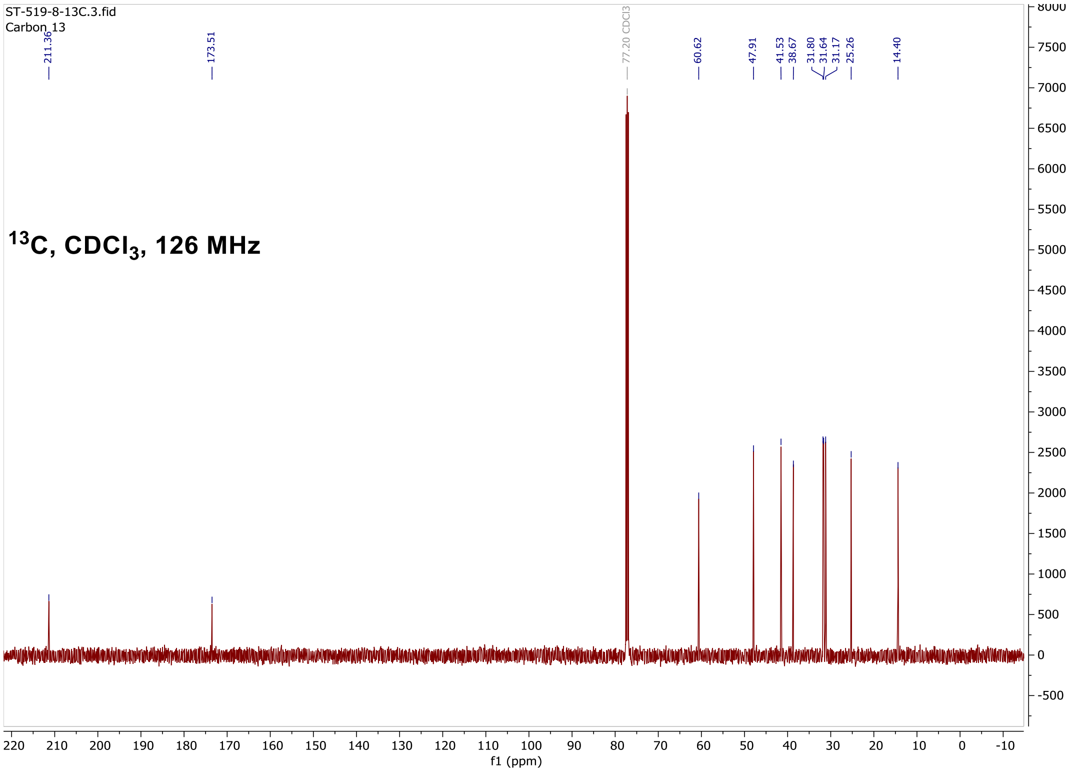

ST-513-1-iso.1.fid
Proton
7.29 7.29 7.29 7.28 7.27 7.26 6.96 6.95 6.95 6.94 6.94 6.94 6.93 6.92 6.92 6.90 6.90 6.89 6.88 6.88 6.88 4.17 4.15 4.14 4.13 4.02 4.01 4.00 2.53 2.52 2.50 2.14 2.13 2.13 2.12 2.11 2.11 2.10 2.10 2.09 1.27 1.26 1.25
O
O
OEt
16
^{1}H, CDCl$_3$, 500 MHz
2.78 0.97 1.95 2.00 2.06 2.04 2.06 3.10
30000 25000 20000 15000 10000 5000 0
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


ST-513-1-iso.2.fid
Carbon 13
173.45 159.00 129.63 120.89 114.65 76.95 66.77 60.62 31.02 24.85 14.42
^{13}C, CDCl$_3$, 126 MHz
8500 8000 7500 7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

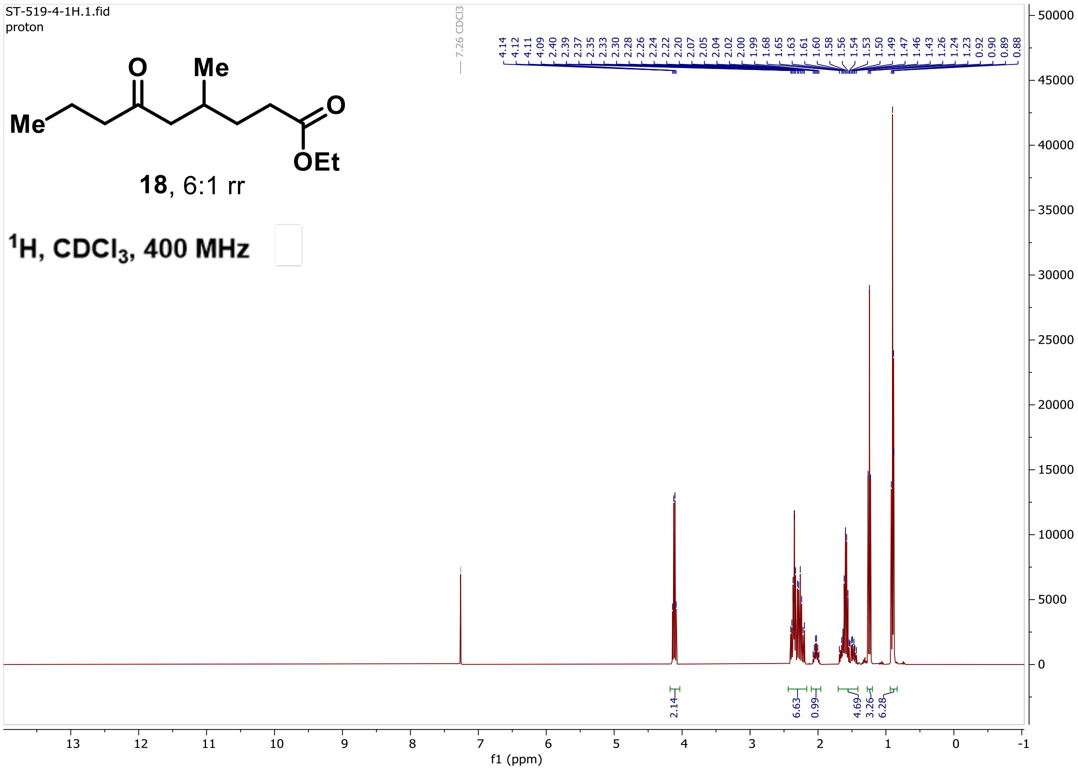


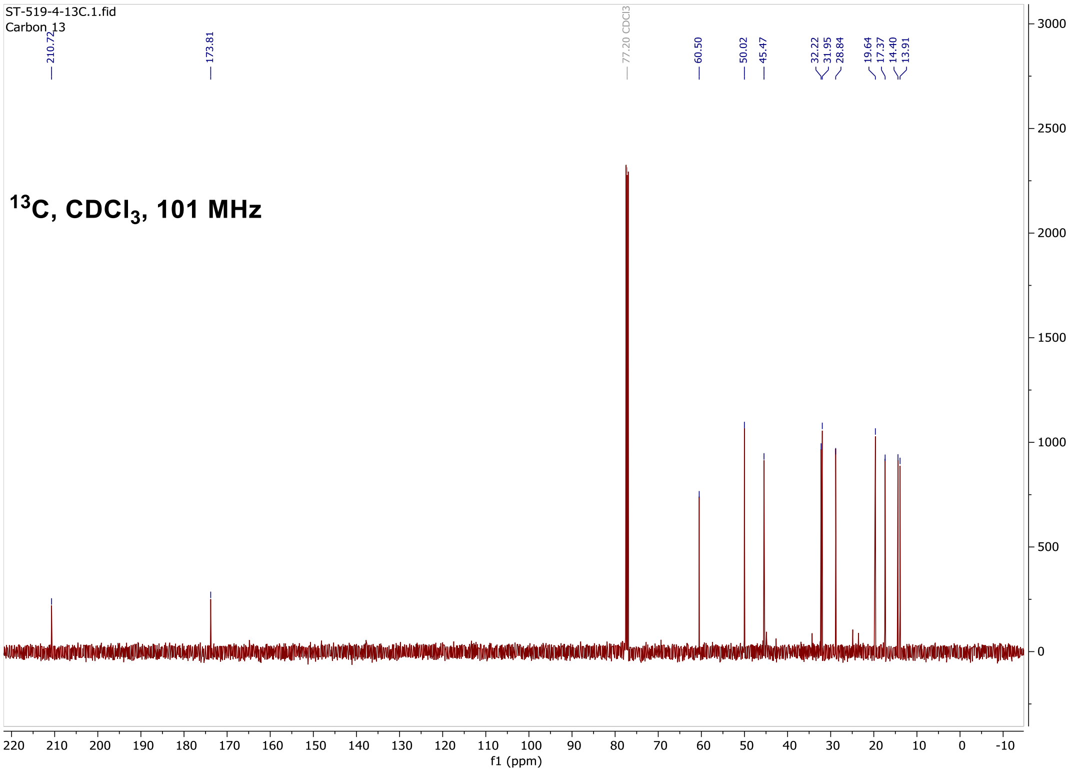
17, 7:1 rr

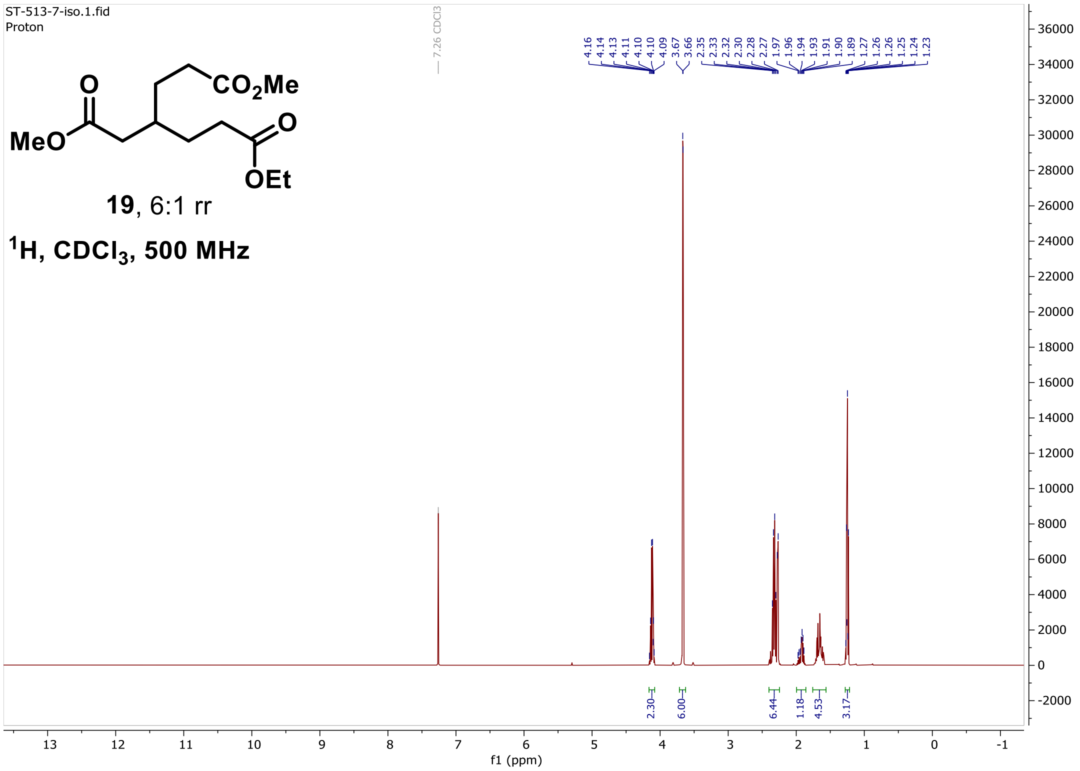
ST-519-8-1H.1.fid
Proton
O
O
OEt
H
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
4.14
4.14
4.13
4.13
4.12
4.11
4.10
4.10
2.44
2.44
2.41
2.40
2.37
2.34
2.33
2.32
2.30
2.29
2.27
2.26
2.25
2.23
2.22
1.79
1.71
1.70
1.69
1.67
1.66
1.64
1.63
1.61
1.38
1.36
1.33
1.27
1.26
1.25
1.25
1.24
1.23
2.00
1.03
4.11
2.05
1.04
4.24
1.06
3.05
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

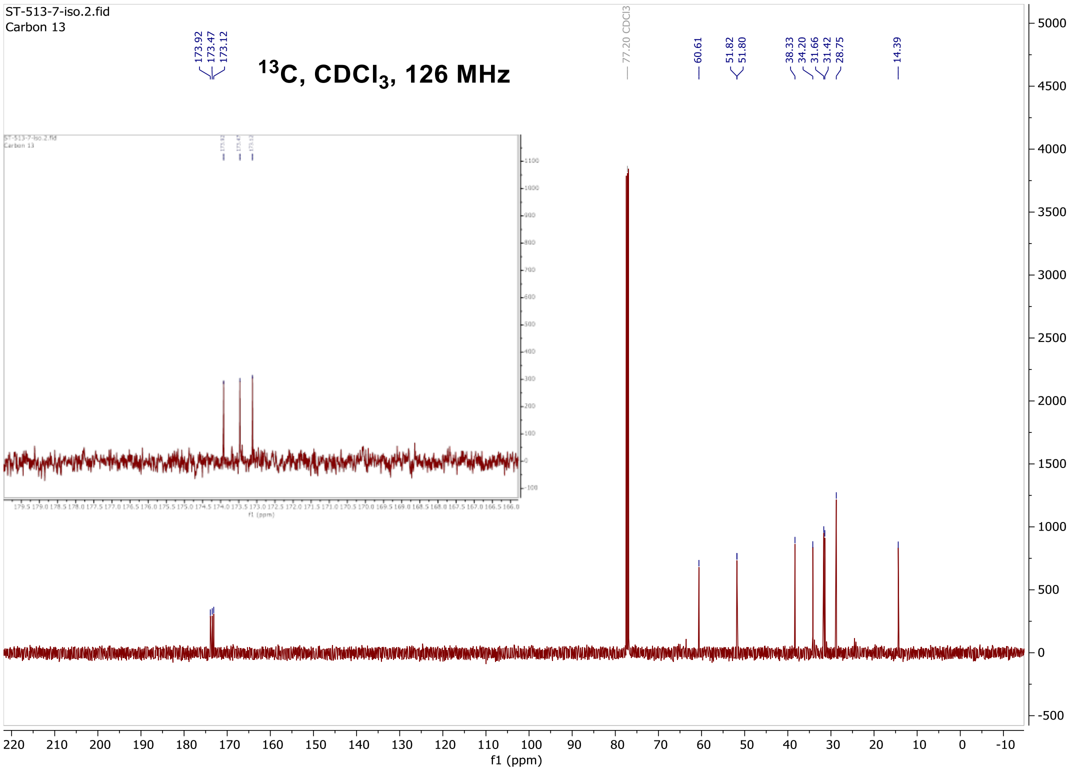


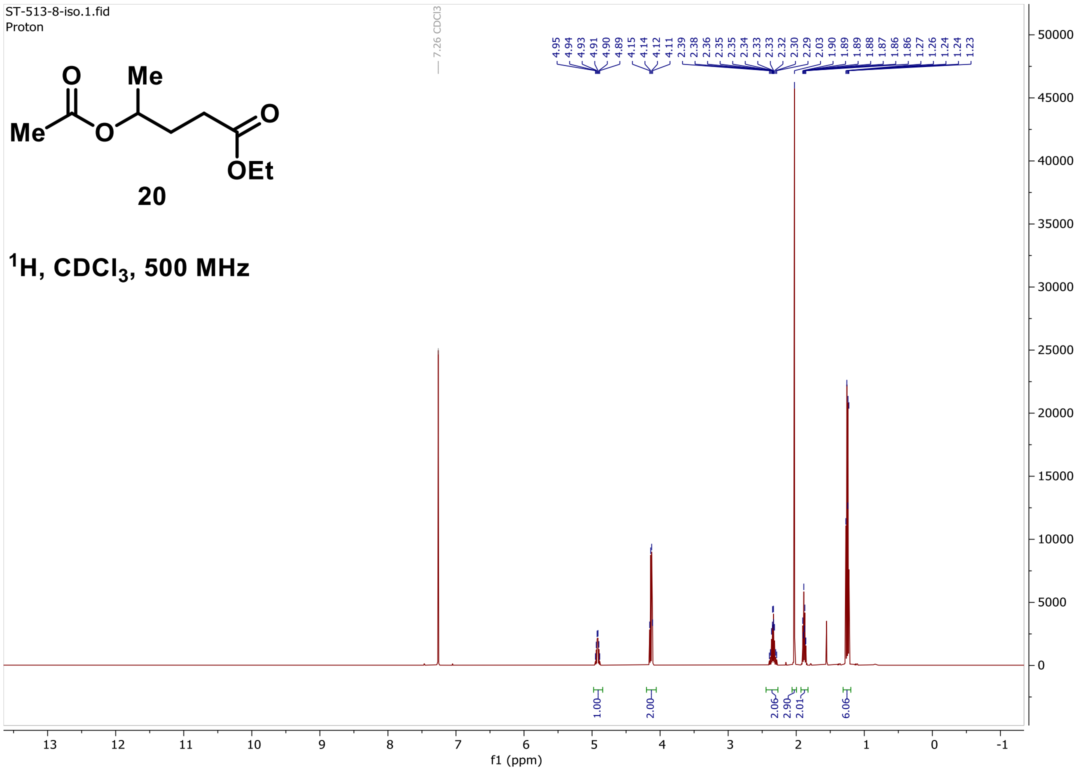
ST-519-8-13C.3.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
211.36
173.51
77.20 CDCl3
60.62
47.91
41.53
38.67
31.80
31.64
31.17
25.26
14.40
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

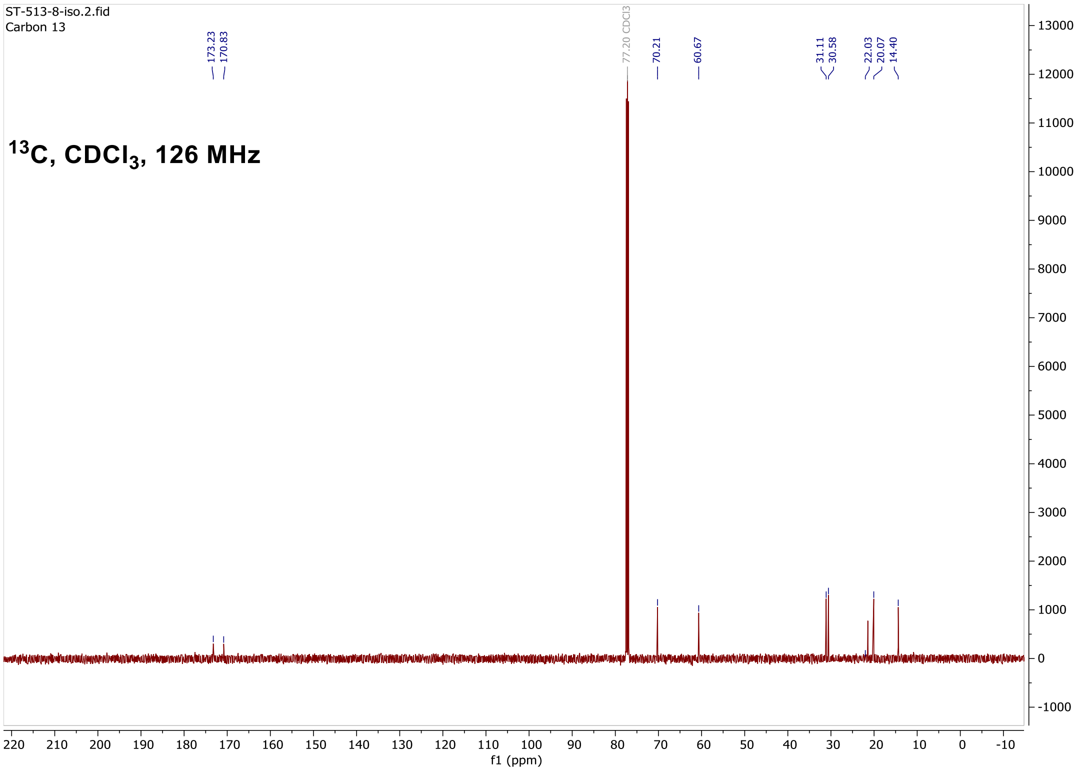

ST-519-4-1H.1.fid
proton
Me
O
Me
O
OEt
18, 6:1 rr
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
4.14 4.12 4.11 4.09 2.40 2.39 2.37 2.35 2.33 2.30 2.28 2.26 2.24 2.22 2.20 2.07 2.05 2.04 2.02 2.00 1.99 1.68 1.65 1.63 1.61 1.60 1.58 1.56 1.54 1.53 1.50 1.49 1.47 1.46 1.43 1.26 1.24 1.23 0.92 0.90 0.89 0.88
2.14
6.63
0.99
4.69
3.26
6.28
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

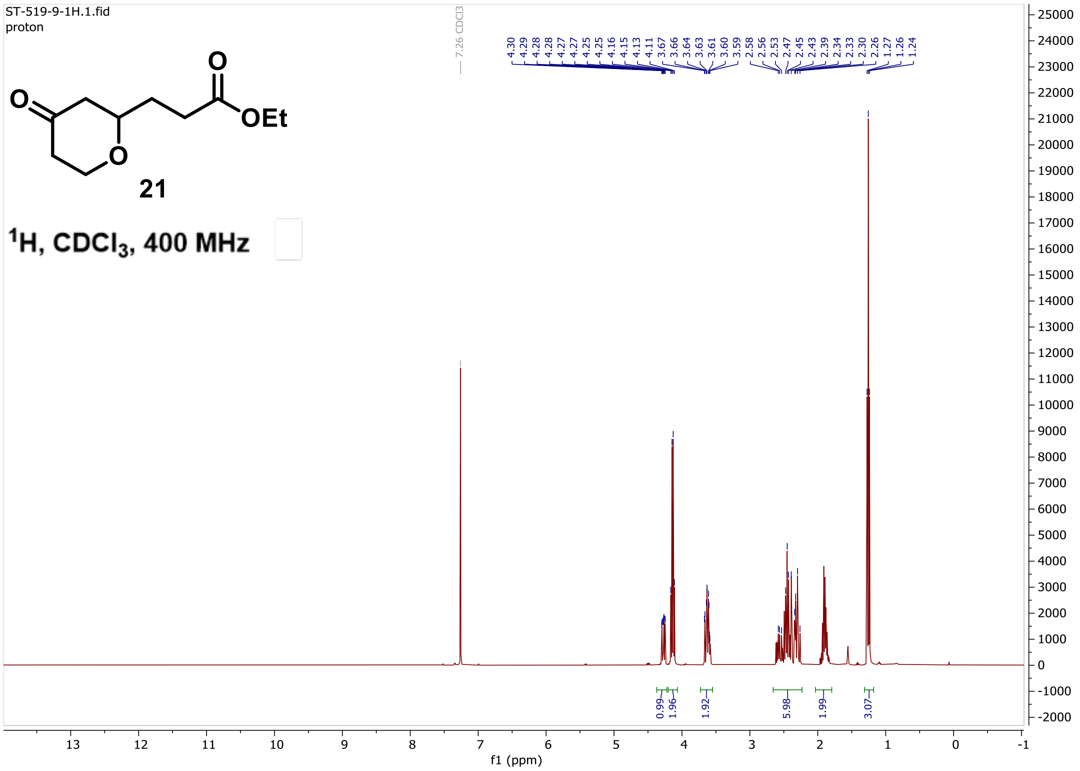

ST-519-4-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
210.72
173.81
77.20 CDCl3
60.50
50.02
45.47
32.22
31.95
28.84
19.64
17.37
14.40
13.91
3000
2500
2000
1500
1000
500
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

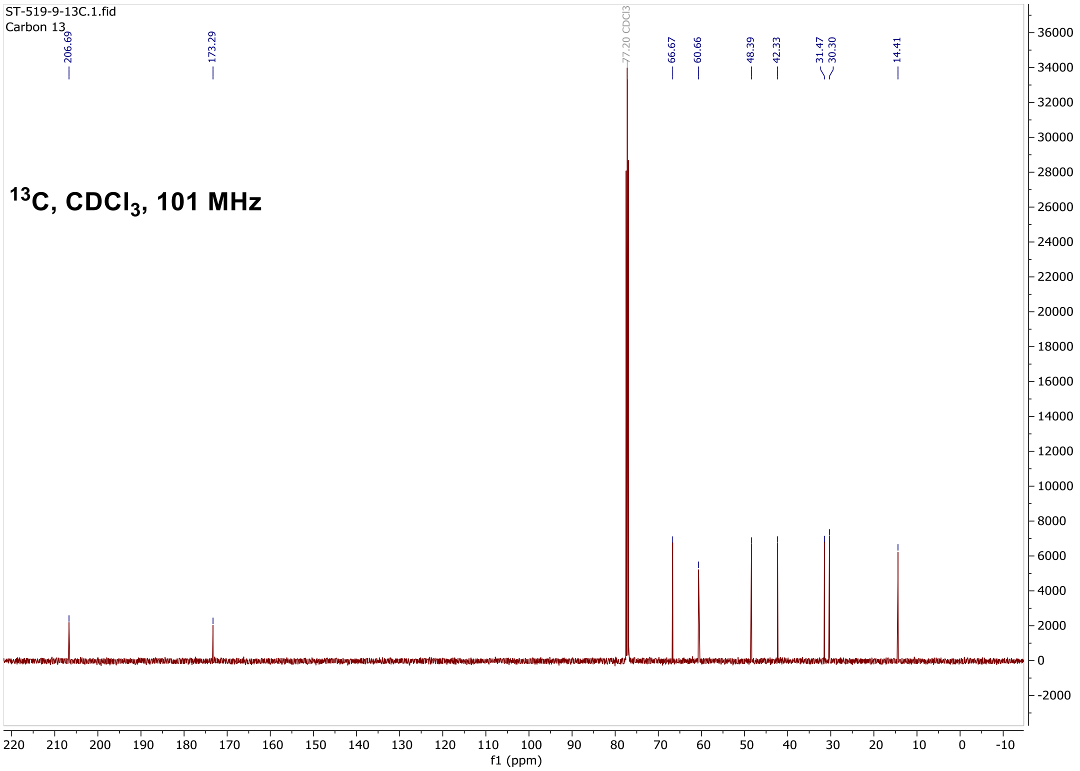

ST-513-7-iso.1.fid
Proton
CO2Me
O
MeO
O
OEt
19, 6:1 rr
1H, CDCl3, 500 MHz
7.26 CDCl3
4.16
4.14
4.13
4.11
4.10
4.10
4.09
3.67
3.66
2.35
2.33
2.32
2.30
2.28
2.27
1.97
1.96
1.94
1.93
1.91
1.90
1.89
1.27
1.26
1.26
1.25
1.24
1.23
2.30
6.00
6.44
1.18
4.53
3.17
f1 (ppm)

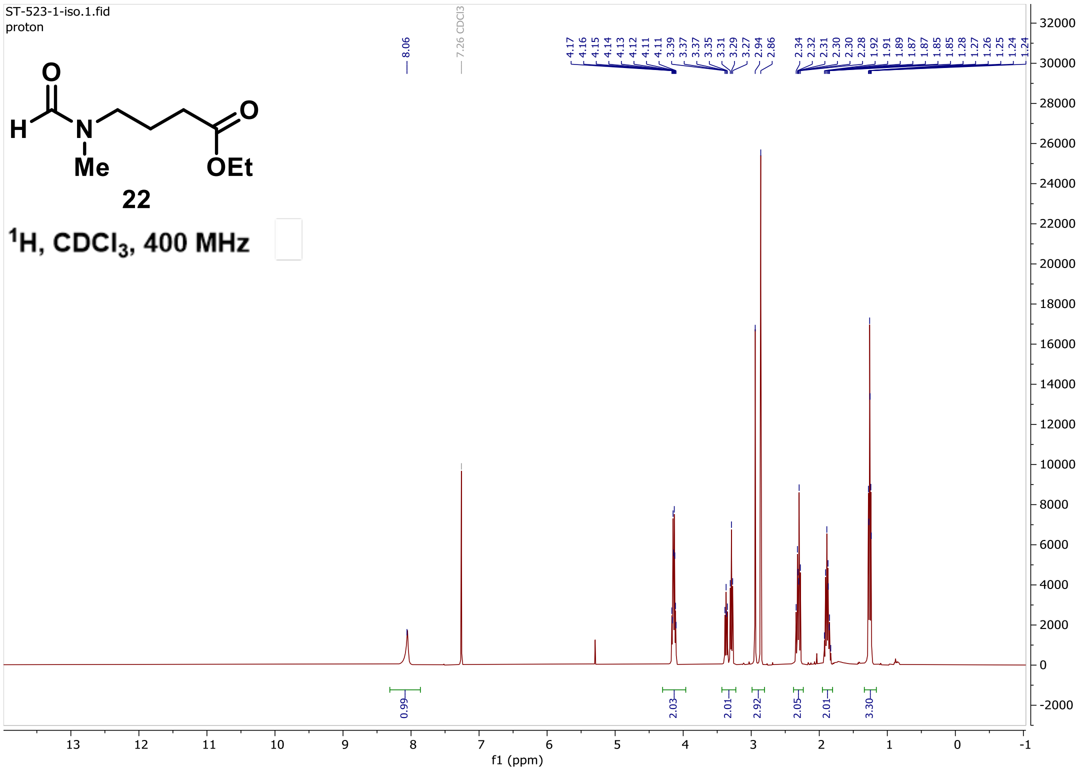

ST-513-7-iso.2.fid
Carbon 13
13C, CDCl3, 126 MHz
173.92
173.47
173.12
77.20 CDCl3
60.61
51.82
51.80
38.33
34.20
31.66
31.42
28.75
14.39
f1 (ppm)

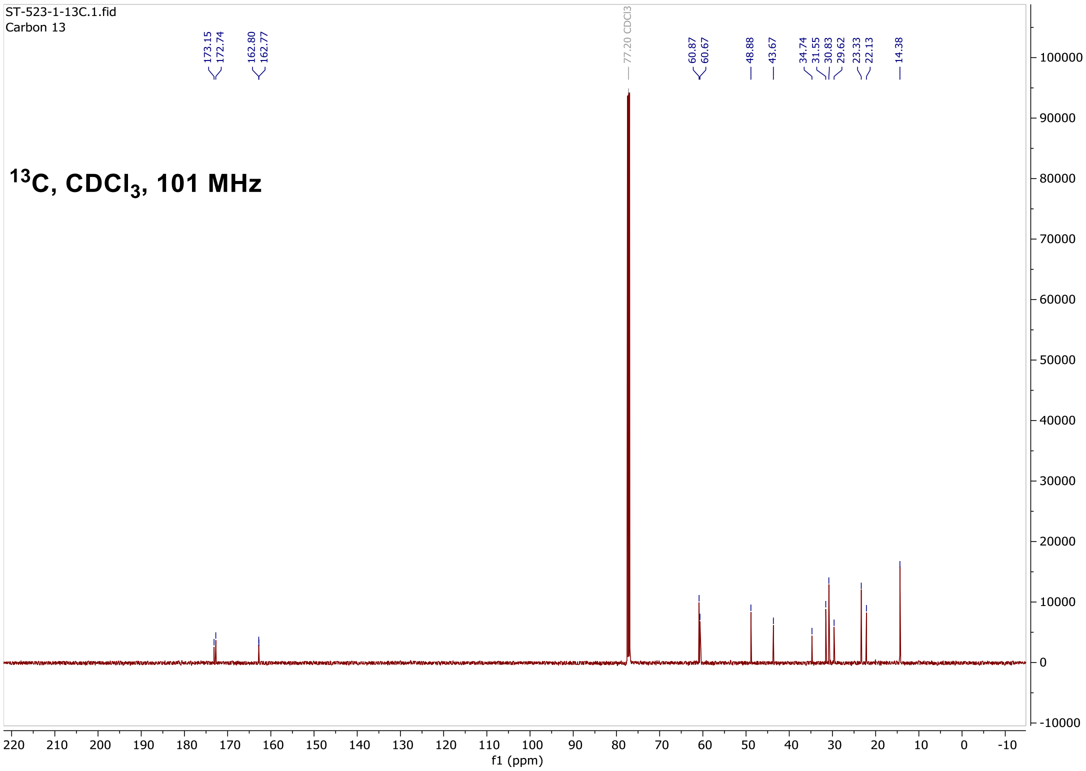

ST-513-8-iso.1.fid
Proton
O
Me
Me
O
O
OEt
20
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
4.95
4.94
4.93
4.91
4.90
4.89
4.15
4.14
4.12
4.11
2.39
2.38
2.36
2.35
2.34
2.33
2.33
2.32
2.30
2.29
2.03
1.90
1.89
1.89
1.88
1.87
1.86
1.86
1.27
1.26
1.24
1.23
1.00
2.00
2.06
2.90
2.01
6.06
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

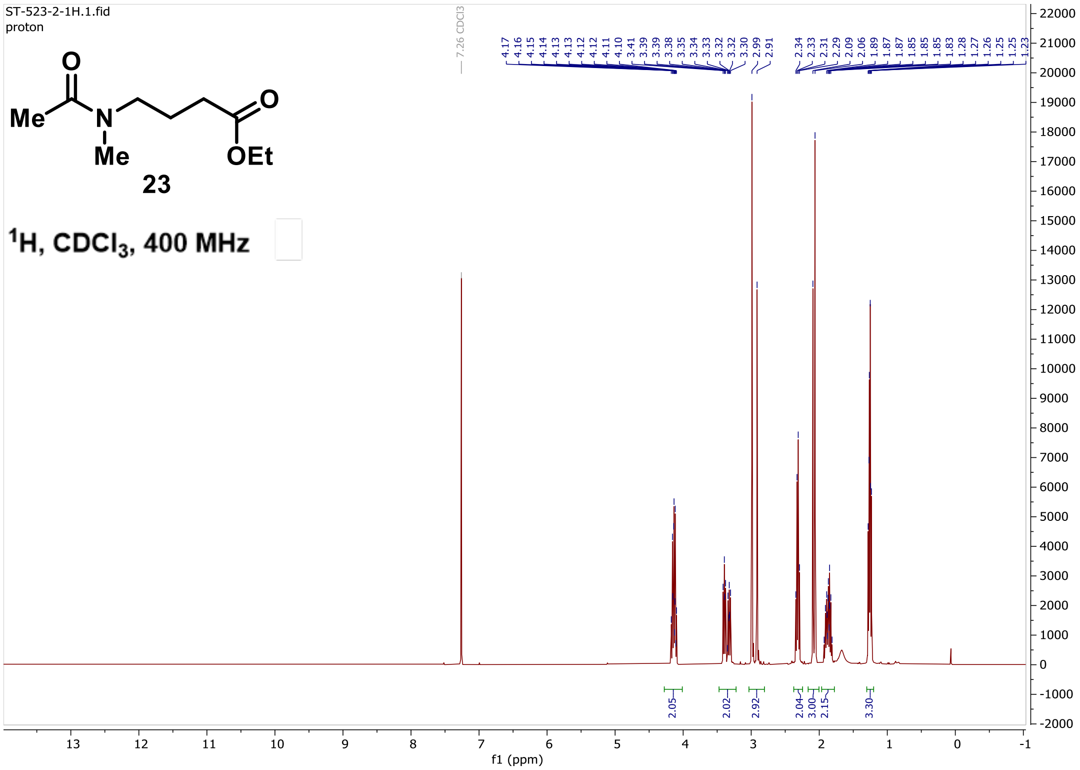

ST-513-8-iso.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
173.23
170.83
77.20 CDCl3
70.21
60.67
31.11
30.58
22.03
20.07
14.40
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


ST-519-9-1H.1.fid
proton
OEt
O
O
O
21
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
4.30
4.29
4.28
4.28
4.27
4.27
4.25
4.25
4.16
4.15
4.13
4.11
3.67
3.66
3.64
3.63
3.61
3.60
3.59
2.58
2.56
2.53
2.47
2.45
2.43
2.39
2.34
2.33
2.30
2.26
1.27
1.26
1.24
0.99
1.96
1.92
5.98
1.99
3.07
25000
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-519-9-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
206.69
173.29
77.20 CDCl3
66.67
60.66
48.39
42.33
31.47
30.30
14.41
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

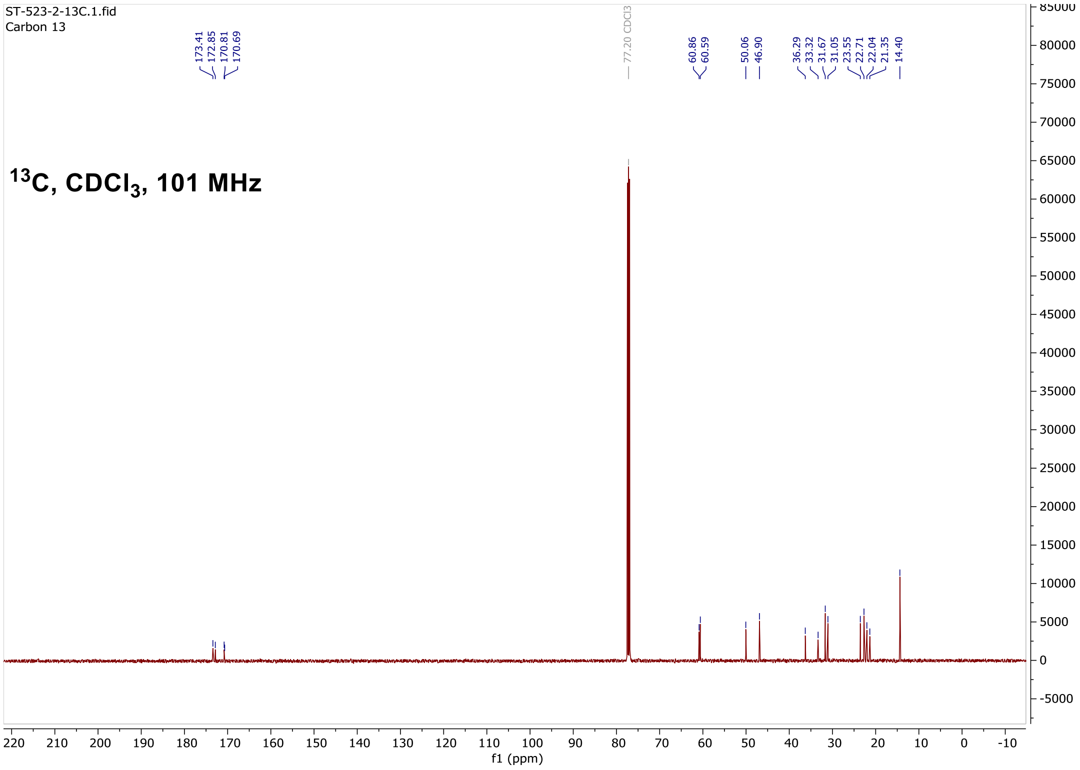

ST-523-1-iso.1.fid
proton
22
^{1}H, CDCl$_3$, 400 MHz
8.06
7.26 CDCl3
4.17 4.16 4.15 4.14 4.13 4.12 4.11 4.11 3.39 3.37 3.37 3.35 3.31 3.29 3.27 2.94 2.86
2.34 2.32 2.31 2.30 2.30 2.28 1.92 1.91 1.89 1.87 1.87 1.85 1.85 1.28 1.27 1.26 1.25 1.24 1.24
0.99 2.03 2.01 2.92 2.05 2.01 3.30
f1 (ppm)


ST-523-1-13C.1.fid
Carbon 13
^{13}C, CDCl$_3$, 101 MHz
173.15 172.74 162.80 162.77
77.20 CDCl3
60.87 60.67 48.88 43.67 34.74 31.55 30.83 29.62 23.33 22.13 14.38
f1 (ppm)

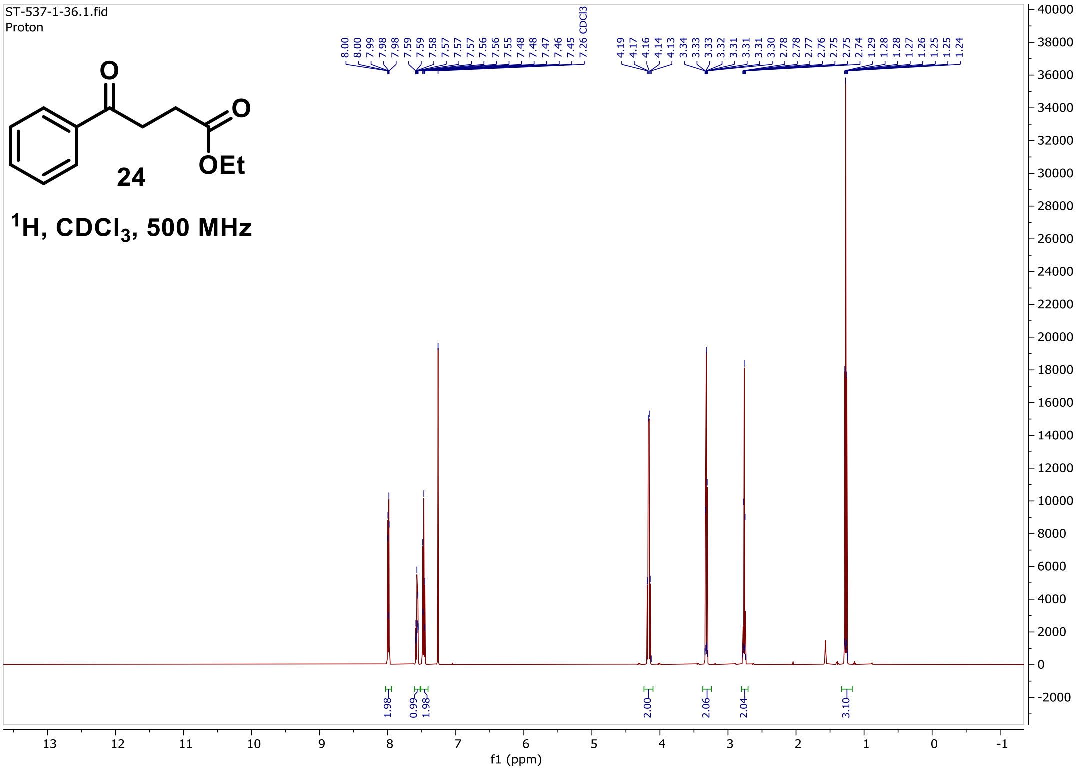


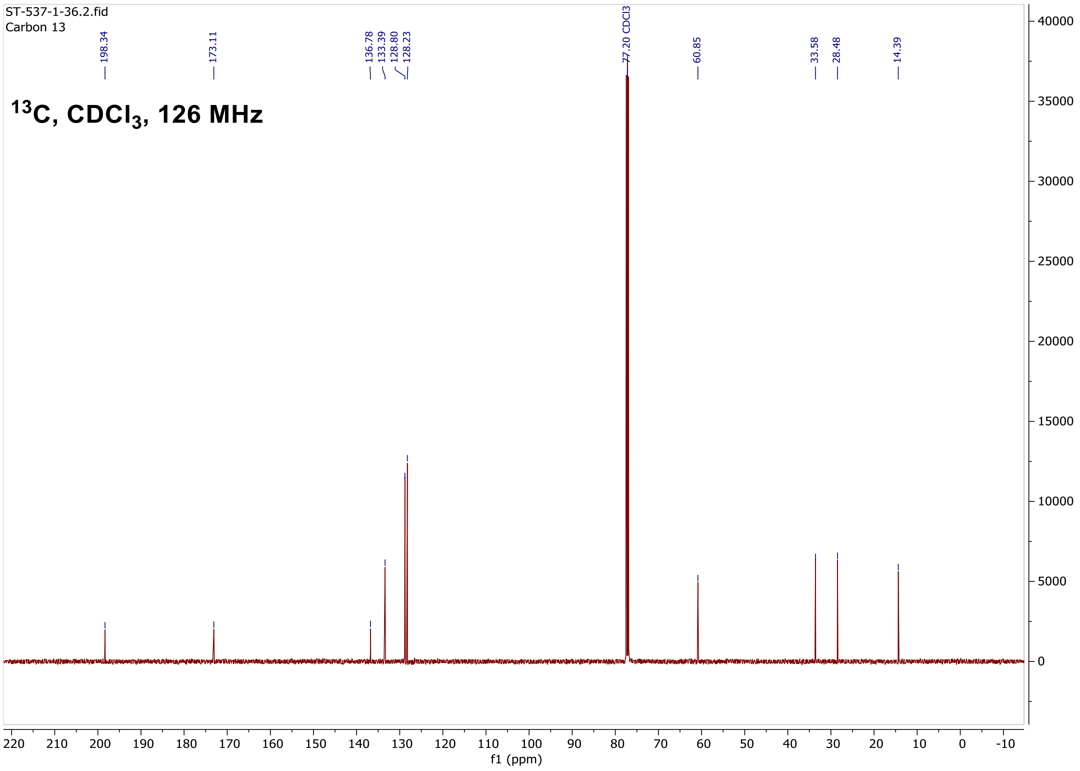
23

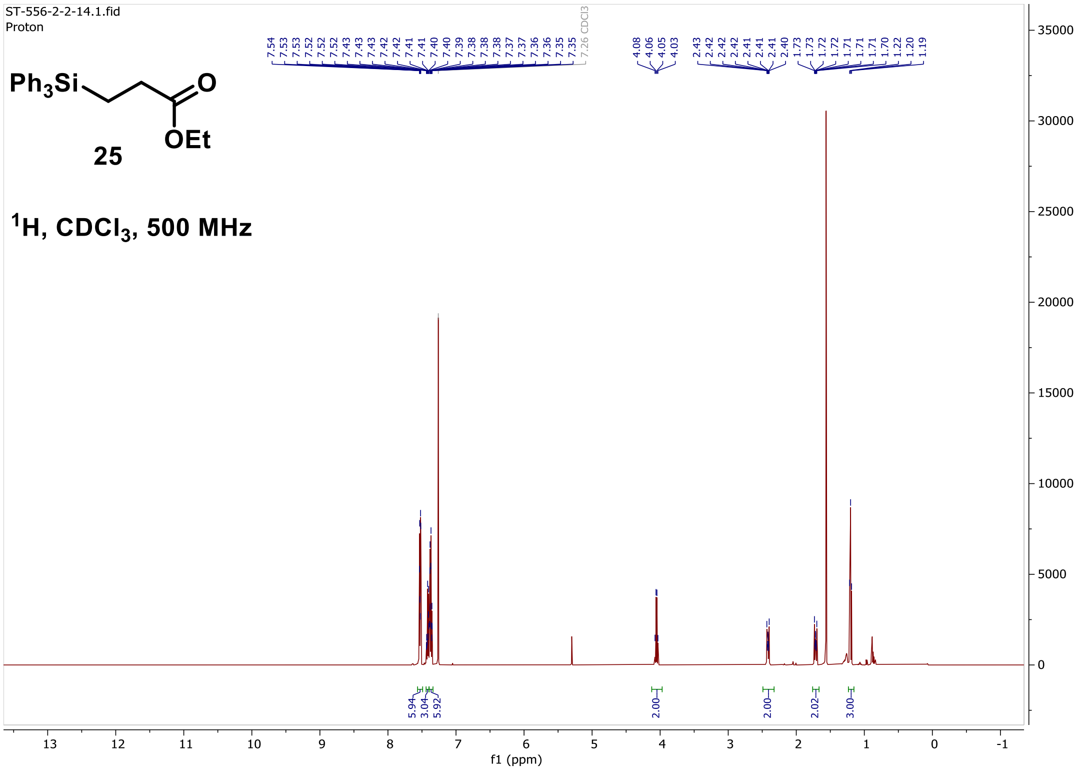
^{1}H, CDCl$_3$, 400 MHz

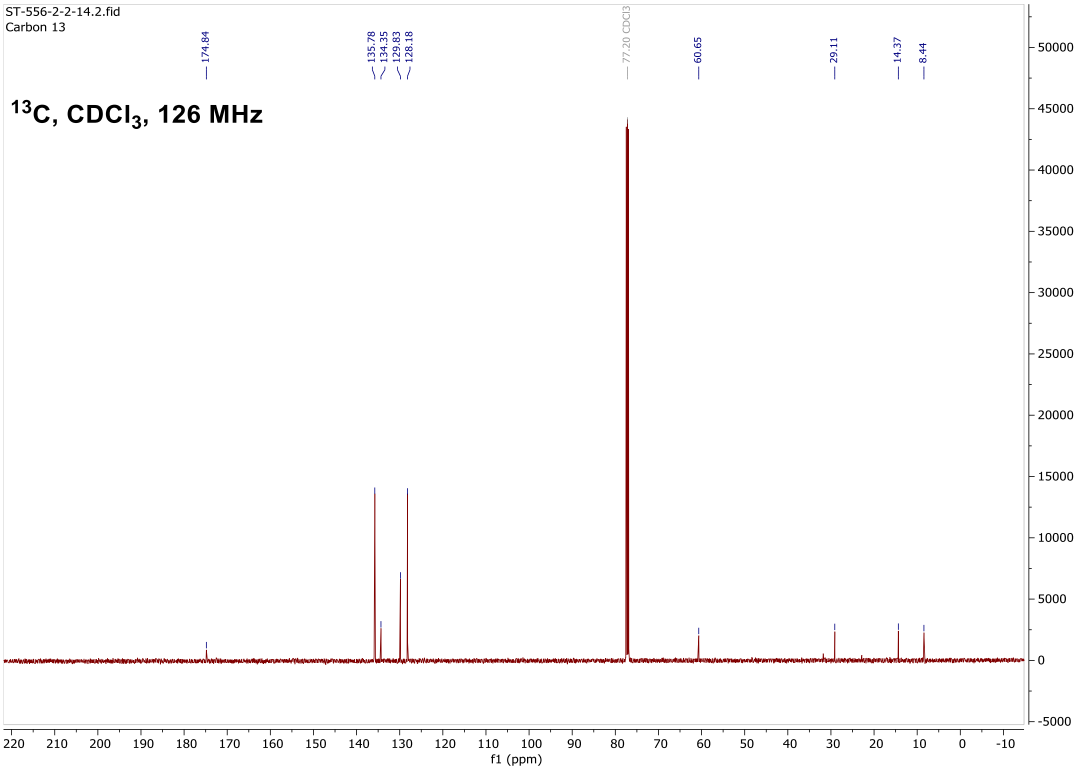


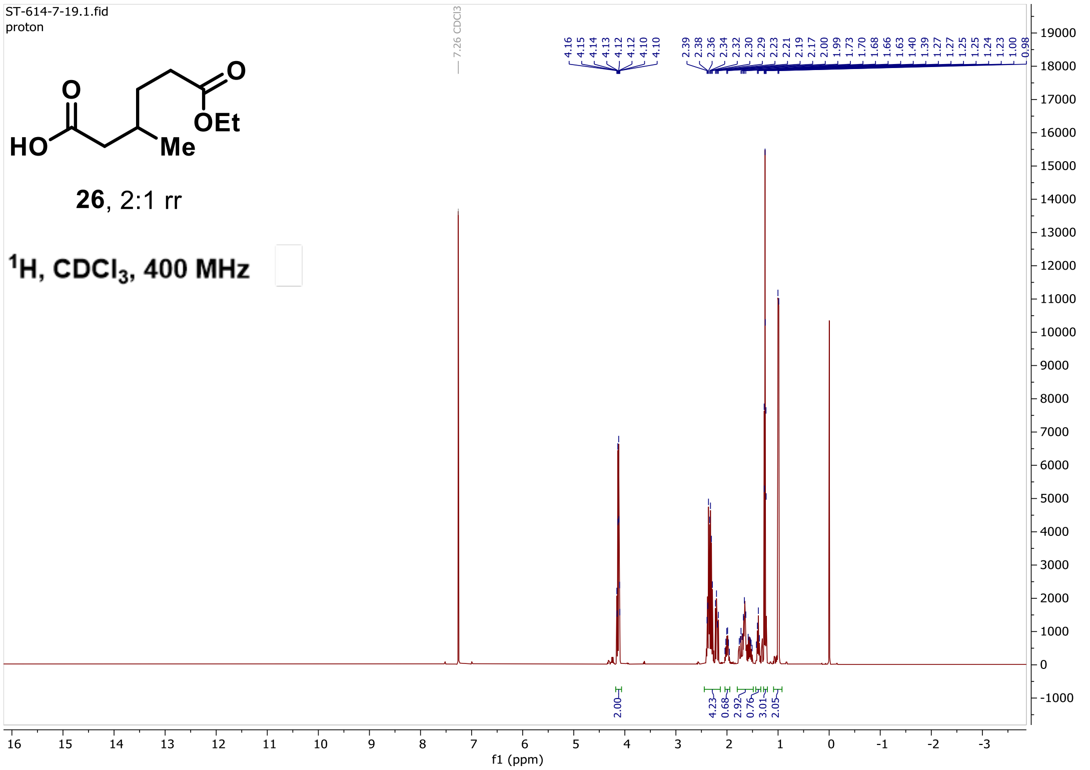
^{13}C, CDCl$_3$, 101 MHz


ST-537-1-36.1.fid
Proton
24
OEt
O
O
^{1}H, CDCl$_3$, 500 MHz
8.00 8.00 7.99 7.98 7.98 7.59 7.59 7.58 7.57 7.57 7.57 7.56 7.55 7.48 7.48 7.47 7.46 7.45 7.26 CDCl3
4.19 4.17 4.16 4.14 4.13 3.34 3.33 3.33 3.32 3.31 3.31 3.30 2.78 2.78 2.77 2.76 2.75 2.75 2.74 1.29 1.28 1.28 1.27 1.26 1.25 1.25 1.24
1.98 0.99 1.98 2.00 2.06 2.04 3.10
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)
40000 38000 36000 34000 32000 30000 28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000


ST-537-1-36.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
198.34
173.11
136.78 133.39 128.80 128.23
77.20 CDCl3
60.85
33.58
28.48
14.39
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
40000 35000 30000 25000 20000 15000 10000 5000 0

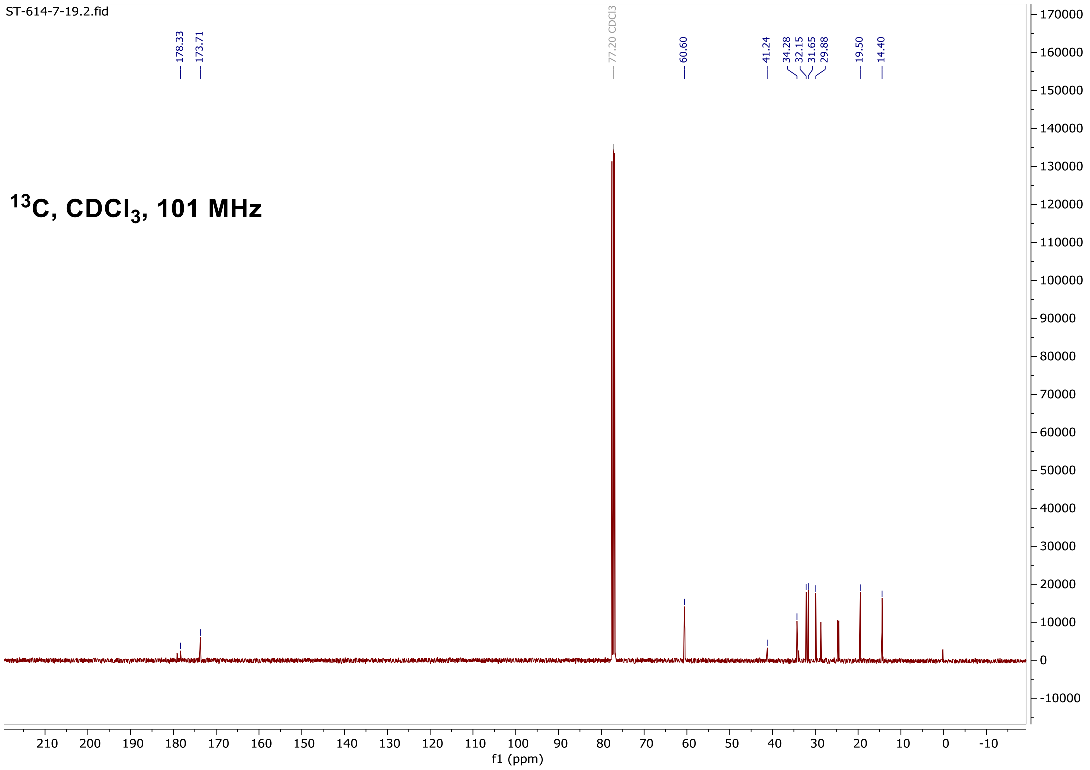

ST-556-2-2-14.1.fid
Proton
7.54
7.53
7.53
7.52
7.52
7.52
7.43
7.43
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.26 CDCl3
4.08
4.06
4.05
4.03
2.43
2.42
2.42
2.42
2.41
2.41
2.41
2.40
1.73
1.73
1.72
1.72
1.71
1.71
1.71
1.70
1.22
1.20
1.19
Ph3Si
O
OEt
25
1H, CDCl3, 500 MHz
35000
30000
25000
20000
15000
10000
5000
0
5.94
3.04
5.92
2.00
2.00
2.02
3.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-556-2-2-14.2.fid
Carbon 13
174.84
135.78
134.35
129.83
128.18
77.20 CDCl3
60.65
29.11
14.37
8.44
13C, CDCl3, 126 MHz
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

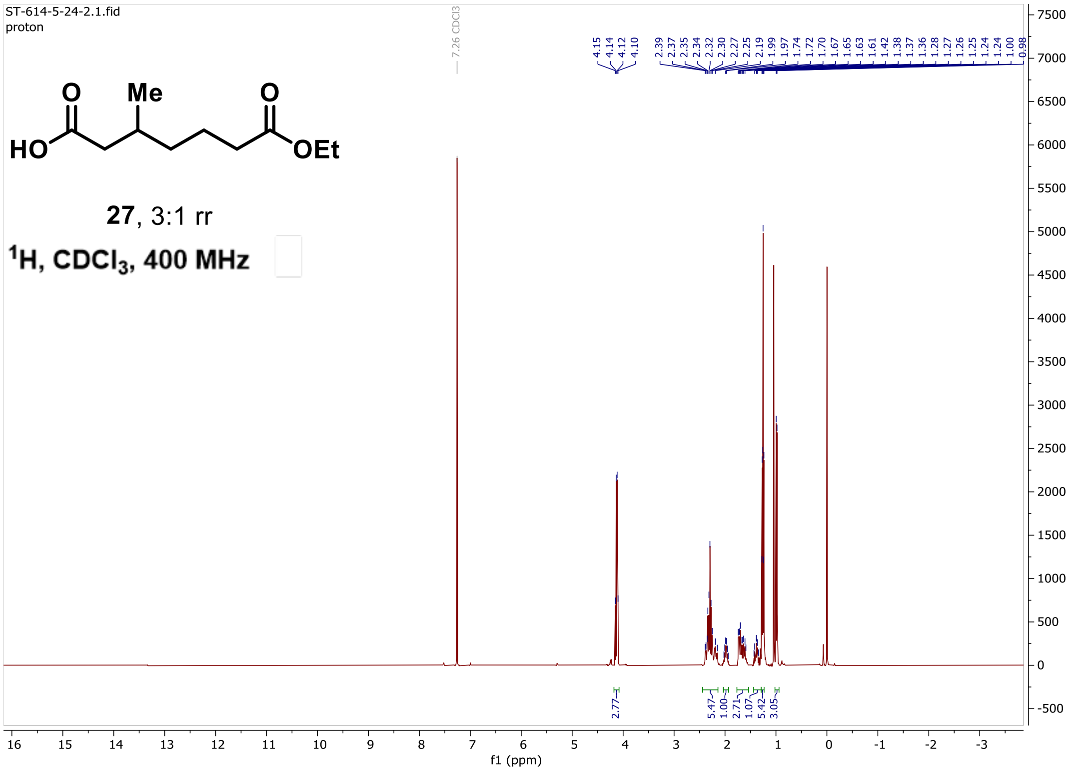


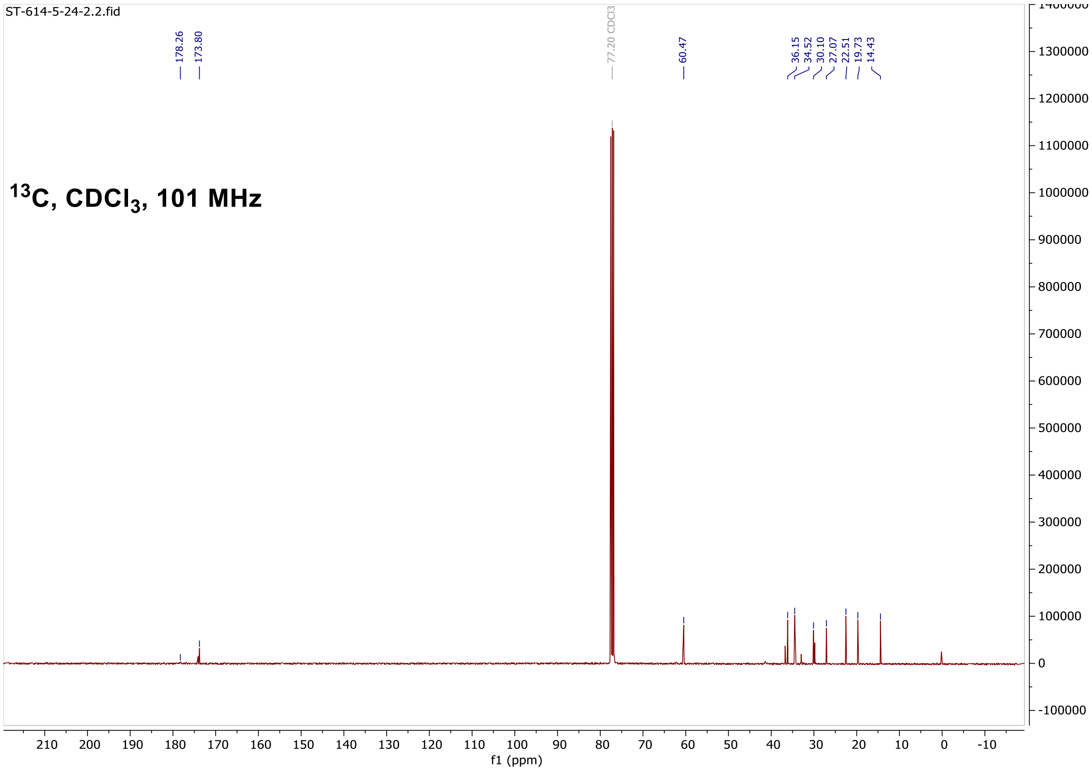
26, 2:1 rr

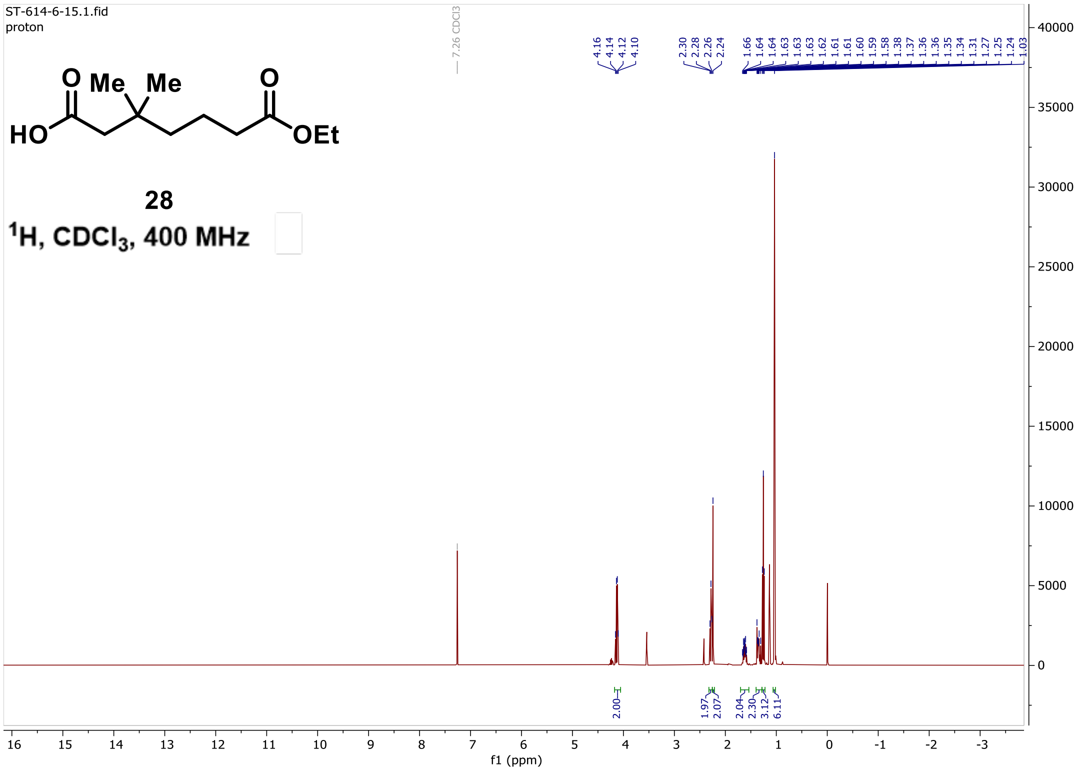
^{1}H, CDCl$_3$, 400 MHz



^{13}C, CDCl$_3$, 101 MHz

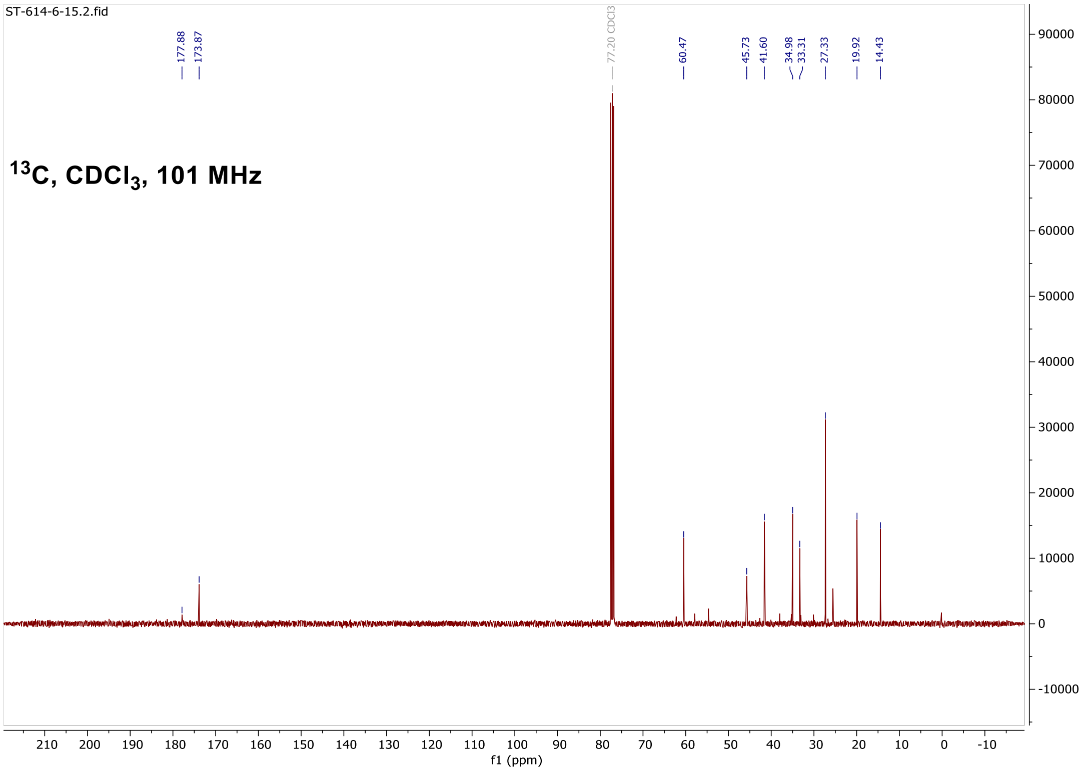

ST-614-5-24-2.1.fid
proton
7.26 CDCl3
4.15
4.14
4.12
4.10
2.39
2.37
2.35
2.34
2.32
2.30
2.27
2.25
2.19
1.99
1.97
1.74
1.72
1.70
1.67
1.65
1.63
1.61
1.42
1.38
1.37
1.36
1.28
1.27
1.26
1.25
1.24
1.24
1.00
0.98
O
Me
O
HO
OEt
27, 3:1 rr
^{1}H, CDCl$_3$, 400 MHz
2.77
5.47
1.00
2.71
1.07
5.42
3.05
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


ST-614-5-24-2.2.fid
178.26
173.80
77.20 CDCl3
60.47
36.15
34.52
30.10
27.07
22.51
19.73
14.43
^{13}C, CDCl$_3$, 101 MHz
1400000
1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

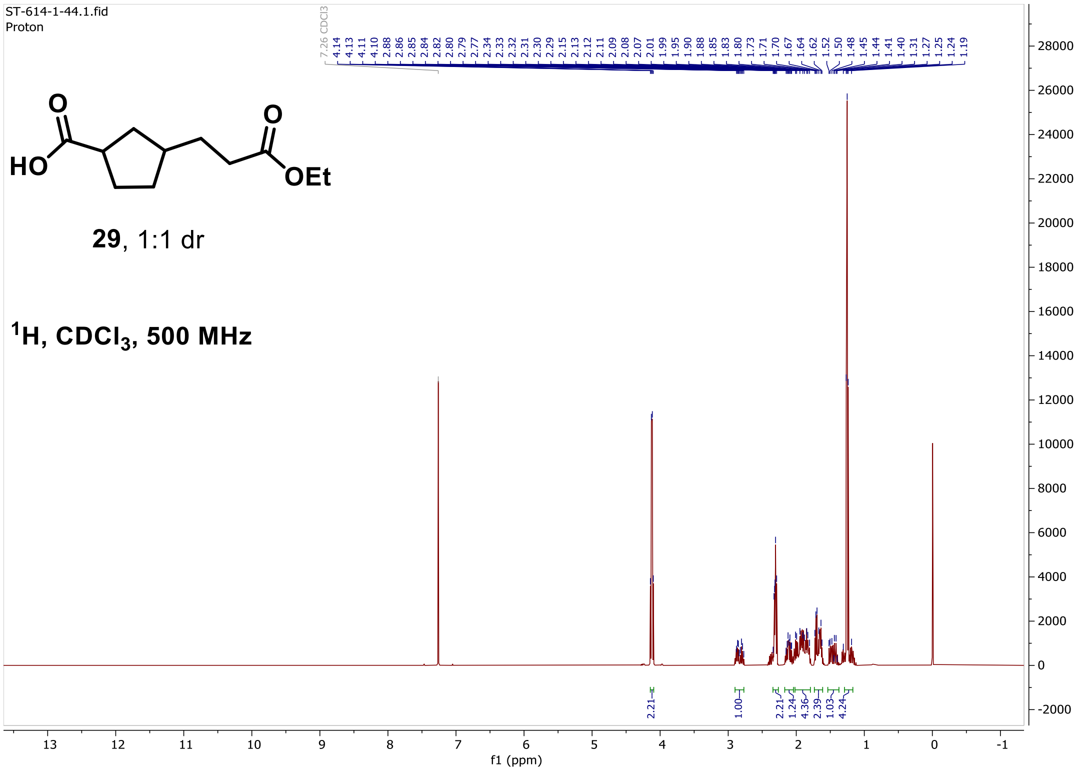


28

^{1}H, CDCl$_3$, 400 MHz

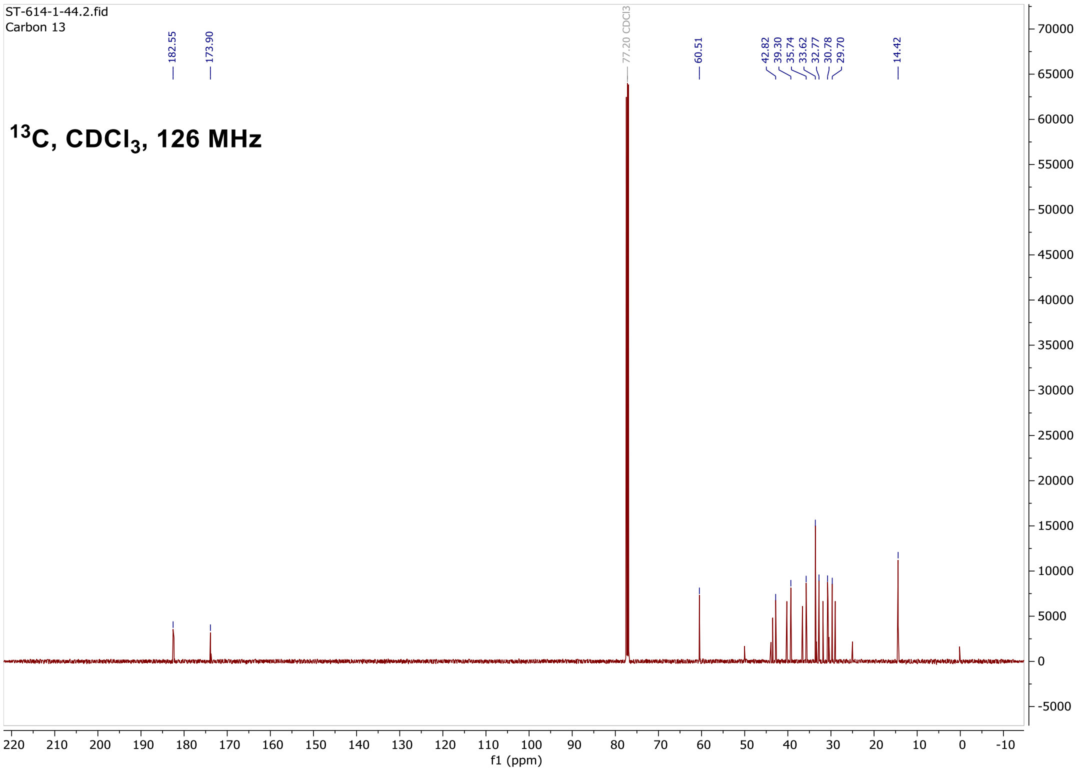


^{13}C, CDCl$_3$, 101 MHz

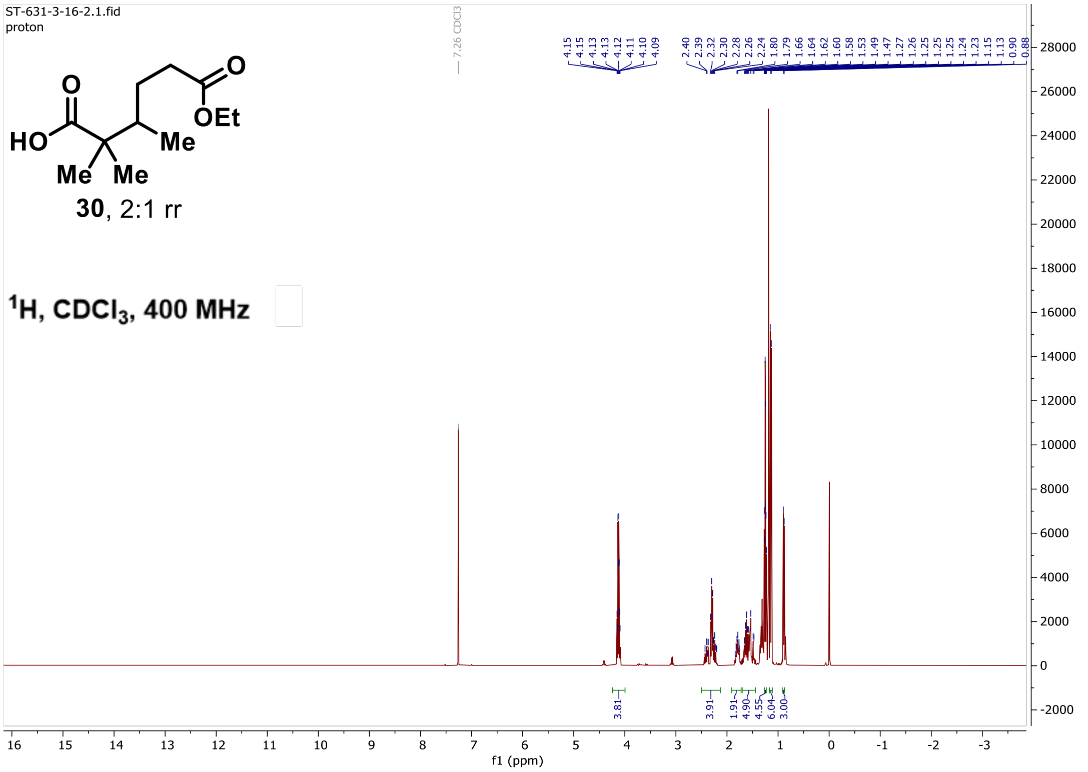


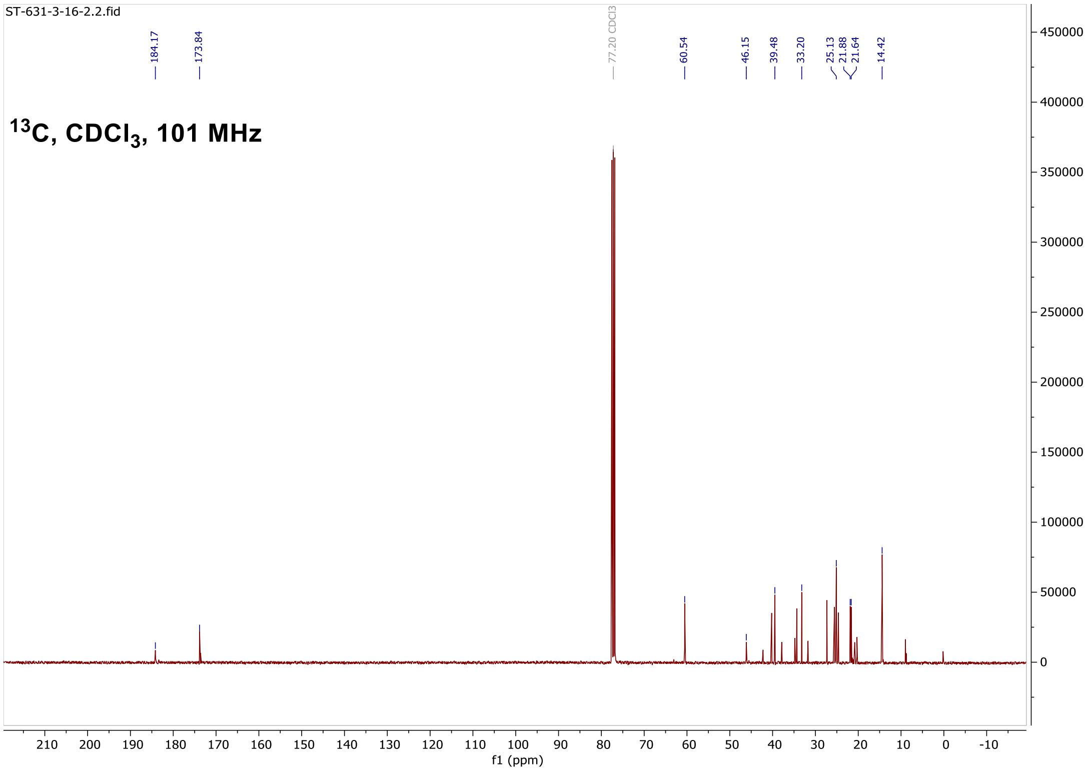
29, 1:1 dr

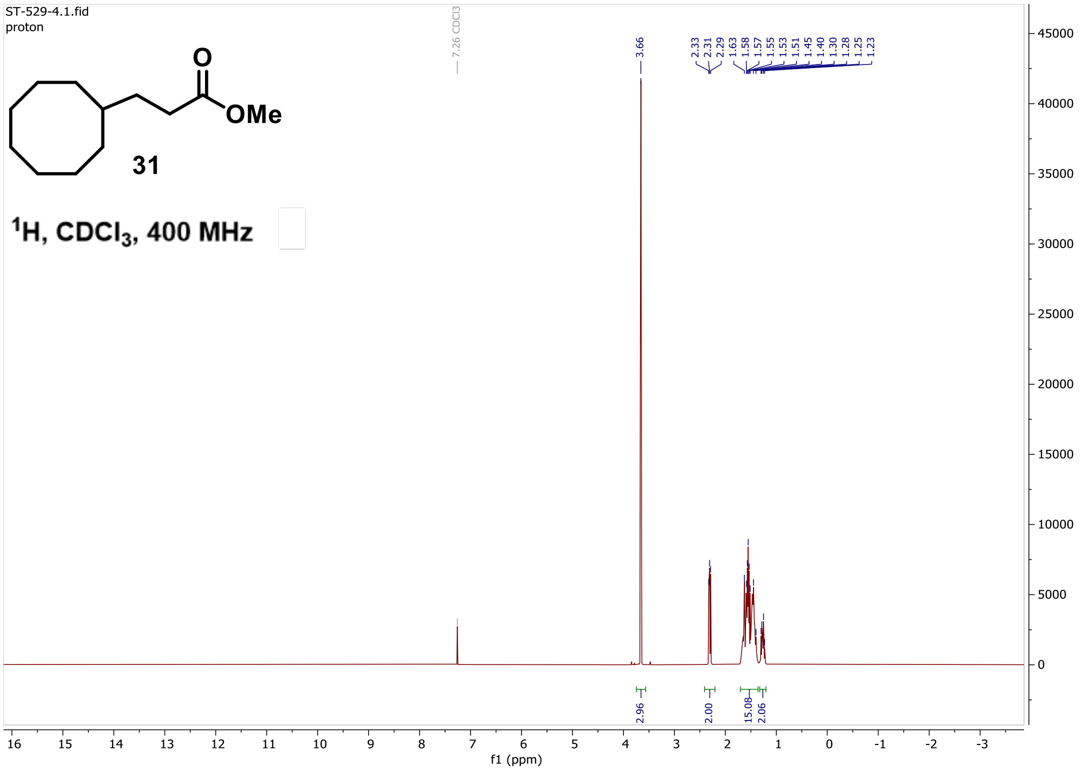
^{1}H, CDCl$_3$, 500 MHz

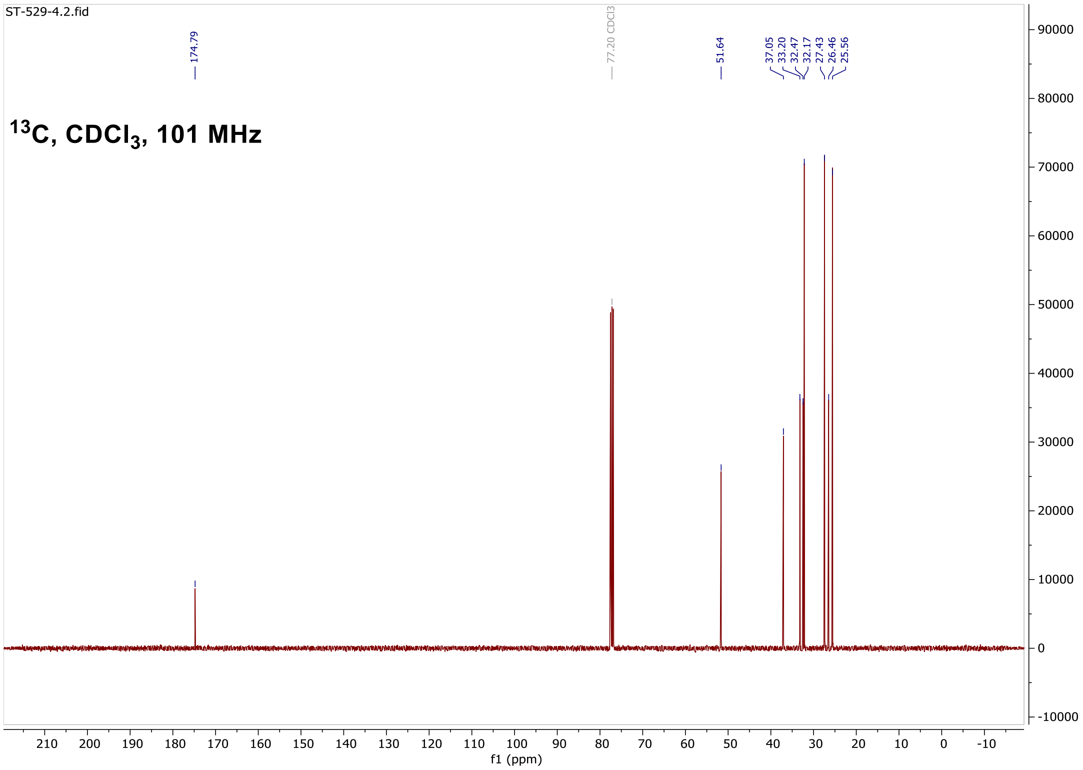


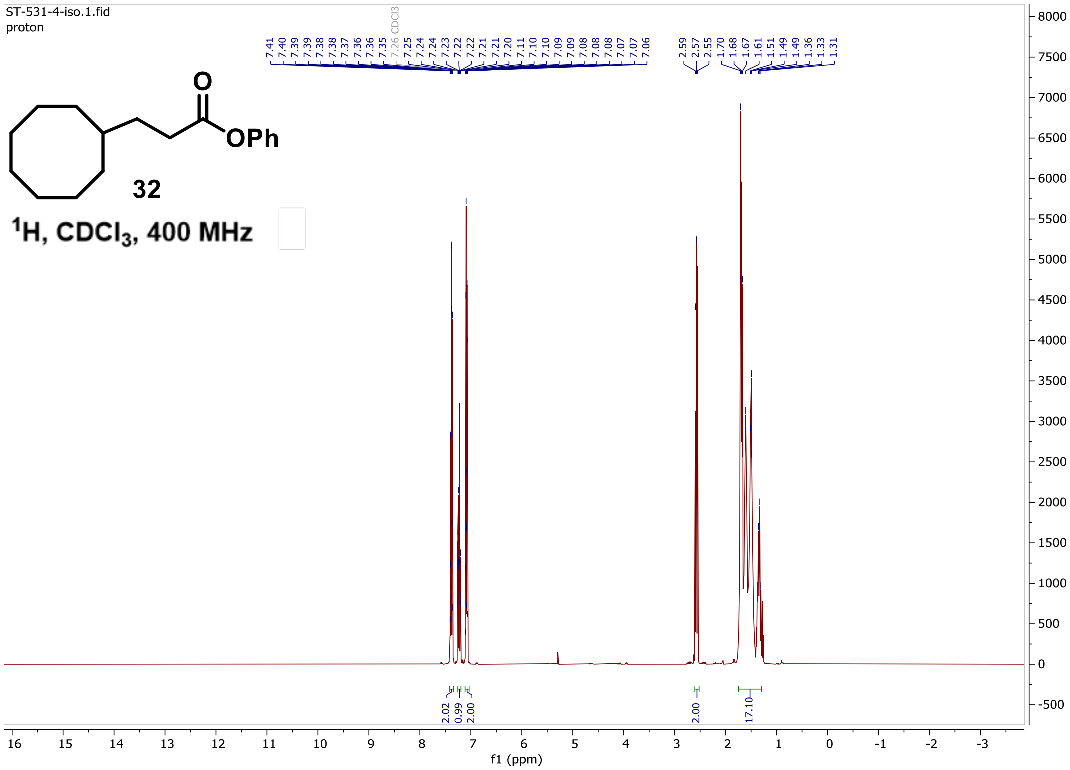
^{13}C, CDCl$_3$, 126 MHz

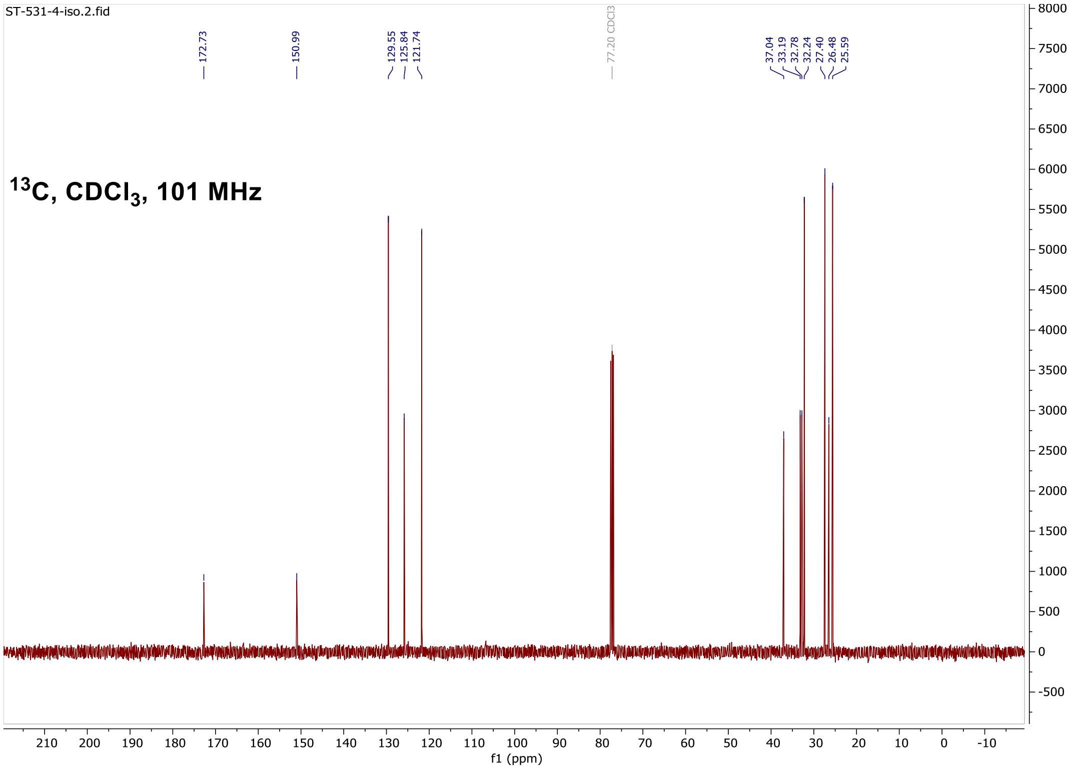

ST-631-3-16-2.1.fid
proton
O
HO
Me
Me
Me
O
OEt
30, 2:1 rr
1H, CDCl3, 400 MHz
7.26 CDCl3
4.15
4.15
4.13
4.13
4.12
4.11
4.10
4.09
2.40
2.39
2.32
2.30
2.28
2.26
2.24
1.80
1.79
1.66
1.64
1.62
1.60
1.58
1.53
1.49
1.47
1.27
1.26
1.25
1.25
1.25
1.24
1.23
1.15
1.13
0.90
0.88
3.81
3.91
1.91
4.90
4.55
6.04
3.00
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

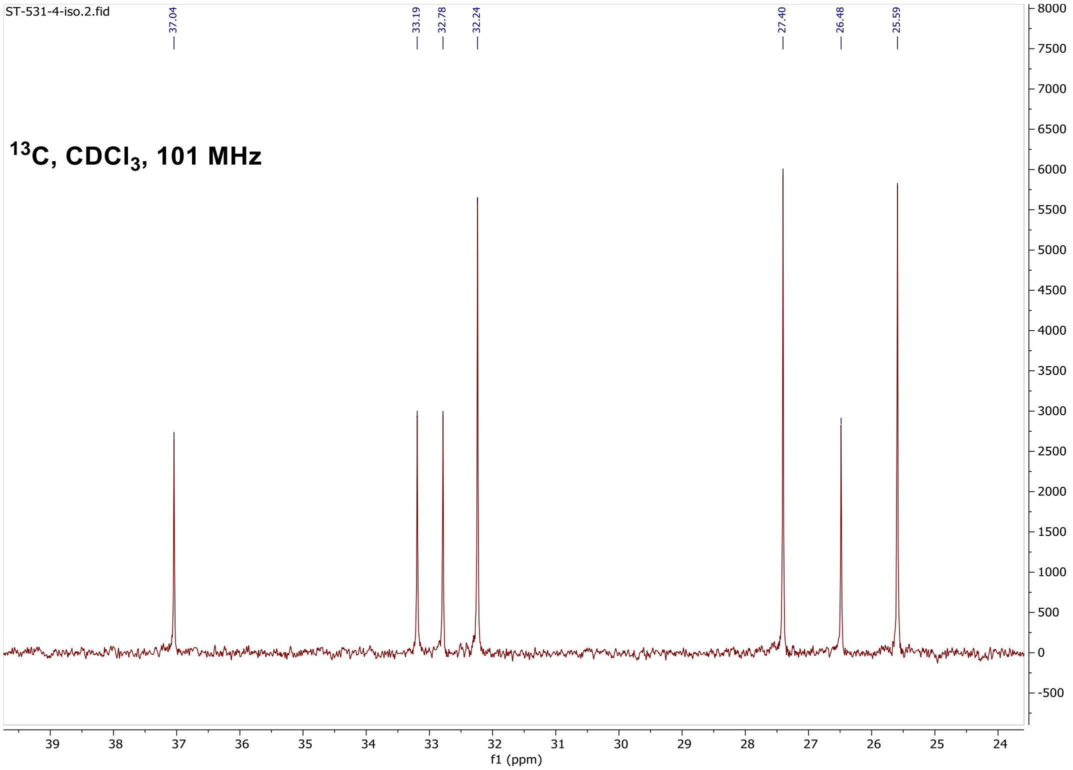

ST-631-3-16-2.2.fid
13C, CDCl3, 101 MHz
184.17
173.84
77.20 CDCl3
60.54
46.15
39.48
33.20
25.13
21.88
21.64
14.42
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

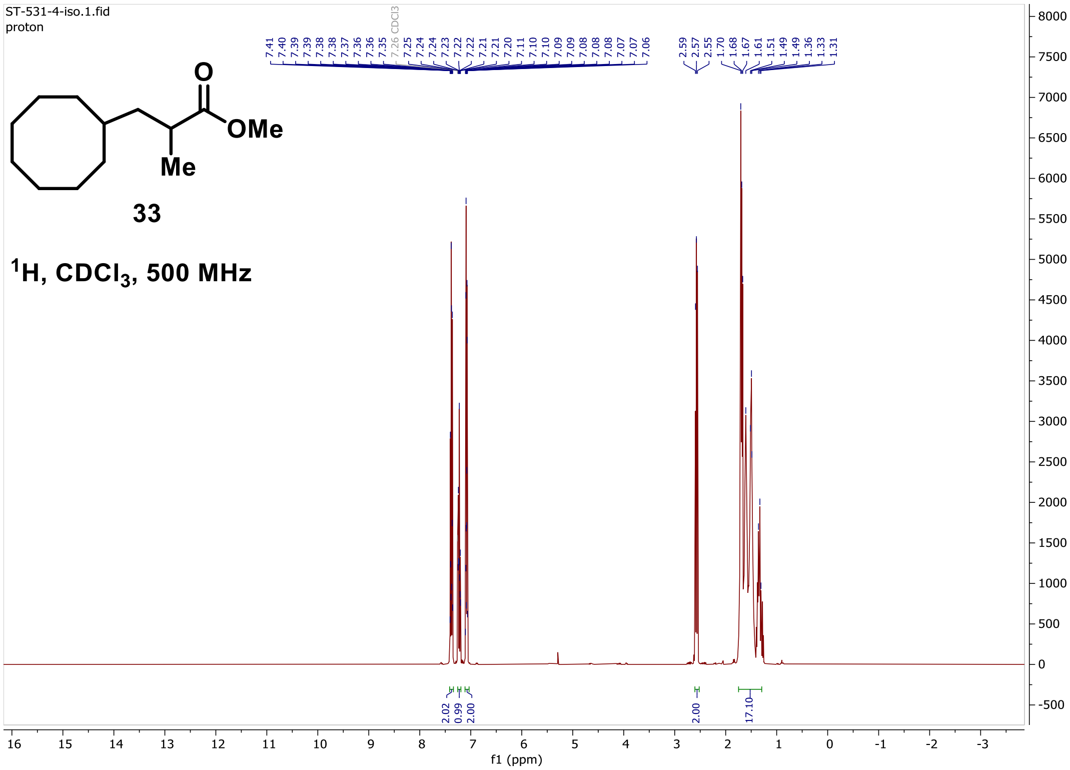

ST-529-4.1.fid
proton
O
OMe
31
¹H, CDCl₃, 400 MHz
7.26 CDCl3
3.66
2.33
2.31
2.29
1.63
1.58
1.57
1.55
1.53
1.51
1.45
1.40
1.30
1.28
1.25
1.23
2.96
2.00
15.08
2.06
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

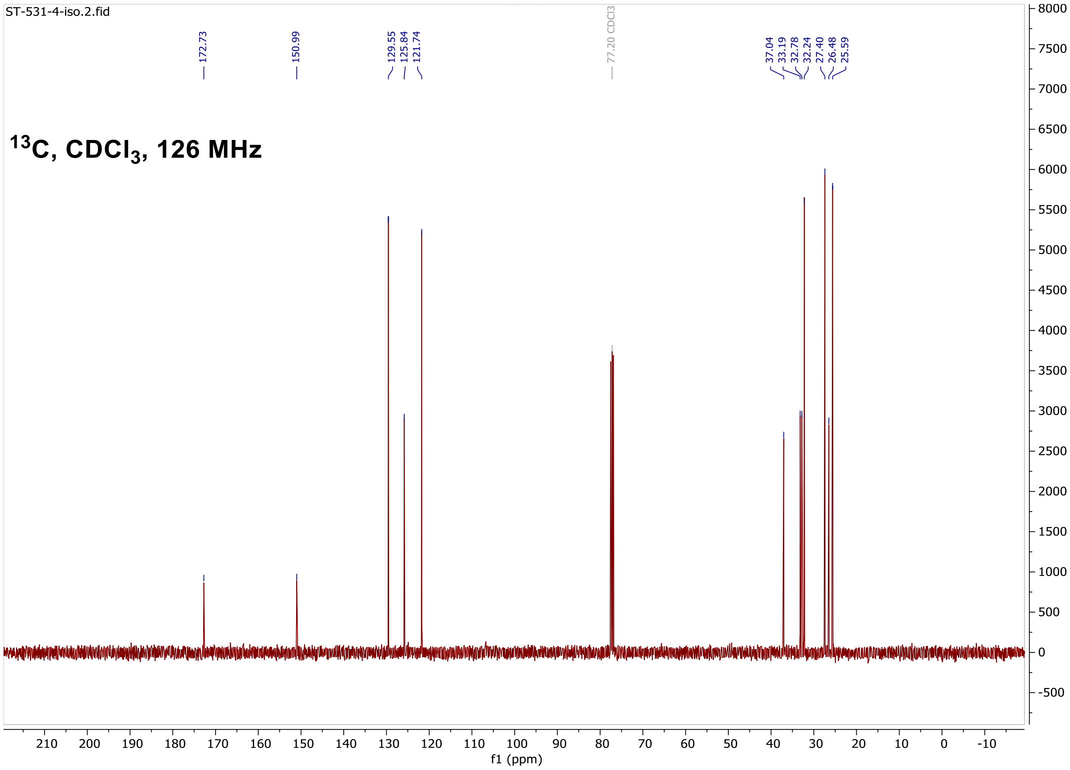

ST-529-4.2.fid
¹³C, CDCl₃, 101 MHz
174.79
77.20 CDCl3
51.64
37.05
33.20
32.47
32.17
27.43
26.46
25.56
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

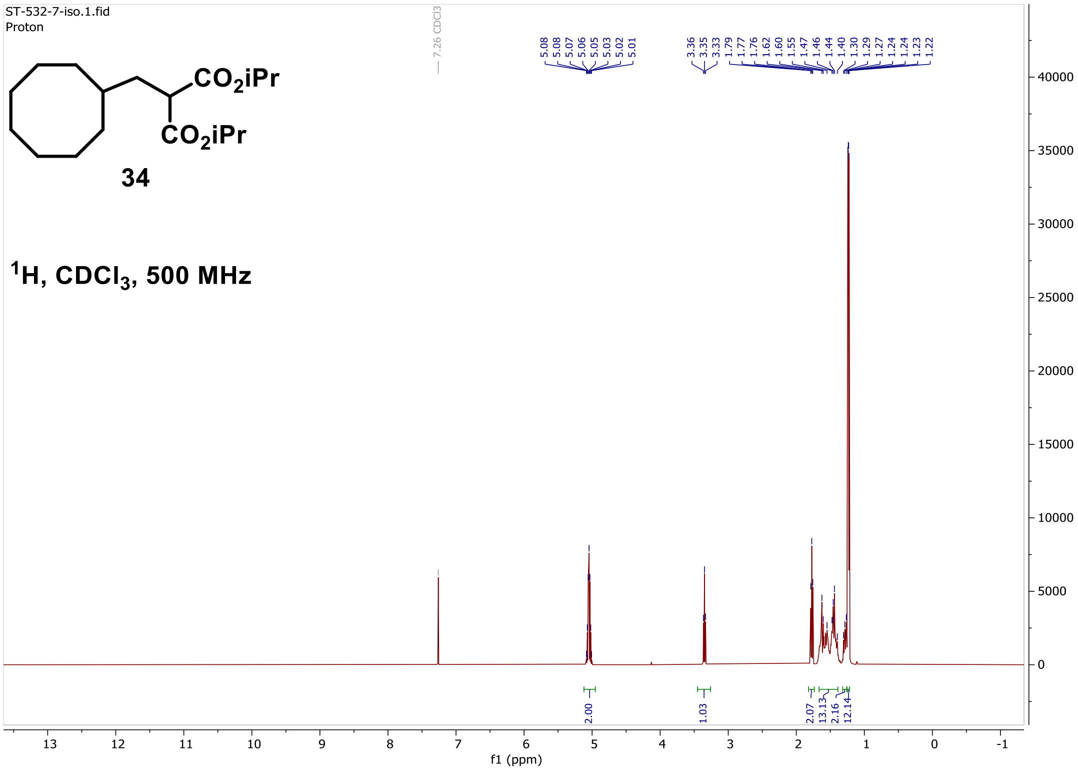

ST-531-4-iso.1.fid
proton
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.36
7.35
7.26 CDCl3
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.11
7.10
7.10
7.09
7.09
7.08
7.08
7.07
7.07
7.06
2.59
2.57
2.55
1.70
1.68
1.67
1.61
1.51
1.49
1.49
1.36
1.33
1.31
O
OPh
32
1H, CDCl3, 400 MHz
2.02
0.99
2.00
2.00
17.10
f1 (ppm)

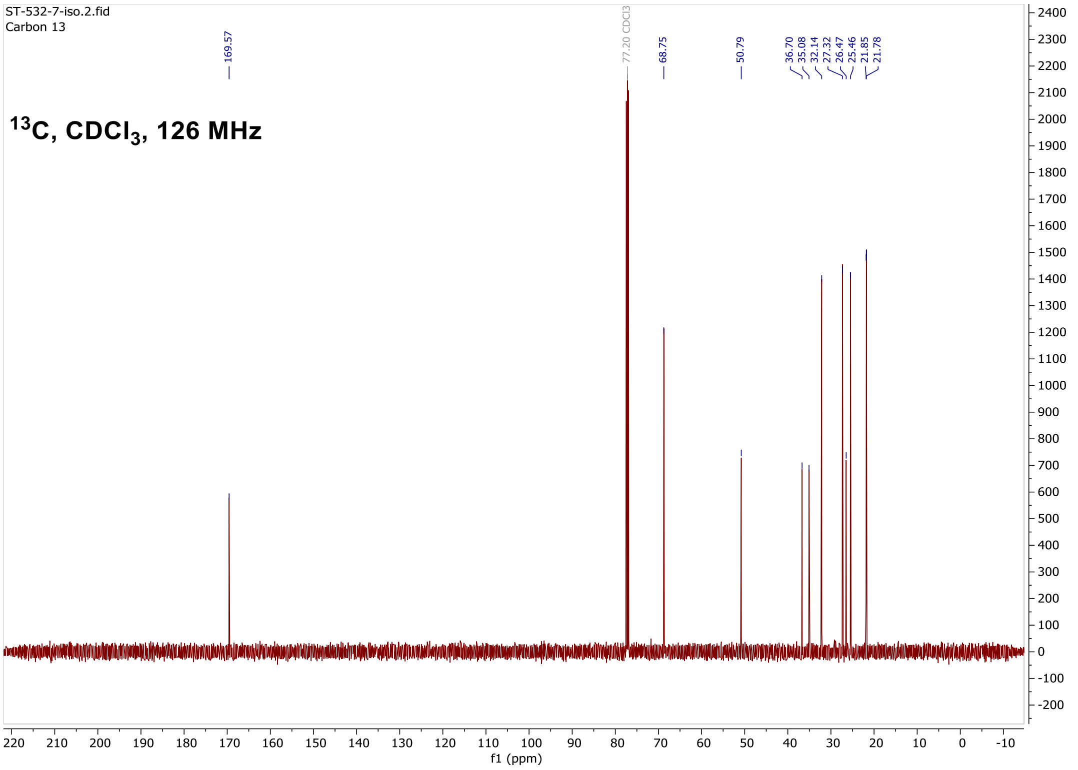

ST-531-4-iso.2.fid
172.73
150.99
129.55
125.84
121.74
77.20 CDCl3
37.04
33.19
32.78
32.24
27.40
26.48
25.59
13C, CDCl3, 101 MHz
f1 (ppm)

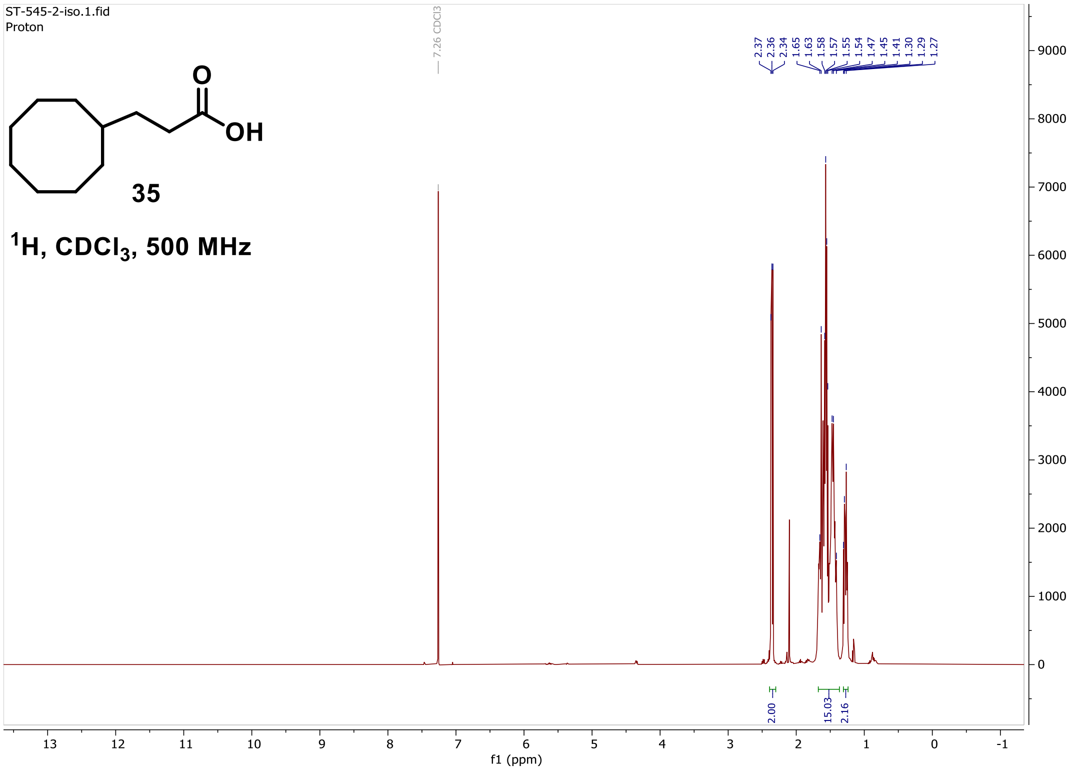

ST-531-4-iso.2.fid
37.04
33.19
32.78
32.24
27.40
26.48
25.59
^{13}C, CDCl$_3$, 101 MHz
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
f1 (ppm)

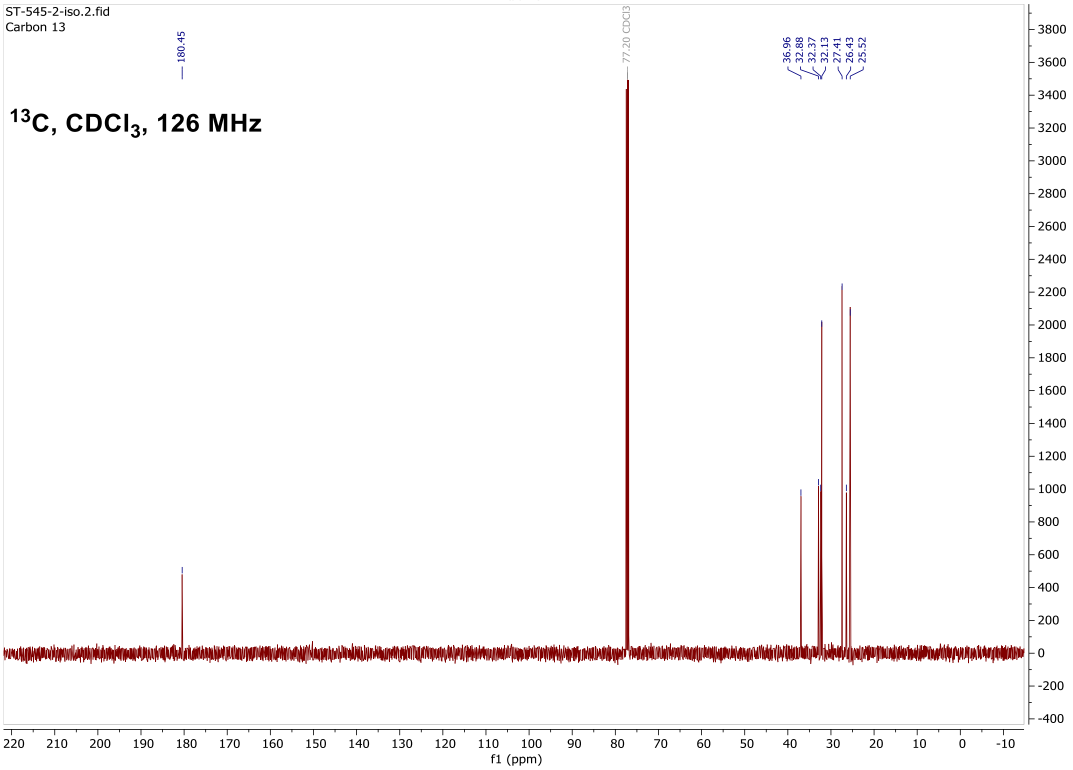

ST-531-4-iso.1.fid
proton
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.36
7.35
7.26 CDCl3
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.11
7.10
7.10
7.09
7.09
7.08
7.08
7.07
7.07
7.06
2.59
2.57
2.55
1.70
1.68
1.67
1.61
1.51
1.49
1.49
1.36
1.33
1.31
O
OMe
Me
33
^{1}H, CDCl$_3$, 500 MHz
2.02
0.99
2.00
2.00
17.10
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

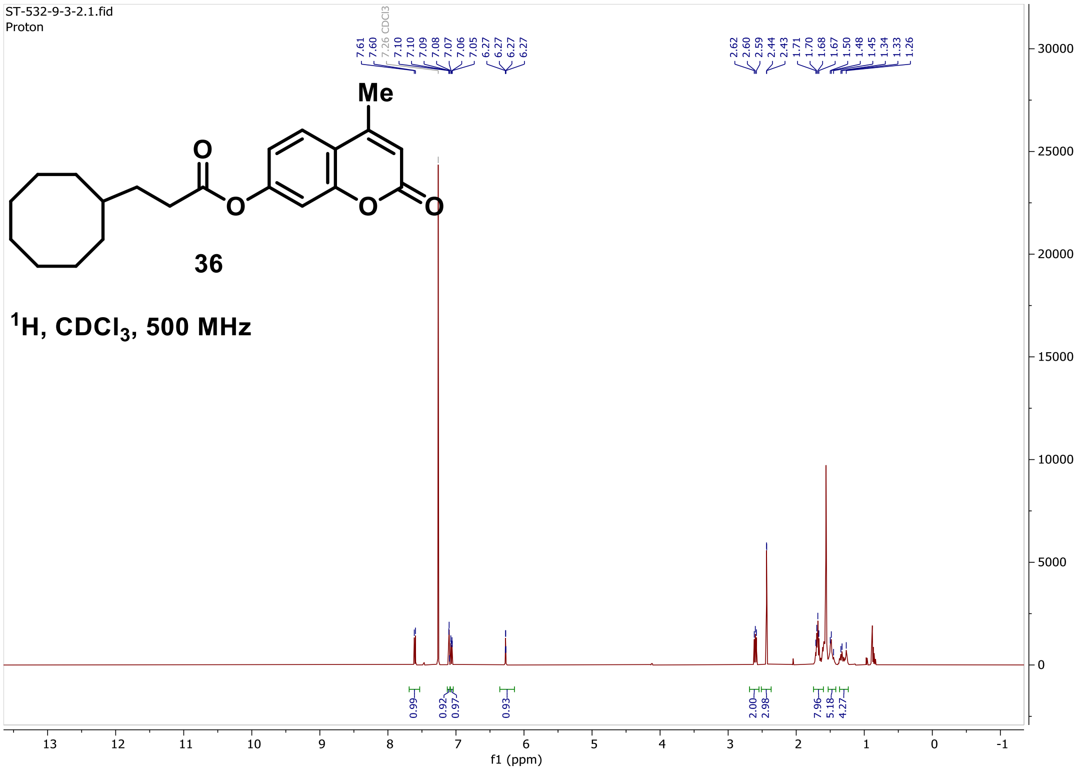

ST-531-4-iso.2.fid
172.73
150.99
129.55
125.84
121.74
77.20 CDCl3
37.04
33.19
32.78
32.24
27.40
26.48
25.59
^{13}C, CDCl$_3$, 126 MHz
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

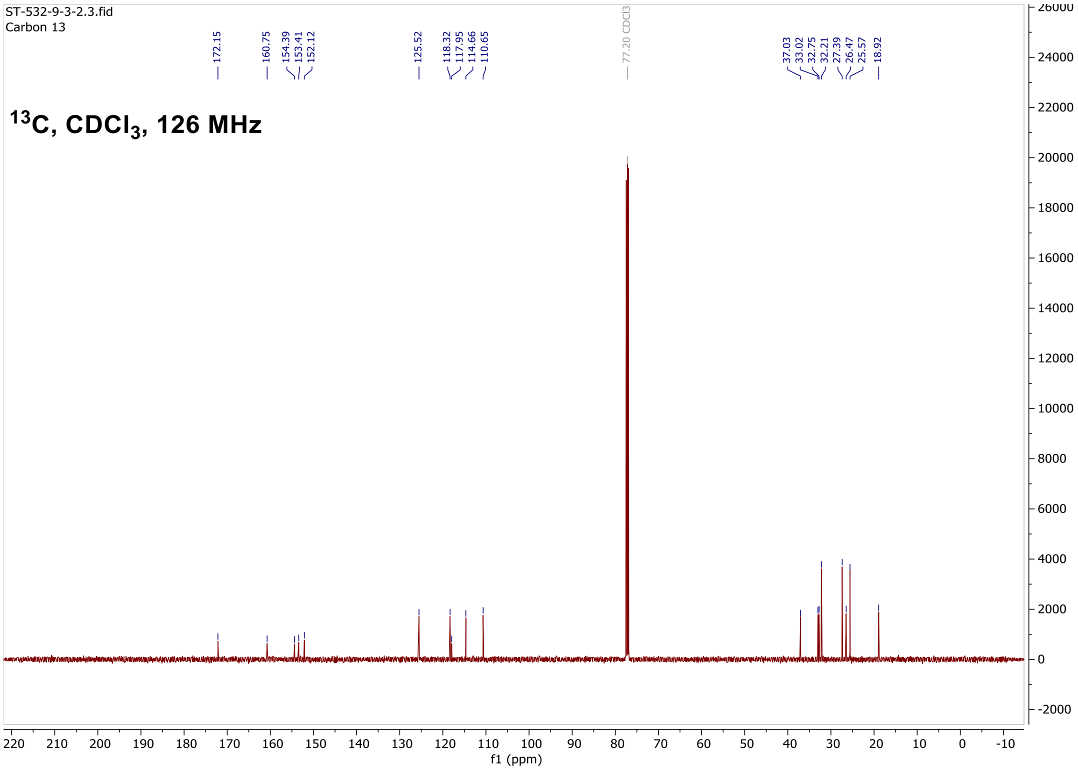

ST-532-7-iso.1.fid
Proton
CO2iPr
CO2iPr
34
1H, CDCl3, 500 MHz
7.26 CDCl3
5.08
5.08
5.07
5.06
5.05
5.03
5.02
5.01
3.36
3.35
3.33
1.79
1.77
1.76
1.62
1.60
1.55
1.47
1.46
1.44
1.40
1.30
1.29
1.27
1.24
1.23
1.22
2.00
1.03
2.07
13.13
2.16
12.14
40000
35000
30000
25000
20000
15000
10000
5000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

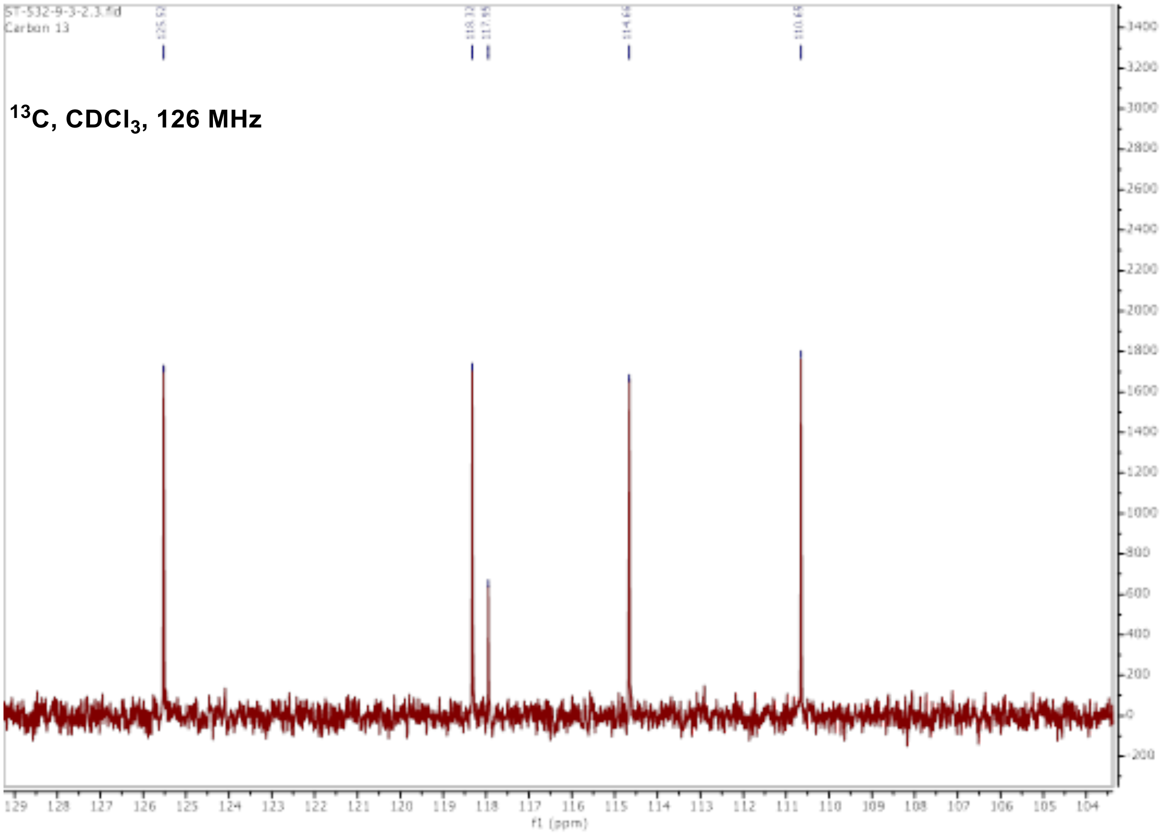

ST-532-7-iso.2.fid
Carbon 13
13C, CDCl3, 126 MHz
169.57
77.20 CDCl3
68.75
50.79
36.70
35.08
32.14
27.32
26.47
25.46
21.85
21.78
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

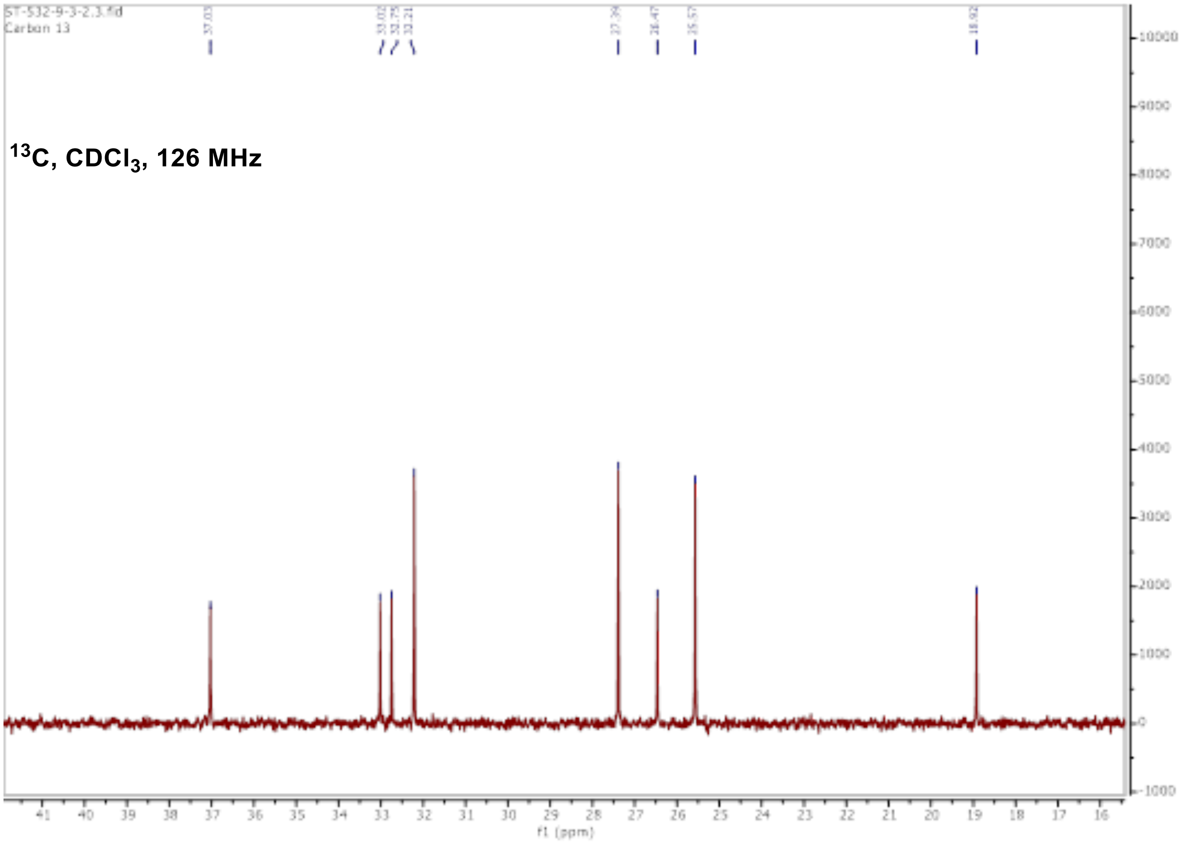

ST-545-2-iso.1.fid
Proton
35
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
2.37
2.36
2.34
1.65
1.63
1.58
1.57
1.55
1.54
1.47
1.45
1.41
1.30
1.29
1.27
2.00
15.03
2.16
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

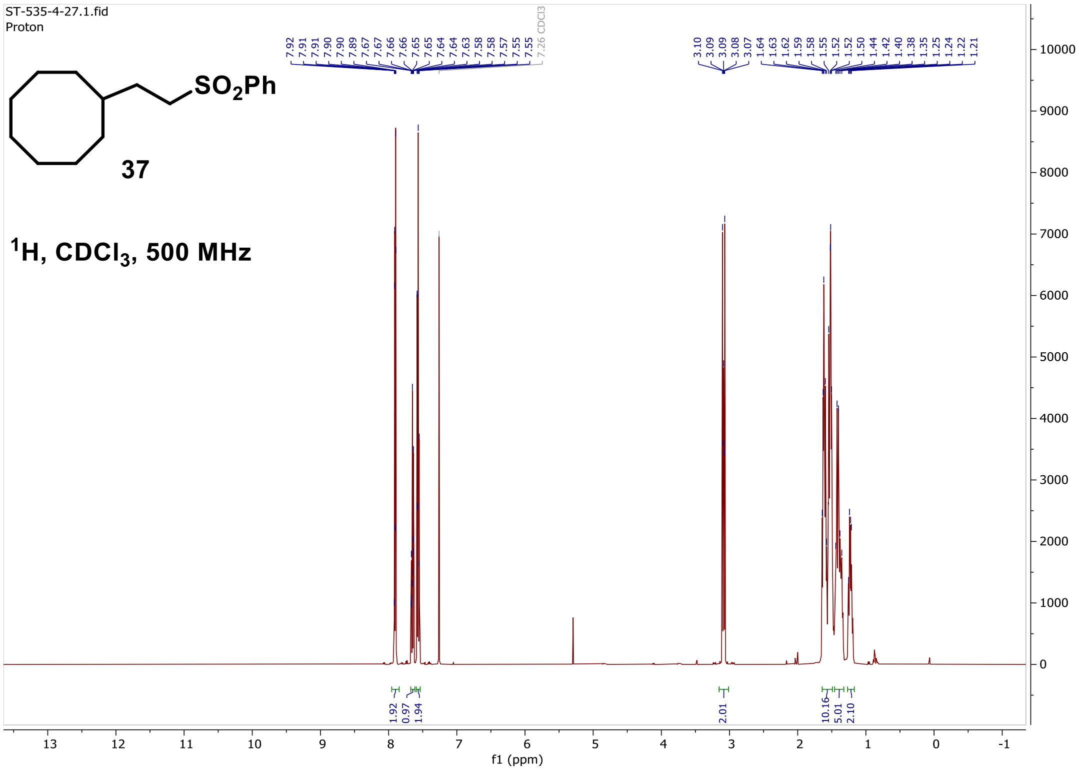

ST-545-2-iso.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
180.45
77.20 CDCl3
36.96
32.88
32.37
32.13
27.41
26.43
25.52
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-400
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

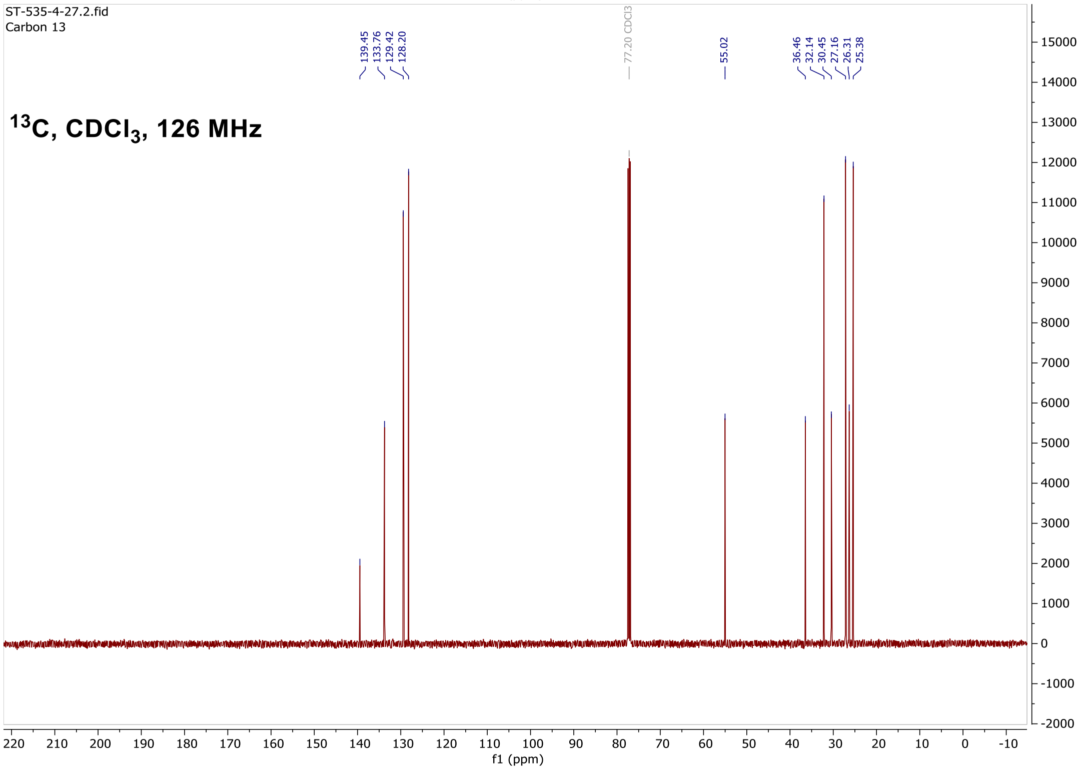

ST-532-9-3-2.1.fid
Proton
7.61
7.60
7.26 CDCl3
7.10
7.10
7.09
7.08
7.07
7.06
7.05
6.27
6.27
6.27
6.27
2.62
2.60
2.59
2.44
2.43
1.71
1.70
1.68
1.67
1.50
1.48
1.45
1.34
1.33
1.26
Me
O
O
O
O
36
1H, CDCl3, 500 MHz
0.99
0.92
0.97
0.93
2.00
2.98
7.96
5.18
4.27
30000
25000
20000
15000
10000
5000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

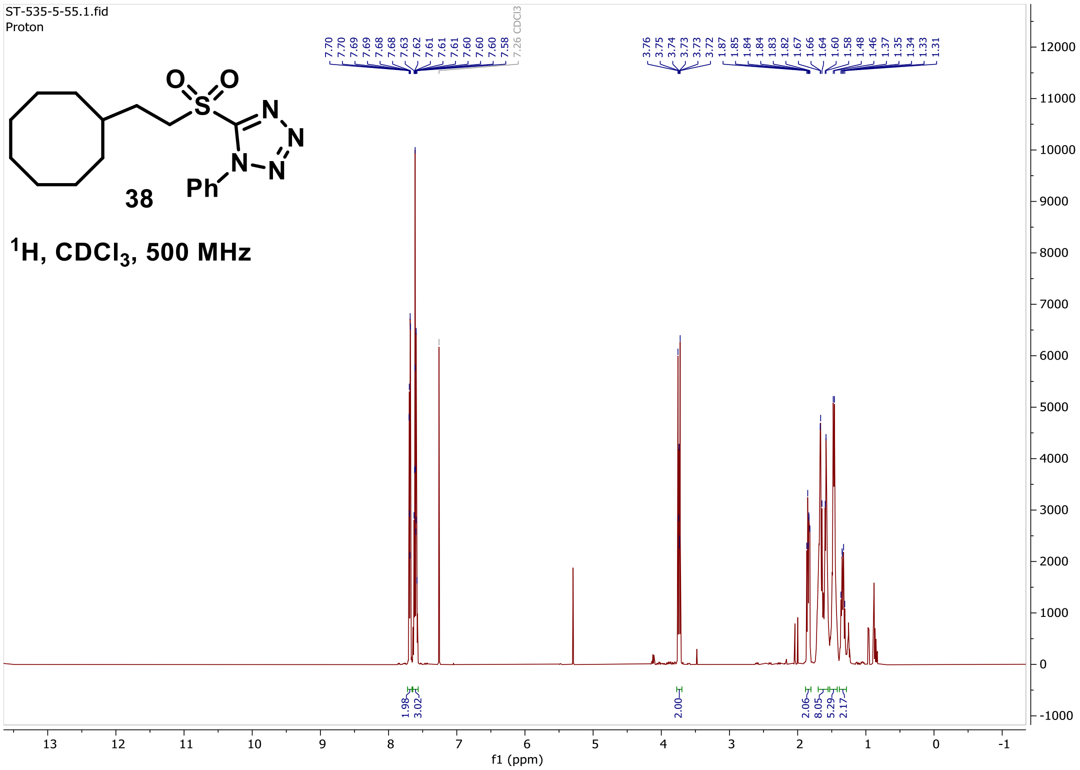

ST-532-9-3-2.3.fid
Carbon 13
172.15
160.75
154.39
153.41
152.12
125.52
118.32
117.95
114.66
110.65
77.20 CDCl3
37.03
33.02
32.75
32.21
27.39
26.47
25.57
18.92
13C, CDCl3, 126 MHz
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

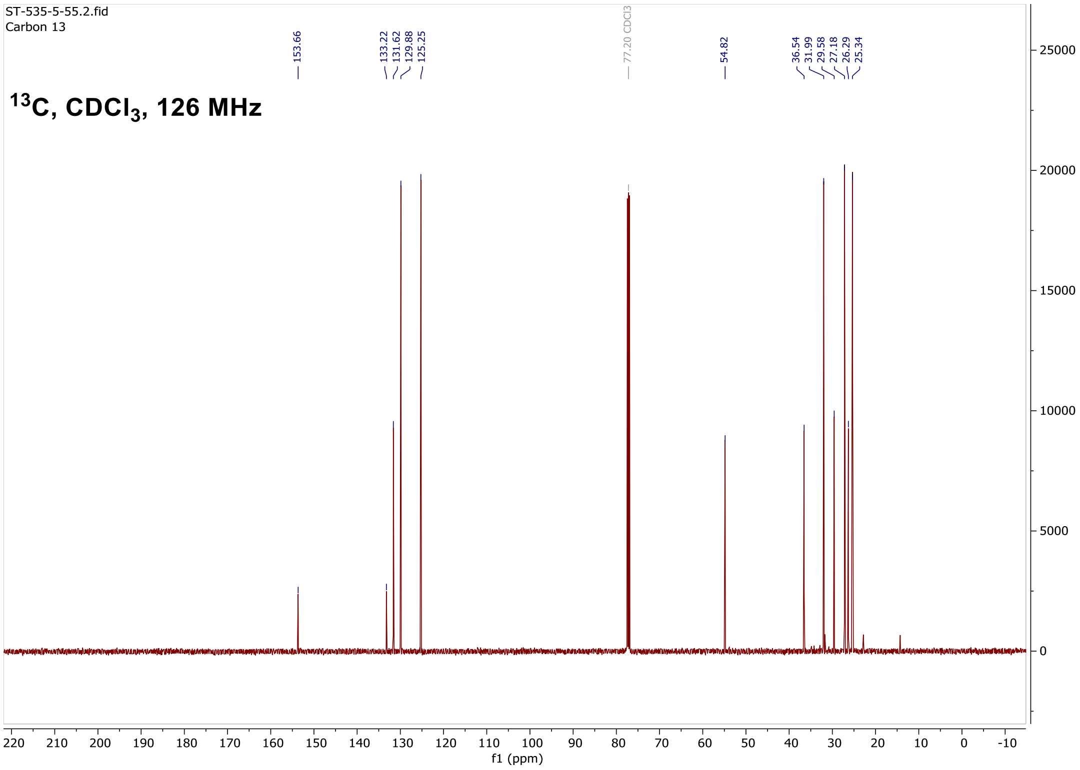

ST-532-9-3-2.3.fid
Carbon 13
125.52
118.32
117.95
114.69
110.69
^{13}C, CDCl$_3$, 126 MHz
f1 (ppm)

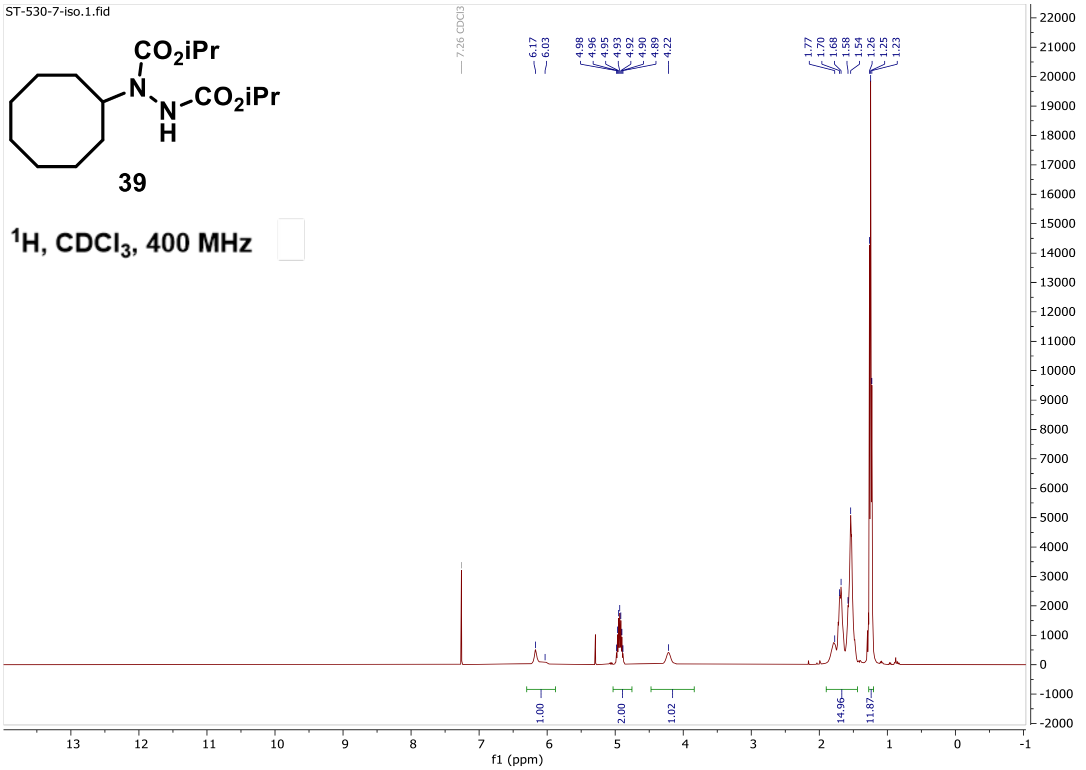

ST-532-9-3-2.3.fid
Carbon 13
37.03
33.02
32.75
32.21
27.39
26.47
25.57
18.92
^{13}C, CDCl$_3$, 126 MHz
f1 (ppm)

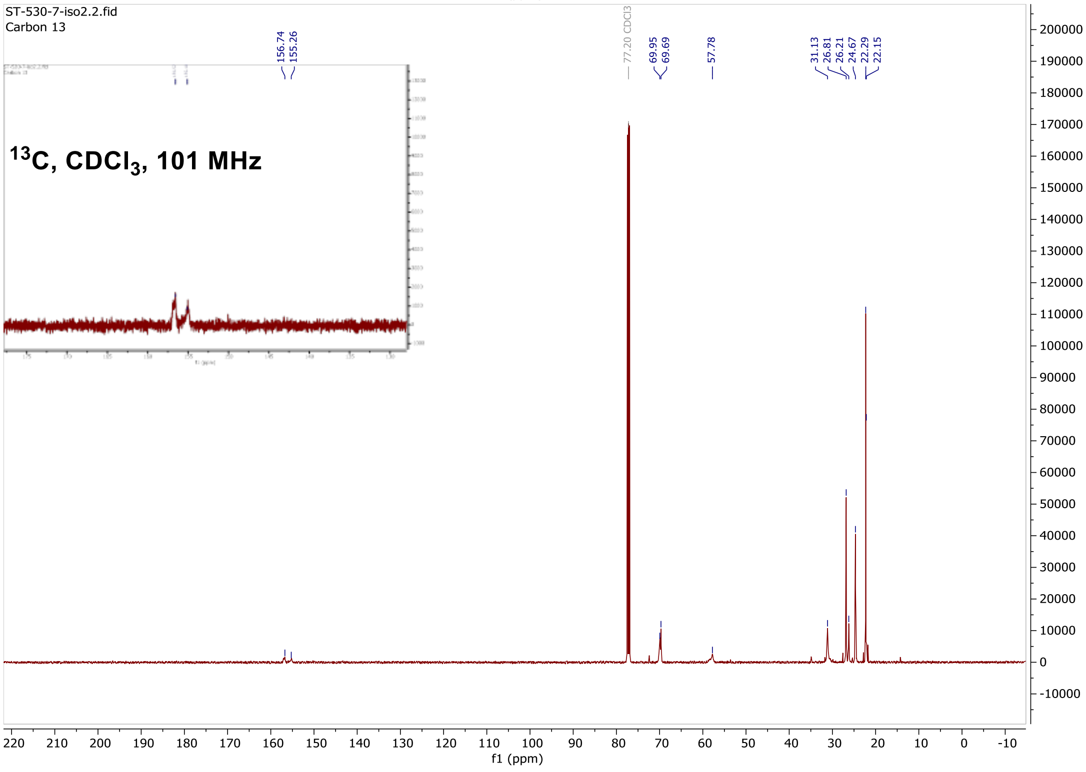

ST-535-4-27.1.fid
Proton
SO$_2$Ph
37
^{1}H, CDCl$_3$, 500 MHz
7.92 7.91 7.91 7.90 7.90 7.89 7.67 7.66 7.66 7.65 7.65 7.64 7.64 7.63 7.58 7.58 7.57 7.55 7.55
7.26 CDCl3
3.10 3.09 3.09 3.08 3.07
1.64 1.63 1.62 1.59 1.58 1.55 1.52 1.52 1.50 1.44 1.42 1.40 1.38 1.35 1.25 1.24 1.22 1.21
1.92 0.97 1.94
2.01
10.16 5.01 2.10
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)
10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0

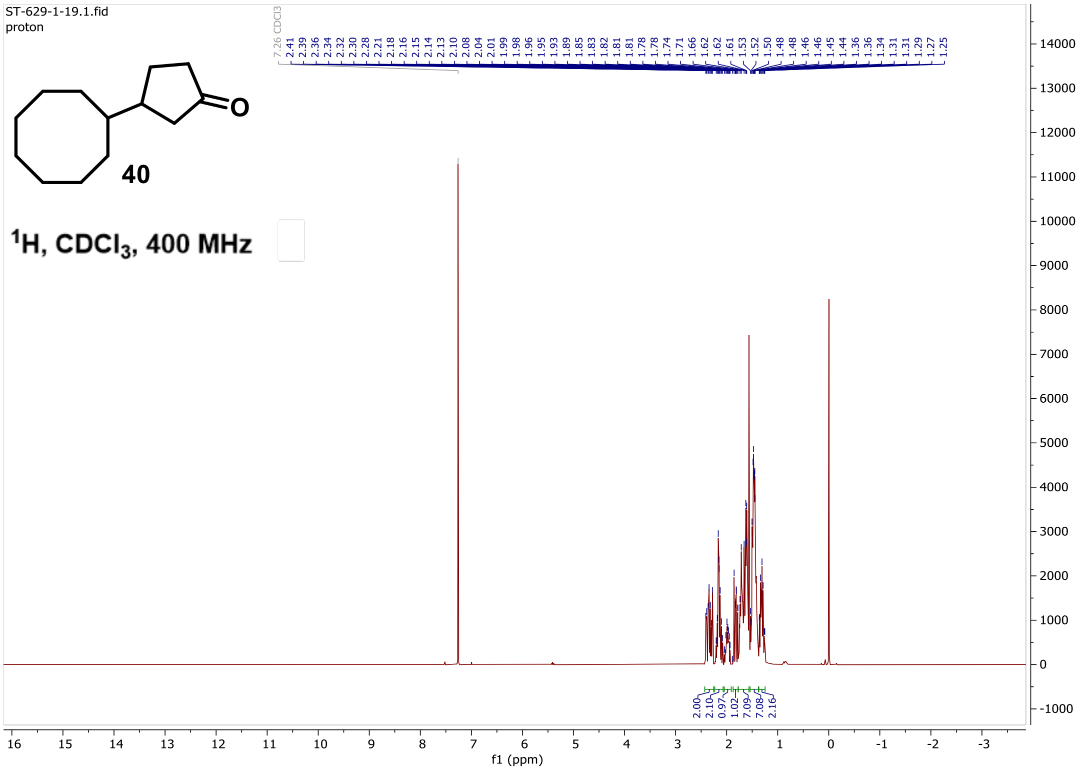

ST-535-4-27.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
139.45 133.76 129.42 128.20
77.20 CDCl3
55.02
36.46 32.14 30.45 27.16 26.31 25.38
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000

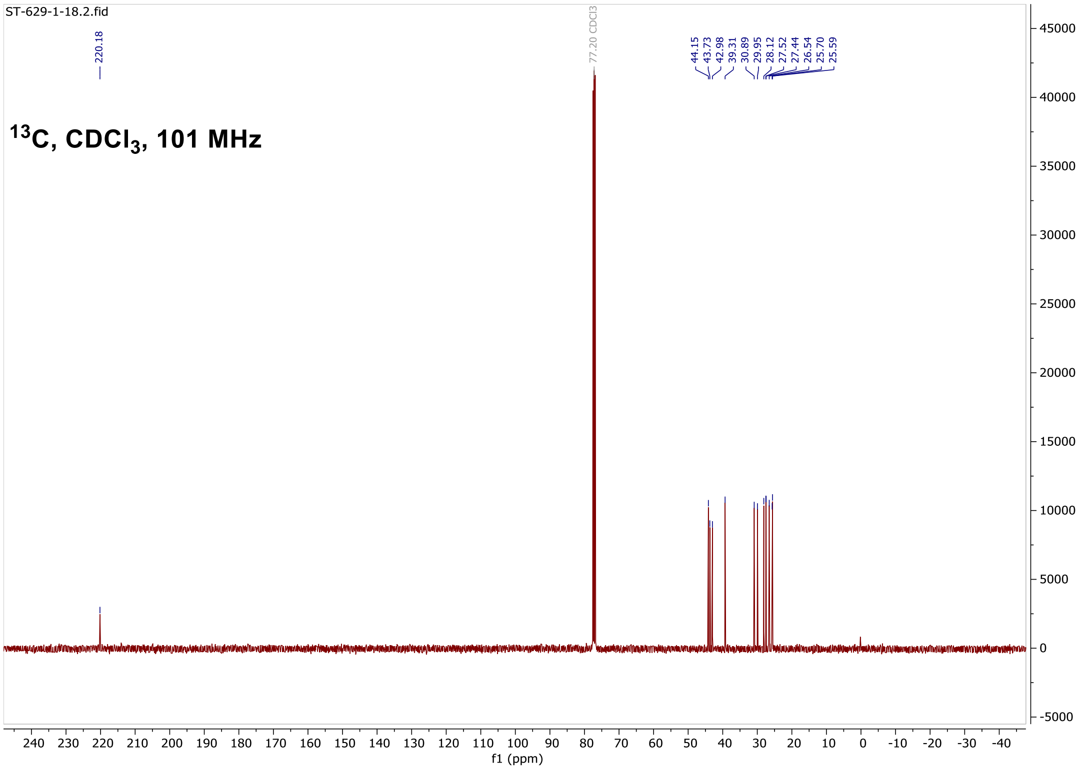

ST-535-5-55.1.fid
Proton
38
Ph
^{1}H, CDCl$_3$, 500 MHz
7.70 7.70 7.69 7.69 7.68 7.68 7.63 7.62 7.61 7.61 7.61 7.60 7.60 7.60 7.58
7.26 CDCl3
3.76 3.75 3.74 3.73 3.73 3.72
1.87 1.85 1.84 1.84 1.83 1.82 1.67 1.66 1.64 1.60 1.58 1.48 1.46 1.37 1.35 1.34 1.33 1.31
1.98
3.02
2.00
2.06
8.05
5.29
2.17
f1 (ppm)

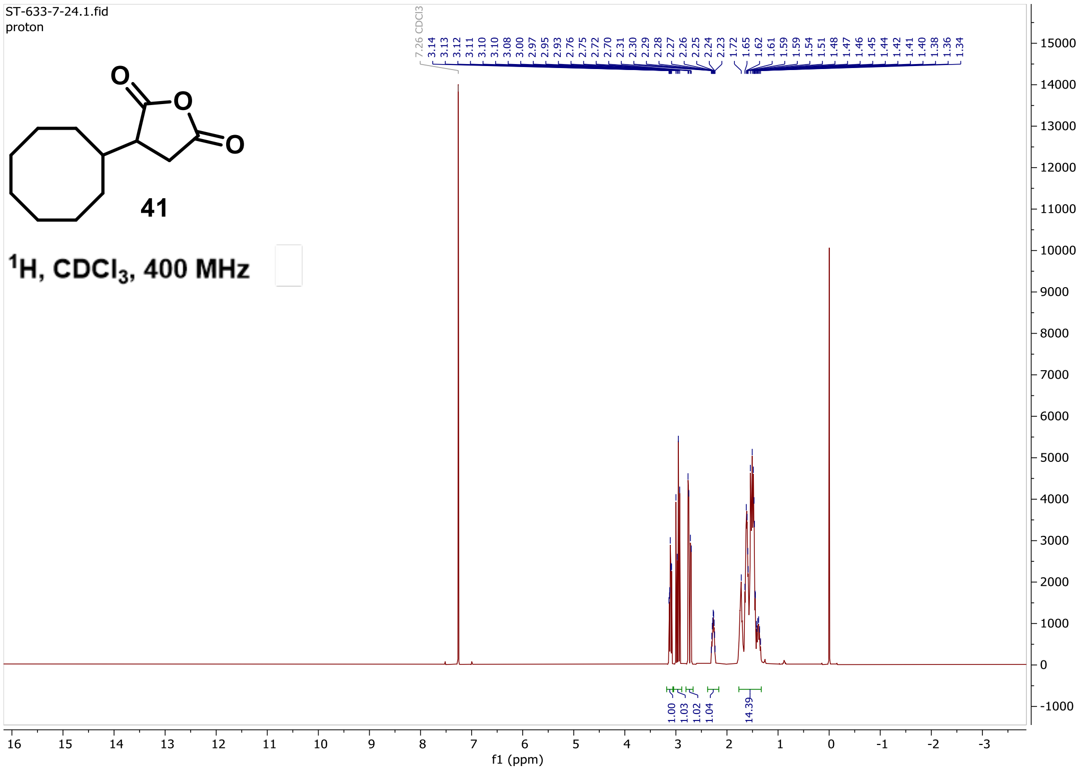

ST-535-5-55.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
153.66
133.22 131.62 129.88 125.25
77.20 CDCl3
54.82
36.54 31.99 29.58 27.18 26.29 25.34
f1 (ppm)

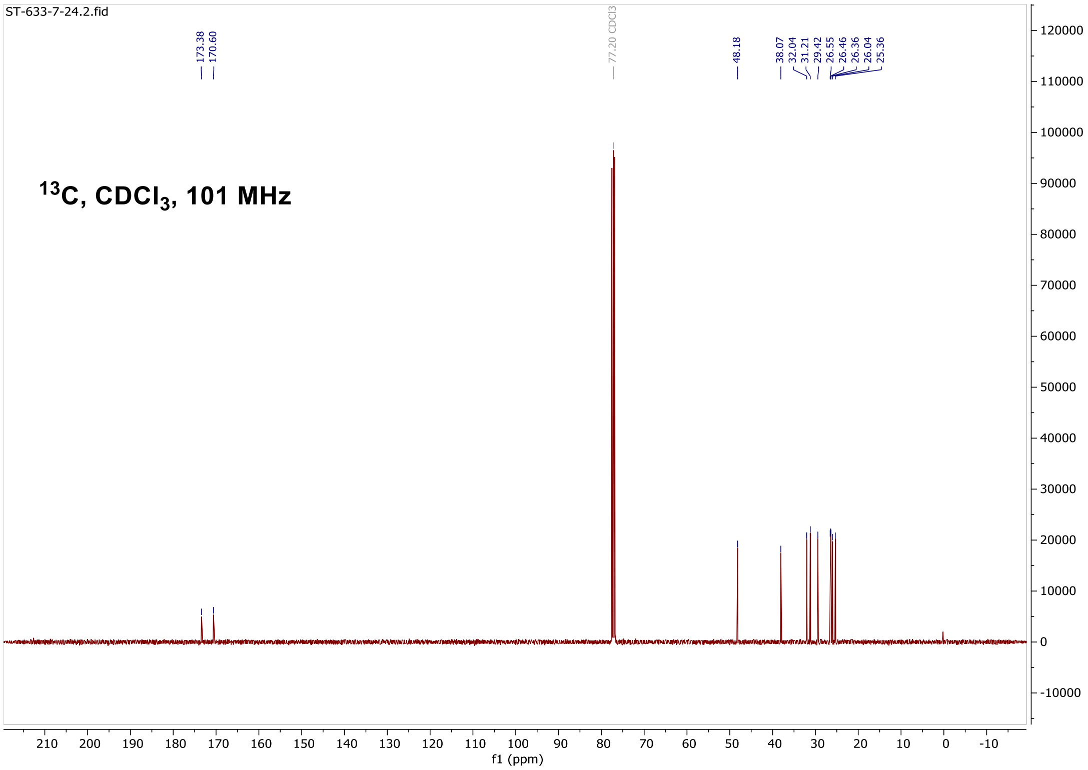

ST-530-7-iso.1.fid
CO2iPr
N
N
H
CO2iPr
39
1H, CDCl3, 400 MHz
7.26 CDCl3
6.17
6.03
4.98
4.96
4.95
4.93
4.92
4.90
4.89
4.22
1.77
1.70
1.68
1.58
1.54
1.26
1.25
1.23
1.00
2.00
1.02
14.96
11.87
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

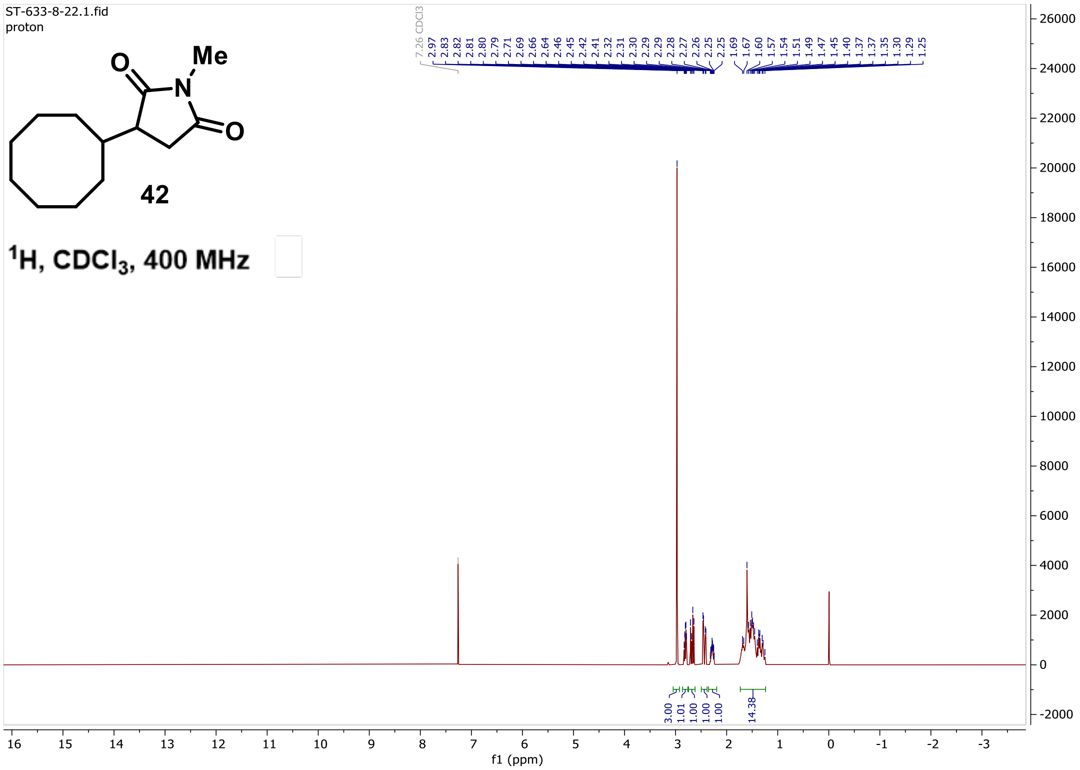

ST-530-7-iso2.2.fid
Carbon 13
156.74
155.26
77.20 CDCl3
69.95
69.69
57.78
31.13
26.81
26.21
24.67
22.29
22.15
13C, CDCl3, 101 MHz
200000
190000
180000
170000
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

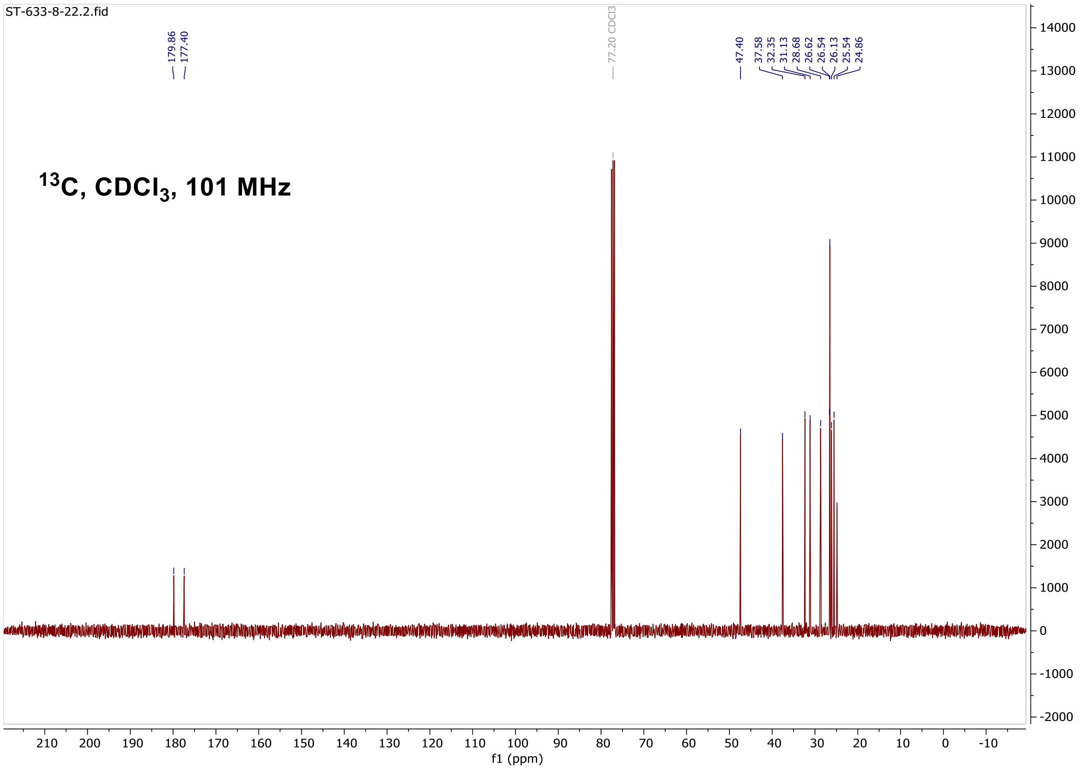

ST-629-1-19.1.fid
proton
7.26 CDCl3
2.41 2.39 2.36 2.34 2.32 2.30 2.28 2.21 2.18 2.16 2.15 2.14 2.13 2.10 2.08 2.04 2.01 1.99 1.98 1.96 1.95 1.93 1.89 1.85 1.83 1.82 1.81 1.81 1.78 1.78 1.74 1.71 1.66 1.62 1.62 1.61 1.53 1.52 1.50 1.48 1.48 1.46 1.45 1.44 1.36 1.36 1.34 1.31 1.31 1.29 1.27 1.25
O
40
^{1}H, CDCl$_3$, 400 MHz
2.00 2.10 0.97 1.02 7.09 7.08 2.16
14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

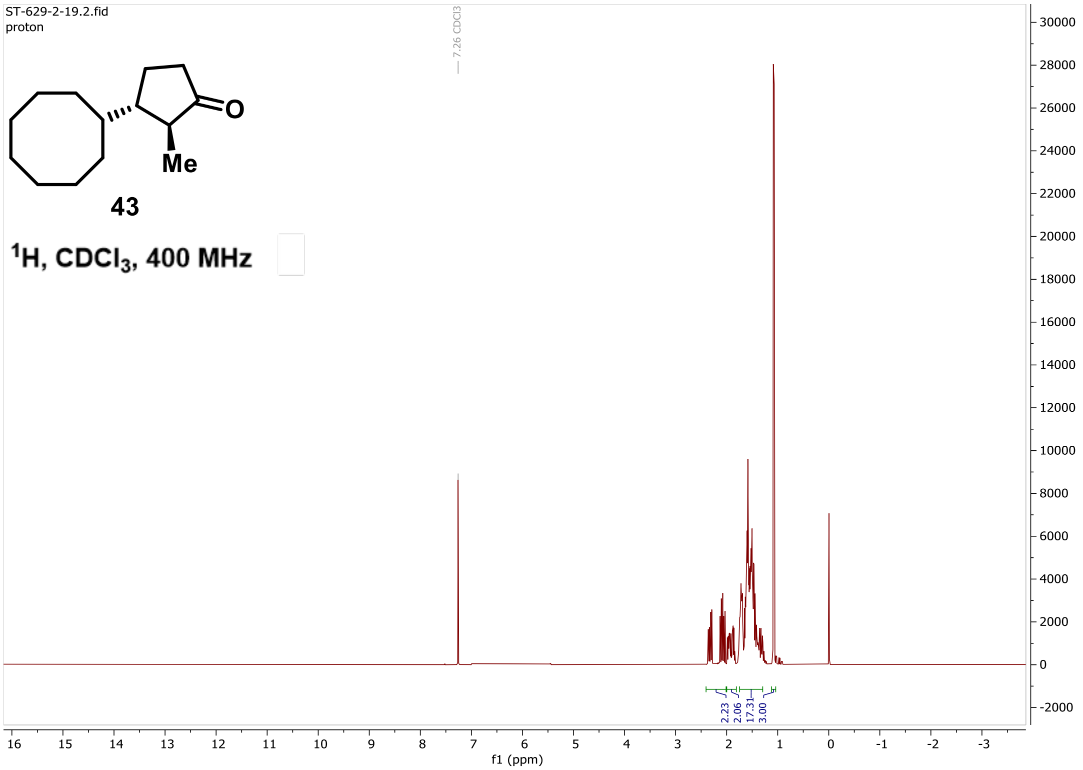

ST-629-1-18.2.fid
220.18
77.20 CDCl3
44.15 43.73 42.98 39.31 30.89 29.95 28.12 27.52 27.44 26.54 25.70 25.59
^{13}C, CDCl$_3$, 101 MHz
45000 40000 35000 30000 25000 20000 15000 10000 5000 0 -5000
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

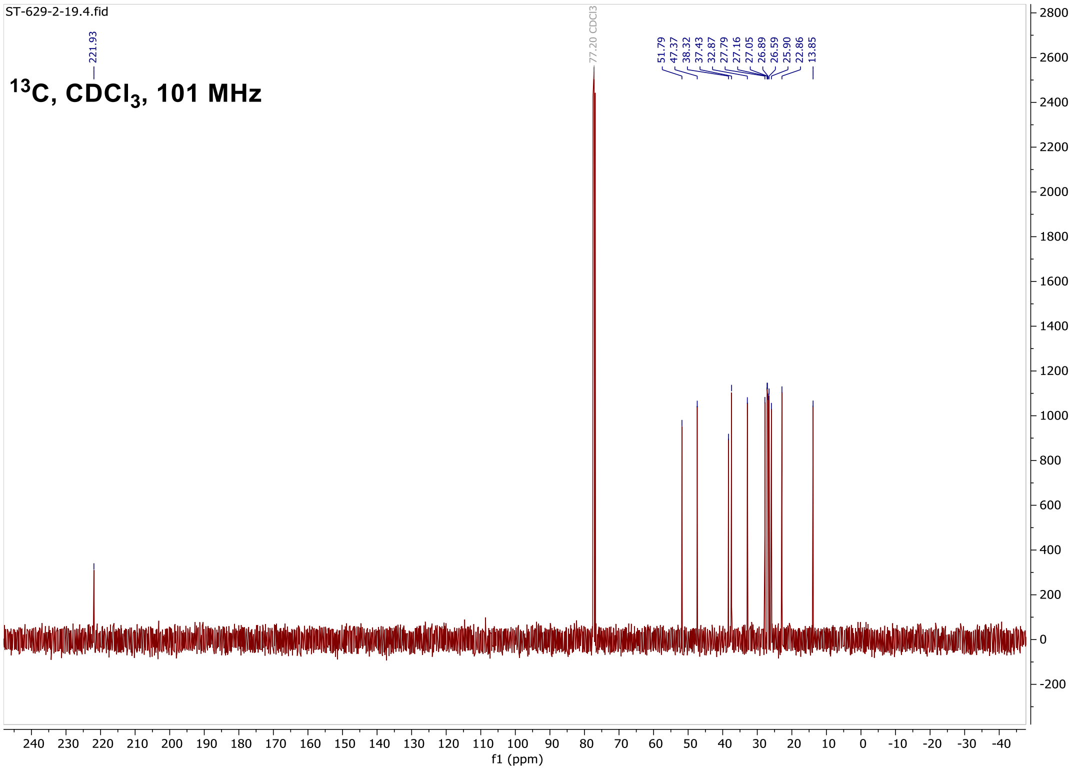

ST-633-7-24.1.fid
proton
41
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
3.14
3.13
3.12
3.11
3.10
3.10
3.08
3.00
2.97
2.95
2.93
2.76
2.75
2.72
2.70
2.31
2.30
2.29
2.28
2.27
2.26
2.25
2.24
2.23
1.72
1.65
1.62
1.61
1.59
1.59
1.54
1.51
1.48
1.47
1.46
1.45
1.44
1.42
1.41
1.40
1.38
1.36
1.34
1.00
1.03
1.02
1.04
14.39
f1 (ppm)

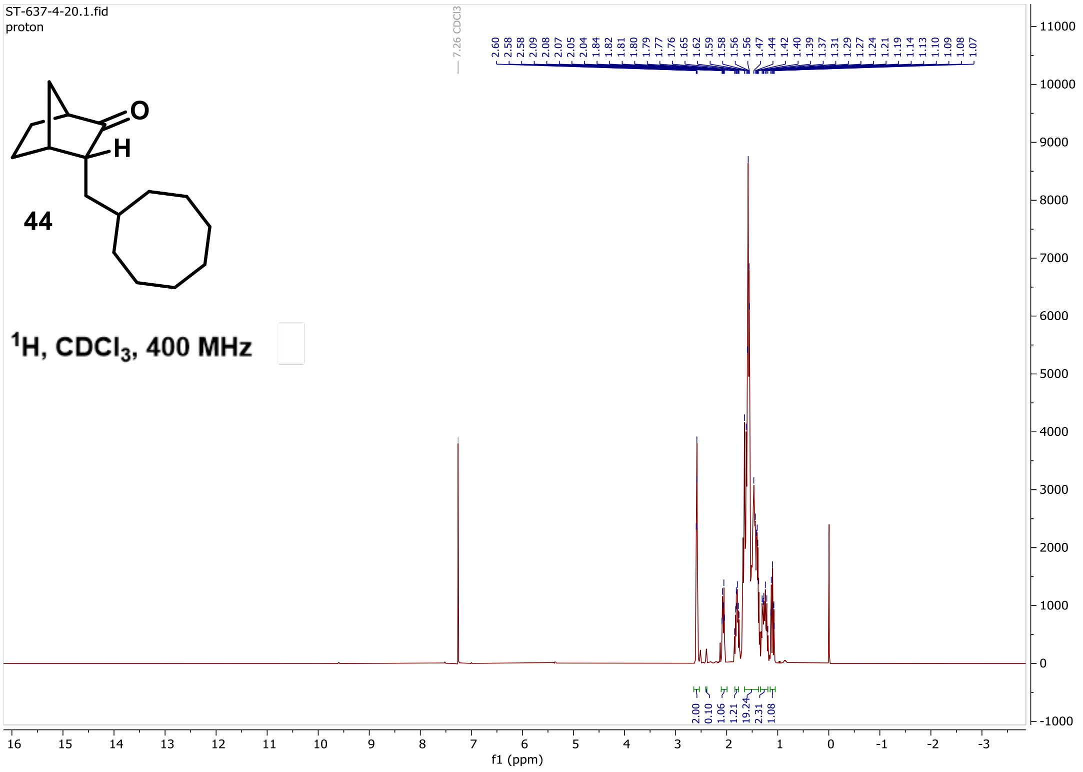

ST-633-7-24.2.fid
^{13}C, CDCl$_3$, 101 MHz
173.38
170.60
77.20 CDCl3
48.18
38.07
32.04
31.21
29.42
26.55
26.46
26.36
26.04
25.36
f1 (ppm)

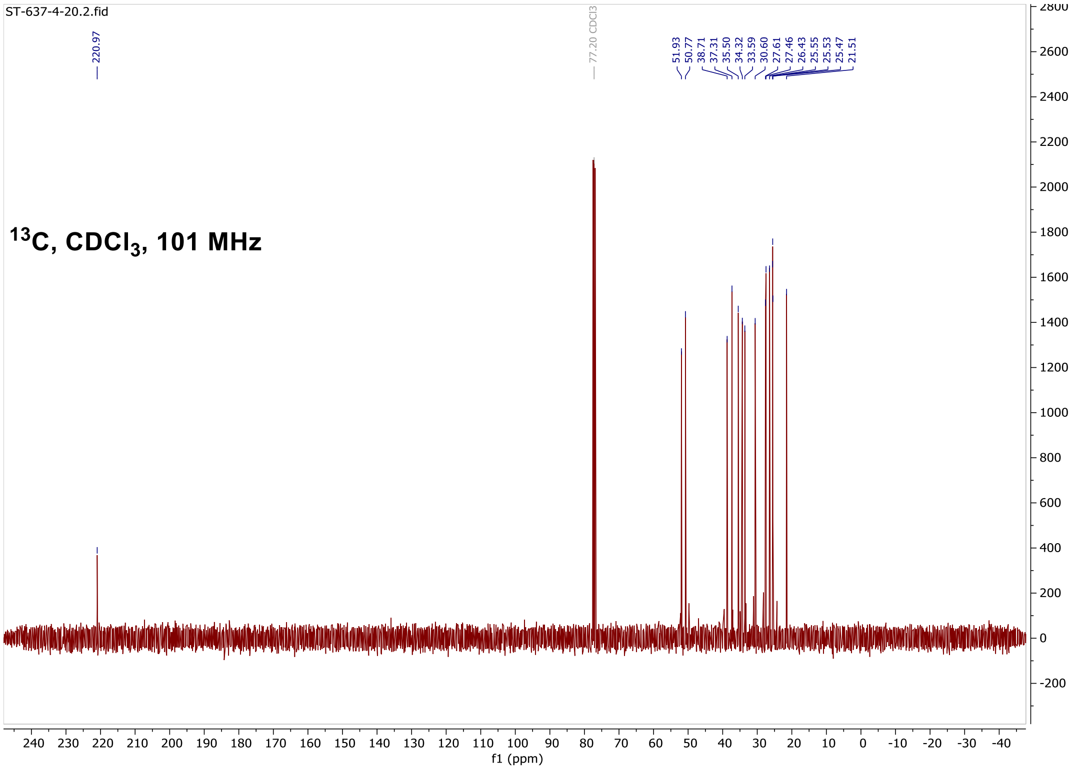

ST-633-8-22.1.fid
proton
O
N
Me
O
42
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
2.97
2.83
2.82
2.81
2.80
2.79
2.71
2.69
2.66
2.64
2.46
2.45
2.42
2.41
2.32
2.31
2.30
2.29
2.29
2.28
2.27
2.26
2.25
2.25
1.69
1.67
1.60
1.57
1.54
1.51
1.49
1.47
1.45
1.40
1.37
1.37
1.35
1.30
1.29
1.25
3.00
1.01
1.00
1.00
1.00
14.38
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

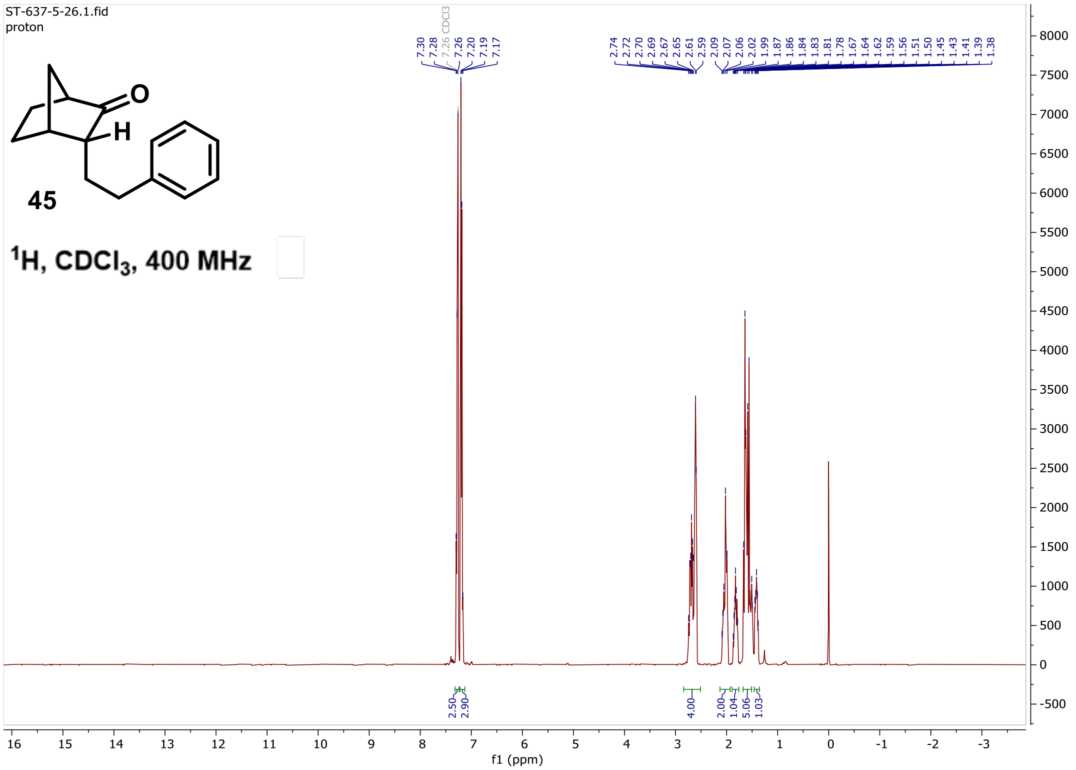

ST-633-8-22.2.fid
^{13}C, CDCl$_3$, 101 MHz
179.86
177.40
77.20 CDCl3
47.40
37.58
32.35
31.13
28.68
26.62
26.54
26.13
25.54
24.86
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

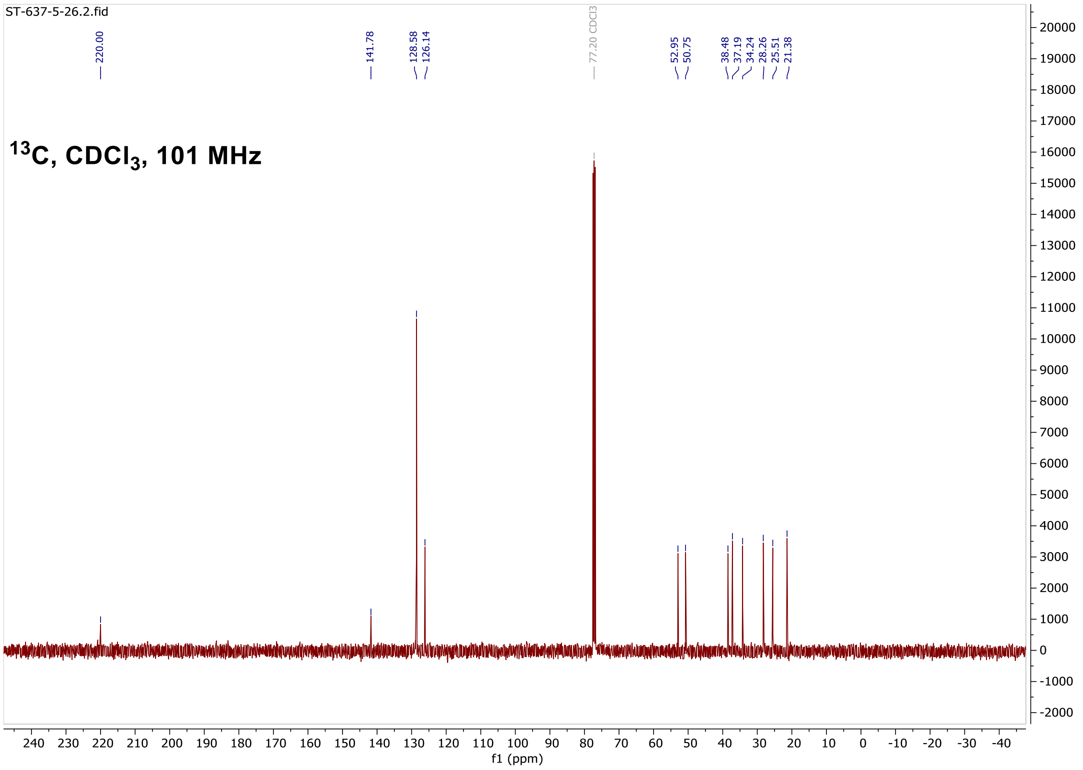

ST-629-2-19.2.fid
proton
43
^{1}H, CDCl$_3$, 400 MHz
Me
O
7.26 CDCl3
2.23
2.06
17.31
3.00
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

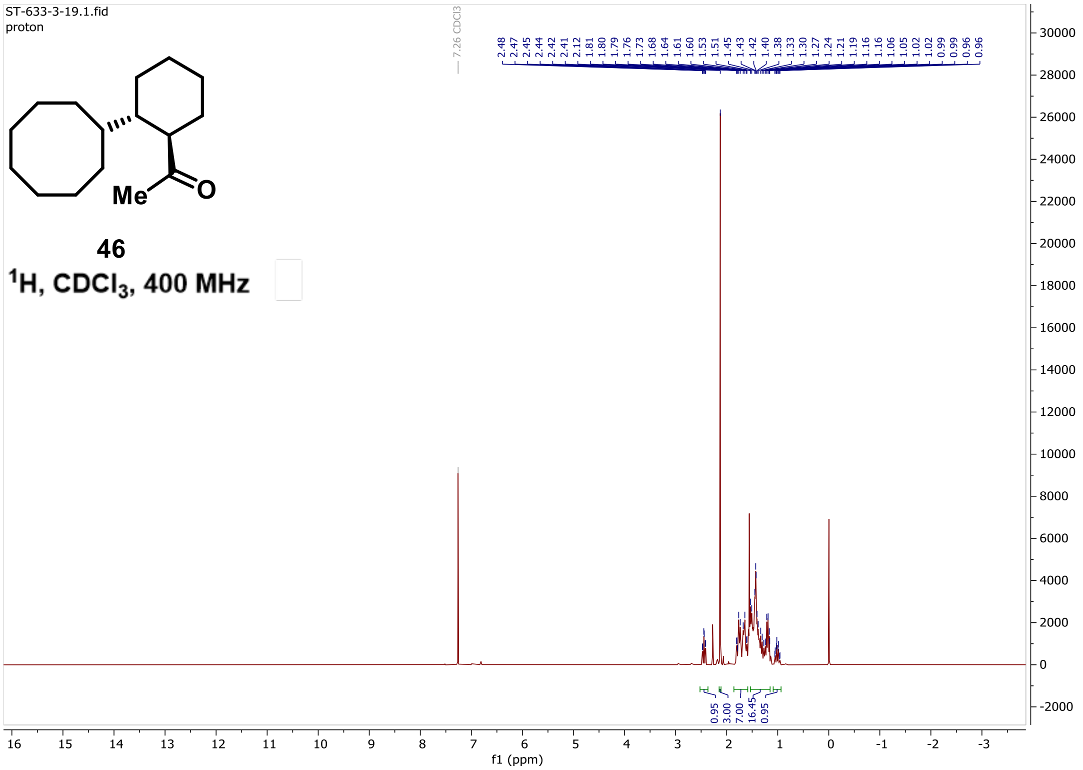

ST-629-2-19.4.fid
^{13}C, CDCl$_3$, 101 MHz
221.93
77.20 CDCl3
51.79
47.37
38.32
37.43
32.87
27.79
27.16
27.05
26.89
26.59
25.90
22.86
13.85
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

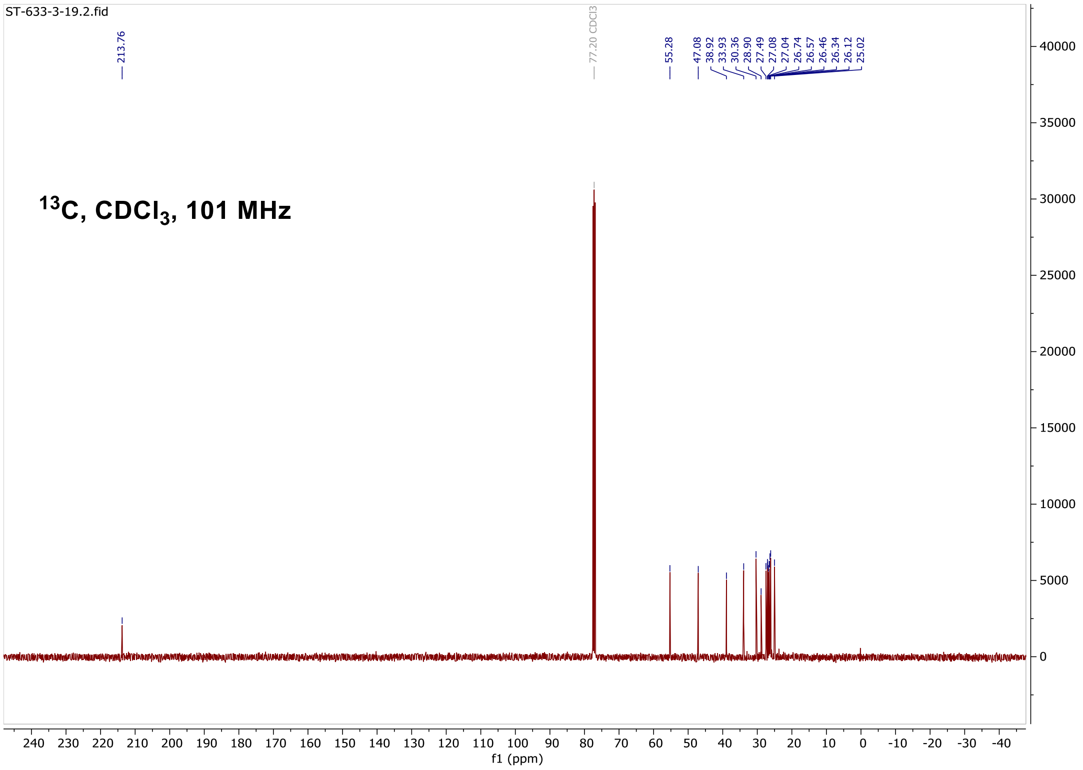

ST-637-4-20.1.fid
proton
7.26 CDCl3
2.60
2.58
2.58
2.09
2.08
2.07
2.05
2.04
1.84
1.82
1.81
1.80
1.79
1.77
1.76
1.65
1.62
1.59
1.58
1.56
1.56
1.47
1.44
1.42
1.40
1.39
1.37
1.31
1.29
1.27
1.24
1.21
1.19
1.14
1.13
1.10
1.09
1.08
1.07
O
H
44
^{1}H, CDCl$_3$, 400 MHz
2.00
0.10
1.06
1.21
19.24
2.31
1.08
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

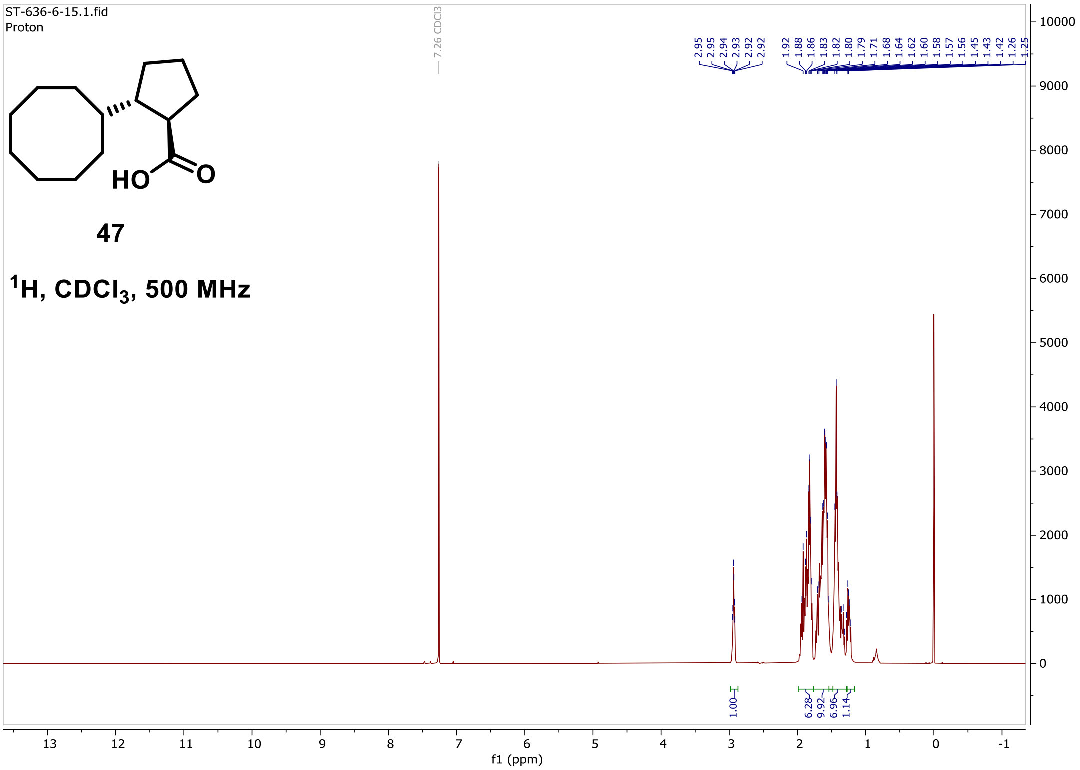

ST-637-4-20.2.fid
220.97
77.20 CDCl3
51.93
50.77
38.71
37.31
35.50
34.32
33.59
30.60
27.61
27.46
26.43
25.55
25.53
25.47
21.51
^{13}C, CDCl$_3$, 101 MHz
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
f1 (ppm)

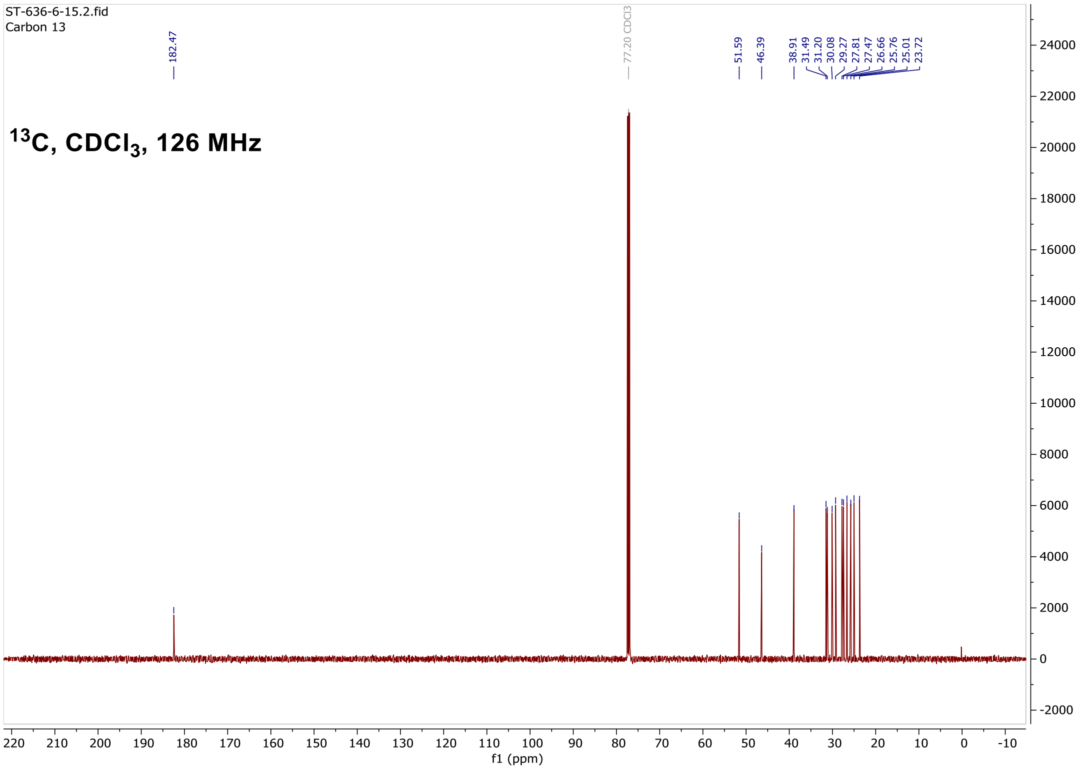

ST-637-5-26.1.fid
proton
45
^{1}H, CDCl$_3$, 400 MHz
7.30
7.28
7.26 CDCl3
7.26
7.20
7.19
7.17
2.74
2.72
2.70
2.69
2.67
2.65
2.61
2.59
2.09
2.07
2.06
2.02
1.99
1.87
1.86
1.84
1.83
1.81
1.78
1.67
1.64
1.62
1.59
1.56
1.51
1.50
1.45
1.43
1.41
1.39
1.38
2.50
2.90
4.00
2.00
1.04
5.06
1.03
f1 (ppm)
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
8000 7500 7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

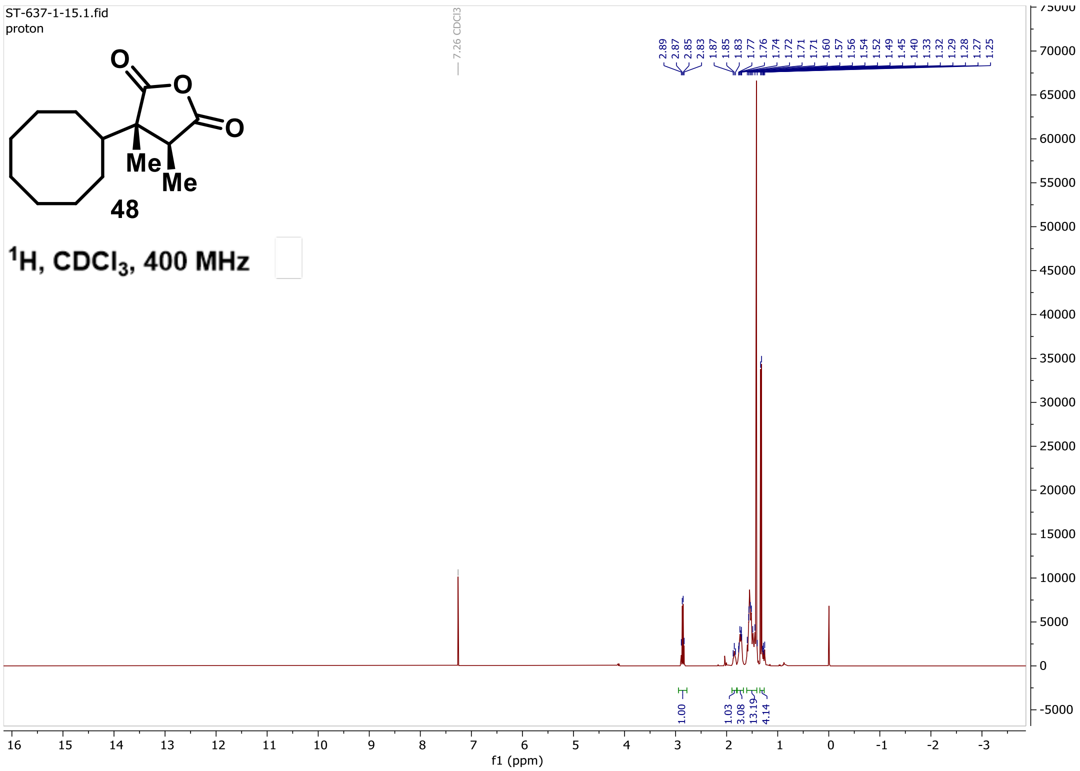

ST-637-5-26.2.fid
^{13}C, CDCl$_3$, 101 MHz
220.00
141.78
128.58
126.14
77.20 CDCl3
52.95
50.75
38.48
37.19
34.24
28.26
25.51
21.38
f1 (ppm)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000

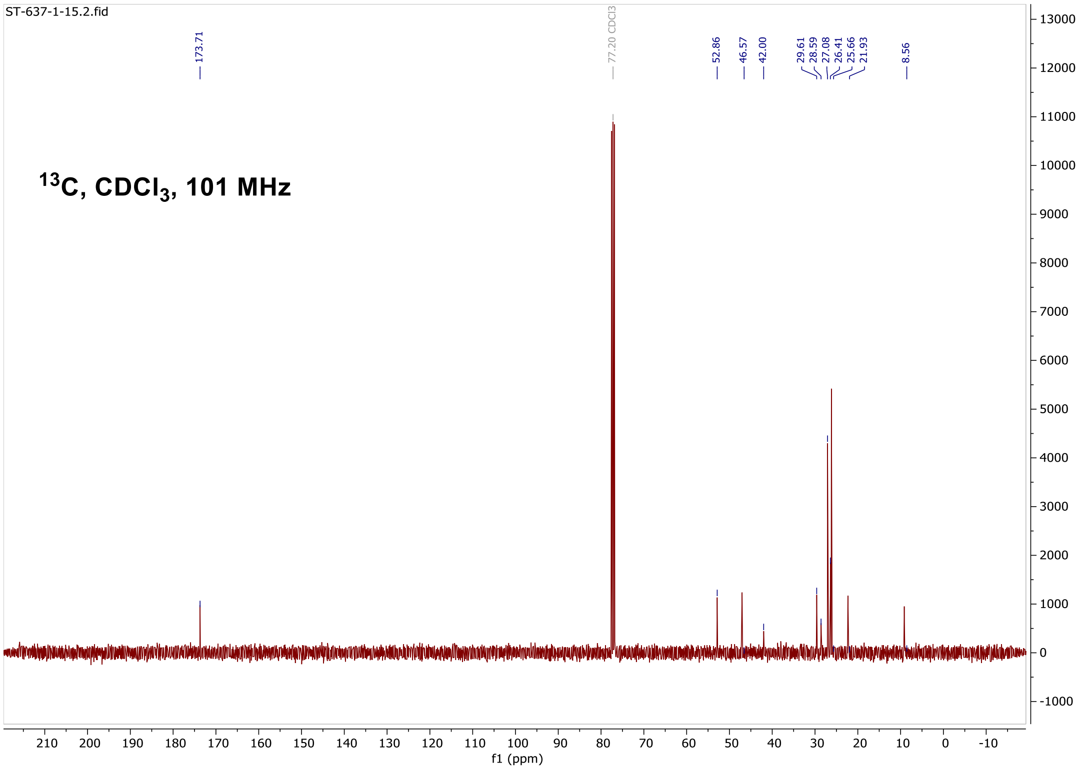

ST-633-3-19.1.fid
proton
7.26 CDCl3
2.48
2.47
2.45
2.44
2.42
2.41
2.12
1.81
1.80
1.79
1.76
1.73
1.68
1.64
1.61
1.60
1.53
1.51
1.45
1.43
1.42
1.40
1.38
1.33
1.30
1.27
1.24
1.21
1.19
1.16
1.16
1.06
1.05
1.02
1.02
0.99
0.99
0.96
0.96
Me
O
46
^{1}H, CDCl$_3$, 400 MHz
0.95
3.00
7.00
16.45
0.95
f1 (ppm)

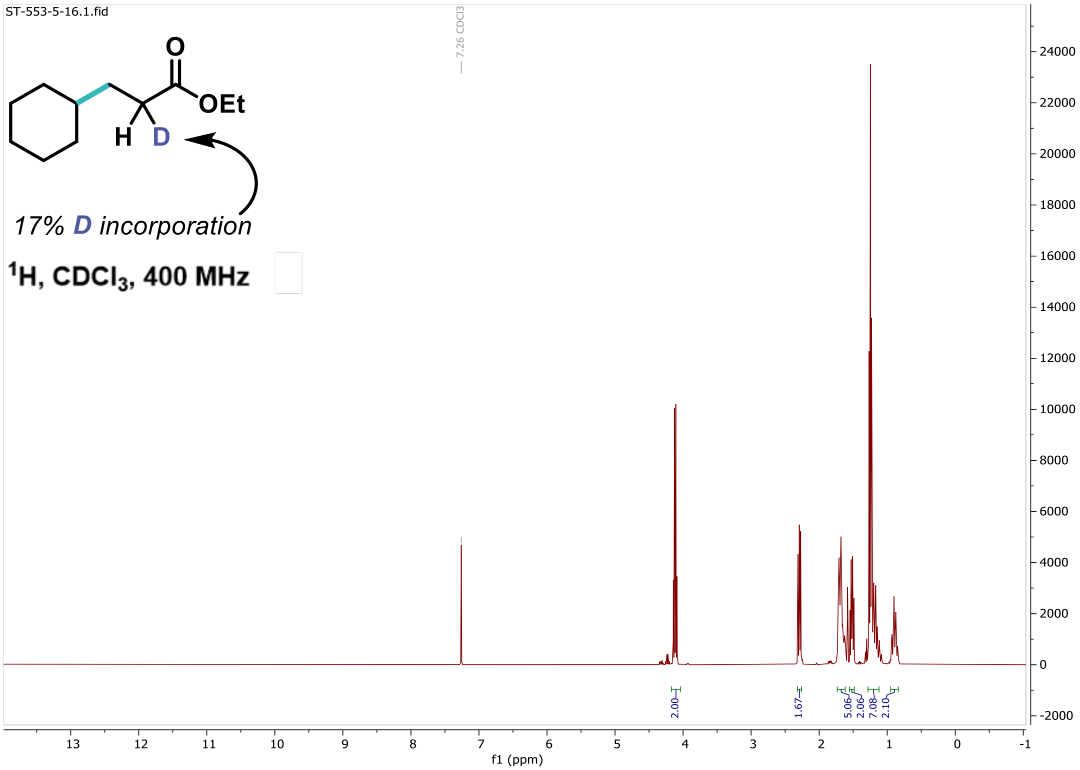

ST-633-3-19.2.fid
213.76
77.20 CDCl3
55.28
47.08
38.92
33.93
30.36
28.90
27.49
27.08
27.04
26.74
26.57
26.46
26.34
26.12
25.02
^{13}C, CDCl$_3$, 101 MHz
f1 (ppm)

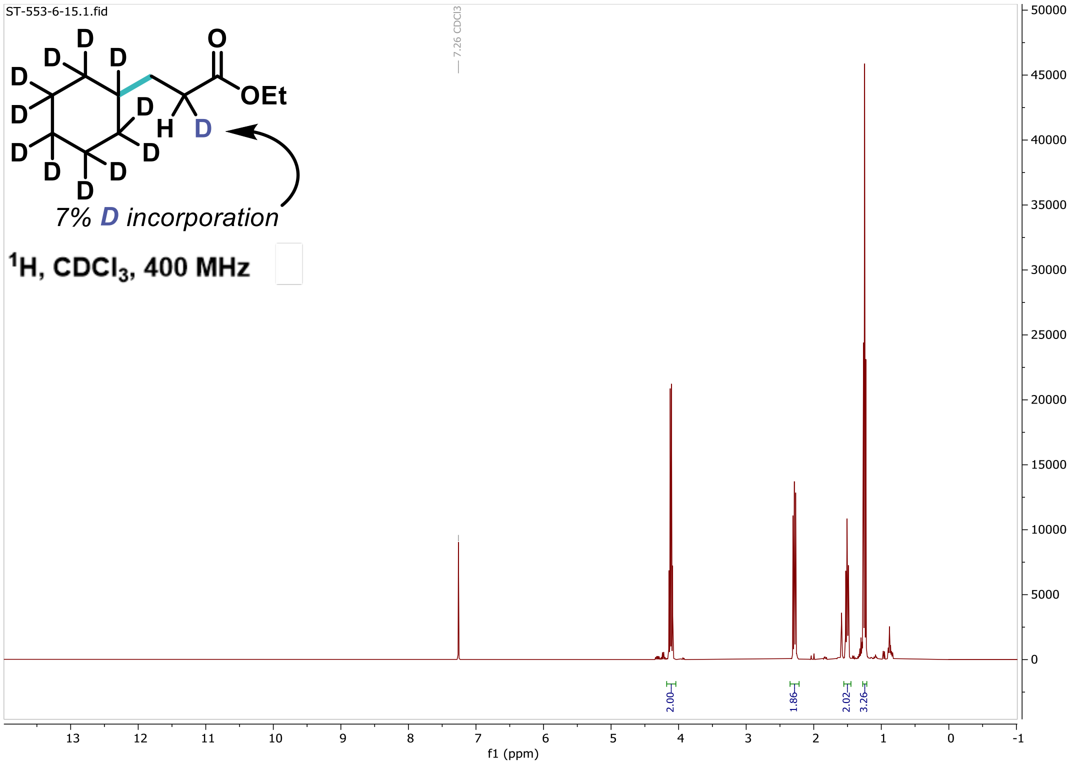

ST-636-6-15.1.fid
Proton
HO
O
47
^{1}H, CDCl$_3$, 500 MHz
7.26 CDCl3
2.95
2.95
2.94
2.93
2.92
2.92
1.92
1.88
1.86
1.83
1.82
1.80
1.79
1.71
1.68
1.64
1.62
1.60
1.58
1.57
1.56
1.45
1.43
1.42
1.26
1.25
1.00
6.28
9.92
6.96
1.14
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-636-6-15.2.fid
Carbon 13
^{13}C, CDCl$_3$, 126 MHz
182.47
77.20 CDCl3
51.59
46.39
38.91
31.49
31.20
30.08
29.27
27.81
27.47
26.66
25.76
25.01
23.72
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


ST-637-1-15.1.fid
proton
48
^{1}H, CDCl$_3$, 400 MHz
7.26 CDCl3
2.89
2.87
2.85
2.83
1.87
1.85
1.83
1.77
1.76
1.74
1.72
1.71
1.71
1.60
1.57
1.56
1.54
1.52
1.49
1.45
1.40
1.33
1.32
1.29
1.28
1.27
1.25
1.00
1.03
3.08
13.19
4.14
f1 (ppm)


ST-637-1-15.2.fid
^{13}C, CDCl$_3$, 101 MHz
173.71
77.20 CDCl3
52.86
46.57
42.00
29.61
28.59
27.08
26.41
25.66
21.93
8.56
f1 (ppm)


ST-553-5-16.1.fid
O
OEt
H
D
17% D incorporation
¹H, CDCl₃, 400 MHz
7.26 CDCl3
2.00
1.67
5.06
2.06
7.08
2.10
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


ST-553-6-15.1.fid
D
O
OEt
H
D
7% D incorporation
¹H, CDCl₃, 400 MHz
7.26 CDCl3
2.00
1.86
2.02
3.26
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)